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# Effects of scale differences of microscopic texture of fine particle peened surface on adhesion behaviour of powders 

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#### Abstract

Texture on the substrate possibly alters powder adhesion behaviour. As anti-adhesion performance was enhanced under the appropriate combination of texture size and powder size, we assumed 'similarity rule' for powder adhesion on the textured surfaces; reduction in the texture size could prevent finer-powders adhesion. This study attempts to examine this hypothesis. Fine particle peening (FPP) was used as the texture creation method. Steel particles (ave. $60 \mu \mathrm{~m}, 25 \mu \mathrm{~m}$ ) and white alumina (WA) particles (ave. $80 \mu \mathrm{~m}, 20 \mu \mathrm{~m}$, $4 \mu \mathrm{~m}$ ) were used as projectiles. Silicon powders with diameters of $5 \mu \mathrm{~m}$, $50 \mu \mathrm{~m}$, and $150 \mu \mathrm{~m}$, were employed to assess anti-adhesion performance. Results indicated the texture created with the smaller projectile showed lower anti-adhesion performance than the other for all adhering powders, except for the smallest WA particle size $(4 \mu \mathrm{~m})$. These results indicate that anti-adhesion performance does not depend solely on the texture scale.


Keywords: shot peening; fine particle peening; FPP; powder adhesion; anti-adhesion; powder detachment; surface texture; surface roughness; dimple; centrifugal force; bridge phenomenon; image processing.

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## 1 Introduction

Recently, the demand for fine powder materials has been increasing such as in the semiconductor industry and powder metallurgy industry. In powder handling, the adhesion of fine powders to the surfaces of a powder conveyor has become a great concern since it may cause contamination of the final product. Mirror finishing or PTFE coating of the base material has been used to solve this problem. However, these methods have problems in terms of wear resistance and processing time.

In this study, we focused on a functional texture on the base material to prevent powder adhesion. In general, van der Waals forces is as known as a possible factor of powder adhesion (Eichenlaub et al., 2004). Van der Waals forces acting on powders are influenced by surface roughness (Masuda, 1997). It has been further suggested that the force can be reduced by decreasing the true contact area between the powder layer and the surface (Katainen et al., 2006).

These ultra-fine powders are used in various advanced materials such as semiconductor materials (Kalyani et al., 2022), while they are difficult to handle due to their high adhesive strength. Anti-adhesion technology of powder based on such theory has been actively studied using simulation, especially for atomically-nanoscale ultrafine powder (Persson and Scaraggi, 2014) or rough surface (Pastewkaa and Robbinsa, 2014). On the other hand, powder materials of micro-metre scale are also important in fields related to powder metallurgy (Heaney, 2012) and pharmacy. However, the adhesion behaviour between the surface and micro-meter scaled powder has not yet been fully discussed, although simulation studies have been conducted (Heaney and Schmidt, 2015).

In addition, many previous studies have considered the adhesion of individual particle (Persson and Scaraggi, 2014; Haarmann and Schmidt, 2015; Braschke et al., 2022).

However, such an examination does not emulate an actual operating environment. It is necessary to evaluate the adhesion of powders under conditions in which interaction forces between powders are active.

Therefore, we would discuss the situation of the adhesion behaviour of 'micro' sized powders in 'non-individual' conditions. Even in the above situation, differences in the shape and scale of the texture affected adhesion behaviour. In fact, previous studies have suggested that surface texture possibly alters the behaviour of the powder to be adhered (Suzumoto et al., 2022). In particular, the substrate texture somewhat smaller for a given powder size effectively decreased the amount of adhesion to the substrate and/or increased the amount of detachment from the substrate. It is assumed due to the effect of reduced true contact area (Katainen et al., 2006). As described above, the relationship between the texture and the powder in their sizes is an important factor for realising anti-adhesion performance, exploring an appropriate texture size depending on the powder to be deposited is a necessary issue. Moreover, if the similarity rule works for the powder-substrate adhesion behaviour, it would contribute to the creation of a highly effective anti-adhesion texture which might differ depending on powder size.

