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Flattening Mechanism in Thermal Sprayed Nickel Particle Impinging on Flat Substrate Surface

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The transition behavior of the splat pattern of nickel particles sprayed onto a flat substrate was investigated. Auger analysis and scanning electron microscopy observation of splats on a gold- coated substrate were examined. It was confirmed that splashing was not formed by flowing on the substrate surface from the impingement center to the periphery, but by jetting away from central disk. The etched splat surface revealed that the bottom part of the central disk of the splat solidified quite rapidly just after impingement onto the cold substrate. The splash pattern was found only in a direction perpendicular to the scratch pattern on the substrate. Therefore, it was confirmed that splashing was caused by some deterrent to the liquid flow, for example, due to effects such as poor wettability at the flow tip or initial rapid solidification of the splat. The drastic change of the splat pattern near the transition temperature seems to occur when the We number of the liquid flow coincides with some critical value.

Keywords disk splat, flattening, splash splat, substrate temperature, thermal sprayed particle, transition temperature

1. Introduction

The flattening behavior of thermal sprayed particles on flat substrate has been of great concern, and many theoretical (Ref 1, 2), analytical (Ref 3, 4), and experimental (Ref 5-8) studies have been conducted. In most of this research, the flattening behavior of the particle is usually evaluated by the flattening degree, $\xi = D/d$, that is, the ratio of the diameter of original spherical particle, d, to that of final disk splat, D. Speculation of the flattening degree is useful even from the practical viewpoint. However, as already shown in previous reports (Ref 5, 6), most metallic particles exhibit a drastic change of the splat pattern from a "splash" splat to "disk" splat on increasing the substrate temperature. A splash splat is defined as a star-shaped splat, whereas a disk splat is a regularly shaped disk splat.

As many properties of the sprayed coatings, especially the adhesive strength to the substrate, depend remarkably on the splat behavior of the individual particle (Ref 9), substrate temperature becomes one of the dominating factors for controlling coating properties. Moreover, investigation of the flattening mechanism of the sprayed particle is significantly meaningful in the practical use of the thermal spray process. The studies previously mentioned, however, have not clarified the transition phenomenon or splashing mechanism as the substrate temperature increases.

This study investigated the splat behavior of nickel particles sprayed onto a flat substrate to clarify the flattening mechanism.

2. Experiments

Commercially available pure nickel powder, 44 to 45 μm (Showa Denko K.K., Japan) was sprayed. The substrate materials were mirror polished AISI 304 stainless steel, mild steel, titanium-alloy, aluminum-alloy, and glass (25 by 25 by 5 mm). To investigate the effect of thermal conductivity of the substrate on the flattening of the particle, these substrates were gold coated by physical vapor deposition (PVD). Nickel, titanium, and aluminum PVD coated AISI 304 steel substrates were also prepared to investigate the effect of wetting on the flattening of the particle. Plasma spraying was conducted in an air atmosphere on the substrate whose temperature was held at the designated temperature for a few minutes. Table 1 shows spray conditions.

Figure 1 shows a schematic of the particle collecting apparatus. In this apparatus, both the fixed steel slit with a 10 mm hole and the moving graphite shutter with a 32 mm hole were installed between the plasma torch and the substrate in order to

Table 1 Plasma spraying conditions

Conditions	Value
Primary operating gas: N_2, L/min	70.8
Secondary operating gas: H_2, L/min	4.72
Arc current, A	600
Arc voltage, V	70
Powder carrier gas: Ar, L/min	5.0
Powder feed rate, rpm	7.0
Spray distance, mm	200

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collect the homogeneous thermal and velocity characteristics of the particles. 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 pass of the shutter was around fifty, and almost no increase of the substrate temperature was detected by this collection method. The substrate was heated by the hot plate, and its temperature was measured with a K-type thermocouple.

