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Paper:

CFD Analysis of Friction-Reduction Effect of Micro-Textured Surfaces in Lubricant

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This paper investigates the effects of geometrical features of tailored textures on friction reduction of a surface with a lubricant-film thickness of several to several-tenths micrometers, using the Navier-Stokes equations and the orthogonal experimental design. The results indicate that the surface textured with the selected sawtooth riblets in lubricant can have up to 93.83% less friction than an untextured surface. The thickness of the lubricant film plays the most important role in friction reduction; the height and the ridge angle of the riblets are the secondary factors. The results and principles obtained can potentially be used in the designing of low-friction surfaces in precision machines with transmission parts.

Keywords: friction-reduction, micro-textured surface, lubrication, orthogonal experimental design

1. Introduction

Adding lubrication is an efficient way to reduce friction and wear between sliding friction pairs. It has been determined that micro-textured surfaces are able to affect the load-carrying capacity of parallel sliding transmission parts [1–3]. Because research on the friction of transmission parts is also needed in many fields [4], some researchers have tried to improve the lubrication performance of parts, such as bearings [5,6] and rod seals [7], by designing and fabricating textures on sliding surfaces. Kawasegi et al. [8] have proposed milling tools with micro- and nanometer-scale textures to improve the frictional characteristics and resulting machining performance. Many experimental studies concerning the friction of textured surface have been carried out [9,10]. Greidanus et al. [9] have successfully decreased friction in the Taylor-Couette flow by pasting a vinyl film with sawtooth riblets on the surface of a rotator. A maximum friction reduction of 5.3% was found at the Reynolds number of 4.7×10^4 , which corresponds to the riblet spacing Reynolds number $s^+ = 14$. Ramesh et al. [10] have machined micro-dimples on stainless steel to obtain a tex-

tured surface that exhibits friction as much as 80% lower than that of an untextured surface.

With the development of computational science, more and more researchers have done theoretical investigations into the effects of geometrical features on tribological performance by using CFD. Several researchers have compared the validities of the CFD and Reynolds equations on textured surfaces [11,12]. Li and Chen published that the Reynolds equation is not suitable for calculating the effect of squared transverse textures when the heights of textures are higher than 10% of the thickness of the film [11]. M. Dobrica and M. Fillon have illustrated the validity of the Reynolds equation in the textured surface [12]. Therefore, some researchers have used CFD to discuss the relationship between the textured configurations and lubricant performances [13–15]. Cupillard et al. [13] solved the Navier-Stokes equations and showed that micro-dimpled textures can minimize surface friction. Sahlin et al. [14] solved the Navier-Stokes equations to optimize the geometrical features of dimples on a surface in order to minimize the friction coefficient. Caramia et al. [15] also explained how to achieve a maximum friction reduction of 20%. Another texture, the sawtooth riblet, has also been used to decrease friction on the surface of aircraft in turbulent air flows [16,17], and a maximum friction reduction of 10% was attained. Compared with the turbulent air, the effects of the fluid compressibility and the vortex distribution on friction reduction are ignored in the lubricant flow, but the friction is also very likely to be reduced by reducing the amount of surface area exposed to the lubricant flow, a Newtonian laminar flow, with a large velocity gradient in the normal direction of the surface. Moreover, sawtooth riblets are easy to produce by precision cutting with geometrically defined structures, unlike micro-dimpled textures, which were widely produced by laser machining and electrical discharge machining [18,19]. The lubricant-film thickness, which plays a sensitive role in the wall shear stress and the velocity gradient, has generally been defined as larger than $1 \mu\text{m}$ in previous simulations [14,15]. However, it is usually in the submicron range in industrial activities.

We therefore investigated the friction reduction of sliding friction pairs having micrometer-scaled sawtooth ri-

blets with micrometer- or submicrometer-thick film. To seek out the optimum shape of sawtooth riblets for friction reduction, a statistical method, the orthogonal experimental design, was used to design the plan of simulations. Because orthogonality exists in the ridge angles, the height of the riblets, and the distance between the surfaces, these three parameters are defined as the orthogonal variables to control the geometry of the calculation field, namely, the geometry of the lubrication film. Additionally, it is essential to define a recommendatory sliding speed for the friction pairs in industrial applications, such as hydrodynamic bearings and rod seals, so the sliding speed of friction pairs was also defined as an orthogonal variable.

2. Mathematical Model

2.1. Governing Equations

The continuity equation and the steady three-dimensional Navier-Stokes equations for an incompressible lubricant flow can be written as follows:

$$\frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} + \frac{\partial w}{\partial z} = 0, \quad \dots \quad (1)$$

$$\rho \left(u \frac{\partial u}{\partial x} + v \frac{\partial u}{\partial y} + w \frac{\partial u}{\partial z} \right) = -\frac{\partial p}{\partial x} + \mu \left(\frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} + \frac{\partial^2 u}{\partial z^2} \right), \quad \dots \quad (2)$$

$$\rho \left(u \frac{\partial v}{\partial x} + v \frac{\partial v}{\partial y} + w \frac{\partial v}{\partial z} \right) = -\frac{\partial p}{\partial y} + \mu \left(\frac{\partial^2 v}{\partial x^2} + \frac{\partial^2 v}{\partial y^2} + \frac{\partial^2 v}{\partial z^2} \right), \quad \dots \quad (3)$$

$$\rho \left(u \frac{\partial w}{\partial x} + v \frac{\partial w}{\partial y} + w \frac{\partial w}{\partial z} \right) = -\frac{\partial p}{\partial z} + \mu \left(\frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} + \frac{\partial^2 w}{\partial z^2} \right), \quad \dots \quad (4)$$

where u , v , and w are the components of the local velocity vector in the directions of x , y , and z in the lubricant film. p , ρ , and μ are the pressure, density, and dynamic viscosity of the lubricant, respectively. In the simulation, the dispersed Navier-Stokes equations are solved by the SIMPLE algorithm [20].

2.2. Geometry of Computational Field

A three-dimensional sawtooth riblets model was built, as shown in **Fig. 1**, to analyze the friction performance. As a typical model of the Taylor-Couette flow, the sliding speeds of the upper movable plate are defined as 0.01, 0.02, and 0.03 m/s. The sliding direction of the movable upper plate is parallel to the ridge of the microstructures, as indicated by the green arrow. Since the Reynolds number ranges from 0 to 0.111, the fluid is considered as a laminar flow. The surfaces around the gap between the upper plate and the lower textured surface, marked with

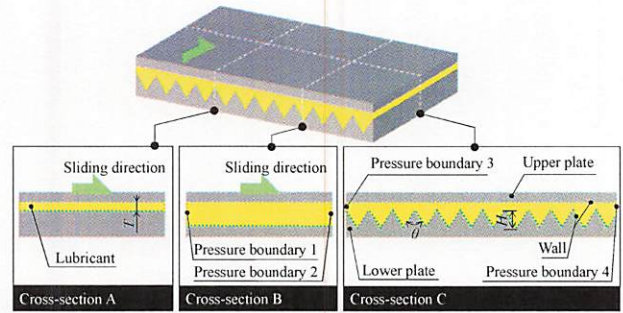


Fig. 1. Schematic of computational field and boundaries.

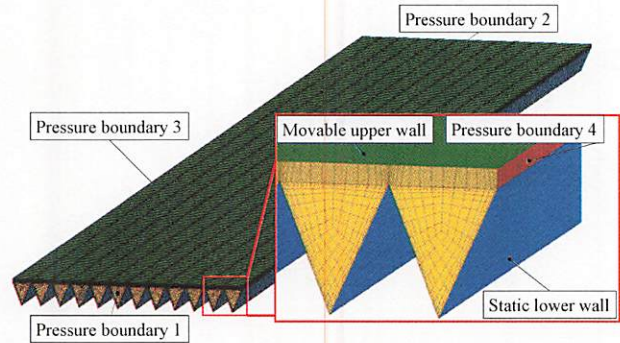


Fig. 2. Structured hexahedral grid and boundary conditions.

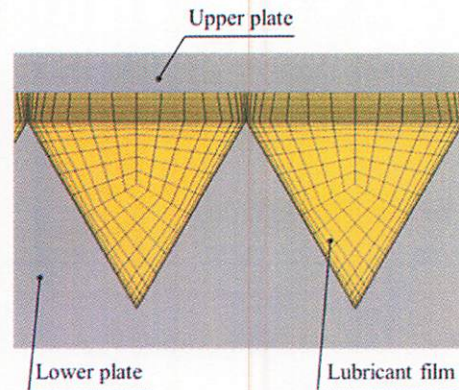


Fig. 3. Dense grids in the gap between the upper plate and the ridges of sawtooth riblets.

red and orange dashed lines in cross-sections, are defined as pressure boundaries of 101,325 Pa.

The structured hexahedral grid shown in **Fig. 2** is set up to disperse the computational field, yellow lubricant film. The pressure boundaries 1, 2, 3, and 4 are defined as 101,325 Pa. The difficulty comes when taking into account that the riblet height (H) is much larger than the thickness of the lubricant film (T). This special height ratio between H and T leads to the large changes in pressure and velocity gradient that occur precisely in the lubricant film on the ridges of riblets. Therefore, dense grids are generated between the ridges of riblets and the upper wall, as shown in **Fig. 3**. The theorem of grid distribution in the

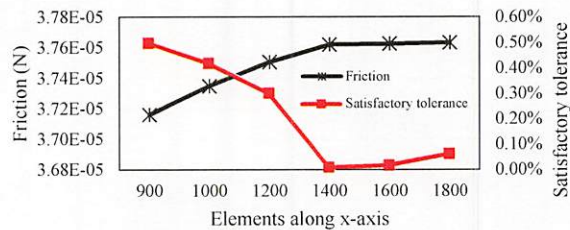


Fig. 4. Relation of calculation stability to the number of grids.

normal direction of the wall used in our calculations can be written as follows:

$$Z_p \sqrt{\frac{u_\infty}{\nu \cdot x}} \leq 1, \dots \dots \dots (5)$$

where Z_p is the distance to the wall from the adjacent cell centroid, u_∞ is the average flow velocity of the lubricant, ν is the kinematic viscosity of the lubricant, and x is the distance along the wall from the starting point of the boundary layer [21].

Based on this theorem, grid-clustering is done near the upper wall and the textured surface. It is observed that the parameter, which is sensitive to the friction of the textured surface, is grid distribution along the direction of flow. By varying the element size along the direction of flow, it is possible to obtain different calculation results [17, 22].

The frictions on the textured surface are calculated for grids with different elements along the direction of flow (x -axis). The result of the grid study presented in Fig. 4 reveals a tolerance between each calculation. A grid with more than 1,400 elements along the x -axis can provide accurate friction, within a satisfactory tolerance of 0.02%. Since calculation time increases with the increase in the quantity of elements, the condition of 1,400 elements along the direction of flow has been used in all of the calculations. The chosen grid consists of 7,271,600 nodes and 7,779,712 hexahedral elements.

3. Orthogonal Design

In order to find the optimal geometry of sawteeth and sliding speed for friction reduction, the orthogonal experimental design [23] and the range analysis are provided to design the plan for the simulations with different conditions. Four orthogonal factors, the ridge angle of riblet θ , the thickness of lubricant film T , the velocity of upper plate V , and the height of riblet H are selected as the variables of simulations. Each orthogonal factor, factor j , is defined as three levels, and $i \in \{1, 2, 3\}$ is the ordinal number of the level. The images and geometrical features of the textured and the flat surfaces are listed in Table 1, and detailed combinations of factors are listed in Table 2. A process for determining the primary factors and the optimal combination is shown in Fig. 5.

The coefficient of friction reduction of a textured surface calculated by number s simulation, Cr_s , is given by

Table 1. Geometrical features of textured surfaces and flat surface.

Level i								
	T [μm]	H [μm]	θ [deg]	T [μm]	T [μm]	H [μm]	θ [deg]	T [μm]
$i = 1$	0.3	10	60	0.3				
$i = 2$	0.5	20	70	0.5				
$i = 3$	3	50	90	3				

Table 2. Designed plan of simulations based on $L_9 (3^4)$.

Sim. Num. s	Factors j				
	T [μm]	θ [deg]	H [μm]	V [m/s]	
1	0.3	60	10	0.01	
2	0.5	60	20	0.02	
3	3	60	50	0.03	
4	0.3	70	20	0.03	
5	0.5	70	50	0.01	
6	3	70	10	0.02	
7	0.3	90	50	0.02	
8	0.5	90	10	0.03	
9	3	90	20	0.01	

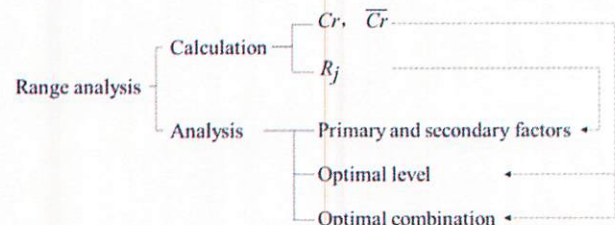


Fig. 5. Process of range analysis. Cr is the coefficient of friction reduction, $\overline{Cr}$ is the average value of Cr , R_j is the range of $\overline{Cr}$ of the factor j (T , H , θ or V).

the following equation:

$$Cr_s = 100 \times \frac{(F_{\text{texture}} - F_0)}{F_0}, \dots \dots \dots (6)$$

where F_{texture} is the friction of a textured surface and F_0 is the friction of a flat surface. The friction, F , can be written as follows:

$$F = \int \tau dA, \dots \dots \dots (7)$$

where A is defined as the wetted area of the flat or textured surface. The wall shear stress, τ , is related to the normal velocity gradient at the wall and given by the following equation:

$$\tau = \mu \frac{\partial u}{\partial n}, \dots \dots \dots (8)$$

Table 3. Results of simulations.

Sim. Num. s	Cr [%]
1	87.77
2	84.22
3	67.65
4	90.20
5	92.22
6	31.00
7	92.91
8	78.74
9	54.99

where n is the normal vector of the surface. For a lubricant, dynamic viscosity, μ is a constant of 7.05 mPa·s. The velocity, u can be obtained by solving the Navier-Stokes equations.

The average coefficient of friction reduction in the level i of factor j , $\overline{Cr}_{ji}$, is given by the following equation:

$$\overline{Cr}_{ji} = \frac{Cr_{s1} + Cr_{s2} + Cr_{s3}}{3}, \dots \dots \dots (9)$$

where s_1 , s_2 , and s_3 are the numbers of simulations with factor j set to the i -th level. Based on the magnitude of $\overline{Cr}_{ji}$, the optimal level of factor j can be determined. The combination of the optimal levels of T , θ , H , and V is the optimal combination for friction reduction.

For the range analysis of orthogonal factors, the range of $\overline{Cr}_{ji}$, R_j , can be written as follows:

$$R_j = \max\{\overline{Cr}_{ji}\}_{i \in \{1,2,3\}} - \min\{\overline{Cr}_{ji}\}_{i \in \{1,2,3\}}. \quad (10)$$

A larger value of R_j indicates more impact of factor j on the extent of the friction reduction. That is, the factor j is more important for friction reduction than other factors.

4. Results and Discussion

4.1. Range Analysis of Factors

The results of simulations are presented in **Table 3**, and the range analyses of Cr are listed in **Table 4**. What calls for special attention in **Table 4** is that the result of range analysis, $R_T > R_H > R_V > R_\theta$, shows that the thickness of the lubricant film, T , plays the most significant role in the process of friction reduction. Additionally, the optimal level of factor j (T , θ , H or V) is defined by the largest value of the $\overline{Cr}_{j1}$, $\overline{Cr}_{j2}$, and $\overline{Cr}_{j3}$ in the same column under factor j . The results indicate that the optimal combination of geometrical parameters and sliding speed for friction reduction is T_1 , H_3 , θ_1 , and V_3 .

4.2. Analysis of Velocity and its Gradient

The friction is related to the velocity gradient near the interface between the surface and lubricant. Therefore, the profiles of the velocity and its gradient in the near-wall region with a distance of 0–0.3 μm from the surface are

Table 4. Range analysis of factors.

$\overline{Cr}_{ji}$	Factors j			
	T	θ	H	V
$\overline{Cr}_{j1}$	90.29	79.88	65.84	78.33
$\overline{Cr}_{j2}$	85.06	71.14	76.47	69.38
$\overline{Cr}_{j3}$	51.21	75.54	84.26	78.86
R_j	39.08	8.74	18.42	9.49
Priority	$T > H > V > \theta$			
Optimal combination	T_1 0.3 μm	θ_1 60°	H_3 50 μm	V_3 0.03 m/s
Optimal Cr	93.83%			

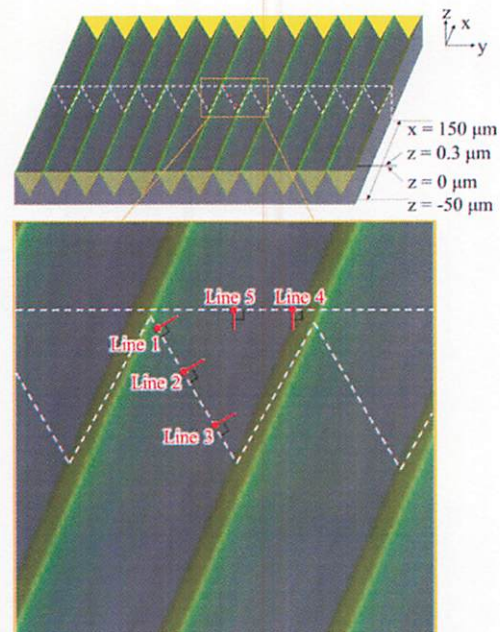
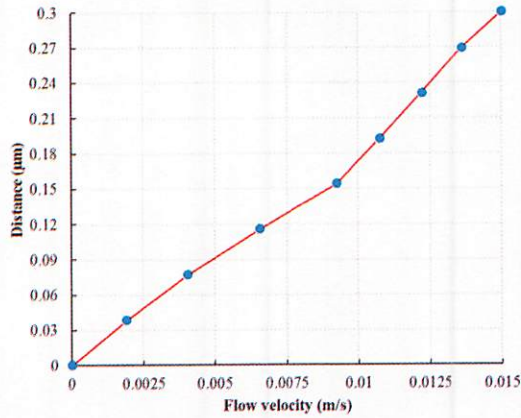


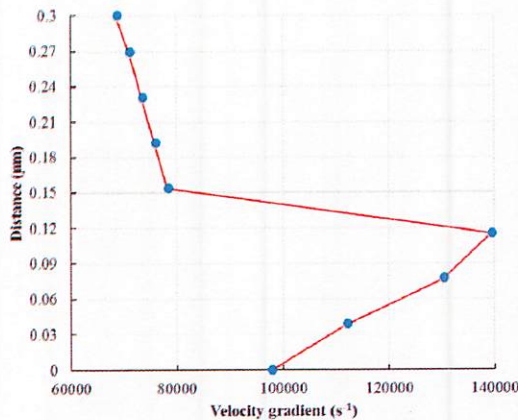
Fig. 6. Positions of lines = 1, 2, and 3 in the fluid field.

analyzed. To obtain the results on the different positions of the interface, five vertical lines with a length of 0.3 μm , lines 1–5, are located in cross-section C, which is defined as the place where $x = 150 \mu\text{m}$ and is parallel to plane YZ, as shown in **Figs. 1** and **6**. The white dotted lines indicate the intersections of cross-section C and the interfaces. As shown in **Fig. 6**, lines 1–5 are perpendicular to the white dotted lines on the different positions on the interfaces.

Equation (8) reveals the relationship between the velocity gradient and the wall shear stress. Moreover, μ is assumed as a constant, so the wall shear stress increases along with the growing velocity gradient. Eq. (7) shows that the friction also increases along with the growing wall shear stress. It means that the velocity and its gradient are directly related to the friction, so the gradient and the wall shear stress are discussed in the following. As presented

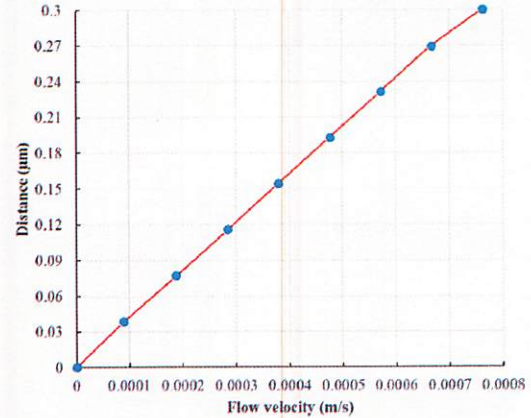


(a)

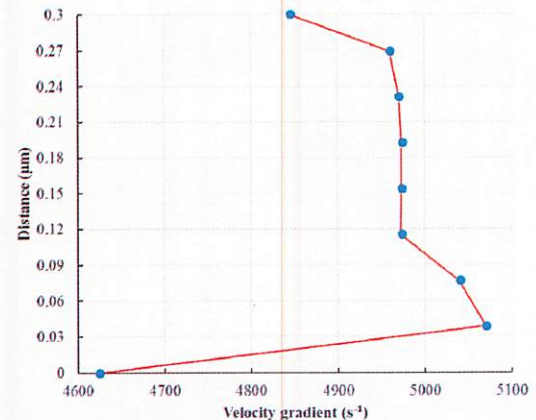


(b)

Fig. 7. Flow velocity and velocity gradient on the ridge of riblet (line 1): (a) velocity, (b) velocity gradient.



(a)



(b)

Fig. 8. Flow velocity and velocity gradient on the sidewall of riblet (line 2): (a) velocity, (b) velocity gradient.

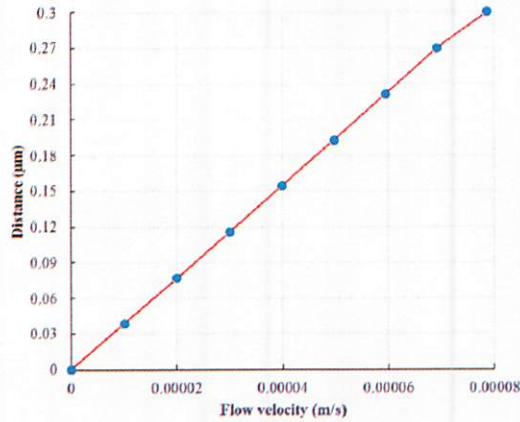
in Eq. (6), the friction on the flat surface which is a benchmark for comparison with that on the textured surface, so the velocity and gradient of a textured surface are compared with those of a flat surface. Based on the predictive results of orthogonal experimental design and range analysis, the velocity and its gradient of the textured surface with the optimal combination of T_1 , H_3 , V_3 , and θ_1 were investigated. **Figs. 7–9** show the details of these parameters at the positions of the lines drawn above the interfaces. Moreover, the velocity and its gradient on the moving flat surface are shown in **Figs. 10** and **11**. In these figures, the vertical axes represent the distance to the surface from the local position, and the horizontal axes represent the flow velocity and the corresponding velocity gradient.

Figure 7 shows that the velocity and its gradient at the position of the ridges of riblets (line 1). As can be seen in **Fig. 7(a)**, the linear velocity increases with the growing distance to the surface from the local position. Otherwise, the velocity gradient is smaller than $140,000 \text{ s}^{-1}$ in this near-wall region, as shown in **Fig. 7(b)**. At the interface between the ridge and the lubricant where the distance is 0 μm , a velocity gradient of $97,000 \text{ s}^{-1}$ is found.

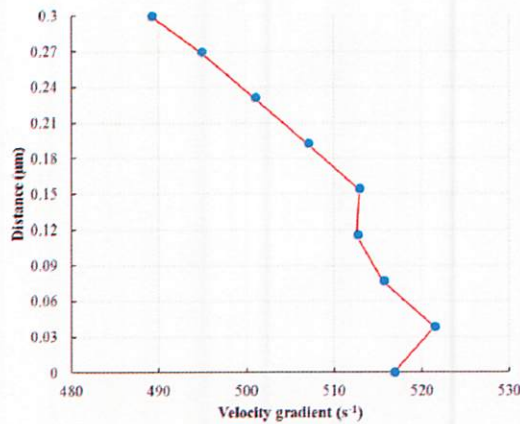
Figure 8 provides entirely different profiles of the velocity and velocity gradient on the sidewall of a riblet (line 2). The velocity near the sidewall also exhibits linear growth, but with a value much smaller than that near the ridge (line 1). Additionally, the velocity gradient of $4,624 \text{ s}^{-1}$ near the wall is also much smaller than that on line 1. On this position of the sidewall, which is 25 μm lower than the ridge, the results show that the velocity gradient decreases along with the increase in depth from the ridge of the riblet to the local position.

As shown in **Fig. 9**, similar trends of the velocity and velocity gradient are also observed in the bottom of the valley of the riblet (line 3), though the velocity gradient of 516 s^{-1} near the wall is much lower than that on the ridge (line 1) and the sidewall (line 2). Actually, those are not of the same magnitude. It should be noted that the flow velocity in most of the region of the valley is nearly 0 m/s . That is, the sawtooth riblet texture lifts the higher-velocity flow off the valley floor and reduces surface area exposed to the higher-velocity flow.

In the following **Figs. 10** and **11**, each curve is named based on its position and kinestate. For instance, in the



(a)



(b)

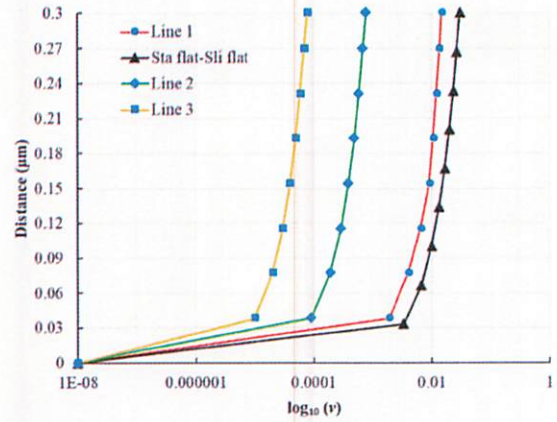
Fig. 9. Flow velocity and velocity gradient in the bottom of the valley between riblets (line 3): (a) velocity, (b) velocity gradient.

friction pairs, “Sta flat-Sli flat” indicates the stationary flat surface opposite the sliding flat surface, and “Sli flat-Sta saw” refers to the sliding flat surface opposite the sawtooth surface. **Fig. 12** is a schematic view showing the positions of the surfaces.

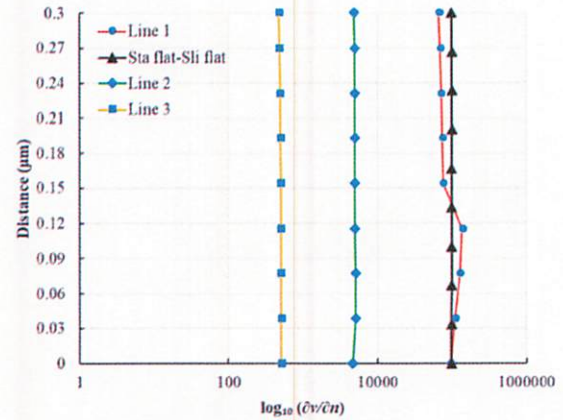
As shown in **Fig. 10**, the velocity and the velocity gradient on “Sta flat-Sli flat” are compared with those on the sawtooth surface (lines 1, 2 and 3). The velocity gradient on the flat surface is not only similar to that on the ridge of a sawtooth (line 1) but also slightly larger than that on the sidewall and the valley bottom of the sawtooth (lines 2 and 3). According to Eq. (8), the larger velocity gradient on the flat surface can lead to more massive wall shear stress than that on most of the area of the sawtooth riblet.

The velocities and the velocity gradients on lines 1, 2, and 3, as shown in **Figs. 7–11**, reveal that the gradient decreases with the decline in height of the local position from the bottom of the valley. It is evident that the valleys between sawtooth riblets reduce friction at the interface of the lubricant and the textured surface.

In order to clarify the effects of the stationary textured



(a)



(b)

Fig. 10. (a) Comparison of flow velocity on the flat surface opposite the sliding flat with that on the riblet, (b) comparison of velocity gradient on the flat surface with that on the riblet.

surface on the velocity and the gradient of the paired sliding flat surface, the friction of the sliding flat surfaces is discussed in the same manner. **Fig. 11** compares the velocities and the gradients on “Sli flat-Sta flat” with those on lines 4 and 5.

Figure 11(a) shows that the velocities decrease as the distance from the interface increases. On line 4, the velocity is lower than 0.005 m/s when the distance from the interface is greater than 0.25 μm . In the near-wall area, the descent rate of the velocity on line 4 is higher than that on line 5. However, in comparison with the “Sli flat-Sta flat,” the descent rates of the velocities on lines 4 and 5 are lower. The velocity gradients shown in **Fig. 11(b)** illustrate the discussions of the velocities. In the near-wall region, the gradient on line 4 is much larger than that on line 5, and smaller than that on the “Sli flat-Sta flat.”

4.3. Analysis of Friction and Wall Shear Stress

The two kinds of friction pairs are shown in **Fig. 13** to present the differences in surface friction. **Fig. 13(a)** presents the distribution of the wall shear stress on “Sli

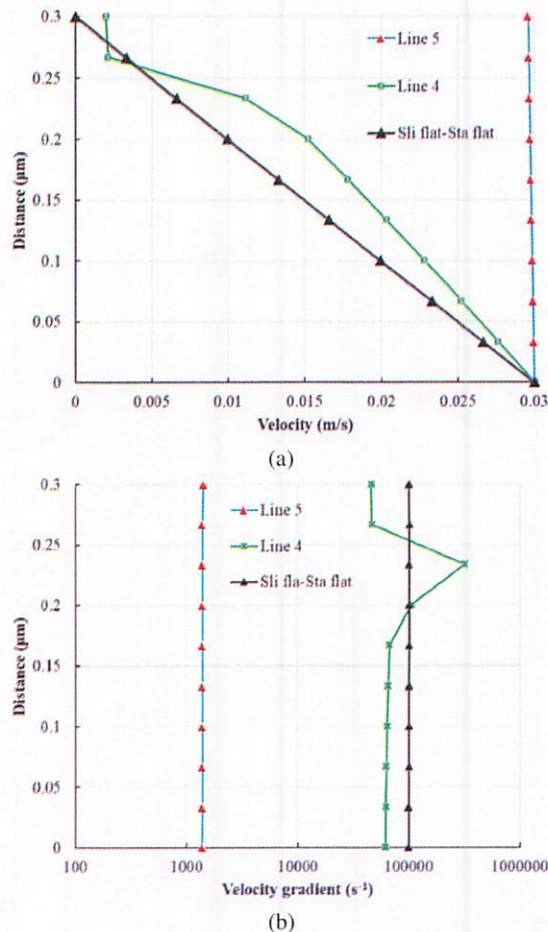


Fig. 11. (a) Comparison of flow velocity on the flat surface in textured friction pairs with that in smooth friction pairs. (b) Comparison of velocity gradient on the flat surface with that on the riblet.

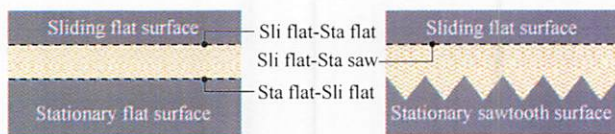


Fig. 12. Positions of the surfaces (Sli flat-Sta flat, Sli flat-Sta saw, and Sta flat-Sli flat).

flat-Sta flat." In Fig. 13(b), the wall shear stress on "Sta flat-Sli flat" is shown. It is clear that the distributions and magnitudes of the wall shear stress on the sliding surface are similar to those on the stationary surface. Figs. 13(c)–(e) provide the wall shear stress on "Sli flat-Sta saw" and the stationary sawtooth surface. As can be seen in the figures, the wall shear stress is nearly 0 Pa on most of the sawtooth surface. The maximum shear stress appears near the ridge of a sawtooth whether on "Sli flat-Sta saw" or a sawtooth surface. If Figs. 13(a) and (c) are compared, it is clear that the wall shear stress on "Sli flat-Sta flat" is larger than that on "Sli flat-Sta saw." Moreover, Figs. 13(b), (d) and (e) reveal clearly that the wall

shear stress of 682.88 Pa on the ridge of a sawtooth is not only less than that on the "Sta flat-Sli flat" but also much greater than that on other positions of the sawtooth riblet. The wall shear stress increases as the local position gets closer to the ridge. Moreover, the area of the ridge, the red area, is only a small portion of the total area of the textured surface.

On the basis of the aforementioned instructions and the expression of Eq. (7), the friction, F , on total textured surface is much less than that on "Sta flat-Sli flat." Moreover, a maximum coefficient of friction reduction of 93.83% is obtained. Sawtooth riblets can provide friction-reduction performance to an opposing flat surface in a sliding friction pair.

5. Conclusion

The effect of the textured surface on friction reduction was investigated by using numerical simulations. The presented research allows for valuable conclusions about the application of textured surfaces to friction reduction for sliding pairs in lubricants. The results of simulations and further discussions lead us to the following conclusions.

- (1) Sawtooth riblets can be used to decrease the friction by changing the normal velocity gradient of the lubricant in near-wall region. The maximum friction on the textured surface is nearly the same as that on a flat surface, and the friction on the other places, such as the sidewalls and bottoms of the riblets, is much smaller than that on a flat surface.
- (2) The sawtooth riblet can provide friction-reduction performance for the paired sliding flat surface in the friction pairs.
- (3) The effects of geometrical features on friction reduction were presented. The geometrical parametric analysis based on the range analysis and the orthogonal experimental design with statistical significance reveals the impact of the texture features on the performance of friction reduction. The thickness of the lubricant film (T) plays the most crucial role. The riblet height (H) and the ridge angle (θ) are the secondary factors in decreasing friction.
- (4) The predicted optimal condition for friction reduction of about 93.83% was successfully verified. Additionally, the recommended sliding speed of 0.03 m/s for the optimal shape of the textured surface was also verified.

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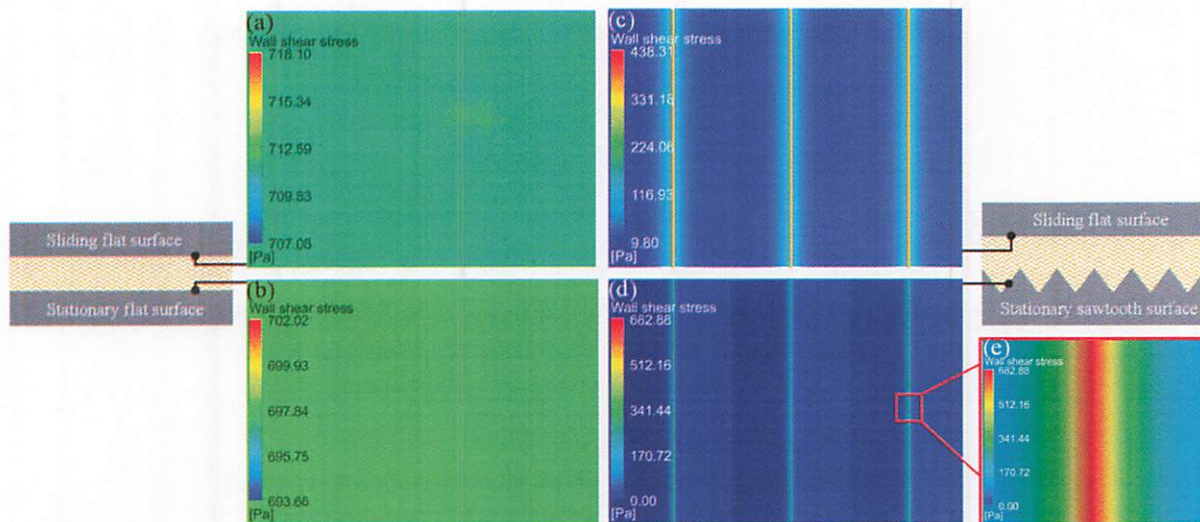


Fig. 13. Distribution of wall shear stress: (a) on the “Sli flat-Sta flat,” (b) on the “Sta flat-Sli flat,” (c) on “Sli flat-Sta saw,” (d) on the sawtooth surface, (e) on the ridge of sawtooth.

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Paper:

Friction Reduction by Micro-Textured Surfaces in Lubrication

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Experimental investigations were carried out to verify if the friction reduction in lubrication can be expanded by a textured surface with sawtooth riblets. Sawtooth riblets were formed by ultraprecision diamond cutting, with a ridge angle of about 60°–90° and height of about 20–50 μm on the contact surface. Six types of textured surfaces with different ridge angles, heights, and sliding directions were tested and compared with the untextured surface. The tribological tests were conducted by a flat-on-flat tribometer in lubrication. The effects of the ridge angle, height, and relative sliding direction on the friction coefficient in lubrication were reported.

Keywords: textured surface, friction reduction, lubrication, ultraprecision diamond cutting

1. Introduction

With the development of the fabrication technology, various functions including optical and friction properties can be obtained by fabricating regular patterns or textures on surfaces [1]. Decreasing the friction and wear of lubricated friction pairs is crucial for improving the service life and accuracy of transmission parts such as shafts, bearings, and gears. Severe wear and high friction occur when a plastic deformation is generated by solid-to-solid contacts in lubrication. The modification of surface topography by artificially produced microtextures influences the deformations and behaviors of lubrication films, and consequently, the wear and friction of rubbing surfaces.

Dimples and grooves have been widely studied to improve the tribological performance of friction pairs [2–5]. To research the friction characteristics of a micro-dimpled surface, some fabrication methods of dimples, such as micro-coating, abrasive jet machining (AJM), laser beam machining (LBM), electrical discharge machining (EDM), and etching, have been developed. Ramesh et al. reported that the dimpled surfaces fabricated by micro-coating have a lower coefficient of friction (COF) compared to the untextured surface under mixed lubrication [6]. Pettersson et al. produced grooves and dimples on the wafer by using etching and coating and studied

the frictional performances of textured surfaces in lubrication. They found that TiN-coated surfaces exhibit a good frictional performance, however the low friction conditions last shorter on the textured surface than on the flat surface [7]. Wakuda et al. [8] fabricated micro-dimples on a silicon nitride ceramic nitride surface by using AJM and LBM and studied the effect of micro-dimples on the friction. They showed that the size and density of the micro-dimples have a significant influence on the friction reduction of friction pairs in lubrication. The dimpled surfaces with a diameter of 100 μm and a texture area ratio of 5%–20% were recommended. Segu et al. used an Nd:YAG laser to fabricate multi-shaped dimples on a scale of hundreds of micrometers in width on steel surfaces, and found that the multi-shape dimpled surface can significantly reduce friction under dry and lubrication conditions when compared to untextured surfaces [9]. Podgornik et al. [10] fabricated the micro-dimpled surface on a steel surface by using an Nd:YAG laser and summarized that the smaller dimples and lower texture area ratio can lead to the lower friction of textured surfaces. EDM was also employed to fabricate the textured surface with low friction [11]. The friction coefficient of the EDM-dimpled surface was found to be 11%–24% lower than that of the non-textured surfaces. The frictional performance of the round, diamond, and ellipse dimples was studied, and the round dimples were found to be the most helpful for decreasing the wear and friction.

Recently, researchers have not only focused on the tribological features of the micro-dimpled surfaces but also on that of the micro-grooved surfaces for development of bearings and shafts. Kawasegi et al. [12] proposed the milling tools with micro- and nanometer-scale grooves to improve the frictional characteristics and resulting machining performance successfully. Scaraggi et al. [13] presented an experimental approach on the friction properties of a laser-grooved surface from the boundary to hydrodynamic lubrication. Suh et al. [14] fabricated the micro-grooved crosshatch pattern on the SKD11 steel by the photolithography process and observed the relations among the frictions of the textured surfaces, the crosshatch angles of micro-grooves, and the width of grooves by using a pin-on-disc tribometer. They compared the frictions of the textured surfaces by changing the cases of the pattern widths and crosshatch angles.

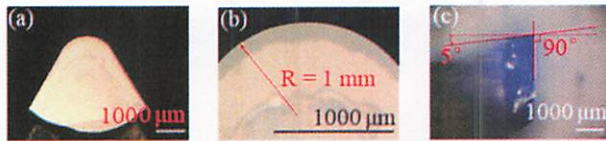


Fig. 1. Geometric features of diamond tool to finish the mirror surface: (a) rake face, (b) enlarged drawing of cutting tip, (c) 0° rake angle and 5° clearance angle.

They, however, did not discuss the friction reduction in comparison with a mirror-finished surface.

Thus, it is evident that the rough parts of the textured surfaces, such as the top of the groove [15], the edges of dimples, and the non-dimpled regions between them [16], influence the friction of the textured surface. However, such parts are difficult to control in the above fabrication methods. Compared to the photolithography process and laser machining, ultraprecision diamond cutting is more efficient, applicable, precise, and reproductive for texture producing various metals. Additionally, as an environmentally friendly processing method, ultraprecision diamond cutting can also be used to fabricate a polygonal shape, which is difficult to be finished precisely by using the above-mentioned methods. Moreover, the friction of sawtooth riblets has not been discussed in detail. Based on above background, this paper aims to analyze the influences of the geometrical features of sawtooth riblets on friction and the friction reduction in comparison with the mirror-finished surface in lubrication. The experiments reported in this paper were conducted on textured surfaces with sawtooth riblets parallel or transverse to the relative sliding direction of the friction pairs. The relation between the sliding friction and direction of relative motion was also studied. Additionally, the load carrying capacity of the textured surface, which is an important factor influencing solid friction in lubrication, was investigated in two sliding forms by using computational fluid dynamic analysis.

2. Experiments

2.1. Surface Texturing

Ultraprecision diamond cutting, a mechanical process used in precision fabrication to efficiently and precisely remove the substrate material, was carried out on the mirror-finished surfaces of aluminum alloy 7075 (A7075). It can not only afford precise control over the shape and size of the sawtooth riblets but also finish the mirror surface and sawtooth riblets over an entire surface simultaneously with high accuracy. To control the roughness of the rubbing surface, the roughness, R_a , was measured using a probe-type roughness tester. The A7075 samples were preliminarily processed to mirror surfaces with roughness (R_a) of 0.005 μm by using the diamond tool shown in Fig. 1. The V-shaped diamond tools shown in Figs. 2–4 were then employed on the mirror-finished surfaces to obtain the sawtooth riblets

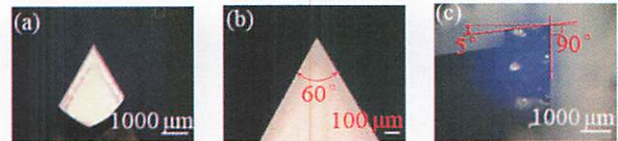


Fig. 2. Geometric features of 60°-V-shaped diamond tool: (a) rake face, (b) enlarged drawing of cutting tip, (c) 0° rake angle and 5° clearance angle.

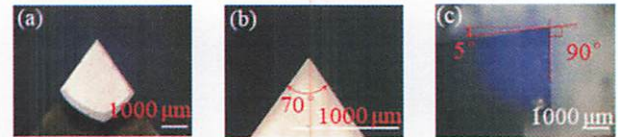


Fig. 3. Geometric features of 70°-V-shaped diamond tool: (a) rake face, (b) enlarged drawing of cutting tip, (c) 0° rake angle and 5° clearance angle.

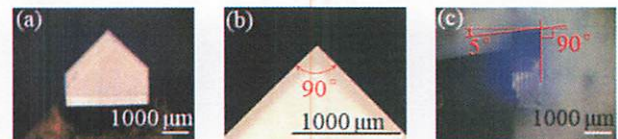


Fig. 4. Geometric features of 90°-V-shaped diamond tool: (a) rake face, (b) enlarged drawing of cutting tip, (c) 0° rake angle and 5° clearance angle.

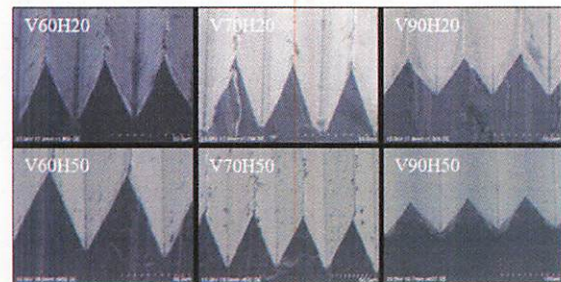


Fig. 5. SEM photographs of the sawtooth surfaces.

with different ridge angles. The rake and clearance angles of the tools were 0° and 5°, respectively. As we know, increasing the cutting speed is a useful way to suppress the burrs and finish cutting with a small cutting thickness is also necessary for improving the shape accuracy of textures. Therefore, the maximum cutting speed of the machine tool, i.e., 10 m/min, was used. Moreover, the cutting depth of the finish cutting was set as 1 μm .

Here, each sample is named based on its degree of the ridge angle with V, the value of the sawtooth height in micrometers with H, and the relative sliding direction with either capital of traverse or parallel, such as “V60H20.T.” The six types of sawtooth surfaces were fabricated by the selected diamond tools, and their SEM photographs are shown in Fig. 5. It can be seen that the qualities of the ridges of the sawtooth riblets differed slightly. However, because the sizes of the burr and the notch of the ridges were much smaller than the depth of the valley and the

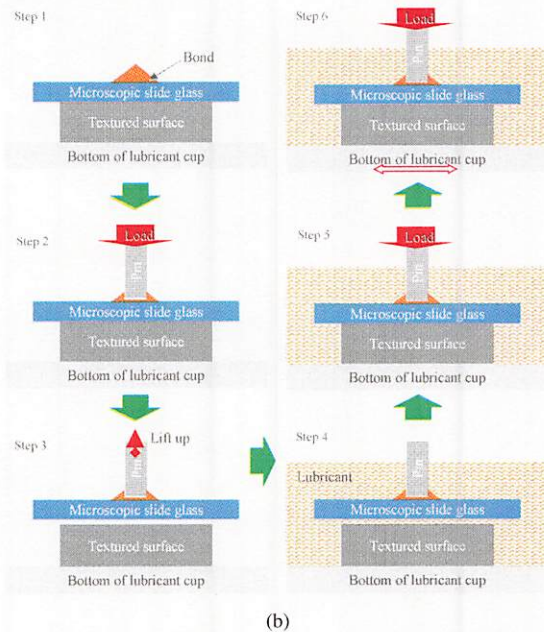
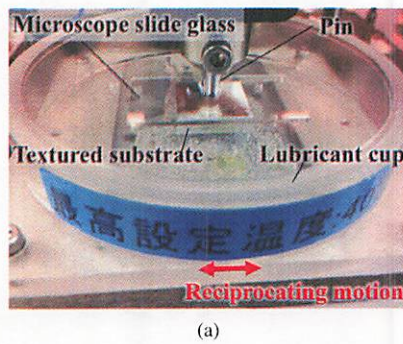


Fig. 6. (a) Flat-on-flat tribometer, (b) diagram of the measuring procedure.

valley and ridge angle played an important role in friction reduction, the quality of the ridge was not considered in this study.

2.2. Tribological Testing

The tribological behaviors of the friction pairs consisting of a ribleted surface and a microscope slide glass were investigated under a reciprocating motion by using a surface contact-type tribometer, as shown in **Fig. 6(a)**.

To keep the upper and lower pairs parallel to each other, the following six steps, as shown in **Fig. 6(b)**, were performed. In Step 1, the bond was painted on the roof of the microscope slide glass, which was placed on the fixed textured surface. In Step 2, the pin that contacted the force sensors was loaded and fixed with the roof of the microscope slide glass by the bond. After this step, the glass and textured surface can be considered as parallel friction pairs. To establish the lubricated friction pairs, the upper glass was lifted (Step 3) and the lubricant was filled into the gap of the pairs (Step 4). In Step 5, the normal

Table 1. Experimental conditions.

Material	Microscope slide glass (upper pair) A7075 (lower pair)
Normal load	50 g
Contact area	400 mm ² (20 mm × 20 mm)
Sliding velocity	10 mm/s (reciprocating motion)
Temperature	25°C
Lubricant viscosity	8.108×10^{-6} m ² /s
Lubricant density	869 kg/m ³

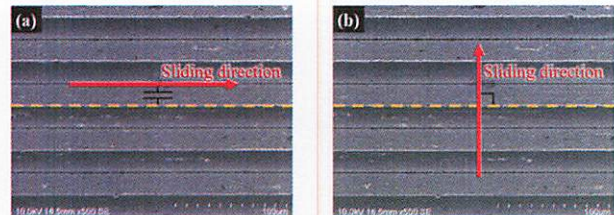


Fig. 7. Sliding directions relative to the ridges of sawtooth riblets: (a) in parallel form and (b) in transverse form.

load acting on the pin was carried out to give an initial lubricant film of the friction measurement. In Step 6, the friction of the friction pairs was measured under reciprocating motion.

Moreover, the substrate was an A7075 with a ribleted surface; the upper sliding flat was a mirror surface formed of the microscope slide glass with a surface roughness (R_a) of 0.002 μ m. During the tribological testing, the friction pairs were immersed in the No. S8P oil lubricant, whose temperature was 25°C. The details of the testing are reported in **Table 1**. In the 600 s of testing time, each testing had a sixty-second pre-run time to prevent the unstable friction signal generated by the starting friction, which has a substantial effect on the average of the sliding friction.

It is well known that the pressure profile of the lubricant plays a major role in improving the load carrying capacity and frictional performance of the friction pairs [17, 18]. Therefore, to clarify the influence of relative sliding direction on the frictions of different sawtooth riblets, the two forms of relative sliding directions of the friction pairs, the transverse and parallel directions, were used in the tribological testing, as shown in **Fig. 7**. The lubricant in different sliding directions could provide different load carrying capacities acting on the upper slide glass to offset the contact pressure on the contact areas between the microscope slide glass and the ridges of the sawtooth riblets.

3. Results and Discussion

3.1. Friction in Lubrication

The COFs (y-axis) were plotted against the ridge angles (x-axis) of the sawtooth riblets in **Fig. 8**. To analyze the friction reductions of sawtooth surfaces, the COF of

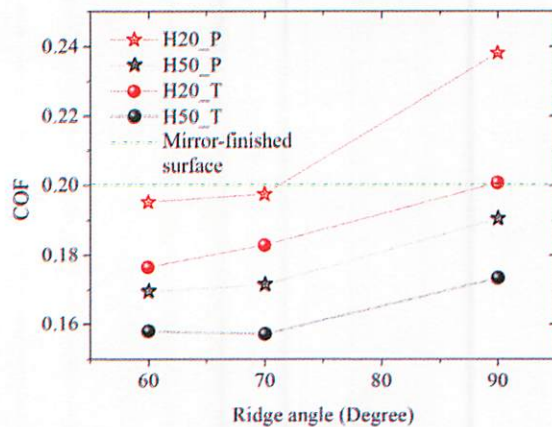


Fig. 8. Relations among the COFs, heights, and ridge angles of sawtooth riblets in the transverse and parallel sliding forms.

the mirror-finished surface, which is equal to 0.203709, was also measured and considered as a standard in comparison with that of the sawtooth surfaces.

Clearly, the COFs increased with the growth of the ridge angles irrespective of being in the parallel sliding form or transverse sliding form. Moreover, a sawtooth height of 50 μm could provide lower COFs than that of 20 μm , as shown in Fig. 8. The curves of “H20_P” and “H20_T” show the influence of the sliding form on friction. It is evident that the transverse sliding form can provide a lower COF than the parallel sliding form regardless of the heights and ridge angles of sawtooth riblets. In comparison with the mirror-finished surface, the selected sawtooth textured surfaces can provide lower COFs. The transverse sliding form is also able to give a deeper improvement to decrease the COFs of the sawtooth surfaces. “V70H50_T” for 70° ridge angle with 50 μm sawtooth height in the form of transverse sliding can provide the largest friction reduction of 22.85%.

Furthermore, the fluctuations in the friction forces during the measuring procedures are listed in Fig. 9. The x-axis shows the measuring times, and the y-axis shows the measured friction force. As shown in Fig. 9, all friction forces were nearly symmetrical with 0 mN, which means that the measurements were performed under stable states.

3.2. Load Carrying Capacity of Lubricant

Because the fluid friction between the lubricant and solid surface is much lower than the solid friction, a decrease in the fluid friction cannot give an obvious friction reduction in lubrication. The solid-to-solid pressure is directly proportional to the solid friction. Therefore, one of the main objectives of the sawtooth surface is to improve the load carrying capacity for offsetting the normal solid-to-solid pressure occurring on the sawtooth ridge. As an important role for friction behaviors of friction pairs in lubrication, the load carrying capacity of the saw-

tooth surface was discussed by computational fluid dynamic (CFD) analysis.

In the CFD model, the continuity equation and the steady three-dimensional Navier-Stokes equations for an incompressible lubricant flow can be expressed as

$$\nabla \cdot V = 0 \quad (1)$$

$$\rho (V \cdot \nabla) V = -\nabla p + \nabla \cdot (\mu \nabla V) \quad (2)$$

where V is the local velocity in the lubricant film, and p , ρ , and μ are the pressure, density, and dynamic viscosity of the lubricant, respectively. In the simulation, the dispersed Navier-Stokes equations were solved using the SIMPLE algorithm [19].

A three-dimensional sawtooth riblets model was built to analyze the load carrying capacity in different sliding forms. As shown in Fig. 10(a), the model was composed of a lubricant (yellow part), glass pane, sawtooth surface, and the interface (orange plane) between the lubricant and the glass flat. The sliding velocities of the movable sawtooth surfaces were 10 mm/s along the directions of parallel and transverse to the ridges of the sawtooth riblets. The ridge angle and height of the sawtooth were set as 90° and 50 μm , respectively. This numerical calculation was performed at an environmental pressure of 101325 Pa. A normal load of 0.5 N was applied on the upper glass surface with an area of 200 $\mu\text{m} \times 200 \mu\text{m}$. Considering that the calculation time increased with the number of elements, the calculational field was discretized into 200,000 elements, as shown in Fig. 10(b).

The load carrying capacity, F , can be written as

$$F = \int p_z dA \quad (3)$$

where A was defined as the area of the interface between the upper flat and the lubricant. The normal pressure acting on the interface, p_z , was not only related to the shape of the fluid field and the relative sliding direction of the friction pairs.

The distributions of normal pressure on the interface between the upper glass and lubricant were shown in Fig. 11. During the transverse sliding of the sawtooth surface, the load carrying capacity centered on the interface near the sawtooth ridge was larger than that on the other place, as shown in Fig. 11(a). The normal pressure from the upper glass acting on the sawtooth ridge could be offset by the load carrying capacity. However, a different condition was found in Fig. 11(b), where the sawtooth surface in the form of parallel sliding could neither give enough normal pressure acting on the upper glass nor offset the solid contact. The lubricant could be easily squeezed out through the valley with sawtooth sliding, so the load carrying capacity was nearly 0 Pa in the form of parallel sliding.

To give an intuitive impression, the load carrying capacity was calculated by Eq. (3) based on the normal pressure acting on the upper flat. The results indicated that the load carrying capacity in the form of transverse sliding was 2.68×10^{-11} N, which is larger than that in the form

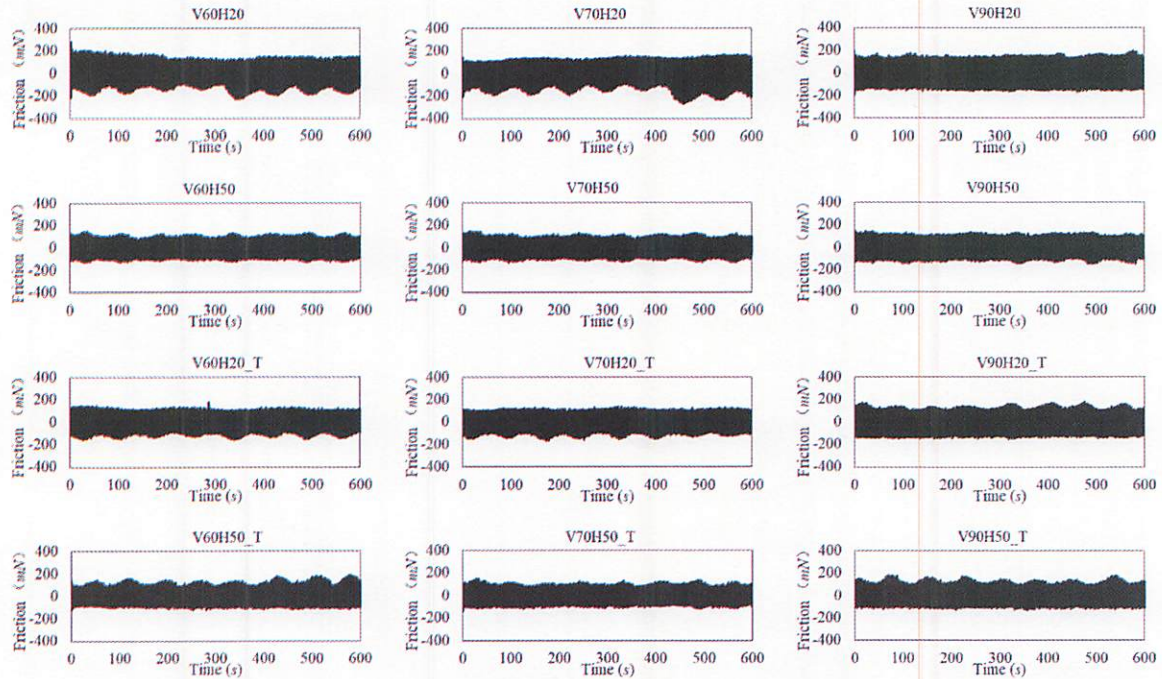
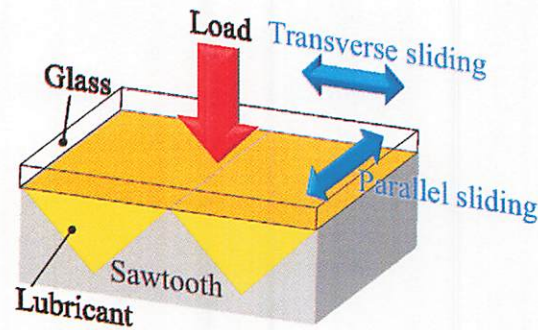
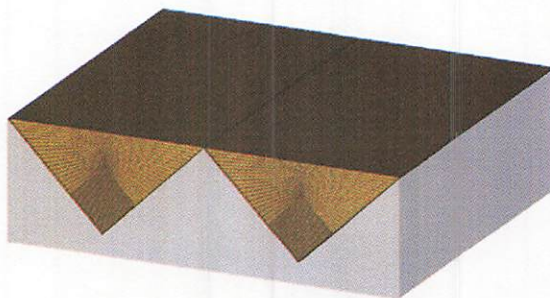


Fig. 9. Relation between the friction forces and measuring times.

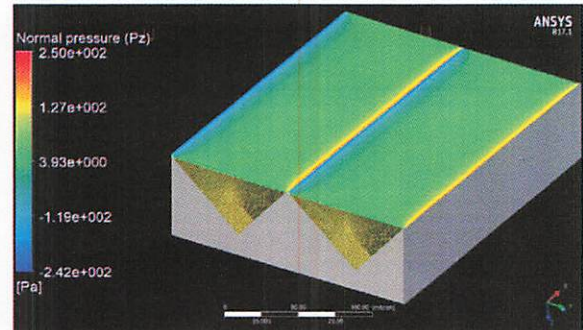


(a)

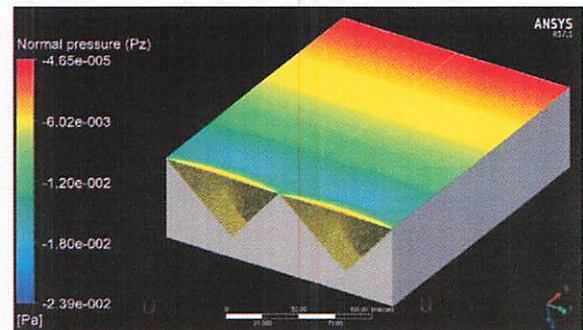


(b)

Fig. 10. (a) Schematic of the computational field. (b) View of the computational elements of the computational field (lubricant).



(a)



(b)

Fig. 11. Normal pressure acting on the upper flat surface in the form of (a) transverse sliding and (b) parallel sliding.

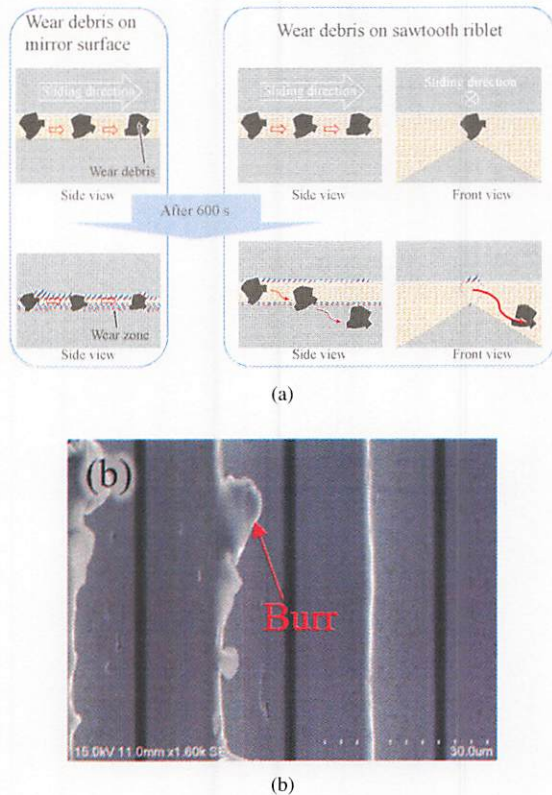


Fig. 12. (a) Schematics of the motion of wear debris on the mirror-finished and sawtooth surfaces, (b) burrs on the ridge of the sawtooth riblets.

of parallel sliding, i.e., -1.6787×10^{-16} N. It is clear that lubricant in the form of transverse relative sliding can provide more load carrying capacity to support the upper flat than that in the form of parallel sliding. With the rise of load carrying capacity of lubrication, the normal pressure, which is proportional to the solid friction, decreased. The above reason resulting in the COF in the form of transverse sliding is obviously smaller than that in the form of parallel sliding.

3.3. Wear in Lubrication

Some previous studies have concluded that the wear in the contact region plays a key role in friction performance. Owing to the existence of roughness at the contact region, the friction and wear were affected seriously [9, 20].

The surface differences between the mirror-finished and textured surfaces result in the difference in the friction and wear mechanisms in lubrication. Schematics of the debris motion during sliding are shown in Fig. 12. The arrows show the route of the debris during the relative sliding of the friction pairs. In the case of the mirror-finished surface, the wear debris is mainly caused by the surface microasperity. Once debris arises in the contact region, it hardly evacuates from there, and such free debris leads to more wear and debris, as shown in the left part of Fig. 12(a). On the sawtooth surface, the wear debris primarily results from the burrs on the ridges of the sawtooth

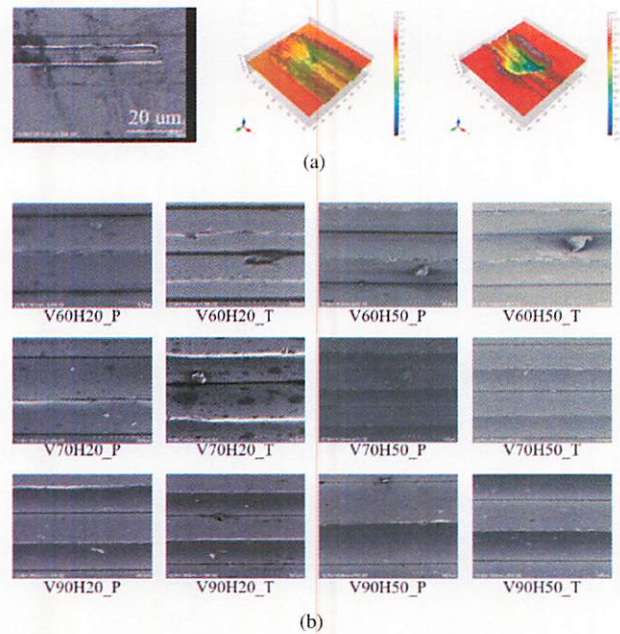


Fig. 13. Debris and scratches on (a) the mirror-finished surface and (b) the sawtooth surfaces.

riblets, as shown in Fig. 12(b). During the relative sliding, some burrs fall off from the ridges of sawtooth riblets and become free wear debris. However, because the valleys of the sawtooth riblets can play the role of channels for discharging the wear debris from the contact region, as shown in the right part of Fig. 12(a), the lower friction and abrasive wear were obtained.

The wear and debris of the mirror-finished and sawtooth surfaces are depicted in Fig. 13. The main difference observed here is that the debris was discharged into the sawtooth valley on the textured surface, as shown in Fig. 13(b); however, it was not discharged from the contact region on the mirror-finished surface, as shown in Fig. 13(a). The debris led to an increasing number of scratches and debris at the contact region on the mirror-finished surface. Meanwhile, the size of the debris decreased during the relative sliding. It can be seen that the size of the linear debris with a length of 1 to 5 μm distributed the edges of the scratches. The average depths of the scratched grooves were near 4 μm. On the sawtooth surface, it is helpful in decreasing the wear and friction that the valleys of sawtooth riblets store and discharge the debris during sliding. It can be seen that the deepest place of the scratched groove on the mirror-finished surface was nearly 6 μm, as shown in the last two figures of Fig. 13(a). However, in Fig. 13(b), the scratches on the ridges of the sawtooth riblets were not so significant as those on the mirror-finished surface. The size of the debris ranged from 5 μm to 20 μm. Moreover, the debris was distributed in the bottom of the valley of the sawtooth riblet and could not influence the sliding friction anymore.

To confirm the mechanism, an energy dispersive X-ray (EDX) analysis for the tested sawtooth surface was

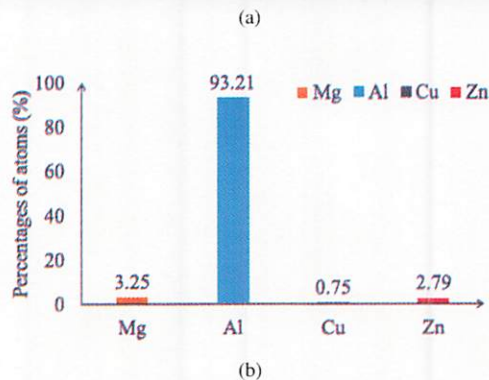
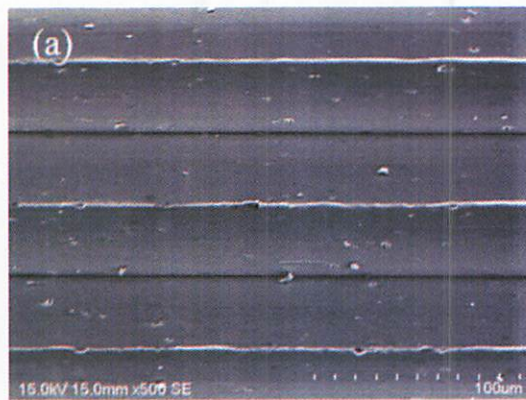


Fig. 14. (a) Tested region and (b) percentages of the elements on the sawtooth surface.

carried out as shown in Fig. 14. Since the microscope slide glass is much harder than A7075, all elements shown in Fig. 14(b) were those that form A7075, and silicon, the main element of the microscope slide, was not found. Namely, the debris was the wear of the sawtooth surface.

4. Conclusions

In this study, the friction reduction of sawtooth riblets was achieved and the mechanism of friction reduction was investigated. The conclusions can be summarized as follows.

1. The sawtooth surface could decrease the sliding friction of the friction pairs by discharging the debris on the contact surface.
2. The sawtooth valley played the role of the channels for evacuating more wear debris from the contact region, which led to a lower friction and longer wear life of the friction pairs.
3. The transverse sliding form provided higher load carrying capacity and lower friction than the parallel sliding form on the sawtooth surface.
4. The friction of the sawtooth surface decreased with an increase in the sawtooth height. The relative small ridge angle was better for reducing the friction of the sawtooth surface based on the experimental results.

As future research, the friction behaviors of micro-patterns such as sawtooth, dimple, and crosshatched grooves under the lubricated condition will be discussed theoretically by using numerical modeling.

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Paper:

Effect of Crystal Structure on Fabrication of Fine Periodic Surface Structures with Short Pulsed Laser

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In recent years, nanostructures have been required for industry and medical services, to perform functions such as reduction in friction, control of wettability, and enhancement in biological affinity. Ultrashort pulsed lasers have been applied to meet these demands, and have been actively studied both experimentally and theoretically in terms of phenomena and principles. In this study, to clarify the phenomenon of the fabrication of laser-induced periodic surface structures (LIPSS), and its application to industry, experiments were conducted on SUS304, titanium, and nickel-phosphorus by a short pulsed laser that has a longer pulse duration, higher cost-effectiveness, and higher stability than ultrashort pulsed lasers. The results confirmed that while LIPSS were fabricated on Ti and Ni-P workpieces, a uniform fine periodic structure was not fabricated on the whole irradiated surface of SUS304, and crystal grain boundaries appeared with low energy density and irradiation number because SUS304 is an alloy composed of Fe, Cr, and Ni. Further, the short pulsed laser has a low power and long pulse duration, inducing the thermal effect. We clarified the effect of crystal structure on fabricating fine periodic surface structures with short pulsed laser.

Keywords: short pulsed laser, crystal structure, crystal orientation, parametric decay, surface plasmon

1. Introduction

Reduction in energy loss, and a safer and more secure society are highly demanded. Hence, particular interest has been shown in the fabrication of fine structures on material surfaces capable of imparting functional properties, such as the reduction in energy loss by reducing the friction of automobile sliding parts [1–3], the improvement in the safety of medical care owing to the enhancement of biocompatibility of implants [4–6], and the improvement in the efficiency of solar light power generation by reducing reflection [7]. For use in the fabrication

of functional surfaces, ultraprecision cutting, ultrasonic-vibration-assisted machining, and photolithography techniques are often employed; however, some disadvantages exist: long processing time, complicated work process, and difficult processing with large surface areas. Therefore, we have chosen the fabrication of fine periodic structures with ultrashort pulsed lasers, hereinafter defined as the less-than-one-picosecond pulse duration laser, as this method creates nanostructures in self-organizing manner by laser irradiation alone [8]. This method can simplify the working processes, shorten the processing time, and significantly reduce energy loss. It will contribute to mitigating the pace of global warming, and therefore, facilitate a safer and more secure society.

Figure 1 shows the processing model when an ultrashort pulsed laser is irradiated on the surface of metal. First, laser irradiation induces surface plasmons causing a deviation in the electric field. Next, ionized atoms cause Coulomb explosions in the large electric field. Finally, ablation occurs in a heat distribution generated on the surface. As mentioned above, fine periodic surface structures are fabricated in self-organizing manner by laser irradiation in this technique [9–17].

The pulse length of lasers is a determinant factor for fabricating nanostructures, as optical phenomena primarily occur when it is shorter than the collisional relaxation time of the object material [15–17]. Therefore, ultrashort pulsed lasers are suitable for the fabrication of nanostructures. However, longer pulsed lasers are favorable from the aspects of cost-efficiency of equipment, and stability of irradiation. Thus, in this study, a short pulsed laser, whose pulse length is 20 ps, is used to fabricate nanostructures on a stainless steel alloy SUS304, titanium, and nickel-phosphorus, and to examine the effects of a picosecond-long pulse length on the processing.

Herein, we report the results considering distorted periodic structures and the non-existence of structures fabricated on the irradiated surface of the alloy.

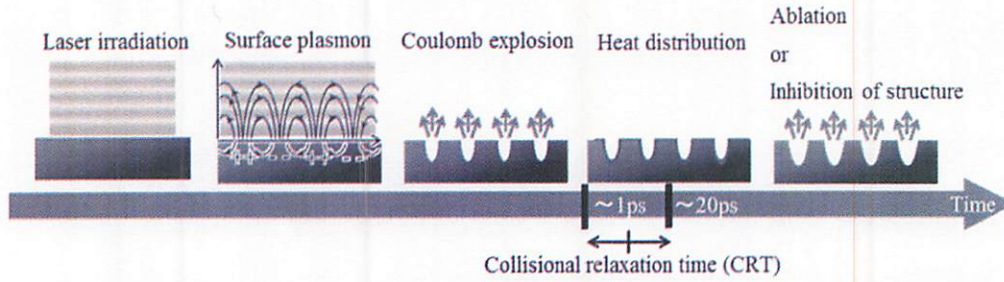


Fig. 1. Processing model of ultrashort pulsed laser.

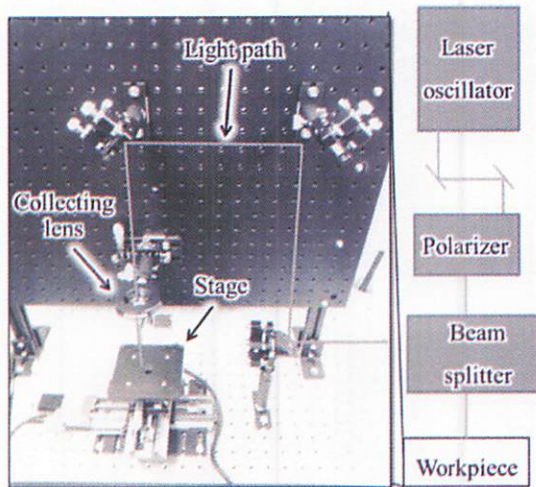
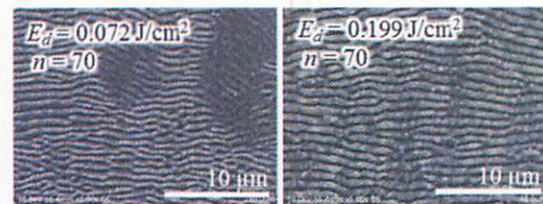


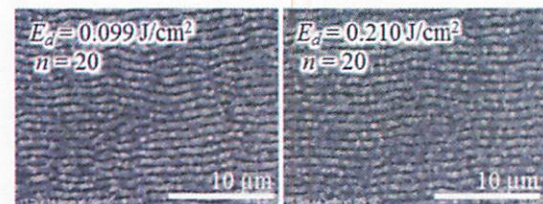
Fig. 2. Setup of laser processing.

Table 1. Experimental conditions.

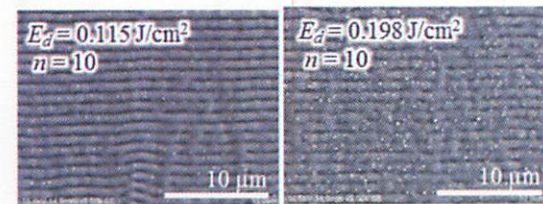
Wavelength	1064 nm
Pulse duration	20 ps
Frequency	50 Hz
Beam spot size	2500 μm
Irradiation number n	1–1000 shot(s)
Energy density E_d	0.011–0.296 J/cm ²
Workpiece	SUS304, Ti, Ni-P



(a) SUS304



(b) Ti



(c) Ni-P

Fig. 3. The SEM images of LIPSS on each material.

2. Fabrication of Fine Periodic Surface Structures on Various Materials via Short Pulsed Laser

Basic experiments were conducted with various materials using the 20-ps pulsed laser to verify the effect of the material.

2.1. Methodology

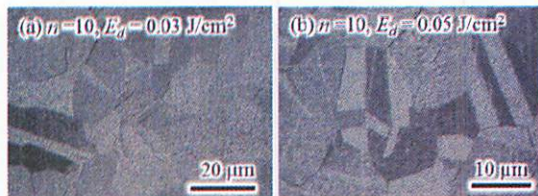
A picosecond pulse laser oscillator (EKSPLA, PL 2250-50P20) was applied in this experiment. Fig. 2 and Table 1 show the setup and the laser irradiation conditions, respectively. The work materials were stainless steel alloy SUS304, titanium, and nickel-phosphorus, which were polycrystalline, single crystal, and amorphous materials, respectively. They were all mirror finished. In an experimental trial, laser with a Gaussian beam profile was irradiated at a fixed point on the sample under the following conditions: the irradiation number was n , the energy density was E_d , and the irradiated points, n and E_d were varied in each trial.