## 2 Experimental method

### 2.1 Prepared specimens

AA1050 aluminium was machined into a rectangular shape measuring $13 \mathrm{~mm} \times 5 \mathrm{~mm} \times$ 3 mm , and one side of the surface was mirror finished using water-resistant abrasive paper as the test specimen. Fine particle peening (FPP) was employed for the texture creation method. The authors have been engaged in researching surface texturing by FPP (Suzumoto et al., 2022; Kameyama et al., 2015, 2019). Therefore, in this study, we prepared several shot particles for FPP varied in sizes was conducted to create textures with similar topographical characteristics but ranged in scales. The test specimens were mounted on a suction projection type FPP apparatus and were projected at a peening pressure of 0.5 MPa , a peening time of 15 s , a particle feed rate of $0.33 \mathrm{~g} / \mathrm{s}$, a distance from the nozzle of 50 mm , and a nozzle angle of $90^{\circ}$.

Five types of particles were employed for FPP: spherical steel particles with an average particle diameter (projected area equivalent diameter) of approximately $60 \mu \mathrm{~m}$ and $25 \mu \mathrm{~m}$, and angulated white alumina (WA) particles with an average diameter of $80 \mu \mathrm{~m}, 20 \mu \mathrm{~m}$, and $4 \mu \mathrm{~m}$. FPP with each particle was performed on eight specimens each. Hereafter, we refer to each specimen prepared with a different particle as mentioned below: specimens with steel $60 \mu \mathrm{~m}$ and $25 \mu \mathrm{~m}$ as DS1 and DS2, and ones with WA $80 \mu \mathrm{~m}, 20 \mu \mathrm{~m}$, and $4 \mu \mathrm{~m}$ as DW1, DW2, and DW3, respectively. 'D' means dimple as FPP at a nozzle angle of $90^{\circ}$ tends to produce a dimple-like texture. In addition, as machined specimen (specimen raw) and a mirror-finished specimen (specimen mirror) were prepared for comparison.

Typical SEM images of the prepared specimens and steel/WA particles are shown in Figure 1. Furthermore, Figure 2 shows the results of observation of the shape of each projected particle by optical microscope image. Figure 3 shows the profile curves and several roughness parameters for each specimen. Specimens with steel particles (DS1 and DS2) show dimple structures on their surfaces, with DS2 having smaller dimples than DS1 due to the smaller diameter of the projected particles. The profile curves also show
differences in the scale of the dimples. The specimens with WA particles (DW1 and DW2) have a crevice-like structure with sharp scratches, rather than the dimple structure observed on DS1 and DS2. A finer texture was produced on DW2 than on DW1. Finally, for the DW3 surface, where the smallest particles were used in the projection, it was revealed that an isotropic pattern was created with very small scars. The SEM image shows that R and M are flatter than the other textures; cutting marks are engraved on R . The Rsm value for M is larger than expected, probably because of the referred waviness.

Figure 1 SEM images of specimens and shot particles, (a) DS1 (b) DS2 (c) DW1 (d) DW2 (e) DW3 (f) raw (g) mirror (h) steel particle (i) WA particle (see online version for colours)


(e)

(f)

(g)

(h)

(i)

Figure 2 Shape of projectiles (average diameter), (a) steel particles ( $60 \mu \mathrm{~m}$ ) (b) steel particles $(25 \mu \mathrm{~m})(\mathrm{c})$ WA particles $(80 \mu \mathrm{~m})$ (d) WA particles $(20 \mu \mathrm{~m})$ (see online version for colours)

(a)

(c)

(b)

(d)

