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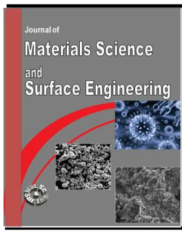

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Compositional Control of Co-deposited TiAl Film using Dual Magnetron System

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Abstract

Titanium Aluminum (TiAl) films have been deposited by physical vapor deposition method using two unbalanced planar magnetrons. Pure Titanium (Ti) and Aluminum (Al) sputtering targets were used for the co-deposition of TiAl films of varying compositions. Precise control of the sputtering yields of target materials were achieved through optimization of applied power. This in turn controlled the composition of the Ti and Al in the grown films.The films were obtained at applied power levels of '50W to 900W' and their compositions were established and affirmed by EDAX measurements. Amorphous TiAl films were formed for a higher Al composition (i.e. from 20 % to 50 Atomic %), while crystalline film was formed for low Al composition (i.e. 10%). Crystallinity of the TiAl film was improved at post annealing temperature of 800°C.

Introduction

Titanium Aluminum (TiAl) alloy coatings are promising materials for a wide range of applications depending on the alloy composition and its corresponding properties. These alloys are used in aerospace application and automobile industries because of its low density, resistance to oxidation and unique thrust to weight ratio[1].These alloys are also considered as promising candidates for high temperature structural materials because of high melting temperature and reasonable oxidation resistance[2]. TiAl is also a candidate as a potential conductor and/or diffusion barrier for high temperature electronics because of their high melting points and high oxidation resistance[2].

Techniques such as co-evaporation, co-sputtering etc. are available to produce coating with one or more elements in the film. These coatings have well defined structural and mechanical properties. Due to the ease of operation physical vapor deposition (PVD) technique, especially magnetron co-sputtering is being increasingly used for deposition of TiAl thin films[3-4]. Hampshire et al.[5-6] have shown that an intermetallic phase of TiAl has been formed for specific composition only. Amorphous TiAl alloy films deposited by sputter deposition from dual target of pure Ti and Al have been found to exhibit extended periods of passivity in chloride-based solution[7]. Synthesis of TiAl thin film by co-sputtering has been performed at different temperatures[1,8]. However, such coatings are usually prepared using composite target material or sectioned targets. These composite targets limit the composition in the grown film due to the differences in the sputtering yields of target material. After long operation material with higher sputtering yield sputter faster and changes the composition of materials in the composite target[9]. Also one kind of composite target can be used to grow a film of only fix composition. Therefore, usually only one or two sets of TiAl composition are reported not a range of composition and their control using magnetron sputtering deposition.

In this research, it is shown that dual Magnetron sputtering system can be configured such that two different target materials are simultaneously sputtered with different powers to control the composition of material in the grown film. In this manner, the desired composition of the target material can be maintained and also composition in the grown film can be varied. For the confirmation of the power controlled sputtering yield, study of Ti and Al co-deposition is presented and TiAl film of various compositions is reported by controlling the power of individual target. The results are interpreted with SEM, EDAX and XRD mappings.

Materials and Experimental

Materials

Experiments are carried out using co-sputtering of Ti (99.99% purity) and Al (99.99% purity) metals to get variable compositions of Al from 10% to 50 % in the TiAl film. Studies are performed to compare the structure of the film at different compositions. Initially deposition rate at different powers for both Ti and Al are measured by using in-situ thickness monitor and the sputtering yield of Ti and Al is calculated independently by using TRIDYN code[10]. Film thickness of ~ 1 µm was kept constant in all the experiments. Based on deposition rate and sputtering yield of Ti and Al, required power to the individual metal target is estimated. TiAl film is deposited at different power levels to get required composition of Al in Ti film. The composition of Ti and Al in the TiAl film is measured by EDAX. Also condition for amorphous and crystalline TiAl film formation is reported with XRD measurements.

