

**In-Situ TEM Observations of Strain-  
Induced Interface Instability in TiAl/Ti<sub>3</sub>Al  
Laminate Composite**

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# IN-SITU TEM OBSERVATIONS OF STRAIN-INDUCED INTERFACE INSTABILITY IN TiAl/Ti<sub>3</sub>Al LAMINATE COMPOSITE

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## Objective/Scope

The stability of interfaces in lamellar TiAl (or TiAl/Ti<sub>3</sub>Al laminate composite) by straining at ambient temperatures has been investigated using in-situ staining techniques performed in a transmission electron microscope in order to obtain direct evidence to support the previously proposed creep mechanisms in refined lamellar TiAl based upon the interface sliding in association with the cooperative motion of interfacial dislocations. It has been reported previously that the mobility of interfacial dislocations can play a crucial role in the creep deformation behavior of refined lamellar TiAl [1,2]. Since the operation of lattice dislocations within refined  $\alpha_2$  and  $\gamma$  lamellae is largely restricted, the motion of interfacial dislocations becomes the major strain carrier for plasticity. Results of ex-situ TEM investigation have revealed the occurrence of interface sliding in low-stress (*LS*) creep regime and deformation twinning in high-stress (*HS*) creep regime. These results have led us to propose that interface sliding associated with a viscous glide of pre-existing interfacial dislocations is the predominant creep mechanism in *LS* regime and interface-activated deformation twinning in  $\gamma$  lamellae is the predominant creep mechanism in *HS* regime. Stress concentration resulted from the pileup of interfacial dislocations is suggested to be the cause for the interface-activated deformation twinning. Accordingly, the creep resistance of refined lamellar TiAl is considered to depend greatly on the cooperative motion of interfacial dislocations, which in turn may solely be controlled and hindered by the interfacial segregation of solute atoms (such as W) or interfacial precipitation. Furthermore, through the in-situ TEM investigation, we also found that the lamellar interfaces could migrate directly through the cooperative motion of interfacial dislocations. That is, the  $\gamma/\gamma$  and  $\gamma/\alpha_2$  interfaces can migrate through interface sliding and lead to the coalescence or shrinkage of constituent lamellae (i.e. microstructural instability), which results in a weakening effect when refined lamellar TiAl is employed for engineering applications. Although it is anticipated that interface sliding and migration are prevalent at elevated temperatures, the present in-situ straining study reveals the instability of lamellar interfaces at ambient temperatures.

## Technical Highlights

Refined lamellar TiAl with a nominal composition of Ti-47Al-2Cr-1Nb-1Ta (at.%) was used for this investigation. Interfacial substructures were examined using JEOL-200CX transmission electron microscope. Dislocation structure and the core structure of interfacial dislocations were also examined using weak-beam dark field (WBDF) and high-resolution (HRTEM) imaging techniques. To investigate strain-induced interface sliding, an in-situ straining experiment was performed at room temperature in a JEOL-200CX transmission electron microscope using a single-tilt straining holder. A gear-drive translation mechanism was activated through the foot pedals with the deformation rate controlled by the speed of y-axis tilt. Plastic deformation took place and was recorded right after a short period of time in which elastic deformation was first distinguished from the motion of bend extinction contours. A hi-8 videocassette recorder attached to a TV rate camera in the microscope was used to record the straining events (e.g., dislocation motion/interaction, interface migration). After completing the experiment, video images of special interest were printed out for analysis.