3. Results and Discussion

3.1 Transition Behavior of the Splat Pattern

Figure 2 shows the typical transition behavior of the nickel splat pattern on a stainless steel substrate with an increase in the substrate temperature. The fractional change of the disk splat with respect to substrate temperature is also shown. The fraction of disk splat was given by counting the number of disk splats in the whole splats on a measured substrate surface area. From the results of several trials of the measurement, the reproducibility of this method was confirmed. It is recognized from the figure that the change to a disk splat from a splash splat increases with respect to the substrate temperature, T_s. The transition temperature defined by Fukumoto et al. (Ref 5), T_t, at which the splat pattern changed from a splash to a disk, was approximately 610 K. This drastic change in the splat pattern near the transition temperature is a quite unique phenomenon.

Both velocity and temperature of the particle, substrate temperature, and wetting at the particle/substrate interface are dominant factors for the flattening behavior of the particle. In this case, however, as the conditions relating to the particle are constant, both particle solidification and interface wetting affect the flattening behavior of the particle on changing the substrate temperature.

3.2 Effect of Particle Solidification and Interface Wetting

To check the effect of particle solidification on flattening while keeping wetting at the particle/substrate interface constant, the nickel particle flattening behavior was observed on various substrate materials. A gold coating was made by the PVD process on the substrate materials in order to keep the wetting condition constant. Figure 3 shows the relationship between thermal conductivity of various substrates and the transition temperature. There is a tendency for the transition temperature to be higher when thermal conductivity of the substrate is greater. The particle is easy to splash for a higher transition temperature; therefore, splashing tends to occur when solidification is more likely.

The flattening behavior of nickel particles was investigated on the specimens with PVD films of several metals on AISI 304 steel substrate to assess the effect of wetting at particle/substrate interface. Figure 4 shows the relationship between the PVD film material and the transition temperature. This indicates that the transition temperature of less active metals such as gold and nickel is low, while that of active metals such as aluminum and titanium is high. Auger analysis of the surfaces of heated PVD films of various metals showed that all films except gold were oxidized. The result suggests that the particle/substrate wetting in this study is similar to the wetting behavior of pure nickel particles to various metal oxidized films. It can be pointed out that the wetting of liquid metal to a solid oxide is related to the thermodynamics of oxide materials, that is, the more thermodynamically instable the oxide, then the wetting behavior becomes easier (Ref 10). Figure 5 shows the relationship between standard formation free energy of each PVD film metal and transition temperature. This figure indicates that a strong correspondence exists between the standard free energy of formation and the transition temperature; splashing is more difficult when wetting between the particle and film is better.

3.3 Splashing Mechanism

As shown in Fig. 2, the final radius of the splash splat is larger than that of the disk 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 flattening of the splash splat. Possible reasons for the rapid flow of the liquid film include (a) sliding of the liquid flow due to the poor wettability at the liquid/substrate interface, (b) interference and jetting away of the liquid flow due to some barriers to the liquid flow, and (c) explosion by the shock wave generated in the impinged spherical liquid.

