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ATOM PROBE FIELD ION MICROSCOPY OF POLYSYNTHETICALLY TWINNED
TITANIUM ALUMINIDE

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Two phase γ -based TiAl alloys are attractive for structural applications at high temperatures because they possess good elevated-temperature mechanical properties, low density, and good creep and oxidation resistance. The microstructures of these alloys consist of plates of the near equiatomic γ phase ($L1_0$ -ordered structure) and the $Ti_3Al \alpha_2$ phase ($D0_{19}$ -ordered structure). It is of great interest to study the details of the lamellar $\alpha_2+\gamma$ microstructure because the interface stability is the key to providing a usable high temperature material.

Polysynthetically twinned (PST) TiAl crystals have been developed in order to systematically study the lamellar microstructure.¹ These PST materials contain no high angle grain boundaries and have a single set of aligned lamellae of α_2 and γ phases, as shown in Fig. 1. Therefore, PST samples facilitate the study of the dependence of mechanical properties on lamellar structure by providing a known, consistent set of aligned lamellae. Previous transmission electron microscopy studies of PST TiAl have shown that Cr and Mo segregation occurs at certain non-coherent γ/γ twin boundaries.² These studies also found a depletion of aluminum at certain γ/γ interfaces, showing " α_2 -like" compositions. However, no evidence was found of even a few unit cells of the $D0_{19}$ -ordered structure at the interface by microdiffraction. This paper investigates the feasibility of applying atom probe field ion microscopy to PST TiAl samples and presents some preliminary characterization results from γ/γ interfaces in PST TiAl.

The materials investigated in this study are a Ti-49.7 at. % Al PST crystal and a near fully lamellar cast Ti-47 at. % Al- 2% Cr-1.8% Nb-0.2% W-0.15% B alloy. Blanks (0.5 x 0.5 x 10 mm) were cut from the PST material with a slow speed diamond saw and then electropolished into suitable field ion specimens by standard solutions. Field ion microscopy was performed with neon as the imaging gas. Energy-compensated atom probe analysis was performed in the presence of 1×10^7 Pa of Ne, with a pulse fraction of 20%, and a pulse repetition rate of 1500 Hz. A specimen temperature of 60K was used for both field ion microscopy and atom probe analysis. Electron microscopy was performed in a Philips CM30 transmission electron microscope operated at 300 kV.

A field ion specimen containing two γ/γ interfaces in the analyzable apex region was prepared from the PST material and is shown in Fig. 2a. The γ/γ interfaces are aligned approximately along the specimen axis of this field ion specimen. This orientation is one of the special orientations that facilitates atom probe analysis. The γ/γ boundaries in the field ion images, Fig. 2b, of TiAl materials are generally evident by both the discontinuity of the atomic terraces (i.e., the concentric rings in the field ion image) across the boundary and by the abrupt change in contrast due to the different orientation of the material on either side of the interface. As oxygen is known to partition strongly to the α_2 phase, the lack of oxygen in an analysis confirms that it is from a γ phase. The γ/γ interface marked T in Fig. 2b, may be a "true twin" (180° rotation) due the quasi-continuous imaging nature of the poles across the boundary. Field evaporation and the orientation of the crystal lattices reveals that the left interface is not a true twin, as seen in Fig. 3. Atom probe analysis from this γ/γ interface indicates a composition of Ti-25.5 at. % Al and the oxygen level was below the detection limit. Therefore, this γ/γ interface also exhibited a depleted " α_2 -like" aluminum level. The field ion image can also be used to investigate the possibility of a thin film of the α_2 phase at the γ/γ interface. In contrast to the thin brightly-imaging α_2 platelet observed between two γ regions in a cast TiAl alloy, shown in Fig. 4, no evidence of an ultrathin α_2 film was observed at these γ/γ interfaces in the field ion images. These observations are in agreement with previous microdiffraction studies and indicate that there was no α_2 phase at the γ/γ interface.^{2,3}

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1. H. Inui et al., *Acta Metall. et Mater.* 40 (1992) 3095.
2. H. Inui et al., *Phil. Mag. A* 74 (1996) 451.
3. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and by an appointment (DJL) to the Oak Ridge National Laboratory (ORNL) Postdoctoral Research Associates Program administered jointly by the Oak Ridge Institute for Science and Education and ORNL. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at ORNL.

