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Penetration Behavior into Connected Porosities of Plasma Sprayed Al₂O₃ Coatings with Liquid Mn

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KEY WORDS: (Penetration)(Liquid manganese)(Al₂O₃ coating) (Plasma spraying)(Heat-treatment)(MnAl₂O₄ spinel)

Plasma sprayed Al₂O₃ ceramic coatings have been widely applied in many industrial fields because of an excellent wear and heat resistance which ceramics possess by themselves. However, the mechanical properties of plasma sprayed Al₂O₃ coatings declined by some defects of the connected porosity, etc., in the coatings such as nonbonded area among the particles and microcracks in particles. In this study, penetration behavior of liquid Mn into connected porosities of Al₂O₃ coatings and densification of the coatings during heat treating Al₂O₃ coatings with liquid Mn in order to improve the properties of the coatings were examined.

Al₂O₃ spraying powder with a diameter 10～40 μm, SS41 steel as the substrate and Mn plates with purity of 99.9% were used in this experiment. Figure 1 shows a schematic diagram of process used for Mn penetration method. After Al₂O₃ coatings with thickness of about 200 μm were sprayed onto sand-blasted SS41 steel by using the plasma spraying, Mn plate contacted with surface of Al₂O₃ coatings was heated at 1573K in 1.33 × 10⁻¹ Pa and 1.33 × 10⁻³ Pa.

Figure 2(a) shows an example of the result of optical microscopic observation for Al₂O₃ coating as-sprayed on a SS41 steel substrate. Fig 2(b) shows a result of a Al₂O₃ coating heated without Mn at 1573K and 10.8ks in 1.33 × 10⁻³ Pa. In both cases, a lot of porosities and defects in the Al₂O₃ coating were recognized.

Figure 3 shows wetting images of pure Cu, Ni and Mn on the Al₂O₃ coatings after heating for 0.3ks at temperature of 50K over melting point of each metal. The contact angles of Mn, Cu and Ni were about 0°, 140° and 115° respectively. It was recognized that the wettability of Mn on the Al₂O₃ coatings is better compared with those of Cu and Ni, because Mn was penetrated into inside of the coatings.

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Fig.1 Processes of thermal spray and heating treatment with Mn.
Figure 2. Microphotographs of Al₂O₃ coatings. (a) as-coat; (b) heat-treatment (1573K, 10.8ks, 1.33 × 10⁻³ Pa).

Figure 4. shows the results of SEM observation and image analysis of Mn, O and Al elements by means of EPMA for the cross section of Al₂O₃ coatings after heat-treatment with Mn at 1573K and 1.33 × 10⁻³ Pa. From this figure, it was recognized that Al₂O₃ coating after heat-treatment with liquid Mn become dencer compared with as-sprayed coating and heat-treated Al₂O₃ coating as shown in Fig.3. Moreover, the interface between Al₂O₃ coating and SS41 steel substrate strengthened. As a result of elements analysis of EPMA, it was observed that thick rich-layer of Mn with Al and O not only existed on surface of the coating, but distributed like net throughout grain boundary (or the connected porosities) inside of the coating. Figure 5. shows XRD results of surface layer and inside of the Al₂O₃ coatings of Fig.4. compared with the as-coat. The new dense surface layer of the Al₂O₃ coating after heat-treatment with Mn was mainly composed of MnAl₂O₄ spinel phase and α-Al₂O₃ phase as shown in Fig.5(b). The inside of the Al₂O₃ coatings, where a little MnAl₂O₄ was existed as shown in Fig.5(c), was composed of α-Al₂O₃ as main phase and MnAl₂O₄ spinel. It may be considered that MnAl₂O₄ is formed by reaction of liquid Mn, contained oxygen in vacuum atmosphere and Al₂O₃, depending on oxygen quantity dissolved in liquid Mn. These results show that, by applying the heat-treatment with Mn to Al₂O₃ coatings at 1573K in vacuum of over 1.33 × 10⁻³ Pa, liquid Mn dissolving oxygen which remained in vacuum penetrated easily into the connected porosities of the coating and formed MnAl₂O₄ spinel around Al₂O₃ particles by reaction between Mn(O) and Al₂O₃. Vickers hardness of Al₂O₃ coatings increased to ~1500Hv from 700Hv ~ 800Hv of as-sprayed and heat-treated Al₂O₃ coatings after heat-treatment with Mn for over 3.6ks at 1573K. The hardness of 1500Hv is similar as that of sintered Al₂O₃. It is considered that such effective increase of hardness is due to the formation of MnAl₂O₄ at the connected porosities in Al₂O₃ coating by the reaction of Mn(O) and Al₂O₃.

From above-mentioned results, it was showed that
Fig. 4 SEM microstructure and EPMA analyses of Mn treated Al₂O₃ coatings.

the improvement of Al₂O₃ coatings is possible by the penetration of the liquid Mn into the connected porosities of Al₂O₃ coatings and the reaction of the liquid Mn and Al₂O₃ particles when Mn heat treatment was done.

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Fig. 5 XRD results of Al₂O₃ coatings after heat treatment with Mn.