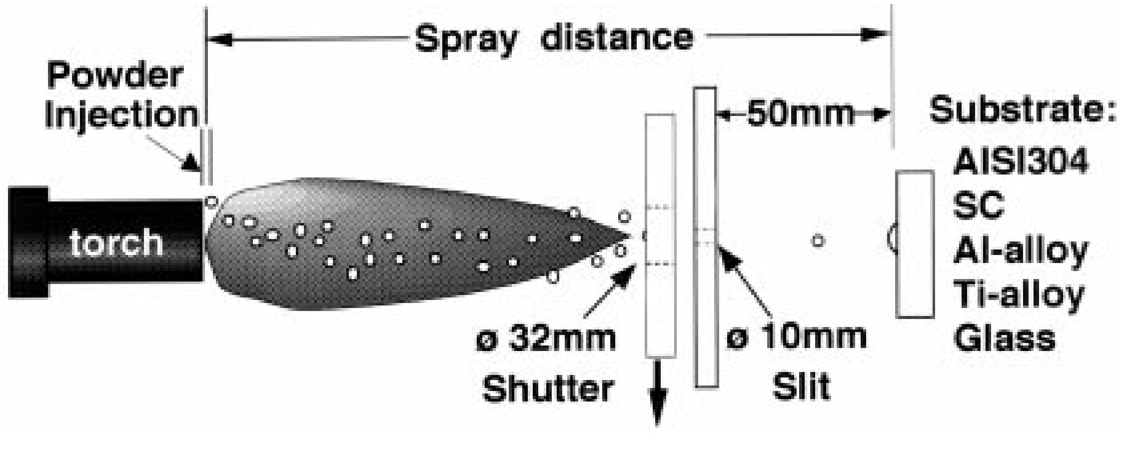

Fig. 1 Schematic drawing of splat collection

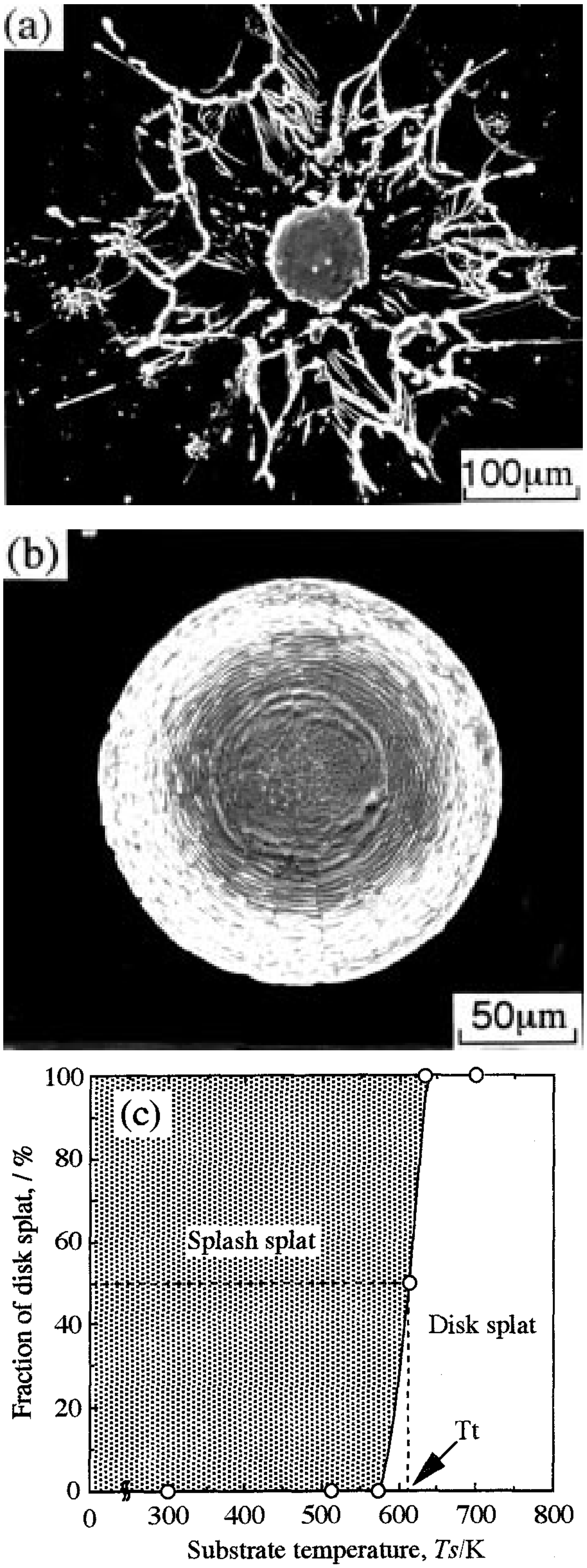

Fig. 2 Nickel splat morphologies on AISI 304 substrate. (a) Splash splat, T_S = 300 K. (b) Disk splat T_S = 673 K. (c) Fraction change of disk splat with substrate temperature

