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TITANIUM – ALUMINIUM INTERMETALLIC THIN FILMS PREPARATION BY DC SPUTTERING AND THEIR CHARACTERIZATION

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Abstract. DC magnetron sputtering is a well-developed deposition technique for coatings and thin films used in industrial applications. The experiments were performed with unbalanced circular magnetron sputtering targets of aluminium (99.999%) and titanium (99.99%). Sputtering of aluminium (Al) and titanium (Ti) was carried out in pure argon (99.999%) atmosphere at base pressure of 4×10^{-6} torr and constant sputtering pressure of 5×10^{-3} torr. Substrate materials were mainly stainless steel (304) and aluminum plates. Characterization of TiAl films deposited onto different substrates was evaluated using XRD, SEM and EDS analysis techniques. The film surface and cross-section was examined using a scanning electron microscope (SEM). The TiAl phase was confirmed using XRD analysis. The composition of the TiAl film was determined using EDS technique. These characterizations revealed the growth of TiAl intermetallic thinfilm with a characteristic crystallite size of 123.9 Å and a lattice strain of 0.1352%. Also a columnar growth perpendicular to the substrate surface was observed repeatedly in our experiment. The microhardness of the TiAl film had an average value of 1873 HV.

Keywords: Magnetron Sputtering, XRD, SEM, Microhardness.

1 Introduction

The surface properties of structural units are greatly influenced by combination of mechanical, tribological, corrosive and thermal atmospheres. Under such circumstances protective layer coatings are employed to overcome modification of surface properties [1, 2]. Due to the presence of coatings with low thermal conductivity, a temperature gradient is established across interface between the bulk material and coating. Hence the temperature in bulk material remains low compared to uncoated region of it and pushes the service temperature limit of the materials above the normal service temperature. The coatings will reduce the material cost and consumption for making such systems. The other advantages of the coatings are superior tolerance against straining, erosion, corrosion, thermal shock etc. Titanium (Ti) Aluminide (Al) intermetallic coating is one of such coating which is widely used for these types of applications [3-6]. The main properties of interest for these materials are high temperature strength and high temperature corrosion resistance. Also they frequently exhibit very good resistance to high temperature corrosion due to the formation of oxide rich films [7]. The Ti Al coatings are of intense interest to the gas turbine and aircraft engine industries where their high temperature properties and low density offer prospectus for tremendous weight savings. Also Ti Al are finding several application in power generation and automotive industries because of their superior mechanical properties such as low density, elevated temperature strength [8], oxidation and corrosion resistance [9,10] with improved wear, friction and creep deformation due to lamellar nature of TiAl compared to super alloys [11]. Titanium aluminide intermetallics are believed to have potential applications as high temperature materials to replace Ni based superalloys due to their low density and excellent high temperature strength. Ti – Al intermetallics form particular crystal structures with ordered atom distribution where atoms are preferentially surrounded by unlike atoms. If the aluminium content is below 50 at.% in the coated Ti Al film forms nonprotective scales of mixed $TiO_2 + Al_2O_3$ at elevated working temperatures.

2 Experimental

In this work intermetallic Titanium Aluminide (TiAl) thin films were prepared by DC magnetron sputtering. The coating of the Ti – Al was done using high purity titanium (Ti – 99.99%) and aluminum (Al – 99.999%) from two different individual sputtering targets. The stainless steel deposition chamber evacuated to a base pressure in the range of $3 - 6 \times 10^{-6}$ torr. After evacuation, Argon 99.999% purity was introduced into the chamber using gas mass flow controller attached to the system. The base pressure in the system during sputtering was maintained in the range of 4.0 – 5.0×10^{-3} torr by using throttle valve. The targets were kept at a relatively low temperature. The targets were presputtered prior to sputtering. The films were deposited onto mirror polished substrates of rectangular discs of stainless steel (304) and aluminium of dimension 10mm X 15mm X 2mm. The deposition rate of each target was controlled separately at an evaporation ratio of Al/Ti in the range of 1:3. The cleaning of the substrates and surface roughness minimization were done using electropolishing. The substrates were fixed to a rotatable and heatable circular (3inch) substrate holder. The substrate temperature was set at 390°C. Then the substrates were subjected to the sputtering process in the magnetron sputtering system.

2.1 Characterization

Studies on the compositional properties of the thin films were performed using X – ray diffractometer (JEOL Model- DX-GE-2P) for composition and phase analysis. The thin films were also studied using energy dispersion spectroscopy (EDS) and scanning electron microscopy (SEM - JEOL / JSM - 6380LA). The focus was given for the composition and hardness of the coated thin film in relation to their growth process. The microhardness of thin films was analyzed using CLEMEX microhardness tester.

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3 Results and Discussion

3.1 X-Ray Analysis

In XRD the coated sample of the material to be analyzed is placed in a sample holder, then the sample is illuminated with x-rays of Copper K_α (30kV, 20mA) wave-length (λ_α = 1.5406Å) with a K_β filter and the intensity of the reflected radiation is recorded using goniometer. The diffracting angles were set to 20° to 80° with a low scanning speed (1°/min) and in continuous mode. The prepared coatings were both amorphous and crystalline in nature. The diffraction peaks, corresponding to that of the Titanium (Ti) – Aluminum (Al) match with the JCPDS data (47-1137) of $Al_{1+x}Ti_{1-x}$ (Orthorhombic). The amorphous nature of the coating was observed in the 2θ range of 20 to 32 degrees. The corresponding peak position, miller indices and the texture coefficients were as listed in the table1. This characterization revealed the growth of TiAl intermetallic thin film with a characteristic crystallite size of 123.9Å and a lattice microstrain of 0.1352%.

