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Paper:

Ridge-Texturing for Wettability Modification by Using Angled Fine Particle Peening

Yutaka Kameyama†, Hideaki Sato, and Ryokichi Shimpo

Tokyo City University
1-28-1 Tamazutsumi, Setagaya-ku, Tokyo 158-8557, Japan
†Corresponding author, E-mail: ykameya@tcu.ac.jp

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The industrial demand for wettability control has been increasing because wettability is a key factor for achieving novel anti-contaminant surfaces and related products. In this paper, the potential of angled fine particle peening (angled-FPP) was explored as a method of surface modification to control wettability. Angled-FPP, which is an abrasive jet machining process conducted using a peening nozzle set at an angle inclined to the material surface, is a potential texturing technique. With it, it is possible to create periodically aligned peaks and valleys ("ridges") on the peened surface. Because control of wettability could possibly be achieved by varying the geometric characteristics of the texture, an attempt was made to vary ridge texture by conducting angled-FPP using three kinds of shot particles: steel, glass, and alumina grit. Thereafter, changes in the wettability owing to creation of a ridge texture on the surfaces were evaluated, with focus on the geometric and morphological features of the ridge texture. The results indicate that the size of the ridges depends on the size and shape of the shot particles, and the angled-FPP conducted using finer angular particles created densely concentrated ridges. The superficial appearance of the ridge texture differed depending on the shot particles used for angled-FPP. The topographies created by angled-FPP using steel particles and alumina grit were "hierarchical" (i.e., the ridge structure was overlapped by finer-scale roughness). In contrast, angled-FPP conducted using glass particles created ridge structure with a quite plain surface. Measurement of the water drop contact angle revealed that the surface became less hydrophilic after creation of the hierarchical topography. It was concluded that the predominant influence on the wettability of the angled-FPP surfaces came from the superficial morphology of the ridge texture.

Keywords: peening, surface texture, periodical structure, wettability, contact angle

1. Introduction

Demand for control of wettability by modification of surfaces has been increasing in industry. For example, wettability-controlled surfaces have reportedly been used to achieve novel micro-fluidic devices [1] and to enhance cell activities on medical implant materials [2]. Excellent anti-contaminant ability could be achieved by creating hydrophobic surfaces [3]. The most common approach used to obtain hydrophilic and hydrophobic surfaces should be applying thin films on substrates. Wet processes such as the sol-gel method, followed by addition of specific agents to control wettability, have been attempted to create hydrophilic and hydrophobic films [4–6]. Those techniques could change the water drop contact angle of the surfaces depending on film morphologies as well as on the choice of agents. However, the effect should disappear if the coating delaminates or is worn away during the service period. Techniques intended to control wettability by improving the material itself would be expected to avoid risks arising from film delamination.

Surface texturing technologies [7, 8] which fabricate micro/nano structures on materials are potential alternatives for controlling the wettability of surfaces. Recently, a variety of studies on surface texturing have been conducted using photolithography [9], laser processing [10–16], self-organization of melted materials [17], micro indentation [18, 19], precise cutting [20–22], and electrical discharge machining [23]. Some of these studies were focused on the wettability of the textured surface and revealed that appropriate geometries fabricated on the surface could affect the contact angle of liquid droplets.

As a new alternative surface texturing method, the authors proposed angled fine particle peening (hereafter angled-FPP) [24], whereby a surface was bombarded with fine particles projected from a nozzle set oblique to the workpiece. This method can create specific "ridge" texture on a surface on which peaks and valleys align periodically. Angled-FPP enables fabrication of patterned structure in a simple manner without using masks. Because ultraprecise and expensive equipment is unnecessary to perform angled-FPP, it is a cost-efficient process. These features provide practical advantages to angled-FPP compared to conventional texturing technology. In addition, angled-FPP enables on-site treatment of large-scale objects. Thus, on-site fabrication of anti-contaminant textures upon civil structures, traffic signs, outdoor luminaries, and so on, would be predictable applications of angled-FPP. Achieving these results with other texturing technologies might be difficult. Simultaneously, angled-FPP can transfer shot particle fragments to a textured surface [25, 26]. This is another specific advantage of angled-FPP; surface characteristics can be changed by changing the composition of the shot particles. In terms of the wettability control, angled-FPP is attractive because it likely modifies the wettability of the surface by means of the patterned structure newly created, as well as by the composition of the fragments transferred.

Because surface geometries affect the wettability of a surface, it is necessary for wettability control to range the surface texture by adjusting the condition of the angled-FPP. In a previous study the authors reported that the pitch and the height of a ridge can be ranged by ranging conditions such as peening time, particle supply rate, and nozzle distance [24]. From a practical point of view in the shot peening industry, it is a common approach to choose specific shot particles to control the surface morphology of the peened surface. However, the effect of shot particle choice on the ridge creation behavior of angled-FPP has not previously been clarified.