While the disk splat is formed by the continuous and stable liquid flow from the impingement center of the particle, the splash splat consists of a small central disk as well as a splash region. Moreover, as shown in Fig. 2, the splash region is not always connected to the central disk. In order to investigate the flow behavior of the flattening metal splat on the substrate surface, Auger analysis of the splash splat on the mild steel substrate was conducted (Fig. 6). From the figure, while nickel was clearly detected at the central disk or the splash part indicated as No. 1 or 4, it was not detected at positions of 2, 3, and 5. This indicates that the substrate surface was exposed at these positions. Figure 7 shows scanning electron microscope observation of the nickel splash splat on the coated substrate. From the figure, it is recognized that the gold film is torn at the position impinged by the nickel splash, while the film still remains on the region between the central disk and the splash. Thus, the splash was not formed by flow on the substrate surface from the impingement center to the periphery, but by jetting away from the central disk.

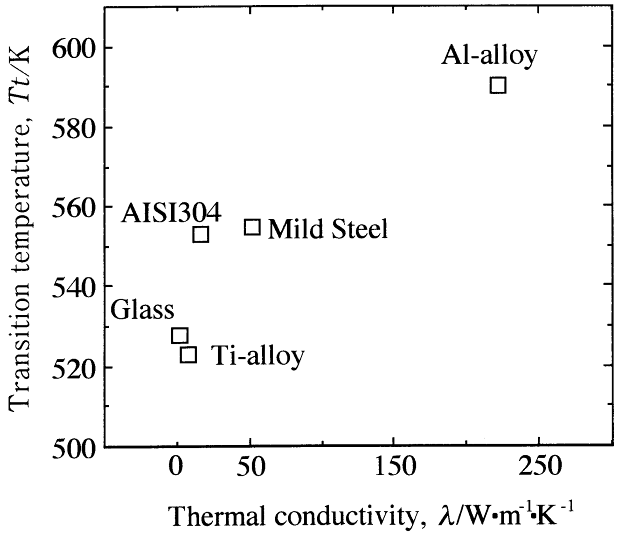

Fig. 3 Relationship between thermal conductivity of substrate and transition temperature

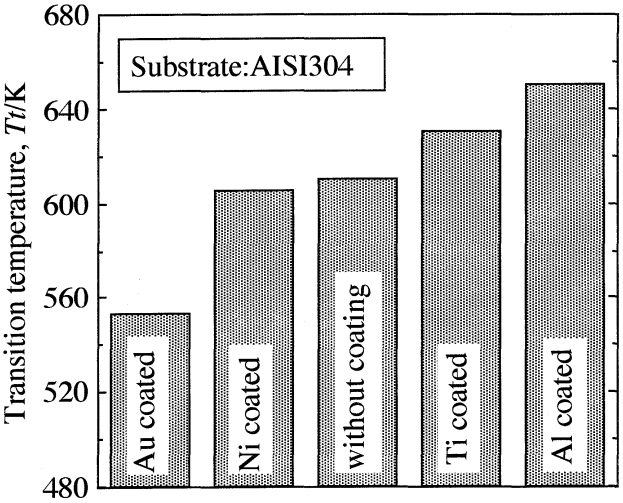

Fig. 4 Transition temperature dependence on physical vapor deposition film material

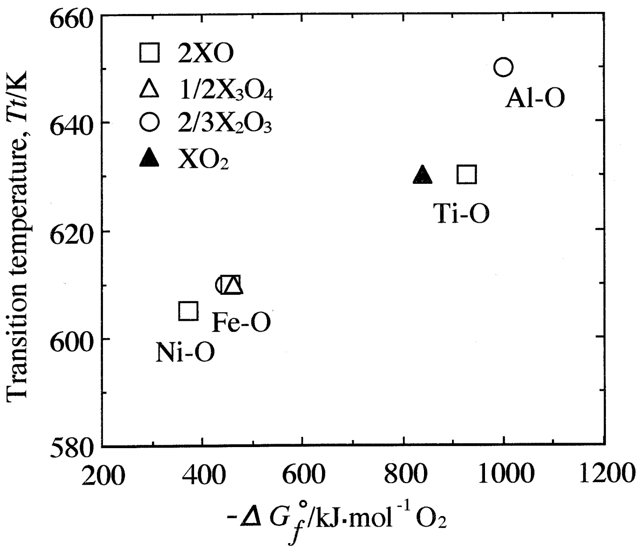

Fig. 5 Relationship between oxide formation free energy and transition temperature

