Plasma-sprayed Coatings of Aluminium Oxide and Mulite

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Abstract

The present report is the investigation of the effects of the HIP treatment on plasma-sprayed ceramic coating of Al_2O_3, Al_2O_3-SiO_2 on the metal substrate. These effects were characterized by phase identification, Vickers hardness measurement, and tensile test before and after HIPing.

Key Words : HIP treatment, plasma-aprayed, Al_2O_3, Al_2O_3-SiO_2

1. Introduction

Plasma spraying is typical of a relatively inexpensive and rapid method of applying thick ceramic coatings on metal components to improve their resistance against wear, heat, and oxidation. A remarkable feature of plasma-sprayed coatings is their good resistance against thermal shock due to the porous state of the coated layer with a consequently low Young's modulus. There have been investigations on such thermal barrier coatings, applied to aircraft gas turbine components [1-2]. However, the existence of many pores with a bimodal distribution and a laminar structure in the coating reduces coating strength and oxidation protection of the base metals [3].

In order to counteract these problems, there have been many efforts to obtain dense coatings by spraying under low pressure or vacuum and by controlling particle size and morphology of the spraying materials [4].

The aim of the present study is to survey the effects of the HIP treatment on plasma-sprayed ceramic coating of Al_2O_3, Al_2O_3-SiO_2 on the metal substrate, AISI type 304 stainless steel. These effects were characterized by phase identification, Vickers hardness measurement, and tensile test before and after HIPing.

2. Experimental Part

Tables 1-2 summarize the typical compositions and particle size ranges of the starting materials and the processing conditions used in the present study. The substrate of stainless steel was a rod 7 mm in diameter and 7 mm high. The cylinders were degreased with trichloroethylene. After temperature and pressure treatment for 1h, they were cooled to room temperature at a rate of 5 ℃/min. Finally, the residual pressure was released.

Table 1. Specification of Coating Materials.

Coating Materials	Other phases (wt %)	Size range (㎛)	Spray Distance (㎜)
No1 Al_2O_3	-	3-5	100
No2 Al_2O_3	2.0 SiO_2	2-4	80

Table 2. Plasma Spraying conditions the same for Al_2O_3, Al_2O_3-SiO_2 films.

Primary gas Pressure Flow rate(㎪) (㎥/h)	Secondary gas Pressure Flow rate(㎪) (㎥/h)	Current (A)	Voltage (V)	Powder Feed rate (g/min)
345 (N_2) 2.1	345 (H_2) 0.4	500	70	30

The phase of the starting powders, as-sprayed coatings, and HIPed coatings were determined by X-ray powder diffractometry (XRD) with crystal monochromatized CuK_{α} radiation. Vickers hardness measurements were performed at a load of 300 g for 30s. The specimens were polished with various grades of alumina polishing films to obtain flat surfaces for the measurements.

Adhesion strength was evaluated by a tensile adhesion test using a testing machine at a cross head speed of 0.5 ㎜/min. Before measurements, the specimens were filled with acrylic resin in a vacuum dryer and cured at 150℃ for 1h to prevent adhesive penetration. After removing the protruded acrylic resin, the specimens were fixed with tensile jigs using epoxy adhesives and then cured at 150℃ for 2h. The tensile strength of the epoxy adhesive was only 60-70 ㎫, so strengths more than those values could not be estimated by this method.

Microstructures of surfaces, cross-sections, and fracture surfaces were observed using optical microscopy and scanning electron microscopy (SEM). Element distribution near the interface between the plasma-sprayed layer and the substrate before and after HIPing was analyzer by using an electron probe microanalyses.

3. Results and Discussion

Alumina starting powder consisted mainly of amorphous phase and of small amount of γ-Al_2O_3. After plasma spraying, η-Al_2O_3 was mainly obtained with a little amount of γ-Al_2O_3 which might be unmelted particles. Distinction between η-Al_2O_3 and γ-Al_2O_3 was examined by the presence of splitting in the (400) and (440) reflections for γ-Al_2O_3. After HIPing, phase constitutions were converted to α-Al_2O_3 having a trace of 9 Al_2O_3 $2B_2O_3$, included in the BN capsule as a binder. There was no change in Al_2O_3-SiO_2 phase composition after plasma spraying (Fig.1) nor after HIPing except for the appearance of a small amount of α-Al_2O_3.

Vickers hardness and tensile strength of Al_2O_3 and Al_2O_3-SiO_2 coatings before and after HIP treatment are shown in Figs. 2-3 respectively. The Vickers hardness of as-sprayed Al_2O_3 and Al_2O_3-SiO_2 coatings could not be measured because of their rough surfaces with a lot of pores even after polishing.

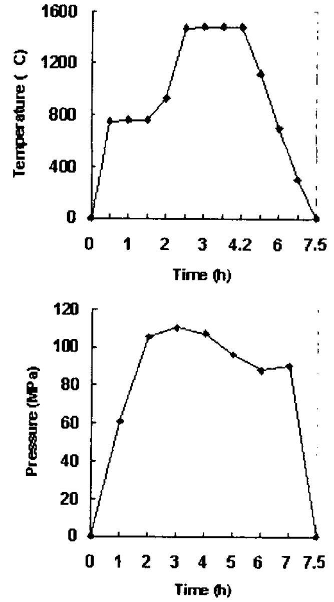

Fig. 1. Temperature (a) and pressure (b) profile of HIP treatment.

