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# Penetration Behavior into Connected Porosities of Plasma Sprayed $\text{Al}_2\text{O}_3$ Coatings with Liquid Mn<sup>†</sup>

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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. Figure 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.

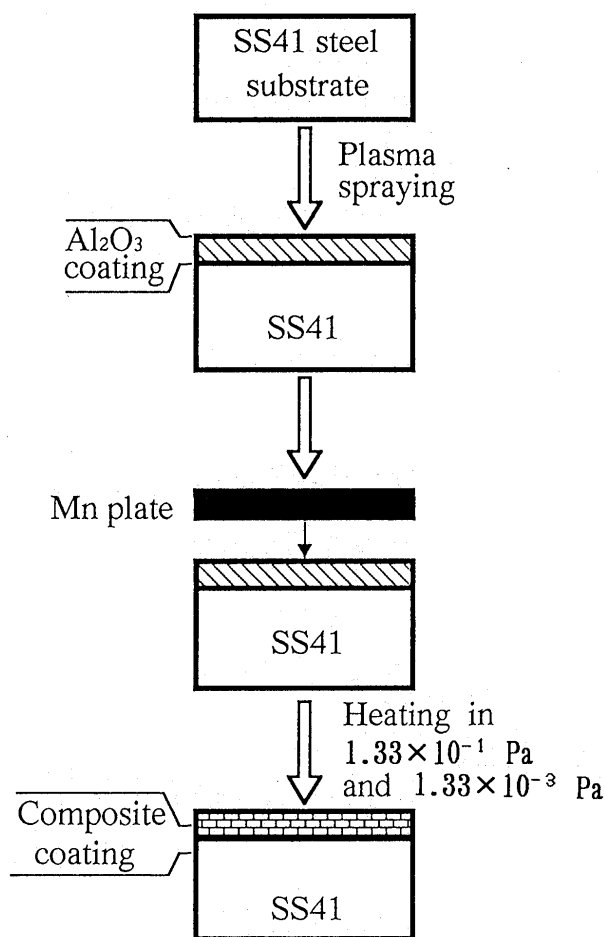


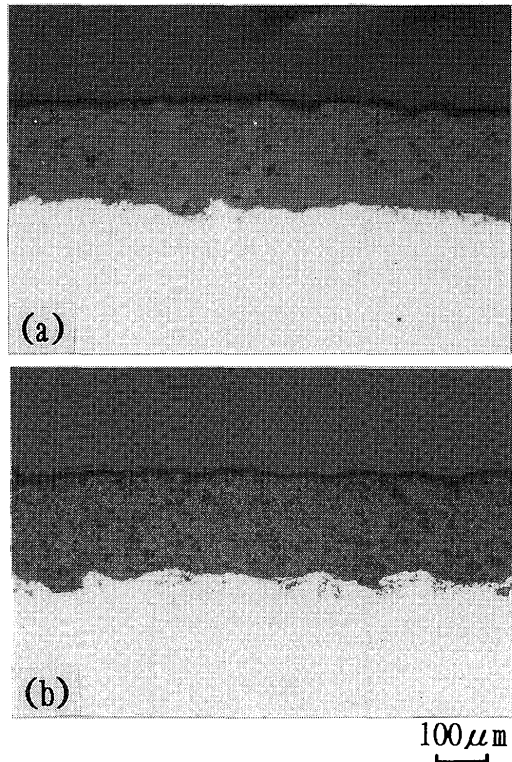
Fig.1 Processes of thermal spray and heating treatment with Mn.

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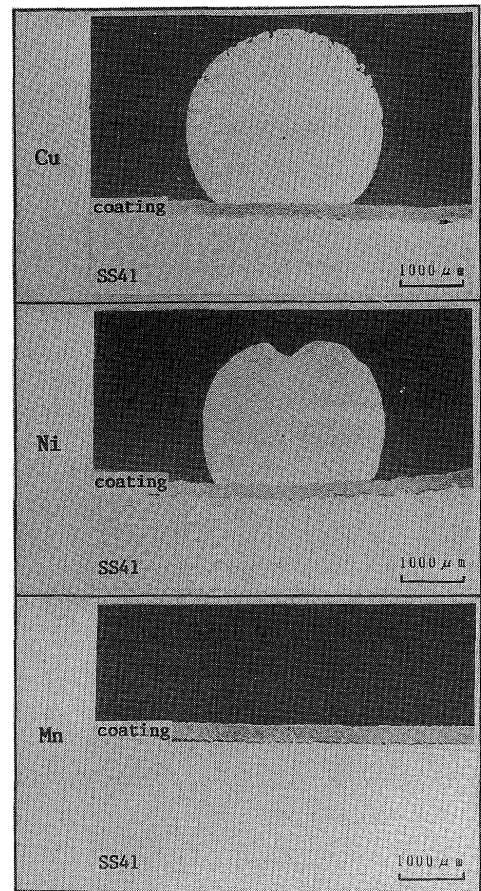
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**Fig.2** Microphotographs of Al<sub>2</sub>O<sub>3</sub> coatings. (a),as-coat;(b), heat-treatment (1573K,10.8ks,1.33 × 10<sup>-3</sup>Pa).

