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# Penetration Behavior into Connected Porosities of Plasma Sprayed $\text{Al}_2\text{O}_3$ Coatings with Liquid $\text{Mn}^\dagger$

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**KEY WORDS :**(Penetration)(Liquid manganese)( $\text{Al}_2\text{O}_3$  coating) (Plasma spraying)(Heat-treatment)( $\text{MnAl}_2\text{O}_4$  spinel)

Plasma sprayed  $\text{Al}_2\text{O}_3$  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  $\text{Al}_2\text{O}_3$  coatings declined by some defects of the connected porosity, etc. in the coatings such as nonbonded area among the particles and microcracks in particles.<sup>1)2)</sup> In this study, penetration behavior of liquid Mn into connected porosities of  $\text{Al}_2\text{O}_3$  coatings and densification of the coatings during heat treating  $\text{Al}_2\text{O}_3$  coatings with liquid Mn in order to improve the properties of the coatings were examined.

$\text{Al}_2\text{O}_3$  spraying powder with a diameter  $10\sim 40\ \mu\text{m}$ , 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  $\text{Al}_2\text{O}_3$  coatings with thickness of about  $200\ \mu\text{m}$  were sprayed onto sand-blasted SS41 steel by using the plasma spraying, Mn plate contacted with surface of  $\text{Al}_2\text{O}_3$  coatings was heated at 1573K in  $1.33 \times 10^{-1}\text{Pa}$  and  $1.33 \times 10^{-3}\text{Pa}$ .

**Figure 2(a)** shows a example of the result of optical microscopic observation for  $\text{Al}_2\text{O}_3$  coating as-sprayed on a SS41 steel substrate. **Fig 2(b)** shows a result of a  $\text{Al}_2\text{O}_3$  coating heated without Mn at 1573K and 10.8ks in  $1.33 \times 10^{-3}\text{Pa}$ . In both cases, a lot of porosities and defects in the  $\text{Al}_2\text{O}_3$  coating were recognized.

**Figure 3** shows wetting images of pure Cu, Ni and Mn on the  $\text{Al}_2\text{O}_3$  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  $0^\circ$ ,  $140^\circ$  and  $115^\circ$  respectively. It was recognized that the wettability of Mn on the  $\text{Al}_2\text{O}_3$  coatings is better compared with those of Cu and Ni, because Mn was penetrated into inside of the coatings.

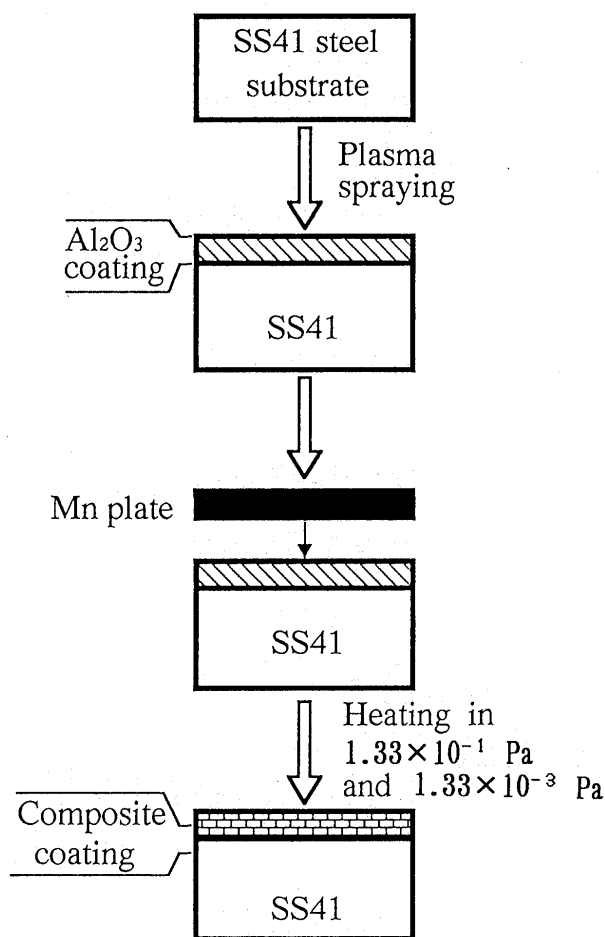


Fig.1 Processes of thermal spray and heating treatment with Mn.