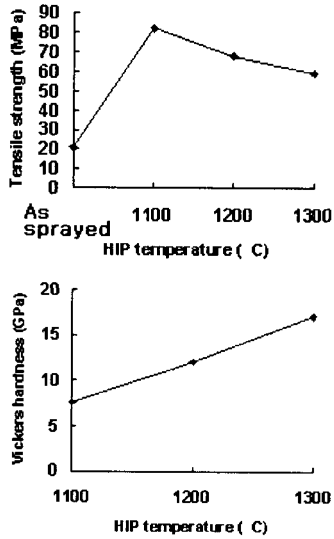

Fig. 2. Variations of Veckers hardness (b) and tensile strength (a) of Al_2O_3 coating HIPed at various temperature.

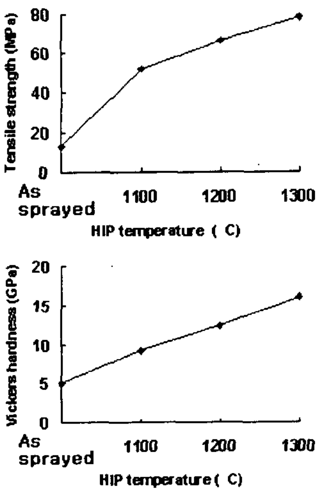

Fig. 3. Variations of Veckers hardness (b) and tensile strength (a) of Al_2O_3-SiO_2 coating HIPed at various temperature.

The Vickers hardness of the Al_2O_3-coated surface showed an improvement to 12.2 GPa after HIPing at 1300℃. This value reaches to 60 % of the hardness of sintered Al_2O_3. The tensile strength also increased from 5 MPa to > 40 MPa.

The fracture path in the as-sprayed specimen was close to the interface between the coated layer and metal substrate. However, after HIPing the fracture occurred mostly at the edge area within the coated layer. The maximum tensile stress seemed to appear in the edge area due to a thermal expansion mismatch between the coated layer and the metal substrate during HIPing. Consequently, the decrease of maximum value of each tensile strength with increasing HIP temperature, as seen in Fig. 2 would be ascribed to the increase of thermal stress with HIP temperature. It is considered that the above mentioned HIP effects would be further improved by introducing some interlayer which can relax the thermal stress.

In case of Al_2O_3-SiO_2 coating, Vickers hardness and tensile strength were remarkably improved, from 5 GPa to 13.3 GPa and from 5 MPa to > 60 MPa after HIP treatment at 1300℃, respectively. The fracture positions before and after HIPing were similar to those of Al_2O_3 coating.

The range of temperature and pressure in HIP treatment was limited, owing to the properties of substrate metal, so the efficient densification of coated Al_2O_3-SiO_2 could not be completed.

Scanning electron microscope photographs of the cross section of Al_2O_3, Al_2O_3-SiO_2 coatings were investigated. The as-sprayed Al_2O_3 coating had a laminar texture consisting of the flattened particles rapidly solidified by spraying, which partially contacted each other. After HIPing, the coated layer changed into a granular structure consisting of α-Al_2O_3 with large voids diminishing remarkably.

When the samples were HIPed at 1200℃, the stainless steel penetrated into the coated layer near the interface by plastic deformation. In such an interface a coating strength is expected to increase by a mechanical interlocking. The substrate metal infiltrated deeply into the coated layers after HIP treatment at 1300℃. The deep infiltration of metal should reduce resistance against wear, heat, and oxidation.

It has been reported that the densification of Al_2O_3 occurred at > 1500℃ by HIPing [4-5]. Therefore, the HIP conditions in the present investigation are not enough to densify the coating. It is considered that the stainless steel penetrated into both the remaining original pores and the spaces produced by volume shrinkage from η- to α-Al_2O_3.

In the Al_2O_3-SiO_2 coating, the pores still existed, but the laminar structure (splat quenched droplets) disappeared after the HIP treatment at 1100℃. The size and amount of pores decreased at 1200℃, and the coated layer was completely densified at 1300℃ by HIPing process.

Particle-like micro-pores near the interface were examined by EPMA. The elements consisting of stainless steel could not be detected in the specimen HIPed at 1200℃ but could be detected in that at 1300℃. The fracture pattern of as-sprayed Al_2O_3-SiO_2 coating was similar to that of as-sprayed Al_2O_3 coating. Trans-granular type fracture was common in HIPed specimens of which particle sizes were 0.2-0.4 ㎛ by TEM observation. In cases of Al_2O_3 and Al_2O_3-SiO_2 coatings, there was neither an apparent reaction layer nor a diffusion layer at the interface between the stainless steel and the coated layers.

4. Conclusions

The effects of HIPing treatment between 1100℃ and 1300℃ on the plasma-sprayed Al_2O_3 and Al_2O_3-SiO_2 coatings onto the stainless steel substrate are summarized as follows:

1) both Vickers hardness and bonding strength were remarkably improved to 12.2 GPa and 40 MPa, respectively, in Al_2O_3; to 13.3 GPa and > 60 MPa in Al_2O_3-SiO_2 coating;
2) each coated layer before HIPing had a laminar texture (splat quenched droplets), but after HIPing it changed into the granular structure with remarkably decreased micro-cracks;
3) the Al_2O_3 coatings could not be densified adequately in spite of decreased large voids. However, the Al_2O_3-SiO_2 coating could be fully densified.

The fracture position by tensile adhesion testing was mainly in the edge area within every coated layer, attributed to the tensile stress caused by thermal expansion mismatches between the coated layer and substrate. The lower tensile strength of specimens HIPed at 1300℃, compared with those HIPed at 1200℃, seems to be due to thermal stress.

These results show that HIP treatment has an advantage for improving adhesive strength and Vickers hardness of plasma-sprayed coatings.

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