2.2. Results

Laser was irradiated on SUS304, Ti, and Ni-P workpieces to fabricate the LIPSS. Fig. 3 shows the scanning electron microscope (SEM) images of the LIPSS on each material. The LIPSS were fabricated on Ti and Ni-P perpendicular to the polarization when E_d was

Table 2. Experimental conditions.

Wavelength	1064 nm
Pulse duration	20 ps
Frequency	50 Hz
Beam spot size	2500 μm
Irradiation number n	10, 200 shot(s)
Energy density E_d	0.03–0.05 J/cm^2
Workpiece	SUS304

**Fig. 4.** Irradiated areas.

0.1 to 0.2 J/cm^2 . In the case of SUS304, however, under a high power of $E_d = 0.199 \text{ J}/\text{cm}^2$, LIPSS of different pitch lengths and directions were fabricated, whereas no LIPSS were observed on the irradiated area under $E_d = 0.072 \text{ J}/\text{cm}^2$ and $n = 70$, as shown in **Fig. 3(a)**.

SUS304 is an alloy composed of Fe, Cr, and Ni, and the short pulsed laser has a lower power and longer pulse duration, thus inducing the thermal effect compared to an ultrashort pulsed laser [18]. Additionally, the LIPSS were not fabricated on the (111) face of polycrystallines with high lattice defect storage [19]. Thus, it is considered that changes in atomic density with crystal orientation affect the fabrication of LIPSS with SUS304, and the effect of crystal structure on LIPSS is studied in the next section.

3. Effect of Crystal Structure on Laser-Induced Periodic Surface Structure

LIPSS were fabricated on crystalline, single crystal, and amorphous materials. However, either different LIPSS or none were observed on the irradiated area of SUS304 with low n and E_d . The effect of crystal structure on the fabrication of LIPSS was studied.

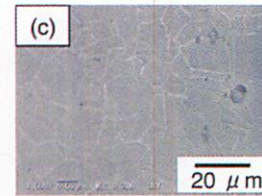
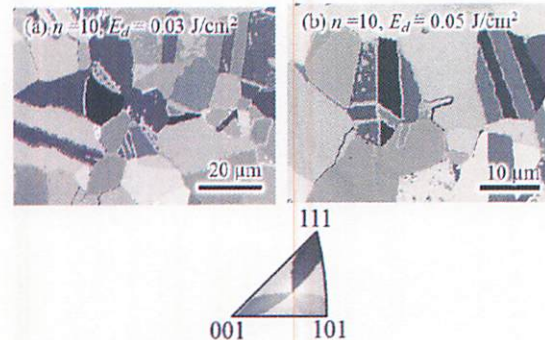
3.1. Methodology

Experiments were conducted using the same device as in the previous section. **Table 2** shows the experimental conditions. Laser was beamed at a fixed point on the workpiece with $E_d = 0.03\text{--}0.05 \text{ J}/\text{cm}^2$ for 10 and 200 shots.

3.2. Results

3.2.1. Irradiated Areas

The irradiated areas on SUS304 workpieces with low n and E_d were observed. **Fig. 4** illustrates the SEM images of the irradiated areas where $n = 10$, $E_d = 0.03$ and

**Fig. 5.** CGBs that appeared with the nanosecond laser [8].**Fig. 6.** EBSD image corresponding to **Fig. 4**.

0.05 J/cm^2 , expressing the surface topographies considered as crystal grain boundaries (CGBs).

CGBs have a lower melting point than crystal grains owing to impurities [20] and high absorption from the disturbed atomic arrangement [21]. Tsukamoto et al. [22] reported that CGBs appeared after nanosecond laser irradiation on SUS304 because the CGBs melted at a low melting point and high absorption, migrated to the top surface owing to the expansion of the surrounding crystal grains, and became an elevated portion after cooling and solidifying. However, as shown in **Fig. 5** [8], the result of CGBs appearing from Tsukamoto et al. is confirmed, in that the appearance is different from the results of our experiment. Therefore, an electron backscattered diffraction (EBSD) pattern analysis is performed to confirm if the structure is caused by the crystal orientation.

Figure 6 shows the EBSD images of the surfaces shown in **Fig. 4**. By comparing **Fig. 4** with **Fig. 6**, we confirmed that the structures appearing after laser irradiation are in accordance with the crystal structures, and it is possible to visualize crystal structure of material surface by short pulsed laser with low n and E_d . Nevertheless, while a nanosecond laser visualizes the CGBs in the previous research, the short pulsed laser expresses crystal structures that are not CGBs. The reason for this will be considered later.

3.2.2. Fabrication of Fine Periodic Structures on Crystal Structures

To confirm the effects of crystal structure on the fabrication of nanostructures with the short pulsed laser, the laser was irradiated on the surface that was preliminarily analyzed with EBSD. First, the laser was irradiated

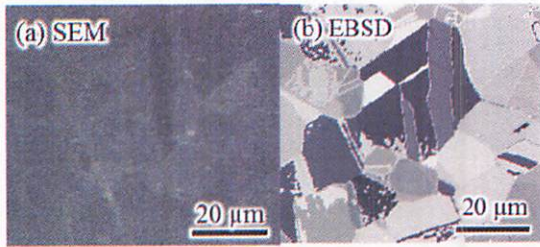


Fig. 7. The surface with $n = 10$, $E_d = 0.03 \text{ J/cm}^2$.

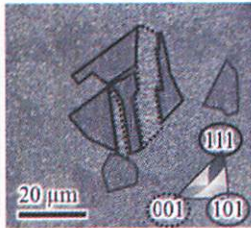


Fig. 8. The surface with $n = 200$, $E_d = 0.05 \text{ J/cm}^2$.

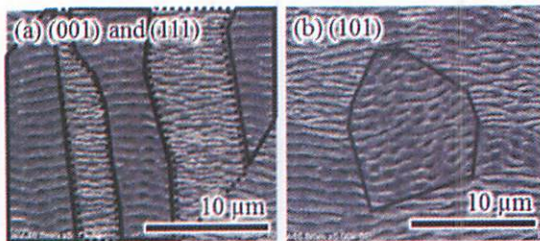


Fig. 9. The large SEM images of face (001), (111), and (101).

with $n = 10$ and $E_d = 0.03 \text{ J/cm}^2$ to decide the irradiation point, whose SEM and EBSD images are shown in Fig. 7. Next, the laser with $n = 200$ and $E_d = 0.05 \text{ J/cm}^2$ was irradiated on the decided point. Fig. 8 shows the SEM images of the result, and the magnified images of faces (001), (101), and (111) are shown in Fig. 9. As shown in these figures, the fabricated structures vary with the crystal orientation planes. They confirm that the pitch length of the nanostructures on (001) are shorter than those on (101) and (111), whose pitch lengths are similar to the laser wavelength, and the structure direction on (101) was not perpendicular to the polarization unlike cases of (001) and (111).

3.2.3. Effects of Crystal Structures on Pitch Length of Nanostructures

The pitch length of periodic structures is calculated by fast Fourier transformation applied to the luminance profiles of the SEM images. While face (001) demonstrated a pitch length of approximately 500 nm, which is half the laser wavelength, faces (101) and (111) exhibited the pitch length of approximately 900 nm, i.e., 0.85 times the laser wavelength.

The reason that the pitch length is 0.5–0.85 times the laser wavelength is explained by the parametric decay by Sakabe et al. [23, 24]: an incident light divides into a surface plasma wave and a scattered wave after changing the surface roughness with laser irradiation. Subsequently, a surface plasma wave interferes with the incident light and nanostructures are fabricated with the pitch length of the surface plasma wavelength. The wavelength of a surface plasma wave is related to the laser power and electron density as follows:

$$\omega_L - \omega_{SP} = ck_{SP} - ck_L \quad (1)$$

$$\omega_L = ck_L \quad (2)$$

$$\omega_{SP}^2 = ck_{SP}^2 - \frac{1}{2}\omega_p^2 - \sqrt{c^4 k_{SP}^4 + \frac{1}{4}\omega_p^4} \quad (3)$$

where subscripts L and SP express the incident light and the surface plasmon, respectively; ω is the frequency; c is the velocity of light in vacuum; k is the wave number; ω_p is the electron plasma frequency. From these equations, the wavelength of a surface plasma wave is half the laser wavelength with low laser power and electron density, e.g., $\omega_p/\sqrt{2} \approx 0$. Meanwhile, it widens to 0.85 times the laser wavelength with an increase in the laser power and electron density, e.g., $\omega_p/\sqrt{2} \approx \omega_L$. It is considered that the pitch length of the nanostructures depends on the electron density of each crystal orientation face.

3.2.4. Expansion of Surface of Each Crystal Orientation Face

The aforementioned previous study suggested that crystal structures are actualized by different expansions of crystal grains and CGBs owing to the difference in melting points. However, the actualizing method in this study is different from that of the previous research. Ito et al. reported that heat conductivity depends on crystal orientation, which increases in the order of (100), (110), and (111), thereby accelerating the electric discharge machining speed in the order of (111), (110), and (100) [25]. They reported that the visualization of crystal structures is attributed to the difference in the expansion rates and heat conductivities among the crystal orientations, and that (111) exhibits the largest expansion. The difference in expansion according to crystal orientation was confirmed by analyzing the three-dimensional topographies with a laser microscope. Fig. 10 shows the laser microscope (KEYENCE, VR-3000) image of the irradiated surface before structure fabrication. It also shows that (001) has a larger expansion than (101) and (111), which is different from the expectation.

The laser microscope image of the irradiated surfaces after structure fabrication is shown in Fig. 11, and shows that (001) has the largest expansion coefficient, the same result as before. From the result, the visualization of crystal structures with the short pulsed laser is due to the different expansion coefficients according to crystal orientation, while the expansion coefficient is different from the expectation.

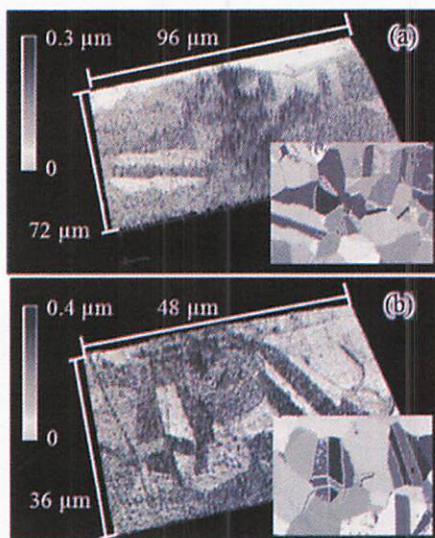


Fig. 10. The laser microscope image of Figs. 4(a) and (b).

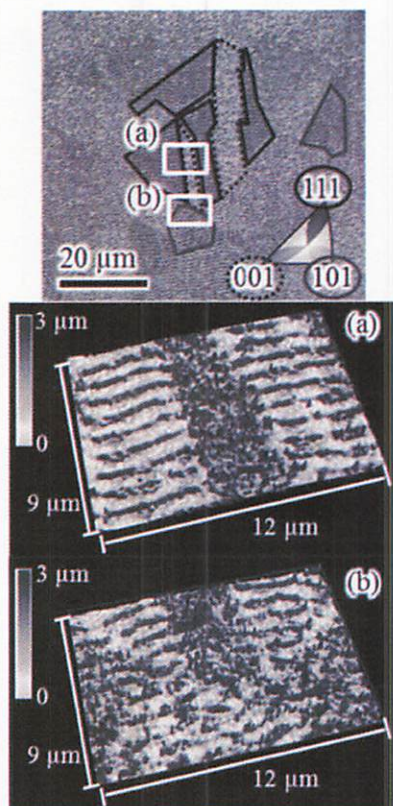


Fig. 11. Laser microscope images of the irradiated surface with $n = 200$, $E_d = 0.05 \text{ J/cm}^2$.

3.2.5. Effects of Crystal Structure on Nanostructure Fabrication

From the atomic structures, it is considered that the expansion coefficients are different from the expectation, and that the fabricated nanostructures vary with the crystal orientation. By comparing (001) with (111), while the

atomic density of the (111) surface is larger, that of (001) in the depth direction is larger [26], thereby rendering the lattice vibration of (001) easier to transfer in the depth direction. Consequently, the evaporation of the (111) surface is larger because the atoms can easily move owing to the layered structure in the depth direction [11, 27]. A (100) surface has a larger heat conductivity as mentioned above. Meanwhile, the heat conductivity of (111) in the depth direction is larger [28]. It is considered that the expansion of the (001) surface is larger because the lattice vibration can easily transform owing to the low heat conductivity and high atomic density in the depth direction.

Although the pitch length of the nanostructures is similar to the laser wavelength, the pitch length of (001) is half the laser wavelength, as explained by the parametric decay owing to the low laser power, low electron density, and high heat conductivity of the surface.

The atomic structure of face (101) is periodic and contains periodic gaps. It is considered that the nanostructures on the (101) surface are not perpendicular to the polarization because Coulomb explosion can easily occur between adjacent atoms, and the periodic atomic structure is not perpendicular to the polarization.

In conclusion, to fabricate uniform nanostructures on the whole surface, it is necessary to increase the laser energy density as various LIPSS with different directions and pitches can be fabricated with only low power, as shown experimentally. Otherwise, the sample of a uniform crystal structure, e.g., single crystal materials and amorphous materials can be used, as LIPSS differ depending on crystal orientation.

4. Conclusion

The effect of crystal structure on the fabrication of nanostructures with short pulsed laser was clarified using the SUS304 alloy in this research. Based on the experimental results and further discussion, the following conclusions were drawn:

1. Crystal structures could be visualized by the irradiation of short pulsed laser with lower irradiation number and energy density on the mirror surface of SUS304.
2. The fabricated structures varied with the crystal orientation as follows:

(001): Nanostructures exhibited a pitch length of half the laser wavelength, and a high aspect ratio owing to the low heat conductivity and atomic density, and large atomic density in the depth direction.

(101): Nanostructures whose pitch length was 0.85 times the laser wavelength was not perpendicular to the polarization because the periodic atomic structure was not perpendicular to the polarization.

(111): The pitch length was 0.85 times the laser wavelength perpendicular to the polarization.

3. It was effective to uniformly fabricate nanostructures on the whole surface by increasing the energy density, and using materials of uniform crystal structure, such as single crystal and amorphous materials.

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Paper:

Unidirectional Wetting Surfaces Fabricated by Ultrasonic-Assisted Cutting

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Surface microstructures can provide various functionalities, and wettability is a typical surface property that can be controlled by surface textures. Unidirectional wetting properties (UWPs) have been garnering attention as a useful wetting function for industrial functions. Thus, in this study, developing UWPs using surface microstructures has been tested. First, UWPs were calculated with the thermodynamic analysis of contact angle (CA). The analytical results predicted that an increased oblique angle of the microstructures, ω_2 , can increase the advancing CA; the receding CAs could not be calculated, and might exhibit the pinning effect. Ultrasonic-assisted cutting was subsequently employed to fabricate hierarchical microstructures for providing UWPs to a workpiece. Although many burrs have been observed on the edges of the structures, microstructures with different oblique angles, $\omega_2 = 5^\circ$, 10° , and 15° , were fabricated in the designed scales. Finally, the UWPs were verified by measuring the CAs and sliding angles (SAs). The anisotropy of CA hysteresis was indicated in each oblique angle structure, and the anisotropy of SAs was confirmed when $\omega_2 = 10^\circ$ and 15° . The retention force ratio of a droplet, r , which indicates the UWPs, was subsequently estimated with two different approaches, and both approaches led a similar value of the attrition rates of r from $\omega_2 = 10^\circ$ to 15° .

Keywords: ultrasonic cutting, functional surfaces, wetting, surface finishing

1. Introduction

Surface microstructures can provide various functionalities including friction reduction, antireflection, and structural colors to materials [1–3], and many examples are shown in nature: moth eyes, shark skin, and gecko feet have provided inspiration for functional surfaces [4–7] and currently biomimetics, which is an academic field pertaining to artificial imitation of such extraordinary natural abilities, has garnered attention to solve human problems [5]. Wetting is a typical surface phenomenon that

is affected by the surface microstructures as well as the surface chemical properties [8, 9]. From the biomimetic viewpoint, natural organisms possess many functional surfaces to control wettability: lotus leaves have ultrahydrophobic surfaces [6], which is widely known as the lotus effect, and the wings of a butterfly (*Morpho aega*) have lamellar-stacked nanometer-sized tips that allow not only waterproof surfaces but also unidirectional sliding such that their bodies can avoid wetting [7]. Zheng et al. [7] explained this unidirectional sliding with the pinning and rolling states of droplets caused by micro- and nanostructures, and indicated that the unidirectional wetting properties (UWPs) can be potentially applied to various smart fluid-controllable interfaces. Chu et al. [10] and Malvadkar et al. [11] fabricated microstructures via selective chemical patterning and the bottom-up vapor-phase technique, respectively, and both studies confirmed UWPs. Additionally, Extrand suggested that the unidirectional wetting in the capillary tube with asymmetric surface structures was generated by the difference in retention forces between both directions [12, 13].

Models and mechanisms to explain the effect of microstructures, or surface roughness, on wetting have been classically studied [8, 9] and the models of Wenzel and Cassie-Baxter are the most famous for describing the effect in the isotropic and static states; however these models cannot express dynamic and/or anisotropic wetting phenomena including the UWPs. To compute such wetting behaviors that cannot be explained with the classic models, a thermodynamic analysis of the contact angle (CA) has been proposed [14–17]. The principle of the analysis is to calculate the change in surface free energies, or surface tensions, of the three-phase boundaries during the droplet transition. The CA on the microtextures is decided where the summation of the free energies, F , minimizes.

Ultrasonic-assisted cutting is a well-known method to reduce the cutting force and improve the quality of the surface finish [18, 19]. Because ultrasonic-assisted cutting is a mechanical removal process, it can be applied on various materials such as metals, ceramics and polymers. Additionally, this method leaves characteristic cutting marks owing to the machining conditions including the vibration frequency, amplitude, and feed rate. Such

micrometer-scaled cutting marks are usually regarded undesirable roughness, but in our study, they have been utilized proactively to shape above-mentioned microstructures [20, 21].

Based on the background mentioned above, we have attempted to calculate wetting with thermodynamic analysis, and subsequently fabricated microstructures [22]. The objective of this study is to fabricate microstructures with ultrasonic-assisted cutting to obtain the UWPs. First, the thermodynamic analysis of the CA was conducted to estimate the effect of the microstructures that will be fabricated. Subsequently, experiments of ultrasonic-assisted cutting were performed to create graded microstructures and their effects on the UWPs were confirmed experimentally.

2. Analysis of Unidirectional Wetting Surfaces

2.1. Fundamentals of Thermodynamic Analysis

The thermodynamic analysis of CA was utilized to calculate the UWPs. As mentioned in the previous section, the summation of the free energy of the system, F , was used:

$$F = \sum_{PB \in \{LG, LS, SG\}} \gamma^{PB} A^{PB}, \dots \dots \dots (1)$$

where γ^{PB} and A^{PB} are the surface tension and area of each phase boundary (PB), respectively; the two-character sets (LG , LS , and SG) indicate two phases forming a boundary; and L , G , and S represent liquid, gas, and solid, respectively. Because this analysis is conducted in a representative cross-section of a droplet, Eq. (1) is modified by neglecting the depth as

$$F' = \sum_{PB \in \{LG, LS, SG\}} \gamma^{PB} l^{PB}, \dots \dots \dots (2)$$

where F' is the free energy in a cross-section, and l^{PB} is the cross-sectional length of each PB. Consequently, the analysis requires the length of the microstructure.

2.2. Details of Analysis

Figure 1 is a schematic of a structure that ultrasonic-assisted cutting is expected to fabricate. The spindle rotation and circular ultrasonic vibration create a trochoid locus; therefore an arc-shaped groove with small grooves remains on the surface during one revolution of the tool. The feed motion allows the fabrication of the same structures intermittently, and the interval of the structure is

$$l_z = \frac{v}{n}, \dots \dots \dots (3)$$

where v is the feed rate and n is the rotation number. The oblique angles of the structures, ω_2 and ω_3 in **Fig. 1(b)**, depend on the shape of the tool; it is noteworthy that the subscripts 2 and 3 are referred to the notation in [12]. The trochoid-shaped grooves shaped in the xz plane are expected to operate as microchannels deciding the flow

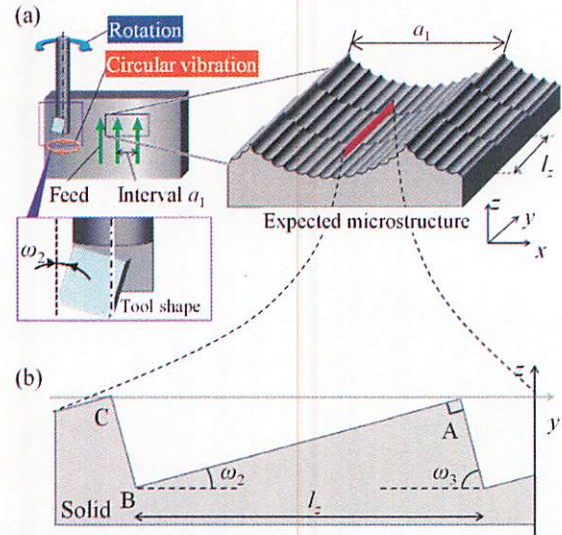


Fig. 1. Schematic of a structure that ultrasonic-assisted cutting is expected to fabricate. (a) Brief fabrication procedure, (b) representative cross-section of yz plane.

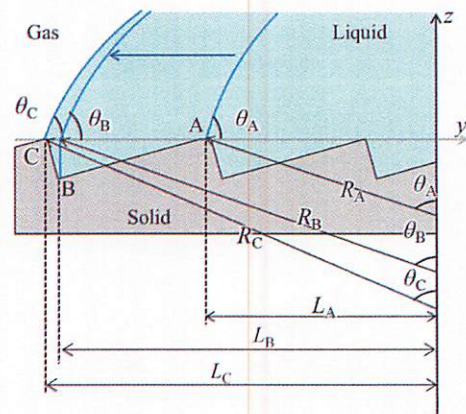


Fig. 2. Advancement of droplet and geometric parameters.

direction, and the oblique structures are expected to provide the pinning and rolling effects mentioned in [7]. Because the objective of this study is to fabricate surfaces with UWPs, the representative cross-section of analysis was set in the yz plane as shown in **Fig. 1(b)**. In addition, the following two assumptions were introduced to simplify the calculation:

1. The cross-sectional shape of the droplet is a circular segment.
2. The cross-sectional area is constant during the droplet transform.

Let us assume that the contact point advances from A to B ($A \rightarrow B$) and subsequently from B to C ($B \rightarrow C$) as shown **Fig. 2**. The change in the free energy can be expressed as follows:

$$\Delta F_{X_1 \rightarrow X_2} = F_{X_2}, \dots \dots \dots (4)$$

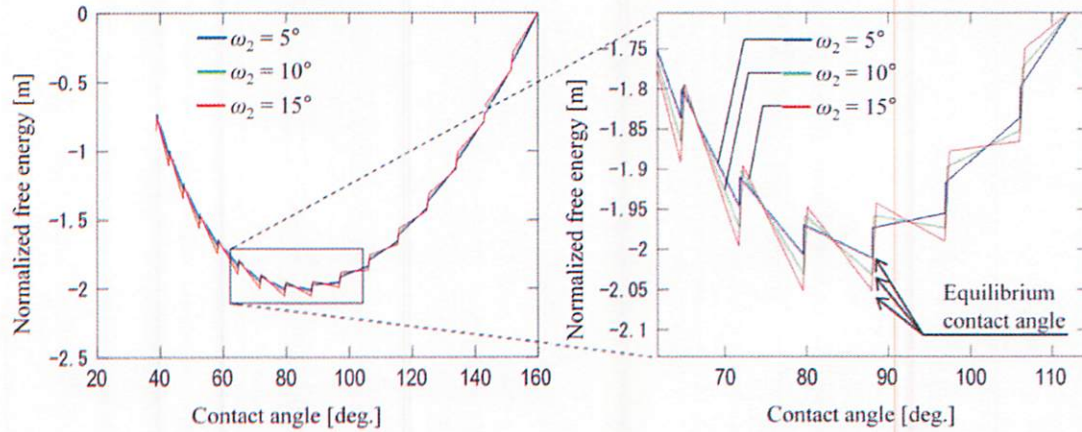


Fig. 3. Calculated relationships between contact angle and normalized free energy.

where F_X is the free energy of the system when the contact point is at an arbitrary point X , which can be written as

$$F_X = \gamma^{LG} l_X^{LG} + \gamma^{LS} l_X^{LS} + C. \quad (5)$$

C is an arbitrary surface energy that is constant during the droplet advance [14]. Firstly, let $\Delta F_{A \rightarrow B}$ be considered. Each l^{PB} related to $A \rightarrow B$ can be obtained geometrically as follows:

$$l_A^{LG} = \frac{2\theta_A L_A}{\sin \theta_A}, \quad (6)$$

$$l_B^{LG} = \frac{2\theta_B L_B}{\sin \theta_B} + 2l_z \sin \omega_2 \cos \omega_2, \quad (7)$$

$$l_A^{SG} = l_B^{SG} = l_z \cos \omega_2, \quad (8)$$

where θ_X is a temporary CA when the contact point is at X ; L_X is from the center of the droplet to X as shown in Fig. 2.

Using the equations above, the normalized change in the free energy is expressed as

$$\frac{\Delta F_{A \rightarrow B}}{\gamma^{LG}} = 2 \left(\frac{\theta_B L_B}{\sin \theta_B} - \frac{\theta_A L_A}{\sin \theta_A} \right) - l_z \cos \omega_2 (\cos \theta - 2 \sin \omega_2). \quad (9)$$

It is noteworthy that the normalized free energy, $\Delta F_{A \rightarrow B} / \gamma^{LG}$, requires the initial CA θ , such that a relative comparison can be performed without actual surface tensions.

θ_B can be calculated with the assumptions that result in the following constraint equation:

$$\frac{\theta_A L_A^2}{\sin^2 \theta_A} - \frac{L_A^2}{\tan \theta_A} = \frac{\theta_B L_B^2}{\sin^2 \theta_B} - \frac{L_B^2}{\tan \theta_B} + l_z^2 \sin \omega_2 \cos^3 \omega_2, \quad (10)$$

where,

$$L_B = L_A + l_z \cos^2 \omega_2. \quad (11)$$

Similarly, $\Delta F_{B \rightarrow C}$ and the constraint equation can be expressed as follows:

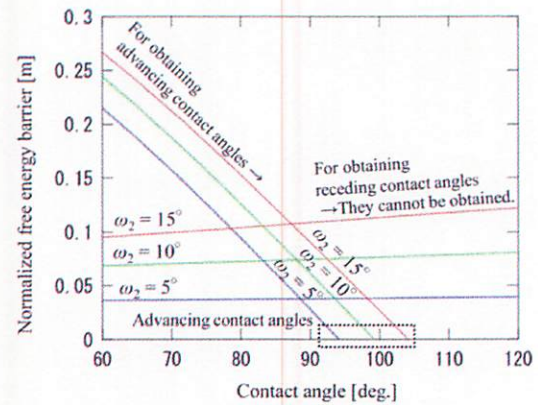


Fig. 4. Calculated normalized free energy barrier.

$$\frac{\Delta F_{B \rightarrow C}}{\gamma^{LG}} = 2 \left(\frac{\theta_C L_C}{\sin \theta_C} - \frac{\theta_B L_B}{\sin \theta_B} \right) - l_z \cos \omega_2 (\cos \theta + 2 \sin \omega_2), \quad (12)$$

$$\frac{\theta_B L_B^2}{\sin^2 \theta_B} - \frac{L_B^2}{\tan \theta_B} = \frac{\theta_C L_C^2}{\sin^2 \theta_C} - \frac{L_C^2}{\tan \theta_C} + l_z^2 \sin^3 \omega_2 \cos \omega_2, \quad (13)$$

$$L_C = L_B + l_z \cos^2 \omega_2. \quad (14)$$

2.3. Calculation Results

Three oblique angles, $\omega_2 = 5^\circ, 10^\circ$, and 15° , were set in the analyses by considering the actual tools. Other analytical conditions were set to the following: $l_z = 200 \mu\text{m}$, the intact CA $\theta = 80^\circ$, $L_1 = 0.5 \text{ mm}$, the initial CA $\theta_1 = 160^\circ$. GNU Octave was used to program and solve the equations.

Figures 3 and 4 show the calculated free energy diagrams and free energy barrier (FEB) diagrams, respectively. The free energy is decided at the minimum point of the diagram, and the x-intercept of the FEB indicates the advancing and receding CAs [14]. The advancing CAs

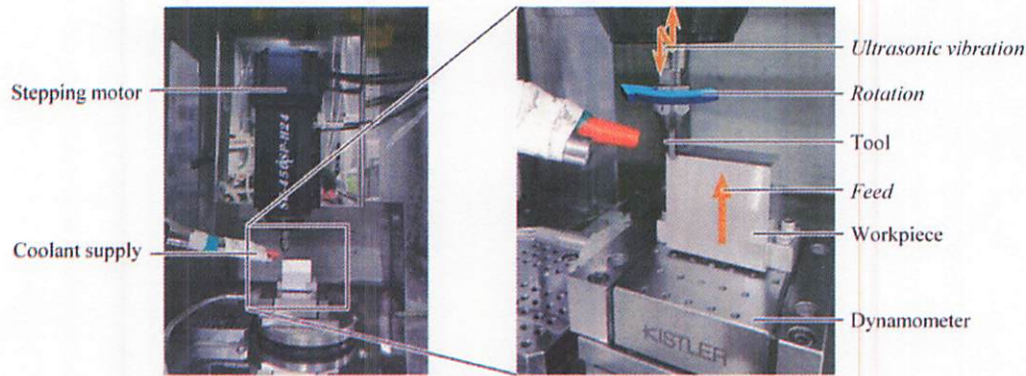


Fig. 5. Setup of ultrasonic-assisted cutting for fabrication of microstructures. Italic characters indicate motion directions.

could be obtained; however, as the receding CAs became less than 0° , they could not be calculated. This result illustrates that receding FEBs exist however small the CA becomes. This means that the receding of the droplet would be blocked by the FEB even if the CA were 0° , which was considered to signify the pinning effect mentioned in [7]. In addition, an increased ω_2 results in an increased FEB, thus the sliding of the droplet will be disturbed.

3. Experiments

3.1. Ultrasonic-Assisted Cutting for Fabricating Microstructures

3.1.1. Conditions

Ultrasonic-assisted cutting was performed using a spindle with three-directional ultrasonic generators (SC-45 SP-H24, Taga Electric Co., Ltd.), which was mounted on a four-axis desktop CNC machining table (Trider-X, Nexsys Corp.). The contact between the tool and the workpiece was detected with a dynamometer (Type 9256A, Kistler Instrument Corp.) placed beneath the workpiece jig. The workpiece material was a tough pitch copper C1100, which is a cheap plastic molding material. Fig. 5 depicts the setup of the experiments and the motion directions. Fig. 6 shows scanning electron microscope (SEM) images of the employed diamond tools. Their nose radii were 0.01 mm and they have different edge angles of 5° , 10° , and 15° , as mentioned in the previous section. Table 1 lists the details of the machining conditions. To avoid excessive friction and wear on the tool tips, the cutting depth d was set to a constant, $20\text{ }\mu\text{m}$, and v was varied.

3.1.2. Results

Figures 7 and 8 are SEM and white light interferometer (WLI) images of fabricated microstructures, respectively. Micrometer-scaled hierarchical structures are shown in each condition. Compared to microstructures attributed to ultrasonic circular vibration, the rightmost figures of Fig. 7, the cutting locus can be observed when $\omega_2 = 5^\circ$;

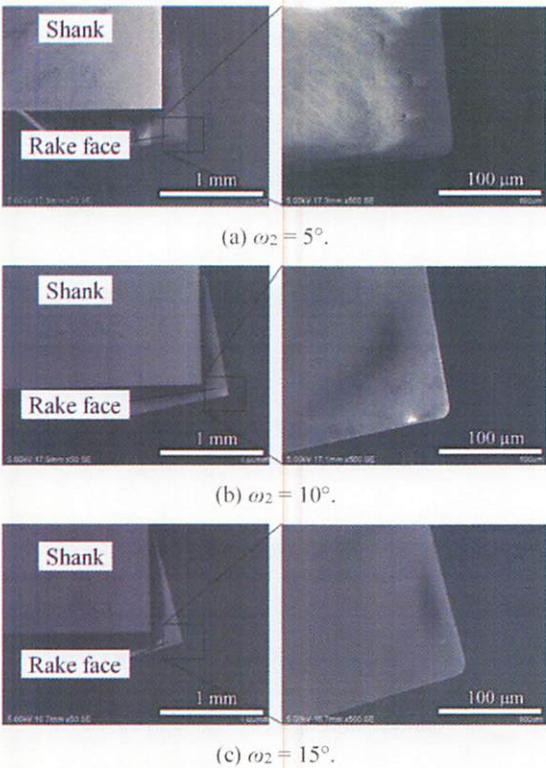


Fig. 6. SEM images of tool tips.

Table 1. Experimental conditions.

Oblique angle ω_2 [deg]	5	10	15
Rotation number n [min^{-1}]		1000	
Feed rate v [mm/min]	200	100	70
Structural spacing l_z [μm]	200	100	70
Height of structure [μm]	17.4	17.6	18.8
Vibration amplitude A_c [μm]		15	
Feed interval a_1 [μm]		400	
Cutting depth d [μm]		20	

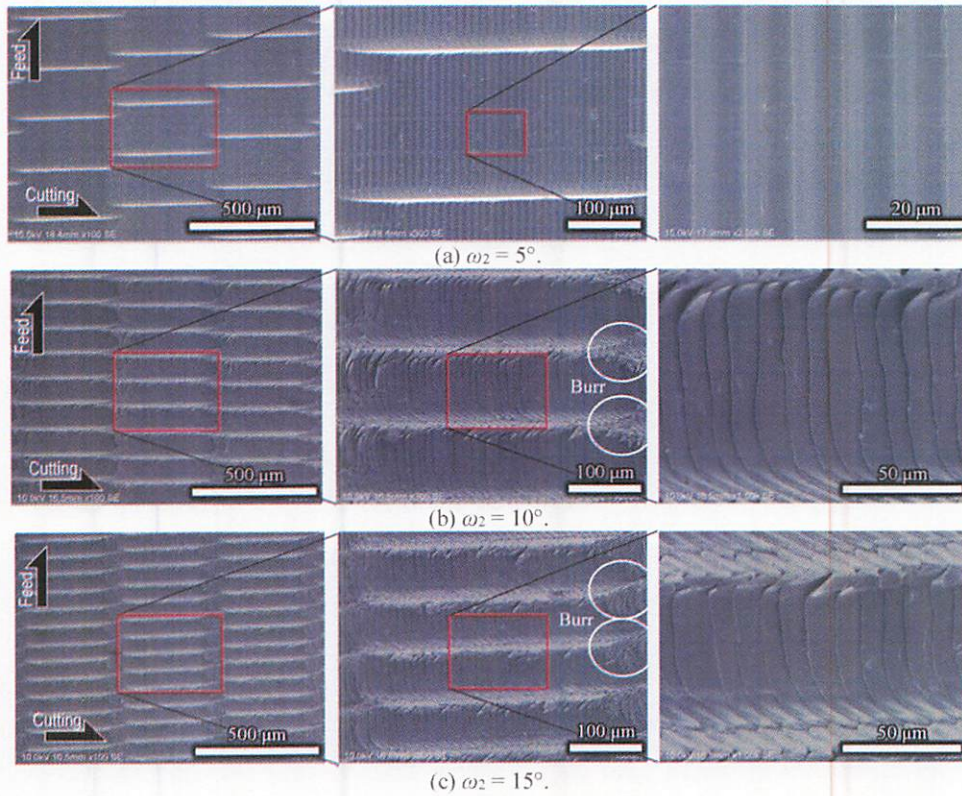


Fig. 7. SEM images of fabricated microstructures.

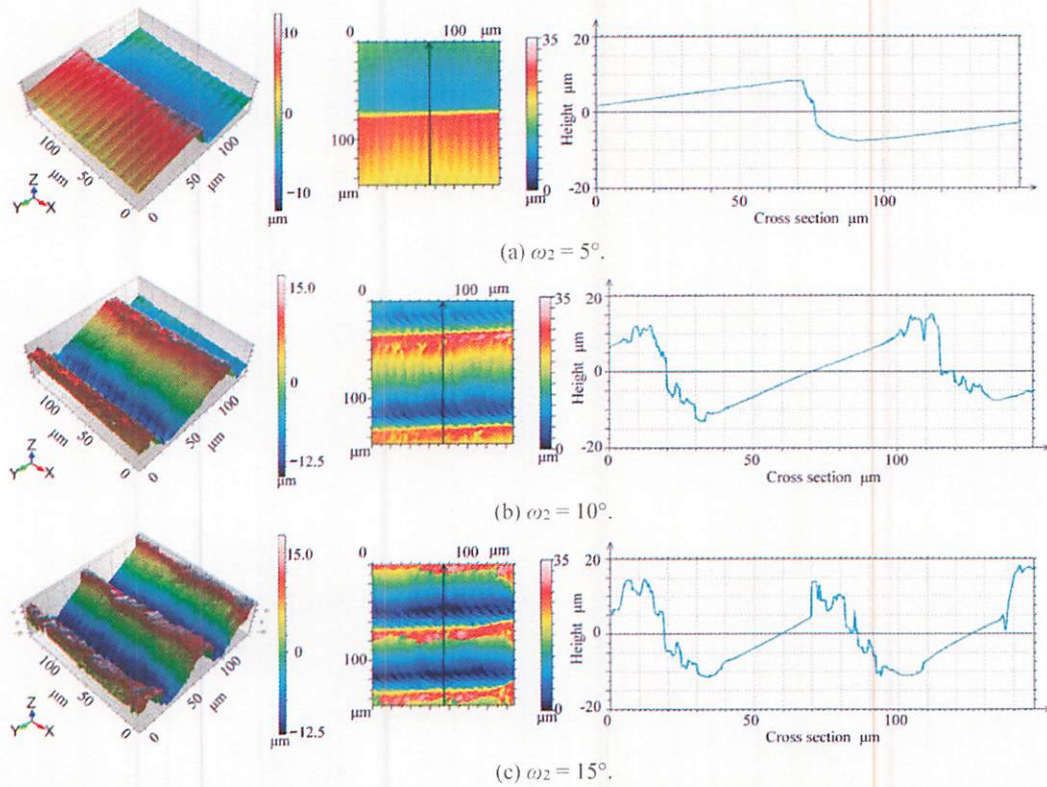


Fig. 8. White light interferometer images and cross-sectional profiles of fabricated microstructures.

meanwhile, lamella structures were formed when $\omega_2 = 10^\circ$ and 15° . These results might be owing to the geometrical constraint of material removal. The more ω_2 increases, the more geometrically limited is the direction of material removal. Thus, the material might remain more easily and be stacked as shown in the rightmost images of Figs. 7(b) and (c).

3.2. Measurement of Unidirectional Wetting Properties

Unidirectional wetting properties were obtained by measuring the CA of the microstructured surfaces. A CA meter (DM-501, Kyowa Interface Science Co., Ltd.) was employed to detect the CA and sliding angle (SA). During SA measurement, the inclination angle of the workpiece table increases from 0° to 90° within 30 s, and a charge-coupled device camera captures the shape of the droplet to compute the advancing and receding CAs at every 1° inclination. The sliding angle was automatically detected and recorded by verifying the advance of the contact point.

Before the measurement, the workpiece experienced ultrasonic cleaning (i) in ethanol for 10 min, (ii) in purified water for 5 min, and subsequently once again (i) and (ii). After cleaning, a fluorochemical release agent (Optool HD, Daikin Industries, Ltd.) was coated on the microstructured surfaces because it had been confirmed in the preliminary experiment that the microstructures enhanced the hydrophilicity of copper and a droplet had spread unmeasurably. The cleaned workpiece was subsequently set on the table and a $5\text{-}\mu\text{L}$ purified water droplet was deposited on it. Each sample was tested 10 times.

The results of the measurements are shown in Fig. 9. The subscript plus and minus signs of y indicates the sliding direction, having definition as shown in Fig. 1. Every microstructured surface became more hydrophilic than the mirror-finished surface whose CA was 106° when the inclination angle was 0° . Specifically, the CA increased by 34% and 29% when $\omega_2 = 10^\circ$ and 15° , respectively. Each surface also shows the CA anisotropic hysteresis. Droplet sliding occurred 10 times out of 10 trials (10/10) for the mirror-finished surface; the mean value of SA was 47.8° , and the CA hysteresis, $\Delta\theta$, was 13.3° . When $\omega_2 = 5^\circ$, the droplet had never slid to either direction, y_+ or y_- . Meanwhile, sliding occurred 10/10 to y_- and 8/10 to y_+ when $\omega_2 = 10^\circ$, and 4/10 to y_- , and 1/10 to y_+ when $\omega_2 = 15^\circ$, which demonstrate the UWPs.

4. Discussion

The thermodynamic analysis of the CA predicted that the advancing CA increases with ω_2 , as confirmed between $\omega_2 = 5^\circ$ and 10° ; however, this did not apply between $\omega_2 = 10^\circ$ and 15° . Meanwhile, the CA hysteresis increased with ω_2 .

From the difference shown in the microphotographs of Figs. 7 and 8, it can be considered that burrs might affect

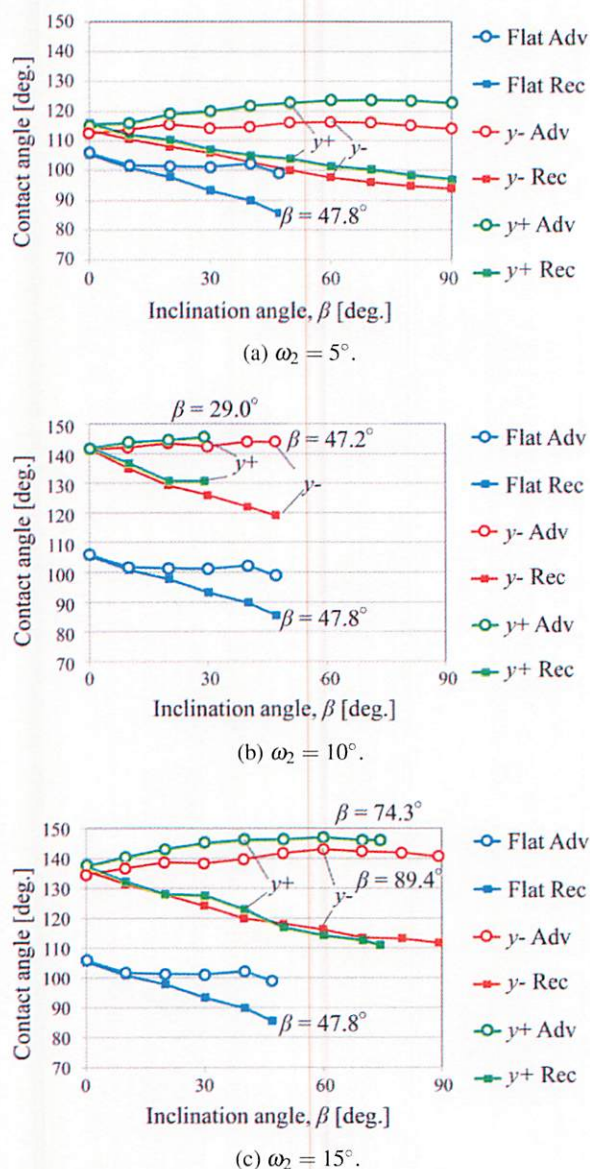


Fig. 9. Inclination angle and contact angle hysteresis.

the sliding behavior of the droplet.

The effect of microstructures on the UWPs was subsequently investigated with Extrand's equation that expresses the retention force anisotropy [12]:

$$r_{\text{Ext}} = \frac{f_3}{f_2} = \frac{\sin\left(\omega_3 + \frac{1}{2}\Delta\theta\right)}{\sin\left(\omega_2 + \frac{1}{2}\Delta\theta\right)}, \quad \dots \quad (15)$$

where $\Delta\theta$ is the CA hysteresis of the inherent surface; f_2 and f_3 are the retention forces whose subscripts correspond to the direction referring to Fig. 1. This equation originally explains the ratio of the retention forces in a capillary tube; however, it is also applied for explaining the anisotropic wetting of a plane [11]. The retention force of a droplet in the sliding test is the adhesion force

against the gravitation. Consequently, when the sliding angle is β , the retention force f_g is expressed with the mass of a droplet m and the gravitational acceleration g as:

$$f_g = mg \sin \beta. \quad (16)$$

The measured retention forces f_{y+} and f_{y-} are represented by the mean values; subsequently, their ratios, r_{exp} , is

$$r_{\text{exp}} = \frac{f_{y-}}{f_{y+}} = \frac{\frac{1}{N} \sum_{i=1}^N m_{i,y-} g \sin \beta_{i,y-}}{\frac{1}{N} \sum_{i=1}^N m_{i,y+} g \sin \beta_{i,y+}}. \quad (17)$$

where N is the number of trials, where the subscript i means the i -th trial.

Using the experimental results and assuming that $\beta = 90^\circ$ when sliding did not occur, r_{exp} was calculated as 1.43 and 1.12 when $\omega_2 = 10^\circ$ and 15° , respectively, and the attrition rate was 22%. In contrast, by substituting the mirror-finished surface $\Delta\theta = 13.3^\circ$ to Eq. (15), $r_{\text{Ext}} = 3.49$ when $\omega_2 = 10^\circ$ and $r_{\text{Ext}} = 2.68$ when $\omega_2 = 15^\circ$. The attrition rate was 23%, similar to the former rate.

5. Conclusion

Surfaces with UWP were used for fabrication in this study. First, the thermodynamic analysis of CA was conducted to analyze the effect of the microstructures on the properties. The analytical results illustrated that the increase in inclination angle of the microstructures increased the advancing CA; however, the receding angles could not be calculated. The inability in calculation might indicate the pinning effect of the structures. Ultrasonic-assisted cutting was subsequently employed to fabricate microstructured surfaces. Although burrs and lamellar structures were observed, the microstructures could be fabricated in the designed scales. Finally, the microstructured surfaces were examined with a CA meter. The anisotropy of the CA hysteresis and the SA was confirmed when $\omega_2 = 10^\circ$ and 15° . The decrease ratios of the retention force ratios exhibited similar values.

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Control of short-pulsed laser induced periodic surface structures with machining - Picosecond laser micro/nanotexturing with ultraprecision cutting

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ABSTRACT

Short-pulsed laser in the fabrication of fine periodic surface structures (hereinafter, referred to as LIPSS: laser induced periodic surface structures) has been studied to reduce friction, improve control of wettability and improve bioaffinity. While it has low cost and stable irradiation compared with the ultrashort-pulsed laser traditionally used, it is difficult to control the fabrication of LIPSS using the short-pulsed laser because of its lower peak fluence than the ultrashort-pulsed laser and the unclarified principle. Optimizing the surface shape before laser irradiation may make it easier to control fabrication using short-pulsed laser since it has been reported that LIPSS is fabricated due to surface plasmons induced by plasma waves after altering the surface roughness during laser irradiation. The proposed method involves cutting the surface to assist with the short-pulsed laser for the control LIPSS: the surface of the workpiece is irradiated after straight microgrooves are fabricated by ultraprecision cutting. The experimental results showed that microgrooves made straight LIPSS with a high aspect ratio and also expanded the effective fluence range of experimental conditions when fabricating LIPSS. The new method was shown to control LIPSS effectively, and highlights the importance of the surface shape before laser irradiation for LIPSS fabrication.

1. Introduction

Fine structures have been studied largely because their characteristics make them well suited for use applications designed to improve environmental issues. They typically are characterized by a reduction in friction [1–3], the antireflection of light [4] and an improvement of bioaffinity [5]. The fabrication of fine structures with a short-pulsed laser has many advantages and solves the problems associated with the traditional fabrication methods. However, because the principles of the phenomenon have yet to be completely clarified [6–9], controlling fine structures with a short-pulsed laser remains a challenge. Fig. 1 illustrates a scanning electron microscope (SEM) image of fine periodic surface structures (hereinafter, referred to as LIPSS) fabricated on nickel phosphorus (Ni-P) plating by a short-pulsed laser. The distorted structures that can be clearly seen in this image result in poor functionality. It has been reported that LIPSS favorably occurs around debris and depressions because plasma waves are readily induced by such surface features, as shown in Fig. 2 [10]. To produce LIPSS with more

desirable characteristics, we focused on the surface texture before laser irradiation. While it has been reported that LIPSS is controlled by using two lasers [11,12], there is a few reports to control LIPSS by changing a surface shape. In our new method, ultraprecision cutting is carried out prior to irradiation with a short-pulsed laser to fabricate straight LIPSS with a high aspect ratio. That is, ultraprecision cutting is employed to produce straight microgrooves several-micrometers in height with nanometer precision on the workpiece before irradiating the surface with short-pulsed laser.

The objective of this study is to control the LIPSS of short-pulsed laser with ultraprecision cutting and to investigate the effect of the surface texture prior to laser irradiation on the fabrication of LIPSS. First, a finite-difference time-domain (FDTD) simulation was performed to investigate the effects of the electric field intensity distributed by a straight microgroove. Then, the workpiece was flattened and straight microgrooves were fabricated on its surface by ultraprecision cutting with a round chip and a triangle chip, respectively. Finally, the prepared surface was irradiated with a short-pulsed laser to fabricate

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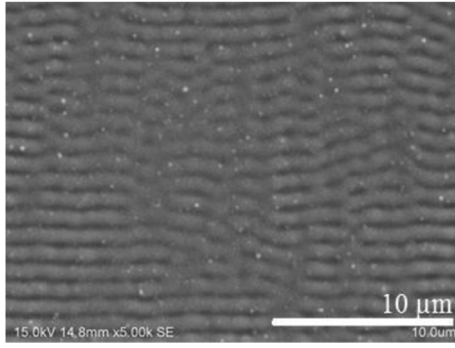


Fig. 1. SEM image of LIPSS with short-pulsed laser.

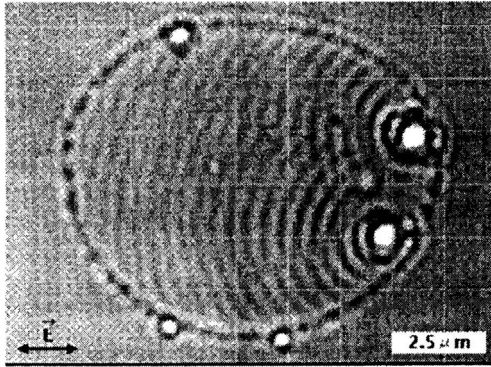


Fig. 2. Micrograph of LIPSS with femtosecond laser [10].

straight LIPSS.

2. Principles

Fig. 3 illustrates the mechanism of LIPSS fabricated by a short-pulsed laser: laser induces the surface plasmons as collective vibration of free electrons due to the plasma wave or the change of electric field intensity distribution due to interference between incident laser and scattered laser. Then, the Coulomb force among the ionized atoms results in a Coulomb explosion and the material surface is removed periodically. After the collisional relaxation time (CRT), heat distribution results in either the ablation or inhibition of the structure depending on the characteristic property of the material. LIPSS is fabricated in this manner [13,14]. It has been reported that LIPSS are fabricated when the pulse duration of the laser is shorter than the CRT, and that the resultant LIPSS has better clarity and is more readily formed when the pulse duration of the laser is in the femtosecond range [15]. This highlights the importance of CRT as a factor in the fabrication of LIPSS. In our study, a pulse duration of 20 ps was employed due to the lower cost of lasers with longer pulse duration and because the irradiation is more stable. At 20 ps, however, it is difficult to fabricate and control

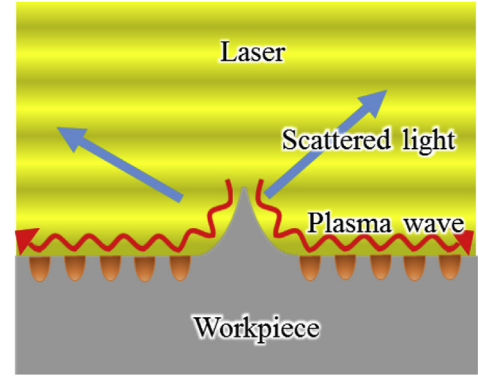


Fig. 4. Geometry of the parametric decay.

LIPSS due to thermal influence and low peak fluence.

Surface plasmons induced by plasma waves are another important factor in the fabrication of LIPSS. Sakabe et al. explained this mechanism in terms of parametric decay [16,17]: laser irradiation alters the surface roughness dividing incident light into scattered light and the plasma wave parallel to laser polarization, and inducing surface plasmons due to an interference between the incident light and the plasma wave as shown in Fig. 4. The surface roughness is clearly an important factor in inducing surface plasmons. The wavelength of a surface plasma wave can be expressed as:

$$\omega_L - \omega_{SP} = ck_{SP} - ck_L \quad (1)$$

$$\omega_L = ck_L \quad (2)$$

$$\omega_{SP}^2 = ck_{SP}^2 + \frac{1}{2}\omega_p^2 - \sqrt{c^4 k_{SP}^4 + \frac{1}{4}\omega_p^4} \quad (3)$$

where subscripts L and SP express the incident light and the surface plasmons, respectively, ω is the frequency, c is the velocity of light in vacuum, k is the wave number and ω_p is the electron plasma frequency. Fig. 5 shows the relationship between the wavelength of a surface plasma wave and the plasma frequency obtained with these equations. Note that the wavelength of the surface plasma wave is 0.5–0.85 times as long as that of the laser.

In addition, since dispersion relations of the incident light and the plasma wave are not in accord when laser is irradiated perpendicular to a flat surface, surface roughness is undoubtedly a determining factor in the fabrication of LIPSS. In order to control LIPSS, ultraprecision cutting prior to the use of a short-pulsed laser is proposed. A laser is irradiated on the straight microgrooves produced by ultraprecision cutting as the laser polarization direction is perpendicular to the microgroove direction since LIPSS are fabricated perpendicular to the laser polarization.

3. Simulation

A finite-difference time-domain (FDTD) simulation was performed to investigate the effect of a straight microgroove on the electric field

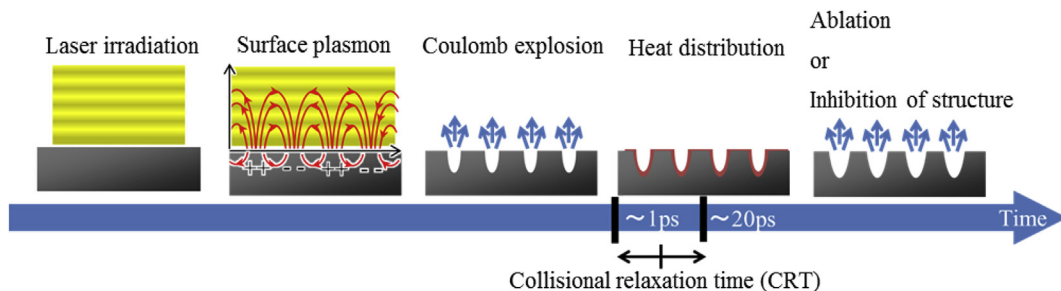


Fig. 3. Processing model of short-pulsed laser.

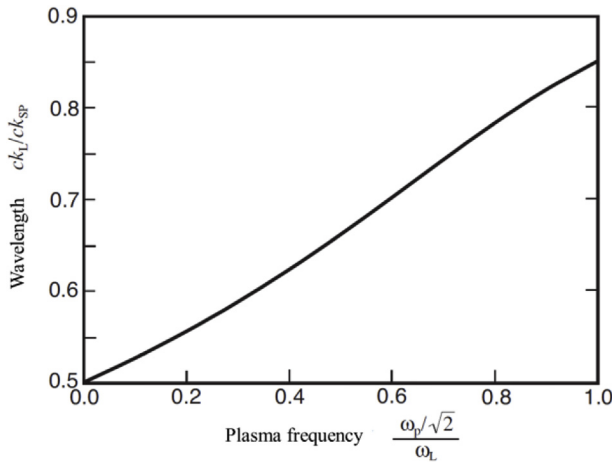


Fig. 5. Wavelength of plasma wave with frequency [16].

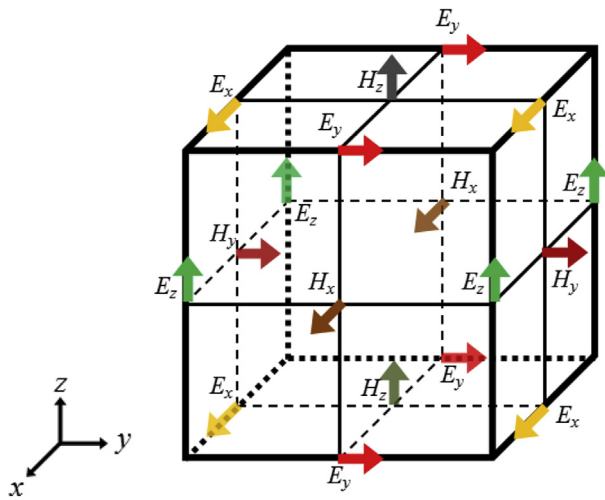


Fig. 6. Spatial arrangement of electric and magnetic fields.

intensity distribution, which brings fabrication of LIPSS. The FDTD is a numerical algorithm for solving the time dependent Maxwell's equations in differential form, introduced by Yee in 1966 [18–22]. The spatial arrangement referred to as Yee cell is shown in Fig. 6.

3.1. Method

FDTD simulation (Lumerical's nanophotonic FDTD simulation software) was used in electromagnetic field analysis, the analytical model of which can be seen in Fig. 7. Laser is irradiated on a flat surface and a surface with a microgroove. The analytical conditions are listed in Table 1. Nickel phosphorus Ni-P, an amorphous material, was used as the substrate.

3.2. Results and remarks

The analytical results are provided in Fig. 8. While the electric field

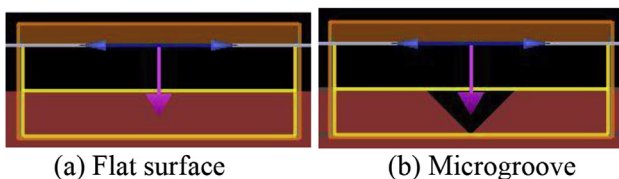


Fig. 7. Analytical model.

Table 1
FDTD analytical conditions.

Laser wavelength	900 nm
Pulse duration	20 ps
Material	Ni-P
Groove depth	0, 1 μ m
Boundary condition	PML

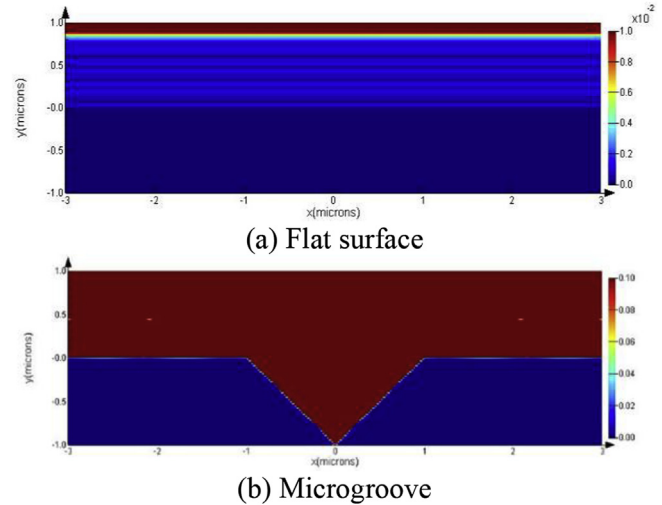


Fig. 8. Analytical result.

was not confirmed on the flat surface, it was periodically detected on the grooved surface. The reason is that surface plasmons are not induced by g perpendicular laser irradiation to a flat surface [23]. On the other hand, surface plasmons are more readily induced on the surface of the workpiece with straight grooves than on the flat surface, and they follow the grooves allowing straight LIPSS to be fabricated. Based on the simulation results, an expected LIPSS on the workpiece with straight microgrooves was drawn as shown in Fig. 9.

4. Experiments

The proposed method to control LIPSS was investigated experimentally. Straight microgrooves were produced on the flattened surface of a workpiece by ultraprecision cutting using an apparatus capable of fabricating microstructures precisely on various materials including nickel phosphorus [24–27], and the surface was then irradiated with a short-pulsed laser. Subsequently, images of the irradiated surfaces were taken with a scanning electron microscope (SEM), and the pitch length and the height of LIPSS were evaluated to investigate the effects of the straight microgrooves on the fabricated LIPSS.

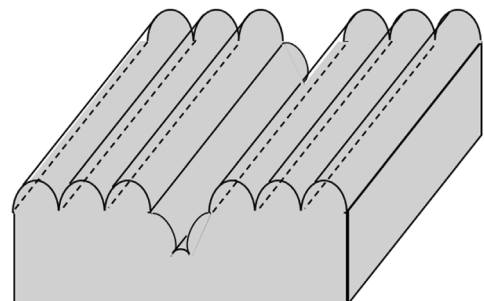


Fig. 9. Expected shape.

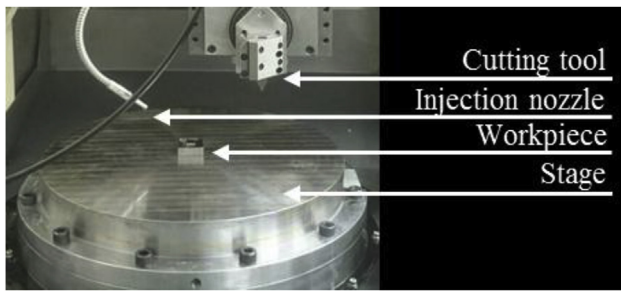


Fig. 10. Cutting setup.

Table 2
Flattening conditions.

Tool	Diamond
Round nose R	2 mm
Material	Ni-P
Depth of cut	10 μm
Pitch f	20 μm
Cutting speed	10000 mm/min
Atmosphere	Oil

4.1. Experimental method

A five axis ultraprecision machine (Nagase Integrex Co., Ltd. MIC300), shown in Fig. 10, was employed to flatten the surface of the workpiece and to produce microgrooves with the conditions listed in Tables 2 and 3, respectively. The work material was Ni-P, an amorphous material. After cutting, experiments were conducted using a picosecond pulse laser oscillator (EXPLA, PL 2250-50P20). Fig. 11 and Table 4 show the setup and the laser irradiation conditions, respectively. The microgrooves with different pitch lengths and depths were prepared to investigate the effects of them on the fabrication of LIPSS. The laser with a Gaussian beam profile was focused on a fixed point on the flat surface and the microgrooved workpiece.

4.2. Results and remarks

The surface roughness R_a and R_z of the flattened workpiece were 5.37 nm and 20.01 nm, respectively. Fig. 12 shows a laser microscope image of the microgrooved surface. SEM images of LIPSS on different pitch depths and lengths are shown in Fig. 13. These figures indicate that a short pitch length is better to control LIPSS. In the case of the pitch length of 50 μm , the LIPSS was destroyed at the area far from microgrooves. It is also clear from Fig. 13 that straight LIPSS were fabricated on the processed surface compared to LIPSS on the flat surface. Fig. 14 illustrates the effective range, where LIPSS were actually fabricated, of experimental conditions when LIPSS was fabricated on the flat surface and the surfaces with grooves of 2 μm in depth and pitch lengths of 10 and 50 μm . From these results, it can be concluded that microgrooves facilitate the fabrication of LIPSS since the parametric decay favorably occurs at the microgrooves and surface plasma waves are readily induced.

Figs. 15 and 16 show the pitch length and the height of the

Table 3
Microgrooving conditions.

Tool	Diamond
Nose angle	90°V
Material	Ni-P
Depth of cut	1, 2 μm
Pitch f	10, 50 μm
Cutting speed	10000 mm/min
Atmosphere	Oil

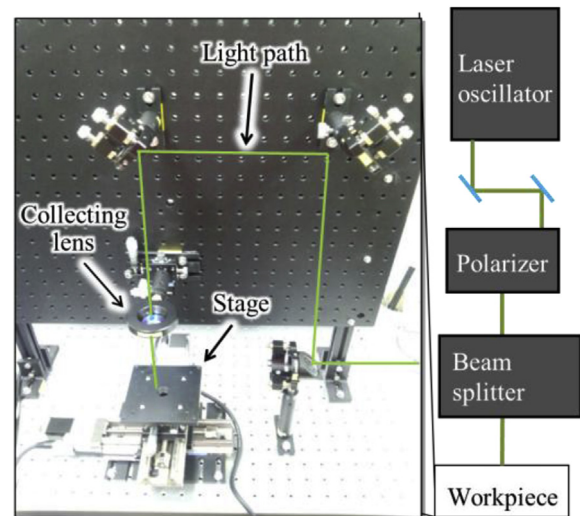
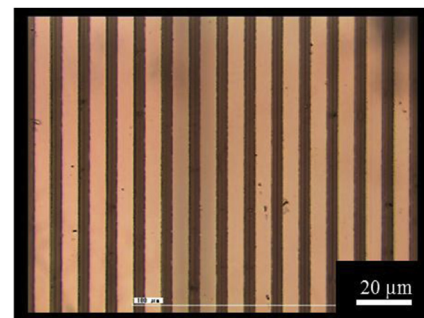


Fig. 11. Setup of laser processing.

Table 4
Experimental conditions.

Wavelength	1064 nm
Pulse duration	20 ps
Frequency	50 Hz
Beam spot size	2500 μm
Irradiation number n	1 - 1000 shots
Energy density E_d	0.072–0.251 J/cm ²
Workpiece	Ni-P

Fig. 12. Laser microscope image of the processed surface with grooves of 2 μm depth.

fabricated LIPSS on the flat and the microgrooved surfaces, respectively. As shown in Fig. 15, compared with LIPSS on flat surface, pitch length of LIPSS is a little small on the grooved surface, although it is considered that pitch length of LIPSS on the grooved surface is larger than that on a flat surface since absorbed energy is increased due to much production of plasma wave by grooves. In case of the 1- μm -deep grooved surface, pitch length of LIPSS is larger on the grooved surface with smaller pitch of grooves since plasma wave is induced more effectively by more grooves increasing absorbed energy. Contrastingly in case of the 2- μm -deep grooved surface, increase of the number of grooves on the irradiated area decreased the pitch length of LIPSS since the deep grooves might disturb surface plasmon. Further studies about effects of grooves on the pitch length of LIPSS are needed. The height of the LIPSS was evaluated by an atomic force microscope (AFM) and Fig. 16 shows that while the LIPSS on the surface with microgrooves of 1 μm depth and 10 μm pitch length is the highest, and microgrooves help to expand the aspect ratio of the LIPSS. In this regard, the microgrooved surface was more suitable for irradiation than a flat surface for fabricating LIPSS. It can be concluded that ultraprecision cutting

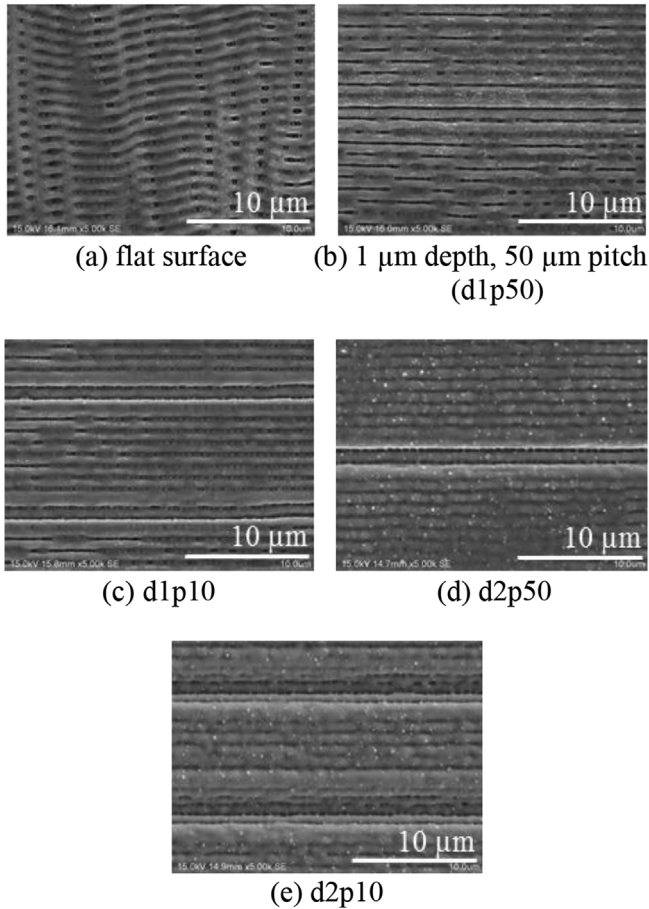


Fig. 13. LIPSS at $E_d = 0.15 \text{ J/cm}^2$ and $n = 10$.

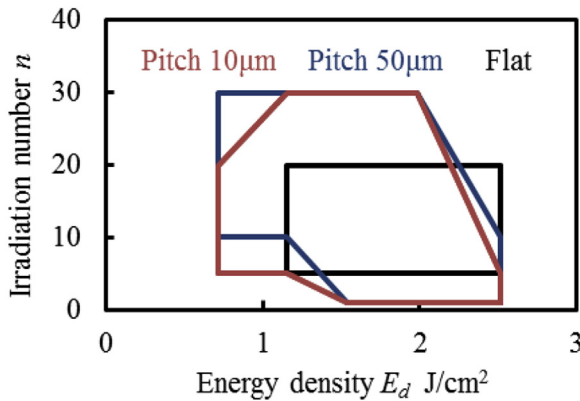


Fig. 14. Effective range of experimental conditions.

prior to irradiation with a short-pulsed laser is an effective method for the control of LIPSS: straight microgrooves likely induce the parametric decay and propagate the surface plasma waves perpendicularly to the grooves, and the surface plasmons occur periodically and linearly, finally resulting in straight LIPSS.

5. Conclusion

The effects of the surface shape on the fabrication of LIPSS were clarified by employing ultraprecision cutting prior to a short-pulsed laser to control the fabrication of LIPSS. The following conclusions can be drawn from this investigation:

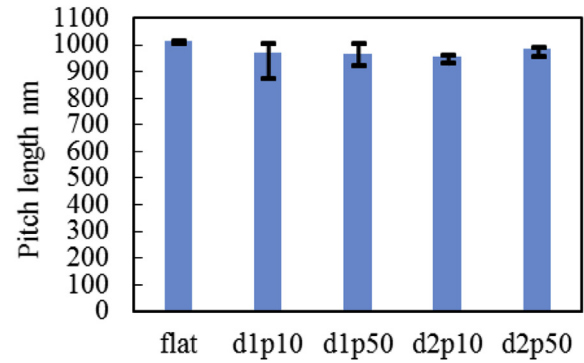


Fig. 15. Pitch length of LIPSS.

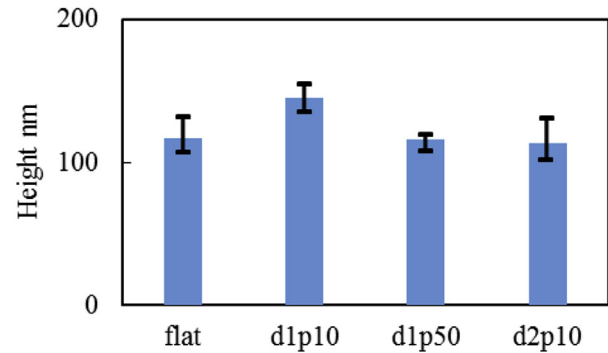


Fig. 16. Height of LIPSS.

1. FDTD simulation shows that a straight microgroove likely induces both a periodic and linear electric field after laser irradiation while there is no electric field on the flat surface irradiated by laser.
2. LIPSS was straight on all surfaces with microgrooves of different pitch lengths and depths. A shorter pitch length provides better since the LIPSS is distorted at the area far from the microgrooves when the pitch length was longer. This difference was not confirmed for LIPSS on grooved surfaces with different depths.
3. The pitch length of LIPSS is close to and depends only on the laser wavelength regardless of the pitch length and depth of the microgrooves.
4. LIPSS fabricated on the microgrooved surface are higher than those on the flat surface. The short pitch length of microgrooves is effective to fabricate high aspect ratio LIPSS.
5. Ultraprecision cutting prior to irradiation by a short-pulsed laser is effective to control LIPSS since the parametric decay is likely caused at microgrooves, and the periodic and linear plasma wave are readily induced and propagated perpendicularly to the grooves.
6. The proposed method is able to make LIPSS straight and improve aspect ratio, providing high functionalities, and has possibility to control LIPSS.

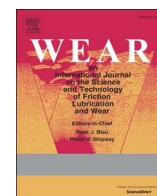
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Tool wear mechanism and its relation to material removal in ultrasonic machining



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ABSTRACT

As a particular important factor in UltraSonic Machining (USM), the tool wear largely affects the material removal process and accordingly influences the machining performance. Although a number of experiments were conducted to monitor and measure the tool wear during USM in previous works, the nature of the tool wear mechanism and its relation to material removal have not been totally understood. Therefore, both simulations and experiments were conducted for studying the influence of tool wear on material removal in this work. Three different tool materials i.e. 304 stainless steel, 1045 carbon steel, and tungsten carbide were used. A numerical simulation model utilizing both Smoothed Particle Hydrodynamics (SPH) meshfree method and Finite Element Method (FEM) was built first to predict tool deformation and fractures of workpiece and abrasive particles. Experiments were then conducted to verify the simulation results. It was found that using tool materials possessing a high flexibility such as 304 stainless steel can slow down the wear of abrasive particles and improve the material removal efficiency. Additionally, work hardening was confirmed to occur in 304 stainless steel and accordingly suppressed the tool wear. On the other hand, when using tungsten carbide as the tool material, even though the tool wear was small due to the high hardness, the abrasive particles were abraded seriously and the material removal efficiency was lowest. Finally, the relation between the material removal and the tool wear was discussed based on these results.

1. Introduction

Hard and brittle materials have a wide variety of industrial applications due to their superior mechanical and physical properties. Various micro-shaped structures are required to be fabricated on these materials to endow them with more attractive performances. Generally, not only the material property would influence the quality and the stability of the final products, the machining precision and potential damages on the machined surfaces can also strongly affect the service performance. For example, in fabrication of quartz accelerometers or gyrometers, damages can generate and propagate in the crystal from a tiny defect even in nanometer scale, which may deteriorate the resolution of the final products [35]. Therefore, to meet the increasing applications of hard and brittle materials, developing precision and efficient micromachining techniques is urgently demanded. However, the high hardness and strength and chemical stability make their machining process extremely hard, costly and time-consuming [5,9]. At present, efficient and economic fabrication of micro-structures with high dimensional accuracy and surface quality on hard and brittle

materials is still a great challenge to keep up with the development of advanced technology.