To achieve further and more efficient control of surface texture, this study was aimed at investigating the effect of the shape and size of shot particles on texturing behavior. Various shot particles chosen from among particles typically used in the peening industry were employed for angled-FPP. In this study, these were investigated in terms of their effects on the geometrical features of the texture created. Then the effects of the ridge texture on the wettability of the surface were investigated. Contact angles of water drops on the ridge surfaces varied in the size and in the geometry were measured and compared to that on a smooth surface. The effect of the surface texture created by angled-FPP on wettability was also investigated.

2. Experimental Procedure

2.1. Angled-FPP

The material examined was AA6061 aluminum alloy. Disks of the alloy 15 mm in diameter and 5 mm thick were machined from a rod and then polished with emery paper. The polished surface was treated with angled-FPP using an air-suction-type peening device.

The experimental setup of the angled-FPP is shown in **Fig. 1**. A peening nozzle with diameter of 6 mm was set at 15° to the polished surface of the aluminum disk. It had already been determined that ridge structure transverse to the particle flow direction is created by conducting angled-FPP at this angle [24]. **Table 1** lists the details of the angled-FPP conditions.

It is well known that the size and shape of the shot particles, affect the surface topography created by conventional FPP because the particle shape is imprinted onto

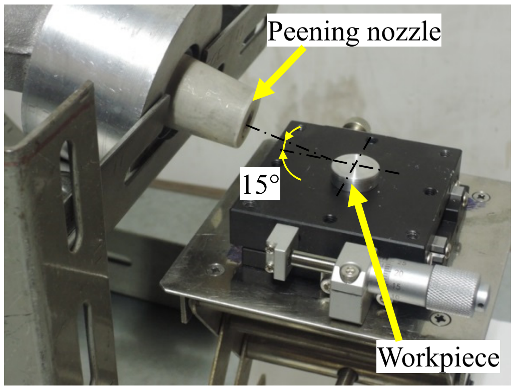


Fig. 1. Close-up view of nozzle setup in the angled-FPP apparatus.

Table 1. Conditions of the angled-FPP.

Peening angle	15°
Nozzle distance	30 mm
Shot particles	Alumina grit #360 (36 μm), Alumina grit #1000 (12 μm), Glass particle #40 (400 μm), Glass particle #200 (80 μm), Steel particle (70 μm)
Peening pressure	0.5 MPa
Peening time	30 s
Particle supply rate	5 g/s

the surface. Therefore, it is expected that shot particles with different shapes and sizes would create a variety of ridge textures. In this study, the five types of shot particles shown in **Fig. 2** were employed for comparison. There were two kinds of alumina grit (AG 36 μm and 12 μm mean diameter; grain mesh #360 and #1000), two kinds of spherical glass particles (GP 400 μm and 80 μm mean diameter; grain mesh #40 and #200), and one kind of spherical steel particle (SP 70 μm mean diameter). AG12 and AG36 grit exhibited similar sharp corners. Angular alumina grit was supposed to create ridges where relatively sharp and deep valleys were aligned. The steel particles, which have been used in previous studies by the authors, and the glass particles were spherical but differed in terms of surface appearance. The steel particles exhibited rough morphology and were partially covered with thick oxides while the glass ones had skin that was totally smooth and plain. These qualities would also affect the resulting surface texture. Hereafter, specimens prepared with angled-FPP using alumina grit, glass particles, and steel particles are referred to as: AG36, AG12, GP400, GP80, and SP70, respectively.

After conducting angled-FPP, specimens were characterized using optical microscopy (OM; VHX-700F, Keyence Corporation), scanning white light interferometry (SWLI; New View 5032, Zygo Corporation), scanning electron microscopy (SEM; TM3000, Hitachi High-

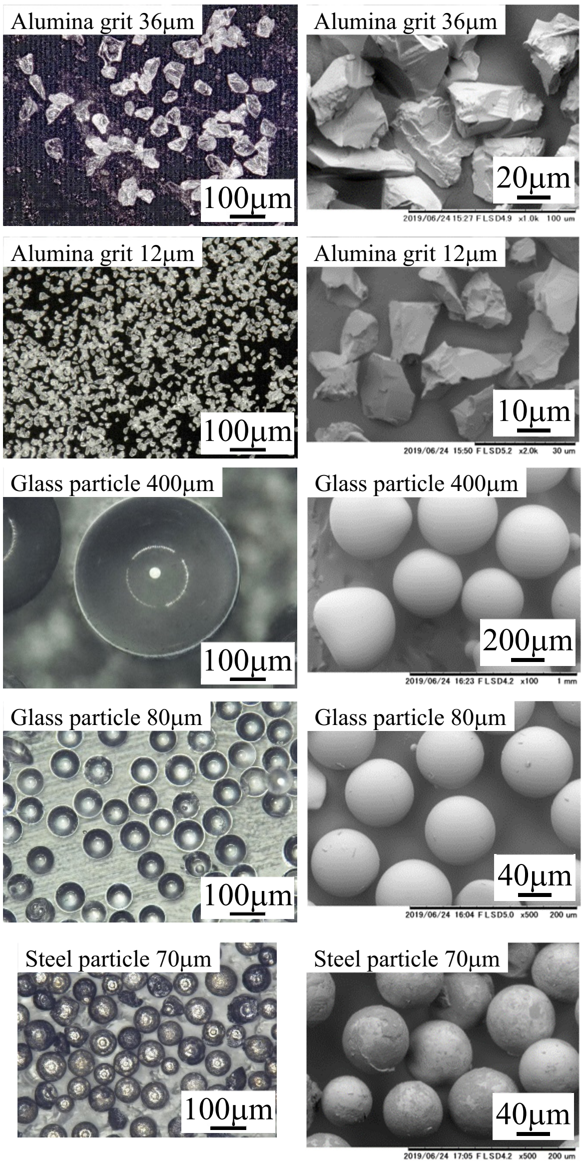


Fig. 2. OM (left) and SEM (right) images of the shot particles used for angled-FPP.

