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Microstructural Characterization of Aluminum Powder Liquid Coating on IN 738 Superalloy

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Abstract

The powder liquid coating method was proposed for aluminizing of nickel-based superalloy grade IN738 in this study. Slurries for the coating consisted of aluminum and alumina (Al_2O_3) powder with various weight ratios in the range from 3:7 to 10:0. The slurries were pasted onto the IN738 specimens, which were subsequently dried and heated under argon atmosphere. Macrostructures of coated samples were observed. Surface and cross-sectional microstructures were studied by OM and SEM. Qualitative analysis and composition mapping were analyzed by EDS. Phase identification of coated surface was done by GIXD, at the incident angle of 2 degrees. The results showed inhomogeneous formation of Ni_2Al_3 on the coated surface. The Ni_2Al_3 phase had the structure of dispersed nodular islands and of thin discontinuous layer on flat area. Size and amount of nodular islands were larger when the ratio of Al:Al_2O_3 increased. Therefore, the amount of Ni_2Al_3 formation was increased with the ratio of Al:Al_2O_3. These nodular islands were formed due to the low wettability of liquid aluminum on nickel surface. Some aluminum oxide and chromium oxide were found in coexistence with Ni_2Al_3.

Introduction

Nickel-based superalloys are used as high temperature resistance materials. One of the most popular nickel-based superalloys is IN738, which is applied as a blade material for turbines and has been widely used in several other industries due to its superior creep rupture strength and corrosion resistance. However, in order to use Ni-based superalloy at high temperature, oxidation and hot corrosion resistance must be considered.[(1-2)]

Aluminum is highly resistant to oxidation, sulfidation, and degradation in chloride-containing aqueous solution. Therefore, aluminized coating is always an appropriate choice to be applied to nickel-based superalloys, resulting in formation of intermetallic compound such as nickel aluminide, which improves high temperature oxidation resistance and hot corrosion.[(3)] Nickel aluminide has a high melting temperature, low density, and excellent oxidation resistance. The high temperature oxidation and corrosion resistance of the aluminide can be explained by the formation of a dense continuous alumina (Al_2O_3) protective film which is formed after exposure to oxidizing environment. This layer acts as a good barrier to oxygen diffusion at high temperature; therefore, oxidation process is suppressed, allowing for a longer life time of the coated material.[(4-5)] In order to form a nickel aluminide film on nickel-based superalloys, a recently developed low cost aluminizing technique, the so-called 'powder liquid coating', is introduced. The process involves heat treatment of specimens in vacuum using a mixture of Al and Al_2O_3 powders as coating materials. In the present experiment, powder liquid coating is performed to coat nickel aluminide on nickel-based superalloy IN738. Microstructure and phases on the IN 738 coated surface are reported.

Experimental Procedure

Sample Preparation and Coating

Nickel-based superalloy grade IN738 was used as substrate. IN 738 superalloy was cut into rectangular shape with the size of 10 mmx10mmx5mm.

All samples were ground by SiC paper to #4000 grade and washed with acetone in an ultrasonic cleaning machine for 0.3 ks. Slurries were prepared with various conditions and pasted onto the samples. The slurries consisted of aluminum powder with the size of 160 μm, alumina powder, and ethylene glycol. The ratio of Al:Al_2O_3 by mass were varied from 3:7 to 10:0. After pasting, the samples were heated up to 473 K for 3.6 ks in order to remove the ethylene glycol. Then the samples were heated in the muffle furnace in argon atmosphere until the temperature reached 1273 K and held at aluminizing temperature (1273 K) for 14.4 ks. After aluminizing, the samples were cooled down in the furnace under argon atmosphere. The coating conditions of these samples are shown in Table 1.

