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INTERFACE DIFFUSION IN POLYSYNTHETICALLY-TWINNED TiAl

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ABSTRACT

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The utility of polysynthetically-twinning (PST) TiAl, which contains a high density of parallel, atomically-flat interfaces within a set of identical crystallographic orientations, as a potential model system for a detailed investigation of interface diffusion is explored. Macroscopic PST crystals were grown in an optical float zone furnace. Thin films were cut from oriented crystals and polished with $\langle 112 \rangle$ directions normal to the film. After sputter cleaning, Ag was deposited on one side of the TiAl thin films. Auger spectra were obtained from these films over a wide range of sputter/anneal conditions. The Al and Ti concentrations were analyzed as well as the important impurity elements, S, Ar, C, N and O. Using the present data and existing knowledge of the microstructure and crystallography of PST TiAl, the potential of this material for providing a detailed understanding of the atomistic mechanisms of interface diffusion is analyzed.

INTRODUCTION

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The limited tensile ductility of fully lamellar polycrystalline TiAl alloys [1, 2] obscures evidence that the lamellar interfaces in this alloy are susceptible to environmental embrittlement at room temperature as seen in experiments on polysynthetically twinned (PST) TiAl single crystals [3]. Promising signs have been reported that the segregation of certain ternary additions to the interfaces can mitigate this environmental embrittlement [4], but with a penalty in other properties. There is also growing evidence that under the moderate stress states expected in service, interface sliding will be the rate controlling creep mechanism in TiAl alloys [5, 6]. At the present time, little is known about the interface diffusion or segregation properties of the lamellar interfaces in TiAl alloys, and almost nothing is known about the effects of these properties on the strength and deformation behavior of these lamellar interfaces.

Yet TiAl is a remarkable alloy system in which to develop an understanding of the relationship between interface atomic structure and properties. The lamellar interfaces are fully ordered and atomically flat with high macroscopic symmetry, which enables the imaging of the atomic structure and the mapping of compositional variations by electron microscopy (EM) techniques. The relatively short periods of the interface structures are ideal for computer-based atomistic modeling. Crystal growth techniques have been developed which allow large PST crystals to be grown in which all lamellar interfaces

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are parallel. At the same time, the introduction of field emission TEMs and the development of interatomic potentials that enable the effective treatment of bond covalency provide the experimental and theoretical tools with which to carry out an effective analysis of diffusion in this interesting alloy. In the present paper, the first results are presented of a study that is investigating the relationship between interface atomic structure and interface diffusion in PST TiAl using electron microscopy techniques, Auger electron spectroscopy and atomistic simulation.

EXPERIMENT

Polysynthetically-twinned (PST) TiAl crystals were grown by directional solidification in an optical float zone furnace at Kyoto University. The feed rod and seed of Ti-49.3%Al were first produced by arc melting of Ti and Al using a non-consumable W electrode in a gettered Ar atmosphere. The growth rate of the PST crystals was 5 mm/hr. Using the float zone method, PST crystals with diameters 1 cm ϕ by 10 cm long are produced. The PST crystals were mounted on a goniometer and back Laue x-ray diffraction was used to orient the crystals for processing. The orientation used in the present study was such that the lamellar interfaces and one set of $\langle 112 \rangle$ directions lying on the interfaces were parallel to the x-ray beam. Oblong disks of approximately 250 μm thickness were cut from the oriented crystal by spark cutting. From these disks, diffusion specimens of three mm diameter were cut using a cylindrical slurry cutter (see Figure 1a). The 3 mm disks were mounted in an automatic polishing apparatus and thinned using a dilute diamond paste solution in oil to a final thickness of less than 50 μm .

The thinned disks were washed using acetone and ethanol. The disks were mounted in a sputter deposition system at Osaka University with a 1 mm collimating aperture centered on each disk. The central 1 mm ϕ area of each disk was sputter

