# Effect of Ambient Pressure on Flattening Behavior of Thermal Sprayed Particles

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Cu splats were thermally sprayed onto the mirror polished SUS304 substrate surface at various ambient pressures ranging from 6.66 to 101.33 kPa. The effect of ambient pressure on the flattening behavior of the particle was systematically investigated. It was observed that only around 10% or less of disk-shaped splats deposited at atmospheric pressure. The splat shape on the flat substrate had a transitional changing tendency from a splash splat to a disk one with a decrease of the ambient pressure. The tendency of splash splat change with increasing the ambient pressure agreed with the BET curve, which indicates that adsorption/desorption of the adsorbed gas/condensation plays an important role on the flattening behavior of thermal sprayed particles. Moreover, a part of substrates were polished to a mirror finish and heated to 573 K for 10 min, then elapsed to air atmosphere for different duration of up to 1 h. The fundamental static wetting behavior of the once heated substrate surface by a water droplet was investigated. The contact angle measurement results agreed well with the splat morphologies. No chemical change and surface topography change took place with the elapse time increasing. Hence, the occurrence of desorption caused by reducing the ambient pressure or by substrate preheating provided good wetting. Wetting of substrate surface by molten particles may dominate the flattening behavior of thermal sprayed particles.

Keywords	adsorption/desorption, ambient pressure, disk-			
	shaped splat, flattening, nano-pore, splash splat,			
	thermal spraying, wetting			

## 1. Introduction

With the continuing development of industry, materials are being subjected to severer and more demanding environments. Surface treatment technologies have been attracting a great deal of attention from various industries, as they present a way to get entirely different material performance from the surface of materials merely through surface treatment (Ref 1). Thermal spraying is one of the surface treatment techniques, which has been increasingly applied in various fields owing to its advantages over other surface treatment methods. It is, however, pointed out that the process controllability or reliability of thermal spraying is still insufficient. As a flattening of an individual thermal sprayed particle on the substrate is a fundamental process for the coating formation, coating properties, such as porosity and adhesion strength, depend strongly on the flattening nature of each splat (Ref 2). In order to improve the coating microstructure and its adhesive strength, it is necessary to study in detail the basic process of flattening behavior of the sprayed particle, not only of scientific interest, but also has technical consequences as well.

A transition phenomenon in a flattening behavior of the thermal sprayed particle on the flat substrate surface was introduced in 1995 (Ref 3), which reported that when the substrate temperature is increased above one critical temperature, the splat shapes of most materials sprayed onto flat substrates undergoes a transition from a distorted shape with splash to a disk-shaped splat. The transition was focused on initially that occurred by changing the substrate temperature. It is quite interesting and practically meaningful, because the transition in the splat shape of an individual particle corresponds well to the transition in the adhesion strength of the coating fabricated at corresponding temperature conditions. Namely, the transition temperature,  $T_{\rm t}$ , as a critical substrate temperature over which more than 50% of splats are disk shaped, can be considered as a useful tool for the process control. Figure 1 shows how the fraction of disk splats varies with substrate temperature for Ni particles sprayed on a SUS304 stainless steel substrate, and how the transition temperature is defined. The main topics should be investigated is a transition mechanism in the flattening behavior of the particle. Hereby, the transition mechanism has been investigated by many researchers. The possible domination for the transition behavior is recognized to be the wetting of the substrate surface by the flattening particle, adsorption, or desorption of adsorbates/condensates on the substrate surface, and so on (Ref 4).

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Moreover, existence of a similar transition was indicated recently, that is, the transition occurs by reducing the ambient pressure (Ref 5). Sampath and Herman reported that more contiguous Ni splats formed in a reduced pressure chamber than at atmospheric pressure (Ref 6). Fukumoto et al. systematically investigated the effect of desorption of adsorbates (for example, water and surrounding air) on particle flattening behavior in low pressure plasma spraying (LPPS), while the substrate temperature being kept at room temperature (Ref 5, 7, 8). Transition pressure,  $P_{\rm t}$ , was defined as the critical ambient pressure at which half of the splats were disk type. The transition pressure is characteristic of the surface: no chemical modification of the surface occurs when pressure is lowered, and desorption is the only possible physical change taking place. However, it is not fully understood yet.