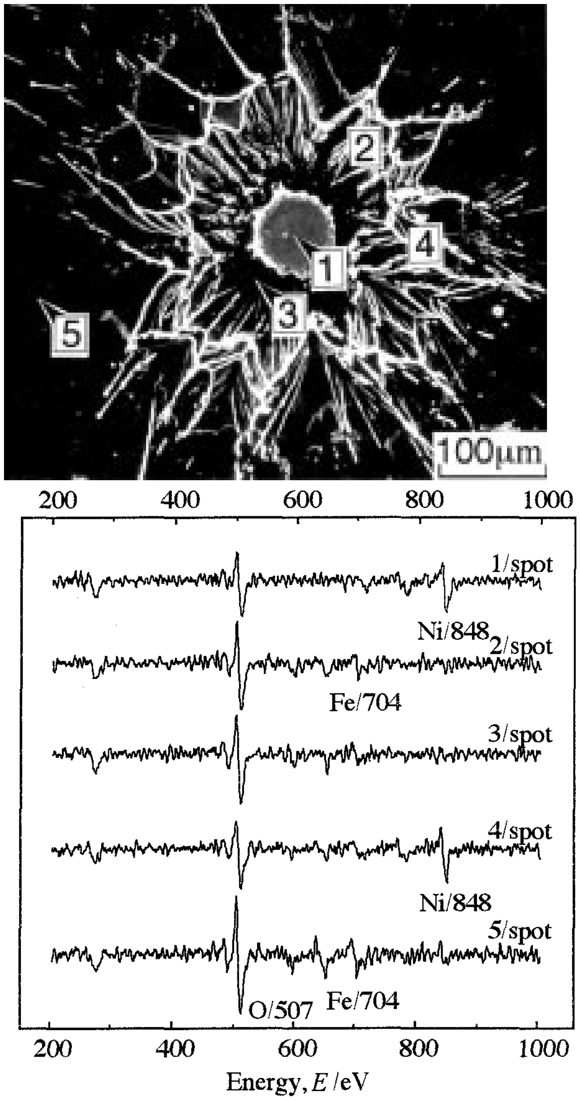

Fig. 6 Auger analysis profiles on nickel splash splat

Therefore, flow and sliding of the liquid just after impingement did not significantly occur on the substrate surface.

Moreover, the nickel splat morphology on the unidirectionally scratched substrate whose temperature was kept at higher than T_t, is shown in Fig. 8. It is clear from the figure that the splat pattern in the direction perpendicular to the scratch is of the splash type, while that in the direction along the scratch is the disk type. This result reveals that splashing tends to occur due to barriers to the liquid flow. This unique tendency of the splat pattern cannot be explained by an explosion in the spherical liquid because such an explosion would spread in all directions on the substrate surface. Therefore, the rapid flow of the liquid film occurred due to flattening of the splash splat on the flat substrate. Also jetting away of the liquid flow arose due to barriers to the liquid flow, such as poor wettability at the tip of the liquid flow or the initial rapidly solidified layer in the splat.

