Visualization of Nanoparticles Behavior Introduced into Gas Flame Spraying

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In a newly developed rod feeding gas flame process, solid resin rod including nanoparticles could be fed into a Rokide® flame gun successfully to create fine ceramics layers without micro cracks and pores applying for electric, magnetic and dielectric components. In this investigation, alumina particles of 170 nm in average diameter were dispersed into acrylic liquid resin at 40 % in volume fraction. The paste materials were injected into a brass mold of φ40×200 mm in inner dimension and thermal cured through heating at 120 °C for 60 min. Formed solid rods were fed into oxyacetylene gas flame coaxially by using the Rokide® spraying gun system. Sprayed particles were cached by water bath for microstructure observations by a scanning electron microscope and crystal phase analyses by an X-ray diffraction spectroscopy. Fine ceramics layer formations will be discussed systematically by the feeding speed of solid rods and gas flame condition of air pressure and oxygen pressure.

Key Words: Rokide® gas flame spraying, solid resin rod feeding, fine ceramics coating, alumina nanoparticle, thixotropic non-Newtonian paste

1. Introduction

Thermal spraying is widely used in current industrial fields to create metals or ceramics layers from micrometer to millimeter thickness on surfaces of practical alloys components. Materials particles of tens of micrometer in average diameter were introduced into high temperature plasma or gas flame, and the molten droplets were sprayed on the substrates. The higher deposition rate could be realized comparing with the other practically used processes, e.g. physical or chemical vapor depositions (PVD /CVD), though considerable micro cracks and pores were included in the surface coated layers consequently. Although these porous coated layers exhibit lower mechanical properties, they are effective as thermal barriers.

Recently, suspension plasma spraying (SPS) was investigated to create the fine coated layers1,2). Nanometer sized particles dispersed in liquid fuel, e.g. alcohol or kerosene were introduced into the plasma or gas flame successfully without powder coagulation caused by using gas blowing feeding machines. However, the deposition rate in SPS was about a tens comparing with the other conventional thermal spraying, because the volume fractions of dispersed particles were only several percent.

In our previous investigations, the high viscosity resin pastes with high dispersion rate of the nanometer sized particles were introduced into the plasma spray effectively3). The resin pastes could be spattered to form micro droplets including the nanoparticles by using compressed air jet, and the micro mists were sprayed into the high temperature and velocity plasma flows continuously. In this study, solid resin rods including nanoparticles formed by thermosetting treatments could be fed into oxyacetylene gas flame coaxially through Rokide® spraying gun system as shown in Fig. 1. The dynamic behavior of the gas flames including nanoparticles will be visualized clearly to discuss the coated layer formations through systematic modulations of the feeding speed of solid rod, air and oxyacetylene gases.

Fig. 1 A schematic illustration of solid rod feeding process in Rokide® spraying gun system. Nanometer sized ceramics particles dispersed in resin matrix with large volume fraction can be introduced into high temperature flame coaxially to realize higher deposition rate of coated layers.

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2. Experimental procedure

Alumina particles of 170 nm in average diameter were dispersed into the acrylic resin at 40 % in volume fraction. Obtained slurries were mixed in airtight containers through configuration of rotation and revolution movements. Fluid characteristics of the slurry pastes were evaluated by using a viscosity to viscoelasticity measuring instrument to realize thixotropic fluid flows to decide the enough mixing time. The obtained slurry was injected into a brass mold of φ4×200 mm in inner dimension, and through heating at 120 degrees for 60 min.

The formed solid resin rod was fed into the oxyacetylene gas flame coaxially at 2.5 to 6.0 mm/s in feeding speed through the Rokide® spraying gun system. The air and oxyacetylene gases pressures were controlled from 0.3 to 0.6 MPa and 0.6 to 0.8 MPa. The SUS-304 stainless steel substrates of 50×50×1 mm in size were placed at 100 mm in distance from the gun. The sprayed particles were collected by a water bath placed at 550 mm in distance from the flame gun. The microstructures were observed by a scanning electron microscopy and the crystal structures were analyzed by using X-ray diffraction. The gas flame behavior including with nanoparticles was observed and visualized by utilizing AccuraSpray (G3C: TECNAR, Canada). Particles velocities were measured between two defined positions along the spraying direction of the gas flame. The intensities of brightness obtained from these measuring points were detected by a sensor head. The particles velocities were calculated from the precise time delays in these measuring points by cross correlation analysis, and the temperature were measured by utilizing twin wavelength pyrometry principle.