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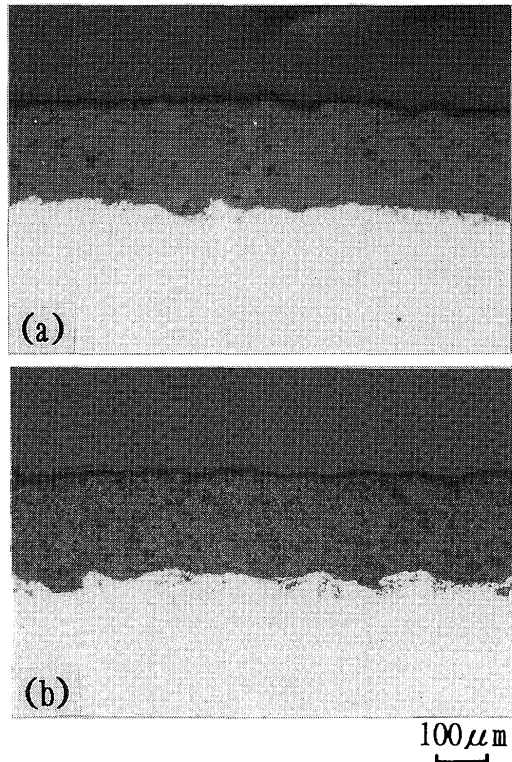


Fig.2 Microphotographs of Al<sub>2</sub>O<sub>3</sub> coatings. (a),as-coat;(b), heat-treatment (1573K,10.8ks,1.33 × 10<sup>-3</sup>Pa).

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 Al<sub>2</sub>O<sub>3</sub> coatings after heat-treatment with Mn at 1573K and 1.33 × 10<sup>-3</sup>Pa. From this figure, it was recognized that Al<sub>2</sub>O<sub>3</sub> coating after heat-treatment with liquid Mn become denser compared with as-sprayed coating and heat-treated Al<sub>2</sub>O<sub>3</sub> coating as shown in Fig.3. Moreover, the interface between Al<sub>2</sub>O<sub>3</sub> 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 Al<sub>2</sub>O<sub>3</sub> coatings of Fig.4, compared with the as-coat. The new dense surface layer of the Al<sub>2</sub>O<sub>3</sub> coating after heat-treatment with Mn was mainly composed of MnAl<sub>2</sub>O<sub>4</sub> spinel phase and α-Al<sub>2</sub>O<sub>3</sub> phase as shown in Fig.5(b). The inside of the Al<sub>2</sub>O<sub>3</sub> coatings, where a little MnAl<sub>2</sub>O<sub>4</sub> was existed as shown in Fig.5(c), was composed

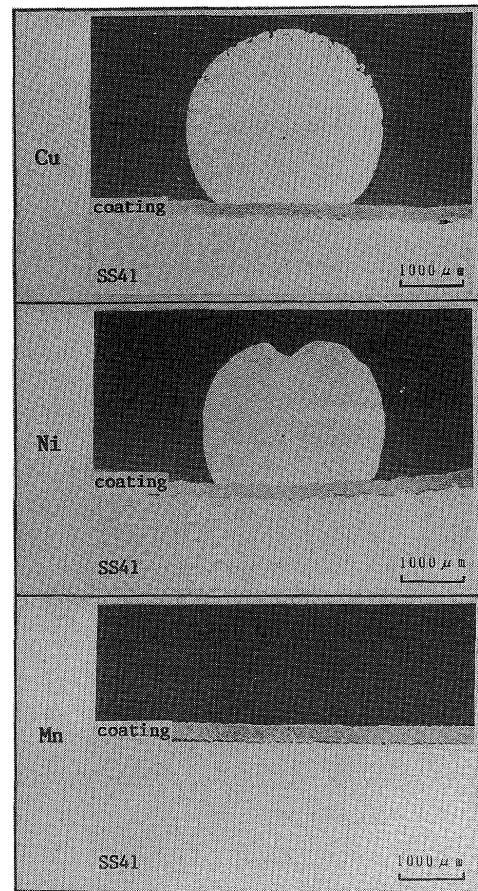
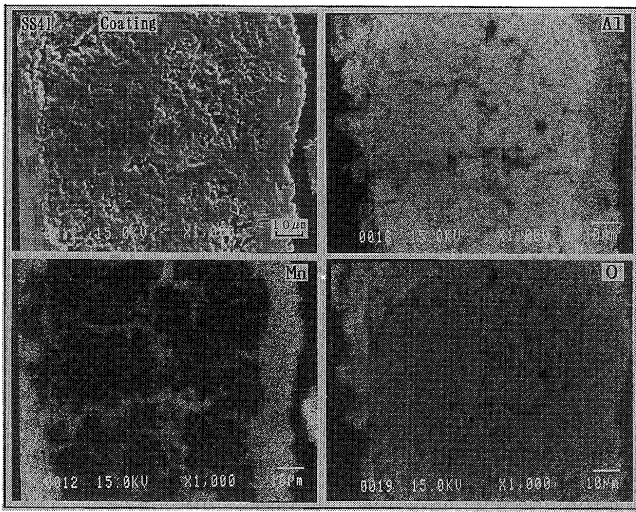


Fig.3 Wettability of Cu, Ni and Mn to the surface of Al<sub>2</sub>O<sub>3</sub> coatings.