Fig. 1 Dependence both of fraction of disk splat and adhesion strength on substrate temperature

Given the limited understanding on the influence of the ambient pressure on the flattening behavior of thermal sprayed particles, it was felt that there is a need for a detailed study of this aspect. In this study, the effect of ambient pressure on flattening behavior of the thermal sprayed copper particles was investigated systematically, focusing on the possible influence factors such as adsorption/desorption of adsorbed gas/condensation and wetting of substrate surface by molten droplets.

## 2. Experimental Procedures

## 2.1 Thermal Spraying Apparatus and Materials Used

Plasma spraying was carried out by LPPS. The schematic of the apparatus is shown in Fig. 2. The spraying conditions are summarized in Table 1. Powders were sprayed onto the substrate kept at various ambient pressure conditions in LPPS chamber. Spraying in LPPS was conducted at the designated pressure after once evacuated to the lowest pressure condition of the equipment. During deposition, the substrate surface was held vertically and spray gun was held horizontally so that the direction of droplet stream was perpendicular to substrate surface. For the particle collection, both of the fixed steel slit with Ø10 mm hole and the moving graphite shutter with

#### Table 1 Plasma spraying conditions

Chamber pressure, kPa	6.66-101.33
Spray distance, mm	150
Arc current, A	800
Arc voltage, V	40
Operating gas flow rate, L/min	
First: Ar	50
Second: He	12
Powder carrier gas: Ar, L/min	4
Powder feed rate, g/min	6
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Fig. 2 Schematic of LPPS equipment



Fig. 3 SEM image of Cu powder used in LPPS process

 $\emptyset$ 32 mm hole were installed between the plasma torch and the substrate in order to collect the particles having the homogeneous thermal and velocity hysteresis. Particles were collected on the substrate by moving the shutter rapidly in one direction. The number of the particles deposited on the substrate in one passing of the shutter was around 50 or more, and almost no increase of the substrate temperature was recognized by this collection.

The mirror polished SUS304 plate with 20 mm  $\times$  20 mm  $\times$  5 mm was used as the substrate. The plates were polished with 0.3 µm alumina (Al<sub>2</sub>O<sub>3</sub>). Commercially available pure copper (Cu) powder with 75 µm or less in diameter was used for the spraying (Kojundo Chemical Lab. Co., Ltd., Japan.) as shown in Fig. 3.

#### 2.2 Evaluation Methods

The splat shape was distinguished from the Nikon Eclipse LV100 optical microscope (OM) images (Nikon Co., Ltd., Japan). Around 50 or more splats were collected, observed on the substrate on every trial in the experiments, and the number of disk splats and splash splats were counted at each condition. The ratio between the number of disk splats and the total splats was defined as the fraction of disk splat. The top surface morphologies of the splat collected at different ambient pressures were observed by scanning electron microscope (SEM). (JSM-6390TY JEOL, Co., Ltd., Tokyo, Japan). Following this, carbon tape was pressed of the sprayed region, and then pulled off, some splats were removed. The bottom surface of the splat collected on carbon tape was examined under SEM (Ref 9). Moreover, the cross section morphologies of the splats were observed as well. Image analysis (UTHSCSA Image Tool) was employed to quantify pore size and distribution on the bottom surface of the splats.