Experimental Method

Schematic diagram of experimental setup is shown in Fig. 1. It consists of cylindrical vacuum chamber connected to diffusion and rotary pump. Base vacuum level of the system is $5x10^{-6}$ mbar. Both the magnetrons are inclined to 39° to the vertical axis of the substrate. The substrate is 10 cm away from the centre of each target. The target materials are positioned such that the sputtered flux combines at the substrate plane. 3" Ti target and Al target of

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equal size and thickness were mounted in the planar magnetron system. Both the unbalanced magnetrons are controlled independently by two 1 kV/3A D.C. power supplies. The composition of Ti and Al film is varied by varying power to each of the magnetron from 50W to 900W. The TiAl films were deposited on Si(111) substrate. The operating pressure during deposition was kept at 5×10^{-3} mbar with argon gas. The deposition time for all the experiments is 30 min. The deposited film was annealed at 500°C and 800°C for 2 h and films were analysed by EDAX, XRD and SEM in the as deposited state and after high temperature annealing.

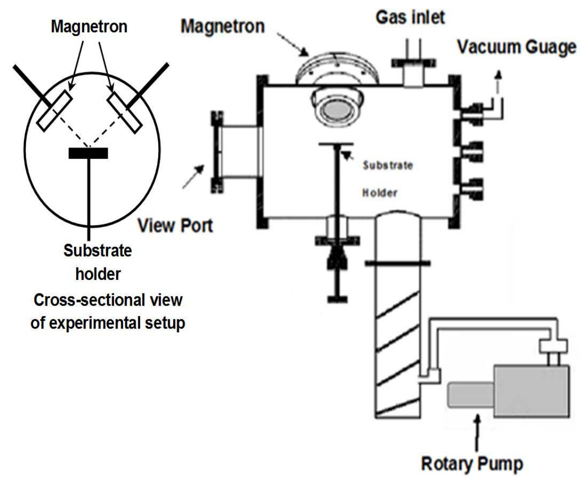

Figure 1: Schematic diagram of dual magnetron experimental setup.

Results and Discussion

Composition results

Initially, deposition rate of Ti and Al is measured by using thickness monitor at the substrate plane. The deposition rates were measured at different power levels from 50 to 900 W in the steps of 100 W. Figure 2 (a) shows deposition rate of Ti and Al at different applied powers. Deposition rate varies starting from 1A/s to 25A/s depending upon the applied power. Corresponding sputtering yield plots of both Ti and Al are shown at the different Ar ion energies in Fig. 2 (b). Sputtering yield is calculated using TRIDYN software[10]. TRIDYN software takes care of the dynamical changes on the surface after ion bombardment and gives more accurate information of the sputtering yield. It shows that Al has higher deposition rate than Ti due to higher sputtering yield of Al. It is obvious from the plot that there is almost a linear relation between the applied power and the rate of deposition for both Ti and Al, respectively. Based on this graph the power required for both Ti and Al target for different Ti and Al compositions was estimated. It is also clear from Fig. 2 (b) that Al has a higher sputtering yield than Ti, therefore if their composite target is used for TiAl film growth, composition will keep on changing at different applied powers and after long sputtering hours.

In the example shown in Fig. 2(a), the same applied power is chosen for both Ti and Al sputtering, however to tune the composition different applied powers to the targets can be chosen. An example is shown in Fig. 3(a) and Fig. 3(b), the atomic percentages of Ti and Al varied from 90/10 to 80/20 as measured by EDAX. Fig. 3(a) and 3(b) clearly demonstrate that by changing the applied power, the Al and Ti concentrations are also changed in the films. The measured atomic percentage corresponding to the plot shown in Fig. 2(a) and EDAX analysis are summarised in table-1. For Al sputtering power of 20 W and Ti sputtering power of 400 W TiAl film of 90:10 ratio is formed , similarly for 20 W Al and 200 W Ti 80:20 ratio TiAl film is formed. In the similar manner other composition of the film are grown as shown in the table-1. Hence it is confirmed that by controlling power of the individual targets, the atomic compositions in the film can be controlled.