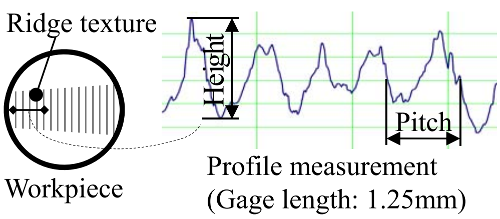


Fig. 3. Profilometry condition to determine the averaged height and the averaged pitch of the ridge texture.

Technologies Corporation), and scanning probe micrometry (SPM; SPM-9700HT, Shimadzu Corporation). Contact profilometry (Surftest SJ-400, Mitutoyo Corporation) was performed on the surface prepared with angled-FPP. The scan direction was across the ridge alignment (see **Fig. 3**). The averaged value of the pitch and the height of a ridge was determined from the profile curves of 1.25 mm gage length. Each peak and valley couple were characterized in terms of wavelength and top-to-bottom height. These results were then averaged to define the averaged pitch and the averaged height, respectively.

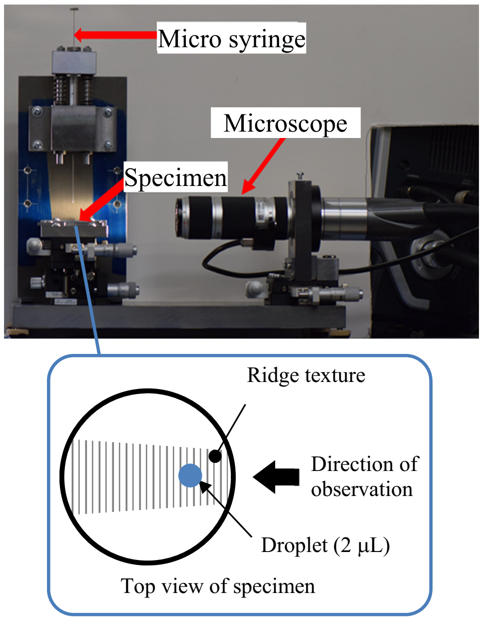


Fig. 4. Equipment and procedure used for contact angle measurement.

2.2. Wettability Evaluation

Measurement of the contact angle between a liquid and the solid surface is a common approach used to evaluate the wettability of the solid surface. In general, surfaces showing contact angles less than 90° are referred as hydrophilic, and in contrast, those greater than 90° are referred as hydrophobic.

The sessile drop method was utilized to determine the contact angle. A droplet of distilled water (2 μL volume) was put on the textured surface, and then was observed by optical microscopy. The approximate size of the droplet measured on the textured surfaces was 2 mm: much larger than the ridge pitch. Measurements were conducted from the direction of particle flow during the angled-FPP process. That is, the observation direction was transverse to the ridge alignment, as shown in **Fig. 4**. The specimens were sonicated in acetone prior to the measurement.

3. Experimental Results

3.1. Characterization on the Textured Surfaces

The typical appearances and corresponding topographic analysis results of the surfaces prepared with angled-FPP are presented in **Fig. 5**. Periodical ridge texture aligned transverse to the particle flow direction was created by angled-FPP regardless of the choice of shot particles. For the GP400 series, which was prepared by