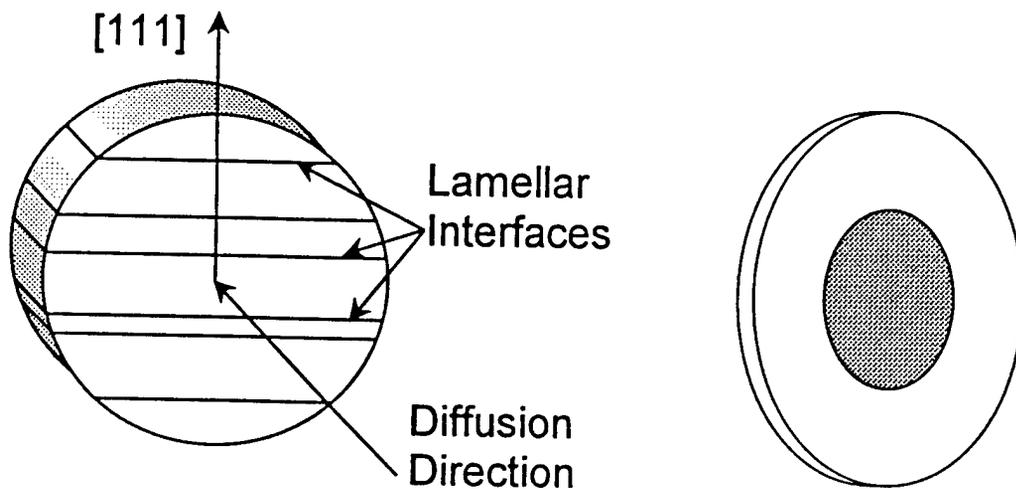


Figure 1 (a) Diffusion specimen prior to thinning showing orientation of the lamellar interfaces with respect to the diffusion direction. (b) The final diffusion specimen after deposition of the diffusing species indicated by the shaded area. The region around this area has not been sputtered and acts as a surface diffusion block.

cleaned using Ar ions and Ag was then sputter deposited on the disks to a thickness of approximately 1 μm (see Figure 1b). The disks were mounted on an in-situ heating holder within an Auger spectroscopy system at the Metals and Ceramics Division of Oak Ridge National Laboratory. The orientation was such that the side of the disks containing Ag, the source side, was mounted away from the electron beam and sputter gun of the Auger system.

Some initial data on Ag diffusion was obtained. However, it was found to be necessary to characterize the sputter and annealing behavior of the TiAl. Therefore, after cleaning some specimens of Ag, sputter/anneal cycles were run at a variety of temperatures. Auger spectra were obtained over an energy range from 50 eV to 1450 eV. This range includes the low energy Al LMM peak, the Ti LMM peaks and the Al KLL peaks. It also includes the important impurity peaks of, in increasing energy, S, Ar, C, N and O.

RESULTS AND DISCUSSION

An initial Auger spectrum was recorded of a specimen at room temperature prior to any processing but after the UHV chamber was baked. The differential spectrum is shown in Figure 2. The peaks due to carbon, titanium, oxygen and aluminum are labeled; the low energy peak due to aluminum is not visible due to the impurity surface layer. This impurity layer, which exists around the deposited diffusant on the visible source surface of Figure 1b provides a barrier to short circuit surface diffusion.

Differential Auger spectra after ten minutes exposure to a 5 keV Ar^+ beam and after a post-sputtering anneal at 600 $^{\circ}\text{C}$ are shown in Figure 3. In the first spectrum, the

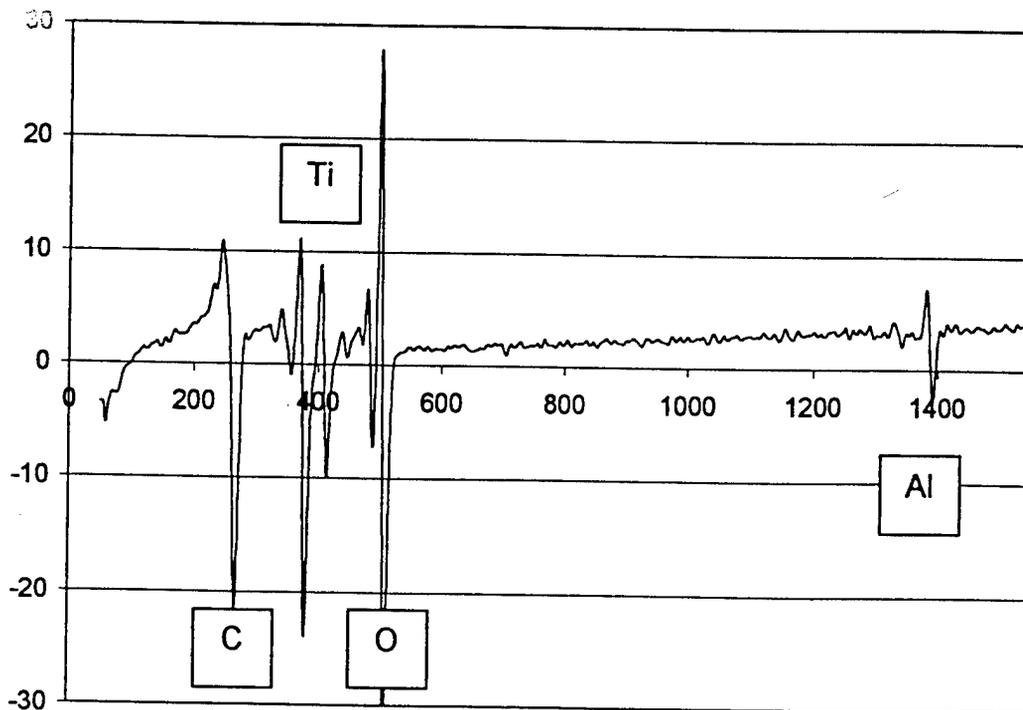


Figure 2 Auger spectrum of the diffusion specimen prior to sputtering or annealing. This spectrum was recorded from the back side of the specimen shown schematically in Figure 1b.

low energy Al peak is now visible and the C and O peaks are reduced in intensity. A small additional peak due to Ar is visible at slightly lower energy than the C peak. Strong preferential sputtering of Al from the surface is indicated by the sharp increase in intensity of the low energy surface Al peak after annealing. The annealing treatment also removed virtually all of the surface C and O as well as the residual Ar from the sputtering treatment.