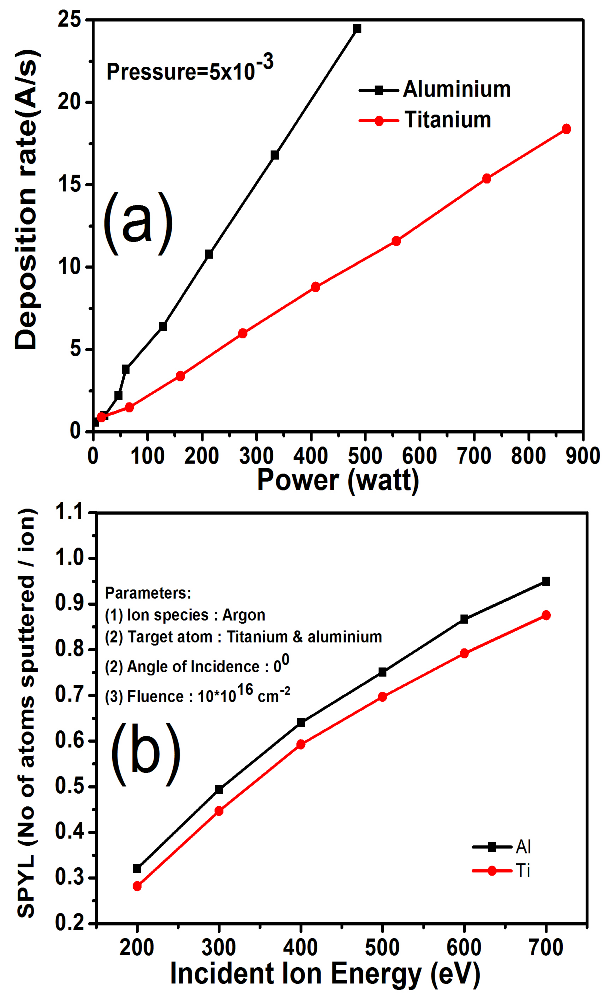

Figure 2: (a) Deposition rates of Ti and Al at different applied powers. (b) Sputtering Yield of Al and Ti calculated using TRIDYN at different energies of bombarded ions at normal incidence to the target.

Table 1: Planed and obtained % composition of Ti and Al at different applied powers to the magnetron targets.

S. No.	% Composition of Al and TI	Aluminum Applied power (watt)	Titanium Applied power (watt)	Obtained % composition of Al and Ti By EDAX
1	10-90	20	400	8-92
2	20-80	20	200	18-82
3	30-70	20	120	29-71
4	40-60	45	190	36-64
5	50-50	45	80	55-45

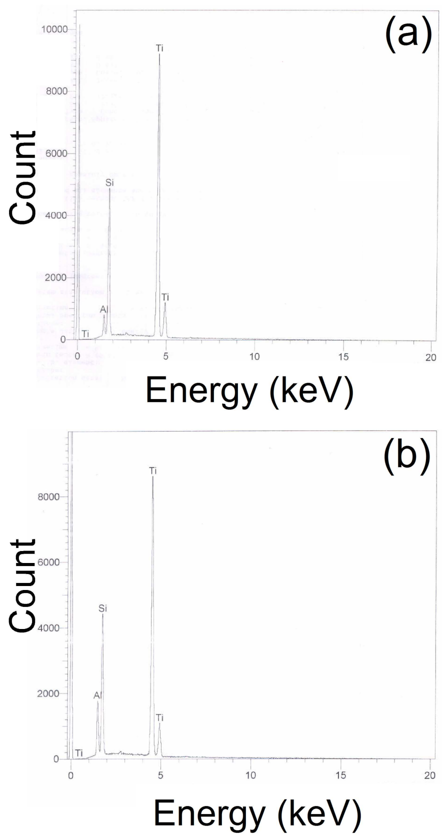

Figure 3: (a) EDAX measurements of 10 % Al and 90% Ti (b) 20% Al and 80% Ti in the grown TiAl film.

SEM Results

Figure 4(a, b) show the cross-sectional view, and Fig. 4 (c, d) shows the corresponding surface morphology, respectively for the 90 % Ti and 10 % Al, TiAl film in as deposited and after annealing at 800°C. As is obvious from Fig. 4(b, d) that after annealing film becomes denser, smoother and columnar structures disappear. This essentially means Al incorporation, and post annealing readjusts the atoms, and gives them additional energy to make denser and smooth film. Same comparison is shown in the surface morphology in Fig. 4 (c) and Fig. 4(d). In as gown film small clusters like grains can be seen on the surface (Fig. 4-c), which are missing in Fig. 4(d) after the annealing.