## Interfacial substructures

There are in general two types of lamellar interfaces within TiAl/Ti<sub>3</sub>Al laminate composite, i.e. (1) the  $\gamma/\alpha_2$  interphase interface which has an orientation relationship:  $(0001)_{\alpha_2} \parallel (111)_{\gamma}$  and  $\langle 11\bar{2}0 \rangle_{\alpha_2} \parallel \langle 1\bar{1}0 \rangle_{\gamma}$ , and (2) the  $\gamma/\gamma$  interfaces which include true twin, pseudo twin, and order-fault interfaces. Types (2) interface is also referred as twin-related interfaces hereafter. A typical TEM observation of lamellar interfaces viewed from the  $[011]_{\gamma}$  edge-on orientation is shown in Fig. 1. Notice that interfacial dislocations (IDs hereafter) can be found in both  $\gamma/\gamma$  and  $\gamma/\alpha_2$  interfaces. Figure 1(b) was imaged with a tilting angle of  $\sim 15^\circ$  from Fig. 1(a). Several dislocation tips (appeared as white dots) in Fig. 1(a), and the corresponding dislocation lines appeared in Fig. 1(b) are marked by arrows. In general, the dislocation density in  $\gamma/\alpha_2$  interphase interfaces is higher than that in  $\gamma/\gamma$  twin-related interfaces as a result of a greater lattice and thermal misfit between  $\gamma$  and  $\alpha_2$  lamellae. The core of each ID in  $\gamma/\alpha_2$  interface contains a small step (ledge) with the step height two layer thickness of the  $(111)_{\gamma}$  plane ( $d_{111} = 0.232$  nm) is shown in Fig. 2(b), which is consistent with the observations reported elsewhere in literature. Accordingly, if an ID moves along the interface, the step moves along with the dislocation and thus, the interface is displaced (advanced) perpendicular to its plane, i.e. the interface will migrate by the distance of a step-height. Therefore, the cooperative motion of IDs will always cause a combination of interface sliding and interface migration as illustrated schematically in Fig. 2(c). Since the Burgers vector ( $b = 1/6\langle \bar{1}\bar{1}2 \rangle$ ) of IDs is parallel to the  $(111)_{\gamma}$  plane, the dislocations need only to glide to cause the interface to slide. Similar properties are also true for  $\gamma/\gamma$  interfaces, and a small step is associated with the core of each ID ( $b = 1/6[\bar{1}\bar{1}2]$ ) in  $\gamma/\gamma$  interface with the step height one layer spacing of the  $(111)_{\gamma}$  plane [Fig. 3(a)]. The IDs in  $\gamma/\gamma_T$  true-twin interface compensate a small angle departure of the  $\gamma/\gamma_T$  interface plane from the exact  $(111)$  twin plane, which is schematically illustrated in Fig. 3 (a). The inclination angle ( $\theta$ ) of the interface plane to the exact twin plane is therefore given by

$$\tan \theta = \frac{h}{s} \quad (1)$$

where  $h$  is the step height, and  $s$  is the average spacing of IDs. Accordingly, as illustrated in Figs. 2(b) and 3(b), it is anticipated that the cooperative motion of interfacial (Shockley partial) dislocations can result in the migration of lamellar interfaces, and thereby lead to the coalescence/shrinkage of the constituent lamellae. The migration rate ( $v_i$ ) of an interface can be expressed as

$$v_i = \rho v_d h \quad (2)$$

where  $\rho$  is the dislocation density,  $v_d$  the dislocation velocity, and  $h$  the step height.