Numerous non-traditional methods such as Laser Beam Machining (LBM), Electrical Discharge Machining (EDM), ElectroChemical Machining (ECM) and UltraSonic Machining (USM), have been developed to solve the problems in manufacturing hard and brittle materials. Among these processes, the micro-USM is considered the most effective from the views of machining efficiency, cost, machined form accuracy, surface quality and material dependence [39]. It does not thermally or chemically damage the work material and does not cause significant levels of residual stress. Fig. 1 shows a schematic view of USM. A tool is set directly above the workpiece and ultrasonically vibrated perpendicular to it. A slurry comprising hard abrasive particles is provided constantly into the working zone, and the tool indirectly impacts the workpiece surface through these abrasive particles. For hard and brittle materials, a large number of tiny fractures occur and the materials are removed by their accumulation. In this system, many process parameters exist which would influence the machining performance. Major parameters including slurry characteristics [36,37,6,9], work material

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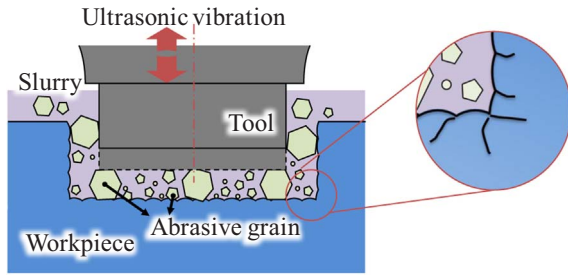


Fig. 1. Schematic view of USM.

[26,28], tool material [32] and some other dynamic conditions [11,32] on the machining performance have been widely experimentally investigated. In addition, many studies were conducted to reveal the involved material removal mechanisms in USM [23,38,4]. However, due to the complexity of this process and the difficulty of observing the machining phenomena directly, a total understanding of USM is still not possible. The relation between the various process factors and material removal should be further studied with a more effective way, which is important for putting micro-USM into practical high quality micro-machining. Therefore, authors of this work firstly proposed to use Smoothed Particle Hydrodynamics (SPH) method to clarify the material removal mechanism [33]. Wear of abrasive particles and crack generation in workpieces were investigated by the calculation in conjunction with the experimental verification [34], and the SPH method is proved effective in studying the material removal mechanism in USM.

As a particular important factor in this system, the tool wear can largely affect the material removal process. The complex tool wear patterns in USM is divided into longitudinal wear and lateral/side/diametral wear [3]. The longitudinal tool wear is the shortening or reduction of the length of the tool, while the lateral wear refers to the diameter reduction of the tool from the original contour. A number of investigations [10,15,22] have been conducted to monitor and measure both of them. The influence of tool wear on machining performance have also been discussed in these works. The results have shown that tool wear is a very important variable in micro-USM, which affects both the machining rate and the machining precision. A worn tool produces a poor form accuracy and a wrong choice of the tool will even lead to failure of the machining. However, as mentioned above, the nature of the tool wear mechanism and its relation to material removal during micro-USM are still not clear. Generally, tool wear in micro-USM depends on several parameters including tool material, workpiece material, static load of the tool on the abrasive particles, amplitude of tool vibration, and dimension and type of abrasive particles [7]. In this paper, the effect of tool material was investigated by both SPH simulations and USM experiments. 304 stainless steel, 1045 carbon steel,

and WC were used as the tool material for comparison. The machining efficiency, surface quality, wear of abrasive particles and tool wear when using different tool materials were studied. We found that the tool material used in USM is not the harder the better, and some interesting features exist in the wear process. Finally, the tool wear mechanism and its relation to material removal was further discussed by using these results, which is significant for understanding USM process and improving the machining performance.

2. Simulation method and results

2.1. SPH to FEM coupling

The simulations were conducted with the computer program AUTODYN from Century Dynamics. The SPH solver has been implemented in it and can be used together with other grid-based Lagrangian techniques including FEM. Grid-based techniques assume a connectivity between nodes while SPH utilizes a density estimation. In SPH, a set of particles endowed with physical properties are distributing in the domain and interacting with each other according to the governing equations to simulate a practical problem. SPH does not require a numerical grid, therefore problems involved in large deformations and fractures which may result in error due to mesh distortion and tangling in the FEM could be solved [25]. Consequently, it is suitable to study the material removal due to fracture failure of hard and brittle materials in USM. However, the SPH is always more expensive in computation time because the algorithm takes more time to find neighboring particles. The combination of SPH and FEM allows material in small deformation to be constructed with grid-based solver and reduces the calculation amount. The joining method between SPH particles and finite element mesh is schematically shown in Fig. 2. The particles for the object can be aligned with the edge of a mesh and does not need to be aligned at nodes. On the other hand, a small gap size is defined to detect the interaction between objects (not joined together) modeled with different solvers. These provide the possibility of calculation among SPH and FEM.

2.2. Material modeling

Glass was used for workpiece modeling; silicon carbide (SiC) was employed as abrasive material; SS304, STEEL1045 and WC referred to stainless steel (AISI: 304), carbon steel (AISI: 1045) and tungsten carbide were utilized for tool modeling. The Mie-Grüneisen polynomial equation of state [1] was employed to describe the initial elastic response of glass and SiC materials. In this material model, the hydrostatic pressure P is

$$P = A_1\mu + A_2\mu^2 + A_3\mu^3 + (B_0 + B_1\mu)\rho_0 e \quad (\mu > 0 \text{ Compression}) \quad (1)$$

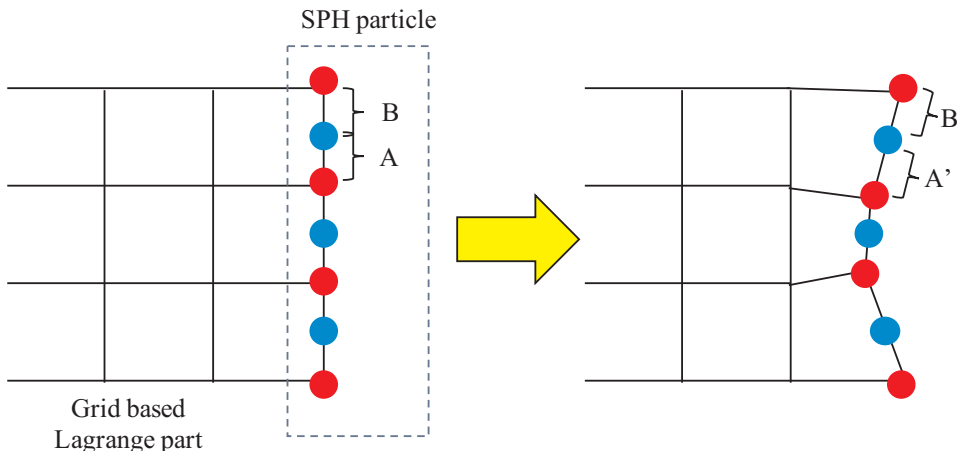


Fig. 2. Joining method between SPH particles and finite element mesh.

$$P = T_1\mu + T_2\mu^2 + B_0\rho_0 e \quad (\mu < 0 \text{ Tension}) \quad (2)$$

where $A_1, A_2, A_3, B_0, B_1, T_1$ and T_2 are constants (A_1 is the bulk modulus); e is the internal energy; and $\mu = \rho / \rho_0 - 1$ for current density ρ and initial density ρ_0 , which is a variation of the volumetric strain.

On the other hand, Johnson-Holmquist material model was used to describe the strength and damage behavior of the two materials which may fracture in the USM. There are two closely related variations of this model. The first version is commonly called JH-1 [18] and the second version is correspondingly termed JH-2 model [19] which incorporated a damage evolution rule. They are suitable to simulate the fracture phenomena of brittle materials and proved effective to visually express the crack extension [13].

The Shock equation of state [1,8] was used for SS304 and WC materials. In this model, the Hugoniot relations [21,29] is chosen as the reference function for the Mie-Grüneisen equation of state. While for STEEL1045, the equation of state simply used a linear description as

$$p = K\mu \quad (3)$$

where $\mu = (\rho / \rho_0) - 1$, and K is the material bulk modulus. This form of equation of state is only used for small compressions.

The strength of SS304 is formed by Steinberg-Guinan model [31]. The model produced expressions for the shear modulus G and yield strength Y as functions of equivalent plastic strain ϵ , pressure P and internal energy T . The constitutive relations for Steinberg-Guinan model are represented in the forms

$$G = G_0 \left[1 + \left(\frac{G_p'}{G_0} \right) \frac{P}{\eta^{1/3}} + \left(\frac{G_T'}{G_0} \right) (T - 300) \right] \quad (4)$$

$$Y = Y_0 [1 + \kappa(\epsilon + \epsilon_i)^n] \left[1 + \left(\frac{Y_p'}{Y_0} \right) \frac{P}{\eta^{1/3}} + \left(\frac{Y_T'}{Y_0} \right) (T - 300) \right] \quad (5)$$

where κ and n are work-hardening parameters.

Johnson-Cook model [17] was employed to model the strength behavior of STEEL1045. The model for the von Mises stress, σ , is expressed as

$$\sigma = [A + B\epsilon^t][1 + C \ln \dot{\epsilon}^*][1 - T^{*m}] \quad (6)$$

where ϵ is the equivalent plastic strain, $\dot{\epsilon}^* = \dot{\epsilon} / \dot{\epsilon}_0$ is the dimensionless plastic strain rate for $\dot{\epsilon}_0 = 1.0 \text{ s}^{-1}$, and T^* is the homologous temperature where $T^* = (T - T_{\text{room}}) / (T_{\text{melt}} - T_{\text{room}})$. The five material constants are A, B, t, C , and m , where A is the yield stress, B and t represent the effects of strain hardening, and C is the strain rate constant.

The strength for WC was simply used von Mises yielding criterion. The yield occurs when von Mises equivalent stress σ is greater than the yield stress Y [20], which is shown as

$$\sigma = \left[\frac{(\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_z - \sigma_x)^2 + 6(\tau_{xy}^2 + \tau_{yz}^2 + \tau_{zx}^2)}{2} \right]^{1/2} = Y \quad (7)$$

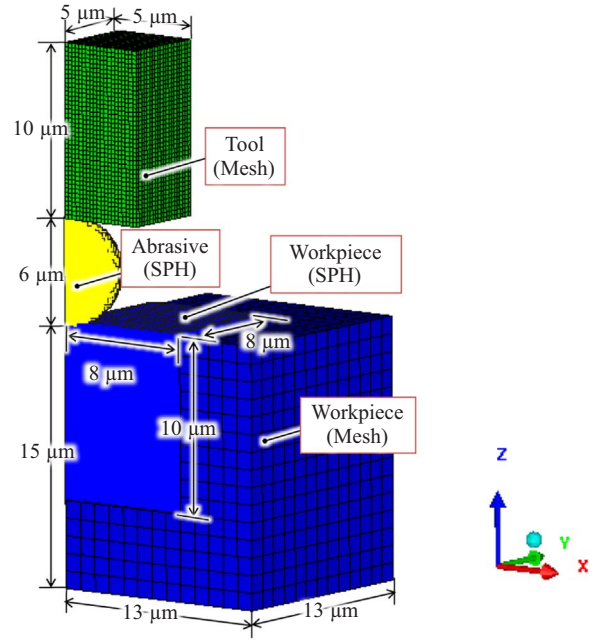


Fig. 3. Snapshot of the initial state of the simulation model.

The important parameters for glass [2,14], SiC [12], SS304 [30], STEEL1045 [16] and WC [30] are summarized in Table 1.

2.3. Construction of simulation model

A snapshot of the initial state of the simulation model is shown in Fig. 3. A one-quarter model with symmetric boundary conditions was used to reduce the calculation amount. The abrasive particle was assumed to be a sphere which was different from those of angular shape in the experiments to simplify the model. In our former work [34], effects of the spherical and angular abrasive particle on USM were discussed by both SPH simulations and experimental verifications. It was found that sharp corners of the angular particle would be worn and turned to be round with the increase of machining time. The generated round particle resisted wear better and played a primary role in material removal. As we are focused on the tool wear in the current work, simulations by using spherical particles are considered suitable for the investigation. The abrasive particle and the partial workpiece areas around the impact site were built with SPH solver, while the tool and the remaining parts of the workpiece were modeled using the Lagrange finite element mesh to improve the calculation efficiency. The smoothing length used in the model was 200 nm. The finite element mesh sizes of the workpiece and the tool were 1 μm and 0.25 μm, respectively. The velocity condition of the tool vibration was applied on the top surface of the tool, which is simplified from ideal condition given in the experiments. The calculation method of the velocity was

Table 1
Material models and important parameters.

	Float glass	SiC	SS304	STEEL1045	WC
Equation of state	Polynomial	Polynomial	Shock	Linear	Shock
Density g/cm ³	2.53	3.215	7.9	7.83	14.9
Bulk modulus GPa	45.4	220	None	159	None
Grüneisen coefficient Γ	None	None	1.93	None	1.5
Strength	Johnson- Holmquist	Johnson- Holmquist	Steinberg-Guinan	Johnson- Cook	Von Mises
Shear modulus GPa	30.4	193.5	77 (G_0)	81.8	254
Hugoniot elastic limit GPa	5.95	11.7	None	None	None
Yield stress MPa	None	None	340 (Y_0)	553.1 (A)	5100 (Y)
Failure	Johnson- Holmquist	Johnson- Holmquist	None	None	None
Hydro tensile limit MPa	150	750	None	None	None

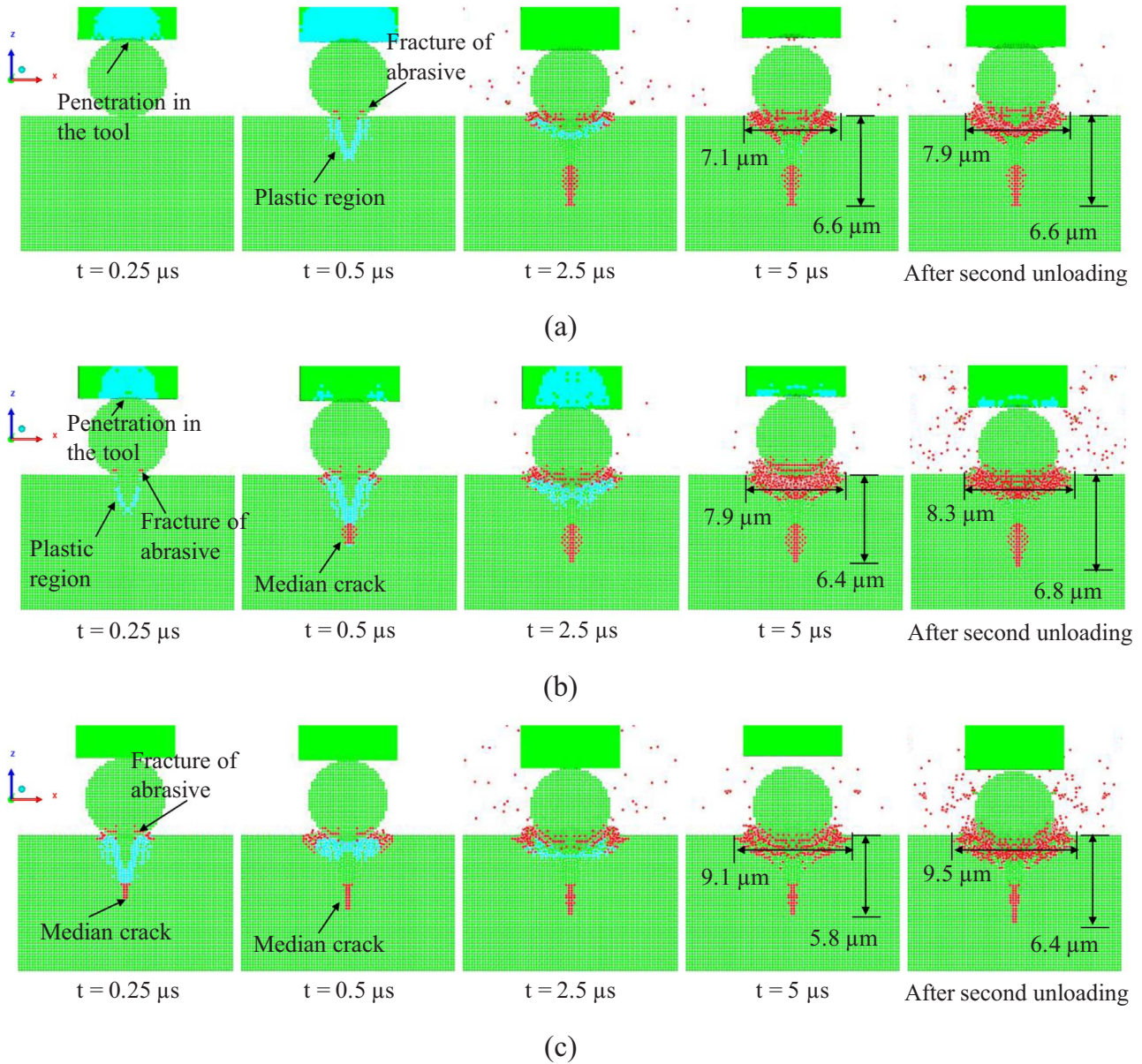


Fig. 4. Time-dependent results of material status with (a) SS304, (b) STEEL1045, and (c) WC tools.

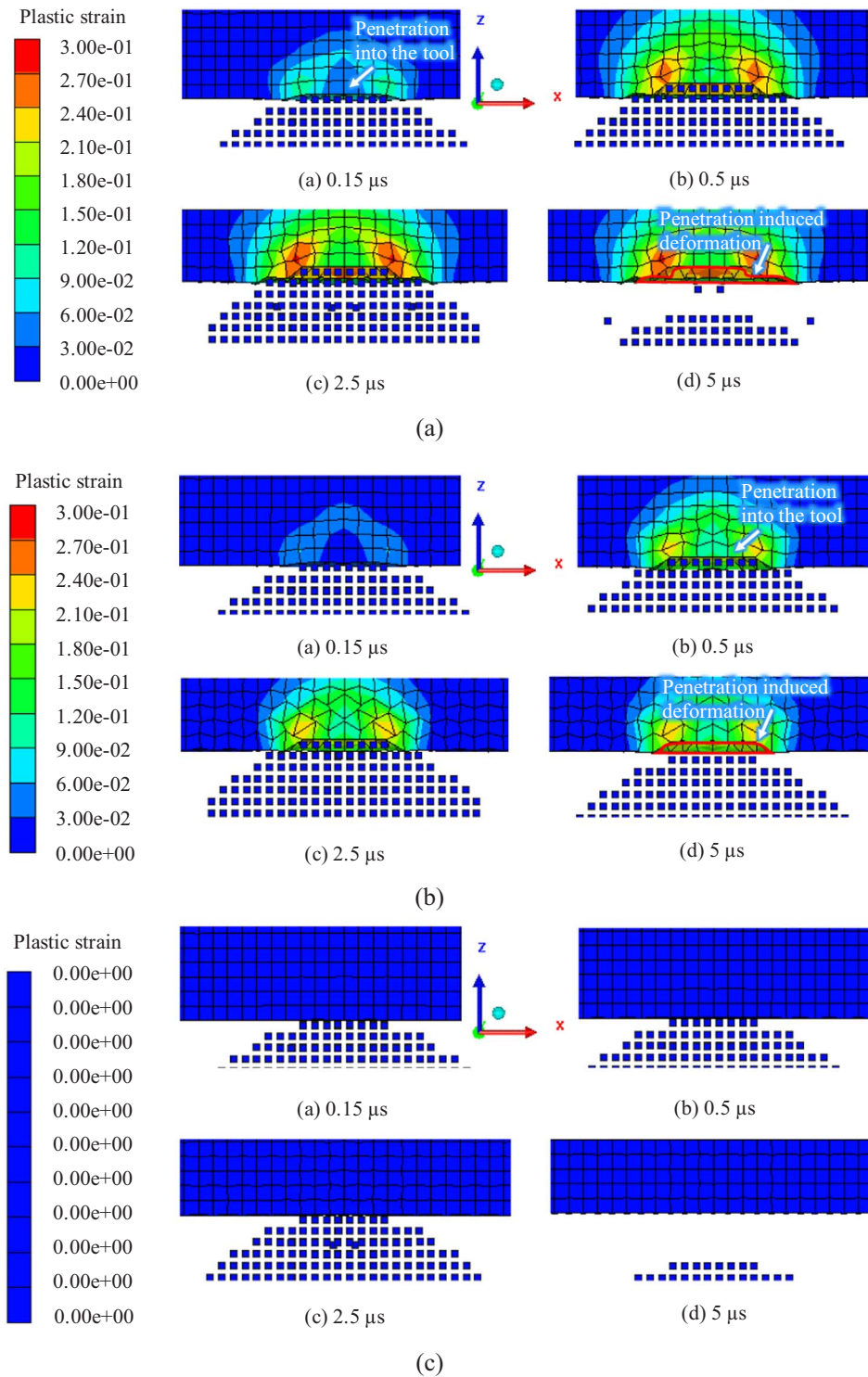
introduced in our former work [33]. Two cycles of the loading boundary conditions with the initial velocities of 0.75 m/s and 0.9 m/s were used. Fixed boundary conditions were applied to the bottom and the side surfaces of the workpiece.

2.4. Simulation results

Time-dependent results of material status with SS304, STEEL1045, and WC tools are shown in Fig. 4(a), (b), and (c), respectively. Red color means material failure. Median/radial and lateral cracks [24] are confirmed to be generated during the loading and unloading cycles for all three cases. The penetrations of the abrasive particles into SS304 and STEEL1045 tools can be seen immediately at the start of the calculations with that being deeper for SS304 tool, while no variation of WC tool can be found in the total calculation cycle. A plastic region (blue color) was firstly generated in the workpiece when subjected to the compression of abrasive particles. It was generated after a calculation of 0.25 μs with both STEEL1045 and WC tools and after 0.5 μs with SS304 tool. Similar lag of fractures in the abrasive was also occurred with SS304 tool. The possible reason is that the large deformation of SS304

due to the abrasive penetration slows down the indentation into the substrate and relieves abrasive fracture. The median cracks grew into 6.6, 6.4, and 5.8 μm in depth at the end of the first unloading cycle ($t = 5 \mu\text{s}$) with SS304, STEEL1045, and WC tools, respectively. The obvious decrease of the crack size with a WC tool is considered as the result of particle fracture. At the time, the total fracture mass (calculated from the red elements) of the abrasive particle was the largest of 21.09 pg with the WC tool, while being 17.9 pg with the STEEL1045 tool and the lowest value of 14.2 pg when using the SS304 tool. During the second cycle, no extension of the median crack occurred with the SS304 tool, while the crack depth increased to 6.8 and 6.4 μm with STEEL1045 and WC tools, respectively. The large deformation of the SS304 tool is considered helpful in suppressing the further crack growth in the depth direction. On the other hand, the lateral cracks with widths of 7.1, 7.9, and 9.1 μm were generated after the first vibration cycle and extended to 7.9, 8.3, and 9.5 μm with SS304, STEEL1045, and WC tools, respectively. The large lateral cracks and their growths are significant for the material removal. In respect to WC tool, the lateral crack was the largest after the first loading cycle, but its growth is low during the second cycle. It is considered that even though the indentation into the

Fig. 5. Plastic strain on different tools: (a) SS304, (b) STEEL1045, (c) WC.



workpiece with the WC tool is larger due to no penetration into the tool at first, the fracture of the abrasive particle decreased the pressure applied on the work surface in the following impact. Therefore, the crack width was only increased slightly, which was not helpful for material removal. Fig. 5 shows the plastic strains on the three kinds of tools. The highest value of the SS304 tool was nearly 1.25 times higher than that of the STEEL1045 tool. Whereas no plastic strain occurred in the WC tool and correspondingly no deformation was generated to relieve the compression pressure applied on the abrasive particles. Pressure change over the time for different tools during first vibration cycle were summarized as shown in Fig. 6. The solid lines depicted the results

at the contact center of the tool and the abrasive, while the dash lines illustrated the results at the point of 0.75 μm above the contact center. The pressure at the contact center was the lowest with the SS304 tool due to its lowest resistance to deformation. Besides, the difference of pressure in the two evaluated points was smallest when using SS304 because of the possible reason that the pressure was transmitted inside the tool material.

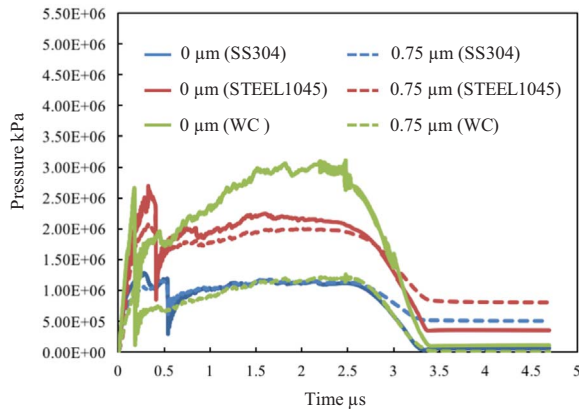


Fig. 6. Pressure change over the time for different tools (evaluated at the contact center of the tool and the abrasive and the point of 0.75 μm above this contact center).

3. Experiments with different tool materials

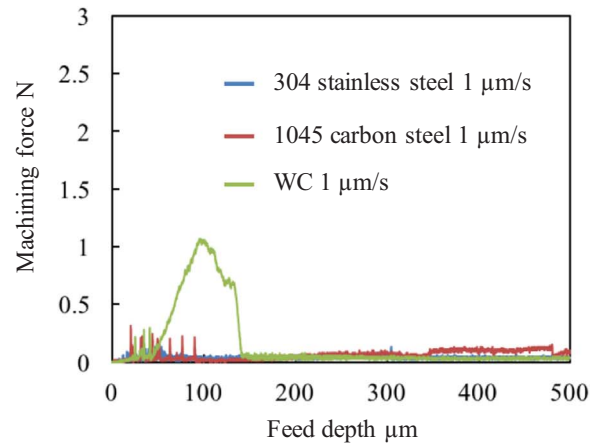
3.1. Machining efficiency with different tools

A stationary ultrasonic drilling machine (SD-100K; Taga Electric Corporation) was used in this work. To verify the simulation results, the same tool materials were utilized in experiments. Blind-hole drilling experiments on glass plates were conducted to compare the machining efficiency. The detailed experimental conditions are listed in Table 2.

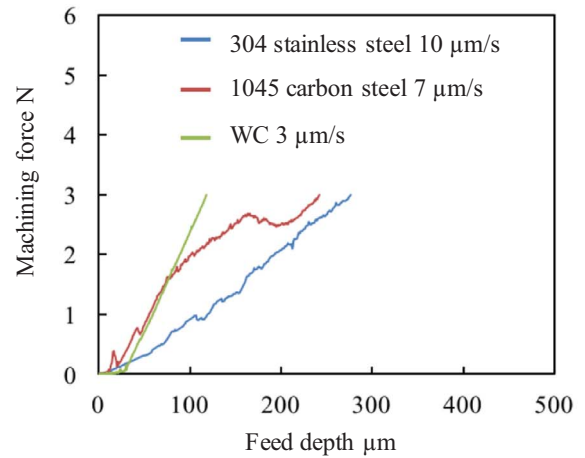
The results of machining force with different tools are shown in Fig. 7. The machining forces for all the three cases were steady and lower than 3 N when the feed rate was low as shown in Fig. 7(a), which means that the material removal was carried on smoothly. On the other hand, the machining forces for all the three cases rapidly increased if a high feed rate was employed as shown in Fig. 7(b), which implies that the material removal rate cannot meet the tool feed rate. The maximum feed depths under different feed rates until the machining force reaches 3 N are shown in Fig. 8. The results show that the tool feed rate can be up to 7 $\mu\text{m/s}$ to finish 500 μm feed with the tool made of SS304, while being 6 $\mu\text{m/s}$ by using the tool made of STEEL1045 and the lowest value of 2 $\mu\text{m/s}$ by using the tool made of WC. Therefore, the machining efficiency when using different tool materials can be ordered from high to low in magnitude as to be SS304, STEEL1045 and WC. In consideration of the simulation results, the lowest material removal efficiency with WC tool may be associated with the large fractures of the abrasive particles. Because of the severe wear of abrasive particles, the crack generation and growth in the workpiece were suppressed, and which accordingly slows down the material removal rate.

3.2. Subsurface cracks

The machined surfaces after etching were observed with an SEM and the crack depths of the remaining cracks were examined. The crack distribution for each case is summarized as shown in Fig. 9. Depths of cracks on the bottom surfaces are less than 6 μm and larger than those



(a)



(b)

Fig. 7. Machining force with different tool materials: (a) low feed rate, (b) high feed rate.

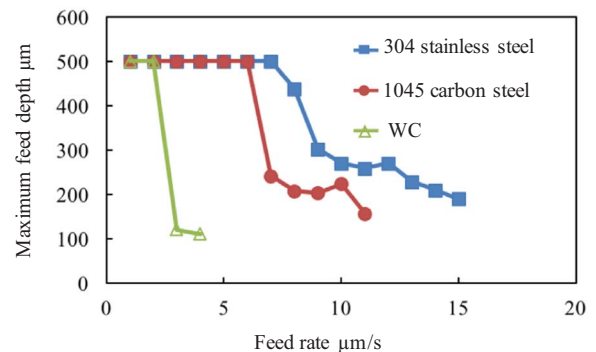


Fig. 8. Maximum feed depth under different feed rates with different tools.

Table 2

Experimental conditions for blind-hole drilling experiments by using different tools.

Vibration frequency	About 61 kHz
Vibration amplitude	About 4 μm (peak-to-peak)
Maximum tool feed depth	500 μm
Machining force	Lower than 3 N
Tool material	304 stainless steel, 1045 carbon steel, WC (ϕ 1 mm)
Abrasive material	SiC (mean size: 8 μm)
Tool feed rate	1 ~ 15 $\mu\text{m/s}$
Flow rate of slurry	50 mL/min
Abrasive volume	10 wt.% mixed with water
Workpiece material	Glass

of cracks on the side surfaces for all the three cases. The bottom surfaces contain a small percentage of cracks larger than 3 μm when using WC tool and it was the lowest among the three cases. The mean depths when using different tools were calculated based on these observations and plotted in Fig. 10. The calculated values are 1.73, 1.70, and 1.68 μm for the bottom surfaces with the SS304 tool, the STEEL1045 tool, and the WC tool, respectively. The corresponding values for wall surfaces are 1.25, 1.27 and 1.21 μm , respectively. The depths of remaining cracks tend to decrease with the WC tool, which again supports the simulation results that the crack growth was slowed down due to the large fractures of abrasive particles.

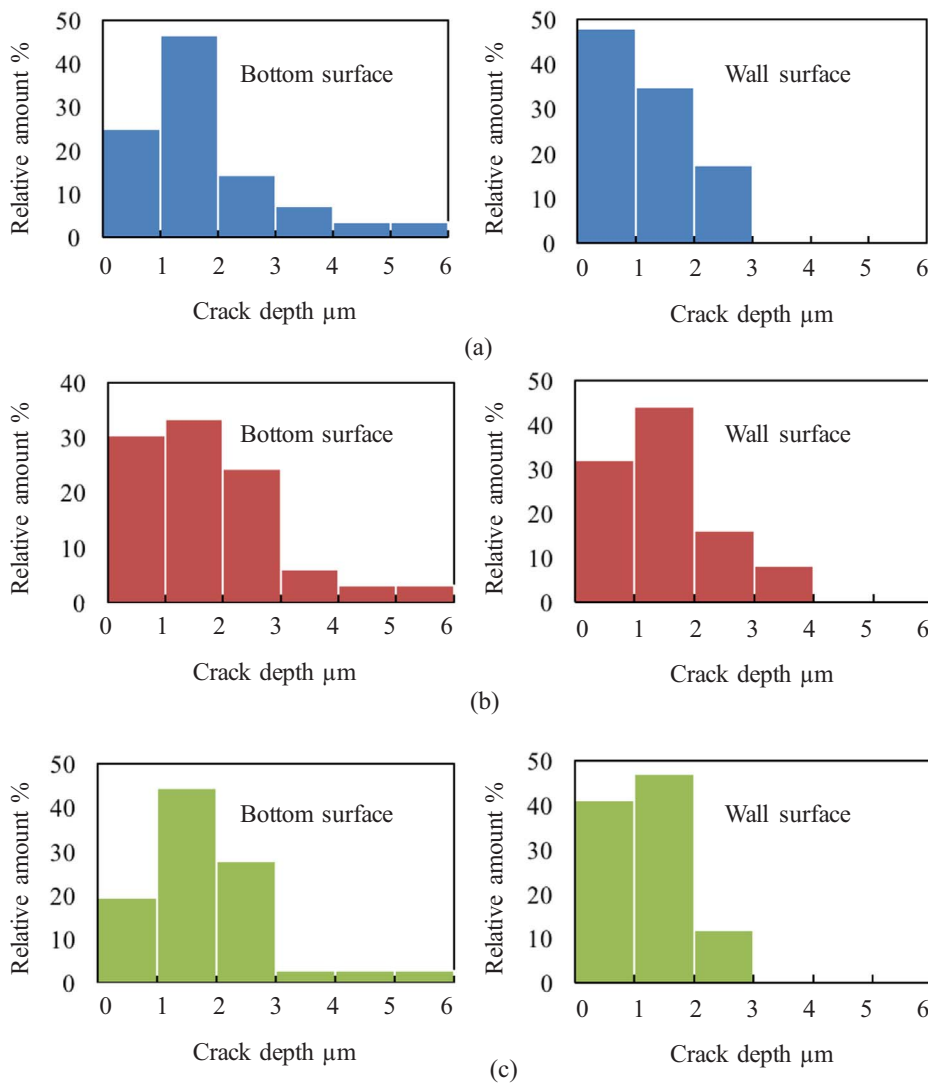


Fig. 9. Crack distribution on the machined surfaces with different tools: (a) 304 stainless steel, (b) 1045 carbon steel, (c) WC tool.

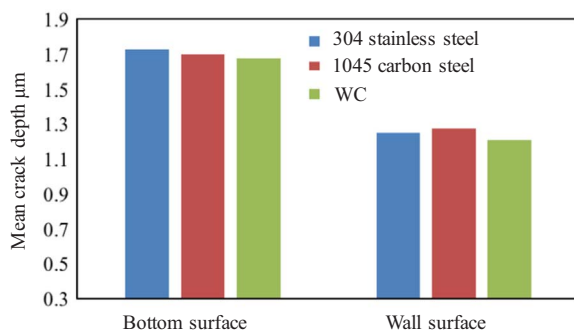


Fig. 10. Mean crack depth with different tools.

3.3. Wear of abrasive particles

To compare the wear of abrasive particles with different tool materials, deep holes up to 1300 μm were drilled on glass plates. Slurry of 20 g in mass (not including the amount in the pump tube) with a low concentration of 5 wt.% was provided at first and no fresh slurries were added in the machining process. The detailed experimental conditions are listed in Table 3. The transition of the mean particle size with the increase of machining time is depicted in Fig. 11. The results show that the particle size decreases with the machining time for all three cases. It also clearly shows a fastest decrease in particle size with the WC tool,

Table 3

Experimental conditions for confirming abrasion of abrasive particles with different tool materials.

Vibration frequency	About 61 kHz
Vibration amplitude	About 4 μm (peak-to-peak)
Maximum tool feed depth	1300 μm
Tool feed rate	1 $\mu\text{m/s}$
Tool material	304 stainless steel, 1045 carbon steel, WC (ϕ 1 mm)
Flow rate of slurry	50 mL/min
Abrasive material	SiC (mean size: 8 μm)
Abrasive volume	5 wt.% mixed with water
Slurry amount	20 g
Workpiece material	Glass

which means that a highest wear rate occurred in abrasive particles with this tool material. Suppression of particle wear can be obtained with the other two materials possessing high flexibility. These results well agreed with the simulation results that the particle fracture can be suppressed due to the deformation of the tools.

3.4. Tool wear

The machining conditions for investigating the wear of different tools are listed in Table 4. Each tool was used for machining five holes successively. The cumulative longitudinal tool wear with the increase of the number of holes is shown in Fig. 12. The tool wear increases almost

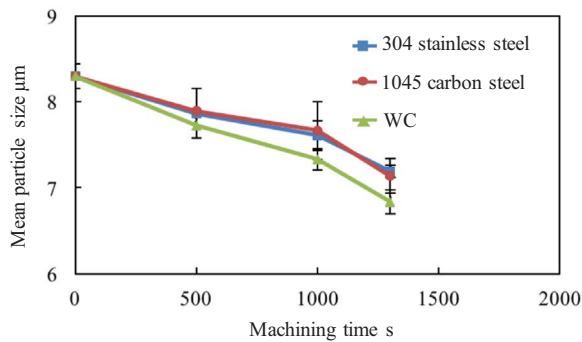


Fig. 11. Decrease of mean particle sizes when using different tools with the increase of machining time.

Table 4

Experimental conditions for investigating the influence of tool materials on tool wear.

Vibration frequency	About 61 kHz
Vibration amplitude	About 4 μm (peak-to-peak)
Maximum tool feed depth	1000 μm
Machined hole numbers of each tool	5
Tool material	304 stainless steel, 1045 carbon steel, WC (φ 1 mm)
Flow rate of slurry	50 mL/min
Abrasive material	SiC (mean size: 8 μm)
Abrasive volume	10 wt.% mixed with water
Workpiece material	Glass

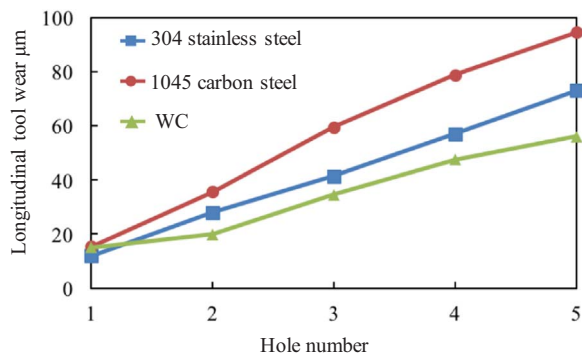


Fig. 12. Longitudinal wear of different tools during holes drilling experiments.

linearly with the number of holes for all cases, which indicates that the longitudinal tool wear during USM grows nearly uniformly with the machining time under the same machining conditions. The wear of the STEEL1045 tool was the highest, followed by the SS304 tool, and it is

the lowest for the WC tool. The cross-sectional profiles of the cutting surfaces before and after machining were measured for the three tools as depicted in Fig. 13, Fig. 14, and Fig. 15, respectively. The results show that the lateral wear of the WC tool is also the lowest. It is believed that the harder the tool material, the larger the lateral wear resistance [22]. In addition, large wear in the center of the cutting surface after machining five holes was confirmed for STEEL1045 tool, the same wear was slight for SS304 tool. In respect to WC tool, no center wear can be found on the cutting surface. All the results show that the tool wear is highest for STEEL1045 tool. As we know, both SS304 and STEEL1045 have good strength, durability, and flexibility, however the SS304 tool shows a much better machining performance than the STEEL1045 tool. Therefore, to find the reason, the properties of the cutting surface of the tools were evaluated.

The hardness of the different tools before and after machining was measured with a microhardness measuring system (FISCHERSCOPE® HM2000 LT). The values for each case were obtained by calculating an average value of five different measured points under a load of 500 mN. Fig. 16 depicts the load-depth curves based on measured results. The figures on the left side show the results before machining, while those on the right side exhibit the results after machining. Fig. 17 shows the measured Vickers hardness and Fig. 18 shows the indentation modulus calculated from the slope of the tangent of the unloading curve. In respect to SS304 tool, the hardness increased dramatically after USM, which explained the result that low wear occurred on SS304 tool during USM. The drastic increase in the hardness of austenitic 304 stainless steel is considered as the result of the work hardening of the cutting face due to repeated impacts of abrasive particles. Commonly, when austenitic stainless steels are deformed or strained, formation of a martensite occurs and the dislocation density within the host material increases in accordance with the amount of deformation [27], which will harden the material. As the cutting surface became harder after impacts of the abrasive particles, the erosive wear of the tool can be suppressed. On the other hand, the indentation modulus of SS304 after USM is still around 200 GPa, which means the tool is as flexible as the one before machining. It is considered that the interior material was not hardened as the cutting surface. The tool still showed flexibility when subjected to impacts of abrasive particles, which is significant to suppress the abrasive fracture and maintain the machining gap. For the other two materials, both the hardness and the indentation modulus decreased after USM, which implies a degradation of surface quality due to repeated impacts. Micrographs of the test surfaces are shown in Fig. 19. All the surfaces were polished with #1200 abrasive paper before machining. After USM, SS304 showed irregular indentations and scratches as marked in Fig. 19(a). Flattened surface can also be found, which is considered as the result of successive impacts of the erodent abrasive particles. In respect to STEEL1045, cracks and large craters are

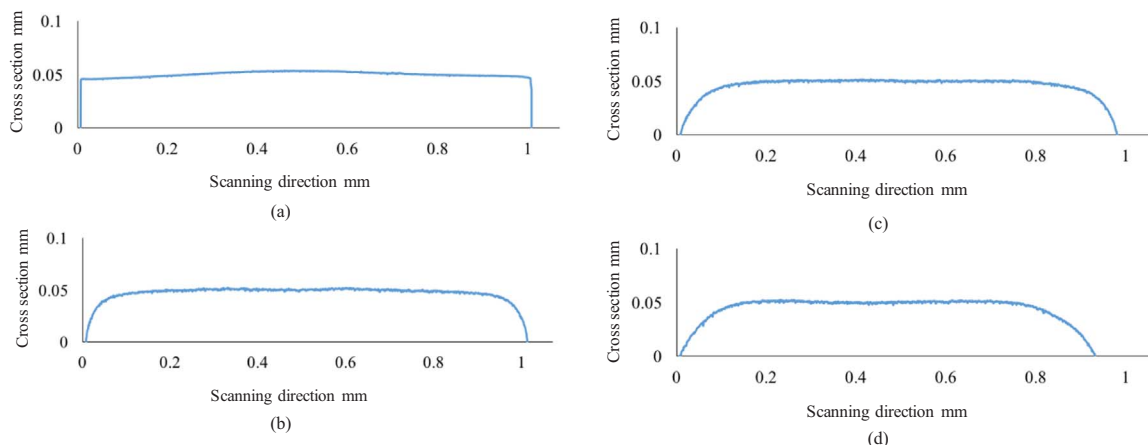


Fig. 13. Cross-sectional profiles of the 304 stainless steel tool tip after holes drilling experiments: (a) before machining, (b) after one hole, (c) after two holes, (d) after five holes.

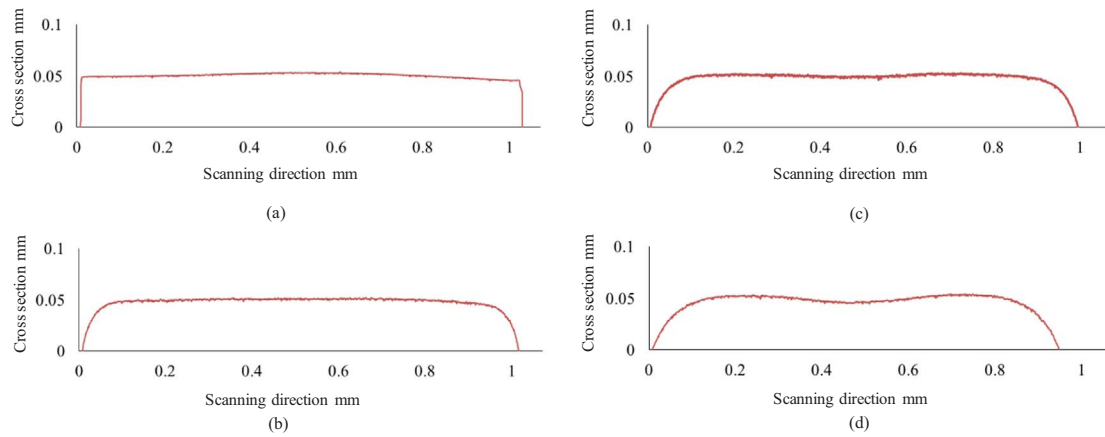


Fig. 14. Cross-sectional profiles of the 1045 carbon steel tool tip after holes drilling experiments: (a) before machining, (b) after one hole, (c) after two holes, (d) after five holes.

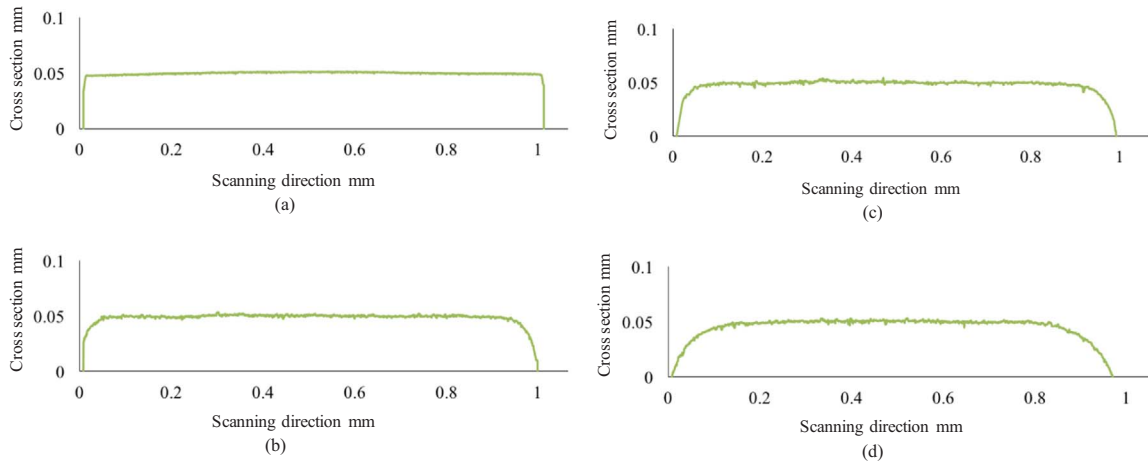


Fig. 15. Cross-sectional profiles of the WC tool tip after holes drilling experiments: (a) before machining, (b) after one hole, (c) after two holes, (d) after five holes.

clearly confirmed on the cutting surface after USM machining, which verified the assumption that the surface quality was degraded due to repeated impacts of the abrasive particles. WC also exhibited a clue of the degradation. Random fractures of WC grains occurred after USM as shown in Fig. 19(c), which is believed to have effects on the hardness and indentation modulus of the test surface.

4. Discussion on tool wear mechanism and its relation to material removal

Longitudinal wear of the tool is a complex phenomenon arising from the hammering action by the abrasive particles at ultrasonic frequency, abrasion by the flow of the slurry, and cavitation effects [22]. Fatigue wear and erosive wear are considered to occur in this process. Generally, the wear is low due to abrasive particles that only indented and did not slide on the material surface. During the successive impacts between the abrasive particles and the cutting surface, the tool material may deform plastically first before fracture.

Fig. 20 shows the relation between the material removal process and the tool wear in micro-USM. Fig. 20(a) depicts the best situation with smooth material removal and small tool wear, which can be considered as the case of using a SS304 tool. In this situation, slight deformation can occur in the tool material due to the penetration of abrasive particles, in turn, fractures of both the abrasive particle and the workpiece can be slowed down. The moderation of particle fracture is helpful to retain the machining gap and ensure further extension of cracks in the workpiece. Therefore, high material removal rate can be obtained. With the machining process going on, the cutting surface

hardened due to the repeated impacts of abrasive particles, which makes it keep away from the risk of fractures or other damages and accordingly reduces tool wear. Besides, the material inside the tool was not hardened as the cutting surface and still shows a good flexibility when subjecting to impacts of abrasive particles, which ensures slow particle wear and high material removal efficiency in total machining process. Fig. 20(b) shows the tool wear due to jammed material removal. In this situation, the tool also has a good flexibility, which can be considered to be made of STEEL1045. At the earlier stage of machining, smooth material removal was conducted and slight deformation occurred in the tool material. However, in this case, the tool did not have a high tendency to work hardening as the SS304 and did not possess sufficient capacity to undergo plastic deformation before fracture took place. Accordingly, the cutting surface exhibited a bad quality with cracks and big craters. The cracks and big craters are considered to hinder the motion of abrasive particles, and fresh abrasive particles in the slurry may be prevented from flowing into the cutting zone. As we know, the machining gap would become narrower and narrower when fresh abrasive particles cannot be flushed into the machining area and this causes the abrasive renewal in the center region more difficult. Therefore, the difference of the material removal rate at the center zone and the peripheral area of the tool would be enlarged with further feed. The fractured particles in the narrow area were not effective to make material removal, but eroded the tool material and led to a concavity on the tool surface. When no further erosion occurs, large plastic flow in the center of the cutting face would be generated inducing a dish shape. On the other hand, if a very hard tool material, such as WC, is used, almost no deformation occurs and the particle wear is fast, which is not

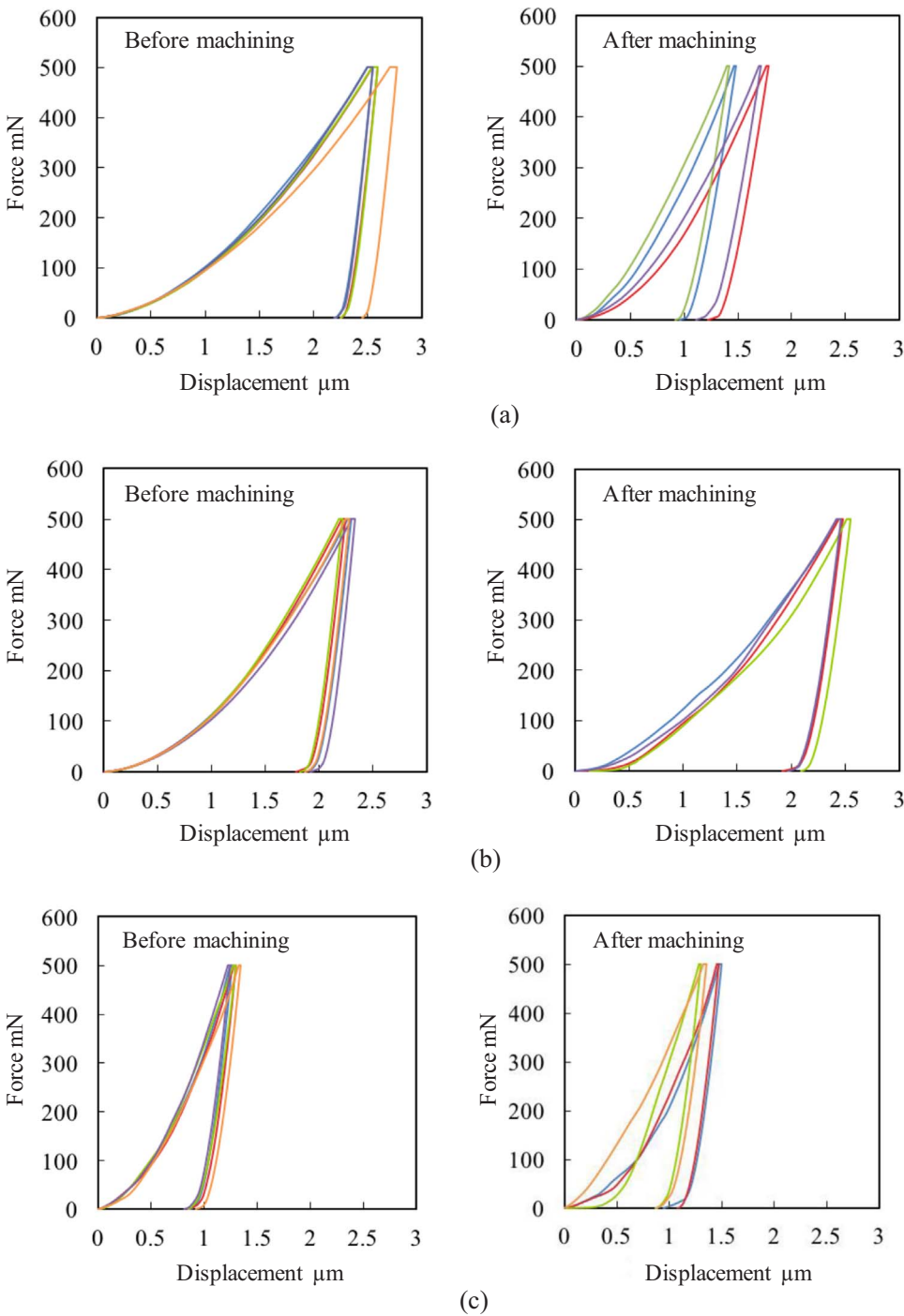


Fig. 16. Load-depth curves of different tools before and after USM: (a) 304 stainless steel tool, (b) 1045 carbon steel tool, (c) WC tool.

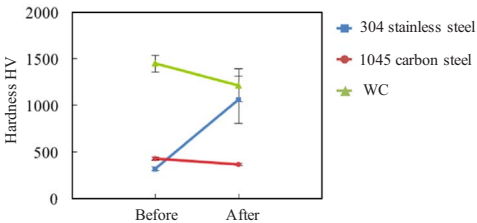


Fig. 17. Vickers hardness of different tools before and after USM.

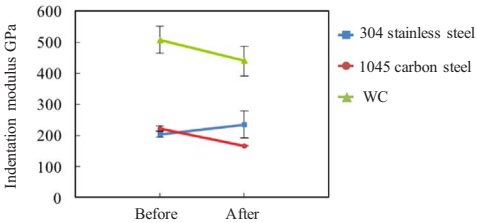


Fig. 18. Indentation modulus of different tools before and after USM.

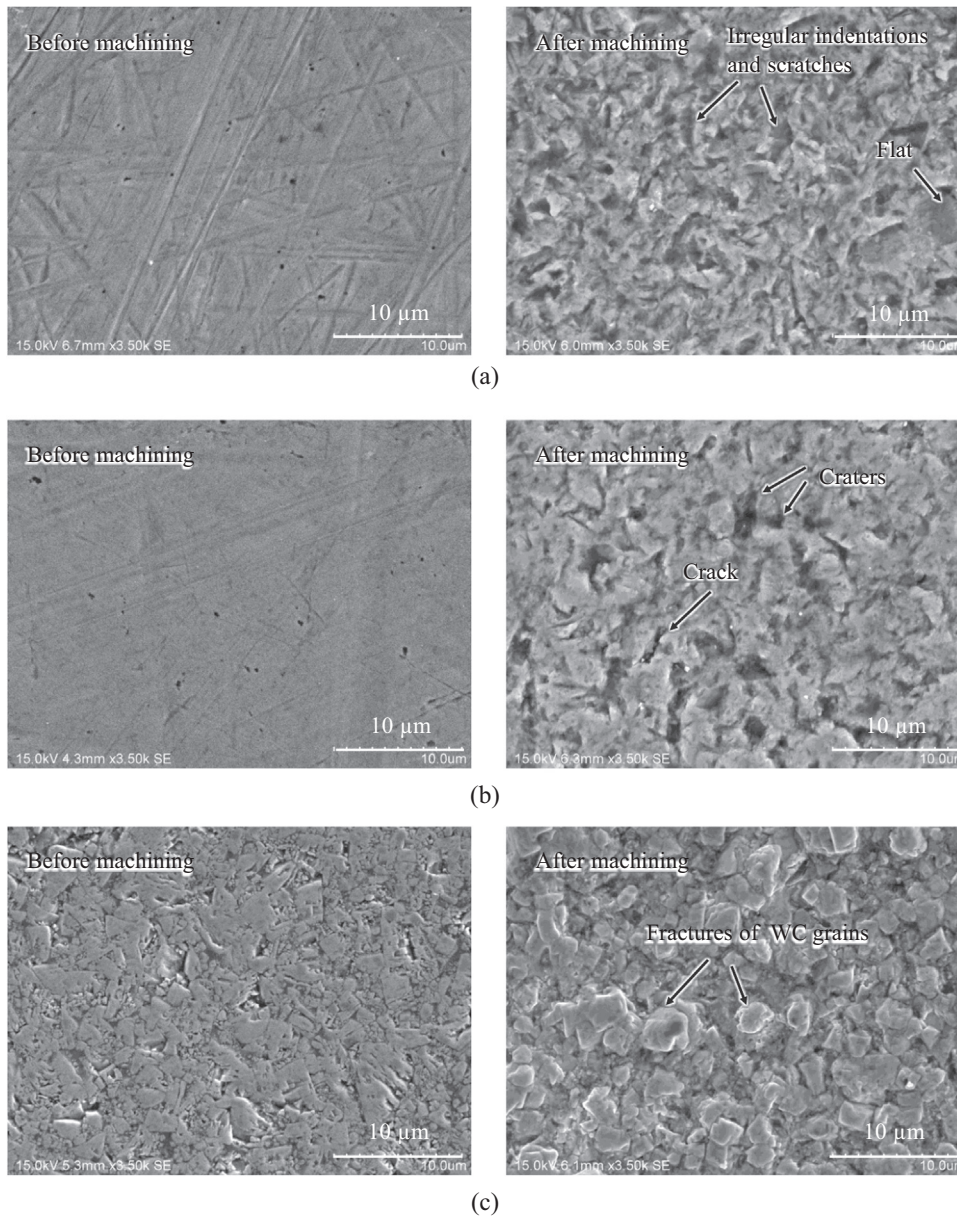


Fig. 19. SEM photographs of samples for hardness measurements: (a) 304 stainless steel, (b) 1045 carbon steel, (c) WC.

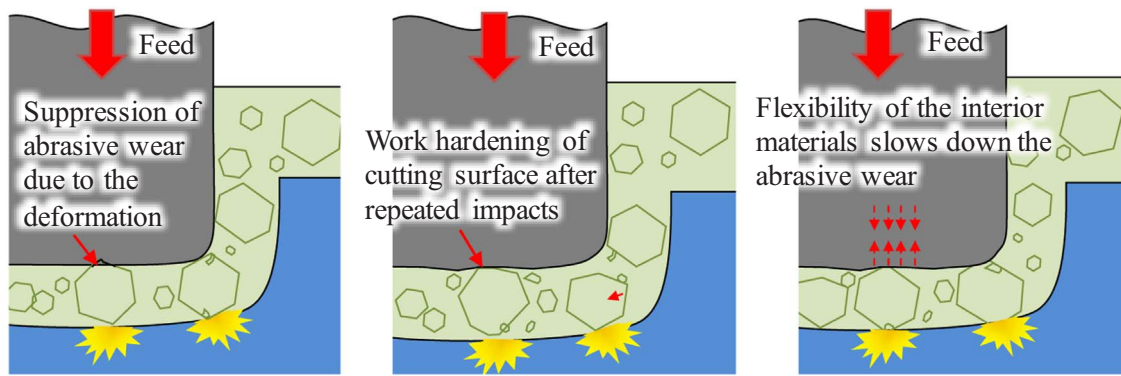
good to maintain the machining gap as shown in Fig. 20(c). The material removal rate for the entire machined area decreases rapidly, so no concavity is formed in the tool. In addition, the high hardness of the tool material can ensure a high resistance to wear.

5. Conclusion

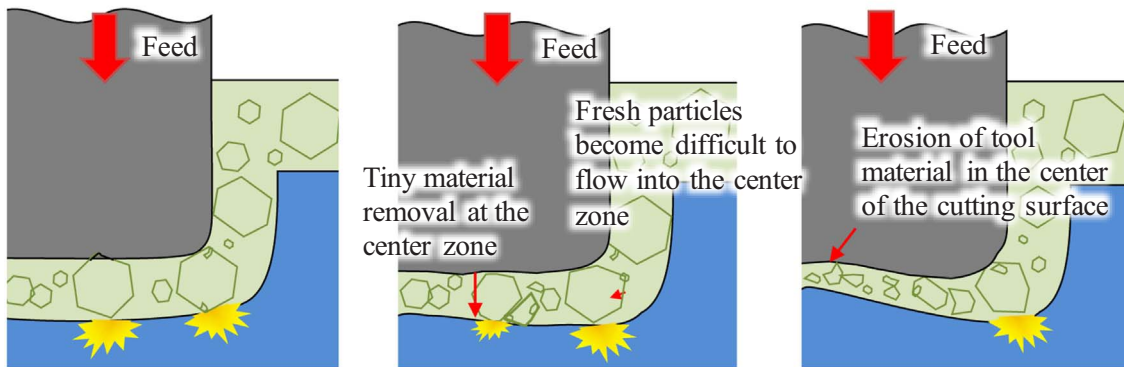
In this work, the tool wear during micro-USM was discussed, which was considered to have an extremely close relation with the material removal process. The tool materials were confirmed to have large effects on the tool wear.

The major conclusions have been drawn as follows:

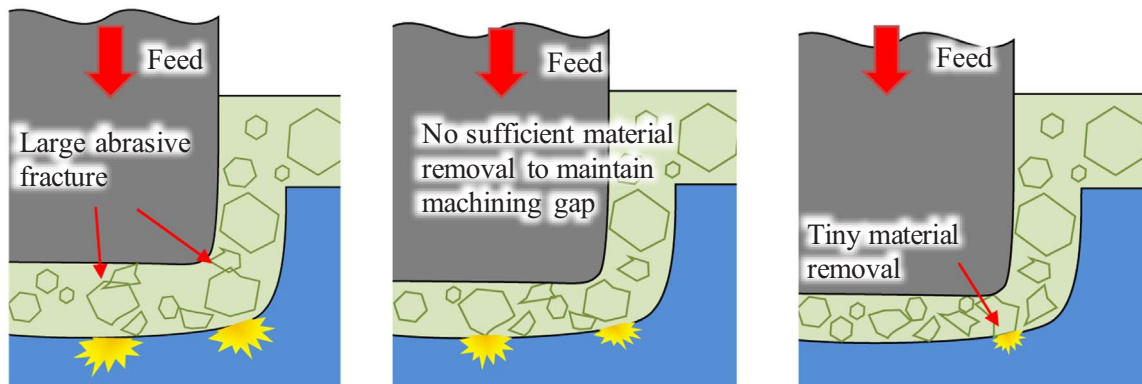
- (1) The tool wear and crack generation were visually demonstrated by using the SPH simulation models, which is significant for understanding the tool wear mechanism and material removal mechanism in USM.
- (2) Steels with a high flexibility such as 304 stainless steel are good choices as tool materials in micro-USM. Slight deformation can occur due to the penetration of abrasive particles, in turn, fractures of the abrasive particles can be slowed down. The moderation of particle wear can retain the machining gap and ensure further material removal.
- (3) Work hardening occurred on the cutting surface of the austenitic 304 stainless steel tool, while the material inside the tool was not hardened and still had a good flexibility. The combination of the two features ensures slow tool wear and particle wear.
- (4) 1045 carbon steel does not have a high tendency to work hardening and does not possess sufficient capacity to undergo plastic deformation before fracture. Cracks and big craters occurred on the tool surface during machining and then prevented fresh abrasive particles flowing into the cutting zone. The fractured particles in the narrow center area were not effective to make material removal, but eroded the tool and led to a concavity on the tool surface. Therefore, the tool material applied in USM should be able to keep a good surface quality under successive impacts of abrasive particles for smooth material removal.
- (5) The wear of abrasive particles is fast when using a hard WC tool because almost no deformation occurs, which is severe to maintain the machining gap. Even though the tool material is too hard to be



(a) Smooth material removal process and small tool wear



(b) Jammed material removal process and large tool wear



(c) Jammed material removal process and small tool wear

Fig. 20. Relation between material removal and tool wear.

eroded, the material removal rate for the whole machined area decreases rapidly. Therefore, the tool material used in USM is not the harder the better.

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ナノ秒パルスレーザによるジルコニアインプラントへの生体適合性付与：微細溝の創成および熱影響の検討

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Imparting Biocompatibility to Zirconia Implants with Nanosecond Pulsed Laser: Formation of Microgrooves and Investigation of Heat Effects

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The purposes of this study were to evaluate the microstructures and chemical composition changes of the surface of tetragonal zirconia polycrystal (TZP) after irradiation of nanosecond pulsed laser and to consider the effects for biocompatibility. Two types of zirconia ceramics, yttria-stabilized TZP (Y-TZP) and ceria-stabilized TZP/alumina nanocomposite (Ce-TZP), were irradiated. To evaluate microstructures, the irradiated samples were observed with a scanning electron microscope (SEM). Laser irradiation blackened the surface of Ce-TZP; contrastingly, it gave no color change on that of Y-TZP. To identify the chemical composition changes, the samples were characterized using X-ray diffraction (XRD), energy dispersive X-ray spectrometry (EDX) and X-ray photoelectron spectroscopy (XPS). To examine the changes in the surface chargeability of zirconia, zeta potential was determined. SEM images showed microgrooves, whose widths were the same as that of laser spot (30 μm), were formed on the surfaces of both samples, and the surfaces of the grooves were roughened by coagulation materials of a few micrometers in size. Such cell-sized grooves with rough surfaces are considered a favorable environment to help osteoblast cells to grow. XRD patterns showed that laser irradiation induced monoclinic-to-tetragonal phase transform. Because the monoclinic phase was induced by machining or polishing, this result means laser irradiation returned the crystal phase to the bulk state. However, XRD patterns did not reveal the reason of the color change. EDX result showed oxygen atoms decreased on the irradiated surface of both samples. In addition, XPS spectra of Zr3d from Ce-TZP showed a part of Zr^{4+} shifted to lower side after irradiation. Considering these points, the color change was caused by the generation of oxygen-deficient zirconia. After laser irradiation, zeta potential of Ce-TZP was decreased. This result suggests the possibility that the amount of deposition of ions and proteins in body liquid is decreased. It is concluded that the grooves to improve biocompatibility can be formed on TZP surfaces by nanosecond pulsed laser, but adverse chemical composition change can be occurred by deficiency of oxygen. Additional process may be needed to prevent the phenomenon. [doi:10.2320/jinstmet.J2018043]

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Keywords: nanosecond pulsed laser, dental implant, zirconia, zeta potential

1. 緒 言

超高齢社会を迎えた日本において、QOL 向上のため歯科におけるインプラント治療は今後需要が拡大することが予想されており、現実には歯科用インプラント材料の生産・輸入数は年々増加している¹⁾。歯科用インプラント(以下、インプラントと称す)において骨との直接的な結合は重要な性質である。インプラントと顎骨が新生骨の形成によって緊密な接合状態を得るには、インプラント埋入手術後数カ月を要す

る。この期間が長期に及ぶと患者の負担が増すばかりか、感染による骨吸収(インプラント周囲炎)のリスクが大きくなり、インプラント脱落に繋がりがかねない。そのため従来は骨と高い親和性を有するチタンあるいはチタン合金がインプラントのフィクスチャー材料として適用されてきた^{2,3)}。しかしながら、チタン材料は金属アレルギーを惹起する可能性がある点やセラミックス製の歯冠修復物と歯肉との間から金属色が露見し美観を損なう点が問題視されている^{4,5)}。そこで近年、金属アレルギーを惹起せず、白色で審美的であるセラミックス材料、中でも、とくに高い強度と靱性を有するジルコニア製インプラントへの注目が高まっている⁶⁾。その中で、ジルコニア表面の新生骨形成を促進するために、生体活

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性を高める表面改質法が検討されている⁷⁾。

ジルコニアの表面改質法は形状変化または化学的組成変化を付与する手法に大別できる。サンドブラスト処理は形状を変化させる代表的手法であり、アルミナや酸化チタンなどの粉末を表面に吹き付けて粗化する⁸⁾。表面粗化による表面積の増大は、細胞が初期接着段階で材料表面に拡散し細胞の分化・増殖することに寄与する^{9,10)}。Yamashita らはブラスト処理によりジルコニア表面に良好な初期細胞接着を得られたと報告している¹¹⁾。しかしながら、ブラスト後に表面に残存した粉体は少量であっても表面特性を変化させ、生体適合性に悪影響を及ぼす可能性がある¹²⁾。

化学的組成変化を付与する表面改質法としては、アパタイトコーティングが挙げられる。骨の主成分であるハイドロキシアパタイトに代表されるリン酸カルシウムをコーティングすることにより、骨の無機質成分の成長を助長し、さらに局所的な Ca イオン濃度を高くすることで骨芽細胞を活性化し、骨形成を促進させる¹³⁾。Sasaki, Hirota らはジルコニア表面にアパタイトを析出させ、骨芽細胞様細胞を培養したところ、良好な増殖を確認している^{14,15)}。この手法はジルコニア表面の生体適合性を向上させる点では有用であるが、工程が複雑で仕上がりを制御することが困難であることに加え、特別な設備や材料、試薬の調整が必要となる。

インプラント材料として用いられる正方晶部分安定化ジルコニア (Tetragonal Zirconia Polycrystal, 以下 TZP と称す) は応力が負荷されると室温で準安定な正方晶から単斜晶に結晶相変態を生じる。この特性は応力誘起相変態機構と呼ばれ、変態に伴い結晶相の体積が 4% 程度増加するため、き裂先端への圧縮応力によりその進展の抑制に寄与している⁶⁾。しかしながら、この強化機構はき裂先端という局所的な領域にのみ有効であり、過度な応力負荷は単斜晶への相変態を助長するため、体積変化に伴うクラックの発生や強度低下の原因となる¹⁶⁾。そのため、機械的接触を伴う手法を施すことは不適切とされている。

本研究では、TZP 製インプラントに対し、骨との適合性を付与する手法としてナノ秒パルスレーザー照射に注目した。ナノ秒パルスレーザー照射では、局所加熱による材料除去により難削材に対して非接触で精密微細加工が可能であり、加えて照射部周辺に数マイクロメートル程度の熱影響層を付与できる改質法である。著者らは、チタンに対して本手法を適用することにより、微細な凹凸形状と熔融物による粗面の創成、および熱反応による酸化膜生成に起因した生体適合性の向上に成功している^{17,18)}。この表面改質は、骨芽細胞の骨形成を活性化させる形状、およびハイドロキシアパタイト析出を向上させる組成を付与し、相乗的な親和性向上を目的に行ったものである⁸⁾。

本研究においては、新規インプラント材料として注目されているジルコニアに対してもこれらと同様な効果の獲得を目的としている。しかしながら、金属であるチタンとセラミックスであるジルコニアとは物性が大きく異なるため、レーザー照射がジルコニア基材に与える影響は明らかになっていない。そこで、本研究ではナノ秒パルスレーザーをジルコニア基材表面に照射し、その改質効果を明らかにするとともに生体

適合性の向上を試みた。本報では、含有物の異なる 2 種類の TZP に対しナノ秒パルスレーザー照射を行い、表面形状および表面組成の変化を確認するとともに、本手法の有用性について検討および考察を行った。

2. 実験方法

2.1 レーザ照射による形状創成

供試材はイットリア添加部分安定型正方晶ジルコニア多結晶 (東ソー社製, TZ-3YB-E, Y_2O_3 3 mol% 含有, 以下 Y-TZP と称す) および、セリア添加部分安定型正方晶ジルコニア/アルミナナノ複合多結晶 (パナソニックヘルスケア社製, NANOZR, CeO_2 10 mol% および Al_2O_3 30 vol% 含有, 以下 Ce-TZP と称す) を用いた。同材を 2 mm × 3 mm, 厚さ 1 mm の平板状に機械加工し、一端面に対し流水しながら耐水研磨紙 (#1200) を用いて算術平均粗さ $Ra < 0.05 \mu m$ となるように仕上げた。

レーザー照射には Nd:YAG ナノ秒パルスレーザー (NEW WAVE RESEARCH 社製, Quicklaze-50 trilit) を用いた。本装置のレーザー照射スポットは Fig. 1 に示すような矩形に整形されており、スポットサイズの調整は光軸の絞りにより行うためスポット内のエネルギー密度はほぼ均一となる。レーザー照射は試料研磨面上に対し垂直に行った。Table 1 にレーザー照射条件

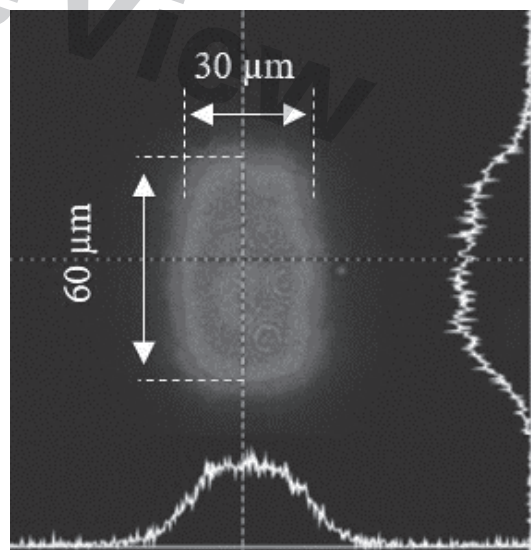


Fig. 1 Diagram of laser spot.

Table 1 Laser irradiation conditions.

Pulse duration	3 ns
Power	150 μJ /pulse
Wavelength	1064 nm
Frequency	50 Hz
Scanning speed	7 μm /s
Laser spot	Rect. 30 μm × 60 μm
Environment	Standard atmosphere

を示す。骨形成は骨芽細胞が分泌するコラーゲン線維にアパタイトが沈着することで生じる¹⁹⁾。基材表面に溝形状を創成することで、骨芽細胞およびコラーゲン線維が溝に沿って伸長し良好な骨形成を得られる^{20,21)}。Fukayo らはナノ秒パルスレーザーによりチタン製インプラント表面に骨芽細胞とおおよそ同じ大きさの幅 $30\ \mu\text{m}$ 程度の溝を創成し、ウサギ大腿骨に 12 週埋入したところ緊密な骨形成を得たと報告している¹⁸⁾。そこでレーザー照射スポットは、骨芽細胞の接着および増殖に寄与されることを目的に幅 $30\ \mu\text{m}$ とし、Fig. 2 のような経路で走査させた。レーザー照射後の溝形状は、形状観察と表面粗さ測定により評価を行った。照射面およびその断面の観察は走査型電子顕微鏡(SEM)を用いて行った。なお断面観察は、試料をレーザー走査方向と垂直に切断した後、切断面を耐水研磨紙で #1200 まで研磨した面に対して行った。また溶融物による粗面の形成を確認するため、レーザー顕微鏡(KEYENCE 社製, VK-X1000)による表面粗さの測定を行った。表面粗さパラメータとしては R_a を適用し、レーザー照射により形成した溝内部の底面に対して Table 2 に示す条件にて測定を行った。

2.2 結晶構造による組成変化

結晶構造の変化による影響を調査するため、レーザー照射後の Y-TZP および Ce-TZP 表面に対し X 線回析(XRD)を行った。X 線回析計(リガク社製, SmartLab3G)において、測定法は $2\theta/\theta$ スキャン法、線源は $\text{CuK}\alpha$ 線を使用し、管電圧は 5 kV、電流 200 mA とし、サンプリング幅 0.02° の条件で測定を行った。以下、対照群はレーザー照射前の Y-TZP および Ce-TZP とする。

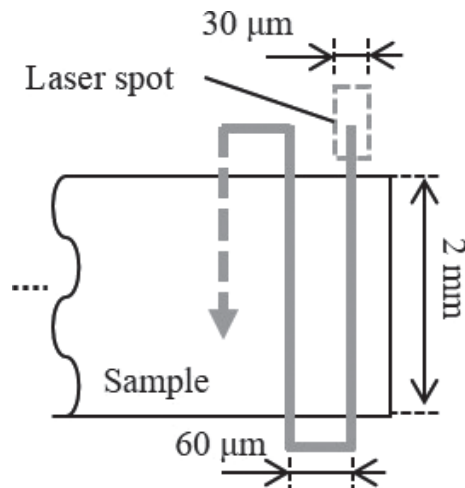


Fig. 2 Laser scanning pass.

Table 2 Roughness measurement conditions.