Nanometer scale surface topography of the substrate may affect friction coefficient between sprayed particle and substrate surface, flattening speed, and thermal conduction. In this study, surface topography and surface roughness of the mirror polished SUS304 substrate before/after preheating in nanometer scale was measured by atomic force microscope (AFM) (SPM-9500J3, Shimadzu Co., Ltd. Tokyo, Japan) covering an area of  $1 \ \mu m^2$ . Moreover, to investigate the effect of re-adsorption with the elapsed time on the flattening behavior of the sprayed particle, substrate materials after heat treatment were exposed to air atmosphere at room temperature. As the substrate material used in this study is stainless steel, it is estimated that no significant oxidation occurred by holding under this situation. Hence, surface roughness of the substrate after keeping in an atmosphere was not re-measured in the experiment.

As the flattening behavior seems to be affected by the wetting, and the wetting is characterized by the contact angle, the contact angle of the water droplet was measured experimentally, based on the standard sessile drop test. Commercially available interface measurement and analysis system (FAMAS), Drop Master 300 was introduced in this study. The droplet volume was  $10^{-3}$  mL, drops were released by a micropipette onto the surface and profile photographs were taken using a camera attached to an optical microscope. The tests were conducted in air at room temperature. Dedicated software was used to calculate the wetting angles.

### 3. Results and Discussion

#### 3.1 Splat Morphologies

To clarify the effect of ambient pressure on flattening behavior of thermal sprayed particles, splats collected at different ambient pressures were prepared, while keeping all the other conditions constant, the top surface, bottom surface and cross section morphologies were observed by SEM in this study.

The top surface morphologies of the Cu particles collected at different ambient pressures are shown in Fig. 4(a) and (c). From the figure, it is clearly observed that with the reduction of ambient pressure, the splat pattern changed from the form with splashing to the one without splashing. The splat obtained at low pressure condition performs a perfect disk-like shape, the diskshaped splat is formed by the continuous and stable flow from the impingement center of the particle. While splash occurred by increasing the ambient pressure, splash fingers were found around the central part, the splash splat consists of a small central disk as well as a splash region, the splash region is not always connected to the center disk. In addition, the central solidification area of the splat was enlarged by decreasing the ambient pressure and the final radius of the splash splat is larger than that of disk-shaped splat. This suggests that rapid flowing of the liquid film of the metal from the impingement center of the splat to its periphery occurs during the flattening of the splash splat. Higher magnified view of the center solidification part was observed as shown in Fig. 4(b) and (d). Nano-pores with smaller diameter can be found from the splat collected at low pressure condition. In addition, the smooth periphery can be attributed to the reduction in the ambient pressure. In contrast, serrated morphologies exist at high pressure condition.



Fig. 4 Top morphologies of Cu particles sprayed onto SUS304 substrate at different ambient pressures



Fig. 5 Bottom surface of Cu splat on SUS304 substrate obtained at different ambient pressures

In order to study the nano-pore more clearly, the precise observations for the microstructures of the bottom surface of Cu splats were conducted. Example of SEM observation result of the particle with splashing obtained at the ambient pressure of 101.33 kPa is shown in Fig. 5(a) and (b), numerous pores can be observed at the bottom surface of the splat. However, it was found that the nanopores separate with each other. The observed pores should

be generated by the existence of adsorbed gas/condensation at the interface between melting particle and substrate during the flattening and solidification process. On the other hand, the observed result of the splat without splashing obtained at the ambient pressure of 6.66 kPa is shown in Fig. 5(c) and higher magnified bottom view of the center part of the splat in Fig. 5(d). In the figures, almost no pore can be observed and solidification structure looks quite homogeneous.

The image analysis was carried out based on the high magnification image of bottom surface of the splat collected at different ambient pressures as shown in Fig. 6. The results are summarized as Table 2. Figure 6(a) and (b) shows the pores of Cu splats sprayed onto SUS304 substrate at atmospheric pressure and low pressure condition, respectively. Note that the splat collected at low pressure produces a lower pore amount, together with lower mean value of pore size and area fraction.