Table 1. Coating conditions

Sample No.	Al:Al_2O_3 powder ratio	Temperature (K)	Atmosphere	Time (ks)
1	10:0 (by mass)	1273	Argon	14.4
2	7:3 (by mass)			
3	5:5 (by mass)			
4	3:7 (by mass)			

Characterization

After coating, the surface microstructure and cross-sectional microstructure were observed by optical microscope and Scanning Electron Microscope (SEM). Quantitative analysis of cross-sectional microstructure was done by Energy Dispersive Spectrometry (EDS). EDS and quantitative analysis were performed by ZAF technique. Phases formed on the surface of coated samples were identified by Glancing Incident-angle X-ray Diffraction (GIXD) at the incident angle of 2 degrees.

Result and Discussions

Surface Morphology

Macrostructures of the sample surface are shown in Figure 1. The surface macrostructure shows some nodular islands on the surfaces of all samples. Large islands are clearly visible on the surface of sample No. 1, which was coated by Al:Al_2O_3 with the ratio of 10:0. At lower ratio of Al to Al_2O_3 in samples No. 2-4, there is smaller number of islands on the surface. The size of the islands on the surface is also smaller when the ratio of Al to Al_2O_3 is decreased.

Microstructures of surface of the samples, taken by SEM, are shown in Figure 2. The islands on the sample surface have a protruding nodular shape. The nodular islands of the sample No. 1 have diameter ranges from one hundred as seen in its microstructure to several hundred microns as seen in macrostructure. In contrast, the diameter of islands on the surface of sample No. 4 is in the range of fifty to one hundred and fifty microns.

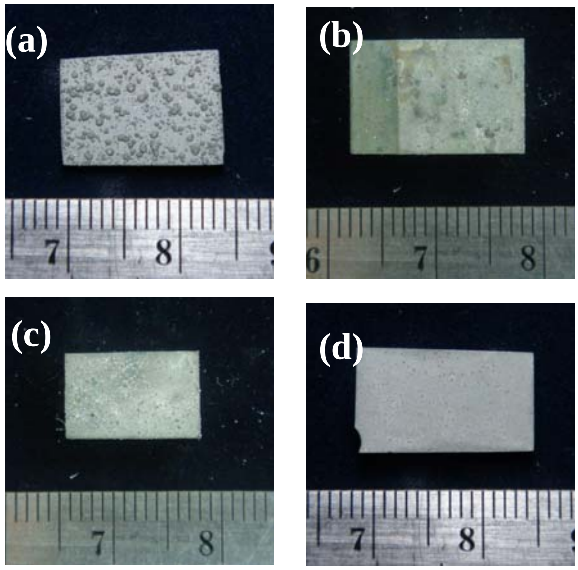

Figure 1. Macrostructures of sample surfaces coated by AlAl$_2$O$_3$ with the ratio of (a) 10:0 (b) 7:3 (c) 5:5 and (d) 3:7.

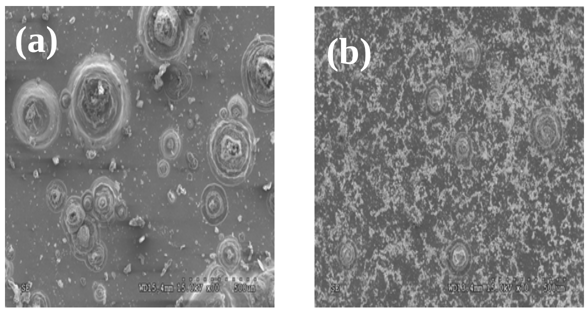

Figure 2. Microstructures of sample surfaces after coating by aluminizing with the ratio of AlAl$_2$O$_3$ (a) 10:0 and (b) 3:7.