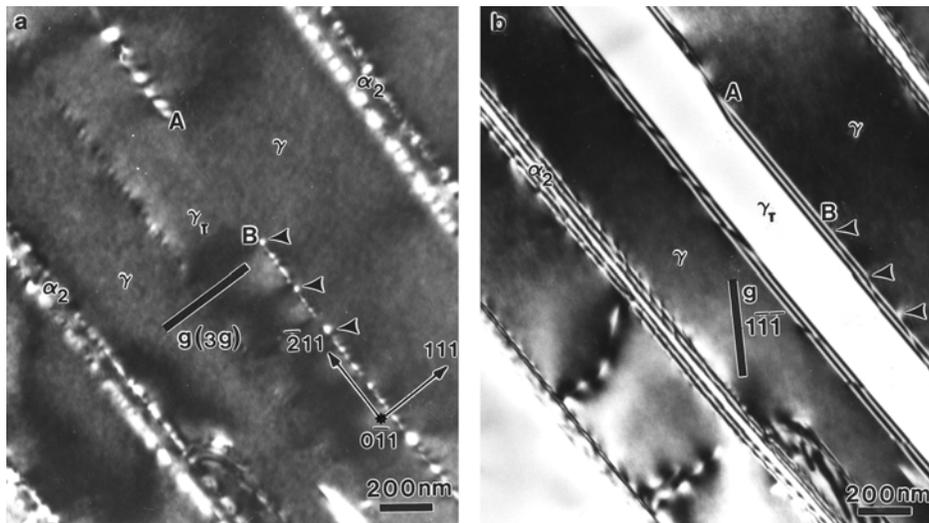


Fig. 1 (a) A WBDF TEM image showing a typical edge-on lamellar structure consisting of  $\gamma$ ,  $\gamma_T$  and  $\alpha_2$  lamellae within an as-fabricated alloy sample,  $Z$  (zone axis) =  $[0\bar{1}1]_\gamma$ . (b) A bright-field TEM image showing the existence of interfacial dislocations in both  $\gamma/\alpha_2$  and  $\gamma/\gamma_T$  interfaces,  $Z = [1\bar{2}1]_\gamma$ .

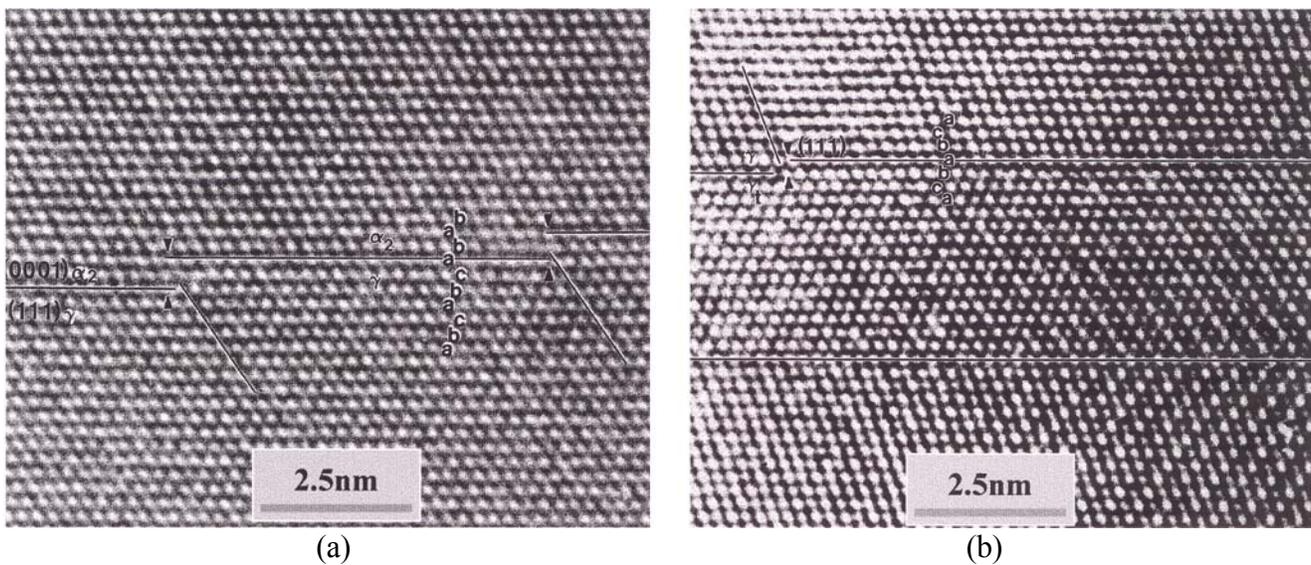


Fig. 2. HRTEM images showing the core structure of interfacial dislocations in (a)  $\gamma/\alpha_2$  and (b)  $\gamma/\gamma_T$  interfaces. The letters **abab** and **abcabc** stand for the stacking sequence of  $\alpha_2$  and  $\gamma$  lamellae, respectively.