Magnification	50x
Sampling areas	$2048\ \mu\text{m} \times 1536\ \mu\text{m}$
Measurement length	$200\ \mu\text{m}$
Number of sampling	$n = 20$
Cutoff value	-

2.3 構成元素による組成変化

構成元素を調査するため、レーザー照射前後の Y-TZP および Ce-TZP に対しエネルギー分散型 X 線分光(EDX)を行った。以下、統計学的検定法には t 検定を行い、有意差を確認した。EDX はレーザー照射により形成した溝の凸部上面と溝底面それぞれについて、Fig. 3 に示すような領域に対し測定を行った。また、構成元素の化学結合状態を確認するため、Ce-TZP に対して X 線光電子分光法(XPS)を行った。XPS 分光計(Thermo Fisher Scientific 社製, ThetaProbe)において、線源は単色化 $\text{AlK}\alpha$ 線を使用し、管電圧 15 kV、電力 45 W の条件で測定した。なお、測定前には試料表面を 10 s エッチングし不純物を除去した。帯電効果は表面吸着の C1s スペクトル(285 eV)を用いて補正した。

2.4 レーザ照射後の表面帯電性評価

レーザー照射が生体適合性に与える影響を検討するにあたり、1 指標としてゼータ電位に着目した。そのため、レーザードップラ法によるゼータ電位測定システム(大塚電子社製, ELSZ-2000)を用いて Ce-TZP 表面のゼータ電位を測定し、表面帯電性を評価した。測定条件を Table 3 に示す。

3. 結果および考察

3.1 レーザ照射による形状創成

レーザー照射後の Y-TZP および Ce-TZP の SEM 像を Fig. 4 に示す。図中の(a)-(d)より、両試料の照射面にレーザー照射スポット幅 $30\ \mu\text{m}$ と同程度の溝の創成が確認できる。このことから、ジルコニア基材に対してナノ秒パルスレーザーの照射スポット幅を変化させることより任意幅の溝形状が創成可

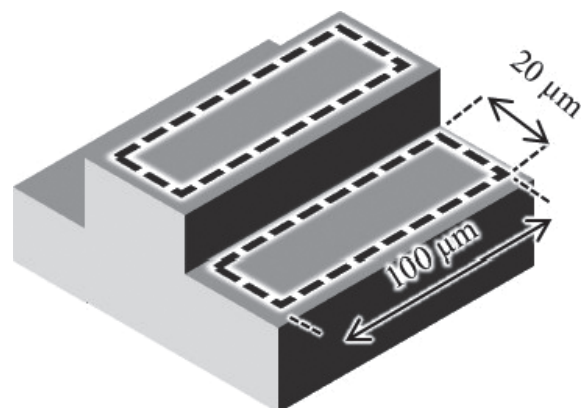


Fig. 3 The region for measurement of EDX.

Table 3 Conditions for measurement of zeta potential.

Solvent	Water
Refractive index	1.3
Viscosity	$8.9\ \text{mPa}\cdot\text{s}$
Permittivity	78.3
Temperature	25°C

能であることがわかった。また、溝内部に溶融凝着物による粗面が形成されていることが確認され、その外観性状は Fig. 4(e) および (f) に示すように、Y-TZP より Ce-TZP の方が凝着物の粒径が大きいことが確認できる。Hayakawa らは、TZP は安定剤の添加が多いほど結晶粒径が大きくなり、とくにセリア添加型 TZP は結晶成長が大きいことを報告している²²⁾。供試材の安定剤添加量については Y-TZP は 3 mol% (Y_2O_3)、Ce-TZP は 10 mol% (CeO_2) であるため、知見に相違ない結果が得られている。

一方、各試料における溝底面の粗さ、および比較として Yamashita らの同材に対し炭化ケイ素粒 (粒径 $125\text{ }\mu\text{m}$) によるサンドブラスト処理面の算術平均粗さ¹¹⁾を Table 4 に共に示す。Yamashita らはサンドブラスト処理面においてマウス頭蓋冠由来骨芽細胞 (MC3T3-E1) を 24 h 培養したところ、研磨面に比べ有意に良好な初期細胞接着を示したと報告している¹¹⁾。このことは、サンドブラスト処理により表面粗さが増加し、細胞の拡散に寄与したことが原因であると考えられている¹⁰⁾。同表より、レーザ照射により創成した溝内部の底面はサンドブラスト処理面より粗いことが確認できる。ゆえに、この溝底面においても粗面が細胞の拡散に寄与し、良好な細胞接着が得られると考えられる。

溝内部の凝着層は溶融過程を経ているため、結晶性や組成

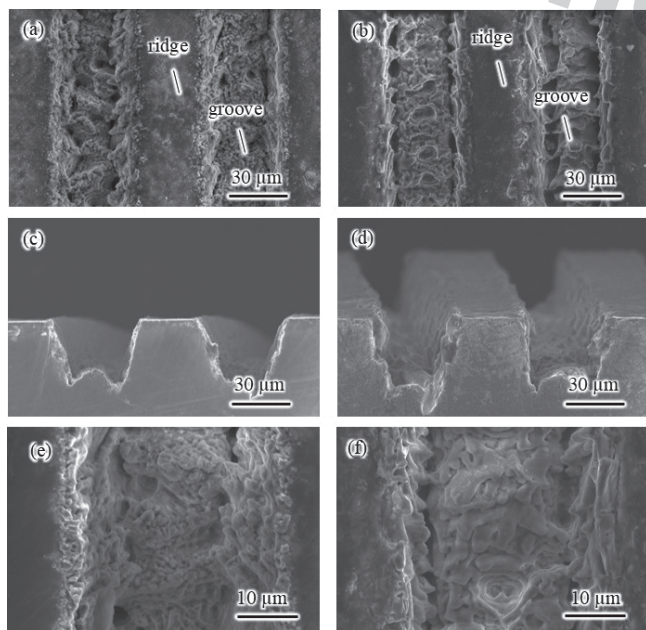


Fig. 4 SEM images. (a) Irradiated surface of Y-TZP; (b) Irradiated surface of Ce-TZP; (c) The cross-sectional face of Y-TZP; (d) The cross-sectional face of Ce-TZP; (e) High magnification image of Y-TZP; (f) High magnification image of Ce-TZP.

Table 4 Surface roughness values of irradiated Y-TZP and Ce-TZP ($R_a \pm$ Standard deviation).

	Y-TZP	Ce-TZP
R_a , μm (Laser-irradiated)	2.42 ± 0.57	1.73 ± 0.47
Skewness	-0.37	-0.83
Kurtosis	0.16	0.10
R_a , μm (Sandblasted) ¹¹⁾	1.01 ± 0.08	1.13 ± 0.10

が焼結体である母材と異なることが推測され、母材との間に異種界面を形成し、界面割れによる層剥離が生じる可能性がある。そこで Y-TZP に対して引掻試験を行い、母材-凝着層の境界近傍を SEM で観察することにより界面の存在を確認した。なお本試験は、Table 1 のレーザ照射条件においてレーザ照射スポットサイズを $60\text{ }\mu\text{m} \times 60\text{ }\mu\text{m}$ に変更し、粗面のみを有する試験片に対して行った。Table 5 に引掻条件を示す。なお、引掻方向はレーザ走査方向と平行に行った。

観察結果を Fig. 5 に示す。同図より、母材-凝着層の境界近傍において凝着層の剥離は確認されなかった。次に、Y-TZP の研磨面 (#1200) に対して破壊が生じるまで引掻試験を行った (荷重 800 g, 往復時間 60 s)。Y-TZP の研磨面に対する引掻痕の SEM 像を Fig. 6 に示す。Fig. 5, Fig. 6 を比較しても、引掻前の粗さにより荷重に差異は生じたが、破壊の様子に変化は確認されなかった。また、Y-TZP のレーザ照射面と研磨面における、引掻試験時の摩擦係数の変化を Fig. 7

Table 5 Scratch conditions.

Pointer	Diamond (Rect. 0.3 mm, R 90°)
Load value	50 g
Reciprocating rate	1 Hz
Reciprocation width	5 mm
Test duration	30 s
Environment	Water (25°)

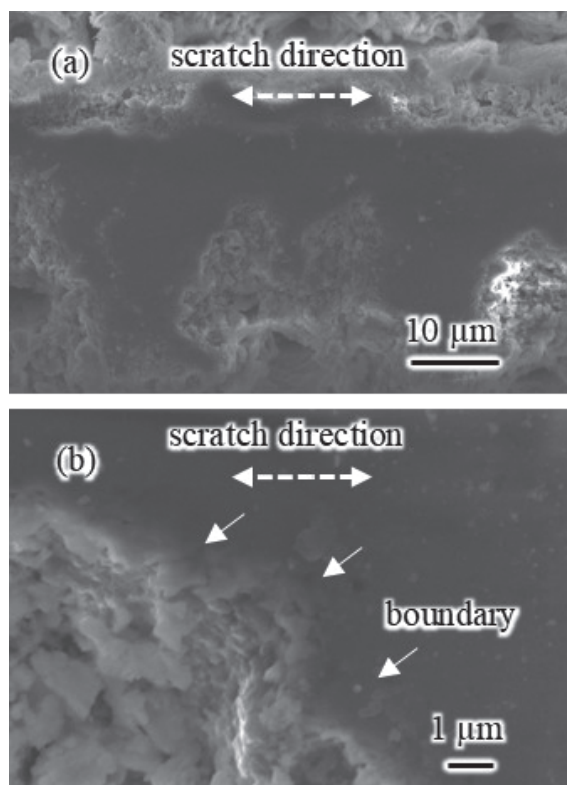


Fig. 5 SEM images of scratch mark. (a) Irradiated Y-TZP; (b) High magnification of (a).

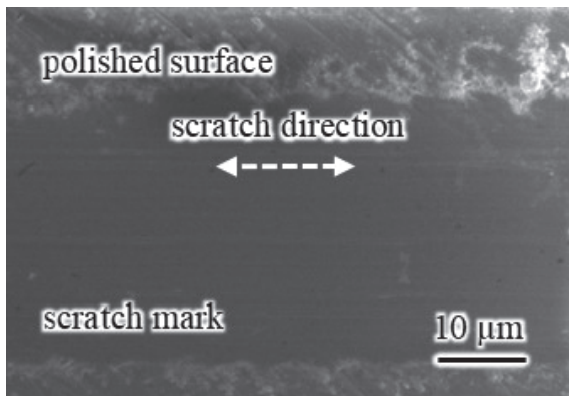


Fig. 6 SEM images of scratch mark of polished Y-TZP.

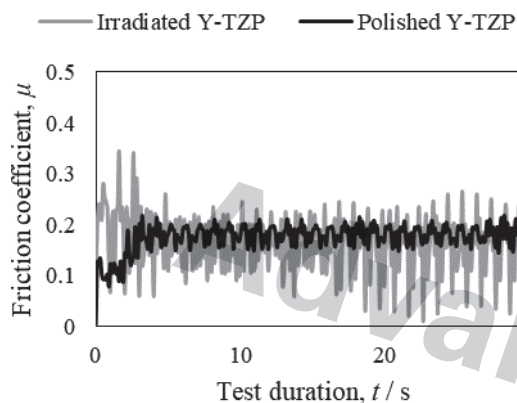


Fig. 7 Friction coefficient of irradiated and polished Y-TZP.

に示す。Y-TZP の研磨面については、試験開始から摩擦係数が増加し、試験時間 10 s 以内に定常値を示した。なお、その後摩擦係数の変化が確認されなかったため、Fig. 7 において 30 s 以降は省略する。研磨面と比較し、レーザ照射面における摩擦係数は上下する。このことは、レーザ照射面が粗いため、圧子から負荷を受け瞬時に破壊が生じたこと、および凝着物による凹凸のため圧子と一定の接触状態を得られず、摩擦力が変化したことが原因であると考えられる。しかし、両者において破壊挙動としての変化は確認されなかった。これらから母材-凝着層間は混合しており、容易に剥離が生じないことが示唆される。Ce-TZP の母材-凝着層間についても、レーザ照射による熔融凝着という点で界面状態は類似しているため、界面は存在しないと考えられる。

3.2 結晶構造による組成変化

レーザ照射後 Ce-TZP の照射面は表層のみ黒く変色した。一方、Y-TZP には変色が確認されなかった。これらのマクロ観察結果を Fig. 8 に示す。この黒化の原因はレーザを照射した際に生じた熱影響による組成変化であり、試料含有物の違いにより熱影響の様子に差が生じたことが示唆される。この原因について調べるため、まずは XRD による結晶構造解析を行った。その結果を Fig. 9 に示す。Fig. 9 より、Y-TZP および Ce-TZP には主に正方晶ジルコニアのピークが確認でき、照射前に見られた単斜晶ジルコニアのピークはレーザ照射後に消失した。また Ce-TZP にはレーザ照射前後を通してアルミナのピークが確認できた。

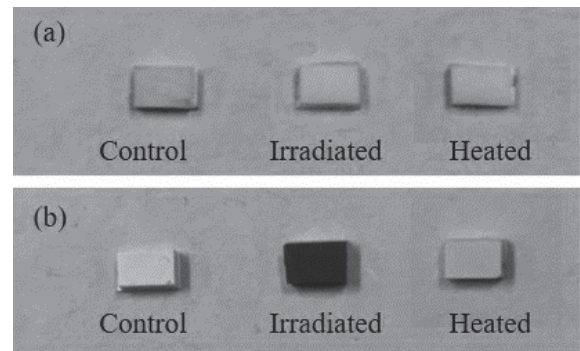


Fig. 8 Photographs of irradiated and heated samples. (a) Y-TZP; (b) Ce-TZP.

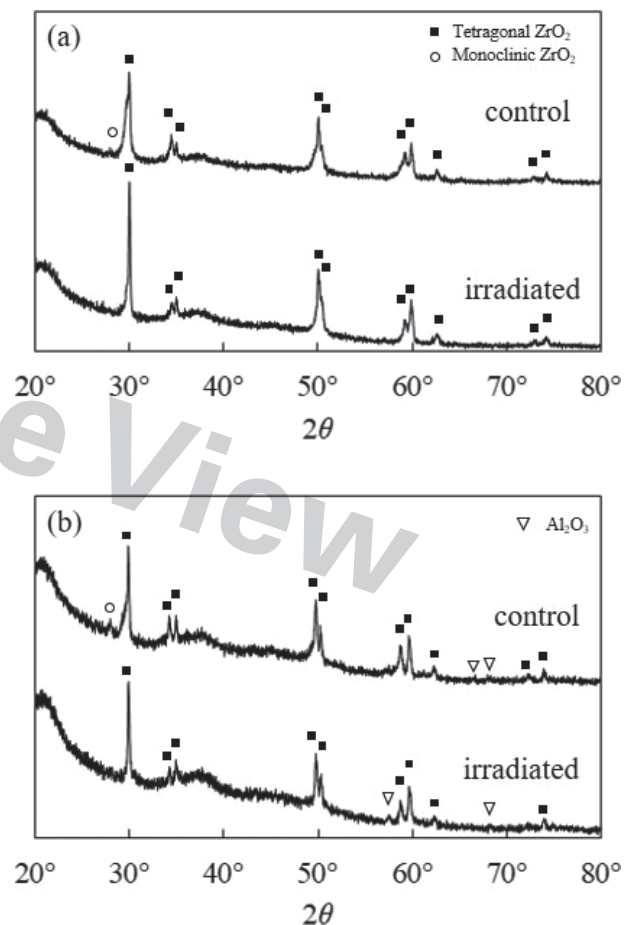


Fig. 9 XRD patterns of TZP surfaces. (a) Y-TZP; (b) Ce-TZP.

正方晶部分安定化ジルコニアは、応力負荷を受けると応力誘起相変態により一部が単斜晶に変態する。そのため、両試料のレーザ照射前に単斜晶ジルコニアのピークが現れたのは、試料の機械加工や研磨時に変態した単斜晶ジルコニアが加工変質層として残存したことが原因であると考えられる。単斜晶ジルコニアがレーザ照射後に消失する現象は Tezuka らが同様に報告をしている²³⁾。純粋なジルコニアは 2370℃ で正方晶から立方晶に変態し、2700℃ で熔融体になることが知られている⁶⁾。レーザ照射部においてジルコニアの熔融が生じていることから、その近傍の熱影響域では単斜晶および正方晶のジルコニアが立方晶に相変態すると考えられる。立方晶ジルコニアは急冷され、再び母材と同様の正方晶で安定するため、単斜晶ジルコニアが消失したと考えられる²³⁾。

また、複合化により Ce-TZP のジルコニア結晶粒内にはアルミナ結晶が析出されているためアルミナのピークが現れた。Fig. 9(b)において、レーザ照射前後におけるアルミナピークの強度のばらつきの原因は、以下2点が考えられる。1点はレーザ照射による材料除去の影響で新生面が現れ、その面を含めたレーザ照射面全体に対してXRDを行ったことである。もう1点は、レーザ照射前の段階で単斜晶ジルコニアが残存しており、レーザ照射によりその単斜晶相が4%程度体積の小さい正方晶ジルコニアに相変態し結晶相の歪みに変化が生じたことである。これらの理由からレーザ照射前後の回折面において結晶の配向性に差異があったと考えられる。しかしながら、以上のことを考慮しても、母材にない結晶相の析出および変態は認められなかった。このことから、Ce-TZP の黒化の原因は結晶相の変化によるものではないといえる。

3.3 構成元素による組成変化

レーザ照射後の組成変化に対する要因として、構成元素の変化、とりわけレーザ照射による温度上昇に起因した酸化状態の変化が考えられる。そこで次に Ce-TZP の黒化の原因が素材を構成する元素の組成変化であると予想し、まずは EDX による元素分析を行った。その結果、レーザ照射後の両試料において酸素原子数濃度の低下が確認された。酸素原子数濃度の変化を Fig. 10 に示す。なお、レーザ照射後の酸素原子数濃度はレーザ照射前の平均値に対する割合を百分率で示したものである。Fig. 10 より、両試料ともにレーザ照

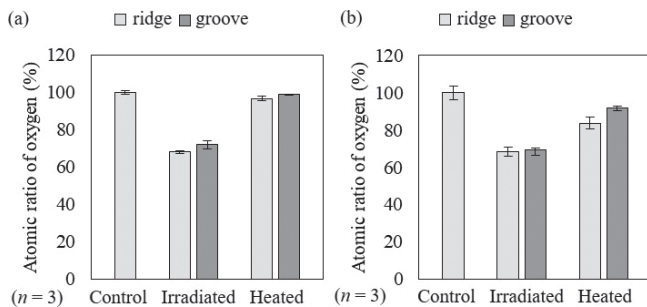


Fig. 10 Atomic ratio of oxygen ($p < 0.01$ when comparing irradiated and control, irradiated and heated, respectively). (a) Y-TZP; (b) Ce-TZP.

射後、酸素原子数濃度が30%程度有意に低いことが確認できる ($p < 0.01$).

EDX の結果を踏まえ、レーザ照射ジルコニア表面の構成元素について、より詳細な検討を加えるため XPS による測定を行った。その結果を Fig. 11 に示す。また Fig. 11 の中で、特にレーザ照射前後に変化があった Zr3d スペクトルをピックアップし、ピーク分離したものを Fig. 12 に示す。ここで、Table 6 に分離した各ピーク位置の束縛エネルギー値、Table 7 に酸化状態別の Zr3d 束縛エネルギー値を示す²⁴⁾。Table 7 を参考とすると、レーザ照射前に確認された 183.0 eV、185.4 eV のピーク (I) はそれぞれ $Zr^{4+}(3d_{5/2})$ 、 $Zr^{4+}(3d_{3/2})$ に由来すると考えられる。Fig. 12 から、レーザ照射後に確認されたピーク (IV) はピーク (I) が低エネルギー側にシフトしたものだと考えられる。また、ピーク (V) はピーク (III) がピーク (II) と同じ束縛エネルギー値までシフトしたものだと考えられる。Table 7 に示されるように、ジルコニアは低価数になるほど、束縛エネルギーが低下する。これらから、レーザ照射によりジルコニアが還元され、レーザ照射前の酸化状態より低価数のジルコニアが生成されたと推測される。

ジルコニアの黒化現象は、レーザ照射および還元性雰囲気での熱処理において確認されている²³⁾。雰囲気中の酸素分

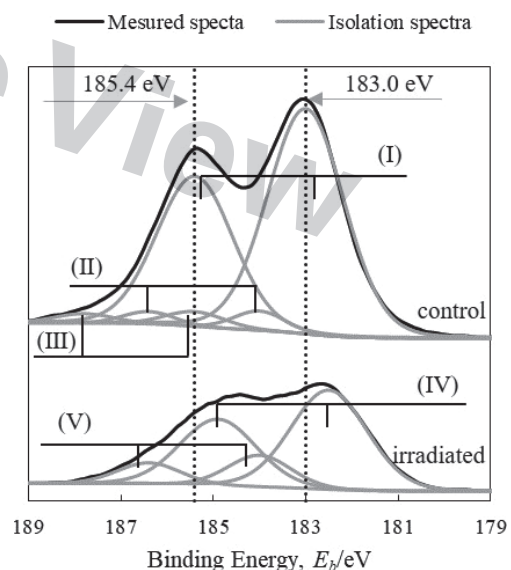


Fig. 12 Core-level XPS spectra of Zr3d from irradiated Ce-TZP.

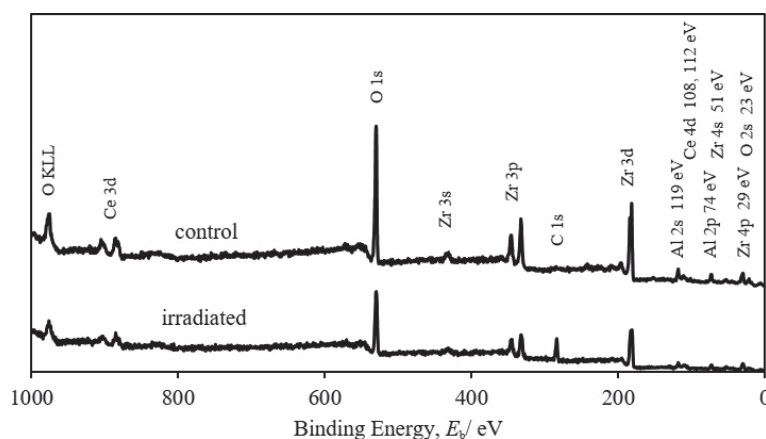
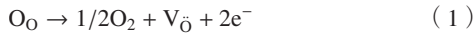


Fig. 11 XPS survey spectra from irradiated Ce-TZP.

圧が低下すると、格子位置にある酸素イオンが空孔と電子を残し酸素ガスとして脱離することで、酸素空孔が増加する^{25,26)}。この反応を以下に示す。



ここで、 O_O 、 V_O はそれぞれ格子位置にある酸素イオン、酸素空孔である。このとき生じる過剰電子により、ジルコニウムイオンの一部が次のように還元する^{25,27)}。



ジルコニアの黒化は、式(2)、(3)のような反応から酸素空

孔を多く有する酸素欠損ジルコニア(ZrO_{2-x})が生成されたため、格子内の酸素空孔が特定波長の光を吸収する、いわゆる着色中心となっていることが原因であると考えられる²⁸⁾。EDXの結果から酸素欠損、XPSの結果からジルコニウムイオンの還元を確認した。したがって、レーザ照射後にCe-TZP表面には酸素欠損ジルコニアが生成している可能性が示唆された。

この酸素欠損ジルコニアに対し、酸素雰囲気または標準大気雰囲気で熱処理を行うことで空孔に酸素イオンを充填し、白色を回復できたという報告がある^{29,30)}。そこで、レーザ照射後の黒化が酸素欠損ジルコニアによるものであることを確認するため、レーザ照射後のY-TZP、Ce-TZPに対し電気炉(光洋サーモシステム株式会社製、KBF442N1型)を用いて熱処理を行った。熱処理条件は室温から1000℃まで1hで昇温し15min保持した後、約100℃まで炉冷した。

熱処理後の試料はFig. 8に示した。Ce-TZPについては、熱処理により照射後の黒化が緩和されていることが確認できる。なお熱処理後、両試料表面の形状については変化がなかった。純粋なジルコニアの融点が2700℃程度であるため、十分に低い温度で熱処理を行うことで形状に変化は生じないと考えられる。また、熱処理後の表面の酸素原子数濃度の変化はFig. 10に示した。熱処理後、両試料はレーザ照射後に比べ酸素原子数濃度が有意に高いことが確認できる($p < 0.01$)。このことからCe-TZPの黒化は還元による表面の酸素欠損が原因であると考えられる。また、Y-TZPもレーザ照射後に酸素原子数濃度は低下しているが、試料の様子に変化は確認されない。このことからジルコニアが黒化に至るための酸素原子数濃度には閾値が存在し、その閾値は含有物によって異なるため、Y-TZPは黒化に至らなかったと考えられる。

酸素欠損ジルコニアの生成については、パルスレーザ照射に伴う下記の機構が考えられる。レーザ照射により材料が気化・蒸発することによりプラズマを形成し、このプラズマがレーザ光と結合し衝撃波を発生させ、爆発的な材料の除去が生じる^{31,32)}。照射部周辺において、昇温による燃焼や衝撃波による飛散のため、瞬間的に酸素分圧が低い領域が発生すると考えられる。酸素欠損ジルコニアの生成機構の模式図をFig. 13に示す。このようにジルコニア表面のレーザ照射部では、高温かつ低酸素分圧という還元雰囲気での熱処理と同様な状態が得られたため酸素欠損ジルコニアの生成に至ったと考えられる。なお、レーザ照射による酸素欠損ジルコニアの生成は、酸素雰囲気下でレーザ照射を行うことで抑制可能

Table 6 Binding energy at the peak position of each isolation spectrum.

Spectrum	Line	E_b / eV
(I)	3d _{5/2}	183.0
	3d _{3/2}	185.4
(II)	3d _{5/2}	184.0
	3d _{3/2}	186.4
(III)	3d _{5/2}	185.4
	3d _{3/2}	187.8
(IV)	3d _{5/2}	182.5
	3d _{3/2}	184.9
(V)	3d _{5/2}	184.0
	3d _{3/2}	186.4

Table 7 Binding energy of Zr3d corrected by assuming the binding energy of gold 4f_{7/2} line at 83.9 eV.

Oxidation state	Line	E_b / eV
ZrO ₂	3d _{5/2}	182.9±0.1
	3d _{3/2}	185.2±0.1
(ZrO) ₁	3d _{5/2}	180.7±0.1
	3d _{3/2}	183.0±0.1
(ZrO) ₂	3d _{5/2}	179.7±0.1
	3d _{3/2}	182.0±0.1
Clean Zr	3d _{5/2}	178.7±0.1
	3d _{3/2}	181.1±0.1

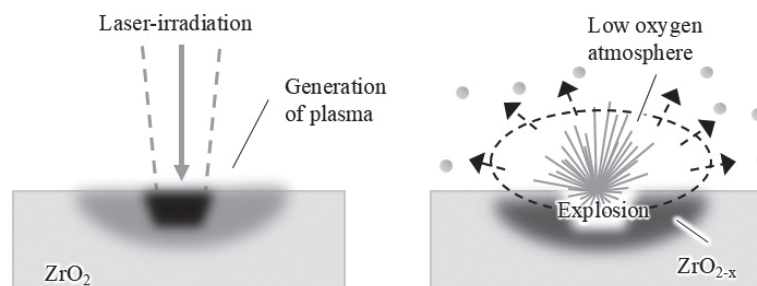
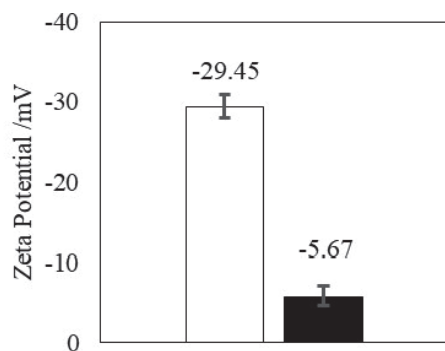


Fig. 13 Generation mechanism of oxygen-deficient zirconia by laser irradiation.

であると考えており、今後の検討項目である。

3.4 レーザ照射後の表面帯電性評価

インプラント表面に電荷をもたせることで、体液中の電荷を有するイオンやタンパク質が静電引力的作用により表面に誘導され、細胞組織の成長促進や接着力強化が期待できる。ゼータ電位は固体表面の帯電状態を表す値であり、生体適合性の表面組成による寄与を評価できる⁸⁾。Fig. 14 にゼータ電位の測定結果を示す。Fig. 14 において、レーザ照射前後でジルコニア表面は負に帯電している。体液中において、負帯電表面には Ca^{2+} など 2 価の正イオンが優先的に吸着し、その後 HPO_4^{2-} などの負イオンや負帯電した骨性タンパク質が順に吸着しアパタイトを析出する³³⁾。この吸着機構を Fig. 15(a) に示す。ここではジルコニア表面の静電引力的作用の大きさを議論するため、ゼータ電位の絶対値に着目した。ゼータ電位の絶対値はレーザ照射後に 5 分の 1 以下まで優位に低下したことが確認できる ($p < 0.01$)。一般に、酸化物表面の帯電に寄与しているのは、水の解離吸着により生じる OH 基の存在である³⁴⁾。このことから、ゼータ電位低下の原因は以下の 2 点が考えられる。1 点はレーザ照射による Ce-TZP 表面の酸素原子数低下であり、もう 1 点は材料内の酸素イオンが酸素欠損により増加した空孔間を移動し、ジルコニア表面に導電性が発現したことである。以上の 2 点により水の解離吸着が生じにくくなり、Fig. 15(b) に示すように OH 基の形成量を減少させたため、ゼータ電位の絶対値が低下したと考えられる。ゼータ電位の絶対値低下により Ce-TZP 表面の静電引力的作用も低下するため、体液中において生体組



(n = 3) □ Control ■ Irradiated

Fig. 14 Zeta potential of irradiated Ce-TZP ($p < 0.01$).

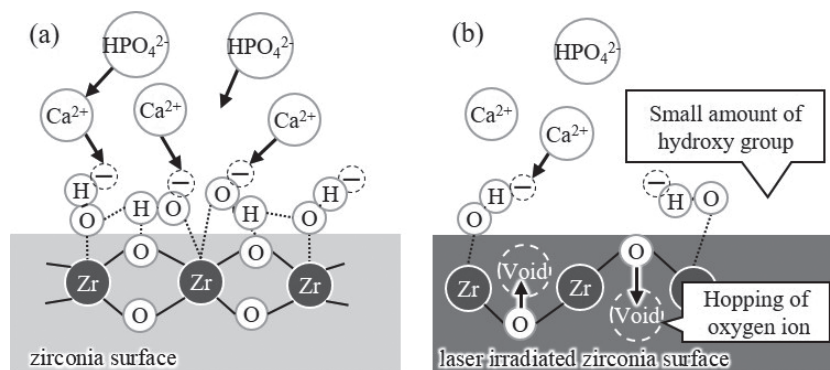


Fig. 15 Difference of deposition behavior. (a) Before laser irradiation; (b) After laser irradiation.

織析出に悪影響を及ぼすことが示唆された。Y-TZP については、Fig. 10 に示すように表面酸素原子数濃度が低下するが黒化には至らないため、ゼータ電位が Ce-TZP 同様に低下を示すかは検討課題である。著者らは 6 週齢 Wistar ラット脛骨に同試験片を埋入し、光学顕微鏡にて埋入 4 週後のインプラント-骨の接触率を測定したところ、レーザ照射を行った Y-TZP の接触率が増加し、Ce-TZP は低下したという結果を得た³⁵⁾。レーザ照射 Ce-TZP インプラントに対して骨接触率が低下した原因が黒化とそれに伴う変化であるならば、3.2 で述べたように、黒化に対し加工雰囲気調整することで解決することが可能であり、それを適切に利用することで、ゼータ電位の調整により生体適合性の改善も可能であると考えている。

4. 結 言

骨芽細胞の骨形成を活性化させる形状、およびハイドロキシアパタイト析出を向上させる組成の付与を目的に、新規歯科用インプラント材料として注目されるジルコニアに対し、ナノ秒パルスレーザを用いた表面改質に試みた。Y-TZP および Ce-TZP に対し、ナノ秒パルスレーザ照射を行い表面形状および表面組成の変化を確認するとともに、本手法の有用性について検討・考察を行った。以下に本報で得られた結論を示す。

- (1) 矩形整形したナノ秒パルスレーザの照射スポットを変化させることで、ジルコニア表面に微細形状の創成と形状制御が可能であることが明らかとなった。
- (2) ナノ秒パルスレーザの照射により、結晶相は試料作成時に変態した単斜晶から母材と同じ正方晶に戻るため、結晶相劣化を緩和できることが明らかになった。
- (3) ナノ秒パルスレーザの照射により、Ce-TZP の照射面は黒化した。これは酸素欠損ジルコニア (ZrO_{2-x}) の生成が原因であり、この黒化は熱処理により緩和できることが明らかになった。
- (4) ナノ秒パルスレーザの照射により、Ce-TZP の表面ゼータ電位は低下するため、体液中において生体組織析出に悪影響を及ぼすことが示唆された。

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論文

プラズマショット法と研削加工による低摩擦・低摩耗面の創成

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Low wear / low friction interface generation by plasma-shot treatment and grinding processing

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著者らは、摩擦面に耐摩耗性を付与する表面処理技術としてプラズマショット法を提案しており、炭化チタン (TiC) 電極を用いることで凹凸形状をもつ高硬度改質層が得られるメカニズムを明らかにしている。本研究では、プラズマショット法と研削加工を単一機械上で適用することで、摩擦面の高硬度化と、摩擦低減効果が期待できるプラトー構造を有する放電ディンプルの創成を同時に実現するプラズマショット研削 (PSG: Plasma shot grinding) 法を新たに提案し、本手法の開発に向けた実験的考察を行う。はじめに、プラズマショット装置と精密平面研削盤を用いて放電ディンプルを作成し、改質面における TiC 残存性評価および形状評価を行った。その結果、摩擦面における TiC の残存および放電ディンプル作成に好適な研削条件を明らかにした。また、同試料の摩擦試験を行い、放電ディンプルのくさび作用による摩擦低減を確認し、研削面粗さによる摩擦特性への影響を明らかにした。最後に PSG 実験を行い、各種実験で得られた知見から本手法の実用可能性を示した。

Key words : friction, wear, plasma-shot treatment, plasma-shot griding, dimple, plateau structure

1. 緒言

機械には摺動部や回転部など多くの摩擦面が存在しており、機能・性能・信頼性などはこれら摩擦面の摩擦・摩耗特性、すなわち広義の潤滑特性に大きく影響される¹⁾²⁾。そのため、低摩擦・低摩耗性を摩擦面に付与する加工・改質手法の開発が多くの研究者によって進められている。一般的に、摩擦部は潤滑油などで満たされていることが多く、油膜を効率的に形成することで摩擦係数の低減につながる³⁾。この油膜形成には表面微細構造の創成が有効とされ、耐摩耗性の向上には摩擦面の高硬度化が有効である²⁾。そこで本研究では、微細構造による摩擦低減および高硬度化による摩耗低減に着目し、それらを実現するプラズマショット研削 (PSG: Plasma-shot grinding) の手法開発を行う。

2. プラズマショット研削法の提案

著者らは、摩擦面に耐摩耗性を付与する表面処理技術としてプラズマショット法 (PS 法) を提案している⁴⁾。本手法は、電極をプラス極性、基材をマイナス極性としてパルス放電を発生させ、そのエネルギーにより基材表面に電極材料の化合物を転移させることで改質層を形成する手法である⁵⁾。また、電極材料としてチタン (Ti) 系電極を用いることで炭化チタン (TiC)

から成る高硬度改質層が得られることが報告されている⁶⁾。本研究で新たに提案するプラズマショット研削 (PSG) は、PS 法と研削加工を複合した加工法である。例えば、図 1 に示すように汎用加工機のスピンドルに軸付き電極を取り付けてプラズマショット法を適用し、その後、一般の軸付き砥石に取り付け替えて研削を行う。これにより、研削時に改質層の盛り上がり部を除去しプラトー部を創成できる。以下、この構造を放電ディンプルと称する。放電ディンプル創成の概略図を図 2 に示す。ディンプルは、くさび作用によって潤滑形態を流体潤滑領域に遷移させることで摩擦低減に有効である³⁾。また、電極に TiC 電極を用いることで放電ディンプル創成と同時に高硬度改質層が得られるため、本手法によって低摩擦・低摩耗面の創成を単一機械上で行うことが可能となる。

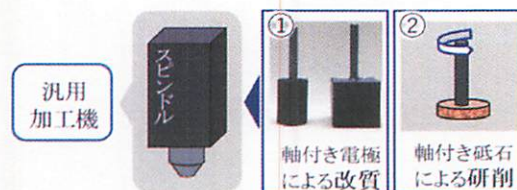


図 1 プラズマショット研削の概要

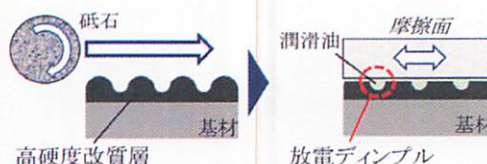


図 2 放電ディンプル創成の概略図

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本報ではとくに、PS 法および研削加工のそれぞれの専用機による加工を行い、放電ディンプルの形成機構や表面改質効果の有無など、本手法の基礎的な知見について検討した。また、それらの知見をふまえ、図 1 に示したような単一機械で PSG を可能とする装置を開発し、その実用可能性について検討を行った。

3. プラズマショット研削の基礎検討に用いた装置

先述のように、本研究ではまず、PS 法と研削加工のそれぞれの専用機を用いて PSG 法に関する基礎的な知見について検討を行った。以下に用いたプラズマショット装置と精密平面研削盤について示す。

3.1 プラズマショット装置

PS 法には、型彫放電加工機をベースとするプラズマショット装置(三菱電機社製, ES041-A)を用いた。本装置は 4 軸制御加工が可能であり、最小位置決め分解能は X, Y, Z 軸で 1 μ m, C 軸で 0.001°である。電極と基材を接触させた際の短絡を検知し位置決めを行う。改質時は TiC 電極をマイナス極性、基材をプラス極性とし、極間にパルス放電を発生させて基材表面の改質を行った。改質は不水溶性の金属加工油中で行った。PS 法における改質条件を表 1 に示す。なお、研削における基準面を明瞭化するため、短時間改質によって単発放電痕が均一に分布する試料を作製した。

3.2 精密平面研削盤

プラズマショット面の研削には、精密平面研削盤(ナガセインテグレックス, SGC-630S4-Zero3)を用いた。砥石にはビトリファイドボンドのダイヤモンド砥石を用い、基材表面での研削音により位置決めを行った。研削時は加工対象へのダメージの軽減および工具であるダイヤモンドの硬度を維持することを目的として、水溶性研削液による湿式研削を行った。研削条件は表 2 に示すとおりであり、砥石半径切込み深さ Δ を異なる 3 条件に設定した。なお Δ の定義は図 3 に示すように未放電加工面を基準とし、正方向を材料に切込む方向としている。

4. プラズマショット面の研削による放電ディンプル創成

4.1 改質面の TiC 残存性評価

PSG では研削により放電痕の盛り上がり部を除去するため、TiC 改質層自体が除去されてしまう可能性がある。そこで本項では、エネルギー分散型 X 線分析(EDX: Energy dispersive X-ray spectrometry)によって、改質面における TiC 残存性に対する Δ の影響について検証する。基材には、摺動部材として多く用いられるねずみ鋳鉄 FC250 を用いた⁷⁾。

研削前後の単発放電痕の EDX 像を図 4 に、Ti 原子数濃

表 1 プラズマショット法の改質条件

使用電極	TiC 圧粉体電極
放電電流	21A
無負荷電圧	260V
パルス幅	8 μ s
休止時間	256 μ s
改質時間	10s

表 2 プラズマショット面の研削条件

砥粒	ダイヤモンド
砥粒径	#800
砥石周速	14m/s
送り速度	4m/min
砥石半径切込み深さ Δ	-5 μ m, 0 μ m, 5 μ m



図 3 単発放電痕形状と砥石半径切込み深さ Δ の関係

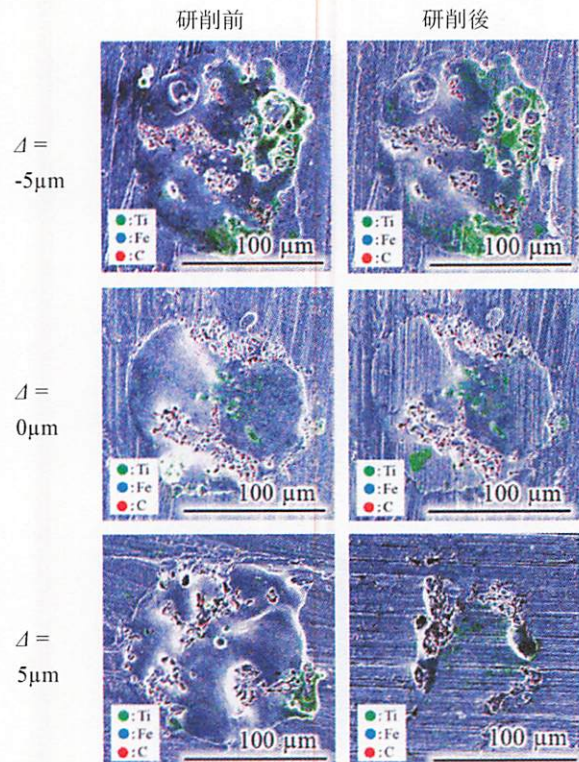


図 4 研削前後の単発放電痕 EDX 像

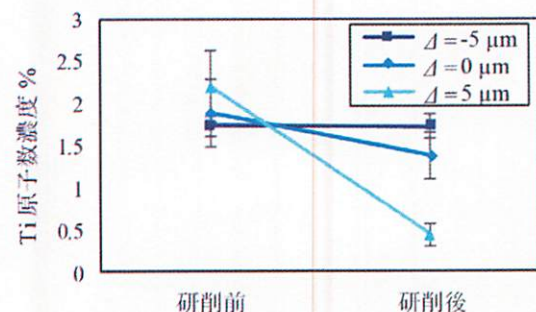


図 5 研削前後のプラズマショット面の Ti 原子数濃度変化

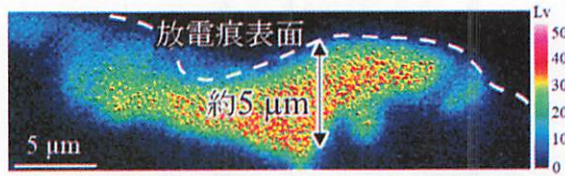


図6 単放電痕断面のTi分布

度の変化を図5に示す。両図より、プラズマショット面を研削した場合でもTiが存在しており、PS法による改質層が除去されることが残存していることがわかる。また $\Delta=0, 5\mu\text{m}$ においては、放電痕の盛り上がり部が除去されている様子が確認でき、所望のプラトー構造を創成可能であることも明らかとなった。

次に、電子線マイクロアナライザ (EPMA: Electron Probe Microanalyzer) によって断面の元素分析を行った。断面試料は、ワイヤ放電加工によってプラズマショット面を切断し、コロイダルシリカを用いて切断面を研磨することで作成した。本改質条件における単放電痕断面のTi分布を図6に示す。同図から、Tiが約 $5\mu\text{m}$ の深さまで存在していることがわかり、先の実験結果を裏付けるものとなった。ただし、TiC改質層がある値の深さをもつことから、 Δ を改質層厚さより小さく設定することによってその除去量を制御する必要がある。例えば図5より、 $\Delta=-5, 0, 5\mu\text{m}$ におけるTi原子数濃度の減少率はそれぞれ0.4%、27.3%、80.7%となっており、Ti原子数濃度が深さ方向に対して減少している。すなわち、本試料においては $\Delta \leq 0\mu\text{m}$ で十分なTiCが研削面に存在するといえる。

4.2 放電ディンプルの形状評価

本項では、非接触式3次元測定装置を用いて、4.1で作成した放電ディンプルの詳細な形状評価を行い、放電ディンプルの作成に最適な Δ について考察する。

研削前後のプラズマショット面のトポグラフィおよびプロファイルシリーズを図7に示す。プロファイルシリーズとは、トポグラフィにおける各断面のプロファイル全てを重ね合わせて表示した画像であり、盛り上がり量 h_p および溶融池深さ、すなわちディンプル深さ d_p を可視化することが可能である。図7(a)より、本条件におけるプラズマショット面は $h_p=10\mu\text{m}$ 、 $d_p=8\mu\text{m}$ であることがわかる。研削後の結果より、 $\Delta=-5\mu\text{m}$ では $h_p=3\mu\text{m}$ および $d_p=8\mu\text{m}$ 程度の放電ディンプルを形成していることが確認された。また $\Delta=0, 5\mu\text{m}$ では、どちらも盛り上がり部が除去され、それぞれ $d_p=8\mu\text{m}$ および $3\mu\text{m}$ 程度の放電ディンプルを形成していることが確認された。

以上の結果は、図8に示す各試料の負荷曲線からもその傾向が定量的に確認できる。 $\Delta=0\mu\text{m}$ において確認された約 $1\mu\text{m}$ の盛り上がりは、高い硬度をもつ放電痕が研削後に弾性回復することで基材表面から盛り上がったためであると考えられる。この負荷曲線における歪度Sskおよび尖度Skuの変化を図9に示す。同図において、 h_p が小さくなるほどSskは負に大きな値となり、 d_p が小さいほどSkuは正に大きな値となるため、 Δ の増加に伴ってこの傾向を取っていることがわかる。

これらの結果より、 $\Delta=-5\mu\text{m}$ では盛り上がり部が除去しきれず残存し、 $\Delta=5\mu\text{m}$ では多くの放電ディンプル部が研削によ

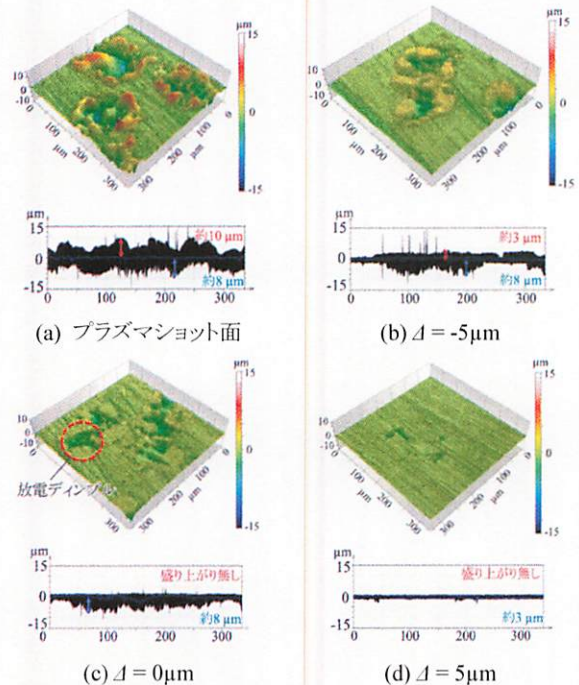


図7 研削前後のプラズマショット面のトポグラフィおよびプロファイルシリーズ

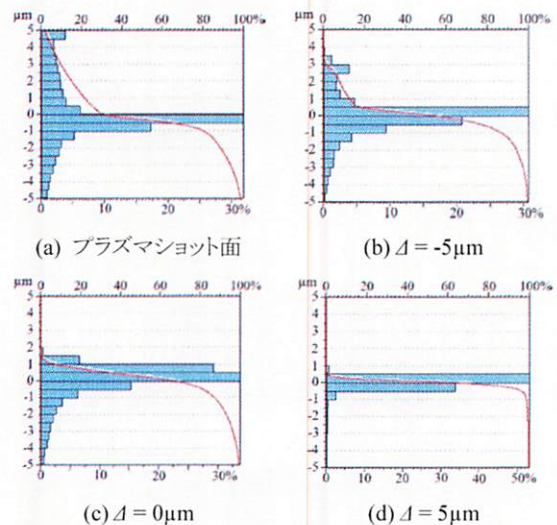


図8 研削前後のプラズマショット面の負荷曲線

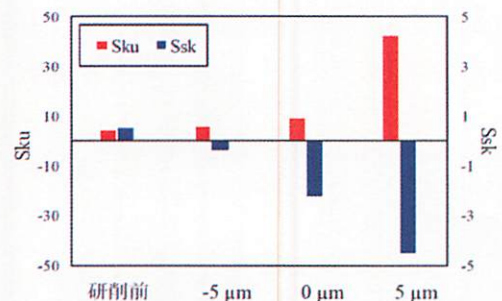


図9 研削前後のプラズマショット面の負荷曲線における歪度Sskおよび尖度Sku

て取り除かれたことから、 $\Delta = 0\mu\text{m}$ が摩擦係数の低減に対して好適とされるブラトー構造の作成に最適であるといえる。

4.3 放電ディンプル試料の摩擦特性評価

本節では、とくに放電ディンプルの形状が顕著に現れた $\Delta = -5, 0\mu\text{m}$ の試料の摩擦試験を行い、放電ディンプルによる摩擦特性への影響を検証する。

摩擦試験の様子を図 10 に示す。本試験では試料上に静置したスライドガラスをピンに接着し、接触面積を増加させることで摩擦面に作用する放電ディンプルを増加させ、その影響を検証する。研削痕の影響を少なくするため摩擦方向を放電痕に対して平行に設定した。試験条件は表 3 に示すとおりである。

$\Delta = 0\mu\text{m}$ 、荷重 10g の時の摩擦係数の経時変化を図 11 に示す。同図より、試験中に摩擦係数が 0.01 以下から約 0.36 まで大きく変化していることがわかる。また、一般的に摩擦係数は、境界潤滑で 0.01 ~ 0.20、流体潤滑で 0.01 以下となることが知られている⁹⁾。本試験においては摩擦面を往復運動させているため、常に摩擦速度が変化する。これにより、試験中連続的に潤滑油膜の厚みが変動し潤滑形態が変化していることが考えられる。放電ディンプルは、くさび作用による動圧効果により潤滑油膜の厚みを増加させ、潤滑状態を流体潤滑へ遷移させるため、本試験で得られた摩擦係数の平均値を比較することでその影響を検討することが可能である。試験によって得られた摩擦係数の平均値を図 12 に示す。同図のとおり、荷重が小さいときは放電ディンプル試料における摩擦係数が減少した。これは、放電ディンプルの動圧効果の影響が荷重に対して大きくなるためであると考えられる。荷重が 500g のとき、 $\Delta = 0\mu\text{m}$ で摩擦係数が増加しているのは、研削による面粗さが基材に比べて大きいためであると考えられる。また $\Delta = -5\mu\text{m}$ においては、ディンプルの周囲に盛り上がり部が残存しているにも関わらず摩擦係数が減少した。これは、盛り上がり部の上面のみがガラスと接触し、接触面積の減少によって境界・混合潤滑での摩擦係数が低減したためであると考えられる。

4.4 研削面粗さによる摩擦特性への影響

PSG 法による摩擦面の面粗さは主に研削に用いる砥石の条件に依存する。また、表面の粗さによって摩擦係数が変化

表 3 摩擦試験条件

荷重	25, 50, 100, 200, 500g
線速度	30mm/s
往復幅	5mm
時間	10min
潤滑状態	湿式(潤滑油添加)

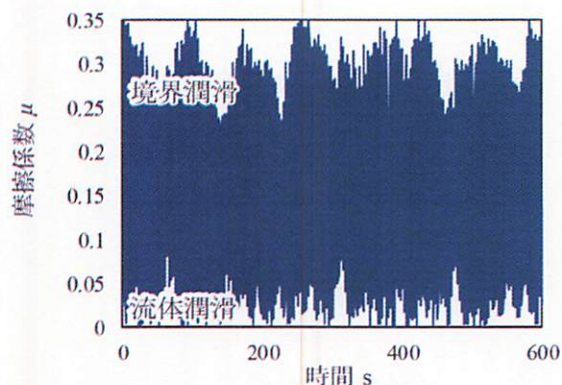


図 11 摩擦係数の経時変化 ($\Delta = 0\mu\text{m}$ 、荷重 10g)

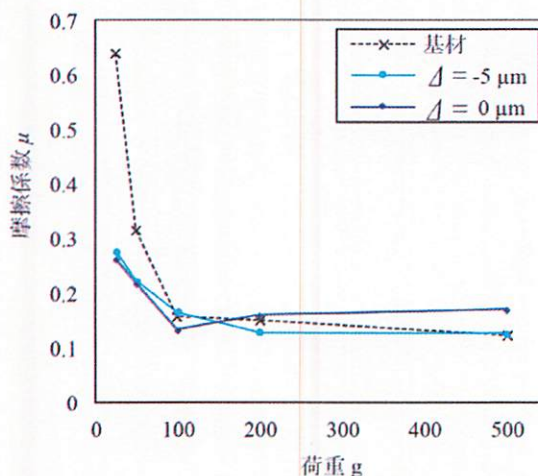


図 12 摩擦試験結果

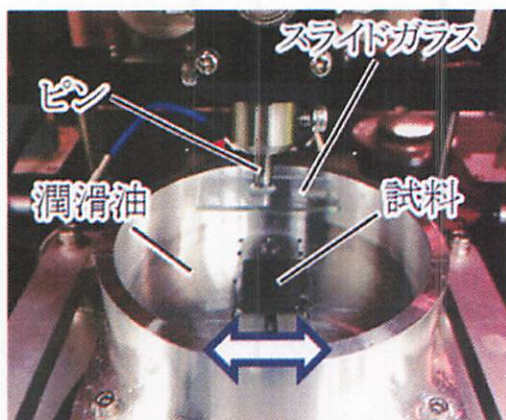


図 10 摩擦試験の様子

する報告例もあるため⁹⁾、本節ではその影響について検討を行う。ダイヤモンド砥石の番手を #200, #800, #1200, #2500 と変化させ、 $\Delta = 0\mu\text{m}$ として放電ディンプルを作成した。放電ディンプル試料表面および研削面の最大高さ粗さ R_z と算術平均粗さ R_a を図 13 に示す。同図より、砥石の番手が大きくなるほど粗さが小さくなることがわかる。

各放電ディンプルの研削面粗さの摩擦特性への影響を検討するため、前項と同様の摩擦試験を行った。試験条件は図 12 で変化の少なかった荷重 200g を除いた 4 条件とし、研削痕による影響を明瞭にするため摩擦方向を研削痕に対して垂直に設定した。試験結果を図 14 に示す。同図より、荷重 25g, 50g のとき、#800 研削面に対して摩擦係数の低減が確認され

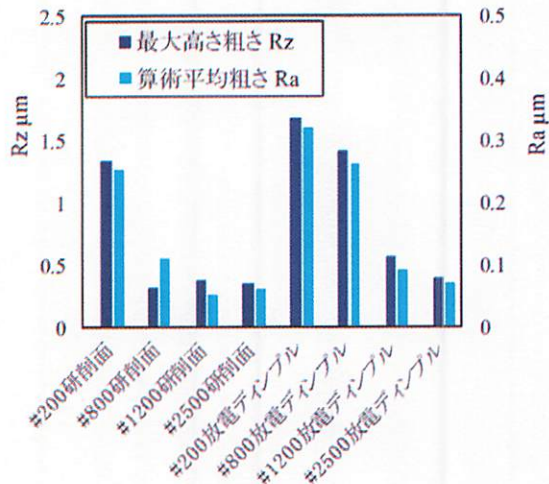


図 13 放電ディンプル試料表面および研削面の最大高さ粗さ Rz と算術平均粗さ Ra

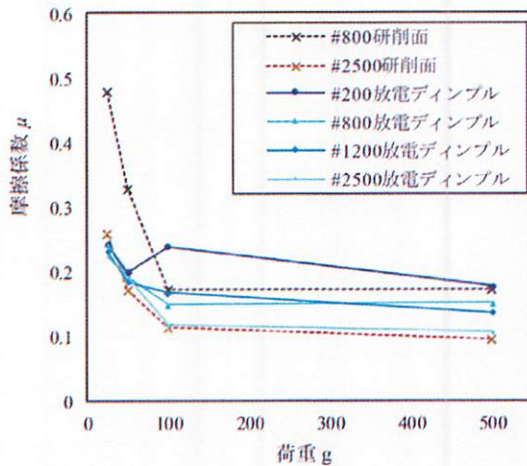


図 14 研削面粗さ変化時の摩擦試験結果

た一方、#2500 研削面に対しては摩擦係数の低減は確認されなかった。これは、本試験におけるガラス・基材間のクリアランスが同試料の最大高さ粗さ $0.35\mu\text{m}$ より大きいことにより、くさび作用による潤滑油膜形成の効果が発現されなかったためであると考えられる。また、#200 放電ディンプル試料では、荷重 100g 以上の条件において摩擦係数が増加した。これは、同試料の研削面粗さが大きいため、高硬度化された研削面の凸部がガラスと接触し、ガラス表面を引っ掻くことで摩擦抵抗が大きくなったためであると考えられる。また、表面粗さが小さいほど摩擦係数が小さくなる傾向となっていることがわかる。

5. プラズマショット研削の手法開発

先述の結果により、放電ディンプル形成に好適な放電および研削条件を明らかにした。次にプラズマショット研削を単一機械上で実現可能とする装置の開発を行い、その性能について検証を行った。

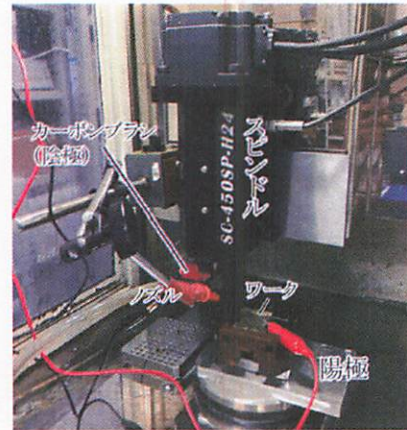


図 15 PSG 装置

表 4 PSG 改質条件

電流制限値	10.50A
電圧	150V
パルス幅	2μs
休止時間	16μs
改質時間	5min

表 5 砥石諸元

ボンド剤	ビトリファイド
結合度	N
砥粒	ダイヤモンド
粒度	#1200
直径	8.0mm
長さ	3.0mm

5.1 プラズマショット研削装置の構築

プラズマショット研削には、図 15 に示すプラズマショット研削装置を用いた。本装置では、駆動系として 4 軸案内装置（新世代加工システム社製）に超音波スピンドル（多賀電気社製、SC-450SP）を取り付けており、スピンドル先端に軸付き電極および砥石の把持が可能である。また、電源系として直流電源装置（高砂製作所社製、ZX-800H）にパルス発生装置（ミクロン精密社製）を接続し、砥石軸および基材に接触給電を行った。以上の仕様から、プラズマショット法と研削加工を本装置上で行うことが可能である。改質時はプラズマショット法と同様に、TiC 電極をマイナス極性、基材をプラス極性とした。基材はアクリル板を介してバイスで挟むことによって案内装置との絶縁を保つように把持し、加工液には精製水を用いポンプにより供給した。

5.2 プラズマショット研削実験

前項で構築した PSG 装置を用いて PSG 実験を行った。はじめに軸付き砥石を用いて基材表面を研削し、平面出しを行った。その後、砥石を軸付き電極に取り付け替え PS 法を適用した。改質条件は表 4 と同様とし、研削に用いた砥石の諸元

は表 5 に示すとおりである。その後、再度電極を砥石に取り付け替え、 $\Delta = 0\mu\text{m}$ として研削を施した。

図 16 に示す得られた改質面のトポグラフィより、図 7(c) と類似した放電ディンプルが作成できていることがわかる。またプロファイルシリーズより、ディンプル深さが約 $5\mu\text{m}$ であることがわかる。PSG 装置は、出力の限界値が小さく放電にかかるエネルギーがプラズマショット装置に比べて小さくなるため、ディンプル深さが小さくなった。これより、ディンプル深さを制御するには PS 法における適当な改質条件を選定する必要がある。

次に、PSG 面の EDX 像を図 17 に示す。同図より、放電ディンプルの周囲に Ti が存在していることがわかる。これは、本装置でプラズマショット法を行うことにより TiC 改質層が形成され、 $\Delta = 0\mu\text{m}$ としての研削によって図 7(c) と同様の改質面が得られたためである。以上の結果から、PSG によって TiC 改質層および放電ディンプルの創成に成功した。

5.3 プラズマショット研削の手法概要

前項までの実験によって得られた知見から、PSG の手法概要を以下にまとめる。なお、実験装置は案内装置と電源装置を組み合わせた前項と同様のものを考える。

- (1) 改質基材を案内装置と絶縁を保った状態で把持し、案内装置に取り付けたスピンドル軸をマイナス極性、基材をプラス極性として電源装置を配線する。
- (2) スピンドルに軸付き砥石を把持し、目標となる形状に研削加工を行う。
- (3) 軸付き砥石を軸付き TiC 電極に取り付け替え、プラズマショット法を適用する。
- (4) 軸付き電極を再度軸付き砥石に取り付け替え、(2) と同様の軌道で仕上げ加工を行う。このとき、用いる砥石の番手は大きいものが好ましい。本加工により、放電痕の盛り上がり部が除去され放電ディンプルが創成される。ただし、大面積の加工においては砥石の摩耗を考慮して加工する必要がある。

以上に示した PSG の改質工程により、摩擦面に放電ディンプルを付与し TiC 改質層による高硬度化が可能となる。よって、PSG を摩擦面に適用することで低摩耗・低摩擦面の創成が理論上可能である。

6. 結 言

本論文では、PSG の手法確立のため、プラズマショット装置と精密平面研削盤を用いて放電ディンプルを作成し、研削後の TiC の残存性および得られた放電ディンプルの形状、摩擦特性評価を行った。最後に、PSG 実験を行い、PSG の手法概要をまとめた。以下に、本章で得られた結言を示す

- (1) プラズマショット装置と精密平面研削盤を用いて、とくに $\Delta = 0\mu\text{m}$ において良好な放電ディンプルの作成に成功した。
- (2) 研削後の TiC 残存性評価から、 $\Delta \leq 0\mu\text{m}$ で十分な TiC が研削面に存在することが明らかになった。
- (3) 放電ディンプル試料の摩擦試験により、とくに荷重が小さいとき、放電ディンプルの動圧効果による摩擦低減を確認した。

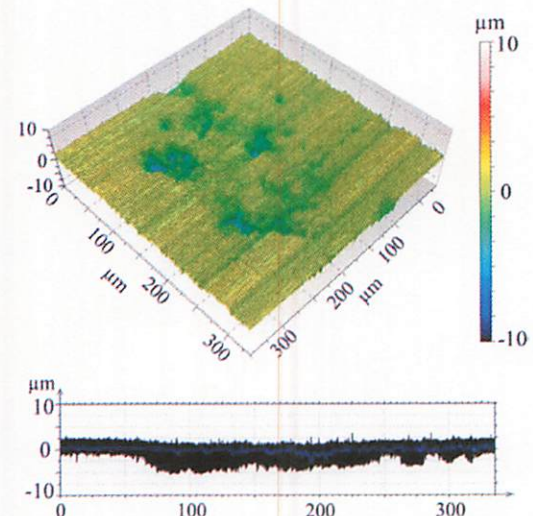


図 16 PSG 面のトポグラフィおよびプロファイルシリーズ

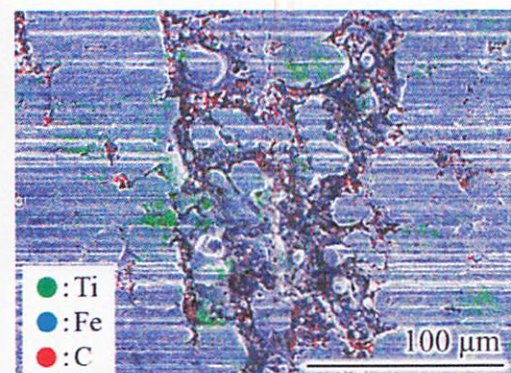


図 17 PSG 面の EDX 像

- (4) 研削面粗さを変化時の放電ディンプル試料の摩擦試験により、研削面粗さを小さくすることで荷重が大きい条件においても摩擦係数の低減が確認された。また、研削面粗さが小さいほど摩擦係数が小さくなった。
- (5) PSG 実験により、単一機械を用いてプラズマショット法と研削加工を適用することで放電ディンプルの作成に成功した。

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レーザ金属積層造形法による微細構造の創成 —純 Ti 単粒子層に対する狭小ビード造形—

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Creation of fine structure by additive manufacturing of metal
Shaping narrow beads to a pure titanium single-particle layer

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Masayoshi MIZUTANI and Tsunemoto KURIYAGAWA

本研究では、レーザ金属積層造形によって得られる純チタン製ラティス構造の微細化を目標としており、その一環として純チタン単粒子層に対する造形幅の狭小化について検討した。CW レーザによる造形では高速度もしくは低平均出力条件によって狭小化傾向を示すが、一定水準を超えると造形体の連続性を損なう欠陥（ボーリング）が発生した。このボーリングを抑制する手段としてパルスレーザを採用し、低出力条件においても造形の連続性を維持しつつ狭小化が可能であることを明らかにし、最小値として 95 μm の造形幅が得られた。さらに、造形中の溶融プールと金属粒子の挙動について高速度カメラを用いて観察し、パルスレーザによる優位性について考察を行った。

Key words: additive manufacturing, pure titanium powder, pulse laser, high speed camera, balling defect

1. 緒言

金属粉末を用いた肉盛溶接を複数層繰り返すことによって立体構造を作り出す金属積層造形技術のなかでも、粉末床溶融結合方法は下層に充填された未溶融の粉末粒子を足場にすることで、急峻なオーバーハング構造や梁構造を造形することができる。さらに柱や梁を狭い間隔で複雑に交差させることで、工具による除去加工では加工不可能なラティス構造（図 1）の造形が可能である。このラティス構造を製品部材の一部に組み込めば、必要な強度を維持したままの軽量化が期待できるため、航空宇宙分野を中心に実製品への適応が進んでいる¹⁾。また医療業界においても、生体インプラントの表面にラティス構造を付与することによって骨細胞に対する侵入性を向上し、自家骨と生体インプラントの接合が効率よく行われることが報告されている²⁾。さらに、その構造設計によっては強度に異方性をもたせる³⁾ことで、単一の部品に複雑な機能をもたせることも提案されている。

ところで、粉末床溶融結合法はその熱源によって電子ビームを用いた Electron Beam Melting 法 (EBM 法) もしくはレーザビームを用いた Selective Laser Melting 法 (SLM 法) があり、形状の精密さおよび微細性の観点では、予熱の必要がない

SLM 法が優位とされている⁴⁾。しかしながら、SLM 法においても市販装置では最小造形幅が 200 μm を超えており、微細性の自由度について限界がある。なお、ナノ粒子を用いるなどの特殊な加工工程によって金属造形の微細化を目指した研究はいくつか報告されている⁵⁾が、すでに製品化され実績のある SLM 法とは手法が大きく異なり、実用化には多くの時間が必要と思われる。これらのことから筆者らは SLM 法をベースとした実現性の高い手法によって造形幅の狭小化について検討し、これまで造形が困難であった微細なラティス構造を作り出すことで、その構造設計の自由度を高めることを目標としている。なお、SLM 法では CW レーザを通常使用することが多いが、K.A. Mumtaz ら⁶⁾はパルスレーザによる造形の狭小化の可能性を示している。しかしながら、CW レーザと比較して、パルスレーザが狭小化に有効であることを示した文献は見当たらない。

本報では、基材上に配置された純チタン粉末による単粒子層にレーザをスキャンすることにより、造形体の一層目にあた

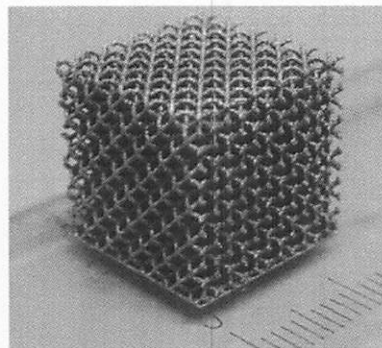


図 1 SLM 法で得られた 12mm 角の立方ラティス構造

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表 1 供試材の化学成分

	化学成分(ppm max.)				
	Fe	O	C	H	Ti
Ti 粉末	50	1300	200	1300	残部
Ti 基材	2000	1500	800	1500	残部

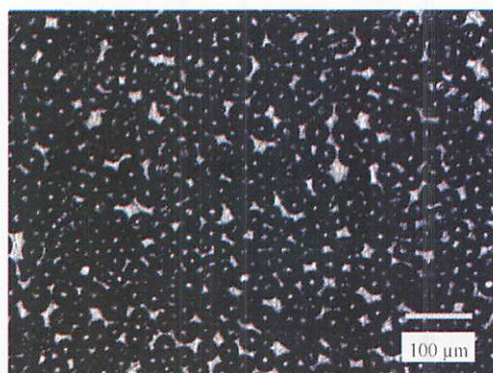


図 2 純チタン単粒子層の充填状態

表 2 発振器および集光特性概要

波長	1,064 nm
最大発振出力	500 W CW ファイバーレーザー
ビームの強度分布	ガウシアン分布
スポット径	φ16.5 μm
焦点はずし量	0 mm (基材上端)
レンズ焦点距離	100 mm

る肉盛りビードを形成し、CW レーザとパルスレーザによる連続性を前提とした肉盛りビードの狭小化について調査した。また造形中の溶融プールと金属粒子の挙動について高速度カメラを用いて観察し、造形体の連続性に必要な要因について考察を行った。

2. 実験方法

2.1 供試材

本研究では、生体・医療用材料として広く用いられている純チタン粉末および基材として純チタン板材を使用した。粉末はガスアトマイズ法によって製造された純チタン粉末を使用した。SLM 法においては粉末輸送の観点から真球に近く、サテライトが少ない粉末が推奨される⁷⁾ため、本実験においてもそのような Ti 粒子で粒径が φ25~38 μm のものを使用した。基材には板厚 4 mm の純チタン 2 種熱間圧延材を使用し、レーザ照射面はバフ研磨を行った。粉末および基材の化学成分を表 1 に示す。

基材上に均一な粒子配列を作るために以下の手順を行った。基材上に約 10 mm 角のケガキ線を入れ、その領域を満たすように水滴を載せ、そこに粉末を散布する。さらに、水滴表面上の粒子が均一に広がるように基材を振動させたのち、十

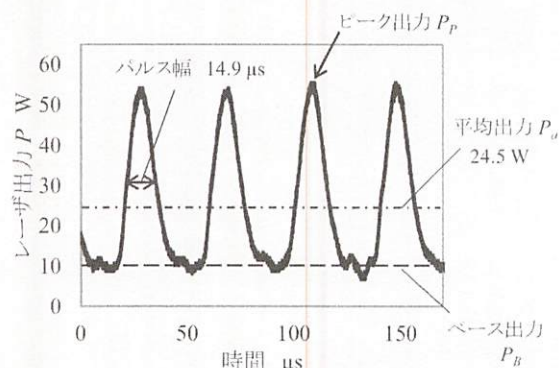


図 3 パルス波形 例

分に乾燥させることで、純チタンの単粒子層を作り出した⁸⁾。この単粒子層の充填状態を図 2 に示す。粒子が重なる部分はあるが、そこを避けるように加工することとした。

2.2 レーザ発振器とパルス波形

使用した発振器の概要を表 2 に示す。本発振器は一般的な金属積層造形装置に採用されている CW ファイバーレーザーであるが、高速変調動作が可能な特徴を有しており、マイクロ秒のパルス波を発振できる。レーザ出力波形はファンクションジェネレータで生成した電圧波形を発振器に入力することで得られた。本論文では CW 発振とパルス発振にて実験を行っているが、パルス発振の場合は発振停止状態からレーザ媒質を急激に励起すると、緩和発振現象⁹⁾によって発振立ち上がりに数 μs 幅以下のスパイク状の高出力部ができる。このスパイク部の出力は指令出力の数倍に達するため、このパルス波を粒子に照射すると、粒子は溶融せずに弾き飛ばされるか、破裂する¹⁰⁾。そこで、本発振器で発振可能な最低出力をベース出力 P_B (約 10 W) とし、これと任意のピーク出力 P_p を交互に繰り返すレーザパルス波を採用することで、緩和発振によるスパイクを防ぐこととした。このパルス波を InGaAs 増幅フォトディテクタで測定し、帯域幅 1 GHz のオシロスコープによってその波形を観察した。その一例を図 3 に示す。図の縦軸はフォトディテクタの電圧値から換算したレーザ出力である。この換算値は十分に長い発振時間におけるフォトディテクタの平均電圧値と、レーザパワーメータによる平均出力により算出した。なお、図 3 のパルス波形において平均出力 $P_a \approx 24.5$ W であり、パルス幅(ピーク出力 $P_p \approx 54.9$ W に対する半値幅)は約 14.9 μs であった。

2.3 実験装置および実験手順

レーザ光は F0 レンズとガルバノスキャナの組み合わせによって駆動させており、レーザスポット位置は常に加工ステージ上にある基材上端となるように設定した。造形時の酸化や窒化を避ける必要があるため、加工ステージごと基材を覆うチャンバを用意し、図 4 に示す構成によってアルゴン環境下の造形を実現した。このチャンバは真上から照射されるレーザ光を透過するコーティングを施した窓と、高速度カメラの撮影用窓、照明窓を備えている。チャンバの配管口に簡便な真空ポンプを接続し、チャンバ内部の空気を強制排出してから、もう一方の配管口からアルゴンガスを流し入れ、十分な時間を経過させることで高濃度のアルゴン環境を作った。さらに造形時に

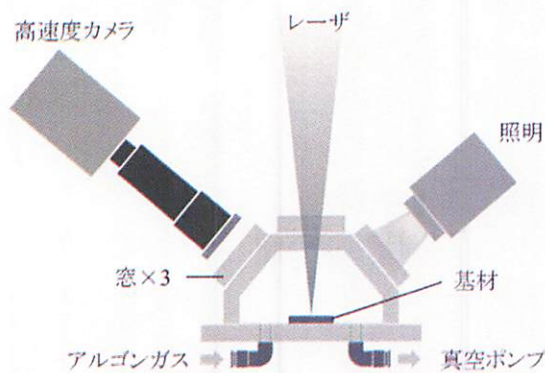
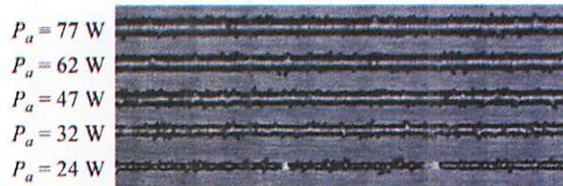


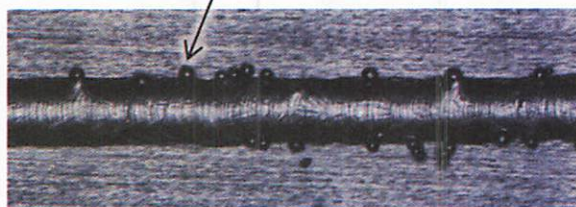
図4 加工チャンバの構成



(a) 全体像

500μm

未溶融粒子

(b) $P_a = 62$ W の肉盛ビード拡大図

ボーリング

(c) $P_a = 24$ W の肉盛ビード拡大図

100μm

図5 CW発振における肉盛ビード(造形速度 100 mm/s)

発生するヒュームを取り除くためアルゴンガスを約 20 NL/min の流量で流し続けヒュームが滞留しないようにした。なお、このときのチャンバ内ゲージ圧は約 0.01 MPa の弱正圧である。

高速度カメラと照明は向かい合せに配置し、ともに基材に対して仰角 40°の角度で設置している。このため高速度カメラ像には Ti 粒子の影が手前に映り込んでいる。高速度カメラには光学倍率 5.0 倍の拡大レンズを取り付け、レンズの先端には使用したレーザー光の波長を吸収する赤外線フィルタを取り付けた。高速度カメラでの観察は撮影速度 210000 fps (1 フレーム約 4.76 μs) にて撮影を行った。

