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SYNTHESIS AND CHARCATERIZATION OF NANOSTRUCTURED Ni-Al COATINGS ON SUPERALLOY

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Abstract - The microstructural characteristics of nanostructured Ni-Al coatings fabricated by DC magnetron sputtering on the superalloy substrate have been investigated in the present work. FE-SEM/EDS and AFM were used to characterize the as deposited Ni-Al coatings and XRD analysis was used to identify the formation of different phases in the coatings..

Keywords - Nanostructured Ni-Al Coatings, DC Magnetron Sputtering, Microstructural characterization.

I. INTRODUCTION

Nanostructured coating is one of the potential materials to provide protection against high temperature oxidation of the superalloys used in high temperature applications such as gas turbine, thermal power plants, etc. **[1-7]**. It is well known that the adequate composition of Al in the conventional coatings imparts good cyclic oxidation resistance to the superalloys at 1000°C due to formation of a continuous alumina scale **[8-12].** The size effect of nanostructured coatings facilitates the enhanced diffusivity of atoms to form the continuous protective scales, which is normally discontinuous in the conventional coatings**[13].** Although the extensive literature is available on the microcrystalline coatings. Very limited literature is available on nanostructured Ni-Al coating on Ni-base superalloy. In order to increase the efficiency and resistance to oxidation for the long term operation of gas turbine, Ni and Al are two main elements for the formation of protective scale such as NiO, $NiAl_2O_4$ and Al_2O_3 in the coatings. Owing to these facts, as a first step, the present work has been focused to investigate microstructural characteristics of nanostructured Ni-Al coating on superalloy, deposited by DC-magnetron sputtering. The coating was fabricated by DC magnetron sputtering method on Superni-718 substrate at 400°C, 600°C and 800°C temperatures. The micro structural features of the coatings were characterized by FE-SEM/EDS and AFM. XRD was used to identify the different phases in the Ni-Al coating.

II. EXPERIMENTAL

2.1 Deposition of Ni-Al coatings by sputtering process

Ni-based superalloy namely Superni-718 (Inconel-718) has been chosen to deposit Ni–Al coatings in the present work. The superalloy was procured from Mishra Dhatu Nigam Limited, Hyderabad, India in annealed and cold rolled sheet form and its chemical composition is shown in **Table 1**. Each specimen measuring approximately: 18 mm (L) × 15 mm (W) × 3 mm (T) was cut from the rolled sheet and grinded by using 220, 320,down to 1000 grit. Subsequently, it is polished on cloth polishing disc by using alumina powder followed by diamond paste. Commercially available Ni Target (99.99% pure) with dimension 50.8 mm diameter and 2.0 mm thickness and similarly, commercially available Al target (99.99% pure) with 50.8 mm diameter and 5.0 mm thickness were fixed at an angle of 45° to each other in the sputtering chamber. With the use of rotator, the substrate heater was rotated between Ni and Al targets continuously to perform a co-sputtering. Firstly one side of the substrate has been coated in chamber at one time, after that it is turned the side of the substrate and do the coating operation. Thus all six faces (both side) of the substrate have been coated. Sputtering chamber is spherical and diameter is 304.8 mm. The target to substrate distance was fixed at 40 mm during deposition. Before starting the deposition, the targets were pre-sputtered for 15 minutes with a shutter located in between the targets and the substrate. The shutter is also used to control the deposition time. All the samples were cleaned in acetone, ethanol and deionized water prior to the deposition of Ni–Al coatings. The process parameters used in DC magnetron sputtering are shown in **Table 2.** At the substrate temperature above 600^0 C, the deposited coatings peeled off due to the induced thermal stress in the coatings, the Ni-Al coatings were deposited on Superni- 718 at substrate temperatures, i.e. 400°C and 600°C .The nanostructured Ni-Al coatings deposited on Superni-718 were subjected to XRD, FE-SEM/EDS, and AFM analysis.