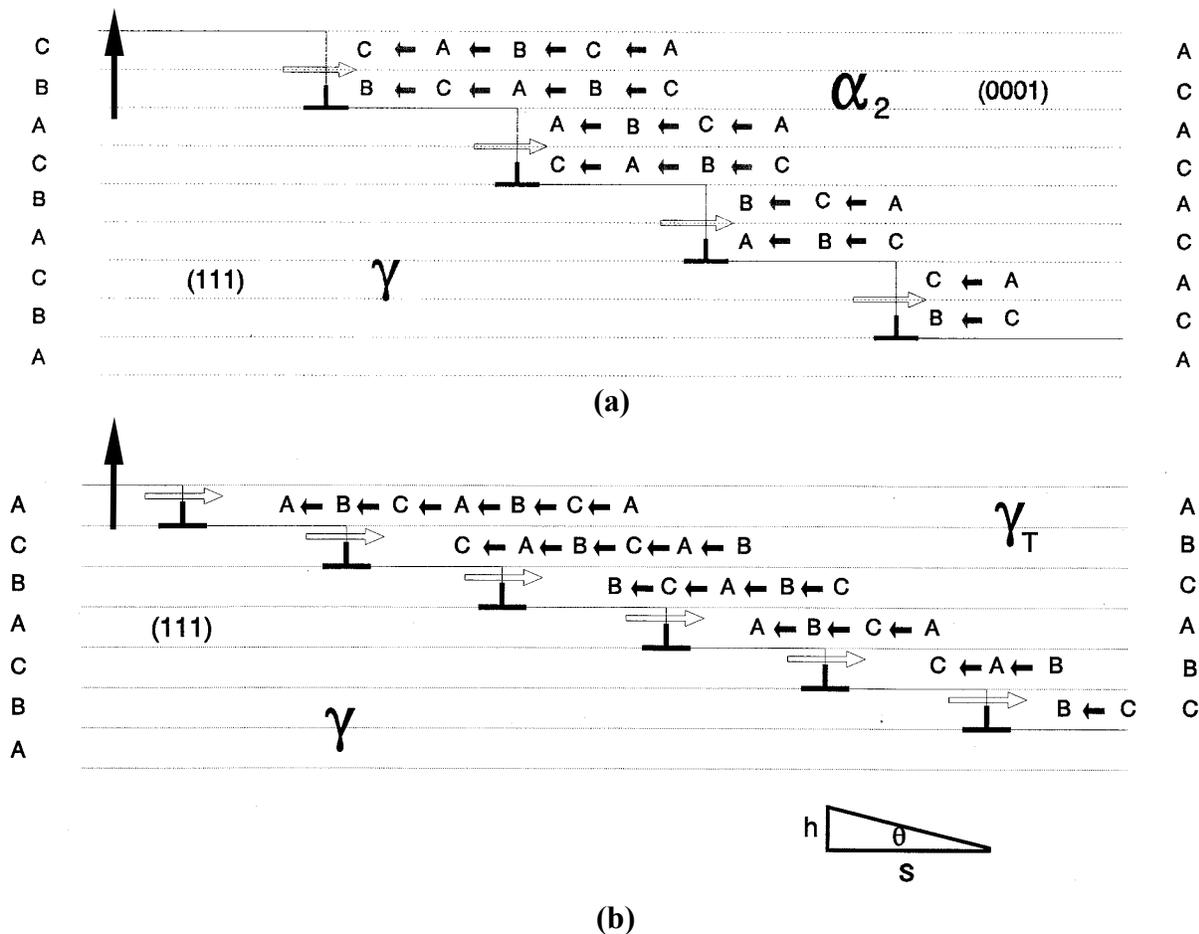


Fig. 3. Schematic illustrations of (a) an array of interfacial (Shockley partial) dislocations forming a glissile  $\alpha_2/\gamma$  interface and (b) an array of interfacial (Shockley partial) dislocations in a glissile  $\gamma/\gamma_T$  interface. Both interfaces can be migrated by the cooperative motion of the interfacial dislocations. The letters ACAC and ABC stand for the stacking sequence of  $\alpha_2$ -lamella and  $\gamma$ -lamella, respectively. The direction of interface migration is indicated by  $\uparrow$ .

