Study of Bonding Strength of Plasma-Sprayed Ti-Al Coating on Mild Steel Substrate

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The effect of the surface roughness of a mild steel substrate on the adhesive strength of a plasma-sprayed Ti-Al coating was examined. The substrate surface was prepared by grit blasting or mirror polishing, and the bonding mechanism between the coating and the substrate is discussed. The adhesive strength of the Ti-Al coatings varied from 55 to 60 MPa with changes in the roughness from 1.2 to 7.5 μm. The adhesive strength of the Ti-Al coating sprayed onto a mirror-polished substrate was 25 MPa. The adhesive strength of a sprayed Ti coating was also affected by the roughness, but that of a sprayed Al coating was not. The bonding mechanism should be a combination of the mechanical anchoring of the Ti compound phase in the coating and the metallurgical bonding of the Al phase to the mild steel substrate.

Introduction

We developed a plasma-sprayed Ti-Al coating as an undercoating for Al2O3 coating with blended Ti-50 mass% Al powder, which consisted of TiN0.3, TiNO (TiN-TiO solid solution), Al and small amounts of Ti3Al. We have previously shown that these Ti compound phases of the Ti-Al coating are synthesized during the particle flight in a plasma jet,[1] and that the double-layered coating on a mild steel substrate has excellent adhesion and corrosion protection properties.

The adhesive strength of coating sprayed onto a metal substrate is usually considered to be due to mechanical anchoring, chemical (metallurgical) bonding, and van der Waals forces.[2] The substrate is generally treated with grit blasting before spraying to roughen and clean its surface. The roughened surface enhances mechanical anchoring of the coating, and the clean surface can also be expected to strengthen metallurgical bonding and physical bonding between the coating and the substrate.[3]

In this study, the effect of the surface roughness of a mild steel substrate on the bonding strength of sprayed Ti-Al coating was examined. The surface of the substrate was prepared by grit blasting or mirror polishing. For comparison, identical substrates with sprayed Ti coating and sprayed Al coating were also prepared, and their adhesive strengths were measured. The mechanical anchoring effect given by grit blasting and the effect of metallurgical bonding on the adhesive strength of the sprayed Ti-Al coating are discussed.

Experimental Part

Ti powder with a powder size distribution of 60–80 μm from Sumitomo Titanium Corporation, and Al powder with a powder size distribution of 40–60 μm from Hikari Sozai Corporation were used as starting materials for the plasma spraying. Ti powder was blended uniformly with the 50 mass% Al powder in advance, and then used for the spraying.

The spraying was performed using Aeroplasma Limited Company APS7050 system in an air atmosphere. Argon gas and compressed air were used as the plasma gas. Mild steel bars (JIS S5400, corresponding to ISO E275A) 25 mm in diameter with different surface roughnesses obtained by grit blasting or polishing were prepared. Three sizes of white alumina grit or...
alumina powder were used for the blasting. The mesh sizes of alumina grit were #24 (average diameter 1 mm), #46 (400 μm), #220 (80 μm), and the average diameter of the alumina powder was 60 μm. The mirror-polished substrates were prepared with a metallographic finish of 0.3 μm alumina polishing. The sample surfaces were cleaned to remove oxides from these finishing treatments. The Ti powder blended with Al powder was sprayed on the end face of the bars to form a Ti-Al coating, which was oversprayed four times, to a thickness of 100–150 μm under an input electrical power to the plasma of 27 kW and a spray distance of 0.1 m. The coated face was bonded to a bar of the same diameter by an epoxy resin adhesive to form test samples for adhesive strength measurement. The number of test samples was 5, to improve the measurement accuracy. Tensile adhesive strength measurement was carried out with an Instron type testing machine.

In addition, the mixed powder was sprayed onto a mirror-polished mild steel sheet (30 × 50 × 3 mm thick) by spraying of a single scan line, and the behaviors of Ti and Al splat formation were observed microscopically by using an optical microscope for an overview image and a SEM for a magnified image.

The surface roughness of the substrate was evaluated by using Form Talysurf S5, (Taylor Hobson Limited Company). Element distributions were measured by an electron probe microanalyzer (EPMA).

**Results and Discussion**

**Deposition Process of Sprayed Ti and Al Particles**

Figure 1 shows the distributions of Ti and Al splats on the mirror-polished steel substrate in a single horizontal spray line. The Al splats were fingered splats, and the Ti splats were disk-shaped splats with microcracks covering their surface. Al splats deposited mainly at the upper area of the line, and Ti splats deposited mainly at the lower area, with both splats being present in the center area.

The masses of Ti and Al particles were 7–9 × 10⁻⁴ and 1.4–2 × 10⁻⁴ mg, respectively, this was calculated numerically from the powder size and the specific gravity. The spraying powder was injected vertical to the direction of the plasma jet flow by means of airflow. So separation of the Ti and Al blended powder to the upper and lower areas of the spray line would be caused by differences in the masses of these particles.

Figure 2 shows a cross-section of the interface between the sprayed Ti-Al coating and the substrate obtained by SEM. The Ti-Al coating has a dense laminated structure composed of the Ti compound phase and the Al phase. In this work, the plasma torch was traversed on the end face of the sample bar from side-to-side in a horizontal direction, and moved down from the upper to the lower area of the face. So, the first particles deposited on the substrate should be mainly Ti powder, resulting in a larger area of direct contact between the Ti compound layer and the substrate.