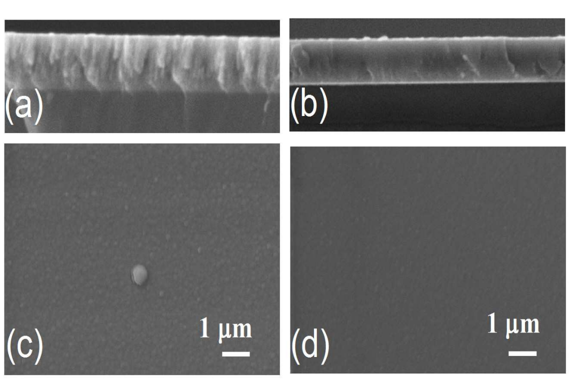

Figure 4: (a) Cross-sectional view of as deposited film, (b) Cross-sectional view of 800°C annealed film. (c) Surface morphology of as deposited film and (d) surface morphology of 800°C annealed TiAl film of 90% Ti and 10 % Al.

XRD Results

XRD results were compared before and after annealing the TiAl film to determine available phases and heat treatment effects on the structure of the film. It was observed that as the Al percentage in the film increases, the crystalline nature of the film decreases as shown in XRD pattern of Fig. 5 (a). The 90% Ti and 10% Al film shows TiAl (111) phase but this peak is suppressed when Al concentration increases to 20 % and makes an amorphous TiAl phase. Intensity of this peak decreases as the Al percentage in the film is increased. No other peaks were observed in XRD results. After annealing at 500°C and 800°C, the width of the TiAl peak decreases indicating larger grain size formation (Fig. 5(c and d)). TiAl film deposited by sputter deposition has the tendency to make amorphous film as ad-atom energy in the sputtering process is usually so high to maintain the crystallinity. The formation of TiAl (111) phase is because of growth parallel to Si (111) substrate[1,11].

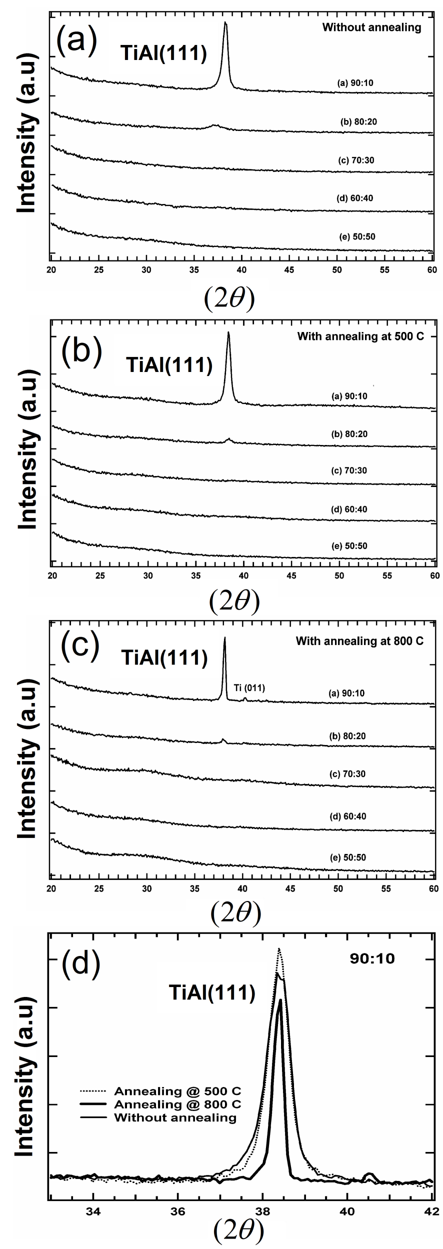

Figure 5: (a) XRD traces for as deposited TiAl films for different Al percentage, (b) XRD traces for films annealed at 500°C for different Al percentage, (c) XRD traces for films annealed at 800°C for different Al percentage, (d) Peak and FWHM decreases with annealing temperature.

Conclusions

TiAl coatings have been produced, using unbalanced magnetron co-sputtering. The composition result shows that through control of the power of individual target, the composition in the film can be maintained. During co-deposition of the Ti and Al, quasi-amorphous film is formed due to kinetically energetic ad-atoms produced after sputtering process. The addition of Al to Ti decreases the crystallinity of the film. After annealing at 800^{o}C temperature the crystalline nature of the film increases.

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