### *In-situ observations of interface sliding and migration*

A direct observation of interface sliding and migration has been obtained from an in-situ straining experiment. Throughout the in-situ straining experiment (conducted at room temperature), the motion of IDs was mainly observed in  $\gamma/\gamma$  twin-related interfaces. This indicates that the mobility of IDs in  $\gamma/\alpha_2$  interface is much lower than that of IDs in  $\gamma/\gamma$  twin-related interfaces at room temperature. Typical video images recorded from the in-situ straining experiment for the motion of IDs in a pair of edge-on twin interfaces are demonstrated in Figs. 4 (a) and (b), in which the identical IDs are marked by arrows. It is seen that dislocations in the left interfaces moved cooperatively about 17 nm downward after 11 seconds, while those in the right interfaces moved cooperatively about an equal distance upward. The average dislocation velocity ( $v_d$ ) is estimated to be 1.5 nm/s. With a known dislocation density at the interface ( $\rho \sim 0.033 \text{ nm}^{-1}$ ), the interface migration rate ( $v_i$ ) can be evaluated [according to eqn. (2)] to be 0.012 nm/s, which is so slow that the interface migration is not detectable within a short period of time. An in-situ observation of interface migration is demonstrated in Figs. 5(a) – 5(d), in which the images were taken in a region close to a crack tip. The velocity of dislocation motion in a  $\gamma/\gamma$  twin-related interface was found to be much faster than that shown in Fig. 4 because of a stress concentration caused by the crack. In fact, the dislocation velocity was so fast that it became difficult to track the motion of each dislocation in the dislocation array.

The twin interface initially migrated with a high rate [Figs. 5(a) and 5(b),  $v_i \sim 19$  nm/sec (1580 times faster than that in Fig. 4!)], and after 10 second the migration became much slower [Figs. 5(c) and 5(d),  $v_i \sim 0.16$  nm/sec]. The interface subsequently migrated close to a  $\gamma/\alpha_2$  interface. This rate change is presumably resulted from the interaction of stress field between  $\gamma/\gamma$  and  $\gamma/\alpha_2$  interfaces. Coarsening of the  $\gamma$  lamella occurred as a result of the migration of  $\gamma/\gamma$  interface.

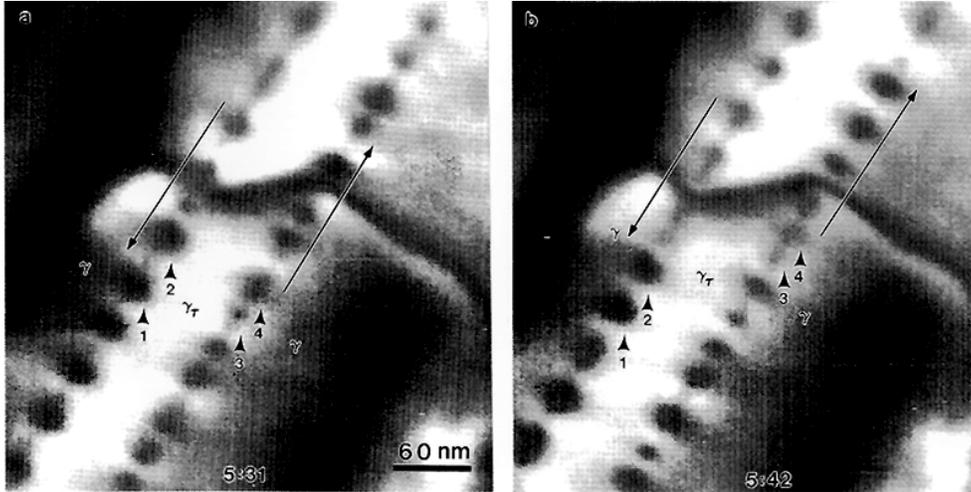


Fig. 4. Two consecutive in-situ video images showing the cooperative motion of interfacial dislocation tips (appeared as black dots) in a pair of  $\gamma/\gamma$  interfaces; the moving directions are labeled by long arrows.