**Correlation Between Surface Roughness and Adhesive Strength**

Figure 3 shows the effect of the average roughness ($R_a$) of the substrate on the adhesive strength of the sprayed Ti-Al coating. The $R_a$ of the blasted substrates was 1.2–7.5 μm,
and the adhesive strengths of the sprayed Ti-Al coatings varied from 55 to 60 MPa with changes in the $R_a$.

Figure 4 shows SEM images of fracture surfaces from the adhesive test. Point A with light contrast is the Ti compound phase, point B with dark contrast is the Al phase, and point C is substrate exposed by the deposit separation. Figure 5 shows the effect of the $R_a$ of the substrate on the ratio of exposed area of the substrate in the adhesive test. As for the coatings on the blasted substrates, the exposed area decreased with an increase in the $R_a$ of the substrate.

When a fracture occurs inside the coating during the adhesive test, the adhesive strength should indicate the tensile strength of the coating itself, and the adhesive strength between the coating and the substrate should be higher than the value of the measured adhesive strength. When the coating delaminates from the substrate, the measured adhesive strength should correspond approximately to the adhesive strength.

For the substrates prepared by grit blasting, the adhesive strength of the sprayed Ti-Al coating was affected slightly by the surface roughness of the substrate as shown in Figure 3, and also the ratio of the exposed area of the substrate decreased with an increase in the $R_a$ as shown in Figure 5. These results imply that the adhesive strength between the coating and the substrate roughened by grit blasting was partly due to the anchor effect caused by surface roughness. In contrast, for the mirror-polished substrate with an $R_a$ of 0.005 μm, the adhesive strength of the sprayed coating was 25 MPa as shown in Figure 3, and the ratio of exposed substrate was 27% as shown in Figure 5. The exposed area was very large in comparison to the coatings on the blasted substrates because the adhesive strength between the coating and the mirror-polished substrate was low.

**Adhesive Strength of Ti Compound Phase and Al Phase**

The adhesive strength of the sprayed Ti coating on the #24 grit-blasted substrate was 22 MPa. This measured adhesive strength should correspond to the tensile strength of the Ti coating, since the test sample was fractured inside the coating. For the sprayed Ti coating on the mirror-polished substrate, the adhesive strength was only 7 MPa. The coating delaminated partly from the substrate, and the ratio of the exposed area of the substrate was 18% on the fractured surface. This measured value of 7 MPa is very low in comparison with the tensile strength of the Ti coating. Therefore, it is reasonable that the starting point of the fracture should be the interface between the Ti coating and the substrate, and that the fracture should propagate inside the coating owing to the Ti coating containing many internal cracks and pores.\[1\] The measured value of 7 MPa would correspond to the adhesive strength of the Ti coating to the mirror-polished substrate. These results indicate that the bonding between the Ti compound phase in the Ti-Al coating and the substrate is mainly mechanical bonding affected by the surface roughness of the substrate.
The adhesive strength of sprayed Al coating on the #24 grit-blasted substrate was 35 MPa, and that on the mirror-polished substrate was also 35 MPa. The test sample of the former fractured inside the coating. The test sample of the latter fractured mainly inside the coating, and the ratio of exposed area on the fractured surface was only 2%. Consequently, the adhesive strengths of both Al coatings on the substrate were speculated to be over 35 MPa. Therefore, the adhesive strength of the Al coating would not be affected by the surface roughness of the substrate. It is expected that not only mechanical bonding, but also metallurgical bonding would contribute to the adhesive strength between the Al phase in the Ti-Al coating and the substrate.

Observation of Interface Between Sprayed Ti-Al Coating and Substrate

The FE-SEM images of the interface between the Ti-Al coating and the substrate show that the boundary is clearly divided at the interface between the Ti compound phase and the substrate. At the interface of the Al phase and the substrate, the boundary seems to be ambiguous compared to that between the Ti compound and the substrate. This might be caused by a reaction layer between the Al phase and the substrate.

The sprayed Ti-Al coating on the substrate was heat-treated at 873 K for 3 h under vacuum, and the interface of the coating and the substrate was observed by EPMA and is shown in Figure 6. A synthesized phase consisting of Al and Fe is identified at point A, so the reaction between the Al phase in the coating and the steel substrate could form a reaction layer during the spray-coating process. From a thermodynamic point of view, intermetallic phases can be formed in an Fe-Al binary system at 973 K. However, the O₂ and N₂ pressures in the plasma atmosphere are too high to form FeTi from Fe and TiO or TiN. This suggests that the Ti compound phase in the coating does not react with Fe in the substrate but that the Al phase does.

Conclusion

The difference in mass between the Ti particles and the Al particles caused separation of the blended powder in the plasma jet. From the plasma torch movements, the first layer of the coating would be mainly the Ti compound phase, resulting in only a small area of contact between the Al phase and the substrate.

The adhesive strength of the sprayed Ti coating was affected by the surface roughness of the substrate. For the sprayed Al coating, the surface roughness of the substrate did not affect the adhesive strength. With the heat-treated Ti-Al coating, synthesis of an Al-Fe phase at the interface of the Al phase and the substrate was observed. The obtained results suggest that the Ti compound phase bonds to the substrate by mechanical anchoring, while the Al phase bonds by metallurgical bonding to form a thin reaction layer.

In conclusion, the sprayed Ti-Al coating would bond to the mild steel substrate by a combination of mechanical anchoring and metallurgical bonding.

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