3. 加工結果および考察

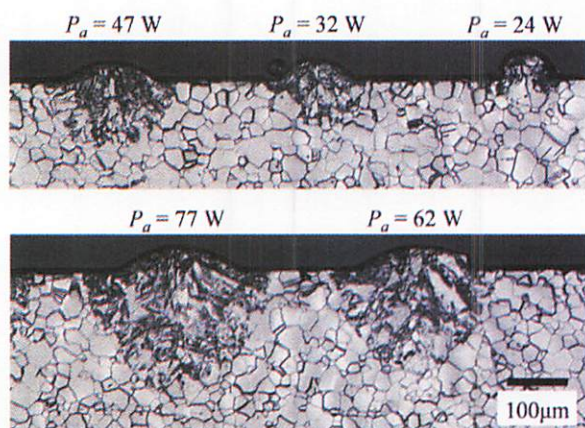
3.1 肉盛ビードの形状による評価

基材表面に形成した単粒子層に対して、CW 発振のレーザービームを直線状にスキャンすることによって純チタン粒子による肉盛ビードを造形した。その一例を図 5 に示す。造形速度およびそのレーザー出力によって肉盛ビードの様相は異なり、造形速度 100 mm/s, $P_a = 62$ W の条件(図 5 (b))では、一部で極微細な未溶融粒子が認められるものの、一様な肉盛が形成できているのに対して、造形速度 100 mm/s, $P_a = 24$ W の条件(図 5 (c))では、レーザー照射ラインに沿って大きな球が形成されていることが確認できる。この現象はボーリング¹¹⁾と呼ばれており、造形速度が速い場合や、造形速度に対してレーザーのエネルギー密度が小さい場合などでみられ、SLM 手法におけるリコータ動作の障害や最終製品の欠陥を引き起こすため避けるべき現象である¹²⁾。

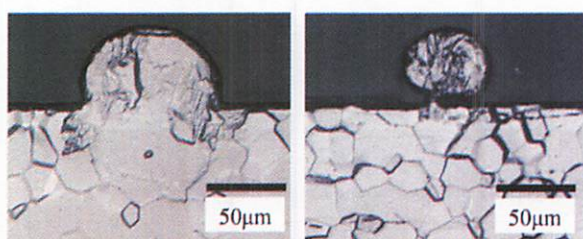
図 5 の各肉盛ビードについて、その断面を精密切断機でカットし、切断の影響を取り除くよう研磨を施し、コロイダルシリカによる鏡面仕上げを行った。その後、ケミカルエッチングによって結晶構造を確認したものを図 6 に示す。ボーリングが起きやすい $P_a = 24$ W の低出力条件では、溶融粒子の塊が基材上に付着しているのみに見え、十分な接合ができていない可能性が高い。さらに低エネルギー密度となる $P_a = 24$ W, 造形速度 200 mm/s の断面では溶融粒子の塊と基材の間に明らかな境界線が残っている。溶融粒子の塊と基材の間に境界線が存在しており接合としては不適切である。溶融粒子を基材に接合させるには基材の酸化膜や吸着層をレーザーエネルギーによって破壊し、互いが液相となり混合する必要があるが、これらの条件では純チタンよりも融点の高い酸化膜を破壊できていないため、界面となって残留してしまうと考えられる。それに対して $P_a = 32$ W 以上の条件では、このような界面は残留しておらず、良好な接合ができています。基材内部の結晶構造より推測される溶融領域をみると、高出力条件ほど溶融領域は深く広くなっており、溶融粒子による半円状の肉盛の幅も連動して広がっている。ところで、基材上の酸化膜は新生面に比べ固体表面張力が小さく、液体金属に対して濡れ広がりにくい性質がある¹³⁾。このため、酸化膜の破壊領域が狭い条件、つまり溶融領域の狭い条件であれば溶融粒子の濡れ広がりを抑えることができ肉盛ビードの狭小化に期待がもてる。

造形条件による肉盛ビード幅の影響を確認するために、造形速度と出力による肉盛ビード幅の十点平均値を図 7 に示す。図中の白抜きプロットに関しては、後述するビードの連続性に関する定義によって、不連続と判断した造形条件を示している。なお、肉盛ビードには図 5 で示した未溶融粒子が側面に付着することがあるため、本図はこれを避けた部分の幅をレーザー顕微鏡で測定した結果である。図によると、すべての条件で肉盛ビード幅はレーザーのスポット径(16.5 μm)よりも十分に大きくなっており、溶融領域が狭くなる低出力、高速度条件において、ビード幅が狭くなる傾向があることが確認できる。

また、100 mm/s の加工条件における肉盛ビードの高さの平均値と最大最小値を図 8 に示す(未溶融粒子部分を避けて測定)。なお、粉末を配置していない基材に直接レーザー加工



(a) 造形速度 100 mm/s における肉盛ビード断面



(b) 造形速度 100 mm/s

 $P_a = 24$ W の拡大図

(c) 造形速度 200 mm/s

 $P_a = 24$ W の断面

図 6 CW 発振における肉盛ビード断面

(ビードオン)したときに形成されるビードオン高さも本図に提示する。図 8 から、 $P_a \leq 25$ W 以下の条件で得られた肉盛ビードはビードオンによる高さ程度もしくはそれ以下の高さしかない領域が発生しており、不連続な造形となっている。なお、本報においては、同じ平均出力で加工した結果、得られたビードオン高さに加えて 5 μ m 以上の肉盛高さがあつたものを連続造形とし、それ以外を不連続造形と定義している。これらのことから、連続性をもった肉盛を前提とすれば、CW 発振のビード幅の狭小化は、110 μ m 付近に限界値があることがわかる。

3.2 パルスレーザによる肉盛ビードの狭小化

電子機器などの伝熱によるダメージが懸念される製品の精密溶接については、熱影響を最小限に抑えつつ深い溶融領域を得るためにパルスレーザが用いられることがある。また、筆者らの過去の調査¹⁰⁾においても、数十 μ s のパルス幅のワンショット発振によって単体粒子と基材がともに溶融し接合することが可能であり、そのときの基材の溶融幅が 100 μ m 以下であることを確認している。これらのことから、2.2 節で示した数十 μ s のパルス幅条件によって造形幅の狭小化を目指した。パルス波形の選定にあたって、パルスの時間間隔が長すぎると、肉盛ビード高さおよび幅の凹凸が大きくなる傾向があることから、平滑性を確保するためにパルスの重なり率を 95 % 以上になるようにした。具体的には 100 mm/s の造形速度では 25 kHz、200 mm/s では 50 kHz を採用している。さらに図 3 で示したピーク出力を 47 ~ 77 W、パルス幅を 10 ~ 30 μ s の範囲で任意に設定することで、複数のレーザパルス波形を作り出し、これらによる造形加工を行った。

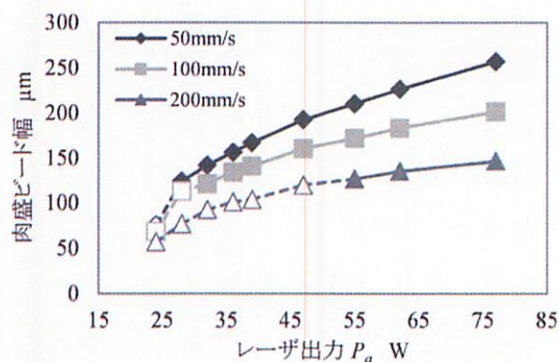


図 7 各種 CW 発振条件における肉盛ビード幅の十点平均値

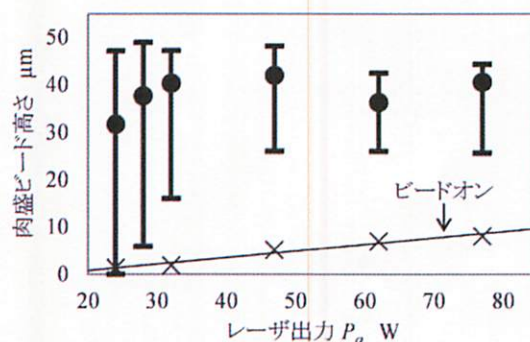


図 8 CW 発振における肉盛ビード高さ (造形速度 100 mm/s)

このパルスによって得られた肉盛ビード幅の十点平均値とパルスの平均出力の関係を図 9 に示す。図中の白抜きプロットは、肉盛ビードの不連続性を示しており、同図には、図 7 で得られた CW 条件の傾向線も記載している。本図より、100 mm/s の造形速度においては、同じ平均出力の CW 条件よりもパルス条件のほうが肉盛ビード幅は狭くなる傾向があることがわかる。連続性を考えた場合でも、最小幅で約 95 μ m を達成できており、パルスによる狭小化の効果が確認できている。それに対して 200 mm/s の条件では幅の減少はみられず、連続性についてもほとんど改善していない。また、今回選択したピーク出力やパルス幅によってさまざまなパルス波形を作り出し、これらの肉盛ビード幅への影響を調査したが、明確な関連性については確認できなかった。その一方で、平均出力と肉盛ビード幅については強い相関性がみられた。

また、造形速度 100 mm/s、 $P_a = 29$ W のパルス発振 ($P_p = 77$ W) で得られた連続性のある肉盛ビードと、同条件の CW 発振で得られた肉盛ビードをレーザ顕微鏡で測定し、その高低差の色別図および高さプロファイルを図 10 に示す。同図より、パルス発振の方が CW 発振よりも肉盛ビード幅が狭く、ビード高さも一定値以上を維持しているのがわかる。ただし、図 5 で示したような未溶融粒子の付着の影響もあり、高さの平均値に対して最大高さ R_z は大きく、このままでは製品表面への適応は難しい。このため、既に形成された肉盛ビードのラインに対してレーザをさらに複数回スキャンし、ビードの再溶融による平滑化などの工夫が必要である。

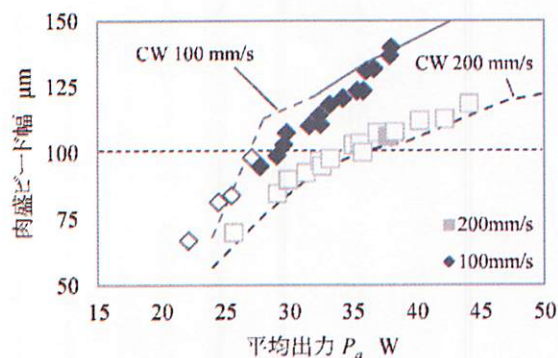


図9 各種パルスレーザー条件における肉盛ビード幅の十点平均値 (粒子径 25 ~ 38 μm)

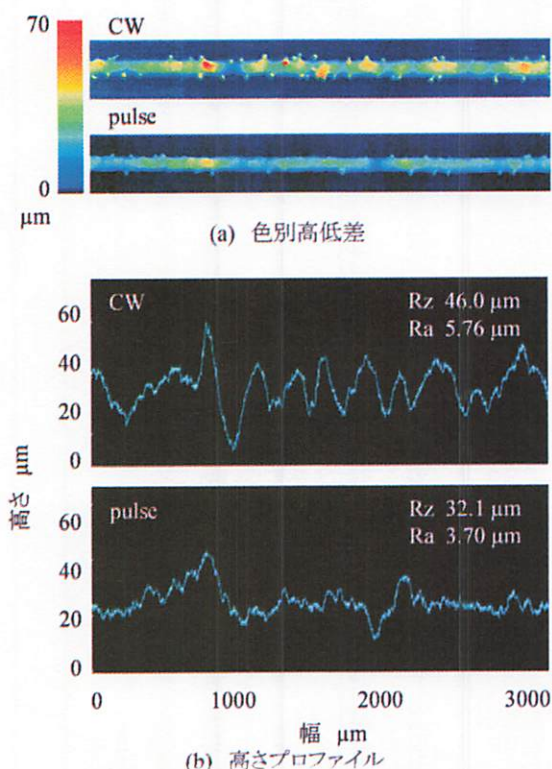


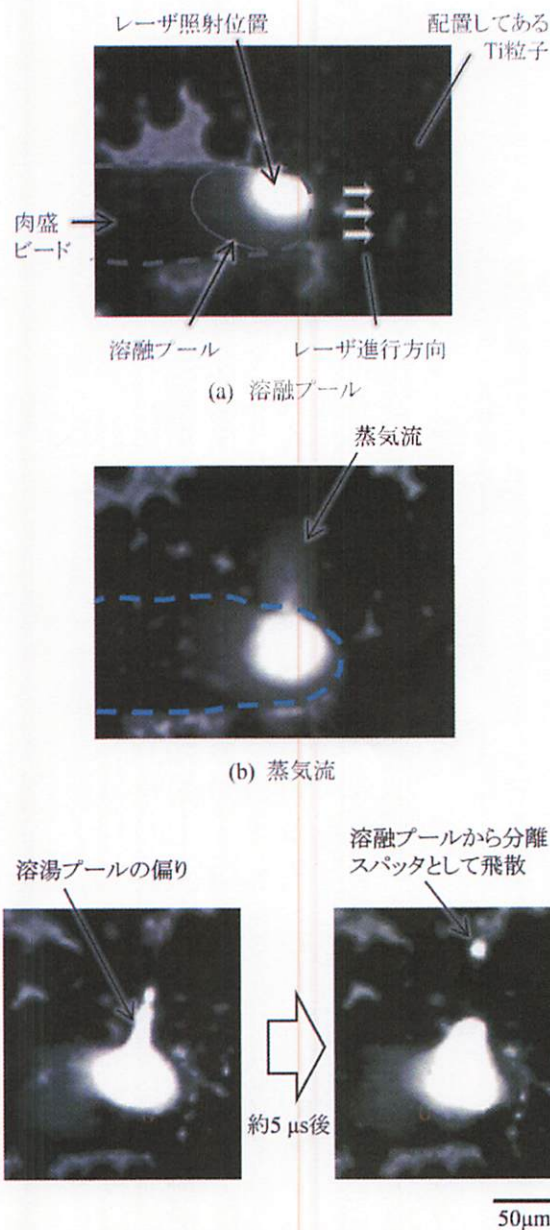
図10 造形速度 100 mm/s $P_a = 29$ W 条件の CW 発振およびパルス発振における肉盛ビード高さ

3.3 造形中の溶融プールおよび周辺粒子の挙動

3.3.1 溶融プールの挙動

前節までの結果より、造形速度とレーザー平均出力が、肉盛ビード幅の狭小化のための有効なパラメータであることが理解できる。しかしながら、肉盛ビード幅を小さくすれば、ビードの連続性が維持できなくなる。この原因を調査するため、肉盛造形中の加工点の挙動を高速カメラで観察した。

溶融プールの挙動を示した画像の一例を図11に示す。図11(a)より、レーザー進行方向および肉盛ビードの周辺は、チタン粒子が密に配置されているため、黒く写っている。これらの粒子が取り除かれると、灰色の基材表面が露出する状態となる。造形中の肉盛ビード先端が最も高い輝度をもっていることから、ここにレーザーが照射されていると考えられる。肉盛ビード



(c) 溶融プールの偏りによるスパッタの飛散

図11 溶融プールの挙動

幅が広がる高出力や低速条件では、この高輝度領域が広くなり、その周囲にある流動性をもった溶融プールの範囲も広くなる。同時に溶融プールからは強い輝度をもつ指向性の強い気体が高頻度で放出するようになる(図11(b))。この気体はチタンもしくは不純物が気化した蒸気流(ブルーム)だと考えられ、溶融プールの流動によりその放出方向は常に変化すると同時に、その反発力に応じて溶融プールをさらに揺れ動してしまう。高出力ほどその動作は激しくなり、これによって溶融プールを押し広げてしまっているようにも見える。また、溶融プールの偏りが大きくなりすぎると、その一部が分離し、スパッタとして飛散することも確認している。レーザー照射位置から離れた溶融プールは自然冷却による凝固を始め、溶融プールの幅がほぼそのまま肉盛ビードの幅となる。

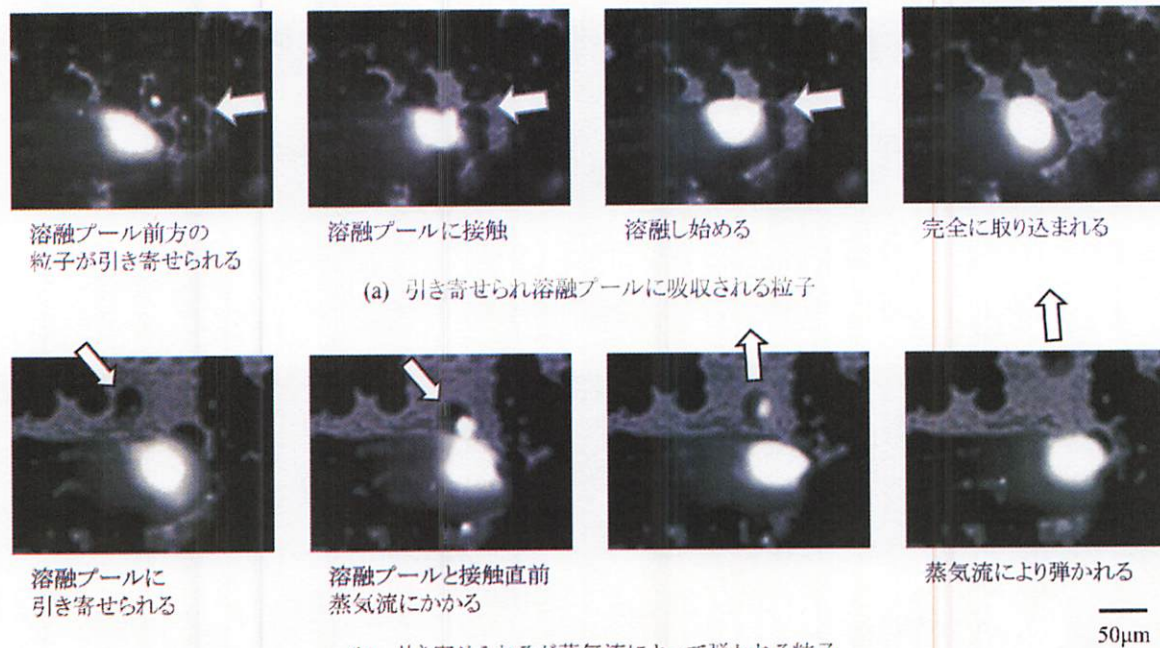


図 12 レーザ進行方向に配置された粒子の挙動

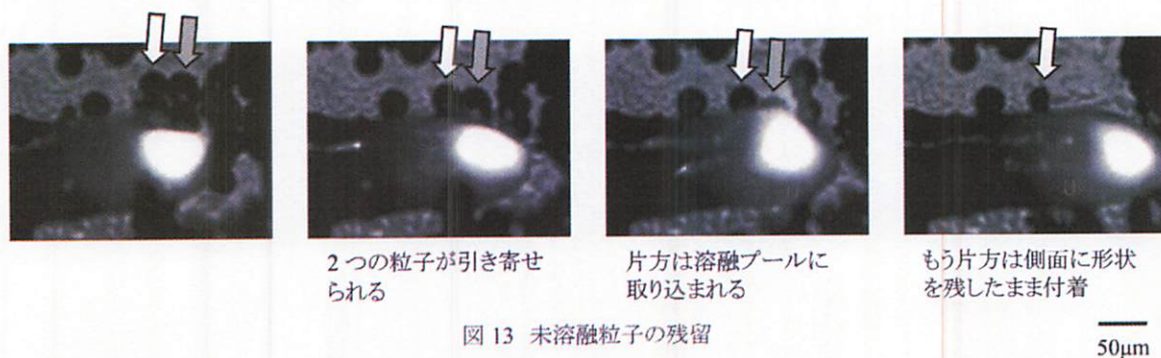


図 13 未溶融粒子の残留

3. 3. 2 レーザ進行方向に配置された粒子の挙動

次に溶融プール周辺の粒子に着目する(図 12). レーザビームの移動にともない溶融プールが、進行方向に配列された粒子に接近すると、粒子はさまざまな挙動を示す。造形速度が 100 mm/s のときには、粒子と溶融プールが十分に離れている状態でも、粒子は運動を始める。その挙動については下記の 3 つのパターンが挙げられる。

- (A) 溶融プールに引き寄せられ、接触し、伝熱によって溶融し、溶融プールに吸収される(図 12 (a)).
- (B) 溶融プールに引き寄せられるが、溶融する直前に反発力が働き、弾き飛ばされる(図 12 (b)).
- (C) 溶融プールに引き寄せられることなく、溶融プールが接近することで弾き飛ばされる。

これらの粒子の挙動において(A)が肉盛りビードを成長させる要因であり、肉盛りビードの連続性を満たすためには、これらの挙動によって定期的に溶融プールへ粒子が供給される必要がある。

一方、造形速度 200 mm/s の場合は、粒子は溶融プールに引き寄せられることなく、配置された位置のまま溶融プールと接触し、溶融プールに吸収される挙動が(A)の挙動よりも多く

確認されている。なお、(A)、(B)における引き寄せられる粒子の速度を、画像解析によって計測したところ、約 200~400 mm/s であり、これが溶融プールによる引き寄せ効果が加工速度によって異なる原因の 1 つであると推察されるが、これに関しては更なる調査が必要である。

溶融プールから離れた粒子に運動を与える現象については、Manyalibo ら¹⁴⁾によって論じられている。これによると溶融プールからの金属蒸気流は、内向きのガス流を誘発し周辺粒子を引き寄せる効果と、蒸気自体の反発力によって周囲の粒子を弾く効果を持つことを指摘している。

3. 3. 3 溶融プール側方に配置された粒子の挙動

前項で示した粒子の引き寄せ効果は、溶融プールの側面や後方にある粒子にも適応され、これは造形速度によらず、運動開始時点の溶融プールに向かって引き寄せられる。造形速度が遅ければ、その引き寄せ速度により溶融プールに追いつくことができ、溶融プールの中心付近の高温領域に付着すれば、そのまま溶融、吸収されるが、進行方向にある粒子と比べればその吸収頻度は少ない。また、溶融プールから離れたビード上に粒子が付着すると、粒子の形状を残したまま、未溶融粒子として残留してしまう(図 13)。とくに造形速度

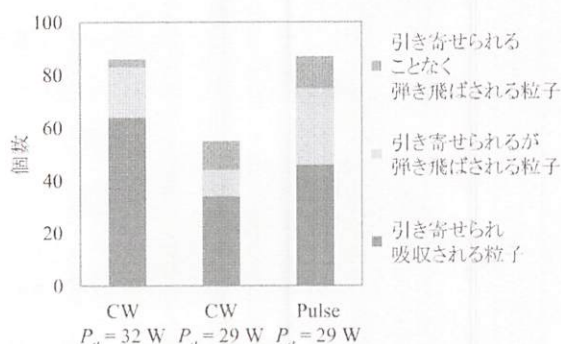


図 14 造形速度 100 mm/s における周辺粒子の挙動

が速い場合は、粒子が溶融プールの高温領域に追いつくことはできないため、その傾向はより顕著である。

3. 4 肉盛ビードの連続性についての考察

3. 4. 1 CW 発振における肉盛ビードの形成プロセス

連続性をもった狭小幅の肉盛ビードを得るには、造形速度 100 mm/s のパルス発振条件が有効なことを 3.2 節で示した。この要因を調査するために、造形中の高速度カメラ像から 4.8 ms 分の映像を抜き出し、前節で説明した造形中の周辺粒子の各挙動についてカウントしたものを図 14 に示す。このときの造形条件は、図 10 で示した $P_a = 29$ W のパルスと CW 条件であり、CW 発振において良好な連続性が得られている最狭小幅条件 ($P_a = 32$ W) についても表記している。 $P_a = 32$ W および $P_a = 29$ W の CW 発振条件と比較すると、溶融プールの進行にともなう、運動を始める粒子の総数は、 $P_a = 32$ W の方が多く吸収される粒子も多い。これらの条件の溶融プールにおける特徴的な挙動を図 15 (a), (b) に示す。図より、溶融プールの先端の勾配を比較すると、 $P_a = 32$ W については基本的に基材に対して濡れ広がっており、粒子の合流時は溶融プールが揺れ動き、一時的に勾配が大きくなるものの比較的緩やかである。

それに対して、CW 発振の $P_a = 29$ W の溶融プール先端は、濡れ性が悪く、多量の粒子が溶融プールに合流した場合、接触角度が 90° に近くなり、急勾配を形成する。これは多量の粒子が合流することで、溶融プールの温度が低下し、温度依存性のある表面張力が大きくなったためと推察される。蒸気流はこの溶融プール先端の勾配に対して法線方向に放出する傾向があるため、勾配の比較的小さい $P_a = 32$ W では垂直か

ら約 45° の傾きの範囲で蒸気流が放出されるが、勾配が大きい $P_a = 29$ W では進行方向前方に大きく蒸気流が倒れ、前方の粒子を弾き飛ばしてしまうことがある。これによって基材が露出し、その直後に合流する粒子が激減する。さらに、この蒸気流にさらされた粒子の一部は高温となって周辺の粒子と合体し大きな球 (ポーリング) になることもあり、これ自体が加工ラインに残ると肉盛ビードの均一性を損なう。配列された粒子と基材に対するレーザ吸収率では、同じ温度でも配列された粒子に対する吸収率の方が高い¹⁵⁾ことと、熱容量の小さい粒子の方が容易に高温になり、吸収率が高くなる。このため粒子によって形成された溶融プールに比べ、粒子が無い基材上に形成される溶融プールは極端に小さくなり、蒸気流も弱まっているようにみえる。これによって周辺粒子はほとんど運動しなくなる。その後、配列された粒子にレーザが直接照射されると、粒子は溶融し、再度肉盛ビードが構築されはじめるが、上記の現象を繰り返すため、CW 発振の $P_a = 29$ W では連続性のある肉盛ビードは得られない。

3. 4. 2 パルス発振における肉盛ビードの形成プロセス

これに対してパルス発振 $P_a = 29$ W の場合は、同じ平均出力の CW 条件と比較して、合流する粒子が増えている (図 14)。ただし運動を始める粒子の総数自体も増えており、そのほとんどが、合流せずに弾かれる粒子である。パルス発振における溶融プールの挙動についても確認する (図 16)。溶融プールの輝度の高い部分、つまり高温領域がパルスの周波数に合わせて拡大縮小を繰り返し、この領域が拡大した瞬間に蒸気流を放出し (図 16 (a))、その後の縮小にともない、蒸気流は霧散する (図 16 (c))。この動作によって溶融プールは、周波数に応じ小刻みに揺れ動いている。CW 条件の溶融プールの動きについては、比較的大きく緩慢で不規則であったが、パルス条件では加工方向に対して前後に規則的に揺れ動くことで、肉盛ビードの均一性を高めている。

また、溶融プール先端は溶融プールの揺れ動きによって瞬間的に勾配が大きくなり、蒸気流が大きく傾斜することがある (図 16 (b))。ただし、短いパルス幅によって蒸気流はすぐに消え去り (図 16 (c))、前方の粒子を弾き飛ばすことは少ない。

さらに、レーザ出力と蒸気圧の関係¹⁶⁾により、高ピーク出力によって強い蒸気流がパルスに応じて断続的に発生するため、引き寄せられる粒子は同出力の CW 発振と比べ多くなっていると考えられる。ただし、引き寄せられた粒子が転がる

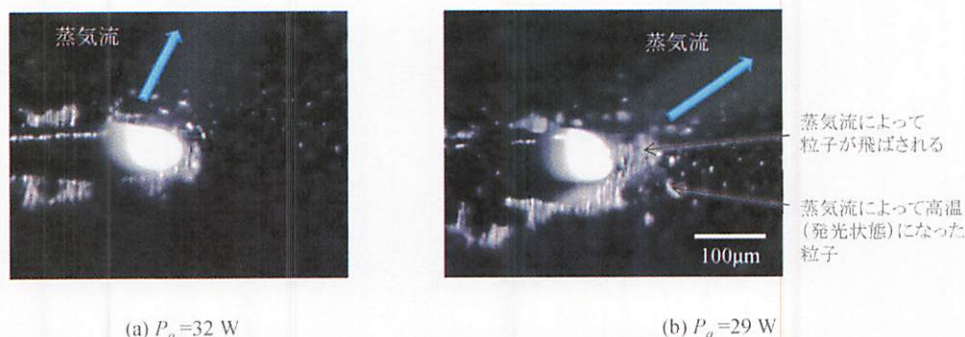


図 15 CW 発振における溶融プール先端の状態

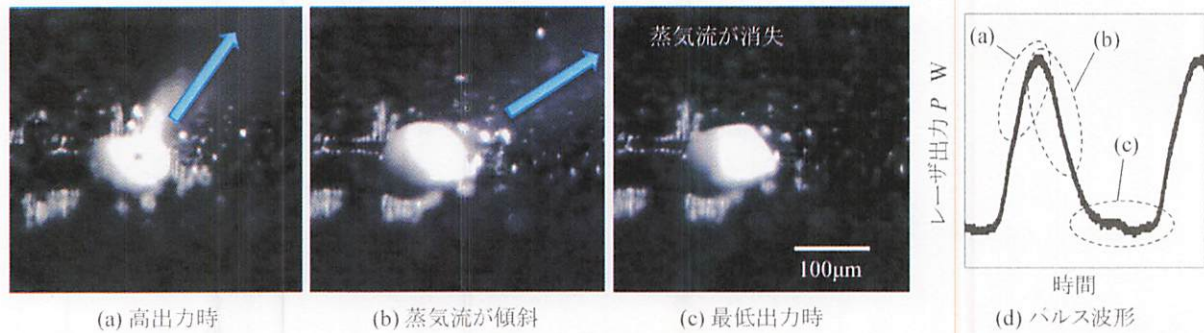


図 16 パルス波形の各時刻における溶融プールおよび蒸気流の挙動

様な軌道で溶融プールに接触することができれば効率よく吸収できるが、パルス発振の場合は、浮き上がった軌道で溶融プールに近づくことが多いため、そのほとんどが蒸気流によって弾かれてしまい、溶融プールに吸い込まれる粒子はそれほど多くはない。それであっても、粒子が定期的に溶融プールに引き寄せられ合流するパルス発振は、連続性のある均一な肉盛りビードを形成するのに有効である。

以上のことからパルス発振によって肉盛りビードの連続性を高めた要因として、溶融プールの規則的な振動、蒸気流の傾斜による前方粒子の弾き飛ばしが少ないこと、強い蒸気流による定期的な周囲の粒子の合流などが考えられる。

4. 結 言

本報では、純チタン粉末を利用した SLM 法における造形体の一層目にあたる基材上の肉盛りビードの狭小化について、CW レーザとパルスレーザを用いて検討を行った。その結果、どちらの発振形態においてもレーザ平均出力が肉盛りビード幅に強い相関性を示していることが確認できた。しかしながら、低平均出力領域の CW レーザではボーリングと呼ばれる欠陥によって肉盛りビードの連続性が損なわれるため、幅として 110 μm 付近に限界があることを確認した。これに対して、パルスレーザでは CW レーザで欠陥が発生した低平均出力領域でも連続性を維持することが可能であり、100 mm/s のスキャン速度において最小値として 95 μm の肉盛りビード幅が得られた。

また、高速度カメラによって得られた溶融プールと粒子の挙動を確認すると、CW レーザでは溶融プールの先端がまれに急勾配な形状となることがあり、これによって溶融プールから立ち昇っている蒸気流が進行方向前方に大きく傾斜し、合流すべき粒子を弾き飛ばしてしまう。これにより造形に必要な粒子が枯渇し、造形の連続性が損なわれてしまう。

これに対して、パルスレーザの場合も CW レーザと同様に溶融プールの先端が急勾配になる場合があるが、短いパルス幅に応じて蒸気流は発生と消失を繰り返すため、前方の粒子を吹き飛ばすことは少ない。またパルスの周波数に応じて

溶融プールが小刻みに揺れ動くことで肉盛りビードの均一性と高めていることと、高ピーク出力時に多くの粒子を周囲から引き寄せていることも重要である。

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ナノ精度機械加工専門委員会

ナノ精度機械加工が拓く新たな世界 —機能性インターフェースの創成—*

High Value Manufacturing As The Next Frontier of Nano-Precision Mechanical Manufacturing Technology

水谷正義**

Masayoshi MIZUTANI

Key words

nano-precision mechanical manufacturing, M⁴ (Micro/Meso Mechanical Manufacturing), non-conventional machining, high value manufacturing, functionalization, surface structuring

1. はじめに (委員会設立趣旨)

日本の大学の研究室では、機械加工技術の研究者が年々少なくなっているのが現状である。このような状況の中で、本委員会(委員長: 厨川常元(東北大学))では企業と強力に連携し、社会実装を想定したニーズを取り入れながら、学学連携を基盤に産業連携を強力に推し進め、これからの精密工学・加工学がどうあるべきか、あるいは企業を支える機械加工技術がどうあるべきかを共に考える場を提供するものである。

これまでの日本のものづくりは、技術と技能の融合の下で高い水準を維持してきた。しかし、人件費の安いアジア諸国の台頭、各企業の海外展開による現地調達進展等により、日本の製造業における国際競争力が低下してきている。例えば、樹脂製非球面レンズのように、製造プロセスが確立した製品においては、超精密加工製品といえども国内では価格競争の波にのまれ、海外に生産拠点が移動し始めているのが現状である。このような状況の中で、日本のものづくりがこれからも世界をリードしていくためには、海外の技術では到達できないような、より高い精度と付加価値をもった製品の生産に転換していかざるを得ない。すなわち、今後はナノオーダーの超平滑な表面ならびにナノ精度の3次元形状を有する構造体を創成する超精密加工技術と、原子や分子の加除を精緻な制御によって達成し、微細な構造体を創成する超微細加工技術の二つを駆使した生産加工技術の開発研究の促進が必要不可欠である。

本委員会ではこれまで、前者の超精密加工技術を発展させたナノ精度機械加工技術や M⁴ (Micro / Meso Mechanical Manufacturing) プロセス技術の開発研究を、産業界のニーズを取り入れながら行ってきた。その一方で、最近では後者に示したような微細な構造体を創成する超微細加工技術の開発研究にも取り組んでいる。これは、

従来の形状創成を主体としたものづくりに加え、加工した表面に任意の「機能性」をもたせるという、新たな取り組みでもある。

2. 機能性インターフェースの創成

本委員会では、前述のとおり、ナノ精度機械加工技術や M⁴ プロセスの開発を基軸とした超微細加工技術を駆使し、さまざまな「機能性」をもったインターフェース(界面)の創成に取り組んでいる。表面への機能性付与の鍵となるのは、材料表面の微細な構造である。表面に対して微細な(ナノ~マイクロメートルオーダー)の三次元構造を創成することにより、その材料が有する機能を飛躍的に向上させる、あるいは今までにもち得なかった機能を付与することが可能になる。本報では、これらの取り組みの中から2例紹介する。

2.1 3D 超音波振動援用マイクロ加工による微細周期構造の創成^{1)~7)}

切削や研削、研磨などのいわゆる「機械加工」は、ものづくりには欠かせない基盤技術として現在までの産業を支えている。最近では、0.1 nm の位置決め精度、つまりピコ精度の分解能を有する工作機械が誕生するなど、機械加工による超精密加工は発展し続けており、微細な形状のアレイや溝など、例えば光学部品製造用の金型のような、極めて高い精度が要求されるような製品に対してその効果を発揮している。一方で、そのような加工を行う場合には、高額な装置やシビアな加工環境の制御が必要であるとともに、素材や加工領域が限定されることも多いという課題も存在している。

そこで、切削や研削という従来の機械加工のプロセスに、超音波振動を加えたプロセスを提案している。この方法では、工具や工作物の並進、回転と、超音波振動の振幅や周波数などの相対運動の要素を任意に設定することにより、その運動に応じた微細な構造を被加工面に対して比較的容易に創成することが可能になる。これまでに、図1に模式的に示すような3軸方向に振動可能な3D 超音波スピンドルを開発するとともに、工作機械の送り運動および

*原稿受付 平成30年7月17日

**正会員 東北大学大学院工学研究科(仙台市青葉区荒巻字青葉6-6-01)

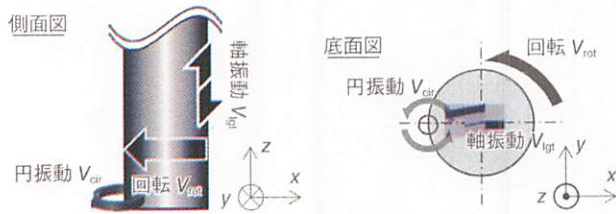


図1 回転・円振動・軸振動の模式図⁷⁾

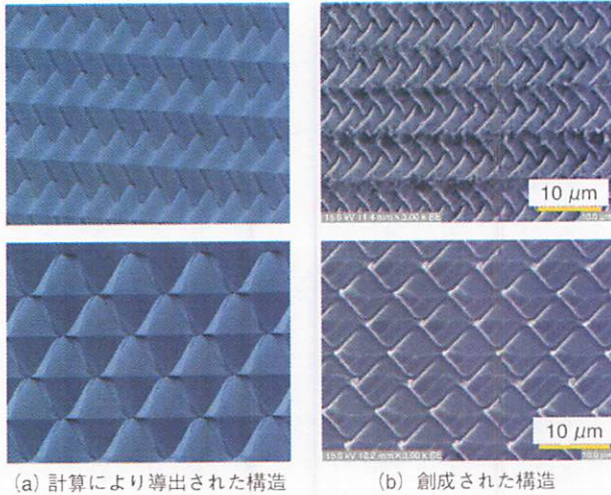


図2 3D 超音波振動援用マイクロ切削加工により創成された微細構造¹⁾

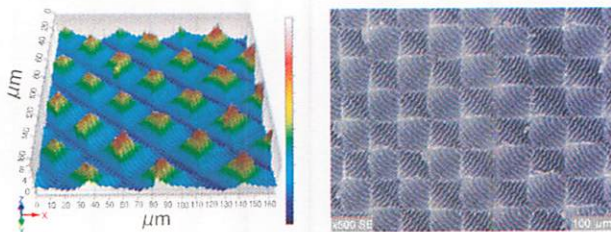


図3 工具デザインと超音波振動制御により創成した複合構造体

主軸回転を制御することで、高効率かつ大面積にサブミクロンからマイクロサイズの微細構造を創成することに成功している。

図2は、得られた微細構造の観察結果である。同図(a)の画像は、上述の相対運動を考慮して創成される構造を計算により導き出したものであり、同図(b)の画像は実際に創成された構造であるが、両者を見比べると、想定した構造とほぼ同様の構造を創成できていることが確認できる。また、図3は特殊な工具を用いた切削プロセスに3次元の超音波振動を援用することで得られた表面である。使用する工具と運動によって同図に示すような、マイクロオーダーのプリズム形状の中に、サブマイクロオーダーの微細な溝が存在する非常にユニークな複合構造体を創成するこ

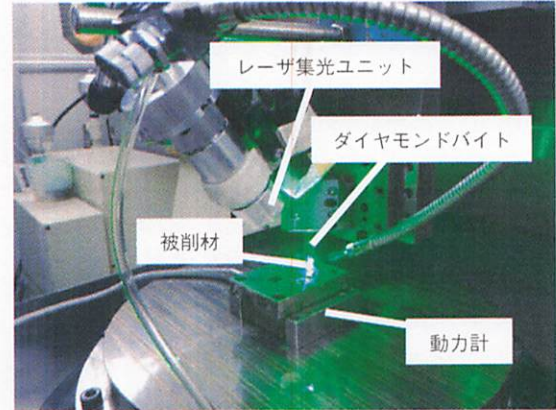


図4 レーザ援用マイクロ切削法 (セッティングの様子)⁸⁾

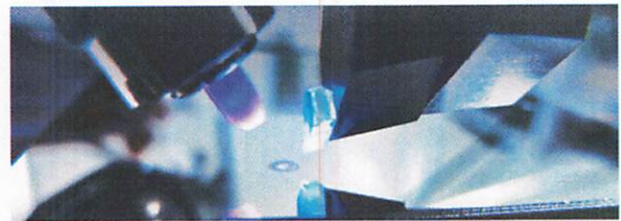


図5 レーザ援用マイクロ切削法 (加工部付近)

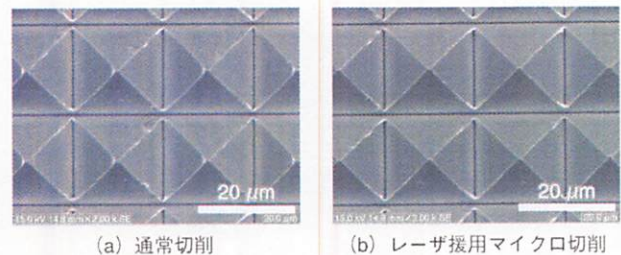


図6 レーザ援用マイクロ切削による欠陥抑制効果 (欠け発生の抑制)⁸⁾

とも可能になる。

現在は、本プロセスで創成される多彩な形状の構造体に対して、形状を考慮に含め、濡れ性をはじめとするさまざまな機能性を記述できる解析手法の構築を行っており、目的とする機能から逆算的に構造をデザインする方法の開発も行っている。

2.2 レーザ援用マイクロ切削法による微細構造体の欠陥抑制¹⁾⁸⁾

光学素子成形などに利用される微細構造を有する金型を超精密切削により作製する場合には、その原理上、バリや欠けといった欠陥がサブマイクロからマイクロメートルオーダーで発生し、形状精度が低下する。さらに対象とする構造の微小化により欠陥の影響は相対的に増大するため、(光学)製品のさらなる微細化、高精度化を実現するためには欠陥の抑制が重要な課題となる。

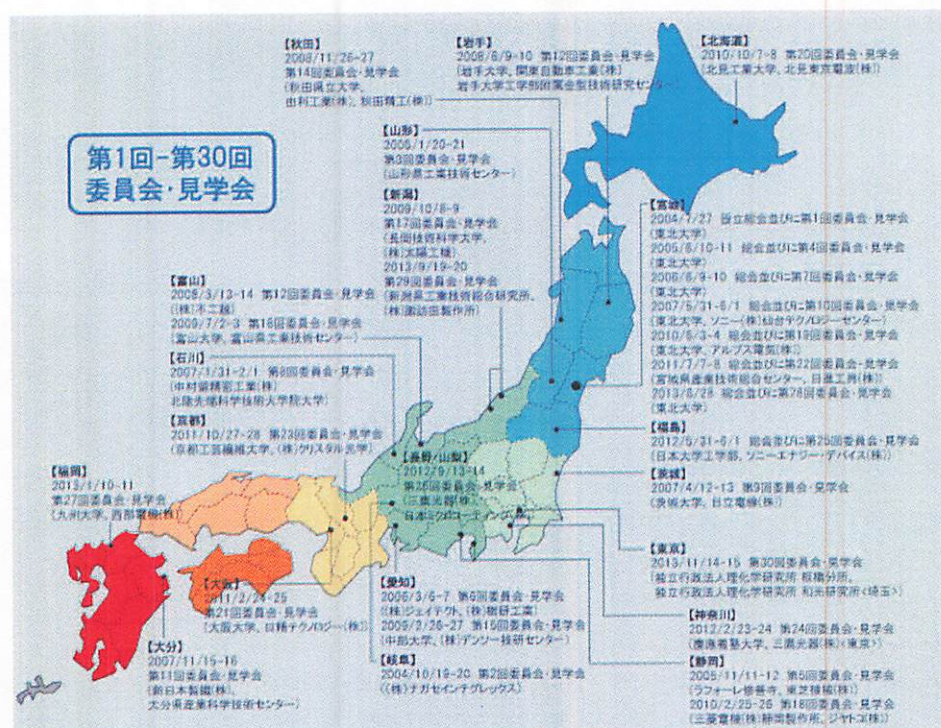


図7 ナノ精度機械加工専門委員会 第1～30回委員会・見学会

この課題を解消する加工法として、レーザー援用切削を微細加工に適用したレーザー援用マイクロ切削法を提案している(図4および図5)。レーザー照射は、材料表層の物性を变化させる手法として非常に有効なツールになる。そのような現象を利用し、機械加工プロセス(超精密切削)にレーザー照射を援用することにより、切りくず生成部分の局所的な材料物性を变化させ、例えば光学的な機能性を有する微細構造体を欠陥レスで高効率に加工するプロセスの構築を行っている。

これまでに、解析と実験によりその効果の実証を行っている。具体的には、レーザー走査を模擬した移動熱源熱伝導解析によりレーザー加熱の効果を定量的に明らかにしている。また、切削実験によってレーザー援用マイクロ切削の有効性について検証を行っており、提案手法による切削抵抗低減効果や、それに伴う欠陥抑制効果(欠け発生の抑制)を明らかにしている(図6)。

3. 本委員会の活動

本委員会は、定例の委員会のほか、技術懇談会や工場見学、あるいは国内・国際シンポジウムなどさまざまなイベントを通じてホットな研究成果をいち早く会員の皆さまに届けるとともに、自由な議論・交流の場を提供している。

特に本委員会では年に3～4回のペースで委員会・見学会を開催しており、2004年の発足から2018年3月現在までに計45回の研究会を行ってきた。例えば図7に第1～30回までに開催された研究会を日本地図上にまとめたものを示すが、全国各地で開催している様子が分かる(ス

ベースの都合上、図中に示した文字が小さいため具体的な内容までは読み取れないと思われるが、もしご興味をおもちいただいた場合には当委員会にお問い合わせいただきたい)。

また、第31～45回までの約5年間の活動についてその具体的な内容を後述するが、これらの情報からも分かるとおり、特に最近では、他の委員会や学協会などと連携して研究会・見学会を開催するなど、本委員会で取り扱う「ナノ精度機械加工」だけでなく、それに関わる材料や計測、分析などの先端技術を交えながら、より深く、自由な議論ができる場を提供している。

さらに、約2年に1度のペースで国際シンポジウムも開催している。図8はこれまでに開催された国際シンポジウムを世界地図上に示したものである。また、具体的な内容も後述するが、本委員会では特にアジア地域を中心にシンポジウムを開催し、そのトレンドの調査と情報交換を行っている。

【委員会・見学会(第30～45回)】

第30回委員会・見学会(東京都 埼玉県)
平成25年11月14日～15日
開催場所:(独)理化学研究所 板橋分所
工場見学:(独)理化学研究所 和光研究所
第31回委員会・見学会(岡山県)
平成26年1月8日～9日
開催場所:岡山大学
工場見学:ナカシマメディカル株式会社



図8 ナノ精度機械加工専門委員会 第1～8回国際シンポジウム

第32回委員会・見学会（茨城県／東京都）

平成26年3月13日～14日

開催場所：大学共同利用機関法人 高エネルギー加速器
研究機構

工場見学：JAXA 筑波宇宙センター

総会ならびに第33回委員会・見学会（宮城県）

平成26年6月6日

開催場所：東北大学

第34回委員会 兼 2014年度精密工学会秋季大会 大会シン
ポジウム（鳥取県）

平成26年9月17日

開催場所：鳥取大学

第35回委員会・見学会（愛知県／岐阜県）

平成27年1月8日～9日

開催場所：名古屋大学

工場見学：三菱マテリアル株式会社

総会ならびに第36回委員会・見学会（千葉県）

平成27年6月25日

開催場所：千葉大学

第37回委員会 兼 2015年度精密工学会秋季大会 大会シン
ポジウム（宮城県）

平成27年9月5日

開催場所：東北大学

第38回委員会・見学会（石川県／富山県）

平成28年1月7日～8日

開催場所：金沢工業大学

工場見学：コマツ NTC 株式会社

総会ならびに第39回委員会・見学会（宮城県／福島県）

※（公社）砥粒加工学会 研削・研磨盤の高度化専門委員
会（GAP）と共催

平成28年7月14日～15日

開催場所：東北大学

工場見学：株式会社 IHI

第40回委員会 兼 2016年度精密工学会秋季大会 大会シン
ポジウム（茨城県）

平成28年9月7日

開催場所：茨城大学

第41回委員会 兼 日本学術会議・公開シンポジウム（東
京都）

平成29年2月3日

開催場所：日本学術会議・講堂

総会ならびに第42回委員会・見学会（神奈川県）

平成29年6月29日

開催場所：慶應義塾大学

第43回委員会・見学会（宮城県）

※トライボコーティング技術研究会と共催

平成29年9月14日～15日

開催場所：東北大学

工場見学：株式会社日立情映テック

第44回委員会 兼 第4回板橋オプトフォーラム（東京都）

平成29年10月18日

開催場所：板橋区立文化会館2F 小ホール

第45回委員会・見学会（宮城県）

平成30年3月9日

開催場所：東北大学

【国際シンポジウム（第1～8回）】

第1回国際シンポジウム（台北・新竹，台湾）

平成17年5月25日～29日

開催場所：機械工業研究所

工場見学：機械工業研究所，利汎科技公司，全敏精密研
磨公司

第2回国際シンポジウム（シドニー、オーストラリア）

平成18年5月10日～14日

開催場所：シドニー大学

工場見学：ANSTO

第3回国際シンポジウム（仁川・ソウル、韓国）

平成19年7月4日～7日

開催場所：KITECH

工場見学：Solutec, OSG Korea

第4回国際シンポジウム

International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM 2012) (桂林、中国)

平成22年8月17日～20日

開催場所：桂林賓館

工場見学：High Precision Machinery Transmission Case Factory, Mini Vehicular Engine Factory

第5回国際シンポジウム

International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM 2012) (宮城県)

平成24年4月18日～20日

開催場所：松島 一の坊

工場見学：トヨタ自動車東北株式会社

第6回国際シンポジウム

International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM 2014) (西安、中国)

平成26年4月21日～24日

開催場所：Xi'an International Conference Center

工場見学：Shaanxi Blower 社、西安交通大学 生産システム工学国家重点研究室

第7回国際シンポジウム

International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM 2016) (秋田県)

平成28年4月25日～28日

開催場所：田沢湖ブラザホテル

工場見学：株式会社斉藤光学製作所、秋田昭和産業株式会社

第8回国際シンポジウム

International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM 2018) (南京、中国)

平成30年5月8日～11日

4. おわりに

本委員会では今後も年4回程度の委員会・見学会を定期的に開催するとともに、国内・国際シンポジウムも精力的に企画する予定である。また、イベントごとに技術交流会を開催し、研究者、技術者どうしの活発な意見交換を行う場を提供する予定である。本委員会に興味をもたれた方々のご参加をお待ちしている。

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S1320206

短パルスレーザーによる高機能切削工具の開発

Development of High Functional Cutting Tool with Nanostructure
Fabricated by Short Pulse Laser○学 福井 一裕^{*1}, 小玉 脩平^{*1}, 清水 康平^{*1}嶋田 慶太^{*1}, 水谷 正義^{*1}, 厨川 常元^{*1}Kazuhiro FUKUI^{*1}, Shuhei KODAMA^{*1}, Kouhei SHIMIZU^{*1},
Keita SHIMADA^{*1}, Masayoshi MIZUTANI^{*1} and Tsunemoto KURIYAGAWA^{*1}^{*1} 東北大学 Tohoku University

In the field of cutting, improvement of friction characteristics and process under the dry condition has been demanded to achieve cost-effective and environmentally friendly process. Hard coating is a well-known method to reduce friction, but limitations of control of film are also pointed out. Micro- or nanostructures on the friction surfaces has also been paid attention because they can reduce friction. The authors have tried to realize a further friction reduction than using only each method by creating nanostructures on a hard coating with a short pulse laser through the phenomenon called laser induced periodic surface structure (LIPSS). In this report, two samples, LIPSS on the hard coating and hard coating on LIPSS, were prepared and these surface properties were evaluated. Then the friction characteristics of them by friction test under the dry condition were evaluated.

Key Words : Friction, Hard coating, LIPSS, Nanostructure, Short pulse laser

1. 結 言

近年、切削加工の分野において作業の高効率化へ向けた加工速度の高速化や環境負荷低減へ向けた切削油を用いないドライ加工などの需要が高まっている。これらの需要に応えるため、次世代の工具にはこれまで以上に耐摩耗性および摩擦特性の向上が要求される⁽¹⁾。耐摩耗性および摩擦特性を向上させる技術として硬質皮膜を表面にコーティングする手法が広く知られている⁽²⁾。また、摩擦面にマイクロメートルスケールの微細構造を施す手法が知られている⁽³⁾。摩擦面に創成された微細構造は接触面積の低減や摩耗粉の排出などの役割を担い摩擦低減に貢献する⁽⁴⁾。一般的に硬質材料が用いられる工具に対して微細加工を施すためには機械加工以外の手法が望ましいことから、本研究では短パルスレーザーによる微細構造の創成手法に注目した。本手法は、直線偏光のレーザーをアブレーション閾値近傍の強度で照射した際、照射面にレーザー波長と同程度からそれ以下の周期を有するLIPSS (Laser Induced Periodic Surface Structure)と呼ばれる微細な溝構造が偏光方向と垂直に創成される現象を利用している⁽⁵⁾。

本研究の最終的な目的は短パルスレーザーと硬質皮膜コーティングの技術を組み合わせ、低摩耗性および低摩擦性を有した次世代の高機能切削工具を開発することであり、本報では微細構造創成後にコーティングを行った試料 (LC 試料: LIPSSed-then-coated sample) およびコーティング後に微細構造を創成した試料 (CL 試料: Coated-then-LIPSSed sample) を作成し、その摩擦・摩耗特性に関して評価・検討を行った。

2. 実験方法

本実験ではピコ秒レーザー発振器 (EKSPLA 社製, PL2250-50-P20) を用いてレーザーを照射した。表 1 にレーザー照射条件を示す。レーザー照射中にステージを移動させることで相対的にレーザーを走査させ微細周期構造の面創成を行った。微細周期構造と硬質皮膜の組み合わせを考慮するため鏡面加工した母材に硬質皮膜を膜厚 1.0 μm でコーティングした後、LIPSS を創成した試料 (CL 試料)、LIPSS 創成後に PVD 法を用いて硬質皮膜を膜厚 0.3 μm で

コーティングした試料（LC 試料）の 2 種類を作成した。レーザ照射前の試料表面における算術平均粗さ S_a 、最大高さ粗さ S_z はそれぞれ CL 試料で $S_a = 0.010 \mu\text{m}$ 、 $S_z = 0.272 \mu\text{m}$ 、LC 試料で $S_a = 0.008 \mu\text{m}$ 、 $S_z = 1.015 \mu\text{m}$ であった。

Table 1 Laser beam parameters.

	Coated-then-LIPSSed (CL) sample	LIPSSed-then-coated (LC) sample
Wavelength	1064 nm	
Pulse width	20 ps	
Frequency	50 Hz	
Laser fluence	0.0715 J/cm ²	0.226 J/cm ²
Number of shots	150	75
Base material	Stainless steel	Cemented carbide

3. 実験結果

3・1 短パルスレーザによる微細周期構造創成

図 1 は各試料のレーザ照射部の走査電子型顕微鏡（SEM: Scanning Electron Microscope）画像であり、それぞれの条件において微細周期構造の創成が確認できる。また、図 2 は各試料表面を原子間力顕微鏡（AFM: Atomic Force Microscope）を用いて測定した 3 次元画像とその断面プロファイルであり、断面プロファイルより算出した LIPSS の寸法は CL 試料で波長 883.8 nm、構造高さ 156.5 nm、LC 試料で波長 921.7 nm、構造高さ 140.8 nm と、波長および構造高さに大きな差異は確認されない。一方で SEM 画像および 3 次元形状より CL 試料ではより微細な構造も創成されているのが確認される。LIPSS の構造周期は照射エネルギーに依存し、照射エネルギーが小さいほど構造周期が狭くなることが知られている⁽⁶⁾。超硬と硬質皮膜でアブレーション閾値が異なり微細周期構造の創成可能エネルギーが異なり、CL 試料の微細構造では照射エネルギーが低いいためより微細な構造が複合的に創成されたと考えられる。

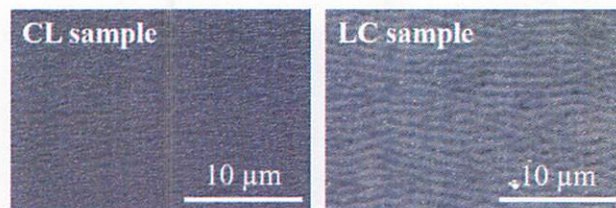


Figure 1 SEM images of microstructures: CL sample, LC sample.

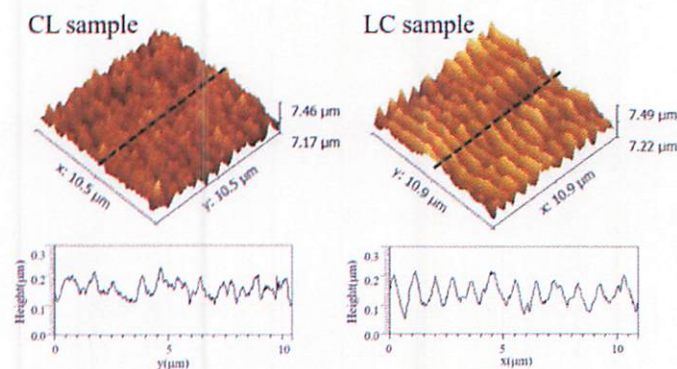


Figure 2 3D AFM topography of microstructures: CL sample, LC sample.

3・2 摩擦試験

本試験ではボールオンディスク方式を用いて摩擦試験を行った。摩擦試験は各母材を鏡面に仕上げてコーティングした試料(C 試料), CL 試料および LC 試料に対し, 直径 5.0 mm のアルミナボールを用いて摺動速度 10 mm/s, 荷重はそれぞれ 50 g, 500 g, 2000 g を加え行った。

図 2 に CL 試料および C 試料に対する各荷重条件における平均摩擦係数を示す。同図より 50 g および 500 g では LIPSS 創成試料において摩擦係数の低下が確認される一方, 2000 g では CL 試料において摩擦係数の増加が確認された。CL 試料に対する摩擦試験後の試料表面の SEM 画像を図 3 に示す。同図より荷重の増加に伴い LIPSS の摩擦が生じ, 2000 g では完全に摩滅しているのが確認される。また, 図 4 に示す摩擦試験後の摩擦ボール表面の画像より, C 試料ではいずれの荷重条件においても摩耗粉の発生が確認されない一方で, CL 試料では 500 g および 2000 g の条件で摩耗粉が発生しているのが確認される。この LIPSS の摩擦は硬質皮膜にレーザを照射したことにより硬質皮膜の組成が変化し, 軟化したためであると考えられる。以上より 50 g および 500 g の条件では LIPSS の摩擦は見られるものの摩滅には至らず構造が残存することで摩擦係数が低減するが, 2000 g では LIPSS が摩滅し多量の摩耗粉となり摩擦係数が増加したと考えられる。

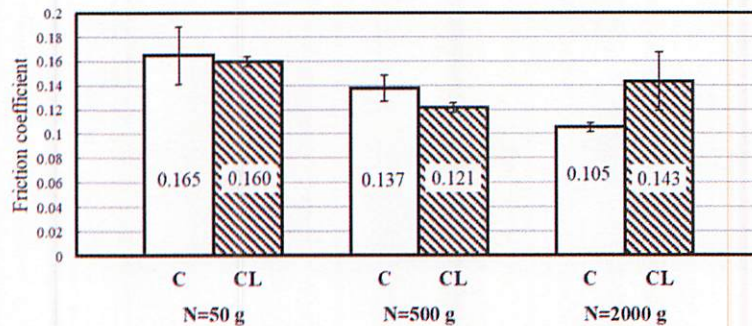


Figure 2 Average friction coefficients of CL samples and coated samples

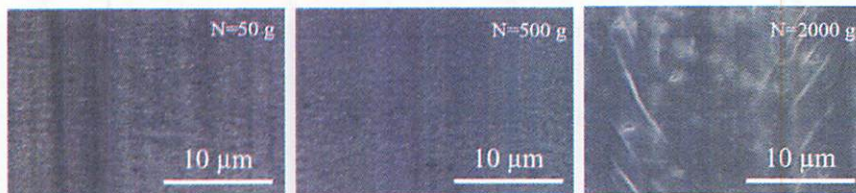


Figure 3 SEM images of CL samples

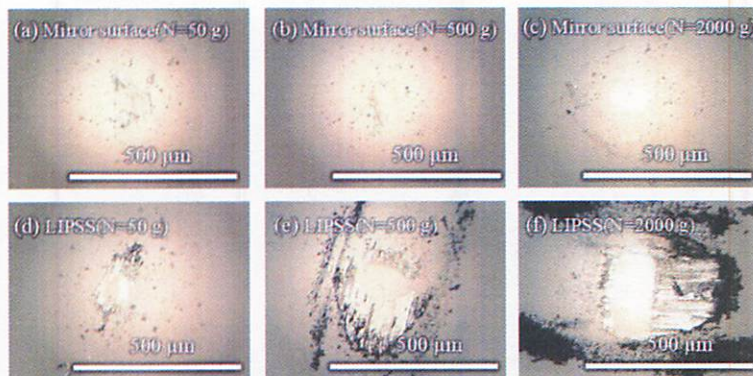


Figure 4 Friction ball surfaces after friction tests

図5にLC試料およびC試料に対する各荷重条件における平均摩擦係数を示す。同図より50gではLC試料の摩擦係数がC試料よりも大きい一方、500gおよび2000gではLC試料の摩擦係数がC試料よりも小さいことが確認された。図6に各荷重条件における摩擦試験後のLC試料表面SEM画像を示す。同図よりいずれの荷重においてもLIPSSが残存しているのが確認される。また、図7に示す摩擦試験後の摩擦ボール表面の画像をよりC試料に対しLC試料において摩擦面に付着している摩耗粉の量が少ない様子が確認される。以上より500gおよび2000gではLIPSSにより摩耗粉の排出・トラップが促進され摩耗粉による耕起を抑制し摩擦係数が低減したと考えられる。また、500gおよび2000gと比較し、50gのみ接触面積が小さくて、摩擦面に付着する摩耗粉の量が多いことが確認できる。50gでは他の荷重とは摩擦状態が異なり摩擦面での摩耗粉の耕起による摩擦が生じにくくLIPSSが残存しているにもかかわらず摩擦低減効果が十分に発揮されず摩擦係数がほとんど変化しなかったと考えられる。

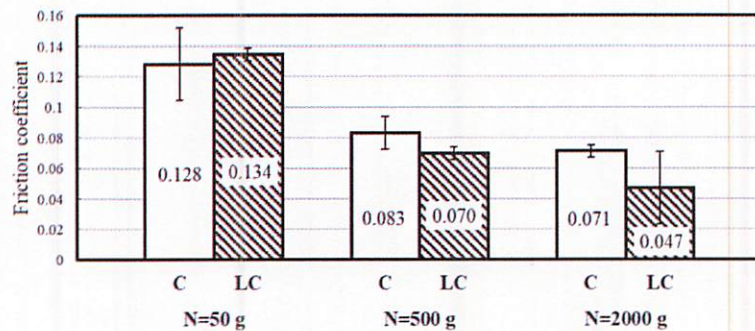


Figure 5 Average friction coefficient of LC samples and C samples

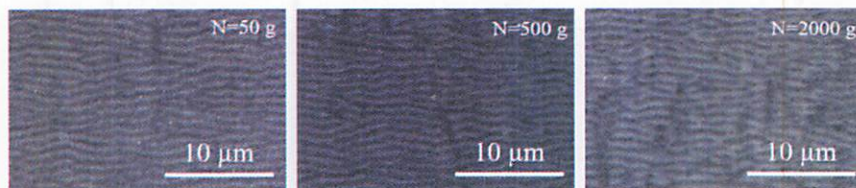


Figure 6 SEM images of LC samples

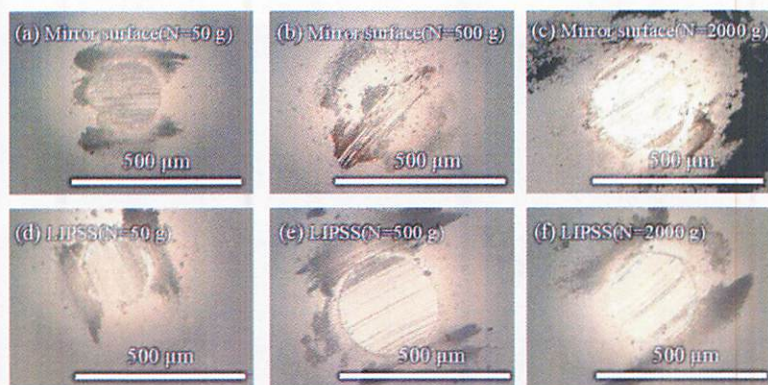


Figure 7 Friction ball surfaces after friction tests

4. 結 言

本研究では短パルスレーザによる微細周期構造と硬質皮膜コーティングを複合した構造創成面の摩擦特性および摩擦試験後の表面について評価した。以下に結論を示す。

- (1) 超硬および硬質皮膜にレーザを走査させることで、照射面端部など一部を除き微細周期構造を一様に創成することが可能である。また、微細構造創成後に硬質膜によるコーティングを行った場合にも同様の構造が再現される。
- (2) 硬質皮膜コーティング後に微細周期構造を創成した試料では、レーザ照射により硬質皮膜が軟化したと考えられ、高荷重条件の摩擦試験において微細周期構造が摩滅し摩擦係数が増加しているのが確認された。
- (3) 超硬に微細周期構造を創成後、硬質皮膜をコーティングした試料では、摩擦係数が低減し摩擦試験後も構造が残存しているのが確認されたため、微細周期構造と硬質皮膜を複合させることにより摩擦特性および耐摩耗性の向上が期待される。

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J0270302

プラズマ放電改質チタンの耐摩耗性と耐食性の評価

Evaluation of Wear and Corrosion Resistance of PDM Treated Titanium

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Titanium alloys is widely used as biomedical implant materials due to its high strength and biocompatibility. However, poor wear resistance has prohibited its application as bearing materials for artificial joints. In order to improve the wear resistance of titanium alloys, much research has been done on surface modification of titanium. Plasma discharge modification (PDM) is one of the promising surface modification methods of titanium. In this study, wear and corrosion resistance of PDM treated titanium, TiC processing and Si processing, was evaluated. The result of ball on disk wear test showed that PDM greatly improves the wear resistance of titanium. This is attributed to the hard TiC layer formed by PDM both on TiC processing and Si processing. The result of anodic polarization tests revealed that the PDM layer shows equal or superior passive current density compared to titanium substrate. However, the pitting potential decreased compared to titanium substrate. Observation of the tested samples show that the corrosion started from the dimple and the interface of the PDM shots created during modification. Therefore, the early pitting potential may be caused by the galvanic coupling of the substrate and the PDM layer at the dimple and PDM shot interface.

Key Words : Plasma Discharge Modification, PDM, Surface modification, Titanium, TiC coating

1. 結 言

超高齢社会を迎えた我が国の人工関節の需要は増加の一途をたどっている。人工関節の摺動部材には耐摩耗性に優れる Co-Cr 合金が適用されている。体内環境で用いられる人工関節には、耐食性も要求されている。Co-Cr 合金の場合、体液との腐食反応により溶出した Co イオンが細胞毒性を生じ、ガン化の懸念がされている¹⁾。したがって、長寿命型人工関節の創製には、耐食性に優れるチタン系材料への代替が提案されている。しかしながら、Co-Cr 合金と比較してチタン系材料は耐摩耗性に劣ることから、耐摩耗性の改善が必要とされている。

本研究では、純チタンおよびチタン合金の耐摩耗性の改善手法として、TiC と Si の電極を用いたプラズマ放電改質（以下 PDM: Plasma Discharge Modification）に注目し、改質材の耐摩耗性と耐食性の評価を行った。

2. 実験方法

2・1 供試材と改質条件

改質基材には、生体用インプラントで汎用される純チタン（CP-Ti）と Ti-6Al-4V 合金を用いた。表 1 に化学組成を示す。PDM（プラズマ放電改質）の電極には、TiC 電極と Si 電極を用いた。それぞれの電極材で改質された素材を TiC 改質材および Si 改質材とする。表 2 に PDM による改質条件を示す。比較材として、鏡面研磨した純チタン（CP-Ti）および Ti-6Al-4V 合金、生体用 Co-Cr 合金（ASTM F799）を用意した。表 3 に Co-Cr 合金の化学組成を示す。PDM で改質した表面性状は凹凸状なため、凸部を 10 μ m 研磨し、供試状態とした（図 1）。

Table 1 Chemical composition of CP-Ti (Grade 2), Ti-6Al-4V alloy (Unit: weight%)

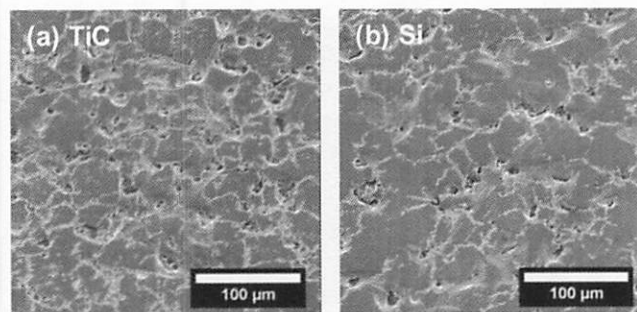
	N	C	H	Fe	O	Al	V	Ti
CP-Ti (Grade2)	0.03 $\geq$	0.08 $\geq$	0.013 $\geq$	0.25 $\geq$	0.20 $\geq$	-	-	Bal.
Ti-6Al-4V	0.05 $\geq$	0.08 $\geq$	0.0150 $\geq$	0.40 $\geq$	0.20 $\geq$	5.50-6.75	3.50-4.50	Bal.

Table 2 PDM parameters

Working parameters	TiC processing	Si processing
Electrode materials	TiC electrode	Si electrode
Discharge Current (A)	10	5.5
Open circuit voltage (V)	320	260
Discharge duration (μ s)	8	8
Pulse interval (μ s)	256	64

Table 3 Chemical composition of Co-Cr alloy (ASTM F799) (Unit: weight%)

	Mn	Si	Ni	Fe	C	N	Mo	Cr	Co
ASTM F799	1.00 $\geq$	1.00 $\geq$	1.00 $\geq$	1.5 $\geq$	0.35 $\geq$	0.25 $\geq$	5.0-7.00	26.0-30.0	58-59

Fig. 1 Surface of PDM treated CP-Ti after 10 μ m polish.

2・2 化学組成の同定、表面性状および組織の観察

被膜組成の同定には、X線回折装置を用いた。被膜を構成している元素の定量分析には、エネルギー分散型X線分光装置(EDX)を用いた。表面性状の観察には、走査型電子顕微鏡(SEM)を用いた。

2・3 耐摩耗性と耐食性の評価

耐摩耗性の評価は、アルミナボールを相手材とした Ball on disk タイプの摩耗試験を行った。試験環境は 37°C の生理食塩水中で実施した。表面粗さ試験機で摩耗痕を測定し、得られた測定曲線から摩耗体積を算出した。

耐食性の評価はアノード分極試験を行った。電位の掃引は-1V から 4V までとした。掃引速度は 20mV/min とした。試験環境は窒素バブリングをした 37°C の生理食塩水中を用いた。

3. 実験結果および考察

3・1 PDM 改質材の化学組成の同定

図2にCP-Ti基材のPDM材とPolish材のX線回折結果を示す。同図より、TiC改質材およびSi改質材ともに基材の α -Tiに加えて、明瞭なTiCの回折ピークが同定された。また、Si改質材においては、Siの回折ピークは同定されなかった。表4にEDXによるPDM材の構成元素の定量分析結果を示す。同表より、チタン元素(Ti)と炭素(C)が同程度含まれていることがわかる。これらより、チタン系材料に対するTiCおよびSi電極で成膜されるPDM材の被膜の主成分は、電極材の種類に関わらずTiCが主体であることがわかった。

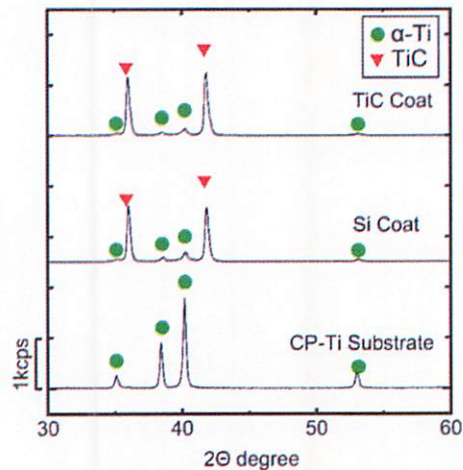


Fig. 2 X-ray diffraction patterns of PDM treated specimens and CP-Ti substrate.

Table 4 Quantitative analysis of PDM treated surface by EDX.

Coat	Substrate	Element (at.%)		
		Ti	C	Si
TiC	CP-Ti	55.0	45.0	-
	Ti-6Al-4V	54.3	45.7	-
Si	CP-Ti	57.3	40.8	1.9
	Ti-6Al-4V	56.4	39.5	4.1

3・2 摩耗試験結果

図3にPDM材とPolish材の摩耗試験結果を示す。同図の縦軸は、粗さ試験機で摩耗痕の断面積から摩耗体積を算出した結果を示している。図3(a)より、PDM材の摩耗量はCP-TiおよびTi-6Al-4VのPolish材と比較して顕著に低減にしていることがわかる。図3(b)より、PDM材の摩耗量は未処理のCo-Cr合金よりも少ないことがわかる。未処理のCo-Cr合金の硬さはHV354、PDM材はHV2000程度である。したがって、硬さの違いが摩耗量に影響したと考えられる。また、Si改質材の方がTiC改質材の摩耗量に比べて少ないことがわかる。改質面の最大高さ粗さ R_z は、TiC改質材では $7.1\mu\text{m}$ 、Si改質材では $5.16\mu\text{m}$ であった。このことは、Si改質材の方がTiC改質材よりも溶融粘度が小さいため、結果として溶融部が扁平に広がる要因と考えられる⁽²⁾。

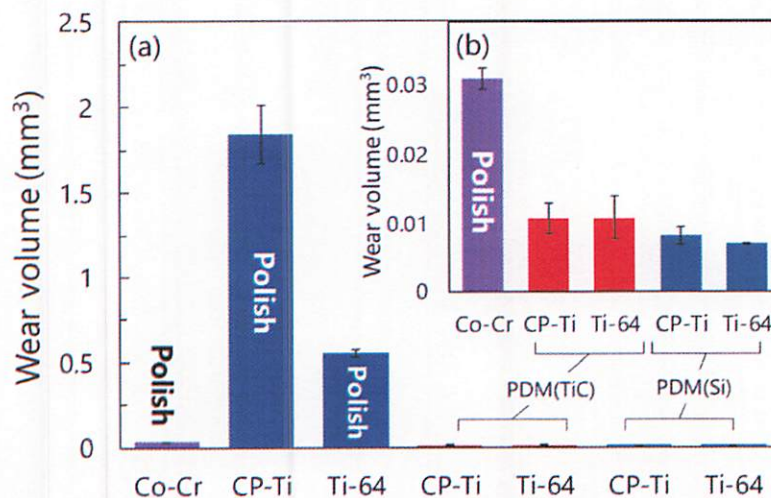


Fig. 3 Wear volume of polished specimens and PDM treated specimens.