**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

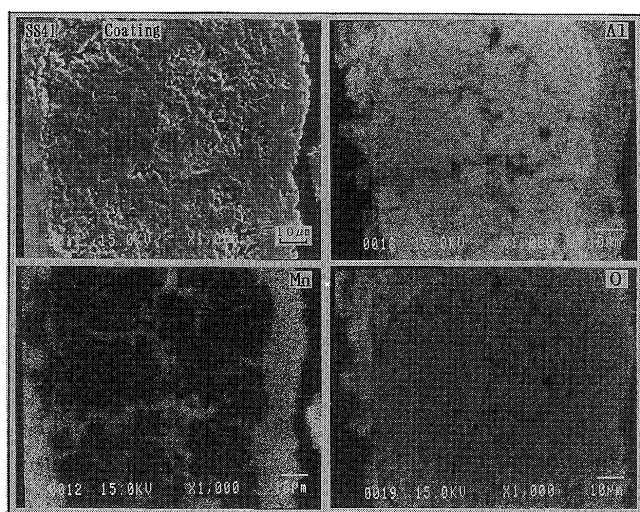


Fig.4 SEM microstructure and EPMA analyses of Mn treated  $\text{Al}_2\text{O}_3$  coatings.

the improvement of  $\text{Al}_2\text{O}_3$  coatings is possible by the penetration of the liquid Mn into the connected porosities of  $\text{Al}_2\text{O}_3$  coatings and the reaction of the liquid Mn and  $\text{Al}_2\text{O}_3$  particles when Mn heat-treatment was done.

#### References

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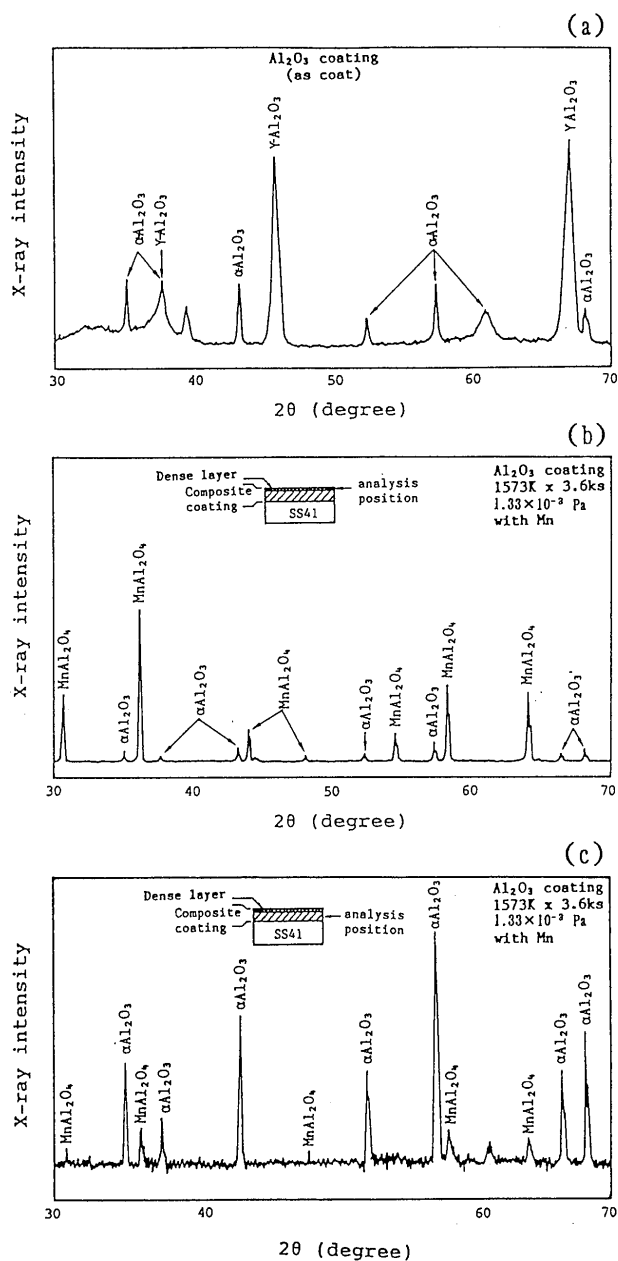


Fig.5 XRD results of  $\text{Al}_2\text{O}_3$  coatings after heat treatment with Mn.