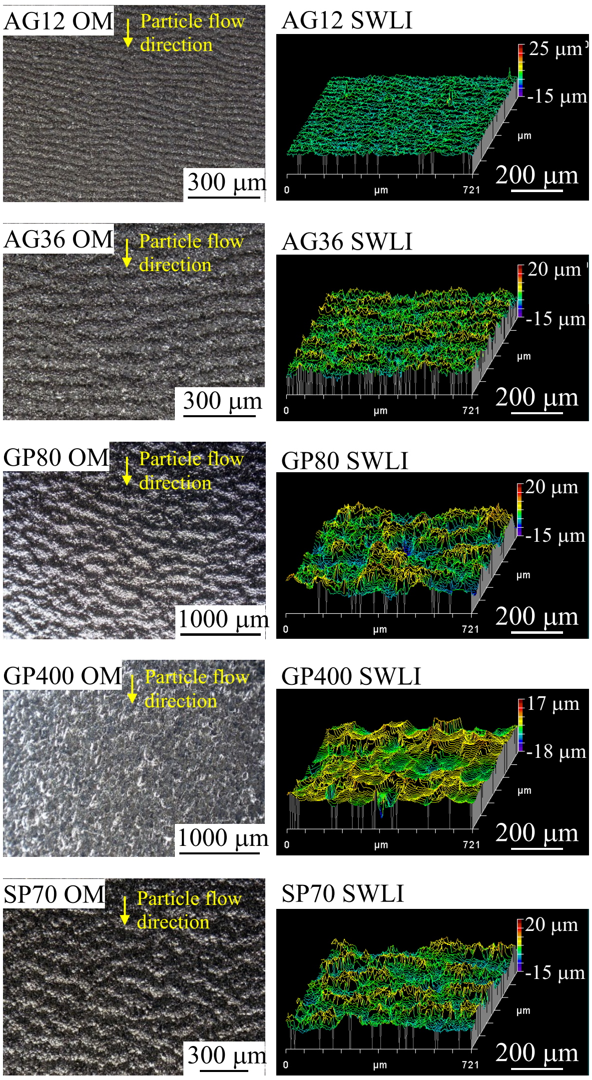


Fig. 5. Typical OM images (left) and topographic analyses results with SWLI (right).

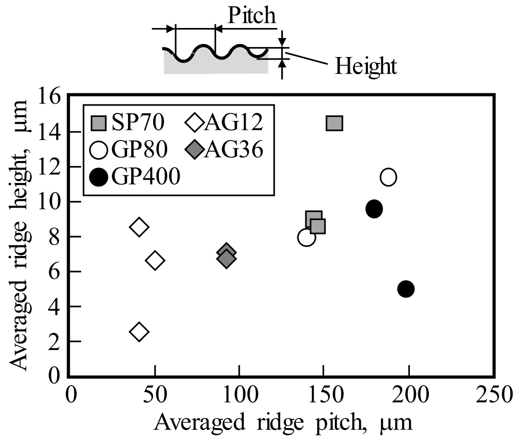


Fig. 6. Comparison in ridge geometry for each angled-FPP condition.

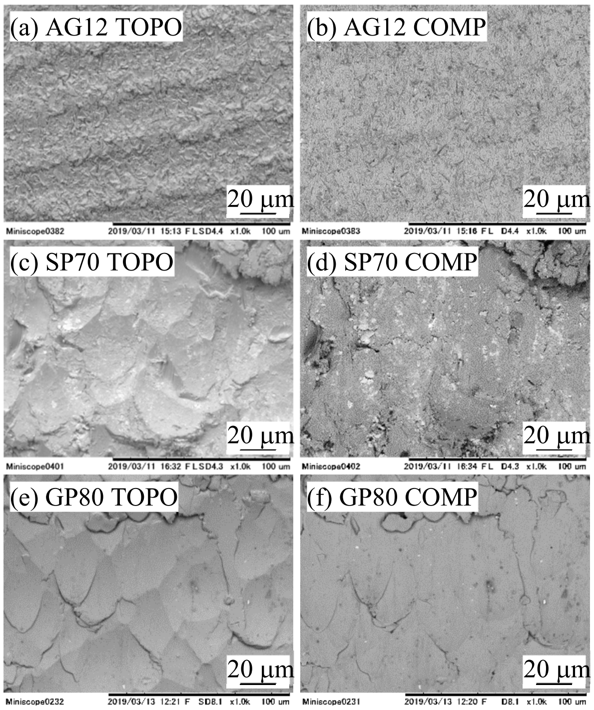


Fig. 7. Typical SEM images observed on the angled-FPP surfaces. (a) and (b) AG12, (c) and (d) SP70, and (e) and (f) GP80 series. (a), (c), and (e) are topographic views and (b), (d), and (f) are typical back scattering electron images showing compositional differences.

using larger glass particles, wave-like geometry was not clearly visible in the OM image. However, aligned peaks could be observed in the topographic map obtained from the specimen. It was revealed that ridge texture was actually created on the specimen.

Figure 6 compares the pitch and the height of the ridge texture. In the experiment, several specimens were prepared for each condition; each plot shows the sizes of the ridges determined from different specimens prepared under the same conditions. Results indicated that the geometric features of the ridge differed depending on the shape and size of the shot particles used for angled-FPP. Alumina grit, which exhibited angular shapes, created ridge texture smaller in pitch than for spherical particles. In particular, smaller grit was more suitable for aligning denser ridge texture.

Detailed observation of the textured surfaces revealed that topographic features of the ridge structure differed depending on the shot particles used to prepare the specimens. **Fig. 7** presents typical SEM micrographs corresponding to the surfaces of AG12, SP70, and GP80. The left images corresponding to each specimen show topographic views that were processed using a back-scattered-electron detector in the SEM. Tongue-like material flow is clearly visible on the SP70 and GP80 specimens. For the AG12 series, abrasion marks finer in size can be observed. The features mentioned above resulted from an abrasive effect induced by the particles, which collided with the surface from an inclined angle. The alumina grit used for AG12 was smaller in diameter and had sharp corners. These particles formed finer abrasion marks.