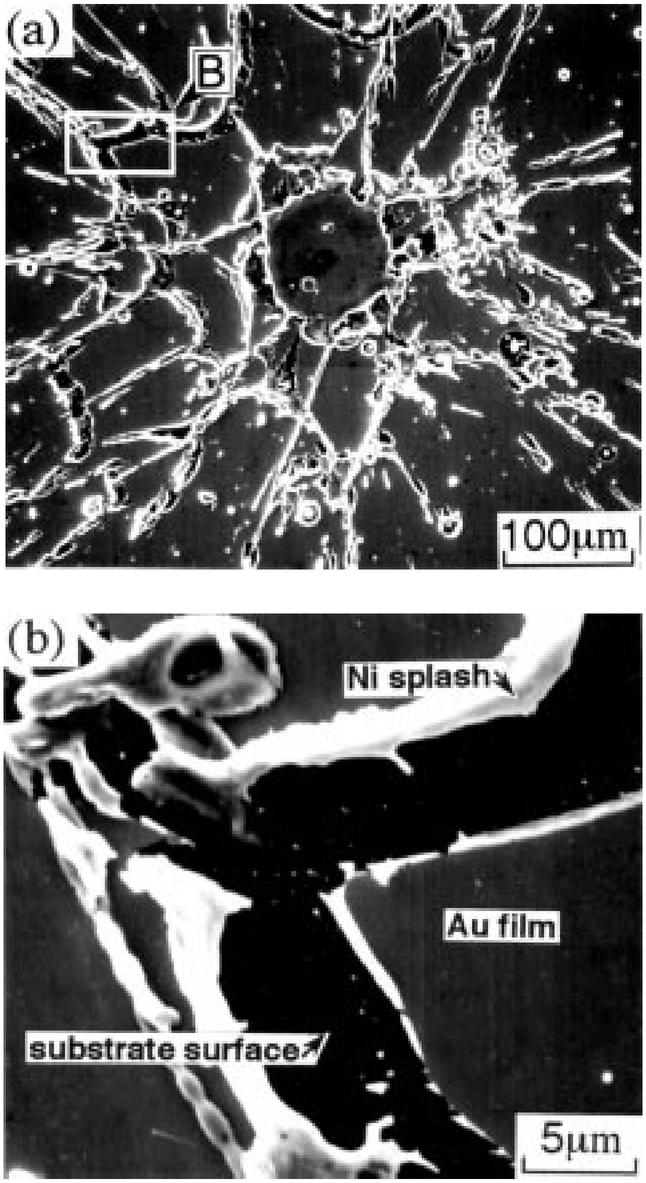

Fig. 7 Section gold physical vapor deposition film at nickel splash. (a) Top surface. (b) Higher magnification of B in (a)

Previous work (Ref 5, 6) has shown that the numerous pores in the central part and the radial structure were observed at the bottom surface of the splash splat. Conversely, almost no pores were observed and the solidification structure looked quite homogeneous over the entire bottom surface for the disk splat. By etching under the same conditions, microstructures of the bottom surfaces of both type splats were observed. As shown in Fig. 9, the radial part in the small central disk of the splash splat is constructed of a very fine grain structure compared to that of the disk splat. This suggests that the central bottom part of the splash splat solidified quite rapidly just after impingement onto the cold substrate, and the resultant solidified region influences the flow behavior of the melt part as indicated by Liu et al. (Ref 4). The formation of the initial solidified layer agrees well with the observation result of Inada and Yang (Ref 11). Consequently, it is inferred that the initial rapid solidified layer formed just after impingement on the substrate plays an important role for the splashing behavior.

3.4 Transition Mechanism of the Splat Pattern

The transition mechanism of the splat pattern near the transition temperature is considered from taking into account the previously mentioned results. When the substrate temperature is higher than T_t, because the wettability at the splat/substrate interface is good, the flow tip of the splat spreads steadily and adheres to the substrate intimately. In this case, the kinetic energy of the particle is consumed effectively as the viscous energy, and finally the splat flattens to a disk shape. The flow speed becomes relatively low because the kinetic energy is consumed as the viscous energy. Figure 10(b) shows the flattening behavior of the disk splat.

When the substrate temperature is lower than T_t, conversely, as the initial rapid solidification of the small central disk occurs just after the impingement on the substrate, liquid film does not spread by flowing on the substrate surface but by jetting away from the initial solidified central disk. In this case, the flow speed becomes higher in inverse proportion to the small consumption of the kinetic energy as the viscous one. Figure 10(a) shows the flattening behavior of the splash splat.