3・3 アノード分極試験結果

図4(a)にCP-Ti, (b)にTi-6Al-4V基材のPDM材とPolish材のアノード分極試験結果を示す。同図より、酸化(腐食)反応が始まる腐食電位 E_{corr} は、電極によらずPDM材の方がPolish材よりも高いことがわかる。すなわち、PDM材が熱力学的に安定で、イオン溶出しにくいことを示唆している。電流密度の急激な上昇を意味する孔食電位 E_p は、基材および電極によらずPDM材は1.4V付近であることがわかる。すなわち、改質層に対する不動態被膜の形成能は電極によらず同程度であることを示唆している。加えて、Polish材の孔食電位 E_p はPDM材よりも高く、PDM材の耐食性が劣ることがわかる。また、CP-Ti基材の不動態保持電流密度 i_p は、TiC改質材とPolish材では同程度であるのに対して、Si改質材では低いことがわかる(図4(a))。Ti-6Al-4V基材では、電極材に因らずPolish材と同程度であることがわかる(図4(b))。これより、Si改質材で生成される被膜の方が、不動態被膜を早期に形成すると考えられる。

3・4 アノード分極試験後の表面観察結果

図5に分極試験前、図6に2.3V分極後の試料表面の観察結果を示す。図6より成膜により生成されるディンプル状放電痕および円形放電痕の縁部が腐食していることがわかる。図7に4.0V分極後の試料表面の観察結果を示す。同図(b)より、TiC被膜が腐食により剥離し、CP-Ti基材が露出していることがわかる。図8にディンプル状放電痕を断面から観察した結果を示す(エッチング処理断面)。同図より、CP-基材が露出していることがわかる。これらより、PDM材の孔食電位の低下は、Ti基材が露出するディンプル状放電痕および、円形放電痕の縁部などが影響していることがわかった。

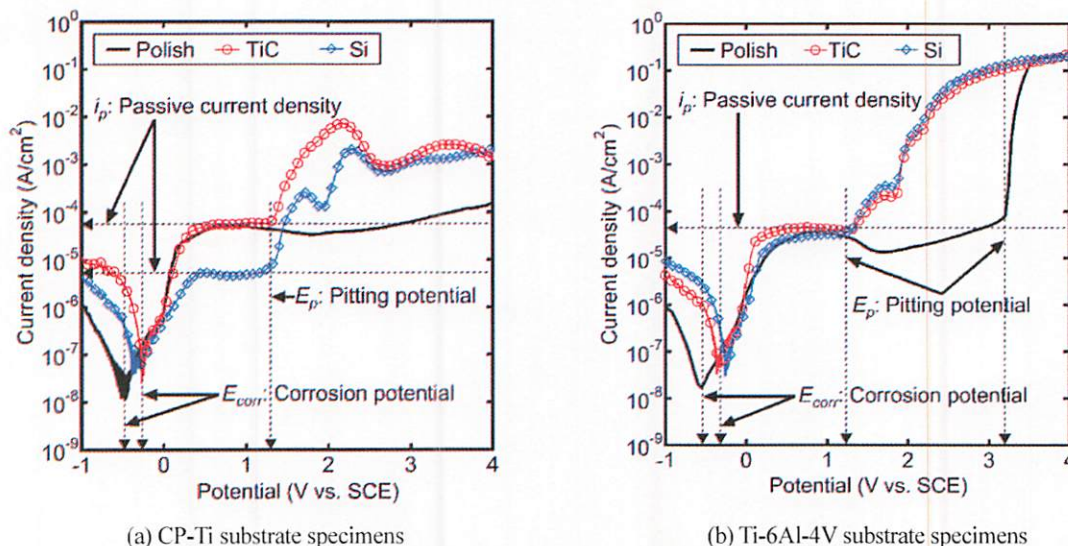


Fig. 4 Polarization curve of polished substrate and PDM treated specimens.

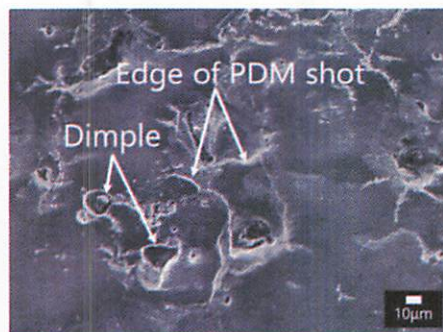


Fig. 5 Surface of TiC processed CP-Ti before polarization.

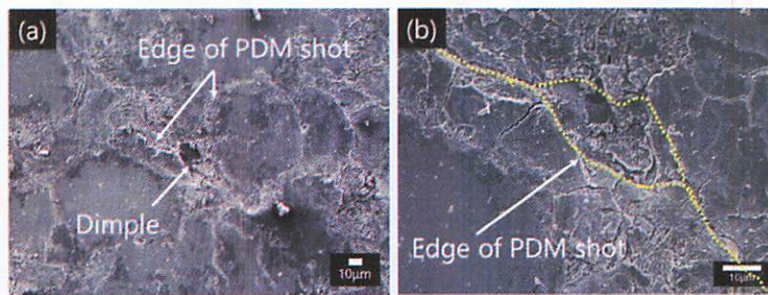


Fig. 6 Surface of TiC processed CP-Ti after 2.3V polarization showing (a)preferential corrosion around the dimple and edge of PDM shot, (b) corrosion around the edge of PDM shot.

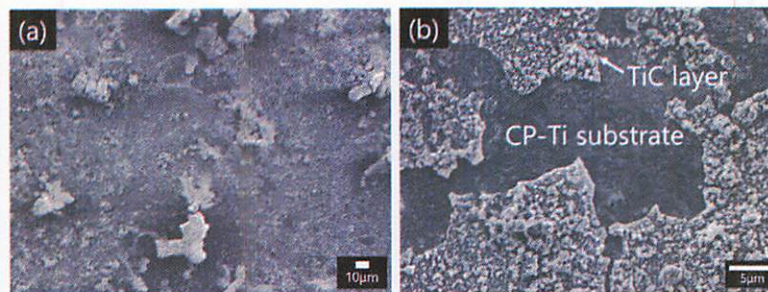


Fig. 7 Surface of TiC processed CP-Ti after 4.0V polarization showing (a)severe corrosion , (b) appearance of CP-Ti substrate.

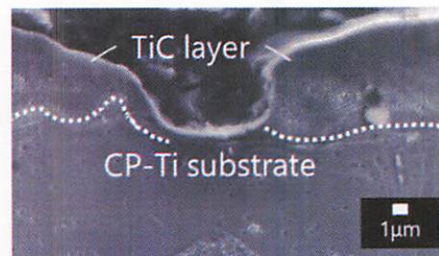


Fig. 8 Cross sectional image of PDM shot interface.

7. 結 語

本研究では、チタン系材料に対して、TiC と Si の電極を用いた PDM 改質を行い、改質材の耐摩耗性と耐食性の評価を行った。以下に結論を示す。

1. PDM 材の耐摩耗性は、大いに改善されることがわかった。
2. PDM 材の耐食性は、孔食の早期化が認められ、改善が必要であることがわかった。
3. PDM 材の腐食反応は、Ti 基材が露出するディンプル状放電痕および、円形放電痕の縁部などが影響していることがわかった。

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歯科用 PJD ハンドピースにおける微粒子流動ダイナミクス

Particles flow dynamics in dental PJD handpiece

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Powder jet deposition (PJD) is a technique to deposit material on the target under atmospheric pressure and room temperature. It has been tried to apply on dental treatment by using particles of hydroxyapatite (HA), the main component of human teeth. Prototype PJD equipment was developed in the previous research, but problems such as unstable and inefficiency jetting of HA were confirmed. In this research, we developed HA compacted powder-adopted PJD system to realize quantitative jetting of HA particles. Moreover, shape optimizations of the internal flow path were tried in order to solve the problems of PJD systems. As a result, it was confirmed that particles scattering causes particles to adhere to the wall, and that the collision of HA particles to the wall surface can be reduced by applying the expansion-and-reduction section at curving part of the flow path.

Key Words: hydroxyapatite, dental, PJD, particles, jetting, handpiece, design, developing

1 緒 言

著者らは新たな歯科治療法として、常温大気圧環境下で粒子を高速で衝突させる成膜法であるパウダージェットデポジション法（以下 PJD 法）を用いて、人歯の主構成成分であるハイドロキシアパタイト（以下 HA）を、人歯に直接成膜する手法を提案している¹⁾。本手法の実現のため、現在チューブ搬送式 PJD 装置によって臨床実験が行われているが、同装置にはチューブ内に粒子が付着・残存することにより定量的噴射が困難とし、また衛生的観点からも問題となりうるなど課題も多く、改善が必要である。本研究では粒子の定量的噴射の実現を主な目的として新型 PJD 装置である HA 圧粉体充填式 PJD 装置を設計・開発し、数値流体解析を用いてノズル部内部形状の最適設計を行うとともに成膜性能を評価した。

2 新型 PJD ハンドピースの開発

緒言で述べたように、粒子の定量的噴射を主な目的とし、衛生的観点からディスポーザブル式を採用すること、重心を手元側にすることを設計方針に加えて圧粉体充填式 PJD 装置の設計開発を行った。図 1 に圧粉体充填式 PJD 装置の外観を、図 2 に初期型の圧粉体充填式 PJD ハンドピースノズルの内部構造を示す。本装置は圧力計や流量計、送り・回転速度調整部を有する切削機構制御部と、図 2 に示したような 3D プリンタで造形したノズル部およびノズル内部に装填する圧粉体充填カップで構成される。実験時にはノズル部と切削機構をはめこみ、切削刃により HA 圧粉体を切削しながら噴射を行う。これにより粒子を定量的かつ安定して噴射でき、また粒子運搬経路が短いため衛生面の改善も期待できる。なお、切削前後の粒子性状の測定から、圧粉体の切削により元粒子と同粒径および同形状の粒子に復元可能であることを確認している。さらに切削機構とノズル部をディスポーザブル式としたことで、装置内の粒子残存やノズル内流路の壁面摩耗といった課題が解決できる。

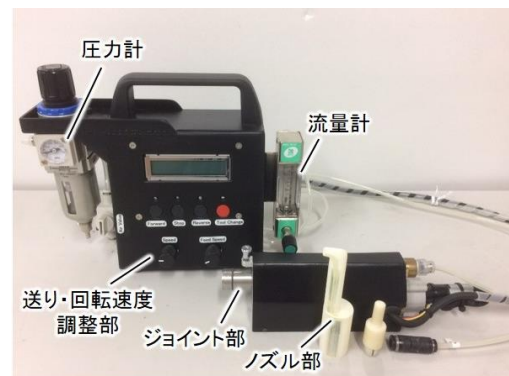


図 1：圧粉体充填式 PJD 装置外観

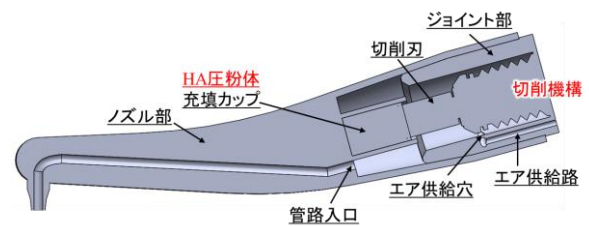


図 2：初期圧粉体 PJD ハンドピースノズル内部構造

3 ハンドピース内部流路における粒子挙動制御

初期型圧粉体充填式 PJD 装置を使用した噴射実験により、HA 粒子の流路壁面への付着、流路湾曲部における粒子衝突による壁面の摩耗、噴射効率の悪さといった問題点が確認された。これらの問題点を解決するため、以下に示すような内部流路の変形による粒子挙動の制御を試みた。

3-1 傾斜構造の導入に関する最適形状検討

壁面への粒子付着を低減させるために、管路入口前流路への傾斜構造の導入を検討した。粒子のハンドピース壁面への

付着の原因として、本研究で用いた HA 微粒子の粒径が数 μm と小さく、体積性質と比較して表面性質が支配的となるためハンドピース壁面に付着凝集しやすいことが考えられる。また壁面と粒子が接触する理由として、そもそも粒子流動が壁面に沿ったものであるほかに、流路が必要以上に大きい場合に粒子流れの方向性が乱れ粒子が飛散することが考えられる。粒子流動性に優れたノズル内部流路の形状を検討するために、流路内部の傾斜角度を変化させたモデルを作成し粒子挙動解析を行った。またその結果をもとに4種類のノズルを造形し、プラスチック袋内部に対して粒子噴射実験を行い、回収前後の重量を測定することで粒子噴射量の変化を検証した。解析および実験の条件をそれぞれ表1、表2に示す。解析から得られた、ハンドピース内部での粒子挙動図の例を図3に示す。粒子挙動計算により、傾斜角度 $\alpha = 0, 5^\circ$ では壁面に沿った粒子の挙動が、 $\alpha = 20, 25^\circ$ では粒子が飛散している様子がそれぞれ確認された。また、粒子噴射量測定実験により、図4に示すように造形ノズルの傾斜角度と粒子噴射量の間に負の相関があることが分かった。これらのことから、 $\alpha = 0^\circ$ のモデルが最も噴射粒子量と粒子流動性に優れ、ノズル壁面に付着する HA 粒子量が少ないと考えられる。さらに、解析結果と噴射粒子量の結果から、壁面に粒子が付着する原因としては粒子の飛散が最も支配的であるといえる。

3-2 流路湾曲部における拡張管形状の適用

ハンドピース湾曲部における粒子衝突を低減させるために、流路断面積変化と速度・圧力の関係性に着目した。等エントロピーかつマッハ数1以下の流れでは、流路断面積の増加により流体の圧力は増加、速度は低下し、断面積の減少により圧力は減少、速度が増加することが知られている²⁾。この性質を利用して、流路湾曲部に拡大管と縮小管を接続した形状（以下、拡張管と称する）を導入し、壁面衝突前に粒子を減速させ、噴射前に再加速させる手法を考案した。まず縦3.2, 4.8 mmの2条件、横5, 10, 15 mmの3条件とし、ノズル付近流路のみを再現した6つの空間モデルを作成し、粒子挙動解析により条件選定を行った。解析条件は基本的に表1に準拠するが、前節で解析を行った際に管路入り口部で確認された、0.25 MPa を供給圧力として管路入り口部に与えた。図5に解析から得られた各拡張管形状モデルにおける粒子位置と平均粒子速度の関係を示す。同図の横軸は拡張管中央部を原点とした場合の管路軸方向の粒子位置を示しており、グラフの負方向に行くほど噴射口部に近い部分での平均粒子速度を表している。またグラフの矢印部分は粒子が流路壁面に衝突する位置を示している。拡張管の採用により湾曲部で粒子が減速し、噴射時に再加速している様子が確認できた。解析結果から、十分な粒子減速が確認された縦4.8 mm、奥行き15 mmの寸法を持つ拡張管を最適形状と定めた。さらに、一様管ノズルと拡張管ノズルを使用し成膜実験を行ったところ、拡張管ノズルの方が流路壁面の摩耗が低減されるだけでなく、より良好な HA 成膜性能を示すことが判明した。これは一様管において壁面に粒子が衝突する際に粒子速度が著しく低下するためであると考えられ、壁面—粒子間の反発係数を変化させた解析を行うことでそれを確認した。

4 結論

本研究では、新たな歯科用 PJD ハンドピースの開発を行い、噴射実験により露呈した新型 PJD 装置の問題点を解決するため、粒子挙動解析と粒子噴射実験により内部流路の最適形状に関する検証を行った。以下に本研究で得られた結論を示す。

- (1) 現行装置の問題点である粒子の定量噴射を可能とする圧粉体充填式 PJD 装置を開発することに成功した。

表1：解析条件

乱流モデル	Realizable $k-\epsilon$
搬送流体	空気
供給圧力	0.5 MPa
HA 密度	3150 kg/m ³
HA 粒径	1, 2, 3 μm
解析モデルの傾斜角度 α	0, 5, 10, 15, 20, 25°
噴射距離	5 mm

表2：噴射実験条件

HA 粒径	2.2 μm
カップへの圧入力	2000 N
切削刃送り速度	3 mm/min
切削刃回転速度	1000 rpm
噴射時間	3 m 30 s
搬送流体	空気
供給圧力	0.5 MPa

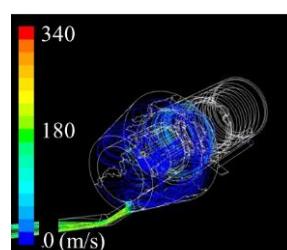


図3：粒子挙動の例

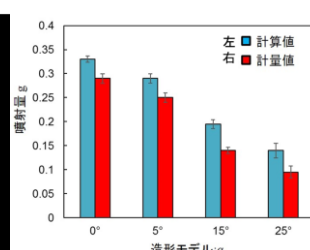


図4：傾斜角度と噴射量の関係

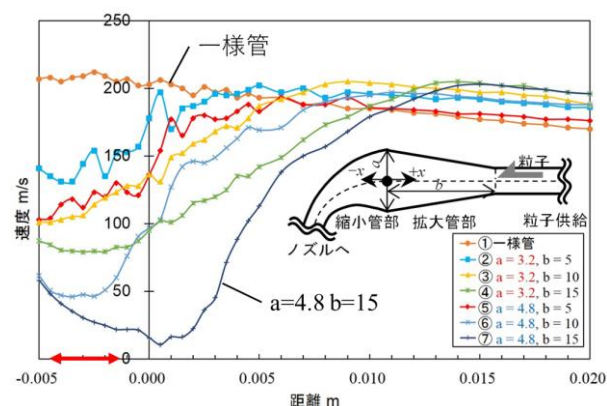


図5：ノズル付近流路における粒子の位置—速度変化

- (2) 内部流路に傾斜構造を適用することで壁面への HA 粒子付着を抑制し、噴射粒子量が増大することを確認した。また、粒子付着の原因として粒子の飛散の影響が支配的であることを示唆した。
- (3) 流路湾曲部に拡張管形状を採用することで粒子速度を制御し、壁面への粒子衝突を抑制しハンドピースの摩耗を低減可能であることを確認した。また、粒子を飛散させることなく、十分な減速および再加速が可能となる、最適拡張管形状を決定した。

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Effect of Crystal Orientation on Short Pulsed Laser-Induced Periodic Surface Structure

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Keita Shimada, Masayoshi Mizutani,
and Tsunemoto Kuriyagawa



Fine structures give new functions



2

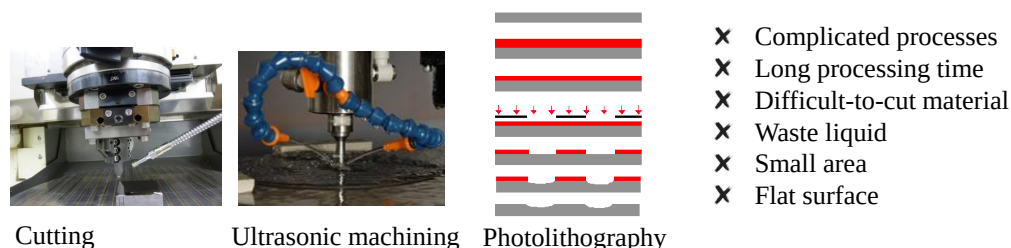


Advantages of USP laser process

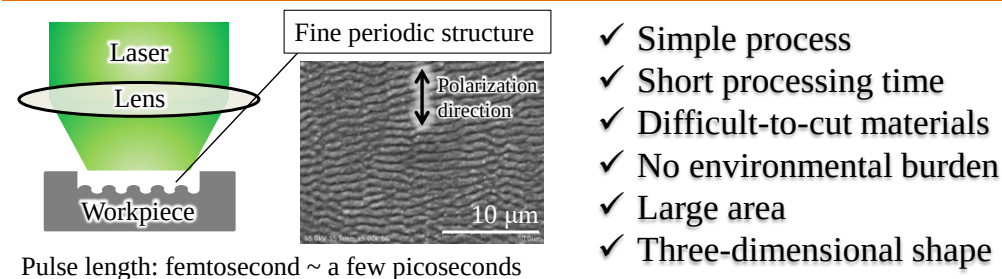


3

Traditional methods



Ultrashort pulsed-laser process



The 5th International Symposium on Micro/Nano Mechanical Machining and Manufacturing (ISMNM2018) May 8th – 11th, Nanjing, China

Fabrication mechanism

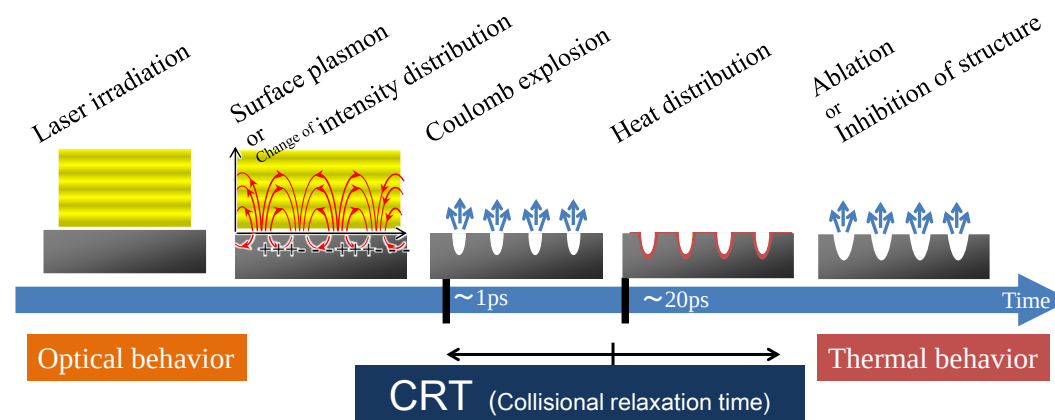


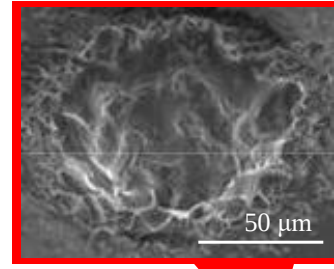
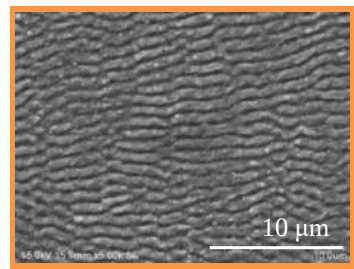
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✗ Low repeatability ✗ Difficult control of structure

➔ **Principle is not clarified** especially in Collisional relaxation time

Powerful theory (as of now)





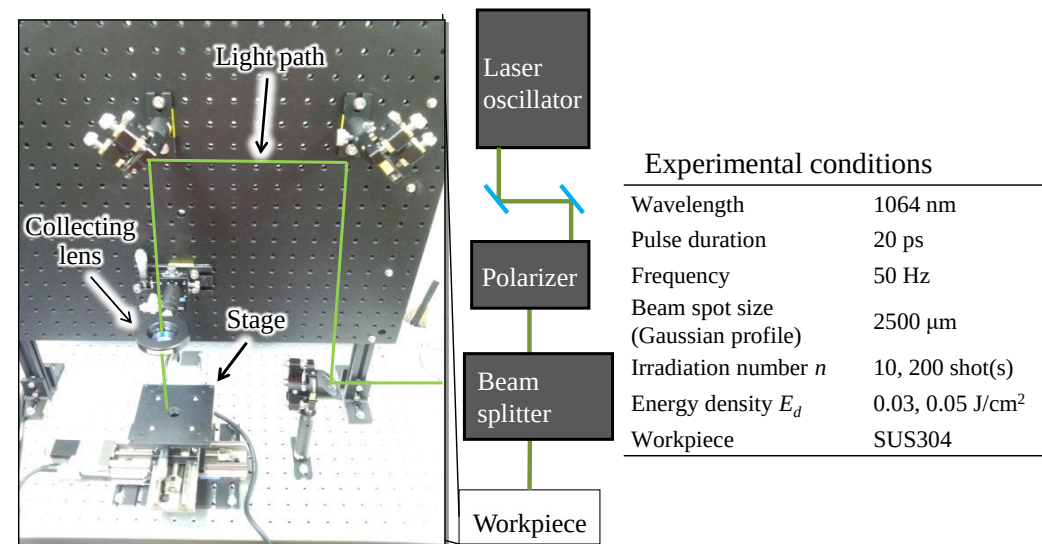
Collisional relaxation time



Ultrashort pulsed laser

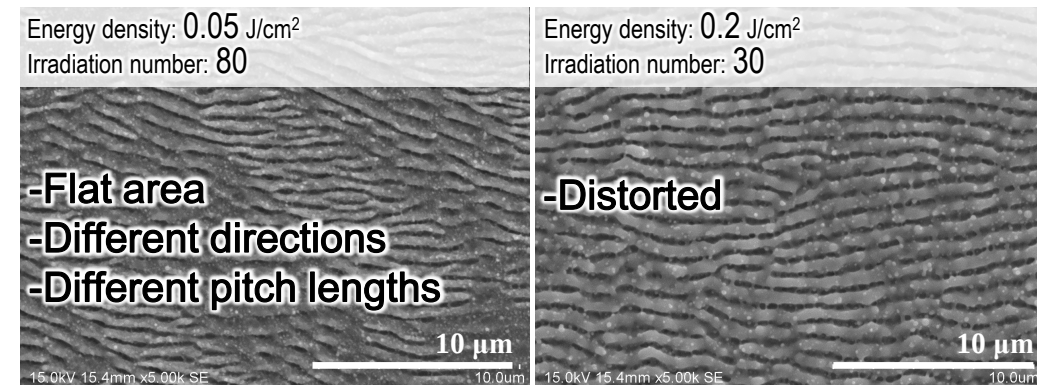
- ✗ High equipment cost
- ✗ Unstable laser irradiation

➤ Short pulsed laser

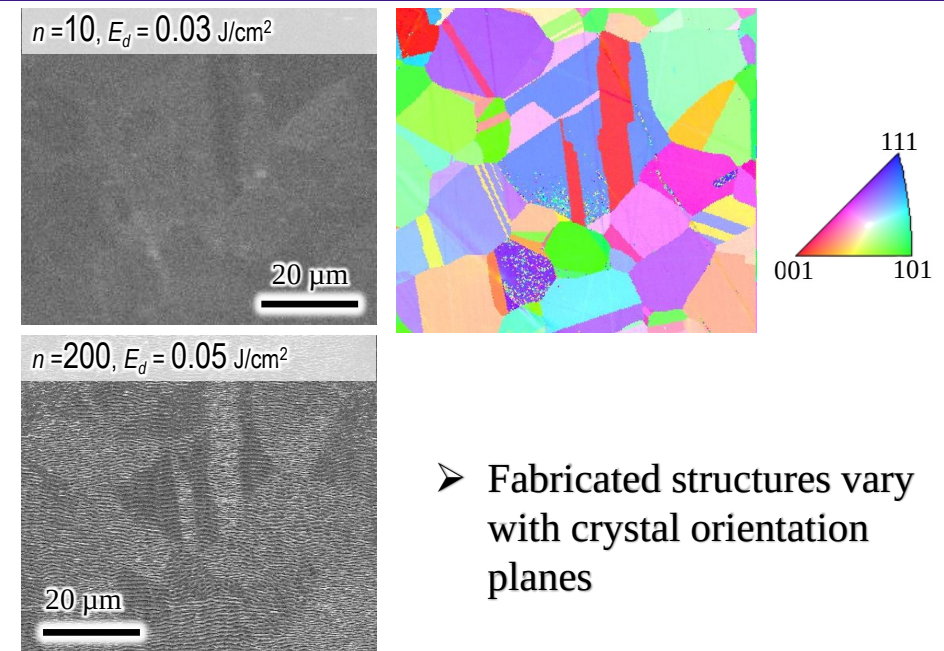


Short pulsed laser process unit and optical system

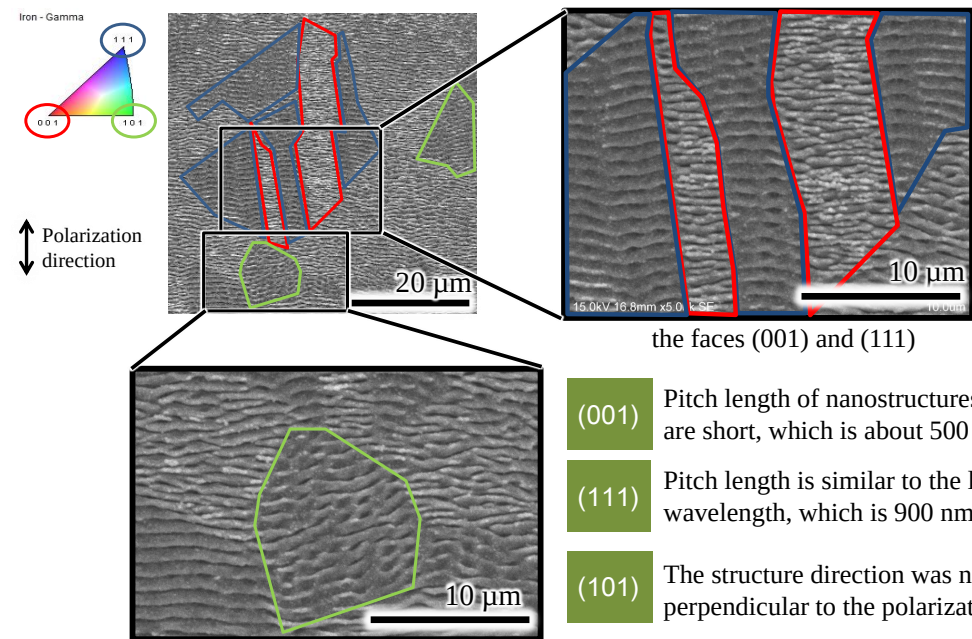
SEM images of the irradiated areas (prev. work)



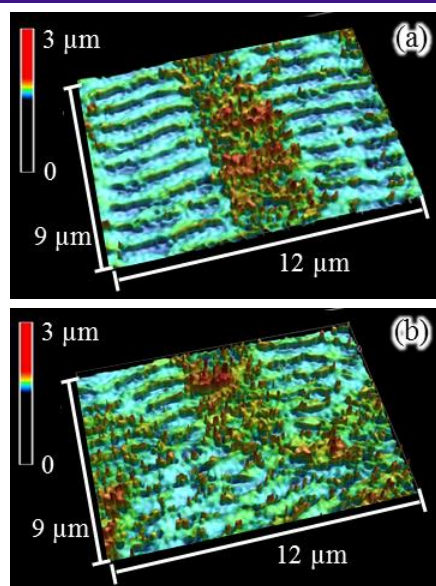
➤ To clarify these phenomena and to control structures



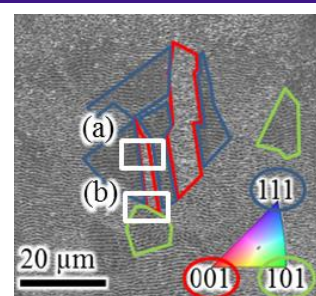
➤ Fabricated structures vary with crystal orientation planes



Expansion of the surface of each crystal orientation face which has nanostructures



Laser microscope images
 $n=200$, $E_d = 0.05 \text{ J/cm}^2$



Large



Small

(001)

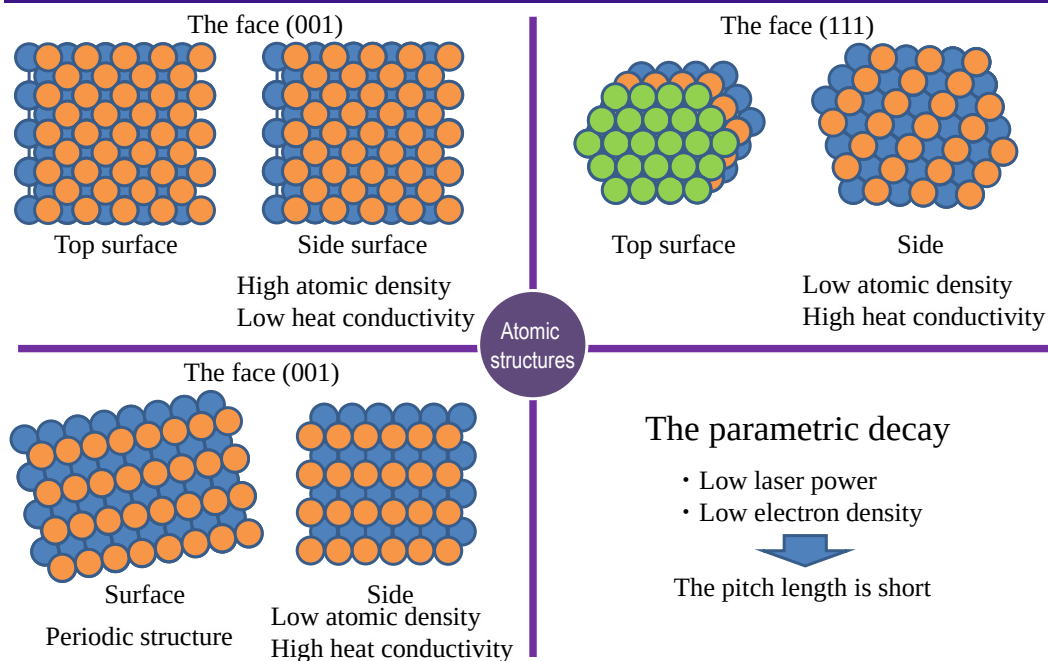
(101)

(111)

Expansion the surface of each crystal orientation face

➤ The expansion coefficient is different from the expectation

Effects of crystal structures on texturing



Conclusions

Fabricated structures vary with crystal orientation as below:

(001): The pitch length and aspect ratio of nanostructures is half the laser wavelength and high respectively due to low heat conductivity and less atomic density on the surface, and large atomic density in a depth direction.

(101): Nanostructures whose pitch length of 0.85 times the laser wavelength is not perpendicular to polarization since the periodic atomic structure is not perpendicular to polarization.

(111): Nanostructures perpendicular to polarization has the pitch length of 0.85 times the laser wavelength.

Improvement of biocompatibility for zirconia dental implant by nanosecond pulsed laser

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Grad. Sch. Eng., Tohoku Univ.
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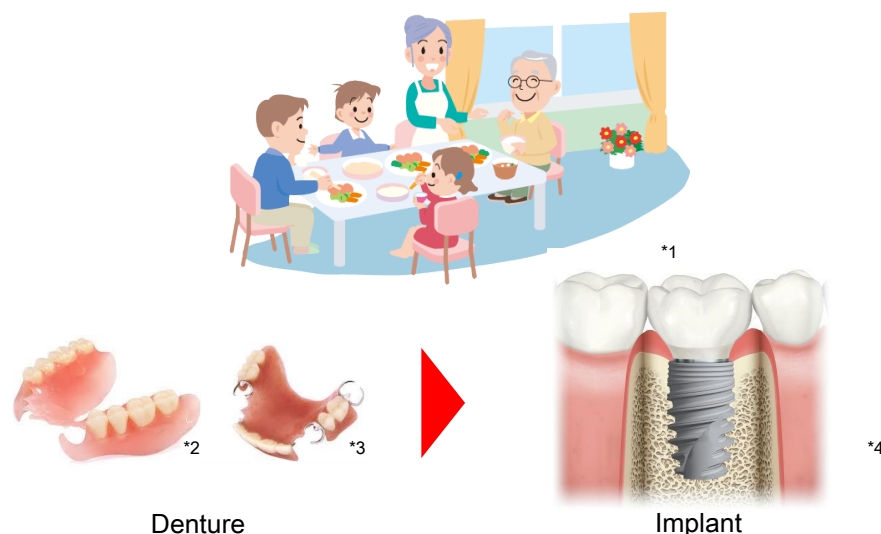


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Improving the quality of life

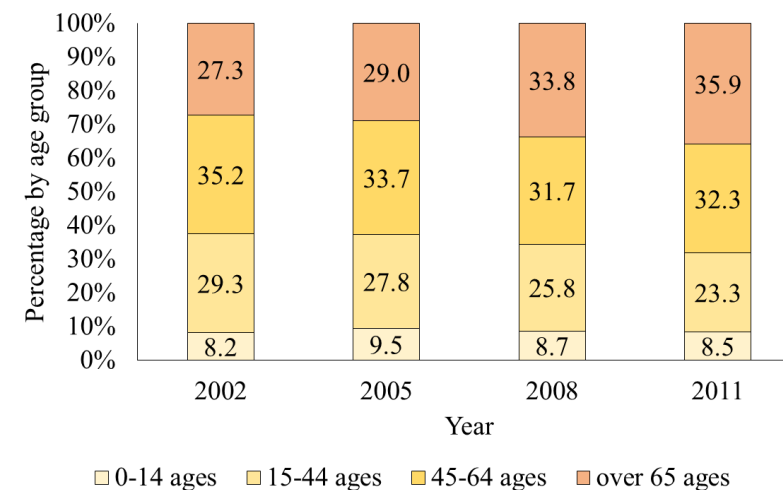


2



*1 <http://www.hosi.co.jp/customer/qol/>
*2 http://www.sanwa-dental.com/web/info/smiledenture_cameroua/
*3 <http://www.sudo-dental.info/price/>
*4 <http://www.medicalexpo.com/ja/prod/nobel-biocare-services-ag/product-73432-656920.html>

◆ Annual trend of estimated dental patients



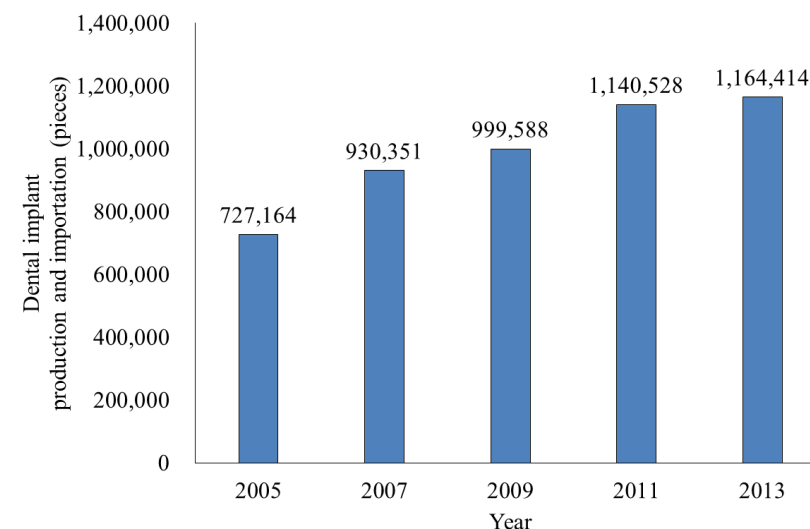
<http://www.mhlw.go.jp/file/05-Shingikai-10801000-Iseikyoku-Soumuka/0000087739.pdf>

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Demands of dental implants in Japan



4



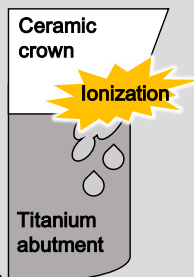
Statistics of Production by Pharmaceutical Industry, Ministry of Health, Labour and Welfare, Japan.



*1



*2



Good points

- High fatigue strength
- High biocompatibility

Bad points

- Metallic color
- Metal allergy

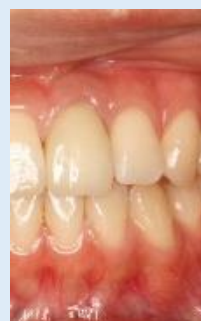
*1 <http://www.japan-implant.info/1510about> *2 <http://www.perio-tokyo.jp/>

Now aesthetic and metal-free implant is needed.

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*1



*2

Good points

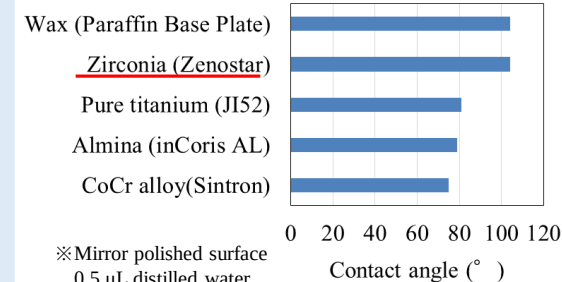
- Aesthetic whiteness
- No risk of allergy
- Higher toughness among other ceramics

* 1 http://dent-implant.jp/05reason/0525implant/oam_implant006.html. * 2 <http://www.iwaki-dental.net/staffblog/629.html>.

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*1



*2

Bad points

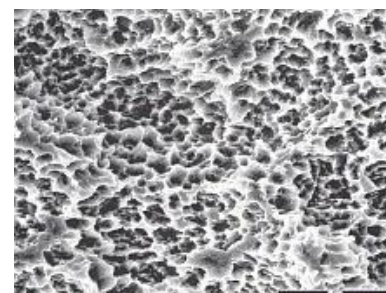
- Hydrophobic surface
- Lower biocompatibility than titanium

* 1 http://dent-implant.jp/05reason/0525implant/oam_implant006.html. * 2 S.Ban: JSB, 34(2016) 62-65.

We need to improve biocompatibility of zirconia implants.

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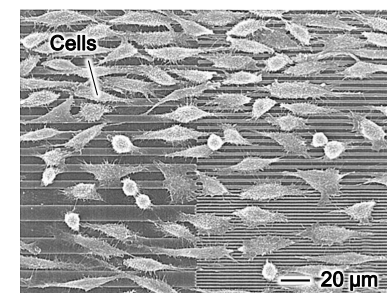
◆ Rough surface



*1

- Increasing surface areas
- Improving hydrophilicity
- Adhering early cells

◆ Groove geometry



*2

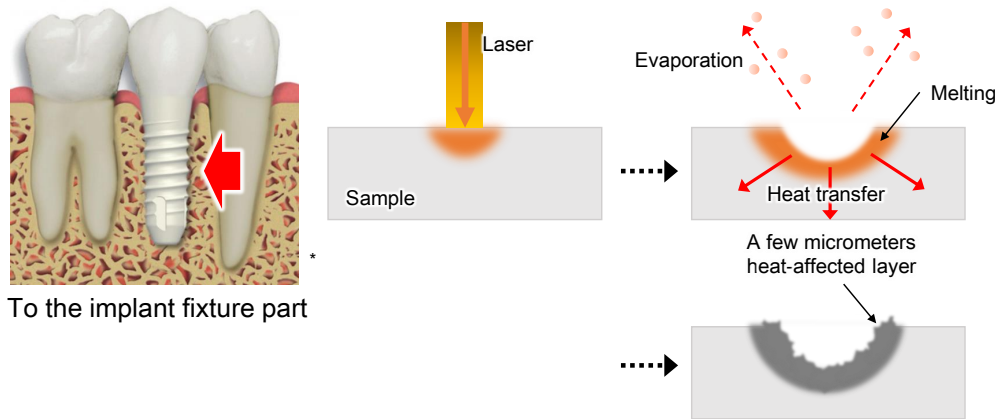
- Catching osteoblasts
- Growing cells along grooves
- Promoting calcification

*1 <http://www.sugiyama-dental.com/implant/senmon2.html>. *2 M.Yoshinari: IRUCA@TDC, 103(2003), 565-572.

We will create both patterns to zirconia surface.

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Microfabrication method for difficult-to-cut materials



*<http://www.sophiahi.com/zirconium-implants/>

- Groove geometry by removal process
- Rough surface by coagulation materials
- Chemical composition change by heat effect.

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Materials

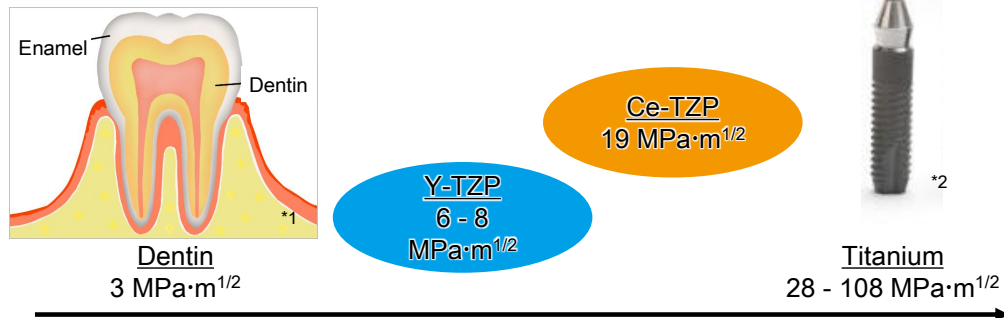
Y-TZP

Yttrium-stabilized
Tetragonal Zirconia Polycrystals

Ce-TZP

Cerium-stabilized
Tetragonal Zirconia Polycrystals
/Al₂O₃ nanocomposite

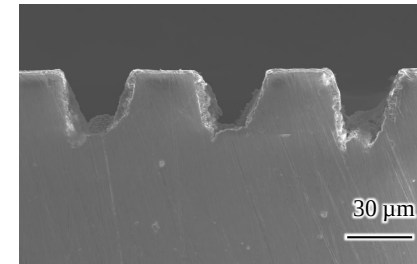
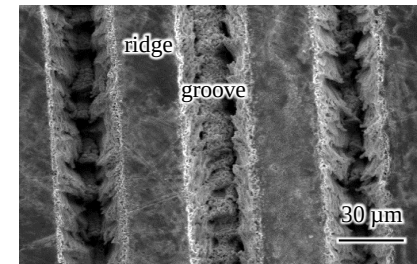
◆ Toughness comparison



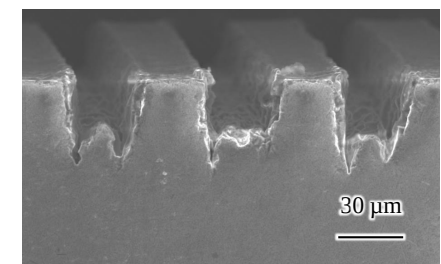
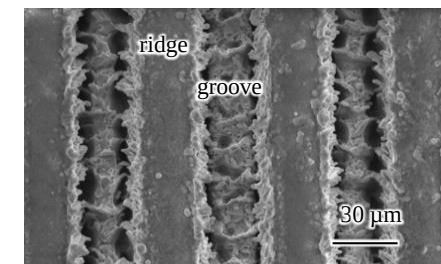
*1 <http://kibami-senshi.com/wp-content/uploads/2017/01/enamerushitsu.png>. *2 <http://www.japan-implant.info/1510about>.

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Y-TZP



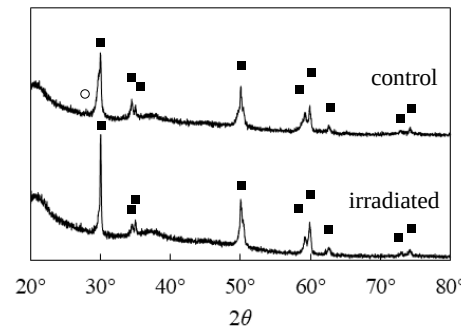
Ce-TZP



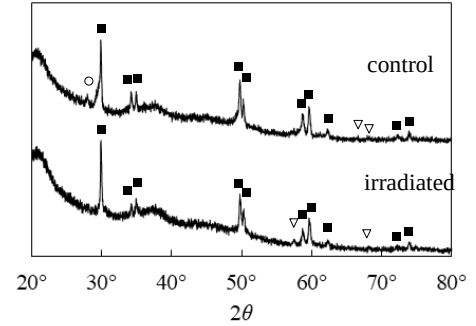
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XRD patterns

Y-TZP



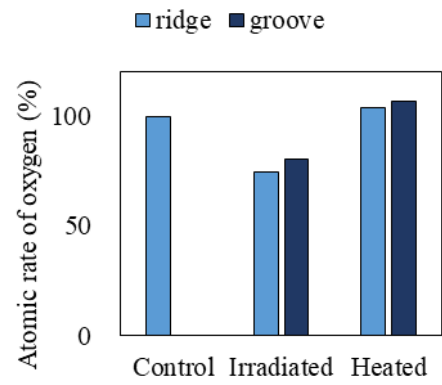
Ce-TZP



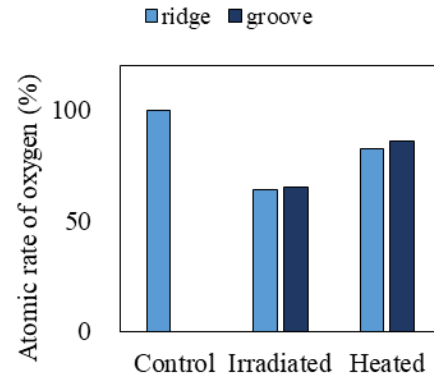
■ Tetragonal ZrO₂ ○ Monoclinic ZrO₂ ▽ Al₂O₃

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Y-TZP



Ce-TZP



Effect of Laser Scanning Conditions on Metal Microstructure of Selective Laser Melting Products

Shinji Ishiabshi*

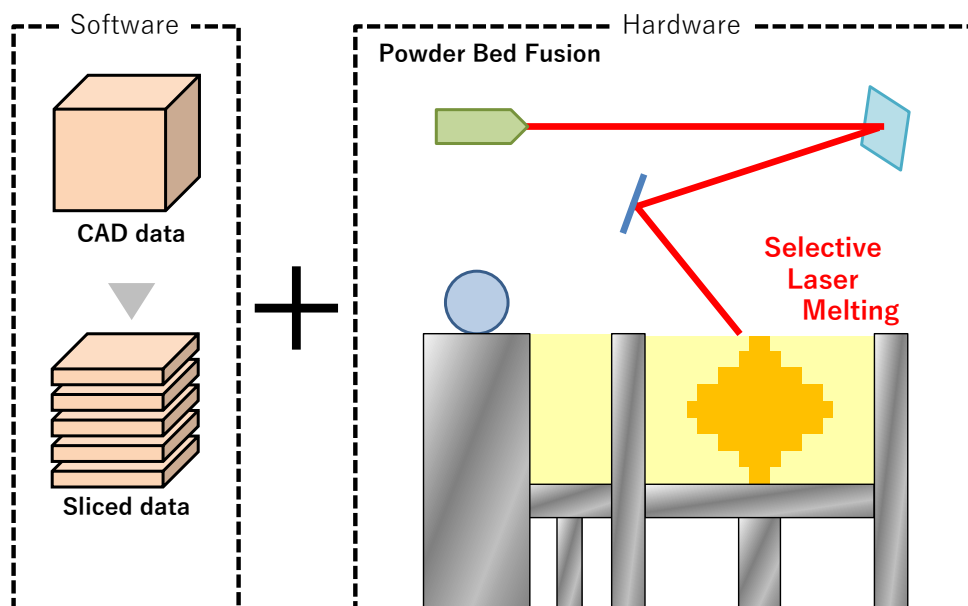
Mizoi Takumi*, Masaki Tsukuda**, Keita Shimada*, Masayoshi Mizutani*, Tsunemoto Kuriyagawa***

*Grad. Sch. Eng., Tohoku Univ., **Grad. from Tohoku Univ. (Currently in NISSAN), ***Grad. Sch. Biomed. Eng., Tohoku Univ.



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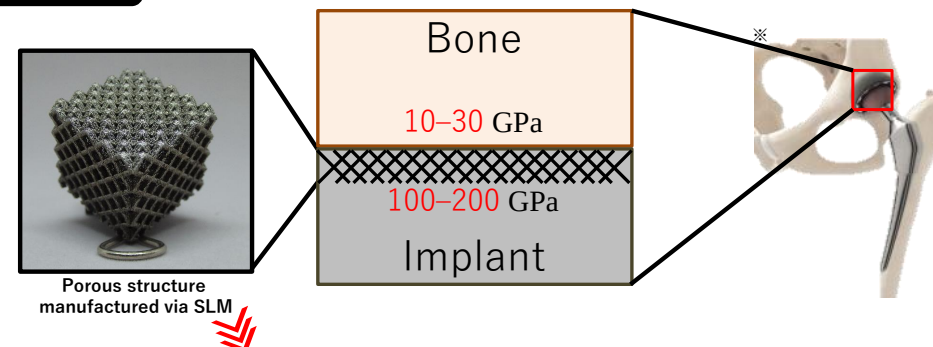
What is SLM



2

Background

Medical



To provide anisotropic mechanical properties on SLM products by controlling microstructure

Purpose

To reveal the effect of laser conditions on the microstructures in SLM products

※http://fukuyamaika.co.jp/struct/wp-content/uploads/Fotolia_50008554_S-300x200.jpg



4

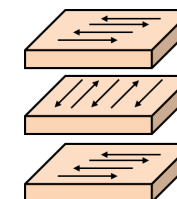
Experimental conditions

Shaping conditions

Equipment	ProX100 (3D Systems)
Atmosphere	N ₂
Laser type	Fiber laser
Laser spot diameter	80 μm
Wave length	1070 nm
power	50 W
Laser strategy	90° alternate
Thickness	30 μm



ProX100



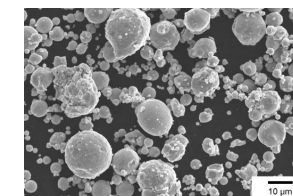
Laser strategy

Metal powder

material	17-4PH stainless steel
diameter	d10=4.4 μm, d50=11.9 μm, d90=25 μm

Chemical composition wt%

Cr	Ni	Cu	Si	Mn	Nb	C	P	S	Fe
16.4	4.3	3.9	0.7	0.7	0.2	0.02	0.018	0.010	Bal.



SEM image of powder

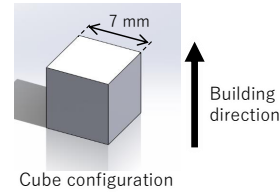
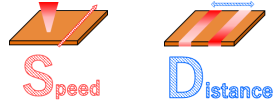


Processing parameters and Crystal structure analysis

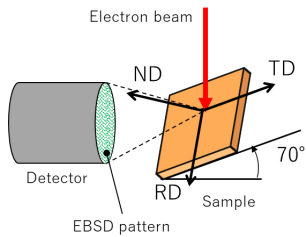
5

Laser conditions

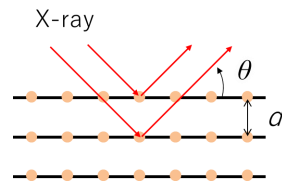
	Scanning speed mm/s	Hatch spacing μm
Cube1 (S100-D40)	100	40
Cube2 (S400-D120)	400	120
Cube3 (S100-D120)	100	120
Cube4 (S400-D40)	400	40



Electron Backscatter Diffraction



X-ray diffraction



RLAB

6

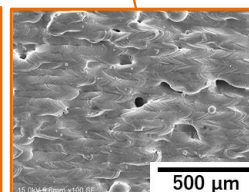
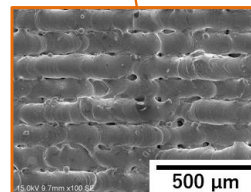
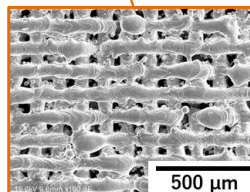
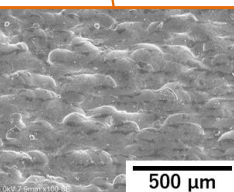
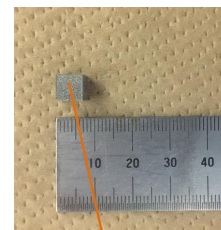
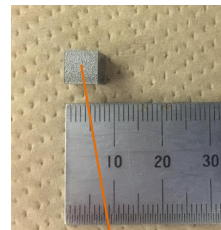
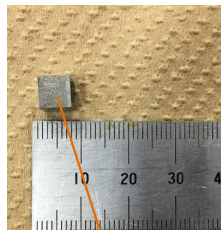
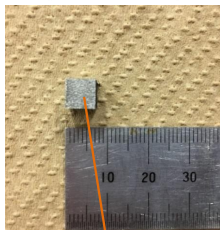
Top views of cubes

Cube1
(S100-D40)

Cube2
(S400-D120)

Cube3
(S100-D120)

Cube4
(S400-D40)

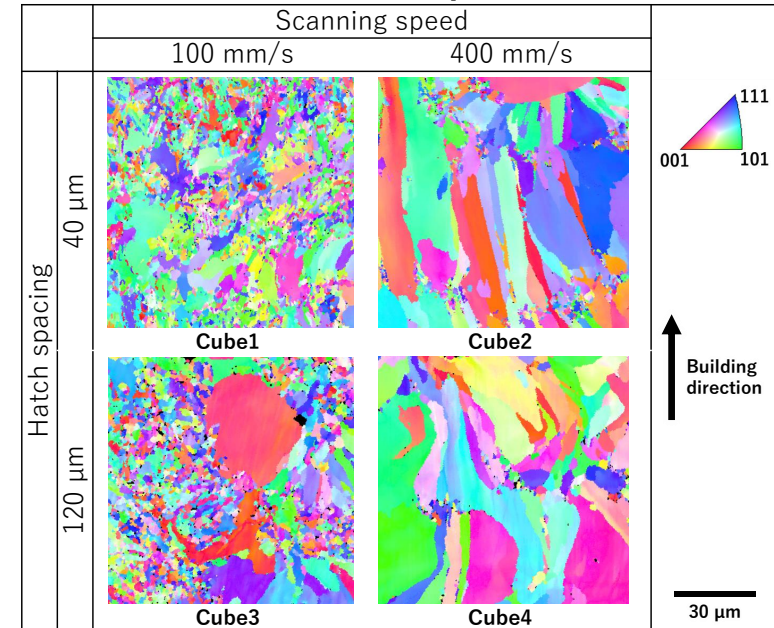


RLAB

Grain morphologies

7

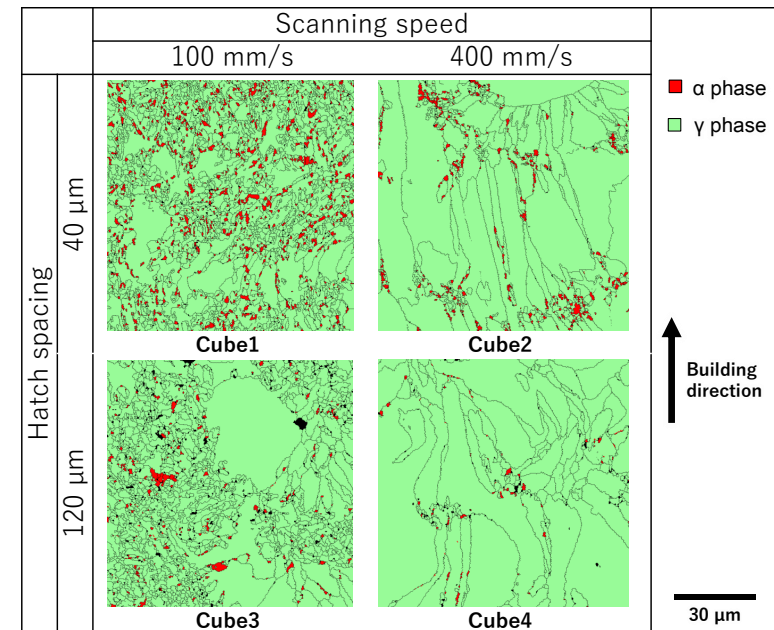
IPF Map



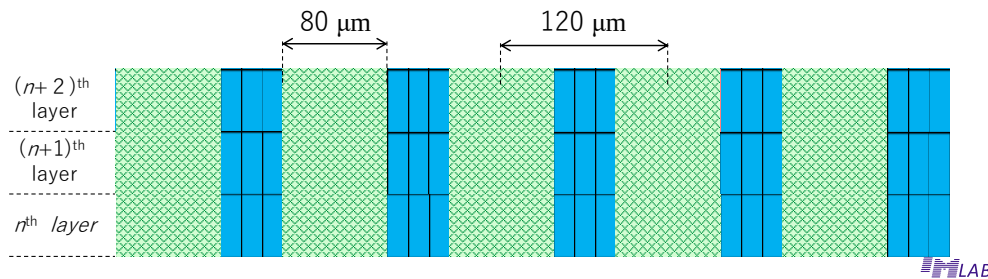
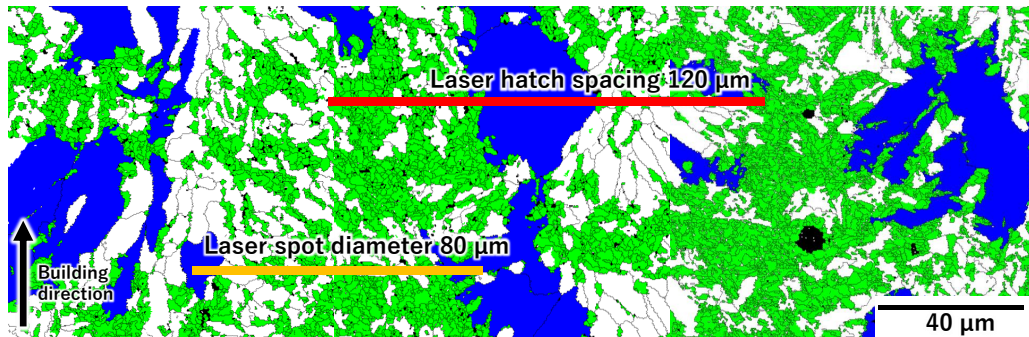
RLAB

8

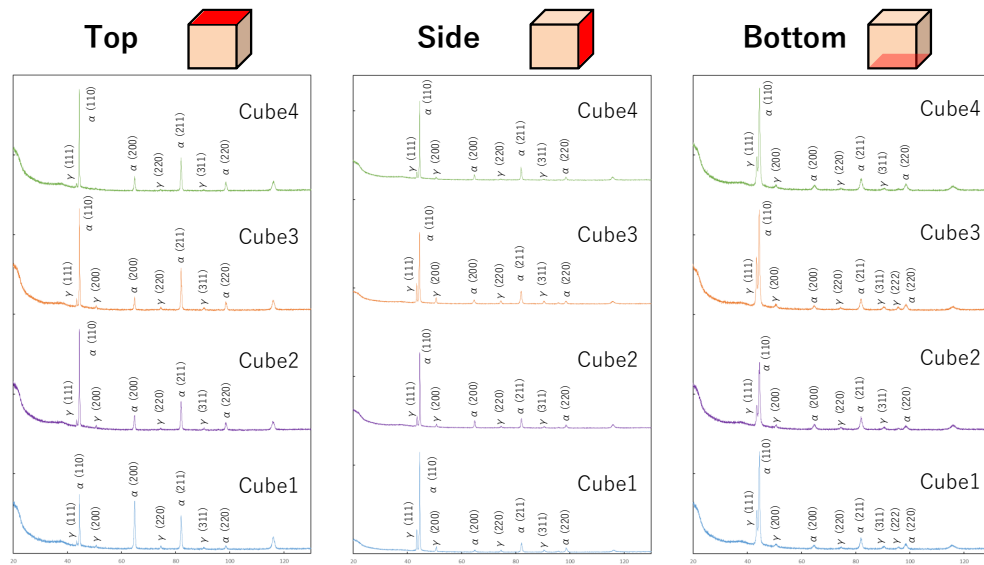
Phase maps



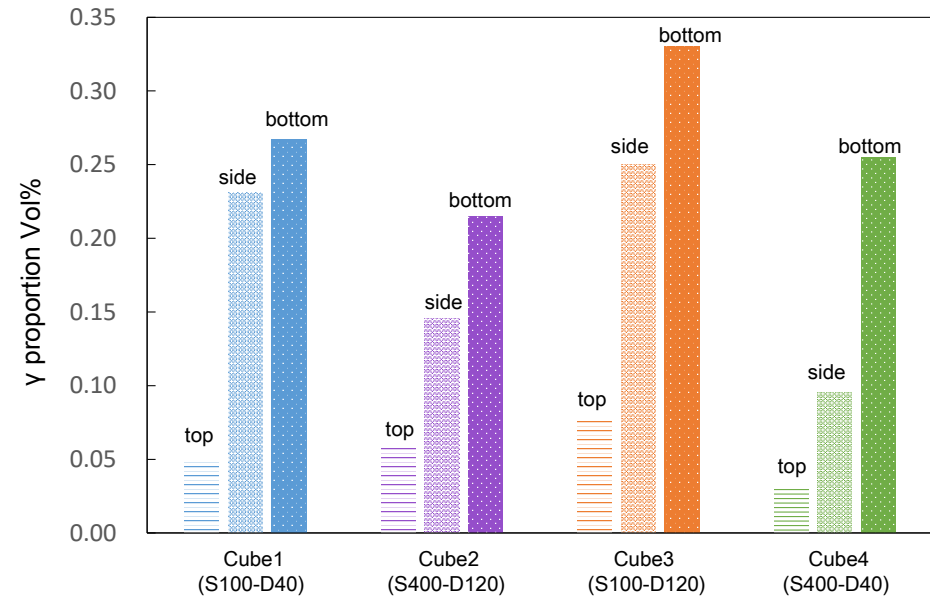
RLAB



XRD diffraction patterns



Volume fraction of γ phase



Conclusions

- 1 Succeeded in creating unique samples consisting of fine grains and/or longitudinal grains.
- 2 Confirmed that the amount and distribution of γ phase varied depending on the energy density and repeated heat input.
- 3 Proposed a mechanism how to generate and transform the microstructure during SLM process.

Fabrication of functional surfaces by ultrasonic-assisted cutting

Keita Shimada*(Presenter)
Takuya Hirai**, Masayoshi Mizutani*, Tsunemoto Kuriyagawa***

*Grad. Sch. Eng., Tohoku Univ. **Grad. from Tohoku Univ. (Currently in AISIN)
***Grad. Sch. Biomed. Eng., Tohoku Univ.



Control of wettability



Wettability = Chemical properties + Geometrical features

Elements & functional group

Hydrophobicity

Hydrophilicity

Emphasis of Wettability

Liquid spreads on & within structures
▶ Contact area ▲

Wenzel's model

$$r\gamma^{LG}\cos\theta + \gamma^{LS} = \gamma^{SG}$$

Change to hydrophobic

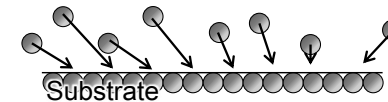
Air is captured
▶ G-S boundary forms

Cassie-Baxter's model

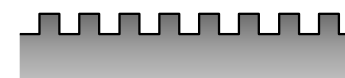
$$\cos\theta_{cb} = f\cos\theta + f - 1$$

Major methods

- Vacuum deposition
- Lithography



deposition



- Control of form
- ✗ Expensive
- ✗ Limit of materials
- ✗ Time-consuming

Ultrasonic texturing

Ultrasonic vibration



Feed



Removal



- Cost-effective
- Versatile
- Effective

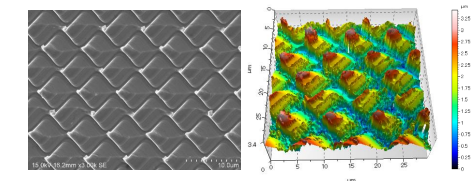
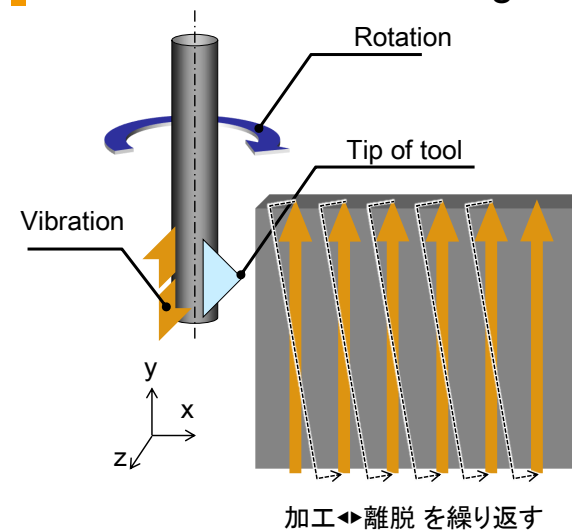
We've focused on
This

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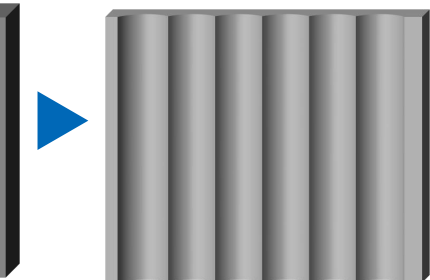
Composite fine structures



Ultrasonic assisted cutting

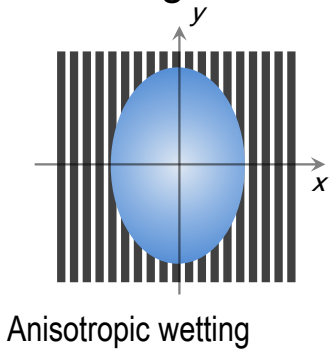


(2) Microstructures (Microscopic)

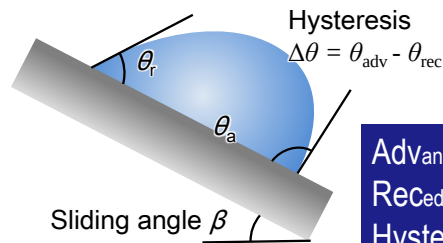


(1) Grooves (Macroscopic)

3D wetting



Dynamics of wetting



! Eqs. Wenzel or Cassie-Baxter cannot explain

$$\gamma^{LG}\cos\theta + \gamma^{LS} = \gamma^{SG}$$

$$\cos\theta_{cb} = f\cos\theta + f - 1$$

Thermodynamic contact analysis

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Thermodynamic contact analysis

Calculation of free energy

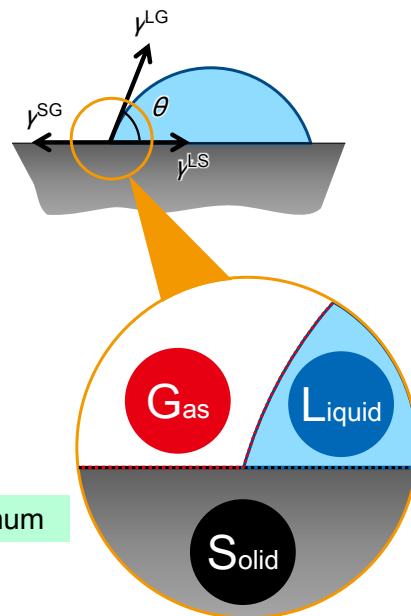
Surface tension γ :
Helmholtz free energy F for a unit area

$$\gamma = \left(\frac{\partial F}{\partial A} \right)_{T,V}$$

Three boundaries ▶ Total free energy F

$$F = (\gamma^{LS} \times A^{LS}) + (\gamma^{SG} \times A^{SG}) + (\gamma^{LG} \times A^{LG})$$

Find out the point where F becomes the minimum



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Machining

Ultrasonic cutting of microtextures for anisotropic wetting

Analysis

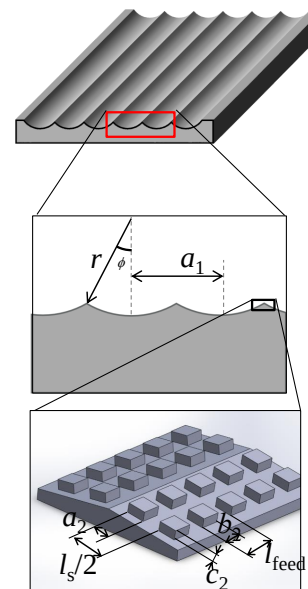
Thermodynamic contact analysis on expected surface textures

Evaluation

Comparing experimental and analytical results

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Modeling of analysis



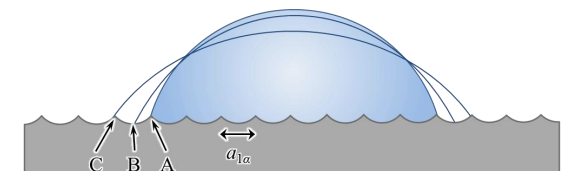
Simplification of model

Envelope of sinusoidal motion

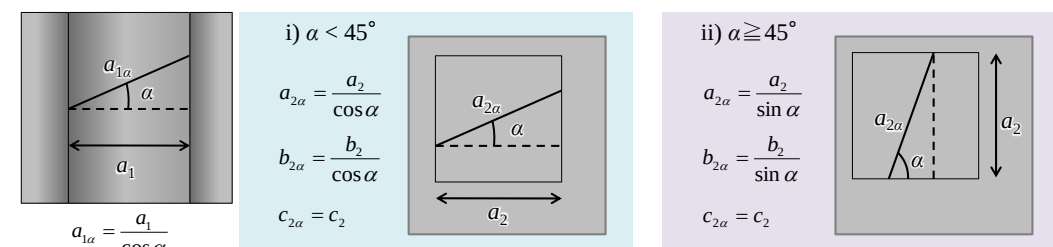
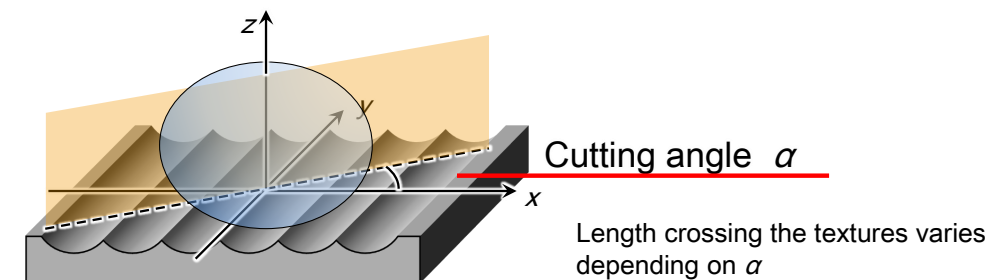
Approximation with line components

Constant volume

- Shape of droplet: spherical cap
- Cross-sectional area: constant
 - ▶ Figure out the directional dependency

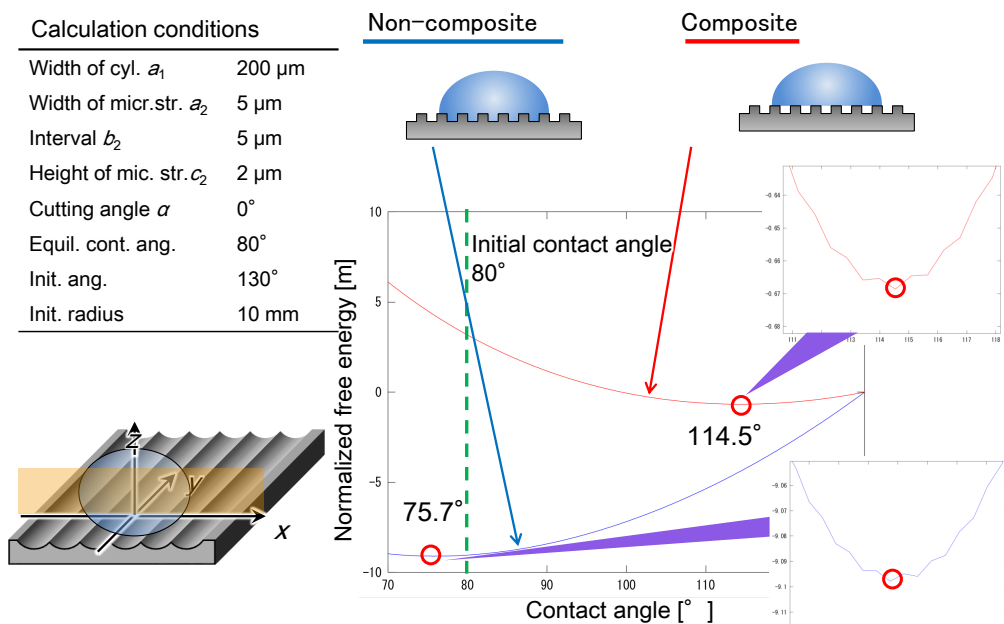


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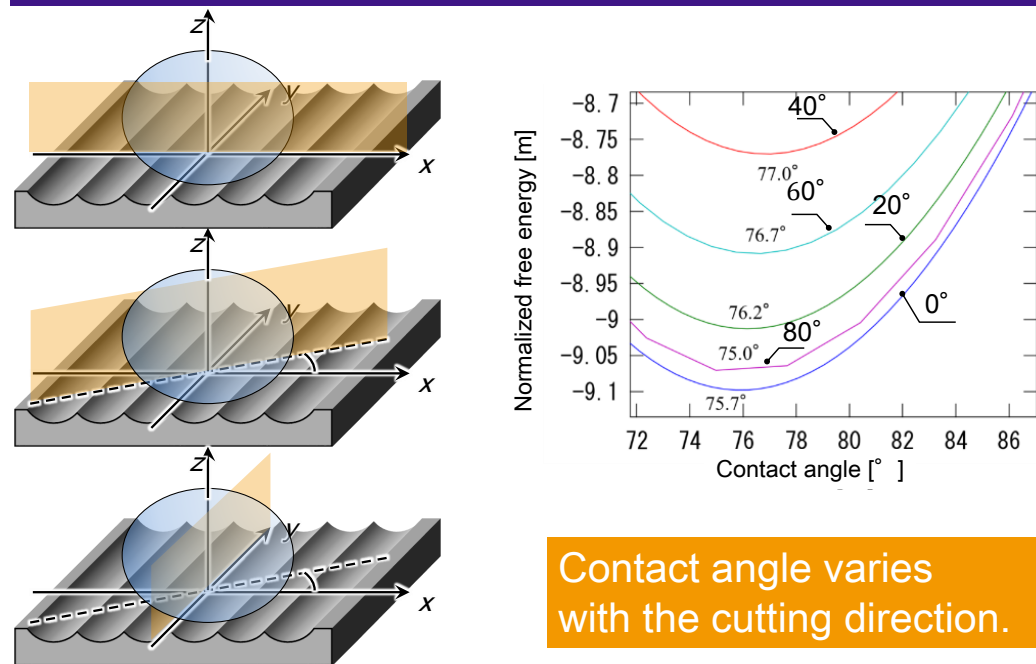
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Calculation result



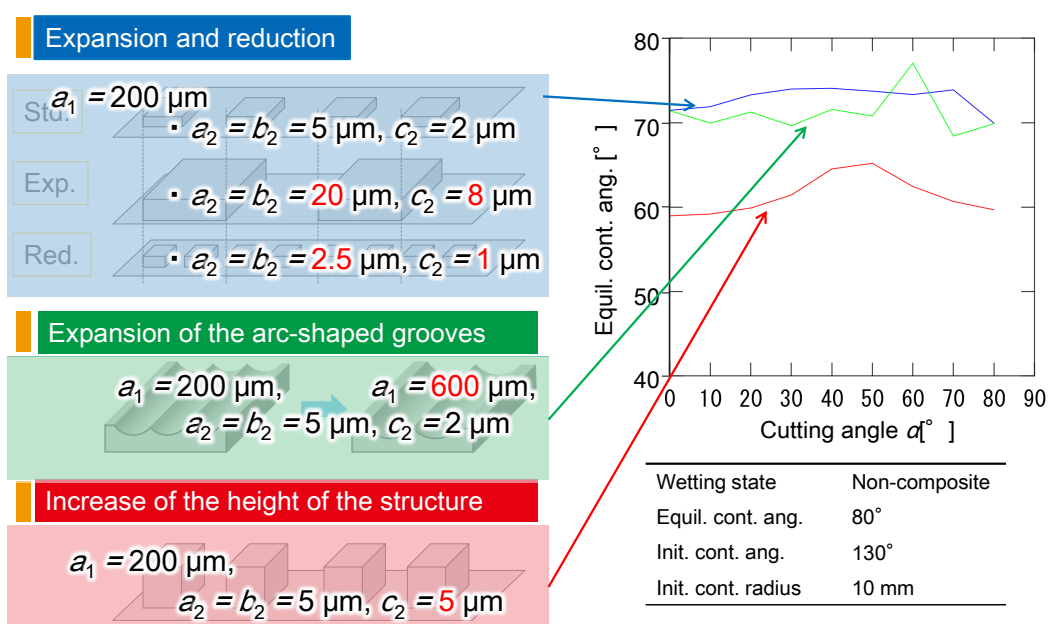
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Cutting angle of cross-section



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Size of structures



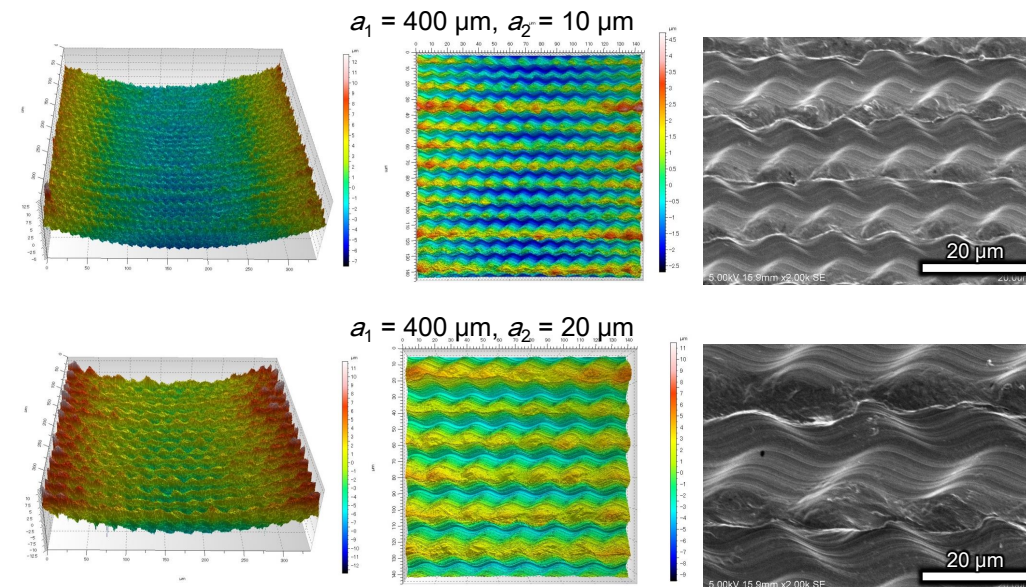
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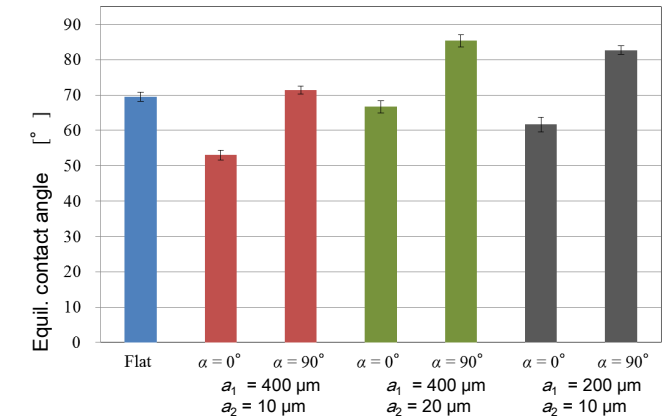
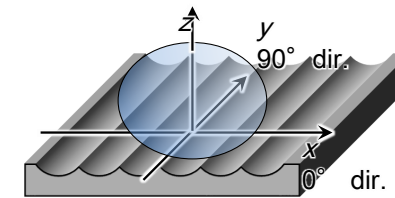
Rotation number n min ⁻¹	1500	3000	3000
Feed rate v mm/min	15	60	60
Frequency f kHz		25	
Amplitude A μ m	1.5	3	3
Surface protrusion a_2, b_2 μ m	10	20	20
Feed pitch a_1 μ m	400	400	200
Cutting depth d μ m		15	

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Images of white light interferometer



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Conclusions

Ultrasonic assisted cutting was applied to form microtextures, and anisotropic wetting was verified with thermodynamic contact analysis.