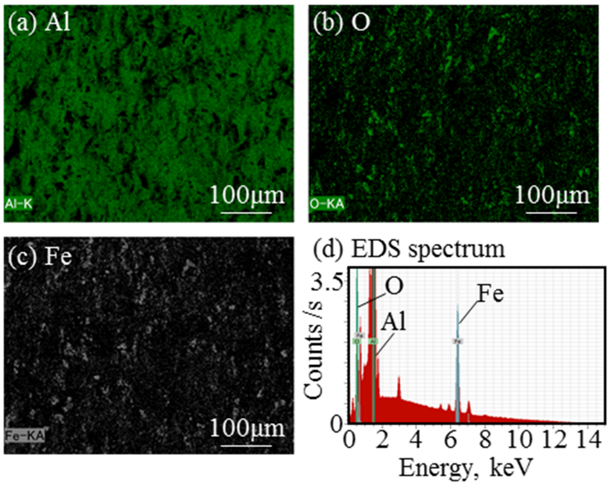


Fig. 8. Elemental maps and corresponding spectrum analyzed on the surface of the SP70 series.

The micrographs on the right in **Fig. 7** show typical back scattered electron images obtained at the same positions in each topographic view. In common, the contrast appeared in these images could be related to the different compositions of the observed materials. It should be noted that clear contrast can be observed in the SP70 series. This implied that fragments of the shot particles were transferred onto the surface of the SP70 series. A bright area in **Fig. 7(d)** should correspond to some transferred fragments. **Fig. 8** shows elemental maps of the SP70 surface analyzed by energy dispersive spectrometer (EDS). The result proved the presence of Fe elements on the surface. Because the distribution of elemental O was similar to that of elemental Fe, the iron oxide that overlaid the steel particles was assumed to have been transferred to the peened surface.

On the other hand, no significant changes in composition resulting from shot particle transfer were found to be induced for the AG12 and GP80 series because no clear contrast appeared in the back scattered electron images.

In terms of superficial appearance observed in SEM images, the AG12 and SP70 series were found to differ from the GP80 series. The surfaces of the ridge structures on AG12 and SP70 were covered with nano-scale irregularities. For detailed investigation, topographical analysis results obtained using SPM are shown in **Fig. 9**. The results demonstrated that deep crevasses occurred on the AG12 and SP70 series. These crevasses could correspond with scratch marks as well as nano-irregularities observed in the SEM images. Based on the profile curves, the depth of the crevasses was estimated to be at least several hundred nanometers deep, while accurate depth could not be determined due to saturation of the profiles. The depth/width ratio of the crevasses was estimated to be approximately 0.6 or more for the AG12 series and 0.4 or more for the SP70 series. It could be supposed that these nano-scale features were imprinted from the sharp corners of the alumina grit and rough surfaces of the steel particles, or perhaps from scratching by the particles during peening. In contrast, relatively smooth morphology was observed in the SEM micrographs and SPM map obtained for the GP80 series. Deep crevasses should be absent on the ridge surfaces created using glass particles because these have plain smooth surfaces.

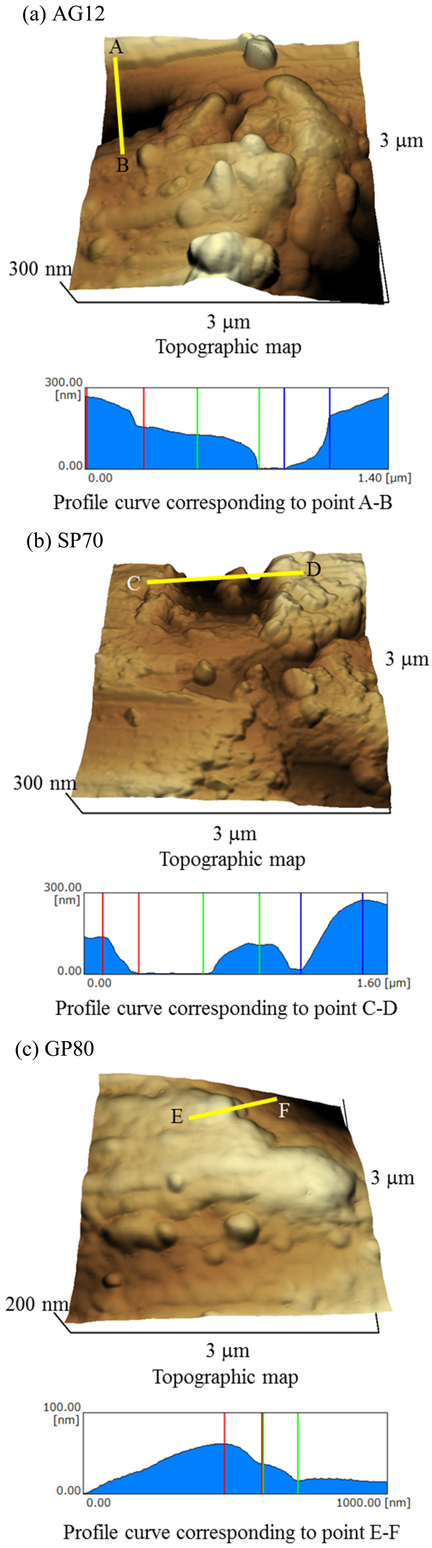
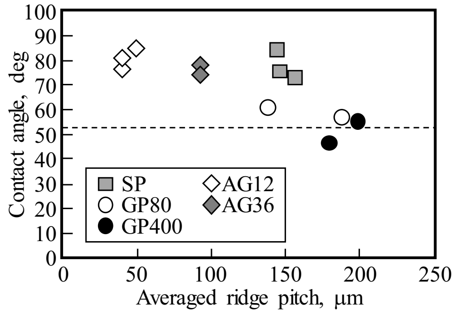
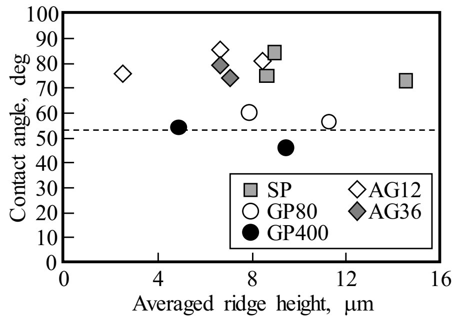