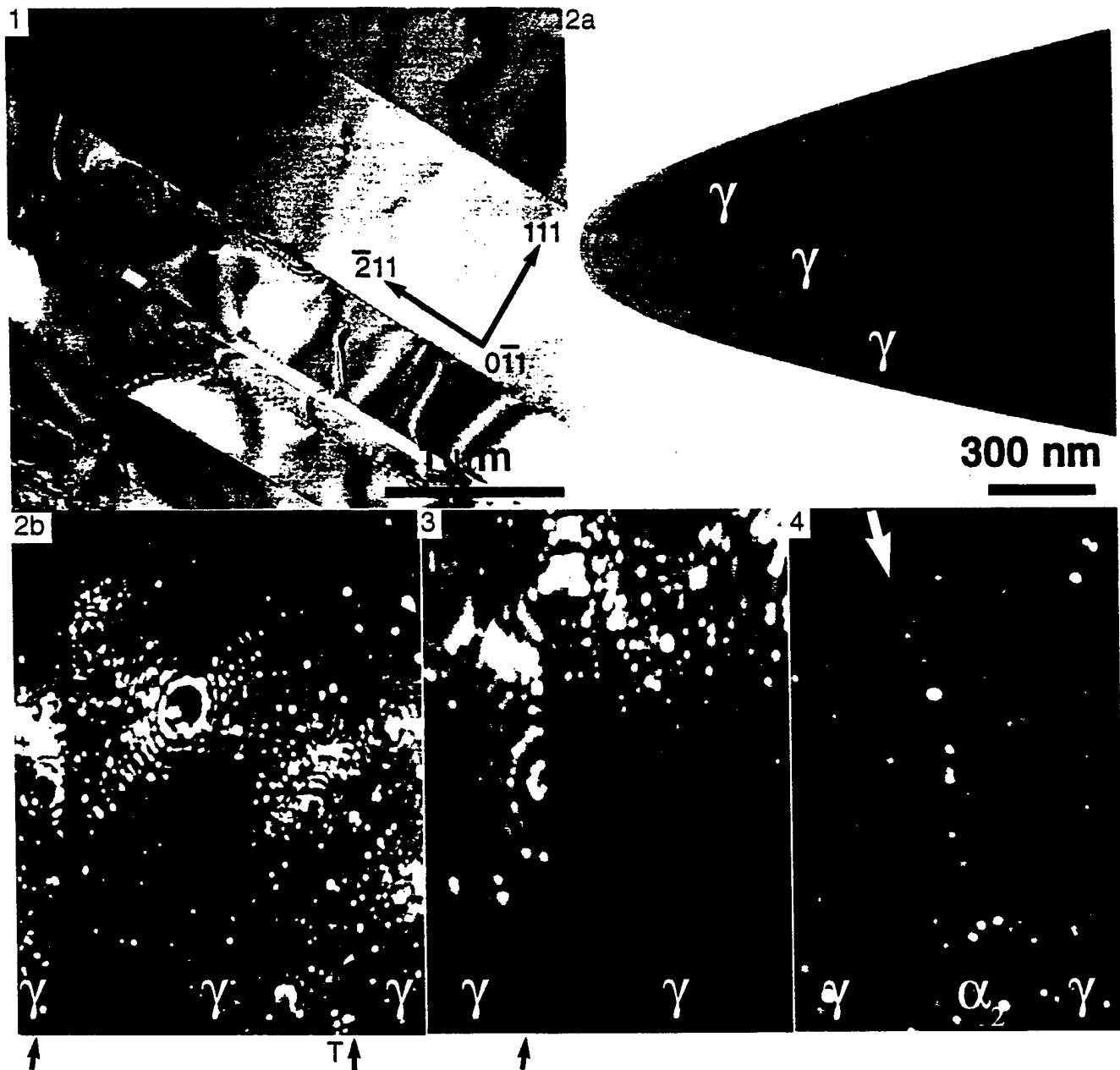


Fig. 1. PST microstructure consists mainly of γ laths of various widths.

Fig. 2. a) TEM image and b) field ion micrograph of PST TiAl showing γ/γ interfaces.

Fig. 3. Higher magnification field ion micrograph of the γ/γ interfaces shown at the left side of Fig. 2 after further field evaporation.

Fig. 4. Field ion micrograph of an ultrathin bright-field imaging α_2 platelet in a cast TiAl alloy.

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