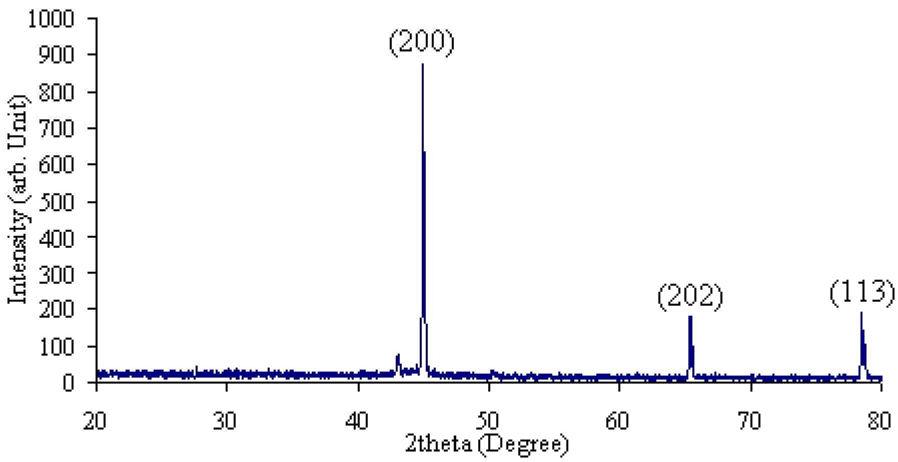

Fig. 1. X-ray Diffractogram of TiAl thin film coated on nanoporous alumina

Table 1. Texture coefficients of the XRD peaks of the TiAl thin films

Sl. No.	Peak Position (degree)	(hkl)	Texture coefficient (T)
1	44.99	(200)	0.832
2	65.56	(202)	0.079
3	78.89	(113)	0.088

3.2 Scanning Electron Microscopy

The scanning electron microscope images have a characteristic three-dimensional appearance and are useful for judging the surface structure of the coated surface. The Energy Dispersive X-Ray Spectroscopy (EDS) is used in conjunction with SEM for chemical microanalysis technique. This revealed the composition of the film and it was found to be 48% titanium and 51% aluminium. The EDS carried out on the cross-sections revealed the presence of TiAl along the film thickness and found to be constant thought the film thickness.

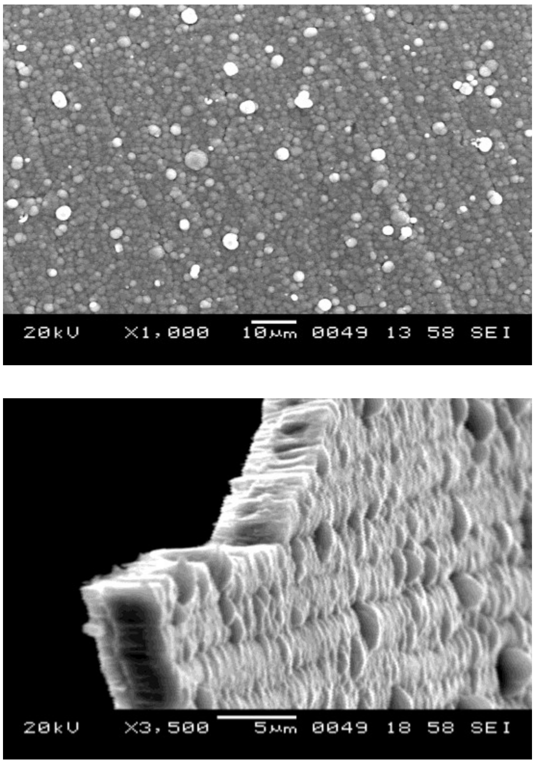

Fig. 2. Scanning Electron Microscopic images of TiAl thin films (a) top view and (b) cross-sectional view

3.3 Microhardness

The microhardness characterization of the thin films coated on different substrates with coating thickness of 1.2µm was carried out using Clemex microhardness tester. A Vickers indenter was impressed into the material at loads of a few grams. The impression length was measured microscopically, and the test loads were used to calculate the hardness value. Microhardness tests differentiated the relative hardness of different substrates of intermetallic TiAl film. The average microhardness values were as listed in the table 2 below and are in agreement with the literature values. It was observed that the substrates are influencing the microhardness values of the coated TiAl films largely.

Table 2. Microhardness values of the TiAl thin films coated on different substrates

Sl. No.	Substrates	Microhardness (HV) of the TiAl film
1	Copper	1837
2	Aluminium Anodisc	1903
3	Aluminum	1600
4	Mild Steel	1858

4 Conclusion

The TiAl films were developed by unbalanced dc magnetron sputtering on various substrates. The experiments carried out yielded the growth of intermetallic $Ti_{1-x}Al_{1+x}$ films which was confirmed by EDS and XRD analysis. The XRD analysis revealed the crystallite size was 123.9Å and lattice microstrain was found to be 0.1352%. The grown $Ti_{1-x}Al_{1+x}$ films were found to be in gamma region of TiAl phase diagram. There were columnar growths of the film on the substrate with good adherence. The microhardness studies of the deposited films were carried out and the values obtained were found to be in accordance with the literature.

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