Cross-Sectional Microstructure

The cross-sectional microstructure of a nodular island is shown in Figure 3. The nodular island consists of two phases which are identified as dark grey and light grey phases. Energy dispersive spectrometry analysis of cross-sectional microstructure of the island is shown in Figure 4. The nodular island consists mainly of aluminum and nickel with a small amount of chromium. The dark grey phase shown in Figure 3 corresponds to the area, which has high nickel and aluminum content while the light grey phase has high chromium content with some nickel and aluminum. EDS analysis shows that the islands formed on the surface of samples after coating are created by droplets of aluminum from molten aluminum powders. After aluminum powder was melted, some amount of liquid aluminum was trapped in the powder mixture, and some drops onto the substrate. Nickel dissolutes into these aluminum droplets and results in formation of nodular islands on the sample surfaces. These nodular islands, which consist of aluminum and nickel, appear to be a nickel aluminide phase with some amount of chromium. Phase identification carried out by GIXD will be shown in the following discussion.

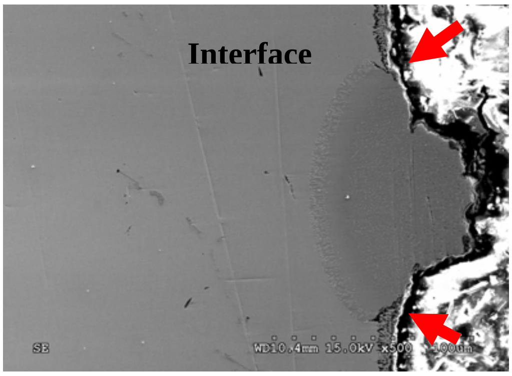

Figure 3. Cross-sectional microstructure of nodular island on the coated surface.

The dark grey phase as identified by the arrows in Figure 3 appears at the interface of the nodular island and substrate a layer of. This area consists of only nickel and aluminum as indicated in Figure 4 (a) and (b). It can be seen that some aluminum is left on the top surface of the nodular island as indicated by aluminum mapping in Figure 4 (b). In the same area, the amount of oxygen is also high as indicated in Figure 4 (d). This means that the aluminum, which has been left on the surface, becomes aluminum oxide after being exposed to ambient atmosphere.

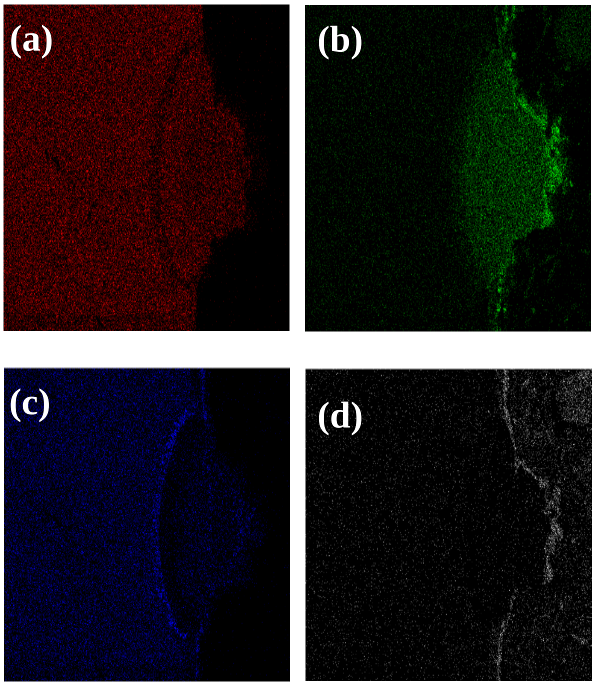

Figure 4. EDS mapping analysis of cross-sectional microstructure of the nodular island on the coated surface (a) nickel (b) aluminum (c) chromium and (d) oxygen.