- ◆ Anisotropic wetting was confirmed experimentally.
- ◆ Calculation and experimental results agreed qualitatively; quantitatively, there is a big gap.
- ◆ The gap might come from the simulation model that cannot handle the composite and non-composite models simultaneously.

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Manufacturing of functional surface with preventing frost accretion effect

Sho Hasegawa
Keita Shimada
Masayoshi Mizutani
Tsunemoto Kuriyagawa

Grad. Sch. Eng., Tohoku Univ.
Grad. Sch. Eng., Tohoku Univ.
Grad. Sch. Eng., Tohoku Univ.
Grad. Sch. Biomed. Eng., Tohoku Univ.



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Outline

1. Background

2. Influence of the surface wettability on the frost growing

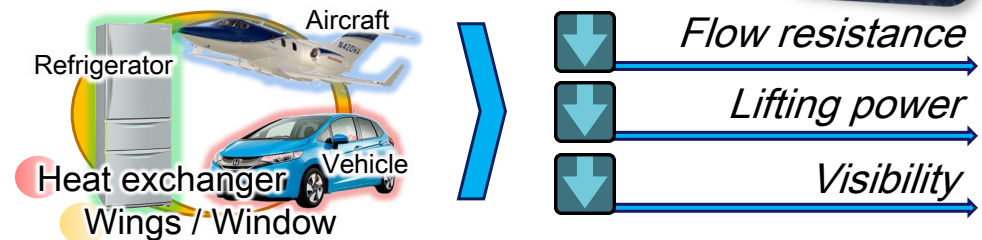
3. Influence of the microstructure on the frost growing

4. Conclusions

Problems of the frost

Frost-formation

- Heat exchange surface
- Cooling surface of cooler

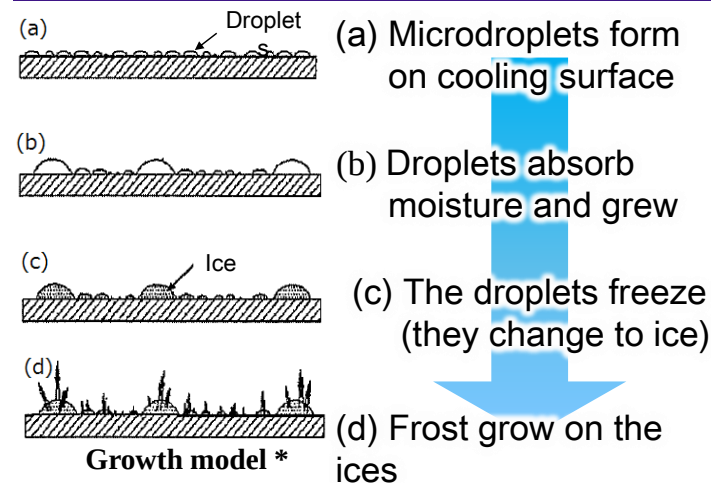


Traditional-defrost-methods



We require further development of **more efficient and economical** defrosting technology.

Frost growth model



! The droplets are indispensable to frost growth

Surface conditions affect the droplets

We focused on **Wettability** and **Microstructure**.

1. Background

2. Influence of the surface wettability on the frost growing

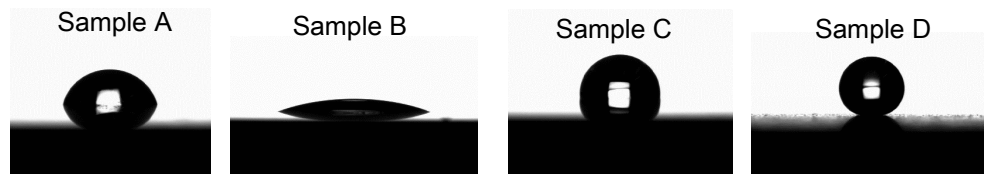
3. Influence of the microstructure on the frost growing

4. Conclusions

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Wettability effect

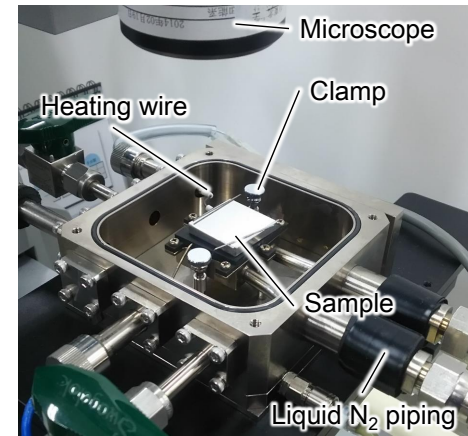
Tow kinds of experiments were conducted to confirm influence of the wettability on frost formation



	Material	Surface	Coating	Contact angle(°)
Sample A	AISI5052	Mirror finished	Non-coating	73.8°
Sample B			Hydrophilic coated	22.0°
Sample C			Hydrophobic coated	92.0°
Sample D			Super hydrophobic coated	159.3°

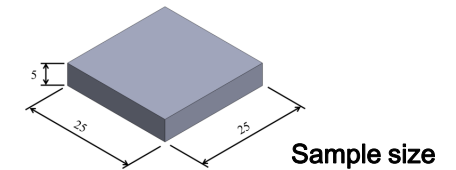
Evaluation items;

Droplet formation **Frost thickness** **Frost strength**



Observing system

Air temperature t_a	21.9°C
Relative humidity ϕ	19.4%
Cooling rate χ_w	-10°C/min
Initial setting temperature t_0	20°C
Target temperature t_w	-30°C
Cooling system	Heating wire, Liquid N ₂

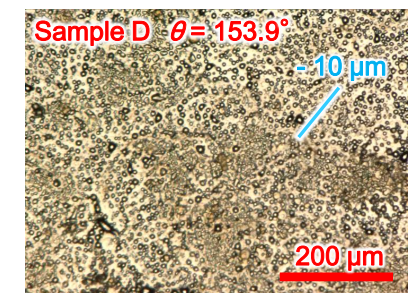
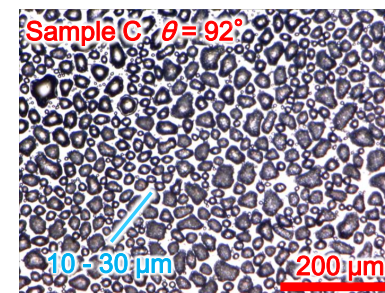
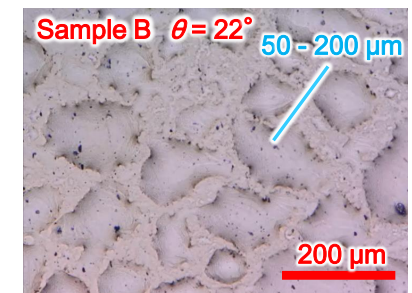
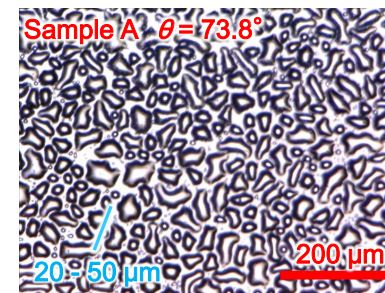


Sample size

Observing the frost on each sample and comparing the droplets formation and the frost thickness

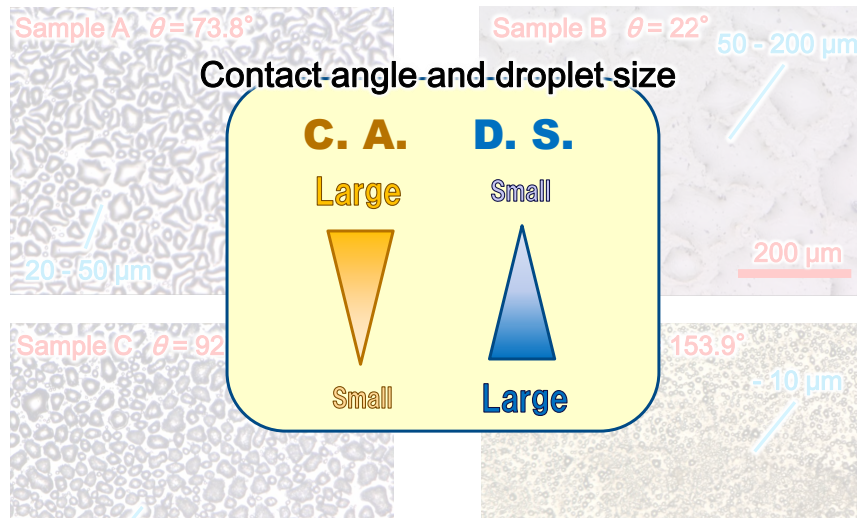
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The droplet before just freezing



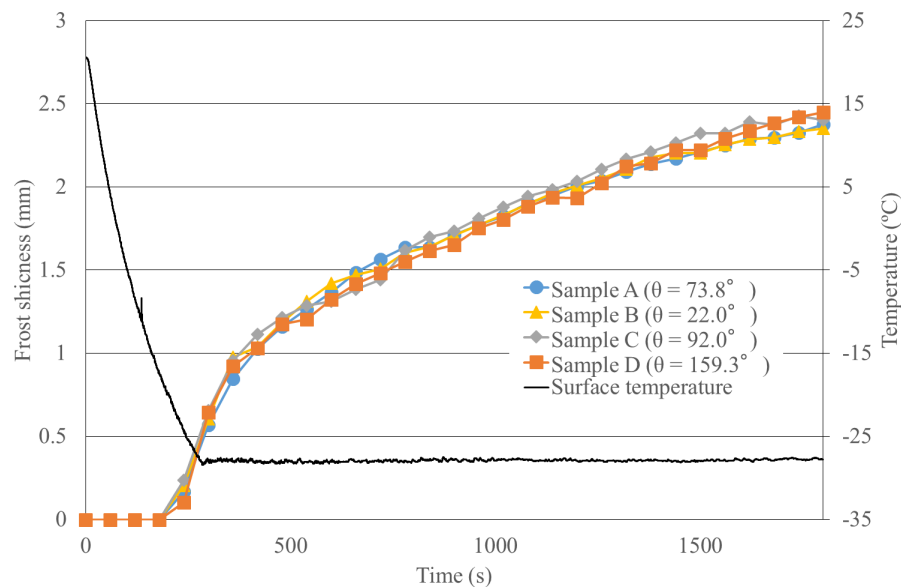
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The droplet before just before freezing



The size of droplet was larger as the surface got higher hydrophilicity

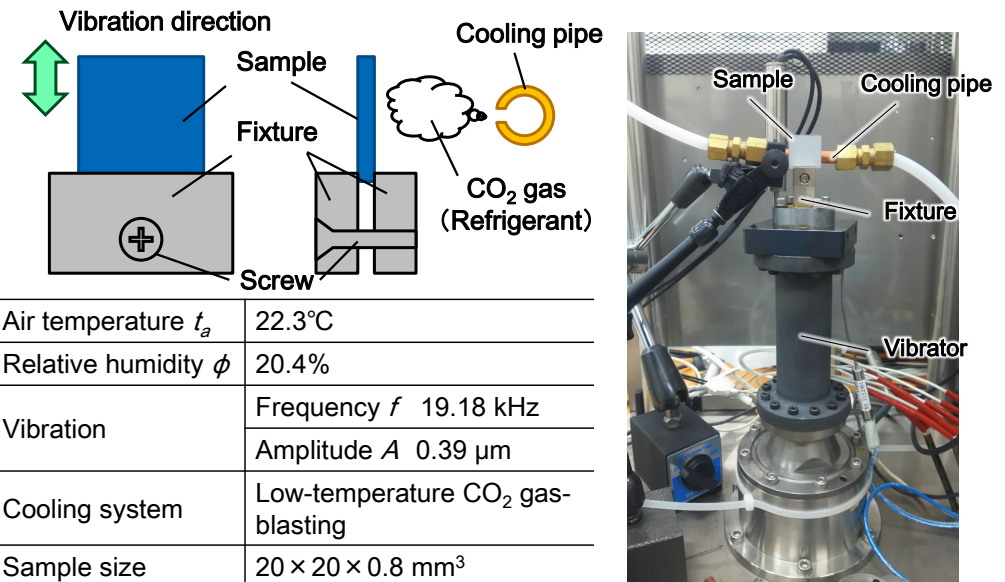
The frost thickness



Wettability did not affect the frost thickness

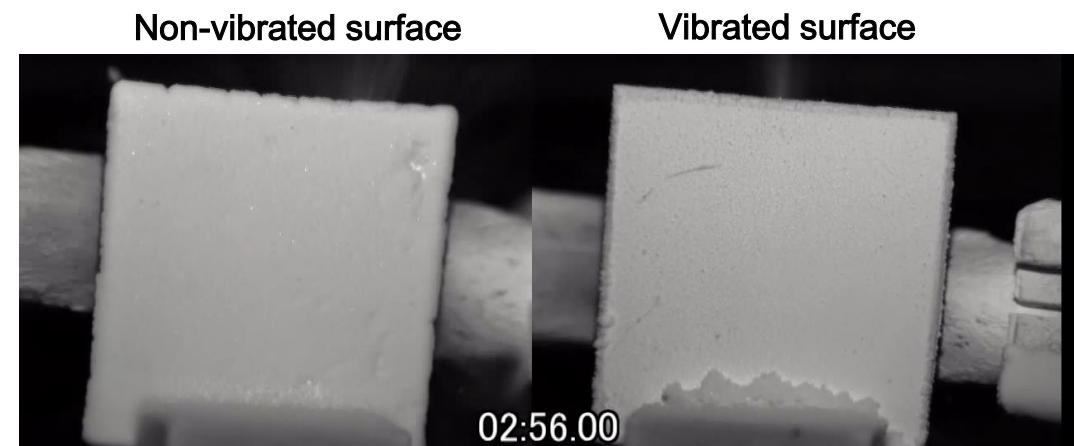
Vibration and cooling

Observation the vibrated cooling surface to evaluate frost strength

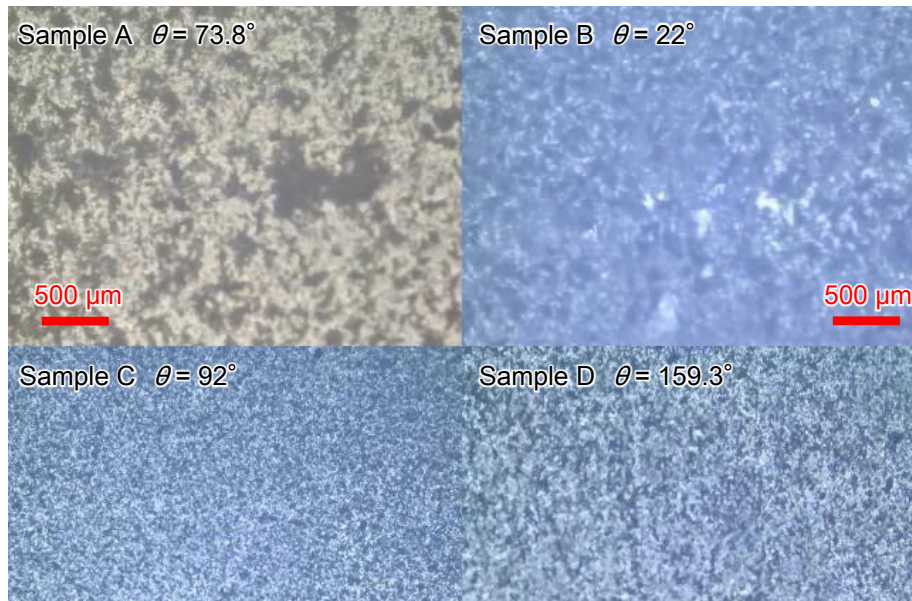


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The vibration broke the frost



Vibration had a capability of breaking frost



High hydrophobic promoted defrost effect with vibration

Wettability affects the frost formation

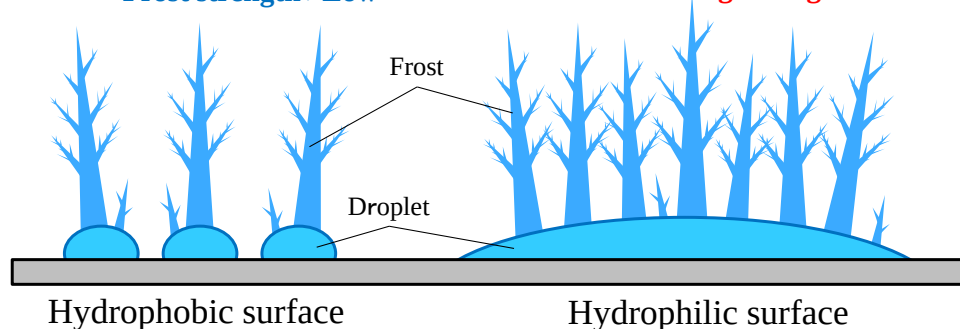
The frost formation depends on the water droplet size.

- Small droplets were formed on the hydrophobic surface.
- On the other hand, large droplets were formed on the hydrophilic surface.

It has been confirmed that **the frost had high density on the hydrophilic surface.****

Frost density: Low
Frost strength: Low

Frost density: High
Frost strength: High



The hydrophobic surface promoted defrost effect

1. Background

2. Influence of the surface wettability on the frost growing

3. Influence of the microstructure on the frost growing

4. Conclusions

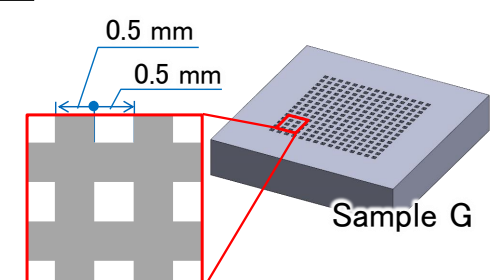
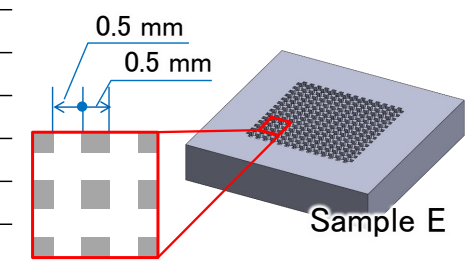
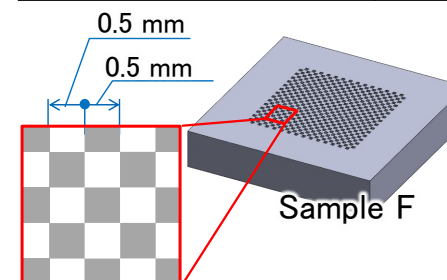
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Influence of the micro-structure on the frost growing

Observation the frost on the microstructures

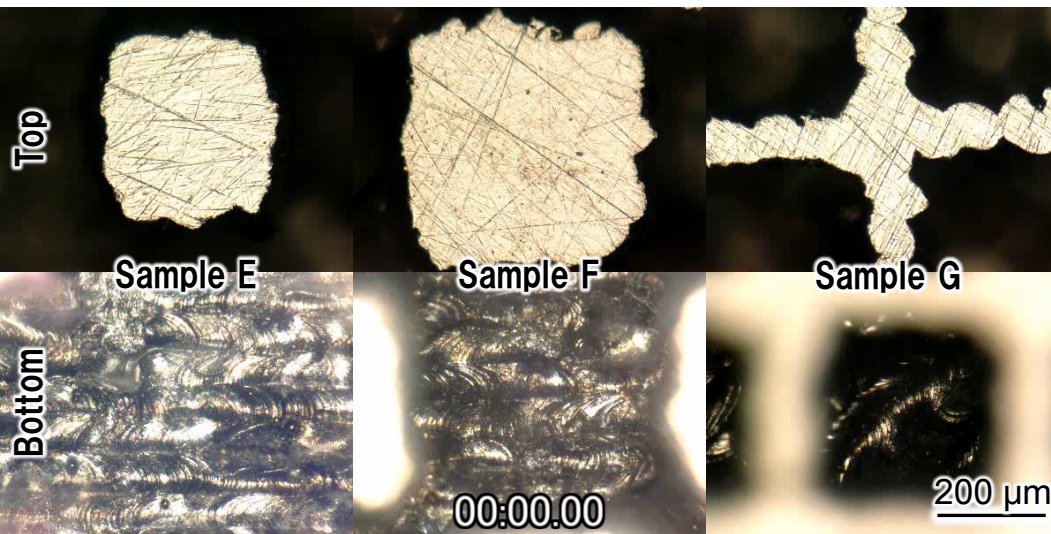
Creating microstructures using a metal 3D printer and comparison frosts of each

Material	AISI S17400
Air temperature t_a	23.6°C
Relative humidity ϕ	49.7%
Cooling rate χ_w	-10°C/min
Initial setting temperature t_0	20°C
Target temperature t_w	-30°C



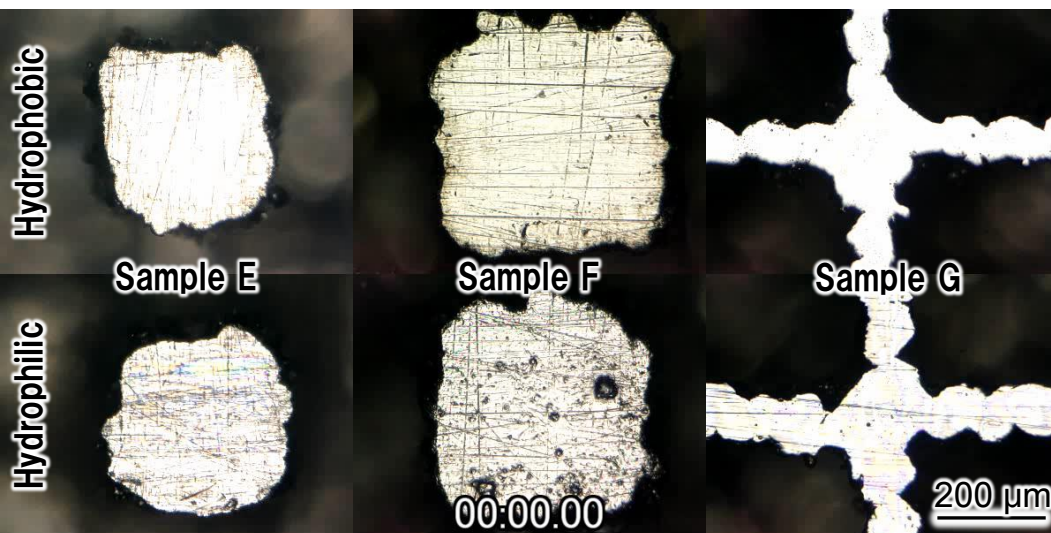
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Top and bottom of the structures



The frost arose intensively at the tops of the structures but there was little frost at the bottoms of the structures

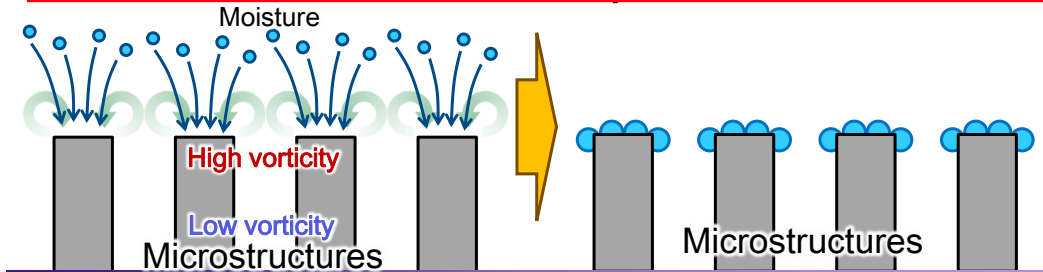
The coated structures



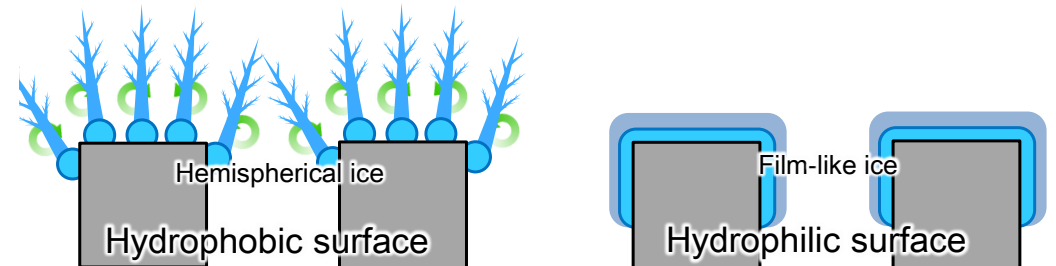
Hydrophobic surface → Columnar shaped ice
Hydrophilic surface → Film-like ice

Discussion

The frost concentrated on the tops of the structures



Observation the frost on the microstructures



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Conclusion

Manufacturing of functional surface with preventing frost accretion effect

The wettability of the surface affects the shape of the water droplet as the starting point of the frost, and the frost arises with low density and low strength on the hydrophobic surface.

Water droplet and frost concentrate in the vertices of the microstructure because the structure promotes moisture supply to the droplets and frosts caused by high vorticity occurrence.

Combining the microstructure and wettability makes it possible to manipulate the frost occurrence location and its growth pattern.

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超音波援用ツルージング/研削における 最大砥粒切込み深さ導出のシミュレーションと砥石の形状創成

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Keita SHIMADA, Ryoichi Nagamatsu, Masayoshi MIZUTANI and Tsunemoto KURIYAGAWA

Key words: ultrasonic-assisted cutting, Maximum grain depth of cut, Monte Carlo method

1. 緒言

研削加工への超音波振動の援用は研削抵抗の低減や加工面粗さの向上の効果が報告された有用な手法であることが知られている¹⁾。研削における重要な因子の1つである砥粒最大切込量 g_m は砥粒1個あたりに作用する負荷を示し、砥石摩耗の形態を推定するために用いることができる。本報では、先行研究²⁾を参考としてモンテカルロ法 (Monte Carlo method, 以下 MC 法) を用いたシミュレーションを行い、超音波円振動を援用した場合を含めた g_m についての導出方法についての検討を報告する。

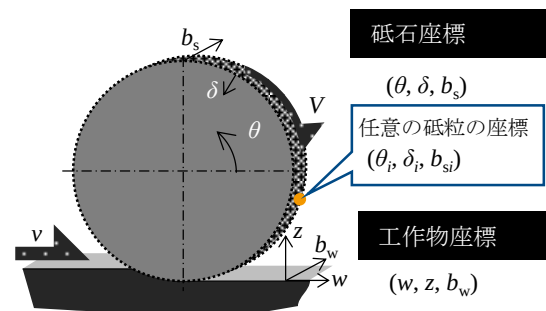


図1 研削モデル

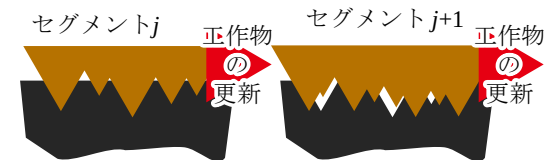
2. 超音波援用研削における最大砥粒切込み深さの解析

2.1 モンテカルロ法を用いた計算概要

図1に一般的な研削モデルを示す。研削加工の理論計算は砥石座標系 Σ_s に含まれる砥粒の座標を工作物座標 Σ_w へ移す写像から考えることができる³⁾。切れ刃の軌跡通りに材料が除去されるという単純なモデルであれば、研削面粗さを考える場合は作用順序については考慮しなくてもよく、各砥粒の Σ_s から Σ_w への写像を取り、包絡線を繋ぎ合わせることで計算できる。これは確率密度の写像としても計算できる³⁾。最大砥粒切込み深さを求める場合も Σ_s から Σ_w への写像を取ることと同様であるが、先行切れ刃が材料をどの深さまで除去しているかが重要となることから、順序性も考慮が必要となる。MC法を用いて単純に乱数によって砥粒を配置する場合、少なくとも砥石の最表面に存在する砥粒分はデータが必要であり、 θ 方向に番号を割り振り、図2(a)のように「…、砥粒*i*による加工 ▶ 工作物形状の更新 ▶ 砥粒*i+1*による加工 ▶ 工作物形状の更新、…」のような単純なループによる逐次計算を行うと計算時間が膨大になる。また、計算のために砥石および工作物の計算領域を薄片化すると、幅方向の影響を考慮できなくなる。そこで本研究では θ 方向を適当な区間に分け(以下、この計算上の区分をセグメントと呼ぶ。セグメント砥石と混同なきよう注意されたい)、セグメント内での砥粒の位置関係により切削に作用する砥粒を選別し、図2(b)のようにセグメントとして作用する形状(作用要素)を求めて、まとめて工作物形状を更新す



(a) 砥粒ごと更新



(b) セグメントごと更新

図2 計算における工作物形状の更新モデル

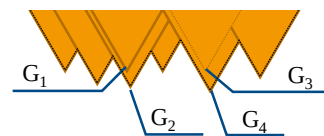


図3 セグメント作用要素の作製と重複の問題

る手法を提案する。図3はセグメント作用要素についての模式図である。作用要素はセグメント内に乱数によって配置した砥粒の先端点を繋ぐことで作成する。本手法の利点は b_w 方向で干渉しあわない切れ刃の作用の並列処理を可能として合理化することであるが、一方で、セグメントの θ 方向幅を大きく取りすぎるとセグメント内での重複する確率が大きくなり、本来は有効に作用するはずの砥粒を無効と判別する確率が増えてしまう。すなわち図3の例では G_1 は G_2 の先行切れ刃となり G_2 の切込み深さを軽減する働きをするはずであったが、この作

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用要素化では無視されてしまう。なお、 G_3 は G_4 の後続切れ刃であることを表しており、 G_4 はセグメントの厚さに関わらず無効である。

2.2 セグメント数の検証

まずセグメント数について検証を行った。表1の計算1がセグメント数検証の条件であり、その結果が図4である。上述の効果のため、セグメント数の増加に対して g_m が計算上減少し、100程度で一定となることが分かる。そこで以降の計算は100に固定し計算を行った。

2.3 超音波援用時の最大砥粒切込み深さ

超音波援用による g_m への効果については振動による幾何学的軌跡を考慮することで求めることができる。表1の計算2の条件で計算した結果を図5に示す。同図が示すように g_m は超音波援用にともなって2倍程度増加している。したがって、目こぼれ型の摩耗を促進することが予想される。一方、ツルレーイングに使用した場合には掘り起こし効果が促進され効率が促進される事が考えられる。

3. ツルレーイング実験

続いて表1の条件で超音波援用と非援用の両条件で軸付き砥石に対して単石ドレッサによるツルレーイング実験を行った。実験では最初に $2\mu\text{m}$ の切込みでツルレーイングを行い、その後ゼロカットを繰り返し、その際の抵抗を計測し、比較することにより実際除去量を推定した。実験結果を図6に示す。横軸0が設定切込み $2\mu\text{m}$ に対応し、その後0カットの回数を増加させている。同図より超音波援用ツルレーイングでは8回程度でツルレーイングが完了した一方、通常のツルレーイングでは15回後も除去が完了していない。これは図5に示すように超音波援用により g_m が増大することにより、砥粒に作用する効果が増大したことが影響していると考えられる。ただし、超音波援用自体に抵抗低減と効率化の効果があるため、今後 g_m についての検証を行う。

4. 結 言

本研究では超音波援用時の最大砥粒切込み深さを求めるモンテカルロ法によるシミュレーション法とツルレーイング実験での超音波援用の効果を検証した。シミュレーションでは多数の砥粒データを取り扱うため計算領域をセグメント化して作用させることにより並列で計算させることを可能とした。ツルレーイング実験では超音波援用により所望の除去量を速やかに得られることを確認した。

5. 参考文献

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- 3) 嶋田ら, 統計的手法による超音波援用研削における研削面粗さの理論解析, 砥粒加工学会誌, 56, 11(2012)7521-757

表1 計算条件

変数		計算1	計算2
砥石	直径 mm		8
	幅 mm		0.1
	砥粒集中度		100
	砥粒番手		3000
超音波振動	振動数 kHz	0	0, 20
	振幅 μm	0	2
研削条件	送り速度 mm/min	50	6 - 100
	切込み深さ μm		1
	砥石回転数 rpm		3000
セグメント数	数	1-400	100
	砥粒数		10000

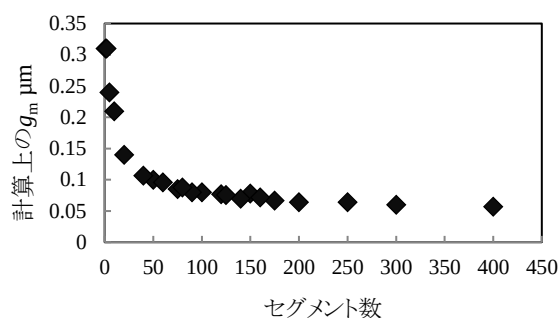


図4 セグメント数による計算上の g_m の変化

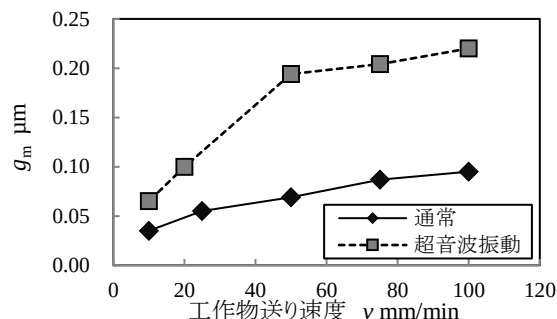


図5 超音波振動援用時の g_m の変化

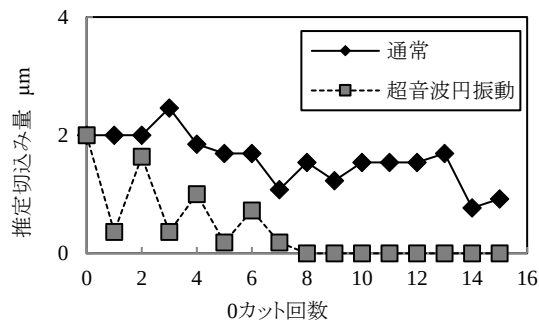


図6 超音波振動援用時の g_m の変化

パウダージェットデポジションによる歯科治療に関する研究

簾内崇彰^{*1}, 山本浩己^{*1}, 泉田一賢^{*2}, 佐々木啓一^{*2}, 嶋田慶太^{*1}, 水谷正義^{*1}, 厨川常元^{*3}

Study on Dental Treatment by Powder Jet Deposition

Takaaki SUNOUCHI, Hiroki YAMAMOTO, Kuniyuki IZUMITA, Keiichi SASAKI,
Keita SHIMADA, Masayoshi MIZUTANI, Tsunemoto KURIYAGAWA

Key words: fast particle impact, powder jet deposition, hydroxyapatite, dental treatment, composite particles

1. 結 言

歯科治療には、齲蝕治療のような機能的な側面と歯の色調のような審美的な側面があり、双方が共に心身の健康に対して重要な役割を担っている¹⁾。現在、金属やレジン、セラミックスなどの歯科材料による齲蝕治療は、修復箇所を経年劣化に伴う2次齲蝕のリスクが指摘されている。この問題を解決するため、著者らは常温大気圧環境下での高速粒子衝突による成膜法(パウダージェットデポジション, 以下 PJD)により、歯と同質のハイドロキシアパタイト(以下, HA)を人歯に直接成膜する治療法を提案している²⁾。これまでに、齲蝕治療を模擬した窩洞へのHA厚膜形成や口腔環境を模擬したサーマルリサイクル試験により、齲蝕治療部への成膜が可能であることを示されており、実用性の高い治療方法である³⁾。

一方、患者の歯科治療に対する高い要望のなかで、近年注目されているのが、口腔に関する審美性である。一般的な審美歯科の手法には、ベニヤ修復法や漂白法などが挙げられるが、修復材の接着不良や劇薬を用いることなどの問題が指摘されている⁴⁾。そのため、良好な密着性があり、人体に対し安全性の高いPJDを審美歯科に適用することで、機能性および審美性の両面を兼ね備えた革新的な歯科治療法を実現することが可能になる。

本研究ではPJDで作成したHA成膜面に対して、歯科材料として一般的に用いられているジルコニアと、審美的な色調の指標として作られたセラミックス製の歯模型であるシェードガイドとの色調評価の比較を行い、PJDによる審美歯科治療の可能性について実験的に検証した。

2. 測色試料用HAの成膜

成膜面の色調評価が目的であることから、基板として透過率が高く背景色の影響がないソーダガラスを採用し

表1 測色用試験片作成のための噴射条件

粒子	HA
粒径	2.2 μm
噴射距離	3.0 mm
供給圧力	0.5 MPa
噴射時間	30 s
基板	ソーダガラス
搬送流体	空気

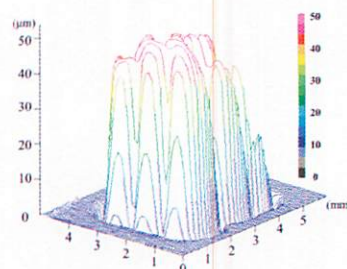


図1 HA成膜面の3次元プロファイル

表2 色調の測定条件

照射径	ϕ 3.0 mm
測定径	ϕ 1.6 mm
光源	ハロゲンランプ
測定時間	3.0 s
測定能力	390 ~ 730 nm

た。噴射条件は先行研究においてHA成膜に用いられた表1の条件を用いた⁴⁾。薄膜の幾何学的形状は色調評価に影響を及ぼすことが予想される。そこで、膜厚を一定にするために成膜面に対して研磨処理を施した。研磨後の表面性状を非接触3次元形状計測装置(三鷹光器, NH-3)により測定した。その結果、図1に示すようなHA粒子の成膜面を得た。膜頂点部のRaの値は約0.01 μm と滑らかな面であることが確認できるため、分光光度計を用いた測定に与える影響は少ないと考えられる。

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3. 色調評価

3.1 測色方法

成膜面の色調評価には分光測色方式に属した高速分光光度計 CMS-35FS (村上色彩研究所) を用いた。成膜実験で得られた HA 膜およびジルコニアは表 2 の条件で測色を行った。

3.2 シェードガイドの測色

審美性評価の基準として一般臨床の審美歯科で最も広く用いられている Lumin-Vaccum (VITA 社) (図 2) を使用し、日本人の平均的な歯の色調といわれるシェードガイド (以下, SG) 番号 A3.5⁵⁾ を測色対象とした。

一般的に人歯は切端部, 中央部, 歯頸部によって色調が異なり, SG も部位によって色調が異なっている。そのため, SG を 3 分割し, 切端部, 中央部, 歯頸部として, 各部位 5 回ずつ表 2 の条件で測色し, それぞれの平均を代表値とした。

3.3 測色結果

各試験片の分光分布を図 3 に示す。HA 膜の分光分布は, ほぼ一定であり, 反射率は 5% 程度と低いことから, 照射された白色光はほとんど反射せず透過もしくは吸収していることが確認できる。一方, SG の各部位は HA 膜に比べて, 高い値を示しており透明性が HA 膜に比べ低い。また, 中長波長領域の反射率が短波長領域に比べて高いことから, 黄色みがかかった色調であることが確認できる。ジルコニアの分光分布は, SG と同様に中長波長領域の反射率が短波長領域に比べて高く, 黄色みがかかった色調であることが確認できる。

次に, この試験片の色調の差を数値化し定量評価するために, 分光光度計から得られた CIE $L^*a^*b^*$ 表色系データ表 3 に示す。 L^* は明度を表す指標であり, 高いほど白色, 低いほど黒色を示す。 a^* , b^* は, それぞれ+〜が赤緑方向, 黄青方向を表している。これらの値を式(1)で表される色差公式に代入することで, 色差 ΔE_{ab}^* を算出した。

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

$$\Delta L^* = L_2^* - L_1^* \quad (2)$$

$$\Delta a^* = a_2^* - a_1^* \quad (3)$$

$$\Delta b^* = b_2^* - b_1^* \quad (4)$$

色差公式から, HA 膜と SG 中央部との色差 ΔE_{ab}^* の値は, 約 25.70, また, ジルコニアと SG 中央部との色差 ΔE_{ab}^* の値は, 約 32.83 であった。歯科領域では SG との色差の許容度として 3.3 を判定基準⁶⁾ としていることを踏まえると, 非常に大きい色差と判断できる。色差を近づけるためには, ΔL^* 値が最も大きいことを踏まえると, HA 膜はより白色化させ, ジルコニアはより明度を抑える必要があると考えられる。

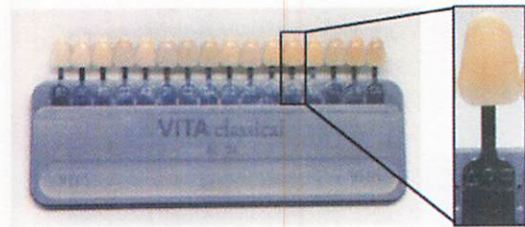


図 2 使用したシェードガイド

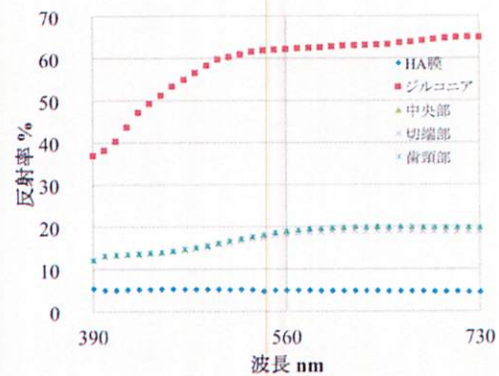


図 3 HA 膜およびシェードガイドの分光分布

表 3 各試料の測色値

	L^*	a^*	b^*
HA 膜	26.47	-0.085	-1.043
SG 中央部	50.06	-0.176	9.156
SG 歯頸部	49.86	-0.164	9.346
SG 切端部	49.19	-0.366	8.398
ジルコニア	82.75	-3.178	9.582

4. 結 言

本研究で得られた結言を以下に示す。

- HA 膜と歯科材料として一般的に用いられているジルコニアを審美性の指標となるシェードガイドとの色差を定量的に評価することが可能となった。
- PJD による審美歯科治療に適用させるには, HA 膜はより白色化させ, ジルコニアはより明度を抑える必要があると考えられる。

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ナノ秒パルスレーザ照射による ジルコニアインプラントの表面改質に関する基礎的検討

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Experimental investigation on surface modification of zirconia implant with nanosecond pulsed laser irradiation
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The aim of this study were to evaluate the microstructures and chemical composition change of tetragonal zirconia polycrystals (TZP) after nanosecond pulsed laser irradiation and to consider the effect on osteocompatibility. Specifically, ceria-stabilized TZP/alumina nanocomposite (Ce-TZP) was chosen in this study. Scanning electron microscopy revealed microgrooves, with the same widths of laser spot. Such cell-sized grooves are considered a favorable environment to promote the differentiation and promotion of osteoblasts. The surface blackened by the laser irradiation was then investigated with energy dispersive X-ray spectrometry and X-ray photoelectron spectroscopy to identify the chemical composition change. The results showed that oxygen atoms decreased on the irradiated surface and a part of Zr^{4+} shifted to lower side after irradiation. Considering these points, the generation of oxygen-deficient zirconia caused the color change. After laser irradiation, zeta potential was decreased. This result suggests the possibility that precipitation of ion and protein in body liquid can be suppressed. It is concluded that nanosecond pulsed laser can form the grooves to improve osteocompatibility on TZP surfaces, but it can occur adverse chemical composition change of oxygen deficiency.

1. 緒 言

近年, アレルギー惹起のリスクがなく, 十分な強度と審美性を有するという理由から, 部分安定化ジルコニア製歯科用インプラントに普及が進んでいる^{1,2)}. しかしながら, その骨適合性については, これを向上する効果的な表面改質の確立に至っていない³⁾. ジルコニアは硬質材料であるため物理的接触を加える改質が困難であることに加え, 化学的に非常に安定であるため化学反応を利用した改質も容易ではない²⁾.

そこで本研究では, ナノ秒パルスレーザ照射法による骨適合性の向上を狙う. 同手法では, 局所加熱により難削材に対して非接触に材料除去を行うことで精密微細加工が可能であり, 加えて照射部周囲に数マイクロメートル程度の熱影響層を付与できる. チタンなど金属材料に対し骨芽細胞の骨形成を活性化させる形状, およびハイドロキシアパタイト析出を向上させる組成を付与し, 相乗的に骨適合性の向上が可能であるとすでに報告されている³⁾. 本研究は, ジルコニアに対しても上記と同様な効果を獲得することを目的としている. セラミックスであるジルコニアは物性が金属とは大きく異なるため, レーザ照射が与える影響もまったく異なることが予想される. 本報では, 表面形状および表面組成の変化を確認し, 特に組成が骨適合性に及ぼす影響について検討および考察を行った.

2. 実験方法

材料はセリア添加部分安定型正方晶ジルコニア/アルミナ複合多結晶体 (Panasonic Healthcare 社製, CeO_2 10 mol%および Al_2O_3 30vol%含有, 以下 Ce-TZP) を用いた. 同材を 2 mm × 3 mm, 厚さ 1 mm の平板に機械加工し, 一方の端面に耐水研磨紙を用いて算術平均粗さ $Ra < 0.05 \mu m$ まで仕上げた. レーザ照射実験は, **Table 1**に示す条件において, 矩形整形した Nd: YAG レーザを基材上面に対し垂直に照射した. レーザ走査パスを**Fig.1**に示す.

Table 1 Laser irradiation conditions.

Pulse duration	3 ns
Pulse energy	150 μJ /pulse
Wavelength	1064 nm
Frequency	50 Hz
Feed speed	7 μm /s
Laser spot	Rect. 30 μm × 60 μm
Atmosphere	In air

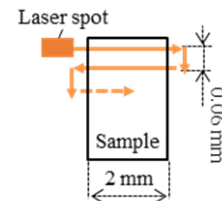


Fig.1 Laser scanning pass.

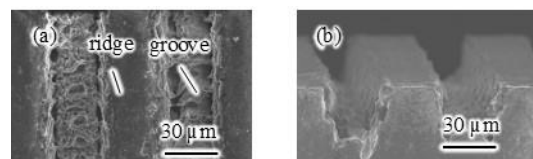


Fig.2 SEM images. (a) Laser-irradiated surface of Ce-TZP; (b) The cross-sectional face of Ce-TZP.

3. 実験結果および考察

3.1. 照射部の形状観察

レーザ照射面およびその断面の形状は走査型電子顕微鏡 (SEM) により観察した. 断面は試料をレーザ走査方向と垂直に切断し, 耐水研磨紙を用いて $Ra < 0.1 \mu m$ まで仕上げ作成した. 観察結果を**Fig.2**に示す. **Fig.2**より, レーザ照射によってスポット幅 30 μm と同程度の溝が形成されていることを確認した. 上記の大きさの微細凹凸の付与により, インプラント埋入時に骨芽細胞が溝に沿って配列し, 分化・増殖が良好になると考えられる.

3.2. レーザ照射による組成変化

レーザ照射後、Ce-TZP の照射面は黒く変色した。試料の外観を Fig.3 に示す。以下、対照群は研磨仕上げ後の Ce-TZP とする。Ce-TZP の黒色化は構成元素の組成変化に起因すると考え、エネルギー分散型 X 線分光 (EDX) による元素分析を行った。その結果を Fig.4 に示す。なお、レーザ照射前の酸素原子数濃度の平均値を 100% とし、レーザ照射後はそれに対する割合を百分率で示した。Fig.4 より、レーザ照射後、酸素原子数濃度が 30% 程度低下したことが確認できる。

EDX の結果に加え、レーザ照射ジルコニア表面の構成元素についてより詳細に検討するため、X 線光電子分光法 (XPS) による解析を行った。なお、線源は単色化 AlK α 線を使用し、帯電効果については C1s スペクトルを 285 eV で補正した。ここでは、特にレーザ照射前後で変化が生じた Zr3d スペクトルを Fig.5 に示す。また、参考値として Table 2 に酸化状態別の Zr3d 束縛エネルギー値を示す⁴⁾。レーザ照射前に確認された 182.0 eV, 184.3 eV のピークはそれぞれ Zr⁴⁺(3d_{5/2}), Zr⁴⁺(3d_{3/2}) に由来すると考えられる。レーザ照射後、ピークが低エネルギー側に移動したことが確認できる。また、Zr⁴⁺ピークの間が平に近づいたことから、価数の低い酸化状態となったジルコニアスペクトルの重複が生じていると考えられる。宗宮らによると、ジルコニア酸素原子が部分的に欠損した酸素欠損ジルコニア (ZrO_{2-x}) が黒色を呈すと考えられている⁵⁾。このことから、レーザ照射によりジルコニアの一部が還元され、酸素欠損ジルコニアが生成されたと推察される。

3.3. レーザ照射によるゼータ電位の変化

組成変化が骨適合性に及ぼす影響について調査するため、純水 (pH 6.3) におけるゼータ電位を測定した。Fig.6 にゼータ電位測定結果を示す。Fig.6 において、ゼータ電位絶対値がレーザ照射後に低下していることが確認できる。酸化物表面の帯電は、水の解離吸着により生じるヒドロキシ基の存在が寄与している。このことから、ゼータ電位絶対値の低下は、以下 2 つの理由が考えられる。1 つ目はレーザ照射により Ce-TZP 表面の酸素原子数が低下したことであり、2 つ目はジルコニア格子中の酸素イオンが酸素欠損により生じた空孔間を移動し、表面に導電性を発現させたことである。以上の理由から、水の解離吸着が阻害され、ゼータ電位絶対値が低下したと考えられる。ここで、体液中におけるイオンの吸着挙動を Fig.7 (a) に示す⁶⁾。ゼータ電位絶対値の低下に伴い、Ce-TZP 表面の静電引力も低下するため、Fig.7 (b) のようにイオンの吸着が減少し、生体組織析出に悪影響を及ぼすと示唆された。

4. 結 言

本研究では、Ce-TZP 表面にナノ秒パルスレーザを用いて微細凹凸形状を付与し、形状の観察、組成分析およびゼータ電位測定を行った。以下に結論を示す。

- (1) 矩形整形したナノ秒パルスレーザの照射により、スポット同程度の幅の微細凹凸を有する表面を創成可能である。
- (2) Ce-TZP のレーザ照射面には酸素欠損ジルコニアが生成され、黒色化する。
- (3) レーザ照射により、Ce-TZP の表面ゼータ電位絶対値は低下するため、生体組織析出に悪影響を及ぼすと示唆される。

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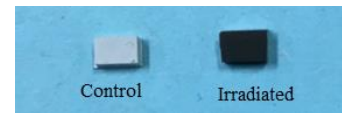


Fig.3 Photograph of laser irradiated Ce-TZP.

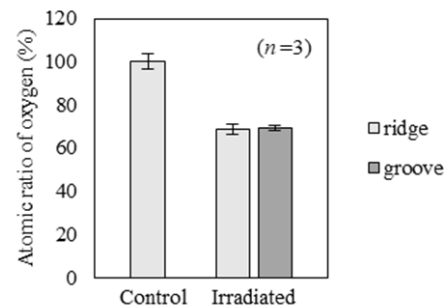


Fig.4 Atomic ratio of oxygen on the surfaces of Ce-TZP.

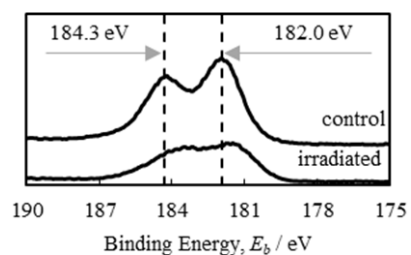


Fig.5 Core-level XPS spectra of Zr3d from Ce-TZP.

Table 2 Binding energy of Zr corrected with the C1s (285.1 eV) peak as a reference⁴⁾.

Oxidation state	Line	E_b / eV
Zr ⁴⁺	3d _{5/2}	182.2
	3d _{3/2}	184.5
Zr ²⁺	3d _{5/2}	181.0
	3d _{3/2}	183.3

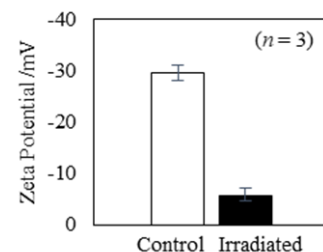


Fig.6 Zeta potential of Ce-TZP.

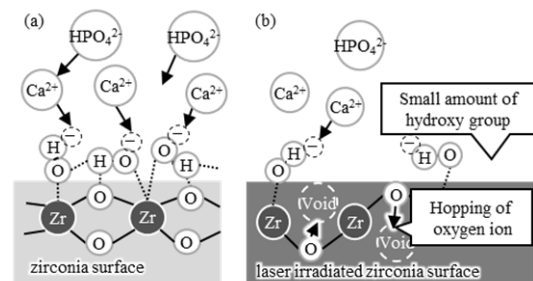


Fig.7 Deposition behavior of ions in body fluid. (a) Before laser irradiation; (b) After laser irradiation.

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レーザ照射によるチタン酸化皮膜の結晶構造制御に関する研究

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Study on crystal structure control of titanium oxide layer by laser irradiation

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歯科用インプラントに対して周囲組織の炎症や本体の脱落等の不具合をもたらす細菌感染を抑制することを目的とし、チタン表面へのレーザ照射により、抗菌性が期待される結晶構造を有する酸化皮膜を形成させる新たな手法を提案する。

1 緒 言

歯科用インプラントは欠損歯の問題解決の手段として需要が高まっている。一方、口腔内細菌によりインプラント埋入部が侵されるとインプラント周囲炎を発症し、インプラントが脱落に至る可能性がある。これを防ぐためには、歯科用インプラントの表面に細菌の付着・成長を抑制する抗菌性の付与が有効である。

現在、歯科用インプラントの材料としては主にチタン系材料が用いられている。同材は表面処理によって酸化皮膜を形成する。酸化チタンの結晶構造は、構成原子の配置の違いにより、アナターゼ型とルチル型に分類される。以下、アナターゼ型酸化チタンとルチル型酸化チタンを単にアナターゼおよびルチルと略す。アナターゼはバンドギャップが大きく触媒作用による抗菌性に優れると考えられており^[1]、チタン製インプラント表面にアナターゼを生成できれば、抗菌性の発現が期待できる。

アナターゼは熱力学的に準安定、ルチルは最安定であることから単純な熱酸化によるアナターゼの優先的な生成は容易ではない^[2]。現在アナターゼを生成する手法としては、陽極酸化法^[3]、ゾルゲル法^[4]、化学蒸着法^[5]などが提案されているが、これらの手法は薬品の使用や長時間の熱処理などが必要である。

そこで著者らはチタン表面にアナターゼを生成する処理法として、レーザを用いた手法を提案している。本研究では、まずアナターゼ構造の酸化皮膜を生成・制御するため、出力と走査幅を変えて純チタンに連続波発振 (CW) レーザを照射し、酸化膜厚と結晶構造の変化を評価した。さらにレーザ照射による表面の温度変化を解析することで、温度変化の観点から照射条件とアナターゼ生成の関係について検討した。

2 実験方法

供試材には#100 耐水研磨紙仕上げした $\phi 12 \text{ mm} \times t3 \text{ mm}$ の純チタン円板 (2 種) を用いた。試料の表面処理は、表1に示すレーザを用いて行った。また比較として表2の条件下で電気炉による大気雰囲気での加熱酸化を行った試料も準備した。処理後の試料表面はデジタルマイクロスコブ (DM) を用いて観察し、表面の結晶構造は X 線回折 (XRD) 分析により同定した。直接測定が困難なレーザ照射部の温度変化の評価には有限要素法による熱伝導解析を用いた。

3 実験結果および考察

3.1 照射後の試料観察

図1に示す DM 像より、レーザ照射による試料表面の変色を確認できる。この色調変化は、表面に生じた薄い酸化皮膜の存在による光の干渉が要因であると考えられる^[6]。また図2に示す電気炉で加熱酸化した試料表面からも同様の色調変化が確認された。

このような色調変化は酸化皮膜の厚さに依存することが知られており^[7]、電気炉加熱試料については温度の上昇にともなって膜厚が増加していることが確認された。レーザ照射試料について

表1 レーザ照射条件

発振方式	連続発振 (CW)
ビームプロファイル	ガウス分布
出力	25, 35, 49 W
走査幅	40, 80, 120, 160 μm
波長	1070 nm
走査速度	100 mm/s
スポット径	80 μm
雰囲気	常温大気圧

表2 加熱条件

昇温時間	1 h
保持温度	200°C, 400°C, 600°C, 800°C
保持時間	1 h
冷却	炉冷

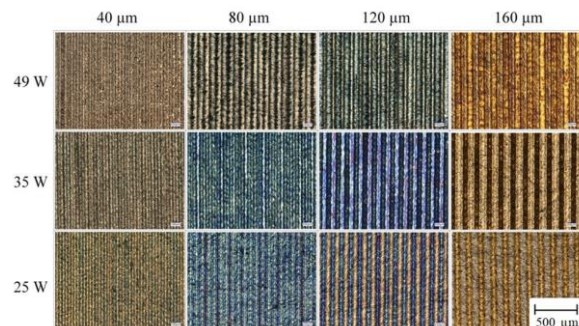


図1 レーザ照射面の観察結果

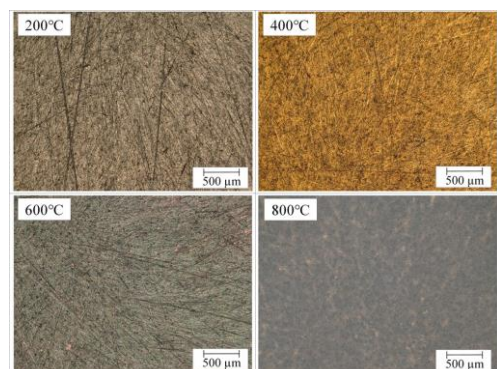


図2 電気炉での熱処理後の観察結果

は走査幅が小さくなる、あるいは出力が大きくなるほど膜厚が増加することが確認された。特に出力よりも走査幅に対する依存度の方が大きい傾向を示した。

電気炉加熱試料の温度と膜厚の関係性から、膜厚は試料表面へ

流入する熱量に依存することが推測される。これをレーザ照射試料で考えると、出力の増加は照射部への流入熱量を直接的に増大し、また走査幅の狭小化は隣接レーザ走査経路からの流入熱量を増大することから、膜厚増加に繋がったと考えられる。さらにレーザが通過する任意の点において、レーザ照射時の熱量の流入は瞬間的である、かつ発生した金属蒸気によりレーザのエネルギーが吸収され表面に到達する熱量が低下する可能性がある一方、隣接照射部からは重複して熱量が流入することから、膜厚は出力よりも走査幅に対して依存度が大きくなったと考えられる。

3.2 結晶構造の変化

レーザ照射と電気炉加熱を行った試料表面の XRD 分析結果を図3に示す。同図レーザ照射試料の回折パターン (a) (b) (c) より、走査幅 120 μm 以下のいずれの出力においてもアナターゼとルチルの両方のピークが確認された。なお、走査幅 160 μm のときも酸化皮膜は生成していると考えられるが、XRD の回折強度は存在量に比例するため、走査幅が大きく酸化皮膜の薄い条件では酸化チタンの顕著なピークが現れなかったと考えられる。

同図 (d) において、電気炉加熱では温度が上昇するにつれて酸化チタンのピークが多く現れ、ルチルの方がアナターゼよりもピーク数が多いことが確認された。これは、高温で保持するほど熱力学的に準安定なアナターゼよりも最安定なルチルが生じやすいためであると考えられる。

次に同図(a) - (c) に示すレーザ照射試料のアナターゼのピークに注目すると、出力が大きくなるほどピークが大きくなっていることがわかる。中村¹⁰⁾は、溶射法による酸化チタン皮膜創成の実験結果から、冷却速度が大きくなるほどアナターゼが形成しやすくなると報告している。また、安岡¹¹⁾は溶射法での冷却速度がルチルへの変態速度よりも短いことがアナターゼ生成に関係していると報告している。以上を踏まえると、レーザ照射による表面処理においても、チタン表面が急加熱・急冷却されるため、最安定なルチルだけでなく準安定のアナターゼが生成されたと考えられる。さらにアナターゼ生成量が出力に比例したことは、冷却速度の大きさに依存することが推測される。そこで次節では、各出力における基板の冷却速度を熱伝導解析により分析した。

3.3 有限要素法による熱伝導解析

有限要素法による熱伝導解析を用いて、レーザ照射部の温度変化について調査を行った。解析モデルは実際の試料寸法、照射条件をもとに作成し、試料中央の温度変化を解析した。なお、本報では走査幅 160 μm での照射の解析結果のみを図4に示す。レーザは 3.53 s 付近で試料中央を通過するが、その際レーザスポットの通過前後において急加熱・急冷却が生じていることから、各出力においてチタンの融点 (約 1670°C) を超えて瞬間的にチタンが熔融すると考えられる。解析結果から、温度が 3.53 s での最高点から凝固点付近まで直線的に冷却すると仮定し、直線変化する領域の温度差と経過時間から平均の冷却速度を求めた。求めた冷却速度を表3に示す。同表より、出力の増加に伴い冷却速度も増加することが確認できる。

以上より、レーザ出力とアナターゼ生成量との相関関係は、出力に依存する冷却速度と関係することが示唆された。

4 結 言

本研究ではレーザ照射によるアナターゼ生成の制御を試みるため、純チタンにレーザを照射し、表面の結晶構造解析と熱伝導解析を行った。以下に結論を示す。

- (1) CW ガウシアンレーザを出力・走査幅を変化させて照射することで、チタン表面に膜厚の異なる酸化皮膜の生成に成功した。
- (2) XRD 測定結果より、酸化膜が厚い試料ほどアナターゼとルチルがともに多く生成されることを確認した。

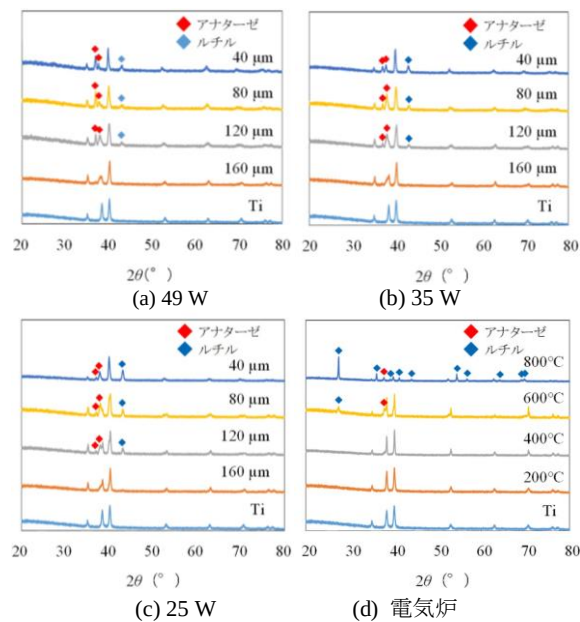


図3 XRD 測定結果

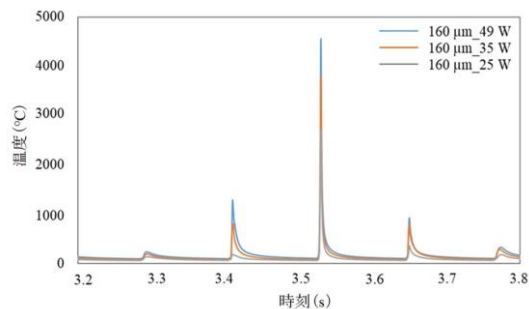


図4 熱伝導解析結果

表3 冷却速度	
出力 (W)	冷却速度 (°C/s)
49	1.58×10^6
35	1.38×10^6
25	0.96×10^6

- (3) 有限要素法によりレーザ照射面の温度変化を解析した結果、高出力であるほど冷却速度が大きいことが確認された。
- (4) XRD 測定結果と冷却速度の解析結果から、冷却速度を大きくすることでアナターゼを多く生成できると考えられる。

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高機能性インプラントの実現を目指した根状多孔質構造体の創成

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Creation of rhizoid porous structure aiming to realize the highly functional implant

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Porous metals are attracting attention for solving problems and improving functions of metal implants. Authors have proposed "rhizoid porous structure (RPS)" utilizing vacancy defects contained in an object produced by selective laser melting (SLM). In this research, we attempted to control the pore forms of RPS by manipulating shaping conditions. The result demonstrates that when the laser scanning is limited in a uniaxial direction, pores are oriented along the direction. Furthermore, the specimen with oriented pores have mechanical anisotropy. These results indicate that the proposed method can realize highly functional implants with mechanical functions equivalent to that of living bone.