Figure 3 Profile curves of each texture, (a) DS1: Ra $3.0 \mu \mathrm{~m}$, Rsm $67 \mu \mathrm{~m}$ (b) DS2: Ra $2.4 \mu \mathrm{~m}$, $R \mathrm{sm} 54 \mu \mathrm{~m}$ (c) DW1: Ra $1.7 \mu \mathrm{~m}, R \mathrm{sm} 57 \mu \mathrm{~m}$ (d) DW2: Ra $1.4 \mu \mathrm{~m}, R \mathrm{sm} 52 \mu \mathrm{~m}$ (e) DW3: Ra $0.23 \mu \mathrm{~m}, R \mathrm{sm} 18.4 \mu \mathrm{~m}$ (f) Mirror: $R \mathrm{a} 0.021 \mu \mathrm{~m}, R \mathrm{sm} 29.6 \mu \mathrm{~m}$ (see online version for colours)


Figure 3 Profile curves of each texture, (a) DS1: Ra $3.0 \mu \mathrm{~m}, R \mathrm{sm} 67 \mu \mathrm{~m}$ (b) DS2: Ra $2.4 \mu \mathrm{~m}$, $R \mathrm{sm} 54 \mu \mathrm{~m}$ (c) DW1: Ra $1.7 \mu \mathrm{~m}, R \mathrm{sm} 57 \mu \mathrm{~m}$ (d) DW2: Ra $1.4 \mu \mathrm{~m}, R \mathrm{sm} 52 \mu \mathrm{~m}$ (e) DW3: $R \mathrm{a} 0.23 \mu \mathrm{~m}, R \mathrm{sm} 18.4 \mu \mathrm{~m}$ (f) Mirror: $R \mathrm{a} 0.021 \mu \mathrm{~m}, R \mathrm{sm} 29.6 \mu \mathrm{~m}$ (continued) (see online version for colours)


### 2.2 Powder adhesion method

Three types of silicon powders with mean particle sizes of 5,50 , and $150 \mu \mathrm{~m}$ were employed as powders to investigate adhesion behaviour (Figure 4). These were stored in desiccators where humidity was maintained under $20 \%$ overnight or longer to dry. After sieve classification, the powders were spread over the specimens through a wire mesh with a 1 mm mesh opening until the powders completely covered the specimens. The humidity at the time of spreading was approximately $40 \%$ or less.

Figure 4 Silicon powder (nominal diameter), (a) $5 \mu \mathrm{~m}$ (b) $50 \mu \mathrm{~m}$ (c) $150 \mu \mathrm{~m}$ (see online version for colours)


After sprinkling, each specimen was gently turned over and the powder was dropped off by gravity (Figure 5). The powder remaining on the specimen texture was then observed using an optical microscope. The entire specimen was photographed, and then the area ratio of the specimen surface where the adhered powder remained was calculated by conducting image processing (Figure 6). The initial powder adhesion rate [\%] is defined as the percentage of the specimen surface area occupied by the powder remaining in the specimen shape after re-flipping. The initial powder adhesion rate represents the ease of static detachment of the powder from the texture.

Figure 5 Dropped off by gravity (see online version for colours)


Figure 6 Example of image processing (see online version for colours)


Figure 7 Powder peeling test apparatus, (a) principal of centrifugal force method (b) actual apparatus (c) experimental setup (see online version for colours)

(a)

(Specimens exposed from under the cover)
(b)
(c)

Dynamic detachment characteristics of the powder from the surface were evaluated by using a specially developed powder peeling test apparatus shown in Figure 7. Centrifugal force method is widely used to measure adhesion forces between powder particles and substrates (Podczeck et al., 1994). This device can install 8 specimens accompanied with silicon powder at once and then rotate under controlled rotational speed. Then the powder is detached from the surface due to centrifugal force. The force to release the powder can be estimated by conducting the test under ranged rotational speed. When measuring the centrifugal force, the mass of the powder was calculated from the mean particle size and density $\left[2.33 \times 10^{-3} \mathrm{~g} / \mathrm{mm}^{3}\right]$. The test is performed at a peripheral speed of $158 \mathrm{~m} / \mathrm{min}$, and the centrifugal force received by each silicon powder particle is $150 \mu \mathrm{~m}: 455 \mathrm{nN}$, $50 \mu \mathrm{~m}: 17 \mathrm{nN}$, and $5 \mu \mathrm{~m}: 17 \mathrm{pN}$, respectively. Powder peeling apparatus was used to perform the peeling test for 60 seconds. After the test, the residual powder on the specimen surface was observed using an optical microscope. The final powder adhesion
rate [\%] is defined as the ratio of the residual powder to the specimen surface area divided by the powder adhesion rate. The powder detachment rate represents the ease of dynamic detachment of the powder from the texture.