Fig. 9. Close-up topographic maps and profile curves analyzed using SPM.



(a) Relationship between the pitch and the contact angles.



(b) Relationship between the height and the contact angles.

Fig. 10. Contact angles as a function of ridge size.

3.2. Wettability of the Textured Surface

Figure 10 demonstrates the contact angle determined on the textured surface as functions of the pitch and the height of the ridge. In **Fig. 10**, the horizontal broken line represents the contact angle corresponding to the polished aluminum surface. Results indicated that the contact angle of the angled-FPP surface was affected by the shot particle employed for preparing the specimen rather than by the averaged pitch and the averaged height of the texture. The contact angles obtained on the AG36, AG12, and SP70 series specimens prepared using alumina grit and steel particles were larger than those on the polished one. Therefore, they became less hydrophilic after treatment with angled-FPP. It should be noted that no significant differences were found in their contact angle, but those specimens did vary in the pitch and the height of their ridges. In contrast, the GP400 and GP80 series showed contact angles almost the same as that of the polished sample. This implied that the ridge texture created using glass particles affected the contact angle very slightly.

4. Discussion

4.1. Factors Affecting Geometric Characteristics of the Ridge Texture

As mentioned in Section 3.1, geometric characteristics of the ridge texture changed depending on the shot particles used for angled-FPP. The pitch was affected by the size and shape of the shot particles: it decreased when angular particles and/or particles finer in diameter were used. For the AG12 and SP70 series, the ridge heights were scattered although the same shot particles were employed for angled-FPP. It is possible that this was due to minor misalignment in the nozzle setting, and resulted in a slight positional error between the shot particle stream and the workpiece that then affected ridge creation. However, the present result clearly revealed that the ridge sizes tended to range depending on the shot particles used. Further improvement in the repeatability of ridge creation will be achieved by providing more precise control in the nozzle position and peening conditions. In addition, a nozzle scan would possibly cancel the errors resulting from nozzle settings. Overall, the choice of shot particles, focusing on shape and size, was found to be an effective strategy to vary the geometries and sizes of the ridge texture.

It should be noticed that the ridge size in the GP400 series was almost the same as that in the GP80 series despite a difference in the shot particle diameters. In particular, the height of the GP400 was even smaller than that of GP80. As a result, the texture on the GP400 series was "shallow"; the ratio of height to pitch of the ridge texture was relatively low. This fact indicated that the size of the ridge texture was not simply increased, even when quite large shot particles were employed for angled-FPP. A reason to be considered for this could be the shot peening condition. In this experiment, the total mass of shot particles to be supplied for the peening nozzle was fixed regardless of the particle size. Hence, employment of larger shot particles would be associated with drastic decrease in the total number of particles that collided with the workpiece surface during the angled-FPP process. The authors considered that the ridge texture formation proceeded in three steps: bump formation beside the collision dent, self-alignment of the bump, and then piling-up of the materials at the bump resulting in growth of the ridge peaks [24]. Because projection of larger particles with fixed peening time could result in insufficient progress in the sequence described above, the resulting ridge texture got smaller in aspect ratio.

An alternative feature of the surface topography revealed by angled-FPP was a difference in the surficial morphology of the ridge texture. This resulted from the fact that the morphology and geometry of the shot particles were imprinted onto the workpiece surface. The specimens prepared using alumina grit and steel particles exhibited "hierarchical topography" because superfine irregularities such as nano-sized crevasses overlapped the wave-like geometry of the ridges for these specimens. In contrast, the ridge texture created by glass particles was smooth (i.e., these were ridges upon which no specific irregularities were observed).

4.2. Effects of Surface Topography on the Wettability of the Ridge Textured Surface

The results shown in **Fig. 10** suggest that the main threshold that varied the contact angles of each specimen, was the difference in the shot particle used for angled-FPP rather than the pitch and the height of the ridge texture. On the one hand, angled-FPP using alumina grit or steel particles increased the contact angles at the surfaces. This means that wettability could be adjusted by means of the texture for the AG36, AG12, and SP70 series. In contrast, the influence of angled-FPP on the wettability was very slight for the GP400 and GP80 series even though ridge textures were created on those surfaces. This fact implies that the existence of the wave-like geometry of the ridge texture was not the predominant factor affecting the water contact angle.