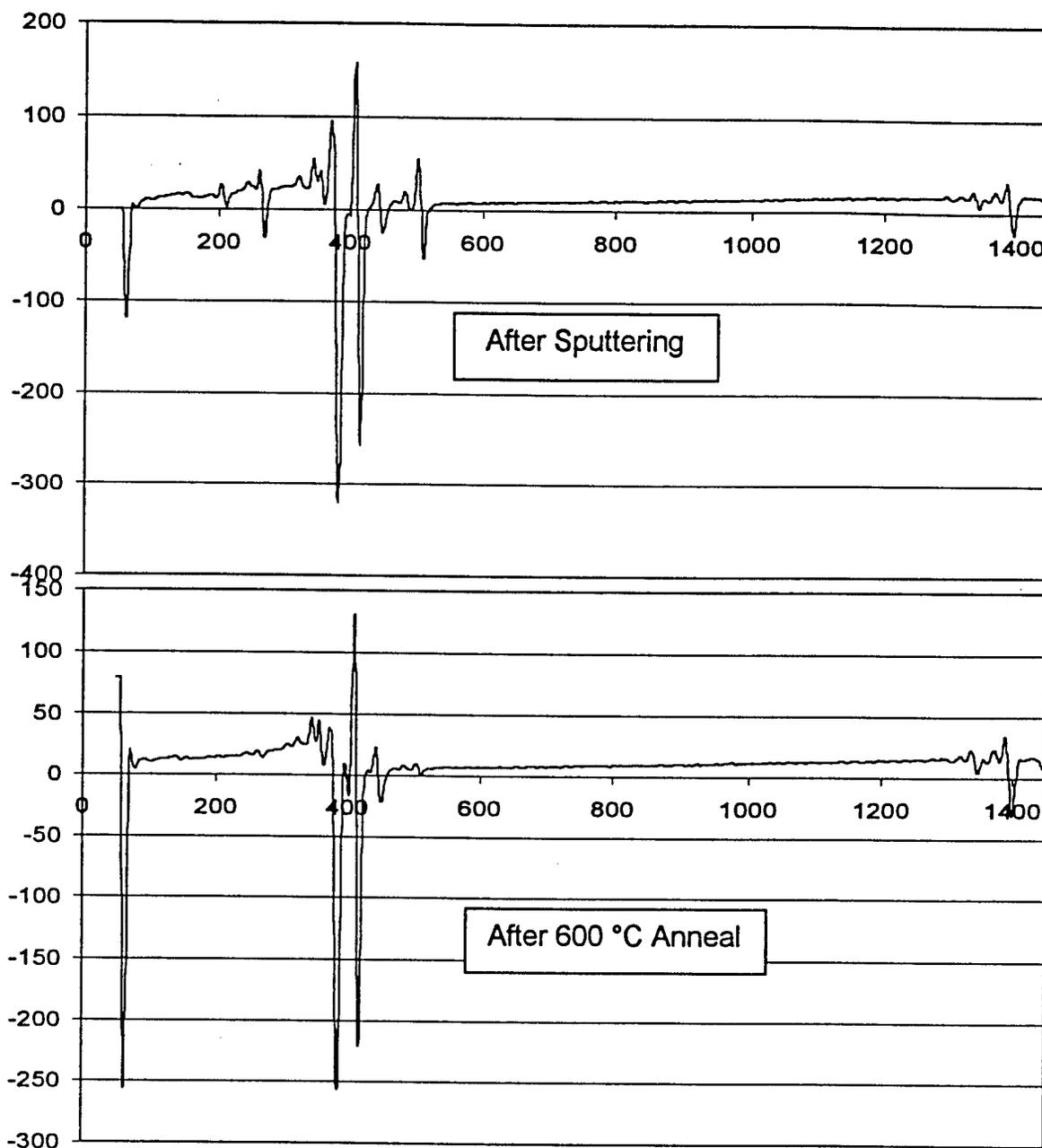


Figure 3 Differential Auger spectra after sputtering (a) and annealing (b).