of α-Al<sub>2</sub>O<sub>3</sub> as main phase and MnAl<sub>2</sub>O<sub>4</sub> spinel. It may be considered that MnAl<sub>2</sub>O<sub>4</sub> is formed by reaction of liquid Mn, contained oxygen in vacuum atmosphere and Al<sub>2</sub>O<sub>3</sub>, depending on oxygen quantity dissolved in liquid Mn. These results show that, by applying the heat-treatment with Mn to Al<sub>2</sub>O<sub>3</sub> coatings at 1573K in vacuum of over 1.33 × 10<sup>-3</sup> Pa, liquid Mn dissolving oxygen which remained in vacuum penetrated easily into the connected porosities of the coating and formed MnAl<sub>2</sub>O<sub>4</sub> spinel around Al<sub>2</sub>O<sub>3</sub> particles by reaction between Mn(O) and Al<sub>2</sub>O<sub>3</sub>. Vickers hardness of Al<sub>2</sub>O<sub>3</sub> coatings increased to ~1500Hv from 700Hv ~ 800Hv of as-sprayed and heat-treated Al<sub>2</sub>O<sub>3</sub> coatings after heat-treatment with Mn for over 3.6ks at 1573K. The hardness of 1500Hv is similar as that of sintered Al<sub>2</sub>O<sub>3</sub>. It is considered that such effective increase of hardness is due to the formation of MnAl<sub>2</sub>O<sub>4</sub> at the connected porosities in Al<sub>2</sub>O<sub>3</sub> coating by the reaction of Mn(O) and Al<sub>2</sub>O<sub>3</sub>.

From above-mentioned results, it was showed that

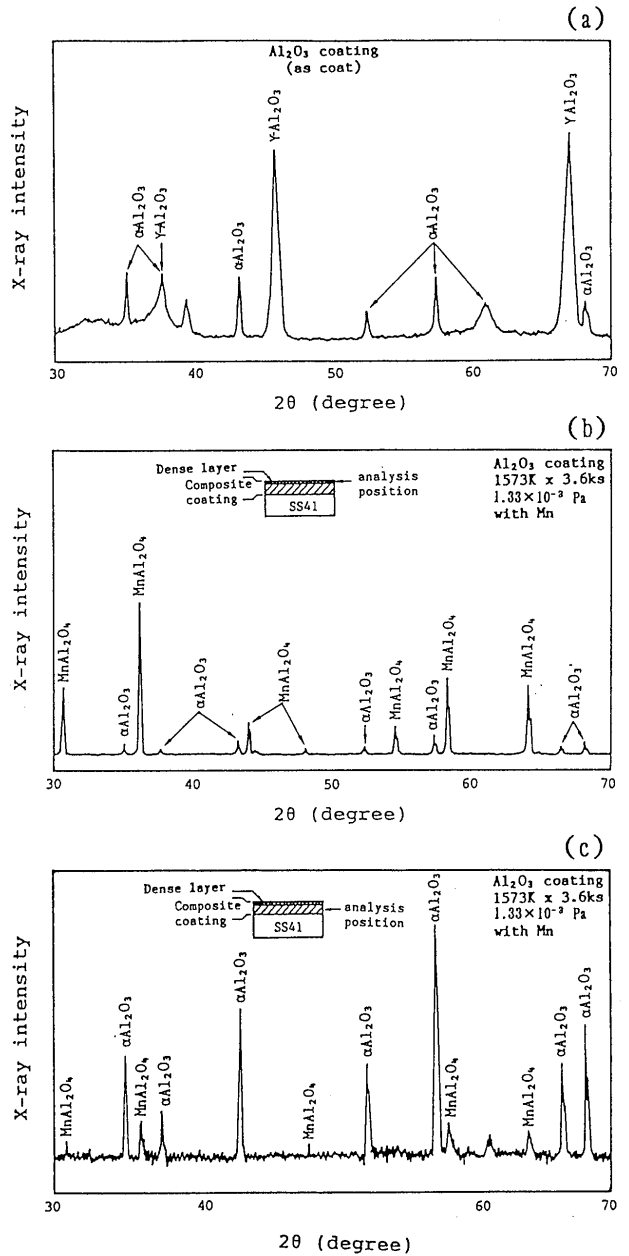


**Fig.4** SEM microstructure and EPMA analyses of Mn treated Al<sub>2</sub>O<sub>3</sub> coatings.

the improvement of Al<sub>2</sub>O<sub>3</sub> coatings is possible by the penetration of the liquid Mn into the connected porosities of Al<sub>2</sub>O<sub>3</sub> coatings and the reaction of the liquid Mn and Al<sub>2</sub>O<sub>3</sub> particles when Mn heat-treatment was done.

**References**

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**Fig.5** XRD results of Al<sub>2</sub>O<sub>3</sub> coatings after heat treatment with Mn.