## 3 Results and discussion

### 3.1 Results of the initial powder adhesion rate

Figure 8 shows the initial powder adhesion rate corresponding to silicon powder of $150 \mu \mathrm{~m}$. The size is larger than the scale of all textures' topography. In this figure, the error bars represent maximum and minimum values, and the plot shows the median value of each measurement. The initial powder adhesion rate tended to be smaller on the peened surfaces than on the smooth surfaces ( $\mathrm{R}, \mathrm{M}$ ). This is considered to be a result of the reduction in the contact area as schematically described in Figure 9. Focusing on the median values, specimens with larger texture sizes (DS1 and DW1) exhibited slightly lower adhesion rates compared to smaller ones (DS2 and DW2). This trend can be attributed to the finer texture, which in turn increases the contact area. The isotropic textures on DW3 have been responsible for the smaller variation in adhesion compared to the other textures. Figure 8 indicates that DW3 did not show a significant change in adhesion relative to the other specimens in the median values. This suggests that ultrafine structure does not lead to a further increase in the contact area. In addition, the much smaller texture compared to the powder size, is not advantageous to preventing adhesion.

Figure 8 The initial powder adhesion rate and the final powder adhesion rate of $150 \mu \mathrm{~m}$ (see online version for colours)


The results of the final powder adhesion rate determined from the dynamic powder peeling test are shown in Figure 8. In total, the final powder adhesion rate did not differ significantly among varied textures, regardless of the difference in the initial adhesion rate. Some of the DS1 specimens demonstrated slightly higher values in the final powder adhesion rate than others. It could be suggested that a higher adhesion rate, which meant lower anti-adhesion performance under loading, was recorded when powders are stacked
into the dimples. Based on this speculation, powders might more likely penetrate in dimples on DS1 specimens since their texture was slightly larger. Conversely, DS2, which has a smaller texture size than DS1, does not show such a high adhesion plot. Moreover, DS1 with a dimple structure and DW1 with a crevasse structure are compared. While DW1 had plots with high adhesion during the static detachment, no such plots were found after the dynamic detachment. The reason for this could be that the surface structure of DW1 is shallower than that of DS1 from Figure 3, so even if the powder gets stuck in the structure, it could be easily ejected.

Figure 9 Examples of reduction in the contact area (see online version for colours)


As described above, $150 \mu \mathrm{~m}$ silicon powder was suppressed to be adhered on DS1 and DW1, exhibiting a relatively larger texture. Then we assumed that adhesion of smaller powders should be prevented on the finer-sized texture, which might be favoured to decrease the contact area. The $50 \mu \mathrm{~m}$ powder is aimed at a scale like the 2 -series, based on the Rsm value of the texture. However, even though it is $50 \mu \mathrm{~m}$, the powder is classified using a $38 \mu \mathrm{~m}$ sieve, so the particle size could be smaller than its texture scale.

Figure 10 shows the results of the initial powder adhesion rate corresponding to silicon powder of a $50 \mu \mathrm{~m}$ grain size. Contrary to the assumption, the results again showed that the DS1 and DW1 were slightly more effective in preventing adhesion than the DS2 and DW2. One of the possible reasons for this is that the powder penetrated in the substrate texture and acted like a spike, making it difficult for the powder to be released (Figure 11). This phenomenon is hereafter referred to as the spiking effect. The size reduction in textures from 1-series (DS1 and DW1) to 2-series (DS2 and DW2) was approximately $20 \%$ while the powder size was reduced from 150 to $50 \mu \mathrm{~m}$. This difference allowed to occur spiking effect.