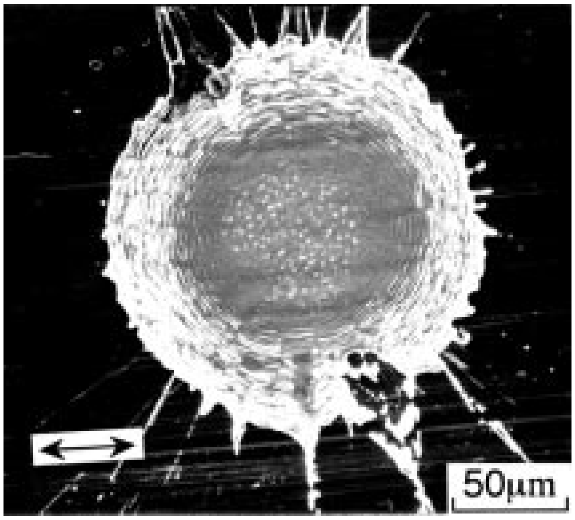

Fig. 8 Nickel splat morphology on unidirectionally scratched substrate. (T_S = 673 K; the arrow shows the scratching direction.)

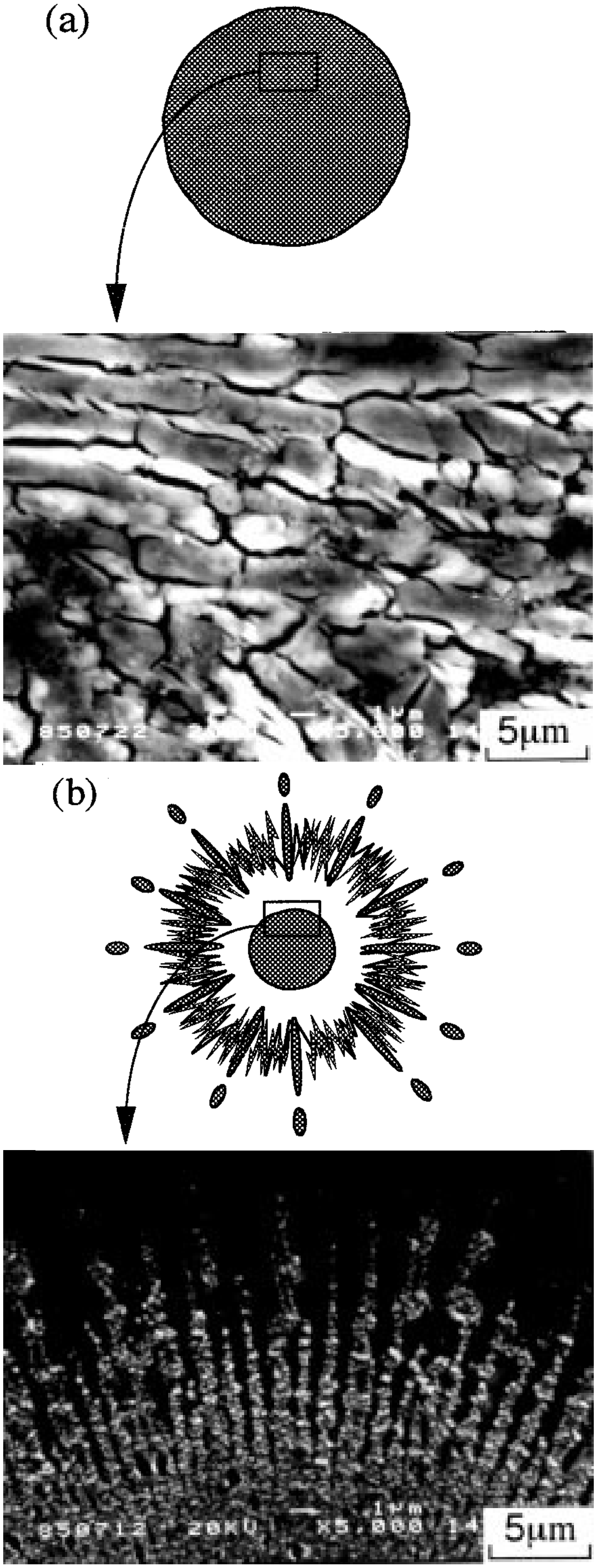

Fig. 9 Etched microstructures of nickel splats. (a) Disk splat. (b) Splash splat

It is known that the break up condition at the flow tip of the liquid film is evaluated by the Weber number, We, which is defined as We = $\rho V^2 d/\gamma$ where ρ, V, d, and γ are density, velocity, diameter, and surface tension of the liquid material, respectively. The breakup of the liquid film, namely, splashing, occurs when We coincides with some critical value. The difference in the We for nickel splats on the substrate at various temperatures is affected by the velocity of the liquid flow. The critical value of the We, We_c, corresponds to the critical value of the flow velocity, V_c. Splashing is more difficult at the small V when the substrate temperature is higher than T_t. Splashing occurs more readily when the substrate temperature is lower than T_t, because V is higher than V_c. The drastic change of the splat pattern near the transition temperature is thought to occur when the flow velocity of the liquid flow coincides with the critical value.