Table 1 Chemical composition of superalloy used in study

	Chemical composition, Wt-%												
Midhani Grade	Fe	Ni	Cr	Ti	Al	Mo	Mn	Si	Co	Nb	P	C	S
Superni-718	19.8	Bal	17.6	0.96	0.53	3.23	0.02	0.03	0.01	4.91	0.005	0.02	0.007

Table 2 Sputtering parameters for Ni-Al coatings

Target	Al (99.99% pure, 2 inch diameter & 5mm thickness) Ni (99.99% pure, 2 inch diameter & 2 mm thickness)
Base pressure	3 X 10^{-5} Torr
Deposition gas pressure (Ar)	10 m Torr
Deposition power	50 W/150 W for Ni/Al target
Deposition time for Ni-Al Coating	90 minute
Substrate	Superni-718
Substrate Temperature	400°C & 600° C
Total Deposition time	90 minute

2.2 Characterization of Ni-Al coatings

XRD (Bruker AXS, D8 Advance) measurements of the as deposited coatings were carried out by using CuK$_\alpha$ radiation. The scan rate and the scan range used were 0.1 step/sec and from 10° to 100°, respectively. An average grain size, d of the Ni-Al coatings is estimated by using its XRD peak broadening according to Scherrer formula **[14]**, as given in Eq. (1)

$$d = \frac{0.9\lambda}{B\cos\theta_B} \qquad (1)$$

Where λ, θ_B and B are the X-ray wavelength (1.54056A°), Bragg diffraction angle and line width at half maximum. Instrumental broadening has been accounted for the calculation of grain size, and its value of 0.1(for standard Si sample) has been subtracted from the full-width half maximum (FWHM) value, from B value.

FE-SEM (FEI, Quanta 200F) has been used to characterize the microstructures of the Ni-Al coatings at an acceleration voltage of 20 kV.

III. RESULTS AND DISCUSSIONS

3.1 XRD Analysis of the coatings

The XRD peak of as deposited Ni-Al coating on superalloy substrate is shown in **Fig. 1**. It shows the formation Ni_3Al_4 and Ni_3Al phases in the sample. The crystallite size of the as deposited Ni-Al coated sample was calculated from the X-ray peak broadening and It was found to be in the range of 20-45 nm and 25.8 nm and 38.7 nm coating at 400°C and 600°C respectively.

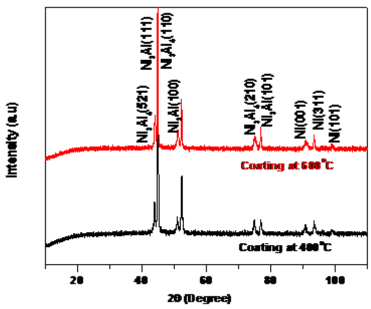

Figure 1 XRD pattern of as deposited Ni-Al coatings on Superni-718 at different substrate temperatures

3.2 AFM Analysis of the coatings

Fig. 2(a-d) shows the 2D and 3D AFM images of the Ni-Al coatings deposited at two different substrate temperatures. 2D image of Fig.2 (a) indicates that coating has some voids and small spherical grains, where as 2D image of Fig.2 (c) shows that it has dense coating and elongated grains.

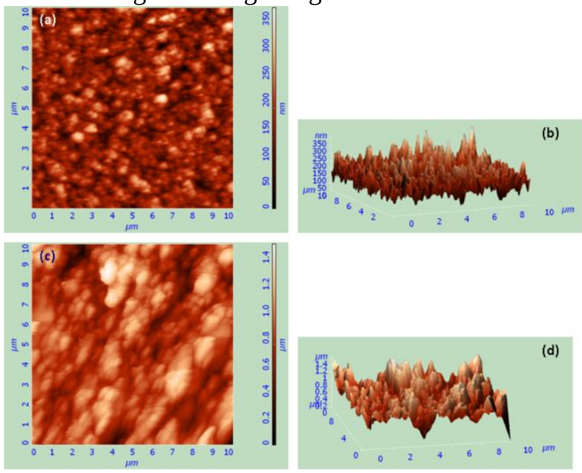

Figure 2 AFM 2D & 3D images of Ni-Al coatings on Superni-718 substrate at different substrate temperatures (a-b) at 400°C, and (c-d) at 600°C

3.3 Microstructural characterization of Ni-Al coatings

Fig. 3(a-b) shows the FE-SEM surface micrographs with EDS at different points on the surface of deposited Ni-Al coatings at 400°C and 600°C substrate temperature. The observed microstructural features of the coatings can be classified by using the structure zone model proposed by Meissier **[15].** The homogeneous temperature, (ratio of substrate temperature to melting point of the coating materials), is used to classify the coating microstructures observed at different substrate temperature during sputter deposition of the coatings. In the present work, the coating deposited at 400°C exhibits a homogeneous temperature of 0.41 and showed small spherical shape grains in bunch form which approaches zone 2 structures as reported in Meissier's work. The homogeneous temperature (T_S/T_m) is 0.51 for the coatings deposited at 600°C and the elongated shape grains, and dense coating are observed at this deposition temperature, which comes under zone 3 structure. With increase in substrate temperature, the surface mobility of condensed atoms increases, which could easily diffuse from island side to lower potential zone of substrate, resulting in denser coating with reduced porosity or voids in coatings. The thickness of coatings was calculated by its cross sectional FE-SEM images and it was found to be approximately 2 µm. The cross-sectional FE-SEM micrographs of as deposited Ni-Al coating on Superni-718 at two different substrate temperatures are shown in **Fig 3 (c-d).** Coating at 400°C substrate temperature has fine grains(Fig.3 (c)), where as coating at 600°C substrate has thick grain(Fig.3