Concerning steel particles, the final powder adhesion rate tended to be higher for DS2 than for DS1. This difference in the dynamic detachment behaviour of powders may be attributed to the porosity fraction of the penetrated agglomerates in the dimples; the dimples on DS2 might be filled with powders more tightly may be due to the size relationship between the dimples and agglomerates. This might maintain the powders stacked in the dimples when centrifugal force was applied. These discussions imply that anti-adhesion properties derived from the texture are affected by the penetration of powders into the dimples.

Regarding the dynamic detachment results, even for $50 \mu \mathrm{~m}$ silicon particles, the final adhesion tended to be less for the WA texture. These results suggest that the crevasse structure may have better detachment performance than the dimple structure when subjected to centrifugal force. The crevasse shape, which is shallower and finely gouged than the dimple structure, is thought to increase the contact area, but it is suggested that it could be less likely to produce a spiking effect.

Figure 10 The initial powder adhesion rate and the final powder adhesion rate of $50 \mu \mathrm{~m}$ (see online version for colours)


Figure 11 Spiking effect and bridging effect (see online version for colours)


Spiking effect


Bridging effect

- Substrate

Powder

Figure 12 shows the initial powder adhesion rate corresponding to $5 \mu \mathrm{~m}$ silicon powder. The size is significantly smaller than the texture scale except for DW3. Similar to the results mentioned above, DS1 showed better anti-adhesion performance than DS2 in terms of steel particles. However, for WA particles, DW2 showed better anti-adhesion performance. Since silicon powders of this particle size are more aggregate than others, it is possible that the bridging effect (Figure 11) is more dominant than the spiking effect. In general, the smaller the particle size, the higher the cohesion (The Association of Powder Industry and Engineering Japan, 1995) and the larger the angle of repose (Zhou et al., 2002). Aggregates get larger so that are less likely to penetrate in the texture. Furthermore, aggregates that build a bridge are less likely to collapse due to gravity because of greater cohesion force (The Association of Powder Industry and Engineering Japan, 1995; Zhou et al., 2002). This is the reason why the bridging effect became dominant. The difference in texture dimensions suggests that DW2, which is more prone to bridging on the substrate, may be better at preventing the adhesion of powders of this grain size. The least amount of adhesion was observed for DW3, suggesting that DW3 has the finest texture of these particles and may have been effective in preventing adhesion even to fine powders.

For the WA surface, no clear difference in the final powder adhesion rate was observed between DW1 and DW2. On the steel surface, the final powder adhesion rate tended to be higher for DS2, even at this powder particle size. Consequently, a smaller texture size is not necessarily more effective in preventing fine powder adhesion.

Figure 12 The initial powder adhesion rate and the final powder adhesion rate of $5 \mu \mathrm{~m}$ (see online version for colours)



## 4 Conclusions

In this study, the effects of texture scale differences on powder adhesion behaviour were investigated. The following are the results revealed in this paper.

1 For powders relatively larger than the texture scale ( $150 \mu \mathrm{~m}$ ), the larger texture scale showed higher anti-adhesion performance. This result may be because a smaller texture scale conversely increases the contact points between the powders and the texture.

2 For powders $50 \mu \mathrm{~m}$ in grain size, which is approximately equivalent in size to the texture scale, the larger texture scale showed higher anti-adhesion performance.

3 For $5 \mu \mathrm{~m}$ powders, adhesion was effectively prevented on DS1 and DW2, which has a much larger scale of texture compared with the powders. Simultaneously, DW3, the finest texture employed in this study, also demonstrated better anti-adhesion performance.

4 Smaller texture dimensions are not necessarily more effective in preventing the adhesion of fine powders.

5 The results suggest that crevice-like textures shallower than dimple textures tend to be less likely to produce a spiking effect during centrifugal force application.

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