Cross-sectional microstructures of the flat area of coated surfaces of samples No. 1 and No. 4 are shown in Figure 5. There is no significant difference in the microstructure of the two samples. This is because most of the applied aluminum in the slurry is in the nodular island, leaving only a small amount on the flat area. The cross-sectional microstructure of the flat area was analysed by energy dispersive spectrometry, as shown in Figure 6. The black area in Figures 5 and 6 consists of nickel coexisting with aluminum. Therefore, it should be nickel aluminide which will be further investigated by GIXD. On the top surface, the white phase in Figure 6 (a) shows coexistence of aluminum and oxygen. This means that some aluminum is left on the surface and reacts with oxygen in air after being exposed to the ambient atmosphere. This same situation occurs on the top surface of nodular islands as discussed above. The EDS mapping results of chromium and oxygen also show coexistence of chromium and oxygen on the top surface, which implies the possibility of chromium oxide formation on the top surface.

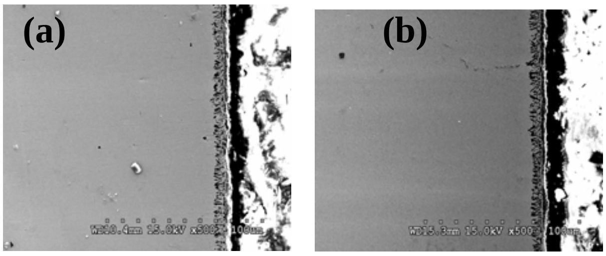

Figure 5. Cross-sectional microstructure of sample after coating by aluminizing with the ratio of $AlAl_2O_3$ (a) 10:0 and (b) 3:7.

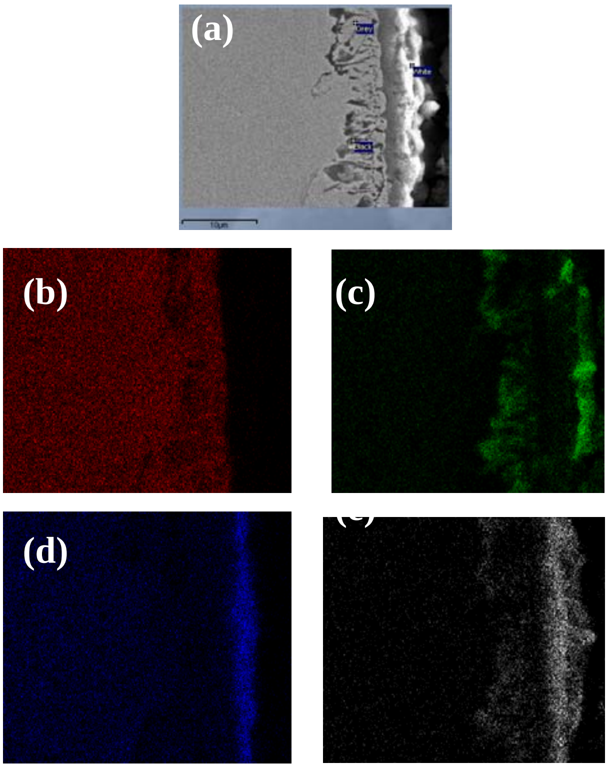

Figure 6. EDS analysis of the cross-sectional microstructure of the coated surface (a) microstructure (b) mapping of nickel (c) mapping of aluminum (d) mapping of chromium and (e) mapping of oxygen.

Phase Identification

Phase identification on the coated surface is shown by GIXD profiles in Figure 7. It is shown that the surfaces of coated samples consist of Ni_2Al_3 compound with a small amount of aluminum oxide and chromium oxide. From EDS analysis, as discussed in the previous section, it can be concluded that Ni_2Al_3 compound is formed mainly in the nodular islands, and to a lesser extent on the flat surface. On the other hand, the oxide film of chromium and aluminum is found mostly on the top of the flat surface. Samples No. 1 and No. 4 both have the same kinds of compound on the coated surface as shown by similarity of peaks in GIXD profiles. However, the intensity of Ni_2Al_3 peaks of sample No.1 is much higher than that of sample No. 4. This result indicates that Ni_2Al_3 volume fraction in sample No. 1 is higher than that of sample No. 4. The large volume fraction of Ni_2Al_3 formed on the coated surface relates to the large number of big nodular islands of Ni_2Al_3 on the surface of sample No. 1 compared to the smaller and fewer islands on the surface of sample No. 4. GIXD analysis corresponds well to the macrostructures and microstructures analysis.

