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Role of substrate temperature on microstructure formation in plasma-sprayed splats

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ABSTRACT

The flattening nature of the individual splat in thermal spraying is the fundamental process for the coating fabrication process, thus numerous studies have been conducted to understand the splat formation process in thermal spraying. In the present investigation, commercially available pure Cu particles with diameter of several tens of micrometers were thermally sprayed onto mirror polished AISI304 substrates held at various temperatures during the spraying. The top surface, bottom surface, and cross section morphologies of splats collected under designated conditions were observed in detail. According to the observation, different splat microstructures could be found varied with the substrate temperatures. In particular, a typical layer composed of fine grains could be found at splat–substrate interface when the substrate was held at high temperature, which has a close relation with the favorable wetting and enhanced heat transfer from molten droplet to substrate. This layer could induce the rapid decrease of droplet viscosity at the bottom surface and prevent the further spreading of the splat, which promoted the formation of disk-shaped splat.

1. Introduction

Thermal spraying is a process that can provide thick coatings over a large area at high deposition rate as compared to other coating processes. The coating is always built-up on the roughen substrate by the impingement of the fully or partly melted particles. The next layer is deposited on the top of the previously deposited one, until a coating of the desired thickness is obtained [1]. This technology has been widely applied in many industrial applications [2–8], due to the cost effective and some other advantages of this process. The coating was fabricated by the individual splat deposited on the substrate or on the previously deposited one, hence the flattening and the solidification of the single splat are the fundamental processes for the coating fabrication. Consequently, coating microstructure and properties, such as porosity and adhesion strength, depend strongly on the flattening nature of each splat [9,10], because it is the first layer of splats that determines the coating–substrate adhesion, while the coating cohesion is determined by the nature of the inter-splat contact. Therefore, it is necessary to clarify the splat formation mechanism of the thermal sprayed particles to establish the controlling way for the coating formation.

The role of substrate temperature on splat formation process has been widely investigated during the past few decades. A transition phenomenon in a flattening behavior of the thermal sprayed particle on the flat substrate surface was firstly introduced in 1995 [11], which reported that when the substrate temperature was increased above one critical temperature, the splat shapes of most materials sprayed onto flat substrates underwent a transition from a distorted shape with splash to a disk-shaped one.

Actually, thermal spraying is a complex and short-period process, it is difficult to clarify the flattening behavior of the thermal sprayed particles directly with preventing technology. Although most of the feedstock materials sprayed onto flat substrate exist such a transition behavior through increasing substrate temperature [12–24], understanding of the processes of splat formation and bonding with substrate is limited up to today because of the difficulties in performing high resolution studies of the splat–substrate interface. The possible influence factors of substrate temperature on splat shape determinant have been widely discussed, and the adsorption/desorption of adsorbates and condensates on the substrate surface [12–15], surface characteristic [15–18] and thermal contact resistance [19,20] might strongly affect the splat formation process.

Most of the previous investigations were conducted by observing the geometry shape of the splat but without knowing the actual flattening and solidification process, the microstructure formation process in plasma-sprayed splats through controlling substrate temperature has not been systematically reported. Consequently, some intensive investigation on this aspect was strongly required. In this research, the splats were collected on the substrate held at various temperatures during the spraying. The splat top surface, bottom surface and cross section morphologies were systematically observed. This article focuses, in particular, on the cross section microstructure and grain distribution at splat–substrate interface.

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2. Experimental procedures

The spraying work was carried out by low pressure plasma spraying (LPPS) with the chamber door open during the splat collection process, a SG-100 plasma torch with anode *diameter* 8 mm (Praxair Surface Technologies, Indianapolis, IN, USA) was used. During deposition, the injector was held perpendicular to spray gun, the substrate surface was held vertically and spray gun was held horizontally so that the direction of droplet stream was perpendicular to the substrate surface. Splats were collected on the substrate by moving the shutter rapidly in one direction. The spraying was operated at a current of 800 A and a voltage of 40 V. The spraying distance between the gun and the substrate was kept at 200 mm. The powder was injected at a feeding rate of 6 g/min. Argon and helium were used as operating gas with flow rates of 50 scfh and 12 scfh, respectively.

Commercially available pure Cu powder with diameter less than 75 μm as presented in Fig. 1 was thermally sprayed onto AISI304 substrates, as the feedstock powder was in a very wide size distribution, only the final depositions with a diameter ranging from 50 to 100 μm were considered for the evaluation. The substrates were polished with waterproof paper of No. 150, No. 400, No. 800, No. 1200 and No. 2000 gradually. The plates were finally polished both with 1.0 μm and 0.3 μm Al_2O_3 buff respectively prior to the spraying. Surface topographies of the substrates once heated to different temperatures were measured by atomic force microscopy (AFM) (SPM-9500J3, Shimadzu Co., Ltd., Tokyo, Japan) covering an area of 1 μm^2.