4. Conclusions

The transition behavior of flattening nickel particles sprayed onto a flat substrate was investigated. The results obtained can be summarized as follows:

- Splashing tends to occur more readily when solidification is more likely. Splashing is less likely when there is good wetting between the particle and substrate.
- Auger analysis and scanning electron microscopy observation of the splash splat on the gold-coated substrate confirmed that the splash is not formed by flowing on the substrate surface from the impingement center to the periphery, but by jetting away from the central disk.
- It is indicated that the initial rapid solidified layer formed just after impingement on the substrate plays an important role for the splash behavior.
- Splashing seems to be a breaking phenomenon at the flow tip of the liquid film and the drastic change of the flattening pattern near the transition temperature is thought to occur when the We of the liquid flow coincides with some critical value.

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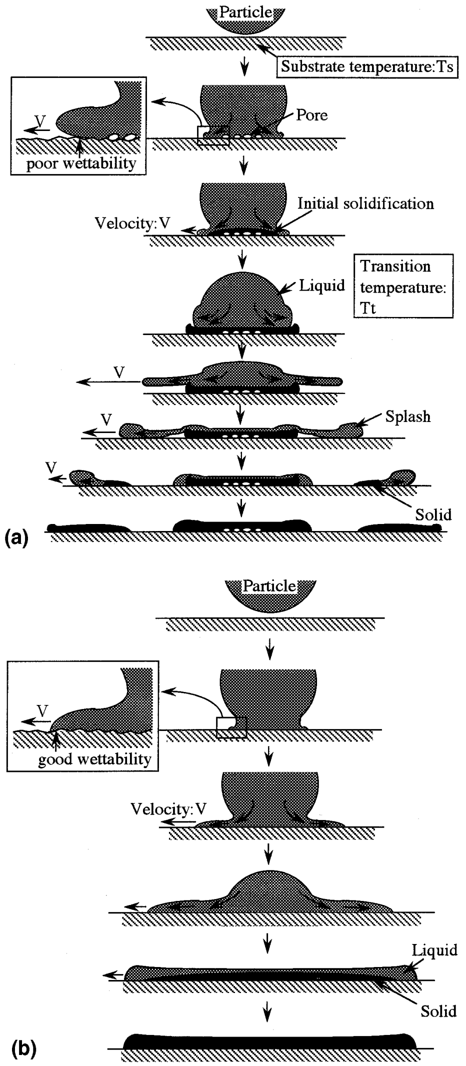

Fig. 10 Proposed splat flattening model. (a) Splash splat $T_s < T_t$. (b) Disk splat $T_t < T_s$