Cassie and Baxter theory [27] is a well-known model that describes the wetting behavior observed on rough surfaces. In the Cassie-Baxter model, it is considered that the liquid droplet on a rough surface is supported by both the solid phase and the vapor phase. The contact angle under this state is given in the following equation:

$$\begin{aligned}\cos\theta_R &= f\cos\theta_S + (1-f)\cos\theta_V \\ &= f\cos\theta_S + (1-f)\cos 180^\circ. \quad \quad (1)\end{aligned}$$

Here, θ_R is the contact angle on the rough surface, f is the area fraction of solid phase, and θ_S and θ_V are the contact angles of solid and vapor phases, respectively. According to this equation, the surface would exhibit a larger contact angle owing to existence of the vapor phase on the liquid-solid interface. The necessary condition for applying the Cassie-Baxter model is that the liquid phase incompletely penetrates the deep channels in the texture. Previous reports indicated that the height/pitch (h/p) ratio of the surface structure was a possible factor that might decide whether the liquid penetration into the structure was allowed or not [28].

Figure 11 represents the contact angles measured on each ridge surface as a function of the h/p ratio of the corresponding surface. Here, the ratio was calculated based on **Fig. 6**. The result indicated that the contact angle slightly increased with increase in the h/p ratio. For some of AG12 specimens on which relatively large h/p ratio resulting from the densely aligned ridge was observed, an increased contact angle might be provided by the larger aspect ratio of the texture. On the other hand, the contact angle did not depend simply on the h/p ratio: SP70 and AG36 showed larger contact angles than GP80 did, despite their similar h/p ratios.

It has been reported that wetting according to the Cassie-Baxter mode appeared in a micro pillar array structure 10 μm in height and with spacing of 12–30 μm (i.e., the h/p ratio was around 0.3–1) [18]. In another case, the Cassie-Baxter mode wetting appeared in a nanorod array structure, the mean roughness was approximately 60 nm and the ratio of root-mean square roughness to average spacing between rods was around 0.9 [28]. Compared with these studies, the h/p ratio obtained on the SP70, AG36, and GP80 specimens was significantly smaller. It seemed reasonable that the ridge texture on the GP400 and GP80 series did not increase the contact angle compared to that of the polished surface. Thus, it was supposed that the Cassie-Baxter mode theory was not

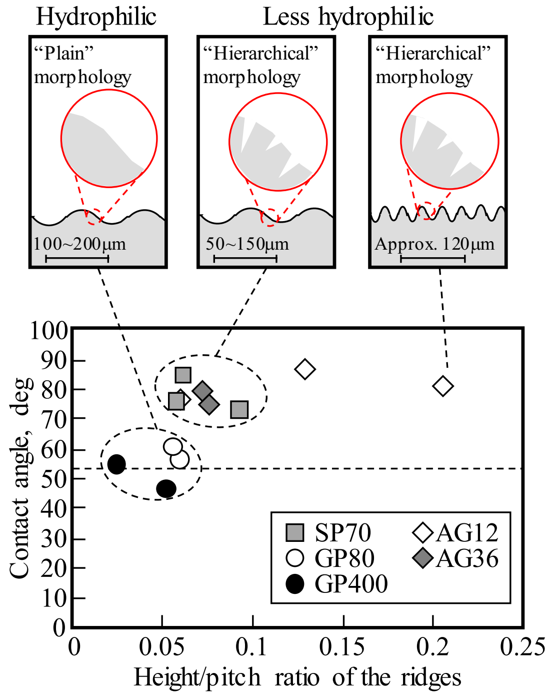


Fig. 11. Influence of geometric features of the ridge texture on wetting behavior. Contact angle as a function of the h/p ratio and schematic illustrations of nano-scale features of the texture that possibly affect the contact angle.

suitable to explain the increased contact angle of the SP70 and AG36 specimens. In addition, the sizes of the ridge created in this study were relatively larger than the structures studied in the literature. This feature should allow liquid to penetrate the wave forms of the ridge texture.

A considerable factor affecting the contact angle of the specimens with h/p ratio around 0.05 should be the nano-scale topography on each specimen. As indicated in Section 3.1 and schematically illustrated in **Fig. 11**, the textures created by using alumina grit and steel particles were "hierarchical" because they comprised submillimeter-scale ridge geometry and finer superficial crevasses. The existence of crevasses was a specific feature of these specimens. Based on the results shown in **Fig. 9**, the approximate value of the depth/width ratio of the crevasses should be relatively higher than that of the h/p ratio of the wave form of the ridges. Such crevasses likely brought about the increased contact angles measured on those specimens. Because the nano-scale topography also existed on the AG12 specimens, there should be an alternative reason for the increased contact angle of these specimens. In contrast, the ridge texture without accompanying finer topography formed by the glass particles showed contact angles similar to that of the polished (untreated) surface because both of these surfaces were quite "plain." Consequently, for the ridge structures where the pitch and the height ranged to around 100 μm and 10 μm respectively, it was concluded that the finer-scale superficial morphology of the ridge was the predominant factor that effectively varied the wettability of the surface.