The splat shapes were indentified from optical microscope (OM, Eclipse LV100, Nikon Co., Ltd., Japan) images. The top surface morphology was observed by scanning electron microscope (SEM, JSM-6390TY JEOL, Co., Ltd., Tokyo, Japan). After the top surface observation, the splats were pulled off using carbon tape, the bottom surface morphology of splat caught on the carbon type was examined using SEM as well. The cross section morphology was observed using a scanning ion microscope (SIM) after cutting by focused ion beam (FIB) microscope (Quanta 200 3D, Czech Republic). The SIM was also employed to examine the grain distribution at bottom surface.

3. Results and discussion

3.1. Substrate topography

In order to clarify the effect of substrate temperature on the substrate topography, the substrates were observed on their surfaces as shown in Fig. 2. The 0.3 μm Al_2O_3 mirror polished AISI304 substrates were preheated to 573 K, and 773 K for 10 min than cooled down to room temperature prior to the observation. According to the figures, it was found that no significant topography and surface roughness change took place on the substrate just heated to 573 K, namely, low preheating temperature didn't produce significant changes in surface topography. However, many projections as oxide were formed on the substrate once heated to 773 K. Thus, the specific surface area, which was defined as the total surface area per unit of mass of the solid material might be significantly enhanced. As a result, more favorable heat transfer from molten droplet to substrate could be obtained due to the more intimate contact. The faster the heat transfer from molten droplet to substrate, the larger the solidification rate in the droplet could be achieved.

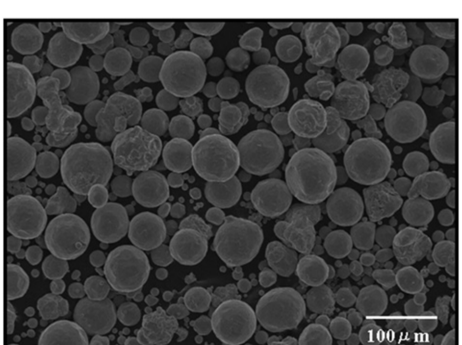

Fig. 1. Morphology of Cu powder used.

It seems that there are two opposite aspects for the influence of surface roughness. Firstly, the roughness would increase the friction of flowing liquid [25]. On the other hand, Wenzel's model proposed that if the original contact angle was less than 90°, a small increase of surface roughness would lead to a reduction of the contact angle [26,27]. Our experimental results verified the latter factor. Meanwhile, similar result has been reported by T. Uelzen et al. [28]. To summarize, surface roughness increase in nano-scale through increasing preheating temperature might promote the wetting.

Meanwhile, McDonald et al. [21] proposed that heating the substrate cleaned the surface and reduced thermal contact resistance. Cedelle et al. pointed out that the thickness of the oxide layer might increase, without changing its composition [18]. However, surface oxidation decreased the ability of a surface to adsorb ambient vapors, therefore less adsorbed gas/condensation existed on the substrate with higher preheating temperature, which might induce the enhancement of wetting. While Hyland et al. reported that the heat treatment prior to the thermal spraying had an effect on the content of the various oxides and hydroxides on the surface [29,30]. However, it is difficult to identify with precision the exact concentration of the elements with the prevailing technology.

In summary, the wetting of substrate by liquid droplet could be strongly affected by substrate preheating. Favorable wetting might be generated by removing the adsorbed gas/condensation through substrate preheating and surface roughness increase in nano-scale.

3.2. Splat top surface morphology

The shapes of splats obtained on the substrates held at various temperatures are shown in Fig. 3. According to the optical images, completely splashing finished splat with circle shaped splash fingers surrounding the central disk-shaped part was found on the substrate without preheating (Fig. 3a-1). Once the substrate was heated up to 573 K, the splat has the typical characteristic of disk-shaped one, and only few short projections were found at the periphery of the central zone (Fig. 3b-1), and the material loss and splat fragmentation were greatly reduced. With the continuous increasing of substrate temperature, a special phenomenon was found from the top surface observation as shown in Fig. 3c-1. That is, typical color change could be confirmed of the splat collected on the hotter substrate. The substrate was held at high temperature during the spraying, and once the particles were thermally sprayed onto substrate surface, disk-shaped splat might be formed in a very short time. However, the splat–substrate system was still kept at high temperature due to the low thermal radiation to the air environment after the splat formation is finished, which might enhance the oxidation of the deposited particle, but has no significant effect on the disk-shaped splat formation process.

As scanning electron microscope allows the observation of details that are not observable by OM, the top surface morphologies of the individual splat deposited on the substrate held at different temperatures were observed using SEM in this study. The splat achieved on the substrate held at room temperature fragmented and splashed, leaving only a small centralized core adhering to the substrate, some pores can be found even from the top surface, and the edge of such disk-liked part was serrated, and ring-shaped splash part was found surrounding the central zone (Fig. 3a-2, a-3). Once the splat was collected on the substrate held at 573 K, the splat shows a typical disk characteristic, most of the splat remained on the substrate, only few short projections was found near the periphery of the splat, and almost no pore was found from the top surface (Fig. 3b-2, b-3). With the continuing increase