The compositions of Ti and Al at the surface was measured as a function of annealing temperature after room temperature sputtering. The results are presented in Figure 4. The concentrations were adjusted using sensitivity values for Ti and Al in TiAl which were obtained from an in-situ fracture specimen at room temperature [7].

Sensitivity values for pure Ti and pure Al were found not to provide reasonable alloy composition values. The results show the strong preferential sputtering of Al from the surface under the 5 keV Ar⁺ beam. The data at 200, 400 and 500 °C were recorded after the specimen had been held at the respective temperature for 1 hour. The data at 600 °C was obtained after the specimen reached the temperature, after an additional thirty minutes and after an additional two hours. The data at 700 and 800 °C were obtained upon reaching these temperatures and after 30 minute anneals at 800 °C. When annealing times are considered, the data of Figure 4 is consistent with the diffusion of Al from the bulk to the surface to replace the Al that was sputtered from the surface.

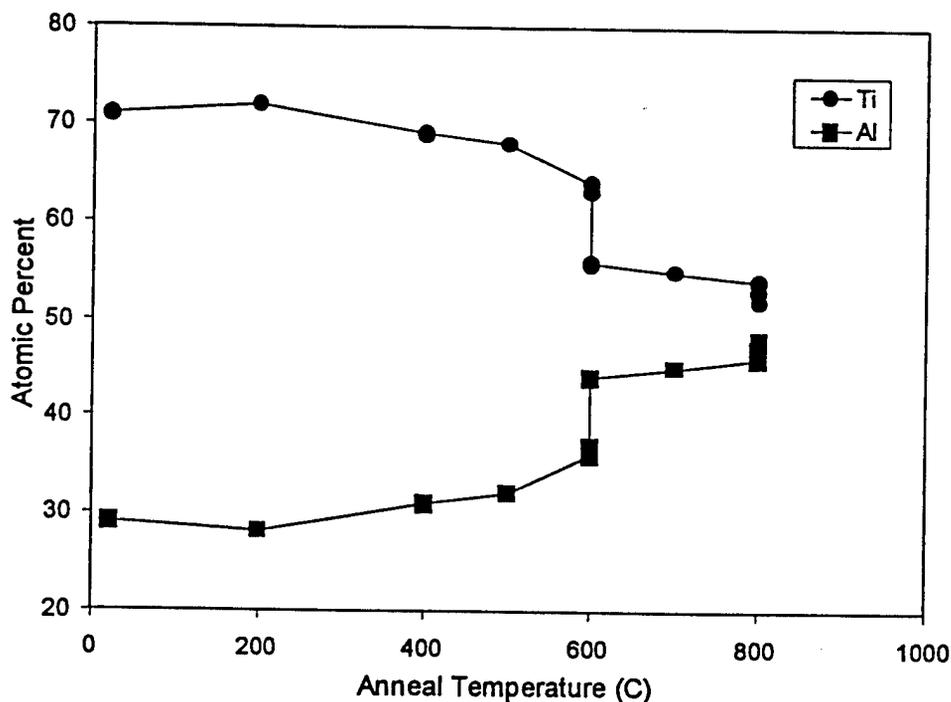


Figure 4 Corrected concentrations of titanium and aluminum on the (112) surface as a function of annealing temperature. Prior to annealing, the specimen was sputtered for 10 m with 5 keV Ar⁺ ions at room temperature. The compositions were corrected using sensitivity data from a virgin in-situ fractured specimen of similar bulk composition.

The preferential loss of aluminum from the surface was greatly reduced when sputtering was done at elevated temperatures. Only a minor difference was found between the differential spectrum recorded after sputtering for ten minutes at 550 °C and that shown in Figure 3b.

The Auger data indicate that the intrinsic sputtering and annealing behavior of the TiAl surface will have to be considered in any interface diffusion analysis that relies upon the measurement of the accumulation of diffusant on the surface. In the temperature range over which interface diffusion is much larger than bulk diffusion, measurements can rely upon a comparison of the diffusant surface peak to the Ti peak as there will be little recovery in the Al surface composition. At higher temperatures, one of two approaches can be used to reduce the effect of the change in surface composition on the apparent surface accumulation of diffusant atoms. A combination of

the Al surface peak and the Ti peak can be used for a reference although it is not clear how reliable this approach can be since these peaks are greatly different in energy and therefore have different surface sensitivities. An alternative approach would use the high energy Al peak as a reference peak to normalize the surface accumulation of diffusant atoms. This peak is much less sensitive to the composition of Al atoms within the surface layer.

CONCLUSION

Polysynthetically twinned TiAl single crystals is an excellent model system for the systematic study of interface atomic structure/diffusion relationships. However, the presence of two elemental bulk species leads to preferential sputtering changing the composition of the surface layer. Therefore, these effects must be included in order to obtain reliable measurements of interface diffusion of solute by the surface accumulation method.

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