The cross section morphologies of Cu splats sprayed onto SUS304 substrate at different pressure conditions are shown in Fig. 7. Splat collected on substrate located at high pressure condition shows that no good contact with the substrate, indicating its intrinsic weak adhesion at interface, while with decreasing the ambient pressure, an increase in the physical contact was recognized. More intimate contact at interface is attained from the cross section observation, which indicates that better heat

 Table 2 Results of bottom surface analysis

Ambient	Count	Total	Average	Area
pressure,		area,	size,	fraction,
kPa		μm <sup>2</sup>	μm <sup>2</sup>	%
101.33	2140	314.26	0.147	42.6
6.66	144	1.549	0.011	0.3



Fig. 6 Image analysis results of bottom surface collected at different ambient pressures



Fig. 7 Cross section of Cu splat on SUS304 substrate obtained at different ambient pressures

transfer efficiency can be expected, followed by a higher cooling and solidification rate during the flattening and solidification process.

#### 3.2 Discussions of Adsorption/Desorption

Figure 8 shows the fraction change of the disk splat on the substrate surface with ambient pressure reduction. As clearly shown in this figure, a significant transition tendency was recognized by reducing the ambient pressure. The transition pressure,  $P_t$ , for the Cu/SUS304 combination is 43 kPa, which indicated that half of the splats were disk type at this pressure.

It is believed that no chemical modification of the surface occurs when the pressure decrease and desorption is the only possible physical change taking place on the surface. It is known that water and other substances can be adsorbed on clean solid surface, the most common condensate is water from moisture. Desorption tends to occur when the substrate temperature rises and ambient pressure decreases. In actually, often molecules do form multilayers, that is, some are adsorbed on already adsorbed molecules. BET isotherm (Ref 10) as shown in Fig. 9(a) suggests that lack of a true chemical bond between adsorbed gas molecular and substrate except the first layer, so that the physical adsorption can be removed easily by substrate preheating or by reducing the ambient



Fig. 8 Fraction change of disk splat with ambient pressure reduction

pressure. Most metal surface exposed to ambient atmosphere will be oxidized to cover a thin oxide film with a thickness over several nanometers. The adsorption to a metal surface will occur usually through the oxide film of the metal at ambient atmosphere.

In this study, when the SUS304 were located in air atmosphere, a chemical adsorption layer and a physical adsorption molecular layer exist on the surface. It is well established that clean surfaces energetically attract foreign species, which results in adsorption and condensation of molecules. Condensed volatiles vaporize when the vapor pressure is lower than the saturation vapor pressure at a given temperature. The evaporation rate increases with temperature and also with a decrease in partial pressure of the condensed species. In general, the equilibrium volume of an adsorbate sharply decreases with an increase in temperature and a decrease in pressure. Multilayer adsorption can take place and the adsorbed volume can be very high and possibly go to infinity as the gas begins to liquefy (Ref 11, 12). Kinetics of adsorption and desorption are mainly controlled by the molecular jump frequency and activation energy, which are determined by species, surface characteristics, volume of adsorption, temperature, and pressure. When the pressure of the system at equilibrium adsorption/desorption decrease, desorption will be dominant until equilibrium is achieved. When the pressure of the system at equilibrium adsorption/desorption increase, adsorption will be dominant until equilibrium is achieved.

When a molten droplet impacts on a polished substrate surface, the heating of molten droplet to the substrate occurs because the heat flow from the droplet to the substrate occurs simultaneously (Ref 13). Desorption of adsorbed water on the substrate surface occurs to form a gas barrier between the splat and substrate due to the intensively rapid heating of the substrate surface. As a result, the physical contact between the splat and substrate reduced. The splat spreads on the gas barrier to a very thin sheet and then easily broken up (Ref 14, 15). In the current experiments, when the substrates were located at low pressure environment, the physical adsorbed water and other condensates, along with a significant fraction of adsorbed species, were removed from the substrate rapidly prior to spraying, resulting in improvement of the physical contact between the splat and substrate as proved in the



Fig. 9 (a) BET isotherm and (b) fraction change of splash splat with ambient pressure increasing

bottom surface and cross section morphologies observations (Fig. 5-7), which enhanced the formation of diskshaped splats (Ref 11, 13). Several mathematical models (Ref 6, 16) were also developed to estimate the heat conduction, cooling, and solidification behaviors between the splat and substrate. By reducing the ambient pressure, most of the physical adsorbed water and other condensates can be removed. Therefore, more intimate contact can be expected and lead to lowering in the thermal contact resistance, which is followed by the high cooling and solidification rate of the splat at low pressure condition.