(d)).Thus, as per AFM analysis and FE-SEM surface and cross-sectional analysis it is observed that coating at 600^{o}C substrate temperature is more dense as compare to coating at 400^{o}C substrate temperature. The detailed chemical compositions of the as deposited Ni-Al coating on Superni-718 substrate has been analyzed at the surface as well as the depth profile as shown in cross-sectional FE-SEM/EDS micrographs (Fig.3(e)).

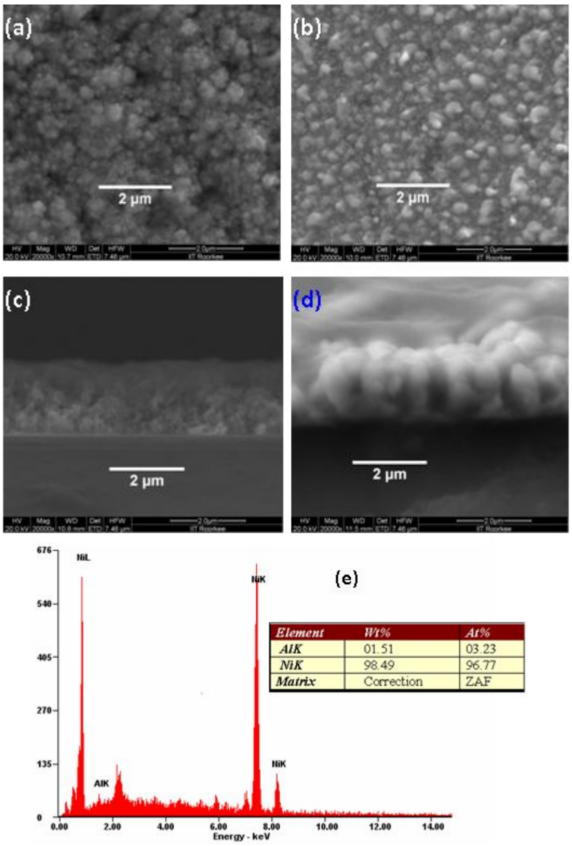

Figure 3 FE-SEM/EDS surface micrographs (a-b) of Ni-Al coatings on Superni-718 substrate at different substrate temperatures (a) at 400^{o}C, and (b) at 600^{o}C , and (c-d) FE-SEM/EDS cross-sectional micrographs of Ni-Al coatings on Superni-718 substrate at different substrate temperatures (c) at 400^{o}C and (d) at 600^{o}C and (e) The detailed chemical compositions of the as deposited Ni-Al coatings, on Superni-718 substrate(Analyzed by depth profile on surface as well as cross-sectional).

The nanostructure Ni-Al coating was designed to resist the high temperature oxidation as reported in the literature **[13].** An alumina scale can form at elevated temperatures due to the high aluminum activity (content) in the coating and the greater negative value of the Gibbs energy for the formation of alumina than that for nickel oxide. Therefore, the nanostructured Ni-Al coatings has been fabricated. It is observed that the coatings exhibit voids and dense morphology at 400^{o}C and 600^{o}C substrate temperature, respectively. The dense morphology of the coating is expected to provide a better corrosion/oxidation resistance. The typical grain size of the coatings is around 20-45 nm, which is essential for facilitating the formation of protective scales at high temperature. The alumina scale in the deposited Ni-Al coated sample may behaves as a diffusion barrier to limit the outward and inward diffusion of the atoms across substrate-coating interface. It may be mentioned that the lifetime of the coated components in gas turbine engines would be enhanced by making use of the nanostructured coatings

CONCLUSION

In the present work, nanostructured Ni-Al coating on Superni-718 has been deposited by DC magnetron sputtering method. As deposited coated superalloy samples were characterized by FE-SEM/EDS, AFM, and XRD. It is observed that coating exhibits porous and dense morphology at substrate temperature 400^{o}C and 600^{o}C respectively. The grain size of the coatings was observed to be around 20-45 nm.

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