1 緒言

各国で社会の高齢化が進行している現代において、高齢患者の健康寿命延伸や QOL 向上に有効な高機能性インプラントの開発は急務であり、その実現に向けて多孔質金属材料に注目が集まっている。インプラントの多孔質化により得られる恩恵としては、応力遮蔽効果の緩和¹⁾や、孔内骨成長による強固な骨結合の獲得²⁾などが挙げられる。現在では選択的レーザー溶融 (SLM: Selective Laser Melting) をはじめとした金属積層造形法 (MAM: Metal Additive Manufacturing) の発展により、多孔質構造体を高い形状設計自由度で作製することが可能となっている。しかし、多孔質構造体の幾何的設計には専用のソフトウェアを介した複雑な作業を要することに加えて、形状再現が可能な限界寸法を制約として加味する必要がある。したがって、設計領域の大きさに左右されず、かつ簡便な多孔質金属材料の作製手法が望まれる。

そこで著者らは、SLM 造形物内部に形成される空孔欠陥を活用した根性多孔質構造体 (RPS: Rhizoid Porous Structure) を提案している。本構造体は数十～数百 μm の空孔を有し、名称はその特徴的な連結孔の形状に由来する。先行研究ではレーザー走査速度による空孔率の制御に成功しており、機械的強度においても従来多孔質材に比べて高い値を得られている³⁾。最終的には、力学的あるいは機能的な事前設計から適切な造形条件を導出し、複数の機能性を相乗的に備えた高機能性インプラントをニアネットシェイプで作製することを目指す。

本報では、RPS の空孔形態をさらに高い自由度で制御するため、空孔の配向性に注目した。まず、造形物最上面の性状が空孔形成に与える影響を検討した。ここで、造形物最上面とは「レーザーによる溶融・固化が完了した最新の面」を指す。得られた結果から空孔の配向性制御手法を提案し、画像処理によって空孔配向角度の評価を行った。また、空孔の配向化による機械的異方性の発現を予想し、引張試験による機械特性評価を行った。

2 最上面の粗さと空孔率の関係

2.1 実験手法

試験片の造形には金属積層造形装置 ProX100 (3D Systems) を用いた。造形材料は Ti-6Al-4V 合金 (以下 64Ti) のガスアトマイズ粉体 (大阪チタニウム) を採用した。試験片は 7 mm 角の立方体とし、Table 1 に示す条件で造形を行った。本実験では造形物最上面の表面性状を変化させるため、2 種類のレーザー走査ストラテジを採用した。X¹Y¹ ストラテジでは層ごとにレーザー走査方向を 90° ずつ回転させ、レーザー走査速度 s は表中の 6 条件とした。X²Y² ス

Table 1 Building conditions

Spot diameter of laser	80 μm
Wave length	1070 nm
Transmission method	Continuous wave laser
Laser power	50 W
Layer thickness	45 μm
Laser scanning distance	70 μm
Atmosphere	Argon

Laser scan strategy and laser scan speed

X¹Y¹ strategy

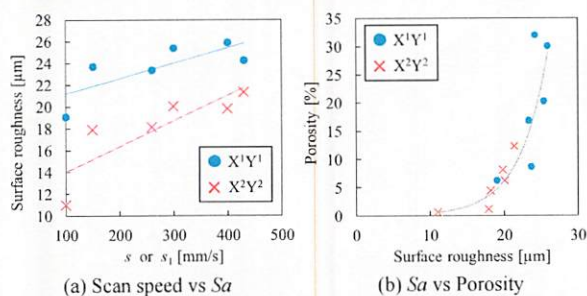


s [mm/s]	100, 150, 240, 300, 400, 430
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X²Y² strategy



s_1 [mm/s]	100, 150, 240, 300, 400, 430
s_2 [mm/s]	100



(a) Scan speed vs S_a
(b) S_a vs Porosity
Figure 1 Experimental result on roughness of top surface

トラテジでは各層において同じ経路で 2 回のレーザー走査を行い、1 回目の走査速度 s_1 は X¹Y¹ ストラテジと同様の 6 条件、2 回目の走査速度 s_2 は 100 mm/s とした。以上、各ストラテジにつき 6 条件、計 12 条件の試験片を得た。造形した試験片の最上面を形状解析レーザー顕微鏡 VK-X1000 (KEYENCE) を用いて観察し、得られた 3 次元形状データから算術平均高さ S_a を算出した。さらに、試験片の体積空孔率を X 線 CT スキャンにて算出した。

2.2 結果および考察

各試験片の走査速度と表面粗さの関係を Figure 1 (a) に示す。同図より、走査速度の低下および重複走査によって表面粗さが低

減する傾向が確認された。最上面の凹凸形状はメルトプールの溶融金属が表面張力によって凝集したことで形成されたと予想される⁴⁾。また、粗さの低減はメルトプールの温度上昇に伴った溶融 64Ti の表面張力の低下、およびレーザによる凹凸の再溶融に起因していると考えられる。

Figure 1 (b) より、最上面の粗さと空孔率との間には明確な正の相関が見られた。さらに Figure 2 に示した最上面の断面プロファイルより、粗さの大きい試験片では顕著に発達した凹凸形状が確認された。このような凹凸形状の谷部では、次に供給される粉体層の厚みが大きくなるため、深さ方向への溶け込み不足による未溶融領域の形成や金属蒸気の残留が生じ易くなり、空孔形成の起点になると考えられる。一方、低速度の試験片では深い谷部の形成が見られないことから、空孔の優先形成領域の減少に伴って空孔率が低減したと考えられる。

3 空孔の配向化と機械的異方性

前節より、レーザ走査によって形成される造形物最上面の幾何的形状が空孔の形態に影響を与えていることが示唆された。本実験ではその仮定を基に、空孔の配向性の制御を試みた。

3.1 実験手法

本実験で採用したレーザ走査ストラテジを Figure 3 に示す。走査方向を一軸方向に限定することで、最上面の凹凸とそれに付随して形成される空孔の配向化が予想される。走査速度は 240 mm/s とし、それ以外の造形条件は前節と同様とした。

空孔の配向性評価には画像処理手法を用いた。まず、X 線 CT によって得られた xy 断面の CT 画像を ImageJ により二値化し、評価の対象となる空孔を抽出した。次に、細線化処理によって空孔を幅が 1 pixel の線に近似し、各線の端点を結んで得られた線分の x 軸正方向となす角度を空孔配向角度 θ ($-90^\circ < \theta < 90^\circ$) と定義した。配向角度の算出は 1 枚の CT 画像中に存在する全空孔に対して行われ、最終的に 1 試料分の画像 880 枚分の値を積算することで配向角度のヒストグラムを作製した。

また、引張試験によって機械特性を評価し、機械的異方性について検討した。試験片は 2 種類とし、引張方向とレーザ走査方向が垂直なものを Vertical 材、平行なものを Horizontal 材とした。

3.2 結果および考察

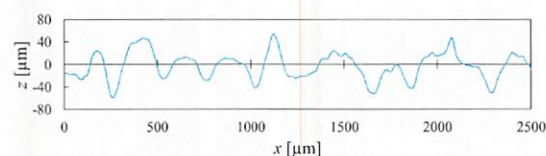
Figure 4 に、本実験で作製した試験片と、比較として同走査速度における X^1Y^1 ストラテジによる試験片の X 線 CT 画像 (二値化後) の一部を示す。同図より、 X^∞ ストラテジでは空孔がレーザ走査方向に沿って伸長している様子が確認できる。また、Figure 5 に示した配向角度ヒストグラムからも、レーザ走査方向と一致した方向への顕著な配向を確認することができた。なお、 X^1Y^1 ストラテジの試験片では、CT 画像およびヒストグラムの両者において空孔の配向は認められなかった。

また、Vertical 材と Horizontal 材の引張強さはそれぞれ 278 [MPa]、368 [MPa] であり、両者の間には有意な差が確認された ($p < 0.05$)。Vertical 材では引張方向に対して垂直に伸長した空孔の存在比率が増大し、空孔端部での応力集中を起点とした開口形き裂進展による破壊が誘起されたために Horizontal 材に比べて強度が低下したと考えられる。

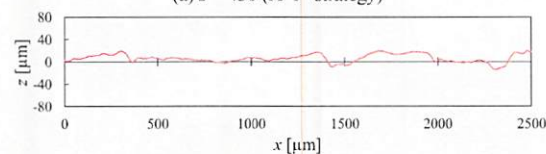
4 結 言

本研究から得られた結論を以下に示す。

- 1) 造形物最上面の粗さと空孔率の間には明確な正の相関が確認された。このことから、最上面の幾何的形状が RPS の空孔形成に支配的な影響を与えていることが示唆された。
- 2) 造形時のレーザ走査経路を一軸方向に限定することで、同方向に空孔が配向化することが分かった。また、空孔が配向



(a) $s = 430$ (X^1Y^1 strategy)



(b) $s_1 = 100, s_2 = 100$ (X^2Y^2 strategy)

Figure 2 Section profile of top surface

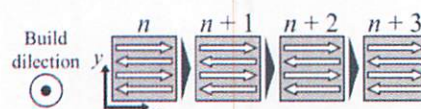
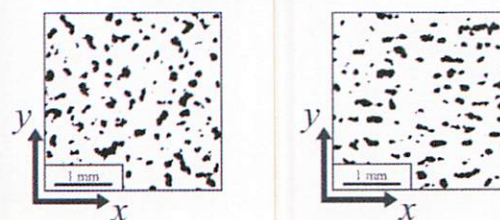


Figure 3 Schematic of X^∞ strategy



(a) X^1Y^1 strategy (b) X^∞ strategy

Figure 4 xy section CT images

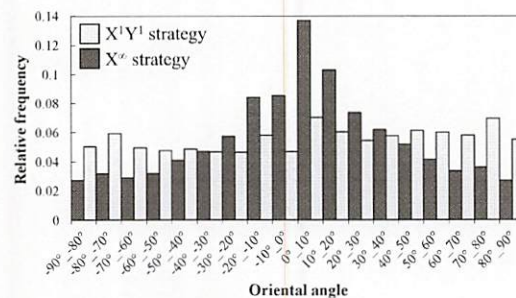


Figure 5 Histogram of orientation angle

化した試験片の引張強さは引張方向に依存して変化した。

以上より、レーザ走査ストラテジによって空孔の配向性を制御することが可能となった。同時に、骨と同様の機械的異方性を有するインプラント用骨代替材料の実現可能性が示された。

謝辞

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超精密平面ホーニング加工のための均一分散砥石の開発

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1. 序論

SiC や GaN をはじめとした次世代半導体基板材料はその材料特性や機能性に優れている反面、硬く脆い性質を持っており、高能率に平滑・平坦面を得ることが難しいとされており、これまでも各種の加工法・砥石を用いた研究が行われてきた。本研究で扱う平面ホーニング加工は、固定砥粒を用いた面接触研削加工であり、他の平面研削法と比較して同時作用砥粒数が非常に多いという特徴がある。そのため、個々の砥粒に作用する研削抵抗は小さく、加えて研削条痕もランダムな方向に入るため加工ダメージが小さい。従って、特に脆性材料を高能率に平坦・平滑に加工するには有利な加工法であるといえる。

2. 従来の平面ホーニング加工の課題

平面ホーニング加工に限らず、面接触式の研削加工では①加工点へ研削液が供給されにくい、②加工点から研削屑が排出されにくい、③滑り距離が非常に長くなり切れ刃が目つぶれを起こしやすい、④砥石を高速回転させた場合、研削液の動圧により工作物が浮上し加工レートが低下するといった種々の問題があり、これらを解決するために例えば①砥石を放射状パターンに配置する、②気孔率が高く結合度の低い砥石を用いるといったことで課題の解決が進められてきた。しかし、こういった複雑な形状配置や、特に微細砥粒を用いた高气孔率の砥石は特殊なビトリファイドボンドを用いることが多く製造コストが高い。加えて砥石摩耗も多くなるため、トータルの加工コストが下がらない課題がある。

本研究では微粒ダイヤモンドを砥粒とし、安価な水溶性フェノール樹脂を結合剤とした有気孔のレジンド砥石を用いて平面ホーニング用パターン砥石を製作し、加工コストの低減と加工品位の両立の可能性について検討した。

3. 製造コストに重きをおいた微粒均一分散砥石の製作

超平坦・平滑加工用の平面ホーニング加工用砥石として望まれる特性は、①砥粒径が $2\mu\text{m}$ 以下であること、②砥粒が均一に分散していること、③結合剤のブリッジ強度が低く均一であるこ

と、④砥石組織の剛性が高く弾性変位量が少ないことが挙げられる。さらに前述のように砥石製造コストを考慮し、水溶性フェノール樹脂・砥粒・硬質な充填剤を湿式で混合・造粒し、従来レジンド砥石のようなホットプレス成型することで図1のような組織の砥石を製作した。組織を観察すると、充填剤と砥粒の樹脂に対する濡れ性の違いから、砥粒は周囲に存在する充填剤によって締め付けられたように砥石内に固定されている。このため製造時、偶発的に砥粒凝集箇所が生じても、砥粒同士は結合していないため加工中すみやかに脱落し、凝集の影響を抑える効果が期待できる。また砥石パターンは先行研究で適とされた図2のような放射状とした。

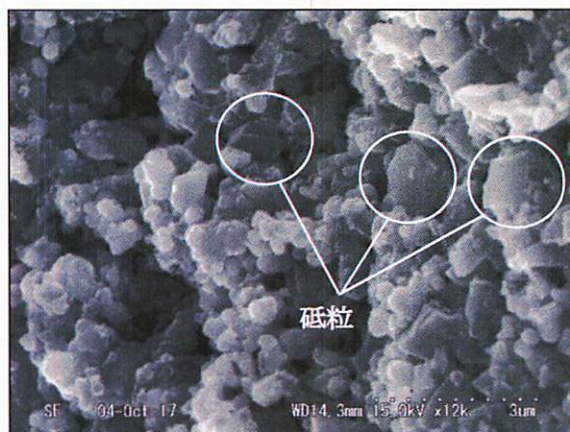


図1. 製作した微粒レジンド砥石組織 (SD10000)



図2. 放射状砥石パターン

4. インプロセスドレス用砥石の製作

実験に用いたナガセインテグレックス社製平面ホーニング盤 MPG-350 は、加工中にドレッサー砥石によるインプロセスドレスが可能な装置であり、これを行うと、常にホーニング砥石の平坦をドレッサーで調整できることや、砥石最表面が常に一定量除去されるため目詰まりや目潰れを起こさないといったメリットがある。一方でドレッサー砥粒の巻き込みによるスクラッチ発生の可能性や、加工条件によってはホーニング砥石の過度な摩耗を生じる恐れがあるため、ホーニング砥石よりも一段細かい GC 砥粒を主とした極低結合度ドレッサー砥石を新たに製作し、インプロセスドレスに用いた。ドレッサー砥石においても GC 砥粒の凝集発生を避けるため、前述ホーニング砥石と同様の製法・組織とした。

5. 加工試験結果

製作したホーニング砥石及びドレッサー砥石を用いて 4 インチ単結晶 SiC 基板の加工を行った。加工条件を表 1 に、加工後基板の状態を図 3 及び図 4 に示す。基板表面粗さの測定には TaylorHobson 製白色干渉顕微鏡 Talysurf CCI HD を用いた。ホーニング砥石の平均砥粒径 1.0 μm 、ドレッサー砥石平均砥粒径 0.5 μm の組み合わせでは加工後面粗さ Ra1.4nm となり、また加工面性状は平面ホーニング加工のモデルに近いあやめ模様の研削面が得られた。

6. 結論

本研究では硬脆材料の高効率平坦・平滑化に有効とされる平面ホーニング加工において、製造コストに優れるレジンボンドを用いて微細粒砥石を製作し、単結晶 SiC 基板の加工を行った。その結果、レジンボンドであっても微細砥粒で有効に研削が行えることを確認し、硬脆材料の加工コスト低減の可能性を見出した。

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表 1. 平面ホーニング加工条件

加工条件	
ワーク	4" 単結晶 SiC(Si 面)
前加工面	SD1000M 研削面
砥石回転数[rpm]	150
ワーク回転数[rpm]	150
ツルア回転数[rpm]	150
ワーク送り[um/min]	3
ツルア送り[um/min]	3
定圧制御荷重[N]	20

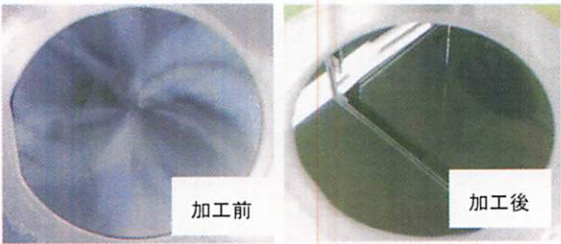


図 3. 加工前後の SiC 基板外観

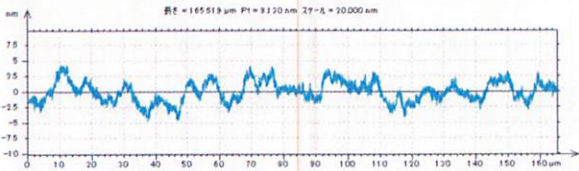
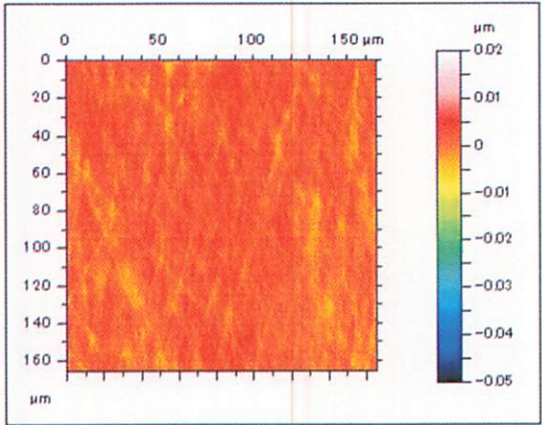


図 4 SiC 加工面 (Ra1.4nm Rz 7.4nm)

ハイブリッドテクスチャリングに関する研究

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Study on Hybrid Texturing

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Key words : hybrid texturing, plasma-shot, LIPSS

1. 緒言

表面処理は材料の硬度, 耐食性, 摩擦・摩耗特性などを向上させる手法である. その中で, プラズマショット(PS)法は電極・基材間のパルス放電により, 電極材料を熔融し基材表面に移着させ, 改質層を形成する表面処理法である¹⁾. PS法では材料の溶け込みのよい改質面を形成できる反面, 放電加工と同様の凹凸形状は摩擦係数上昇や摩耗促進の原因となる. 一方で凸部のみを効率的に除去できればプラトー構造面として摩擦・摩耗特性の向上が期待できる.

そこで本研究では凸部除去のみならず, 更なる高機能化を目的として, パルスレーザ照射を試みる. パルスレーザは短い時間幅にエネルギーを集中させるため高ピークパワーであり, 熱損傷が起こる前に加工を行うことが可能である. また短パルスレーザ照射によりLIPSSと呼ばれる微細周期構造が創成されることが知られており, LIPSSにより摩擦・摩耗低減など表面の高機能化が報告されている.

本研究では, PS法により形成した改質面に対して, パルスレーザを照射し盛り上がり部を除去する手法とLIPSSを創成する手法の2手法を提案する.

2. 実験手法

PS法には型彫放電加工機を使用し, 基材である粉末ハイス鋼 (C: 2.0%, Si: 0.5%, Mn: 0.3%, Cr: 3.8%, Mo: 2.5%, V: 5.1%, W: 14.3%, Co: 11.0%) をプラス極性, TiC 電極をマイナス極性として基材表面と電極材間にアーク放電を発生させ改質を行った. 実験条件の詳細を表 1 に示す. その後, ナノ秒パルスレーザによる改質面の凸部の除去, およびピコ秒パルスレーザによる改質面上へのLIPSSの創成を試みた. 改質面の表面粗さと摩擦特性の関係を検討するために白色顕微鏡, 走査型電子顕微鏡 (SEM), 摩擦試験機を用いて, 観察と評価を行った. それぞれのレーザ照射条件を表 2 に示し, ガラスによる面摩擦試験条件を表 3 に示す.

3. 結果と考察

3. 1 プラズマショット法の効果

表 1 放電条件

無負荷電圧 V	320 V
パルス幅 τ	8 μ s
休止時間 T	256 μ s
放電電流 I_p	3, 21, 51 A
電極消耗量 V_e	0.1 mm

表 2 パルスレーザ照射条件

装置略称	ナノ秒レーザ	ピコ秒レーザ
レーザ強度/ フルエンス	9.25 μ J	0.39 J/cm ²
走査速度	1000 μ m/s	600 μ m/s
走査幅	50 μ m	92 μ m
周波数	400 kHz	50 Hz
パルス幅	10 ns	20 ps

表 3 摩擦試験条件

荷重	25, 50, 100, 200, 500 g
サイクル数	180 cpm
線速度	30 mm/s
往復幅	5 mm
測定時間	600 s

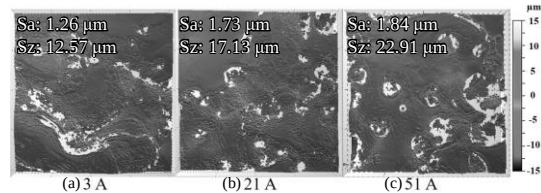


図 1 放電改質面のトポグラフィ

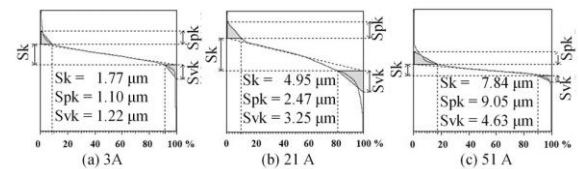


図 2 放電改質面の負荷曲線

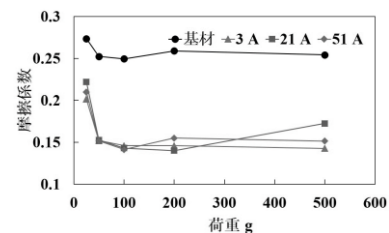


図 3 放電改質面の摩擦試験

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設定放電電流 $I_p = 3, 21, 51$ Aの放電改質面のトポグラフィを図1に、負荷曲線を図2に示す。これらの図に示すように、 I_p の増加に伴い表面粗さが増加していることが分かる。また図2から I_p の増加に伴い、突出山部高さSpkと突出谷部深さSvkのいずれも増加していることが分かる。さらに図3の摩擦試験結果から基材と比べ改質面の摩擦係数が低下しており、PS法により摩擦係数が改善されたと言える。

3. 2 プラズマショット法とナノ秒レーザ照射

次に $I_p = 51$ AのPS法による改質面に対しナノ秒パルスレーザを照射したSEM像を図4に、レーザ照射前後のトポグラフィと負荷曲線を図5に示す。図4より、ナノ秒パルスレーザの局所加熱によって改質面の様子が変化したことが確認できる。また図5と図2(c)の比較よりレーザ照射によるSpkの減少とSvkの増加、すなわち凸部が除去され熔融池が残存していることが確認された。また、図6のレーザ照射前後の摩擦試験結果より、25–200 gの軽負荷下において摩擦係数増加傾向の抑制が確認された。これは摩擦係数を上げる要因となる凸部がレーザ照射により除去され、残存した熔融池が油だまりとして作用したためであると考えられる。上記より、PS法による改質面へのナノ秒レーザ照射によりプラトー構造表面化することができ、表面の高機能化において有用であるといえる。

3. 2 プラズマショット法とピコ秒レーザ照射

図7は $I_p = 3, 21, 51$ Aの改質面に対してピコ秒パルスレーザを照射した際のSEM画像であり、盛り上がり部や熔融池内部にもLIPSSの形成が確認された。また盛り上がり部の周辺では構造が波立っており、LIPSSの構造が被照射体の形状に依存することを示唆している。

図8に基材、 $I_p = 3, 21, 51$ Aの改質面それぞれに対して、LIPSS創成前後を比較した摩擦試験結果を示す。図8(a)より、基材にLIPSSを創成した場合、摩擦係数が低減されたことが確認された。また図8(b)においても、荷重が100 gの時に摩擦係数が低下している。一方、図8(c)(d)から、 $I_p = 21$ Aおよび51 Aの改質面ではLIPSS照射前後での摩擦係数の変化がない、もしくは上昇が確認された。

摩擦係数の減少はLIPSSによる潤滑油の供給や動圧の発生作用によると考えられる。一方、LIPSSと比較してPS法による粗さは大きいことから、PS法の粗さが大きい場合には摩擦低減の効果が得られないことが考えられる。これらより、レーザ照射前の表面粗さの度合いによりLIPSSの発生パターンならびに摩擦特性に及ぼす影響が変化することが確認された。

4. 結 言

本研究では、放電改質面に対してナノ秒パルスレーザとピコ秒パルスレーザを照射することによって、更なる表面処理を施し、改質特性と摩擦特性を解明した。結言を以下に示す。

- (1) PS法により摩擦係数が減少するが、放電電流 I_p を増加させることで、粗さが増加することが確認された。
- (2) ナノ秒レーザを照射することで、PS法による改質面の盛り

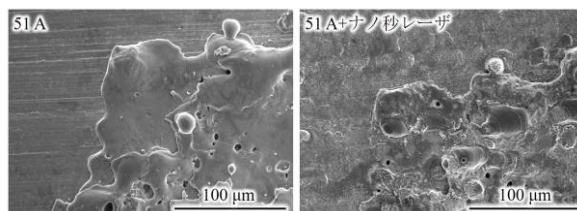


図4 51 A改質面へのナノ秒レーザ照射前後のSEM像

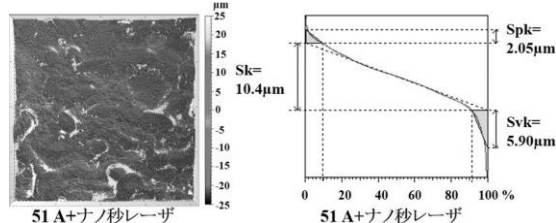
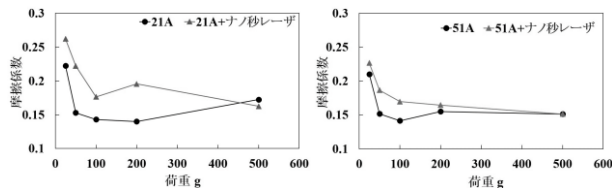


図5 51 A放電改質面レーザ照射後のトポグラフィと負荷曲線



(a) $I_p = 21$ A (b) $I_p = 51$ A
図6 ナノ秒レーザ照射前後の摩擦試験結果

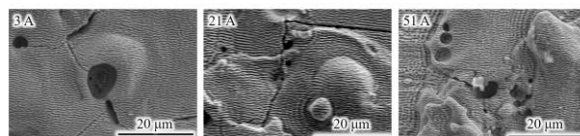
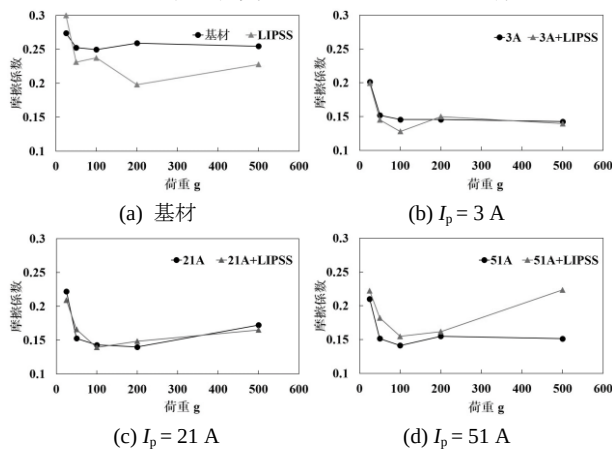


図7 放電改質面上のLIPSSのSEM像



(c) $I_p = 21$ A (d) $I_p = 51$ A
図8 LIPSS創成前後の摩擦試験結果

上がり部の除去が可能であり、摩擦特性が向上した。

- (3) ピコ秒パルスレーザによりPS法による改質面のような凹凸形状に対してもLIPSSの創成が可能であることが確認された。またLIPSSの摩擦低減効果は粗さの度合いにより変化することが確認された。

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金属積層造形法による根状多孔質構造体に関する研究

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Study on Rhizoid Porous Structure by Metal Additive Manufacturing

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Key words: additive manufacturing, selective laser melting

1. 緒言

多孔質金属材料は緻密材にはない特異な性質を有しており、軽量構造材料、断熱材、衝撃吸収材などとして多くの分野での活用が期待されている¹⁾。その作製手法はこれまでにいくつか提案されているが、全てに共通して重要視されるのは、空孔形態(空孔率、形状、孔径など)の制御性である。

著者らは、金属積層造形法の1種である選択的レーザー溶融(SLM: Selective Laser Melting)を利用し、造形条件の制御により一般的に欠陥とされる SLM 造形物内部の空孔を積極的に活用する新たな多孔質金属材料の作成手法を提案している²⁾。得られる構造体を根状多孔質構造体(RPS: Rhizoid Porous Structure)と称している。この名称は図1に示すような本構造の数十～数百マイクロンオーダーの特徴的な連結孔に由来する。先行研究は、造形条件のエネルギー密度および操作速度の変更により空孔率を任意に操作できることを明らかにした²⁾。さらに、SLMの極めて高い形状設計自由度と併せ、種々の機能性がデザインされた多孔質部材をニアネットシェイプで作製することを目指す。

本報では、SLMによる空孔制御における空孔率の制御可能範囲を把握するため、RPSにおける空孔率の上限値を検討した。また、造形したRPS試験片を用いて圧縮試験を実施し、その機械特性を評価した。

2. 造形条件と内部空孔の関係

2.1 実験手法

金属積層造形装置 ProX100 (3D Systems) を用いて Ti-6Al-4V 合金(以下 64Ti)製の SLM 試料(7mm 角立方体)をアルゴン雰囲気中で造形した。固定した造形条件はレーザースポット径 80 μ m、レーザー走査幅 70 μ m、積層厚 45 μ m であり、レーザー走査経路は層ごとに 90°ずつ回転させた。変更した条件はレーザー出力とレーザー走査速度であり、各パラメータから式(1)によって求められるエネルギー密度 E_d を表1にまとめる。なお後述するが、下線付き数値は造形不可であった条件をあらかじめ示している。

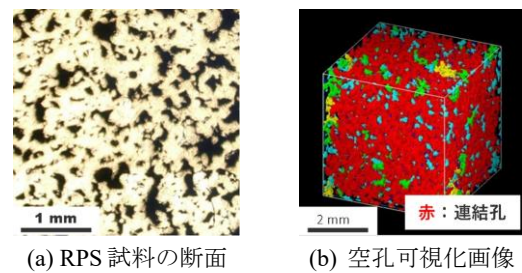


図1 RPSの内部空孔

表1 造形条件とエネルギー密度の関係

エネルギー密度 J/mm ³	走査速度 mm/s		
	430	500	550
出力 W	25	18.5	15.8
	30	22.1	19.0
	35	25.8	22.2
	40	29.5	25.4
	45	33.2	28.6

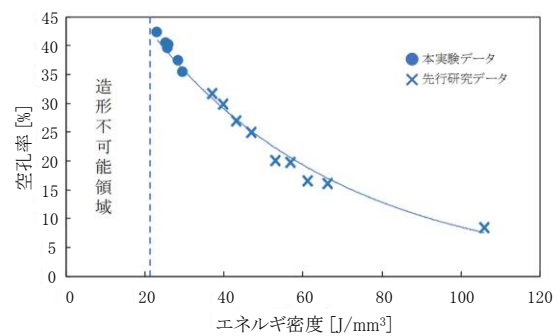


図2 空孔率とエネルギー密度の関係

$$E_d = \frac{W}{t \cdot d \cdot s} \quad (1)$$

また、試料の空孔率は X 線 CT スキャンにより算出した。

2.2 結果と考察

各試験片のエネルギー密度と空孔率の関係を図2に示す。一部の空孔率は先行研究²⁾を引用した。同図より、本実験にて検討した低エネルギー密度の範囲を含め、空孔率はエネルギー密度の増加に伴って指数関数的に減少していることが分かる。これは、投入熱量が増加したことにより粉末の溶融が促進

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され、安定したメルトプールの形成が誘起されたためであると考えられる。なお、 $E_d = 14.4\text{--}22.2\text{J/mm}^3$ の 8 条件では試験片がその形状を保持できず、実質的に造形が不可能であった。したがって、本実験系での RPS の造形可能なエネルギー密度下限値 E_{dl} は 23J/mm^3 程度であった。SLM において造形が成功するためには、粉末層の熔融と同時に、既に凝固が完了しているため下層との接合が必須となる。すなわち、 $E_d \leq E_{dl}$ では良好な層間接合が得られず、粉末供給時に付与される外力によって容易に形状が崩壊したと考えられる。この E_{dl} は粉末の粒度分布や粉末層の積層厚によって概ね決定されることが考えられる。以上より、本実験環境によって得られる空孔率の上限値は 42%程度であることが明らかとなった。

3. RPS の機械特性

3.1 実験手法

試験片は直径 5mm、高さ 10mm の円柱とし、表 2 に示す 6 条件で造形を行った。なお、表中に示されていない条件は前節と同様である。各条件によって得られる空孔率は走査速度の小さい方から、それぞれ 8.56、16.3、20.2、25.2、29.7、31.9%である。圧縮試験の試験速度は 1mm/min とし、得られた応力-ひずみ線図から弾性率と最大圧縮応力を算出した。また、試験片の破断面を走査型顕微鏡 (SEM) で観察した。

3.2 結果と考察

本試験により得られた空孔率と最大圧縮応力および弾性率との関係を図 3 と図 4 にそれぞれ示す。両値は空孔率の増加に伴って線形的に減少した。この傾向は、空孔率の増加に伴った 64Ti 基底領域の減少によって空孔近傍での応力集中が増長されたことに起因すると考えられる。また、本実験によって得られた弾性率は最大でも 20.1GPa であり、64Ti バルク材の弾性率 110GPa 程度³⁾と比して 1/5 以下である。これだけ低い弾性率は、例えば、金属製インプラントと生体骨 (弾性率 20~30%程度⁴⁾)との弾性率の乖離に起因して生じる応力遮蔽問題の解決に一定の効果をもたらすと考えられる。また、破断面の SEM 画像の一部を図 5 に示す。同図 (a)、(b) より、空孔率が低い試験片の破断面ではディンプルパターンが主体的に存在し、延性的な破壊形態であることが示唆された。一方、同図 (c)、(d) ではディンプルパターンはほとんど確認されず、すべりを伴った脆性的な破断面が認められた。これらの結果は、空孔形態の違いが構造の局所的な変形プロセスに影響を与えることを示唆している。

4. 結 言

本研究から得られた知見を以下に示す。

- 1) 本実験環境において、RPS を有する造形物を良好に造形するために必要なエネルギー密度の下限値はおおよそ 23J/mm^3 である。
- 2) 現状の手法で得られる RPS の最大空孔率は約 42%である。
- 3) RPS 試験片の最大圧縮応力と弾性率は空孔率の増加に伴って単調に減少した。

- 4) RPS 試験片の弾性率は従来バルク材と比べて 1/5 と非常に小さい値を示した。

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表 2 造形条件

レーザ出力 W	50
走査速度 mm/s	150, 240, 300, 340, 400, 430

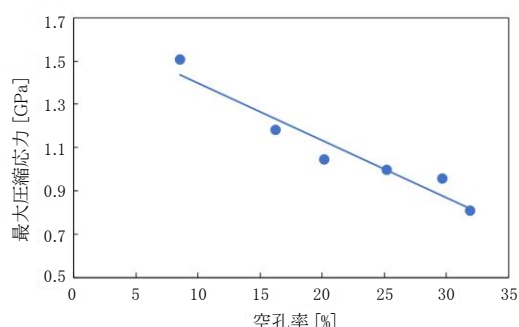


図 3 最大圧縮応力と空孔率の関係

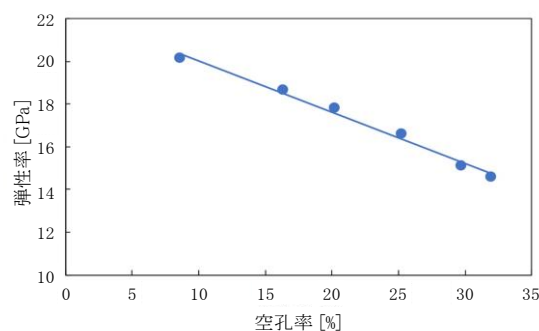


図 4 弾性率と空孔率の関係

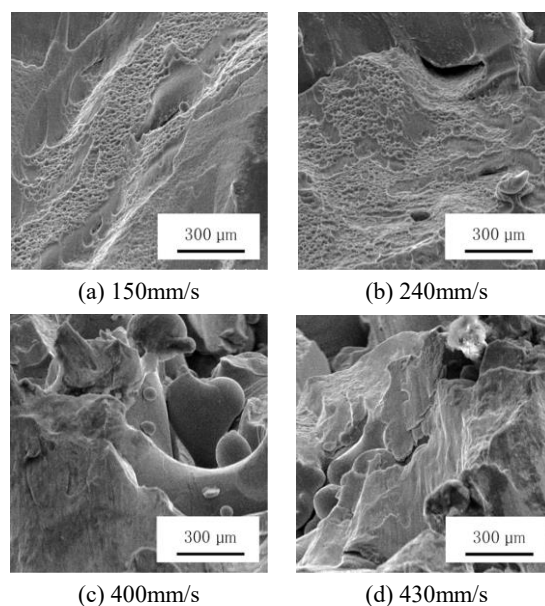


図 5 破断面 SEM 画像

Study on Short-Pulsed Laser-Induced Periodic Surface Structures Assisted by Mechanical Processing

Shuheï Kodama

Abstract

Fine structures have been studied largely to provide materials with such functionalities as a reduction in friction, change of the wettability, a reduction of light reflectance and an improvement of biocompatibility. Ultraprecision cutting, ultrasonic machining and photolithography have been widely used to create these functional surfaces, however, these traditional methods have many disadvantages such as complicated processing steps, environmental burden and difficulty processing large area and three dimensional shapes.

A laser is also a suitable method to fabricate fine structures, and an ultrashort-pulsed laser (USPL) can fabricate fine periodic surface structures called as LIPSS (laser induced periodic surface structures) through self-organizing phenomena. A USPL is a promising method for solving problems associated with traditional methods and providing functions by LIPSS.

However, this method still has such problems as low repeatability and difficulty controlling LIPSS since the principles and the phenomena of fabricating LIPSS have not been clarified completely. Moreover, a USPL which unstably irradiates at high cost have mainly been used to fabricate LIPSS since it has been reported that the laser with shorter pulse duration than the collisional relaxation time (CRT) can fabricate LIPSS. Industry needs a laser with as long pulse duration as possible to stably fabricate LIPSS at low cost. Hence, in this dissertation, fabrication of LIPSS using a short-pulsed laser (SPL) with 20 ps pulse duration was proposed and investigated to clarify principles and phenomena of fabricating LIPSS. In addition, the machining-assisted SPL was proposed to investigate the effects of the surface geometry before laser irradiation on LIPSS and to control LIPSS.

This thesis consists of seven chapters.

Chapter 1 gives the introduction of this dissertation. Various functionalities produced by fine structures on material surfaces and fabrication techniques of them are elucidated. A USPL, capable of fabricating LIPSS in an efficient way, is stated as an effective method to fabricate fine structures. On the other hand, its issues concerning stability of laser irradiation, equipment cost, principles, phenomena and control of LIPSS are pointed out. In addition, principles and phenomena of fabricating LIPSS were stated, and the effects of laser irradiation conditions and environments on fabrication of LIPSS were studied. The interference between incident lights and scattered lights, surface plasmons or the parametric decay, one of them changes the electric field distribution and ionizes atoms inducing the Coulomb explosions periodically on the material surface. After the CRT, the heat distribution causes either the ablation or the inhibition of LIPSS, resulting in LIPSS. Dependency of the pitch length of LIPSS on the laser wavelength, the laser fluence and the incident angle is pointed out. Next, the pulse duration of a laser used in this study was decided to 20 ps, about the upper limit of CRT, via calculation of the CRT to clarify the principles and phenomena of fabricating LIPSS. Moreover, low-temperature environment experiment is introduced to fabricate sharp LIPSS easily and effectively due to extension of the CRT. Finally, the objectives and organization of this dissertation were drawn.

In Chapter 2, fabrication of LIPSS on a 304 stainless steel (SUS304) surface was investigated using the SPL with the pulse duration of 20 ps, and LIPSS perpendicular to the laser polarization were

fabricated. Next, the finite-difference time-domain (FDTD) simulation of the electromagnetic field analysis was conducted to investigate the effects of the electric field (EF) distribution on fabrication of LIPSS and to predict the effective conditions and the shapes of LIPSS. The simulation demonstrated that the decrease of the laser wavelength increased the EF intensity at the bottom of grooves and shortened the pitch length of LIPSS, and the EF intensity is large just at the bottom of grooves under a low temperature. Subsequently, experiments under various laser irradiation conditions and the initial material temperature demonstrated that the pitch length of LIPSS depends on the laser wavelength, the laser fluence and the incident angle, and the low temperature environment is effective to create sharp and high-aspect-ratio LIPSS.

In Chapter 3, the effects of material characteristics on fabricating LIPSS were investigated analytically and experimentally with titanium (Ti), nickel-phosphorus (Ni-P) and SUS304. LIPSS were fabricated on all material surfaces and the aspect ratio of LIPSS depended on the extinction coefficient, the electron density, the ionization energy and the thermal conductivity. On the other hand, the ununiformed LIPSS were fabricated on the SUS304 surface, and the effects of crystal structure on fabrication of LIPSS were investigated. The geometry of LIPSS depended on the atom arrangement which varies with the crystal orientation.

In Chapter 4, the SPL assisted by the magnetic abrasive finishing (MAF), capable of creating straight nanogrooves, was proposed to investigate the effects of the initial surface geometry on fabricating LIPSS and to fabricate straight and high-aspect-ratio LIPSS since LIPSS follow depressions and debris. The effects of nanogrooves on fabrication of LIPSS were investigated analytically and experimentally. The FDTD simulation showed that the EF intensity decreased with increasing the depth of the grooves since deep grooves prevented the propagation of surface plasma waves. The experiments demonstrated the effectiveness of the MAF-assisted SPL to control LIPSS and fabricate straight and high-aspect-ratio LIPSS. The pitch length and the height of LIPSS increased with the increase of surface roughness. The aspect ratio of LIPSS on the Ni-P and SUS304 surfaces fabricated using the MAF-assisted SPL are approximately 0.2–0.25 and 0.4–0.7, respectively. From analytical and experimental results, the surface geometry before laser irradiation as the key factor is pointed out to control LIPSS since grooves facilitate the induction and the propagation of the surface plasma waves.

In Chapter 5, ultraprecision cutting prior to SPL irradiation was proposed to investigate the effects of the groove geometry on fabricating LIPSS and to fabricate micro/nanostructures with microgrooves and LIPSS. The effects of straight microgrooves on the geometry of LIPSS were investigated analytically and experimentally. The FDTD simulation showed that straight microgrooves with short pitch induces the high EF intensity. The experiments demonstrated that LIPSS fabricated around microgrooves were straight and the short-pitch grooves effectively help to fabricate straight LIPSS since decreasing the induction of the surface plasma waves causes distorted LIPSS at the area far from microgrooves. Moreover, the decrease of the pitch length of microgrooves increased the pitch length and the height of LIPSS since the microgrooves induced much surface plasma waves and increased the electron density. Finally, micro/nanostructures were successfully fabricated using the SPL assisted by ultraprecision cutting.

In Chapter 6, the effects of the groove angle on LIPSS were investigated by laser irradiation on the microgrooved surfaces with changing the angle of laser polarization to control LIPSS freely and to fabricate complex LIPSS. LIPSS followed the direction of microgrooves regardless of the laser polarization when the angle between grooves and the laser polarization was from 45° to 90°. Based on the results, an SPL was irradiated on the surfaces with zigzag and crosshatch microgrooves created by ultraprecision cutting to control LIPSS freely and to fabricate zigzag LIPSS and micro/nano-crosshatch structures. Zigzag LIPSS were fabricated as following zigzag microgrooves when the wavelength of the zigzag grooves was 10 μm and the amplitude of zigzag grooves was 3 and 6 μm . Crosshatch LIPSS were fabricated as following crosshatch microgrooves when $\phi = 60^\circ$ and 45° , $E_d = 0.09 \text{ J/cm}^2$ and $n = 10$; though LIPSS were distorted under the other experimental conditions. This chapter demonstrated the importance of the initial surface shape for fabrication of LIPSS and the effectiveness of the machining-assisted SPL to control LIPSS freely and to fabricate complex fine structures due to the propagation of the surface plasma waves from bumps and dents on the material surface.

In Chapter 7, the general conclusions of this research were given out.

In this dissertation, an SPL was proposed to clarify principles and phenomena of fabricating LIPSS and to fabricate LIPSS effectively. The SPL capable of fabricating LIPSS and the geometry of LIPSS depending on laser irradiation conditions, material characteristics and crystal structures were stated. Moreover, the machining-assisted SPL irradiation was proposed to investigate the effects of the initial surface geometry on fabrication of LIPSS and to control LIPSS. The simulations and experiments demonstrated the initial surface geometry as the key factor for control of LIPSS and the effectiveness of machining-assisted SPL to fabricate straight and high aspect ratio LIPSS. Finally, micro/nanostructures and complex fine structures with zigzag and crosshatch shapes can be fabricated using the machining-assisted SPL.

Study on nanocrystalline metal reforming by local heating

Kohei Shimizu

Abstract

Material with high saturation magnetic flux density, which allows to pass 1.5 T, is currently required for soft magnetic material. The material also has to possess high permeability and low iron loss for energy saving. These characteristics are difficult to be compatible because they are general the contradictive factors. However, Fe-Si-B-P-C type nanocrystalline alloys (hereinafter simply called “Fe-based nanoalloys”) have high saturation magnetic flux density since it is a high iron concentration material containing 93–94 wt% iron. They also have high permeability and low energy loss owing to soft magnetic material. This material is expected to reduce the current domestic electricity loss by 72% if the existing material is replaced with it. It is manufactured in the amorphous state and this state is transform into the nanocrystalline state with useful characteristics via heat treatment. Since the nanocrystalline state is brittle, processing is carried out in the amorphous state. However, since amorphous alloys generally possess characteristics such as high toughness and high strength, this processing cause problems such as tool wear during high precision machining. Therefore, it is necessary to devise ways to process the amorphous alloy with high efficiency and high accuracy and locally embrittling was proposed as a new method by utilizing the embrittlement due to structural change.

With these backgrounds, this thesis demonstrates locally reforming and embrittling the amorphous alloy by local heating and laser irradiation and stamp heating were proposed as new methods for local heating method.

This thesis is composed of six chapters.

Chapter 1 is the introduction of this study. The background of the present study and problems of the Fe-based nanoalloys are reviewed in this chapter. There is an increasing demand for new processing methods for amorphous alloys that can finish in high efficiency and high accuracy and can apply to press processing. This thesis, therefore, focuses on laser irradiation and stamp heating as local heating method.

In Chapter 2, heat conduction simulations were performed to determine appropriate conditions for local heating with both the optical laser irradiation and stamp heating. The previous investigation revealed that the Fe-based nanoalloys transform after heating under 400°C for a certain duration. First, the criterion duration for the completion of the crystallization was assumed as the time during which the sample exceeds 400°C, and the criterion was determined by comparing the results of stamp heating experiment and simulation, both of which were performed with a plate of whole surface at 600°C. Based on the criterion, the laser irradiation conditions and the stamp heating conditions for local heating were determined by performing heat conduction analyses. Laser irradiation analysis demonstrated that the local crystallization would occur under the conditions where the heating energy was 0.6 W, scanning speed was 500 or 1000 $\mu\text{m/s}$; meanwhile the stamp heating analysis demonstrated that the local crystallization would occur under the conditions where the stamp temperature T was 600°C and the heating duration τ_h was 1.0–3.0 s.

Chapter 3 discusses the thermal effects of the laser irradiation on Fe-based amorphous alloys. The irradiation marks were smaller than 50 μm of the laser beam diameter. Heat deformation was confirmed under all the

conditions from the surface shape measurement. The height of the thermal expansion increases with the deceleration of the scanning speed. The height becomes constant and the width of it increases when the scanning speed becomes slower. It is expected that these are caused by the movement of Fe atoms due to the absence of the binding force.

Chapter 4 discusses the thermal effects of the stamp heating on Fe-based amorphous alloys. Three types of color transition were confirmed in stamp heated sample. The width of the color transitions became longer as the contact time increased. This transition is caused by the formation of an oxide film. This results under the conditions where $\tau_h = 1.0$ s compared with the analytical result in Chapter 2 and the comparison suggested that this material became steel color via temperature rise at 450°C and blueviolet color via temperature rise at 350°C. With these data, temper color table in it is created.

Chapter 5 clarifies the crystallization and the embrittlement of the samples applied by the laser irradiation or the stamp heating for local heating. First, the crystallization of them was measured by XRD. Since the laser irradiation area of 50 μm was not analyzed, samples having it of 1000 μm were prepared by irradiating with operation. Contrary to the result of the analysis, the crystallization was not confirmed under the condition where $\tau_h = 1.0$ s. It can be inferred that it is due to the measurable range of XRD. The peak of the XRD pattern was confirmed at $2\theta = 45^\circ$ under the conditions where $\tau_h = 2.0$ or 3.0 s and $V = 500$ $\mu\text{m/s}$. This peak is similar to the XRD pattern of α -Fe. Subsequently, the tensile test was conducted on these samples and each sample was broken from the processing trace where $\tau_h = 2.0$ or 3.0 s and $V = 500$ –2500 $\mu\text{m/s}$. The breakage test force decreased with the increase of the heat quantity. It can be expected to use the local heating method by the laser irradiation or the stamp heating for press working. However, it is necessary to deeply study the heat effect of the width about the stamp heating, because there is a risk that affect the product part.

Chapter 6 presents the general conclusions obtained in this study.

Study pn surface active effect by ultra fine bubble

Kazuki Suzuki

Abstract

In recent years, miniaturization, weight reduction, and power saving have been promoted with various kinds of electronics devices, and improvement of "power semiconductors" used for power control has been required. However, the physical properties of silicon that has been mainly used in semiconductor substrates are reaching the theoretical limit. Therefore, such materials as gallium nitride (GaN) and silicon carbide (SiC) have been attracting attention as new semiconductor substrate materials. Currently, much study and development of GaN and SiC have been being carried out actively because the electronics devices with these can be expected to improve efficiency significantly and miniaturization compared with silicon. However, the residual damaged layer in rough processing and the low efficiency in finish processing have become problems in the wafer manufacturing process of the next generation semiconductor substrate. This thesis introduced ultrafinebubble (UFB) assisted tape grinding as a new processing method in order to realize high efficiency and low damage processing of GaN.

Chapter 1 introduces the backround and the objective of this thesis.

Chapter 2 describes the behavior of UFB water over time, the usefulness of UFB water as a working fluid, and confirmed the surface active effect of UFB by carrying out friction test. The particle size distribution measurement of UFB one week after production revealed that about 100 million bubbles remained in 1 mL. From this, it was shown that the UFB has a sufficient life span available for processing. A friction test on GaN was conducted under the respective conditions of purified water and UFB water to evaluate the processing rate and surface properties. This result showed the removal amount and the surface texture of both UFB water and purified water were not changed. From the above, UFB could not form a sufficient oxide layer in the GaN surface layer, therefore a new method to irradiate ultrasonic waves to collapse UFB more is proposed.

In Chapter 3, Ultrasonic assisted UFB water particle size distribution measurement was conducted, the influence of ultrasonic wave on UFB was investigated, Electron Spin Resonance (ESR) measurement was carried out, and OH radicals were quantitatively measured, and the surface active effect of ultrasonic assisted UFB water was investigated. It was confirmed from the particle size distribution of UFB water before and after ultrasonic assistance that the particle size of UFB remaining was reduced due to the influence of ultrasonic wave. From the ESR measurement result of ultrasonic-assisted UFB water, collapse was not confirmed with UFB water alone. In addition, when comparing ultrasonic assisted UFB water with ultrasonic assisted purified water, ultrasonic assisted UFB water produced more hydroxy (·) radicals, and the larger the UFB concentration, the more OH radicals were generated. In the next chapter, while ultrasonically assisted UFB water is continuously supplied to the processed surface, GaN tape grinding processing is performed to evaluate processing rate and surface properties, and the utility of ultrasonic assisted UFB water for GaN processing is examined.

In Chapter 4, effect of UFB water was irradiated with ultrasonic on GaN tape grinding were verified experimentally. The processing rate, the surface roughness, and the process alteration layer were evaluated. Compared with the using purified water, the processing rate using the ultrasonic assisted UFB was able to obtain a processing rate of 2.7 times. Surface roughness and work-affected layer did not change under any working fluid

condition. From this fact, we have succeeded in improving the processing rate without deteriorating the surface quality by utilizing ultrasonic assisted UFB water for GaN tape grinding.

Chapter 5, the general conclusions of this study are summarized.

Study on Powder Jet Deposition with composite particles

Takaaki SUNOUCHI

Abstract

Dental treatments including caries treatment based on minimal intervention are essential to improve the quality of life. In the conventional caries treatment, a dentist removes caries part of tooth enamel and dentin by using a dental drill and then fills the removed parts with an inlay material such as dental gold-silver-palladium alloys, ceramics, or resin. The inlay materials, however, differ from natural teeth in the coefficients of the thermal expansion and the tensile strength. The difference causes to deterioration at the interface between the filling and tooth and finally induces the secondary caries. Additionally, aesthetics dentistry has been highly demanded for patients in recent years. In the conventional aesthetics dentistry has had problems such as poor adhesion of restorative materials and treating harmful chemicals to human bodies. For these reasons, it has been expected that a new dental care technology that can keep the quality of our oral function is innovated.

Powder jet deposition (PJD) is a film formation technology by high-speed collision of fine particles under the atmospheric and room temperature conditions. PJD has been tried applying to dental treatment with hydroxyapatite (HA) fine particles. The HA is the same ingredients as nature teeth; therefore, it is highly safe to human, can be applied to aesthetic dentistry. Consequently, PJD with HA is expected to perform a dental treatment both functional and aesthetic characteristics. In this thesis, particles of zirconium oxide (ZrO_2) that is a material mainly used as dental crowns in the aesthetic dentistry were composited on the HA particles for aesthetical purposes and the composite particles were designed to improving aesthetics effectively. Moreover, the relationship between the film structures made by the composite particles and the color tone of the films was investigated.

The objectives of this thesis is to reveal the following contents: (1) evaluating color tones of the HA film and ZrO_2 for designing the ideal composite particles, (2) the film formation mechanism of the composite particles of HA and ZrO_2 (3) the relationship between the film structure made by composite particles and the color tone of the film.

Chapter 1 is the introduction of this thesis. The background of this study and current problems of the aesthetics dentistry are reviewed in this chapter. The achievements of PJD and the composite particles of the previous study are summarized, together with the objectives of this study.

In Chapter 2, the ideal color tone in the CIE $L^*a^*b^*$ color space was investigated. Spectrophotometry was performed on an HA film sprayed by PJD, a ZrO_2 disk, which is a reference of aesthetic evaluation, and the shade guide (SG), which is a tooth-shaped color referencesimulating natural tooth colors;; subsequently, their spectra were modified in the CIE $L^*a^*b^*$ color space. The CIE $L^*a^*b^*$ is expressed by the three-dimensional orthogonal coordinates: L^* represents the lightness, a^* and b^* represent the combined attributes of hue and chroma, so that the color difference between two colors is obtained by calculating the coordinate distance. The

results illustrate that the L^* value of the HA film was lower than the SG; meanwhile, the ZrO_2 disk possessed a higher L^* value than the SG. order to calculate a composite ratio between the HA and ZrO_2 which is an ideal color tone. These results is the design guideline of the ideal film made by composite particles.

In Chapter 3, the deposition mechanism of the composite particles and a mechanical property of films made by composite particles were investigated. The composite particles were prepared with the mechanochemical treatment generating plasma the plasma in a RF source. The influence of the composite parameters revealed that plasma promotes the composite. In addition, the longer the composite time was, the lower the film formability became. These composite particles was revealed that the longer composite time made the composite particles the more rounded by calucuating the circularity. The previous researches suggest that rounded particles possess less ability to deposit than angular particles. Additionally, it was confirmed that the larger the amount of ZrO_2 , the lower the film formability. Moreover, ZrO_2 has a risk of damaging meshing-teeth because it possesses higher hardness value than human teeth. Therefore, the Vickers hardness test was carried out in order to confirm the hardness problems occurred when made into the composite particles. This test showed that the film made by composite particles was possible to adjust the hardness, so that the possibility of application to clinic was shown.

In Chapter 4, the relationship between the film structure made by the composite particles and the color tone of the film was investigated. The color tone of the films made by the composite particles was evaluted in the CIE $L^*a^*b^*$ color space. As a result, the L^* value of the film made by the composite particles was higher value than the HA film, so that the film made by the composite particles whitened by the composite treatment of ZrO_2 particles was revealed. In addition, transmission electoron microscope (TEM) and scanning transmission electoron microscope (STEM) observations were revealed that ZrO_2 exists in the film made by the composite particle. Energy dispersive X-ray spectroscopy (EDS) analysis the rate of HA and ZrO_2 in the film. As a result, there is a correlation between the ZrO_2 particle volume and the L^* value in the film. From this calculation result, the composite parameters for the target color tone are shown. Moreover from the TEM/STEM observations showed that the effectiveness of control of the film surface structure by composite particles prepared with the mechanochemical treatment.

Chapter 5 gives the general conclusion of this research. In addition, academic and industrial significance of this study were stated.

Imparting biocompatibility to zirconia implants

with nanosecond pulsed laser

Tomohiro Harai

Abstract

Japan has been currently experiencing a super-aging society. Implant treatment in dentistry is becoming the mainstream for improving the quality of life (QOL). In recent years, zirconia implants have garnered attention due to their high strength and toughness among ceramics materials, aesthetic whiteness and no risk of metal allergy. It usually takes several months after implantation for forming a tight connection with growing new bone between the implant and the jawbone, and if a sufficient connection cannot form within a certain period, bone tissue around the implantation site is resorbed to fall off the implant. Thus high biocompatibility is a vital material property for implants, and surface modification of zirconia has been developed.

The research of this thesis focused on nanosecond pulsed laser (NPL) as a potential treatment to improve biocompatibility of zirconia implants. The NPL can perform microfabrication on difficult-to-cut materials by removal of material caused by local heating. In addition, a heat affected area is formed about several micrometers around the irradiated spot. The purposes of this research were to evaluate the microstructures and chemical composition changes of the surface of tetragonal zirconia polycrystal (TZP) after the irradiation of the NPL and to verify the effects for biocompatibility.

The present thesis is composed of six chapter. Chapter 1 is the introduction of the research background and objectives.

In Chapter 2, the zirconia samples were irradiated using NPL with a top flat profile. Two types of zirconia ceramics, yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) and ceria-stabilized tetragonal zirconia polycrystals/ alumina nanocomposite (Ce-TZP) were used because stabilizing substances may make differences in heat effects. The microtextures of the irradiated samples were evaluated through the observation with a scanning electron microscope (SEM). The laser irradiation significantly blackened the Ce-TZP surfaces. To identify the chemical composition changes, the samples were characterized using energy dispersive X-ray spectrometry (EDX) and X-ray photoelectron spectroscopy (XPS). The SEM images showed that grooves whose widths were the same as that of the laser spot (30 μm) were formed on the surfaces of both samples, and the surfaces of the grooves were roughened by aggregate debris of a few micrometers in size. Such cell-sized grooves with rough surfaces are considered a favorable environment to help osteoblast cells to grow. The EDX result showed oxygen atoms decreased on the irradiated surfaces of both samples. In an additional experiment, the color of the irradiated Ce-TZP was whitened by heating in air. Considering these points, the color change was caused by the generation of oxygen-deficient zirconia. The Al2p spectrum from the XRD patterns, however, slightly showed the Al-C binding spectrum. Although this result can not clearly say the generation of aluminium carbide, the possibility that laser irradiation altered the composition of Ce-TZP was suggested.

In Chapter 3, the zirconia samples were irradiated using NPL with gaussian profile to compare the shape of the grooves to the result in Chapter 2. Controlling irradiation parameters gave groove textures in any width and depth. Wider grooves were formed on the Ce-TZP than those on the Y-TZP in the same irradiation conditions, which results might be caused by a higher thermal conductivity of the Ce-TZP. The grooves were rounded comparing to the result in Chapter 2. This shape can be expected to reduce stress concentration.

Chapter 4 demonstrates the trials to suppress the oxygen deficiency of zirconia using laser irradiation with blowing two types of gases, oxygen and nitrogen, and evaluation of the chemical composition changes by EDX and XRD. Blowing oxygen gas suppressed blackening and blowing nitrogen gas accelerated blackening compared with the irradiation in air; however, EDX analysis and XRD patterns gave no change among irradiated samples. The surface color differences appeared, but no chemical effects might occur. This phenomenon needs additional researches.

Chapter 5 elucidates the evaluation of biocompatibility of the laser-irradiated zirconia. The zeta potential was measured to examine the surface chargeability of the samples. The contact angle was measured to examine the wettability affected by the textures and the surface energy states. Cell culture experiments with MC3T3-E1, an osteoblast precursor cell line derived from mouse calvaria, were conducted to examine the cell behavior on the samples. The zeta potential values of the irradiated samples shifted to the positive direction and the absolute value became smaller in the most of them. This result suggests the possibility that laser irradiation suppresses the absorption of ions and proteins on zirconia in body. The contact angle values of the irradiated samples got higher than the polished samples, and indicated hydrophobicity. Subsequently, cell attachment and morphology on each surface were observed a day after seeding. SEM images and confocal laser scanning microscopy images (CLSM) showed that the cells on the polished samples extended randomly; contrastingly, the ones on the irradiated samples along the processing direction. Particularly on the laser-grooved samples, the cells attached to the sides of the grooves and extended three-dimensionally. Three days after seeding, the cells proliferated better on the laser-grooved Y-TZP than polished and the laser-roughened ones ($p < 0.05$). This result shows the effectiveness of the groove texturing. Contrastingly, the cell proliferation got worse on the irradiated Ce-TZP, among which the laser-roughened Ce-TAP significantly suppressed it ($p < 0.01$). The chargeability and wettability are thought to be irrelevant to the cell proliferation result because of no significant difference in them between the Y-TZP and Ce-TZP. Thus, the alteration of the constituents in the Ce-TZP may adversely effect to the cell proliferation.

Chapter 6 summarizes the main conclusions.

Study on Tribological Characteristics of Laser-Induced Periodic Surface Structures

Kazuhiro FUKUI

Abstract

Friction and wear cause energy loss and life reduction of machines and lubricant and hard coating have been widely used as countermeasures against those tribological problems; however, the restriction on the use of chemical substances have gotten stricter due to prevention for large environmental burden and the coating alone cannot impart enough wear-resistant properties against current severe friction environments where manufacturing machines experience. Meanwhile, microtexturing onto the friction surface is a well-known approach to improve the tribological properties. The structure on the friction surface contributes to lowering friction due to reduction in the actual contact area and trapping of the wear particles. Short pulsed laser (SPL) irradiation has attracted attention as a microtexturing method; specifically, a linearly polarized SPL can generate a periodic groove structures, which is generally referred to as LIPSS (laser-induced periodic surface structures), and their spatial period is close to the laser wavelength and their direction is perpendicular to the laser polarization. The method possesses some advantages including applicability to various kinds of materials and ease of fabrication.

The purpose of this thesis is to improve the tribological characteristics of LIPSS and to control them. First, two types of samples, lasered-before/after-coating (LbC and LaC) samples, and these surface properties were evaluated; subsequently, friction tests were performed under the dry condition. In addition, the relationship between adhesion of materials and structure direction of LIPSS against friction direction were clarified and this enabled control of adhesion of material.

This thesis is composed of five chapters.

Chapter 1 states the background and the objectives of this thesis.

In Chapter 2, the two kinds of samples, the LbC and LaC samples, were prepared to examine the difference of the procedure of combining the LIPSS and hard coating. Diamond-like carbon was applied as the coating material and the base material was cemented carbide. These surface features and topographies were evaluated by a scanning electron microscope (SEM) and atomic force microscope (AFM). The spatial period of the LIPSS on the LbC sample was approximately equal to the laser wavelength, while that of the other was approximately 1/5 of the laser wavelength and the height of LIPSS was about a half of the LbC sample. The difference in the LIPSS topographies was assumed due to the irradiation conditions and the material properties. Subsequently, the friction test were performed under the dry condition. The result of experiments clarified that the LbC sample improves the tribological characteristics; meanwhile the LaC sample was worn extensively. These result caused by the small LIPSS and deterioration of the composition of DLC by the laser irradiation. The form of LIPSS perpendicular to the friction direction was more effective in reduction of friction. Moreover, the wear powder trapping by the valley parts of LIPSS and the reduction of the actual contact area were confirmed via the observation of the track of the LbC sample.

In Chapter 3, the effect of LIPSS on the adhesion of material causing seizure or wear was evaluated by observing the tracks after the friction test using an SUS304 ball. The friction tests were performed on cemented carbide with and without LIPSS, DLC-coated cemented carbide and LbC samples. The track was analyzed with energy dispersive X-ray spectrometry (EDX), and the amount of adhesion was determined by the atomic concentration of Fe that is the main element of SUS304. The result of the friction test on the cemented carbide samples clarified that the LIPSS could decrease debris adhesion than the mirror surface and the amount of the adhesion was smaller as the angle between the structure direction and the friction direction was small. The result of the friction test on the DLC-coated samples clarified that the LIPSS brought no new adhesion because all tracks had no adhesion.

In Chapter 4, since the wear debris adhesion depends on the structure direction, the control of the adhesion was attempted by creating groove patterns. The two groove patterns, alternating vertically and horizontally grooves, zigzag grooves, were created on the base material, cemented carbide, by controlling the polarization direction at laser scanning utilizing LIPSS directions. Friction tests were performed on the LIPSSed cemented carbide and LbC samples with each groove pattern to evaluate the effect on debris adhesion by observing the wear tracks. The result of experiments of the LIPSSed cemented carbide samples clarified that local adhesion and the amount of adhesion is controllable on alternating vertically and horizontally grooves, and adhesive particles were not affected by the LIPSS direction. Meanwhile, the result of experiments of the DLC samples clarified that groove patterns brought no new adhesion because all tracks had no adhesion. Further control of adhesion is expected because a more complicated pattern could be created depend on the irradiation condition because crossing grooves were created at the structural boundary of both patterns.

In Chapter 5, the general conclusions of this study are summarized.

Study on influence of ultra fine bubble at friction interface

Sotaro Matsuda

Abstract

Various problems concerning the occurrence of friction between elements and the accompanying occurrence of wear are extremely important tasks in the field of machining, and many researches are being conducted in the field of tribology. The ultrafine bubble (UFBs) is a bubble with the diameter of 1 μm or less and has been actively researched in recent years because it has various effects such as cleaning effect, catalytic effect, sterilizing / disinfecting effect, deodorizing effect, fine particle adsorbing effect and bioactive effect. Friction reduction is also reported as an effect of the UFBs; however, its mechanism has not been established yet. The objective of this thesis, therefore, to elucidate the mechanism.

Chapter 1 demonstrates the background and the objectives. The previous study of the UFBs and microbubbles (MBs) are briefly reviewed and the objectives of this thesis are elucidated.

In Chapter 2, the viscosity measurement experiment of UFBs water was carried out to verify the influence of UFBs on the viscosity of purified water. The experiment was recorded with a high speed camera for 2.18 s with 1/12000 s of the shutter speed and 250 frame per second every five minutes to capture the change in the appearance of UFBs water and pure water. The results showed that MBs and submillimeter bubbles stuck on the transducer of the viscometer and expanded temporally in UFBs water; meanwhile, no changes were observed in pure water. Consequently, the appearing bubbles might be generated with UFBs agglomeration induced by the external vibration of the viscometer. In addition, it became clear that the viscosity nominally increased by submillimeter bubble generation on the oscillator.

In Chapter 3, plate-on-plate friction tests were performed to verify the influence of UFBs at the friction interface between the planes. Two specimens of the mirror-finish and the fine-grooved surfaces were attached on the bottom of the tribometer to examine the influence of UFB on the coefficient of friction depending on the presence or absence of fine groove structure. To observe the behavior of the bubbles at the friction interface, soda lime glass was used for the upper test piece. The test with the mirror-finished surface showed no differences in appearance or friction coefficient between purified water and UFBs water. Meanwhile, the test with fine-grooved specimens showed that MBs and submillimeter bubbles appeared in the microchannels when the UFB water was applied and the friction coefficient was increased; no bubbles appeared in the microchannels with the purified water and the friction coefficient was not changed significantly. These results reconfirm the outcome of the previous chapter: UFBs are agglomerated by the external force and turned to visible-sized bubbles; additionally, it was suggested that the submillimeter bubbles might arise the direct contact between the two bodies to increase the friction coefficient.

In chapter 4, the influence of the UFBs on friction coefficient on the pin-on-disc friction test was investigated. The results indicated that the friction reducing effect may be obtained by the lubricating action of both the UFBs water and the purified water in the test, but the conclusion that the friction reducing effect tends to be the inclusion of UFBs in purified water was not obtained. In addition, it was clarified that the fine groove structure does not contribute to the friction reduction effect in the pin-on-disk friction test.

In chapter 5, the general conclusions of this research are summarized.

Development of the highly functional implant

by metal additive manufacturing

Takumi Mizoi

Abstract

Metallic medical implants are rapidly spreading as effective countermeasures against motor disorders and oral diseases; therefore, they play an important role in elderly patients' prolongation of healthy life expectancy and improvement in quality of life. The demand for them is expected to grow in developed countries with the aging population; however, they have several problems to be overcome: “stress shielding” is a major problem, which is caused by a great difference in the elastic modulus between a metal and living bone. A famous method to reduce the elastic modulus is filling pores into metal and that kind of metal is called porous metal. The porous structure can also provide strong retention ability between a bone and an implant because of the bone ingrowth inside the pores. In other words, porous metallic implants eliminate both mental and physical burden of patients due to complications and implant replacement surgery as well as prevent the stress shielding.

Many methods for making porous metals have been proposed so far, and metal additive manufacturing (MAM) has been gathering attention in recent years. This method can realize a complex porous structure like a lattice structure with high design flexibility. However, such structures require complicated procedures, and it is necessary to consider the reproducible minimum dimension as a design restriction. Therefore, in order to disseminate the porous implants manufactured by MAM, simple and size-independent method is necessary.

Thus, the author has proposed “rhizoid porous structure (RPS)” utilizing vacancy defects contained in the objects produced by selective laser melting (SLM), which is one of representative MAM methods. The RPS has tens to hundreds of micrometers of pores and the pores connect complicatedly like the root of a plant. Because porosity of RPS can be controlled by the shaping conditions, it is expected to develop the highly functional implant with multiple functions by deriving the optimum shaping conditions from functional or dynamical designs. The aim of this thesis is to show feasibility of the highly functional implant. In this thesis, the porosity and orientation of pores in RPS were manipulated by varying the shaping conditions. The pore shapes were evaluated by X-ray computer tomography (CT) and image processing. Additionally, the mechanical properties, bond strength with resin and biocompatibility were evaluated. The thesis consists of following six chapters.

Chapter 1 introduces the background and the objectives of this thesis.

In Chapter 2, the relationship between the top surface textures of SLMed samples and the porosities were investigated to clarify the mechanism of the RPS creation. The surface of the samples became smoother under conditions where the laser was irradiated at a lower scanning speed or twice in each layer. In addition, the surface roughness and the porosity clearly showed a positive correlation and the smoothest sample reached the density of 99.7%. These results show that the geometric shape of the laser scanned surface has a dominant influence on the formation of the pores inside the RPS material. Furthermore, a uniaxial laser scanning provided the orientation along the scanning direction of the surface irregular shape and the pore shape. These results demonstrate that manipulation of non-numerical shaping conditions enables highly free control of the pore forms.

In Chapter 3, in order to evaluate the mechanical properties of the RPS material, a tensile test was carried out using specimens shaped under different conditions. The results validated that the tensile strength of the specimen decreased with increasing the porosity. Compared to conventional porous materials at the same porosity, the RPS

specimens showed a higher tensile strength. This fact suggests that the extremely fine acicular metallic structures, unique on the SLMed products, contribute to improvement of the static strength. In addition, the elastic modulus was a very low value as around 8 to 11 GPa close to a living bone, and a correlation with the porosity was not observed. Furthermore, mechanical anisotropy appeared in the specimens in which the pores were oriented by the method in Chapter 2. These results show that the RPS can combine mechanical functions such as low elastic modulus and high strength and mechanical anisotropy to implants.

In Chapter 4, the characteristic surface structure of the RPS material was investigated whether it sufficiently possesses both functions of "bonding with resin material" and "strong bone retention" required for dental implants. The bonding strength test with a dental resin demonstrated that as-built RPS samples possessed a superior bonding strength to the sample with metal beads that are a conventional retention structure. The cell culture experiments demonstrate the RPS samples with specific porosities possessed cell proliferation properties equal to or higher than those of non-porous materials, and the shape of the cells on the surface was high aspect ratio. Furthermore, the result confirmed that the cells entered the pores and formed networks with each other. These results suggest that the RPS implants may achieve strong bonding with bones because their pores advance bone growth and retention ability.

In Chapter 5, the internal structure of the highly functional implant using the RPS was proposed and the graded porous structure was built by SLM. Design areas of the model were divided at regular intervals along the laminating direction or radial direction, and different laser scanning speeds were assigned to each area. This method realized to change the porosity of the sample gradually along each direction. In addition, the tensile test results for the graded porous structure showed that all the specimens were broken in the highest porosity region, however, their tensile strengths were higher than those of the specimens having a high porosity shaped in Chapter 3. This result indicates that the low porosity region in the graded porous structure can support the stress effectively.

Chapter 6 summarizes the results and conclusions of this thesis.

Study on the Fabrication of Micro-texture by Ultrasonic Vibration Micro-cutting

Ziqi Chen

Abstract

Solid surfaces with micro- or nanometer-scale textures possess many kinds of functional performances. Among the functionalities, controlling of the surface wettability with tailored textures have especially caught extensive attention in recent years. At the same time, the surface wettability controlling provides appearance of valuable effects such as water repellency, self-cleaning property, ideal droplet-sliding action and frost prevention in various fields of industry. To fabricate wettability functional surface effectively, characterization of wetting behaviors on the textured surfaces and clarification and manipulation of wetting performances related to microtexture geometries are need.

When talking about how to change the surface wettability, there are two feasible way to achieve the goal: change the surface chemical property and modify the surface geometry, while texturing approaches have some unique features. To investigate the wetting problem on a textured surface, developed wetting phenomena have observed and studied. Because of the complex geometry of the microtexture, the phenomena cannot analyze by the foundational theories of the wetting features on the textured surface: the Wenzel equation and the Cassie-Baxter equation, thus previous researches proposed two different kinds of methods, the thermodynamic analyses and the finite element methods (FEMs) to analyze the problem. These methods allow calculating developed wetting properties including texture geometries, so that the texturing processes are desirable to contain ability of controlling texture morphology.

It is currently possible to make the tailored textures by chemical approaches, such as lithography, etching and chemical vapor deposition. However, these chemical methods have shortcomings: restriction of material, costly and relatively longer processing time. Ultrasonic vibration assisted machining was developed in the previous studies, to become a texturing process covering the shortcomings of the conventional methods. This is a mechanical removal process using cutting tools with ultrasonic vibration. The texture geometry features are simply determined by tools shapes and processing parameters

In this study, the microtexture on the metal surfaces that are able to be fabricated by ultrasonic vibration assisted machining like rotary ultrasonic texturing (RUT) and ultrasonic vibration assisted micro-cutting were applied the thermodynamic analysis and CFD-FEMs to evaluation on the developed wetting phenomena with the effect of geometry in terms of variables: the textures size and vibration conditions. Subsequently, the calculated textured surfaces with different microtextures were fabricated on the Ni-P surface with two different ultrasonic assisted texturing, and the contact angles of the textured surfaces were measured to find out the change of surface wettability. The objective of this thesis is a further and deeper development of ultrasonic assisted machining.

This thesis is composed of five chapters.

Chapter 1 is the introduction of this thesis. The background of this research is described. The shape of the microtexture is important in the wettability control, indicating that the fabrication of the functional surface is expected by ultrasonic texturing technique, and finally, the research objectives is introduced.

Chapter 2 applies the thermodynamic contact angle analysis to the shape of the microstructure created by the ultrasonic texturing and analyze the characteristics of the wetting function of the textured surface. In addition, different kinds of CFD FEMs, such as the volume of fluid (VOF) method, level-set method (LSM) and phase field method (PFM) were applied to analyze the problem. The balance contact angle was obtained from the energy calculation, and the dynamic contact angle was obtained from the three different simulations.

According to the results, the VOF and PFM simulations and the energy calculations show the same trend: the textured surface with a smaller pitch possesses a larger contact angle, and show a certain hydrophobicity; however, LSM shows the opposite result. The textured surface with pyramid alloy whose pitch is below 40 μm shows more hydrophobicity than 60 μm and 80 μm , and as the pitch becomes larger, the contact angle also increases at a greater rate than larger pitch. When the value of pitch reaches 60 μm , the contact angle shows little difference.

In Chapter 3, diverse microstructures on metal surfaces were fabricated by using the RUT. Furthermore, these microtextures on the surface were observed by a white light interferometer, and the contact angle of the textured surface was measured. From the observation results, the RUT technique can be used to fabricate micro/nanometer scaled surface textures by using one-point diamond tools, and this process has an influence on the change of surface wettability because it can be confirmed that the textured surfaces exhibit a larger contact angle than the flat surface.

In chapter 4, ultraprecision machine tool (MIC-300) was used to create pyramid and frustum microtexture surfaces with different processing parameters and the wettability change of the textured surfaces was also measured. According to the observation results obtained by the SEM and laser microscope, different pyramid and frustum microtexture could be fabricated by using ultraprecision micro-cutting process, and their geometric parameters can be strictly controlled. In addition, the submicrotextures on the pyramid's wall area could be generated by ultrasonic vibration during the machining processing. Comparing to the flat surface, the textured surfaces with pyramid or frustum microtextures show obvious hydrophobicity. Besides, comparing the RUT process in Chapter 3, ultraprecision micro-cutting processing may create relatively stronger hydrophobic surface. It could also be confirmed that within a certain range, much smaller microtextures on the textures surfaces can lead to larger contact angle and better hydrophobicity.

Chapter 5 presented the general conclusions of this thesis.

This study clarified the relationship between microtexture size and surface wettability change, and created functional surfaces by applying ultrasonic vibration assisted machining. These results help to elucidate the wetting mechanism, and is expected that realization of advanced wettability control in many engineering fields

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【研究室だより】

昨年度の仙台は雪も少なくあつという間に春を迎えることができた気がします。

研究室では社会人ドクターとしてさらに 3 名増え、さらに昨年 5 月よりドイツのフラウンホーファー研究所へと旅立たれた水谷先生がいよいよ研究室へ復帰されます。皆様からさまざまな経験・知識を研究室へと持ち込んでいただき、より一層研究を強化していくことを期待しております。

年号が平成から令和へと変わり、時代の節目を迎えた今年度ですが、私たち厨川・嶋田研究室/水谷研究室一同、日々の研究やその他の活動に励み成果を積み重ねられるよう励んでまいります。

令和元年 5 月
助教 嶋田 慶太