The previous studies always consider about the fraction of disk splat which can improve the coating properties. In the current study, the fraction change of the splash splat on the substrate surface with ambient pressure increasing was calculated as shown in Fig. 9(b), it is clearly recognized that the fraction of splash splat increase significantly with increasing the ambient pressure, which agrees with the BET isotherm (Fig. 9a). This indicates that the adsorption/desorption of adsorbed gas/condensation plays an important role on the flattening behavior of thermal sprayed particles.

#### 3.3 Discussions of Wetting

It is believed that a good wetting can be obtained by removing the adsorbed gas/condensation by ambient



Fig. 10 SUS304 substrate thermal treatment history

pressure reduction. However, it is difficult to detect the wetting behavior of the sprayed particles directly at low pressure with the current technology, because the flying particle has a high temperature and high velocity, the flattening and solidification occurs in a very short time. Instead, the wetting behavior of water droplet onto flat surface of the once heated metal substrate was investigated. As the contact angle provides an inverse measure of wettability (Ref 17), the contact angle of water droplet was measured on the SUS304 substrate with different elapsed time after preheating.

A part of substrates were polished to a mirror finish and heated to 573 K for 10 min, then exposed to air atmosphere for different duration of up to 1 h as shown in Fig. 10. First of all, to confirm the effect of substrate preheating on the substrate topography and surface roughness change, these substrates were observed on their surfaces by atomic force microscope (AFM) covering an area of 1 µm<sup>2</sup> as shown in Fig. 11. It was clearly recognized that no significant topography and surface roughness change took place. The SUS304 substrate after heat treatment were stored for appropriate periods in an atmosphere, namely, at room temperature not in high humidity, but in a dry condition. As the substrate material used in the study is stainless steel, it is estimated that no significant oxidation occurred by holding under this situation.

Figure 12 shows the typical contact angle measurement results of water droplet on mirror polished SUS304 substrate with different elapsed time in an air atmosphere after preheating. The contact angle of water droplets was 91.6° on the substrate as-polished condition. Meanwhile, the effect of adsorbate/condensate on the wetting by preheating was investigated. To do this, the static contact angle of water droplet was measured on the substrate once heated to 573 K for 10 min and kept in an air atmosphere at room temperature for a long time. In other words, the effect of elapsed time after preheating on the static contact angle change was investigated. Elapsed time was 1, 4, 24, and 48 h, respectively. One hour after preheating, the smallest contact angle was maintained. When 4 h or more time had passed since the substrate was heated, however, the contact angle began to increase. Finally, the contact



Fig. 11 SUS304 substrate topography and surface roughness without/with preheating

angle increased near the value before preheating over 48 h and more. Measurement result of Ra of once preheated substrate kept in an air atmosphere at room temperature for a long time revealed that the value was almost the same as the substrate just after cool down from heating, regardless of the elapsed time (Ref 18). Thus, some other factors, for example, change in the condition of adsorbates/condensates on the substrate surface, may have an effect.

A careful study of adsorbates and condensates at the substrate surface has been done by Li et al. (Ref 13). Often, water is the main component at the surface. Its adsorption on the surface is usually the result of one of three possible mechanisms, or combinations of them, depending on the temperature of the measurement, intrinsic reactivity of the surface, and the number of defect sites at the surface:

- 1. Physisorption of molecular water;
- 2. chemisorption of molecular water; and
- 3. chemisorptions of molecular followed by dissociation.