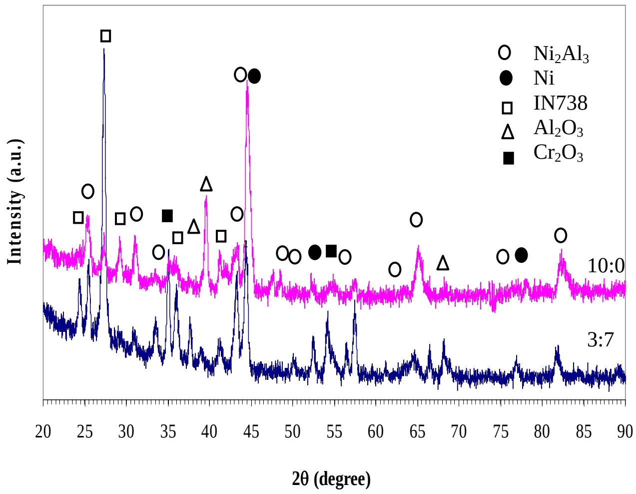

Figure 7. GIXD profiles at an incident angle of 2 degree of coated IN738 with different ratios of $AlAl_2O_3$.

The formation of nodular islands on the surface shows that liquid aluminum has a low wettability on nickel surface. The low wettability of aluminum on nickel surface is due to the formation of intermetallic compound Ni_2Al_3 which has a high melting temperature of 1406 K. Then, the liquid aluminum droplets are no longer in contact with the nickel surface but are sitting on top of the intermetallic layer, and are in effect, trapped by the Ni_2Al_3 intermetallic layer.[6] After that, the continuous upward growth of the Ni_2Al_3 phase takes place. This explanation clarifies the appearance of a cross-sectional microstructure which shows the formation of a Ni_2Al_3 layer at the interface of aluminum droplet and nickel surface, as indicated by the arrows in Figure 3.

Conclusion

1. Powder liquid coating of aluminum on nickel base superalloy grade IN738 shows the formation of Ni_2Al_3 as nodular islands on the surface. Some chromium oxide and aluminum oxide is found on the surface, coexisting with Ni_2Al_3.

2. The ratio of Al:Al_2O_3 affects the surface morphology and the amount of Ni_2Al_3 formed on a coated surface. The larger the ratio of Al:Al_2O_3 mixture of the slurry, the larger is the number of Ni_2Al_3 nodular islands that are formed and the larger is the diameter, also.

3. Formation of Ni_2Al_3 phase has structure of nodular islands due to poor wettability of liquid aluminum on nickel substrate.

Acknowledgement

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References

1. Pomeroy, M. J. 2005. *Mater. Design* **26** : 223-231.

2. Cheruvu, N. S. 2000. *Proceeding of 2000 International Joint Power Generation Conference*, July 23-26 : 1-8.

3. Murakami, K., Nishida, N., Osamura, K. and Tomota, Y. 2004. Aluminization of High Purity Iron by Powder Liquid Coating. *Acta Mater*. **52** : 1271-1281.

4. Houngninou, C., Chevalier, S. and Larpin, J. P. 2004. Synthesis and Characterisation of Pack Cemented Aluminide Coatings on Metals. *Appl. Surface Sci*. **236** : 256-269.

5. Chien, A., Gan, D. and Shen, P. 1996. Microstructures of Two-Stage Aluminized Coatings on Inconel 600. *Mater. Sci. Eng*. **A206** : 215-224.

6. Ip, S. W., Sridhar, R., Toguri, J.M., Stephenson, T.F. and Warner, A. E. M. 1998. Wettability of Nickel Coated Graphite by Aluminum. *Mater. Sci. Eng*. **A244** : 31-38.