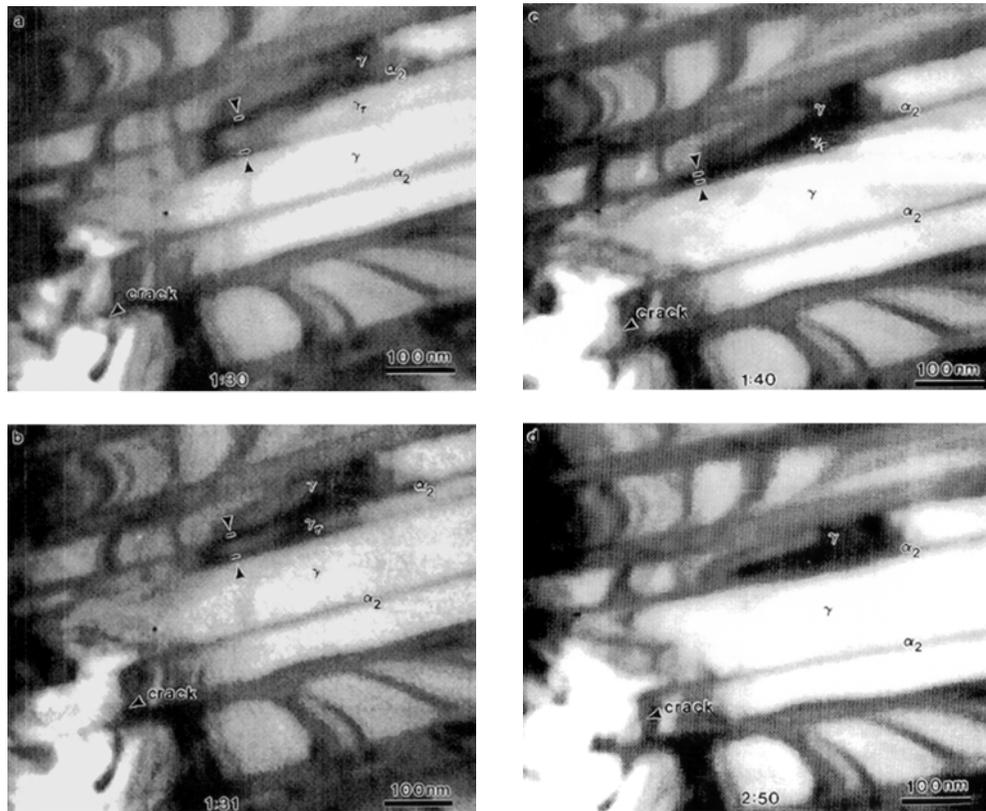


Fig. 5. Consecutive in-situ video images showing the migration of a twin-related interface near a crack tip; (a)  $t = 0$ , (b)  $t = 1$  s, (c)  $t = 10$  s, (d)  $t = 80$  s.

### **Status of FY 2003 Milestones**

Milestone: “Conduct in-situ TEM experiment to record a direct observation of interface sliding” was completed.

Milestone: “Continue to collaborate with ORNL (Dr. C.T. Liu) to fabricate refined TiAl/Ti<sub>3</sub>Al laminate composites from cast Ti-46.5Al-3Nb-1.0W-0.1B and Ti-46.5Al-3Nb-2W-0.1B alloys using hot-extrusion processing techniques” was completed.

Milestone: “Continue TEM characterization and microanalysis to measure the extent of solute segregation at lamellar interfaces” was on schedule and will be completed in June 2003.

Milestone: “Continue characterize the effect of alloying modification on creep resistance of the composite materials” was on schedule and will be completed in September 2003.

### **Travel**

2003 TMS Annual Meeting, San Diego, CA (3/2 – 3/6, 2003).

### **Publications**

L.M. Hsiung, A. J. Schwartz, and T.G. Nieh “In-Situ TEM Observations of Interface Sliding and Migration in a Refined Lamellar TiAl Alloy,” presented in International Symposium on Intermetallic and Advanced Metallic Materials – A Symposium Dedicated to Dr. C. T. Liu, TMS Annual Meeting San Diego, CA, March 3, 2003; to be published in *Intermetallics*.

### **References**

- [1] L. M. Hsiung and T. G. Nieh, *Intermetallics* **7**, 821 (1999).
- [2] L. M. Hsiung, T. G. Nieh, B.W. Choi, and J. Wadsworth, *Mater. Sci. Eng.*, A329-331, (2002), 637.
- [3] M. Yamaguchi and Y. Umakoshi, *Progress in Materials Science*, **34**, 1 (1990).

### AUSPICE

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