Physisorption corresponds to very weak interaction between the substrate and adsorbates, while chemisorption is much stronger. When the substrate is heated adsorbed, water species will be desorbed. Physisorbed molecular water is completely removed by preheating up to 423 K (Ref 19). However, with chemisorbed water, the



**Fig. 12** Relationship between the static contact angle of water droplet on SUS304 substrate and elapsed time after preheated to 573 K for 10 min

thermal desorption temperature depends on adsorbed features and the subsequent product. In this study, physisorption is completely removed by the substrate preheating, good wetting can be obtained on the cleaned substrate surface, so that the static angle significantly decrease on the once heated substrate. With the substrate elapsed to atmospheric pressure, the re-adsorption will be occur and recover the surface gradually, that is the reason why contact angle increase gradually with the increasing of elapsed time.

It is believed that similar re-adsorption occurs by increasing the ambient pressure. In other words, desorption takes place just by reducing the ambient pressure, good wetting can be expected in this condition. However, this is just an assumption in the current study, experimental evidence is necessary in future study.

As a confirmation to the above, thermal sprayed Cu particles were collected on the once heated substrate with different elapsed time in an atmospheric pressure as shown in Fig. 13. Different splat morphologies were observed. It is observed that the splats prepared on the unheated substrate (Fig. 13a) have highly fragmented shapes which generated a large quantity of debris, the splash fingers always do not connect with the center solidification area. Splats prepared 1 h after substrate preheating (Fig. 13b) show a contiguous, disk-like shape, only few short splash fingers can be observed even at atmospheric pressure. Splats prepared 48 h after substrate preheating show different morphologies (Fig. 13c). Most splats show a uniform morphology with clear flow pattern and long projections along the periphery of the splat. However, the splash fingers always connect with the center solidification part without fragmentation, which can be treated as the development of the short splash fingers obtained 1 h after substrate preheating. The splat morphologies agree well with the contact angle measurement results, which indicate that wetting of substrate surface by molten particles may dominate the flattening and solidification behavior of thermal sprayed particles.

The common fact through all observation results mentioned previously is that the disk-shaped splat appeared only on the substrate once heated in air and within a short period after the heating at atmospheric pressure. Namely, elapsed time in a scale of hour may bring about some kind of change on the substrate surface condition. It is suggested that one of the probable



Fig. 13 Top surface morphologies of splats collected on the substrate without/with preheating at air atmosphere

dominating factors in the flattening behavior may be the adsorbate/condensate on the surface by preheating the substrate (Ref 18), which results in a good wetting but not the surface roughness character or topography change. However, as the existence of adsorbates on the substrate surface cannot be detected directly in most practical case, the role of the factor has to be investigated more precisely in future study. Moreover, the contact area between the metal droplet and the substrate shifted arbitrarily prior to final solidification during the deposition process, but many aspects of the dynamic wetting are still unclear at present, intensive research in future is necessary.

## 4. Summary

To evaluate the effects of ambient pressure on flattening behavior of thermal sprayed particles by the low pressure plasma spray technique, the Cu particles flattening and solidification on SUS304 substrate have been investigated systematically. The results obtained in this study are summarized as follows:

- (1) Adsorption/desorption of the adsorbed gas/condensation plays an important role on the flattening behavior of thermal sprayed particles.
- (2) Good wetting generated by removing the adsorbed gas/condensation through substrate preheating or by reducing the ambient pressure, may dominate the flattening behavior of thermal sprayed particles. However, the contact area between the metal droplet and the substrate shifted arbitrarily prior to final solidification during the deposition process, but many aspects of the dynamic wetting are still unclear at present, intensive research in future is necessary.
- (3) By selecting the optimum operating conditions both in substrate temperature and ambient pressure in thermal spraying, we can control the coating microstructure and the corresponding properties of the fabricated coating.

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