3. Results and discussion

The acrylic resin with alumina nanoparticles showed the thixotropic non-Newtonian fluid though the mixing with 700 and 300 rpm in revolution and rotation speed for 900 s. This result involves the fine particles could be covered with acrylic monomers and oligomers, and dispersed uniformly in the organic medium. The obtain slurry was injected into a brass mold. The formed rod by the heat treatment is shown in Fig. 2. The acryl rods with alumina particles were solidifed through the thermosetting without the major shrinking and cracking.

The formed rods were smoothly fed into the gas flame, and supplying speed of the nanoparticles could be controlled exactly by using a motor drive system in the gun. Fig. 3 shows the sprayed gas flame from the Rokide® spray gun nozzle. Optimum spray conditions were determined by observing was sprayed coating on the stainless steel as shown in Fig. 4. Firstly, we were investigate whether resin rod was supplied continuously into gas flame. When the rod feeding speed was set for the slower range, the lump accretions of coagulated particles were formed on the nozzle tip, and introduced into the gas flame discontinuously by the compressed air flow. Consequently conditions rod feeding speed was fast, i.e. 5.6 mm/s was best. Next, we were investigated the conditions under which the alumina layer was deposited without carbon contaminations. The resin ingredient should burn in the flame and increase the temperature, and these phenomena are considered to contribute to the alumina layer formation without the carbon contaminations. The enough oxygen gas supplying with high pressure could promote the effective burning of the resin rods and increase the particles speed directly in the flame. And, the compressed air blow to centralize the flame profile and to concentrate the particle distributions should decrease temperature of the high speed flow. Consequently, to reducing the carbon contaminations, spraying conditions were successfully optimized 0.3 MPa and 0.8 MPa in air and oxygen gases pressures.
Feeding speed of the rod | Air pressure | Oxygen gas pressure
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2.5 mm/s | 0.3 MPa | 0.7 MPa
5.6 mm/s | 0.6 MPa | 0.8 MPa

Fig. 4 The alumina coated layer was formed on the SUS-304 stainless steel substrate. Black spots of the alumina layer surface are carbon.

Fig. 5 Oxyacetylene gas flame profiles including with the alumina nanoparticles visualized by AccuraSpray. The flame temperature and velocity can be measured and analyzed. [A] Rokide® spray gun top, [B] Bulls eye circle of measuring point of particles velocity and temperature, [C] Particle velocity gauge (m/s), [D] Particle temperature gauge (°C)

Fig. 5 shows the observed and visualized gas flame profiles by using the AccuraSpray. The maximum gas flame temperature is measured at 2080 °C. This value is slightly lower level to the alumina melting point of 2050 °C. The particles velocity in the gas flame is 375 m/s. This value is similar level comparing with the conventional plasma spraying. Therefore the heating time of the alumina nanoparticles are considered not to be enough to elevate the temperatures above the melting point. Consequently, the introduced nanoparticles are considered not to be melted in the gas flame region. Fig. 6 shows the X-ray diffraction pattern of solid droplets collected from the gas flame. The crystal phase was analyzed as α-alumina. In the conventional thermal spraying processes using coarse particles, formed layers were composed of the γ-alumina through the rapid solidification of the molten material⁵,⁶). Because the particles could not be melted in the spraying method used in this investigation, the crystal structure of alumina is considered to be still remained as α-phase.

The surface microstructure of collected alumina from the flame is shown in Fig. 7. The grain boundaries are observed in the micro bulk⁷,⁸). And the nanoparticles as raw materials are remained on the bulk surface. In the oxyacetylene gas flame with the high flow velocity, the solid phase alumina particles are considered to be heated and sintered. Subsequently, the micro bulks and remained nanoparticles should collide to the substrate with the dynamical sintering. Fig. 8 shows the cross sectional microstructure of the alumina coated layer formed on the stainless
4. Conclusions

Alumina nanoparticles could be contained with acrylic liquid resin with higher volume successfully. Obtained thixotropic slurry was injected into brass molds to create solid resin rods. We have successfully introduced alumina nanoparticles into oxyacetylene gas flame coaxially by resin rod feeding technique in Rokide® type thermal spraying system. The gas flame behavior including nanoparticles was observed and visualized by AccuraSpray to optimize the spraying process condition. The formation mechanism of alumina layer was discussed according to the X-ray diffraction and SEM. It was found that sprayed particles did not melt because the crystal structure was α-alumina and grain boundaries were observed in the micro bulk. This coating process was successful alumina layer deposition despite the unmelted particles. Inserting the nanoparticles dispersed resin rods into the gas flames was effective for controlling and increasing the deposition rate in the coating process. The solid rod feeding process will be effective supply method to introduce nanoparticles into various thermal spraying heat sources. In the future, try to clarify the deposition mechanism of thermal spraying using nanoparticles.

Reference

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