Another view point about a factor to which the wettability might be attributed, could be the surface composition. As discussed in Section 3.1, the SP70 specimens were accompanied by transferred elemental Fe while no additional components were detected on the other specimens. Thus, the enrichment of Fe on the SP70 surface could be a probable factor affecting the wettability. However, the influence of Fe-enrichment on the SP70 specimen was assumed to be limited because no clear difference was observed in the contact angle compared to the other specimens without transferred shot particles. Should a material showing low surface-free-energy be employed for the shot particle of angled-FPP, achievement of a further increase in the contact angle is expected.

5. Conclusions

In this study, various shot particles were employed for angled-FPP to create a periodic ridge texture varied in size and geometry. Ridge textures created by angled-FPP using a variety of shot particles were compared and examined in terms of their wettability by measuring the contact angle for pure water. The conclusions drawn from this study are listed below.

1) The pitch and height of the ridge structure can be ranged by choosing shot particles of angled-FPP. The sizes of the ridges got finer when finer shot particles were chosen. Angular particles tended to create densely aligned texture. In this study, alumina grit of 12 μm (grain size mesh #1000) was found to be able to create the finest ridges, of which the size was about 50 μm in pitch and less than 10 μm in height.

2) The nano-scale topography of the ridge structure varied depending on the surface morphology of the shot particles. Angled-FPP using steel particles and alumina grit created "hierarchical" topography: finer roughness overlapped the wave-like geometry. Glass particles with smooth surface created ridge structure of which the skin was "plain."

3) Ridge textured surfaces exhibiting "hierarchical" topography were less hydrophilic than were the polished ones. In contrast, no clear differences in wettability were observed between the ridge structure with "plain" surface and the polished surface.

4) For the ridge textures with a submillimeter-scale pitch and h/p ratio less than 0.2, the predominant factor affecting the contact angle was assumed to be the superficial topography of the ridge texture rather than the size and geometric features of the wave-like structure of the ridge texture.

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Name:
Yutaka Kameyama

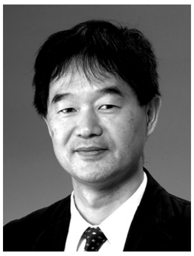
Affiliation:
Associate Professor, Department of Mechanical Engineering, Tokyo City University

Address:
1-28-1 Tamazutsumi, Setagaya-ku, Tokyo 158-8557, Japan

Brief Biographical History:
2007 Received Ph.D. from Keio University
2007- Post-doctoral Fellow, RIKEN
2010- Lecturer, Department of Mechanical Engineering, Tokyo City University
2014- Associate Professor, Department of Mechanical Engineering, Tokyo City University

Main Works:
• "Fabrication of micro-textured and plateau-processed functional surface by angled fine particle peening followed by precision grinding," CIRP Annals – Manufacturing Technology, Vol.64, No.1, pp. 549-552, 2015.
• "Effect of fine particle peening using carbon-black/steel hybridized particles on tribological properties of stainless steel," Tribology Int., Vol.78, pp. 115-124, 2014.

Membership in Academic Societies:
• Japan Society of Mechanical Engineers (JSME)
• Japan Society for Abrasive Technology (JSAT)
• Japanese Society for Tribologists (JAST)



Name:
Hideaki Sato

Affiliation:
Associate Professor, Department of Mechanical Engineering, Tokyo City University

Address:
1-28-1 Tamazutsumi, Setagaya-ku, Tokyo 158-8557, Japan

Brief Biographical History:
1987- Research Associate, Musashi Institute of Technology
1995 Received Ph.D. from Keio University
2008- Associate Professor, Musashi Institute of Technology
2009- Associate Professor, Department of Mechanical Engineering, Tokyo City University

Main Works:
• "Filtration of Machining Fluids by Microfiltration Membranes," Trans. of the Japan Society of Mechanical Engineers Series C, Vol.65, No.637, pp. 3845-3852, 1999.

Membership in Academic Societies:
• Japan Society of Mechanical Engineers (JSME)
• Japan Society for Abrasive Technology (JSAT)
• Japanese Society for Dental Materials and Devises (DMD)

Name:
Ryokichi Shimpo

Affiliation:
Professor, Department of Mechanical Engineering, Tokyo City University

Address:
1-28-1 Tamazutsumi, Setagaya-ku, Tokyo 158-8557, Japan

Brief Biographical History:
1983- Research Associate, The University of Tokyo
1986 Received Ph.D. from The University of Tokyo
2006- Professor, Musashi Institute of Technology
2009- Professor, Department of Mechanical Engineering, Tokyo City University

Main Works:
• "Mechanical Properties of Anodic Oxide Films on Aluminum," J. of the Surface Finishing Society of Japan, Vol.54, No.11, pp. 794-797, 2003.

Membership in Academic Societies:
• Japan Society of Mechanical Engineers (JSME)
• Japan Institute of Metals and Materials (JIM)
• Surface Finishing Society of Japan (SFJ)