



High temperature microsample tensile testing of γ -TiAl

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Abstract

Dislocation activity in two-phase commercial TiAl alloys occurs most readily in the γ -TiAl phase, and measurements of the physical and mechanical properties γ -TiAl are needed to provide a solid foundation for modeling the mechanical performance of these alloys. The recent development of elevated temperature microsample tensile testing is outlined and the application of this technique to elevated temperature tensile testing of γ -TiAl described. Microsample single crystal specimens of Ti–55.5% Al have been tested in tension at temperatures ranging from 400 to 1000 °C. Measurements of the Young's modulus, coefficient of thermal expansion, and flow strength have been made as a function of temperature and crystal orientation. The Young's modulus and coefficient of thermal expansion measurements are compared with the limited data set that is available in the literature. The flow strength data evidences a clear violation of Schmid's law and highlights a significant tension/compression asymmetry, both of which appear to be related to the underlying dislocation mechanisms that govern plasticity in γ -TiAl. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Titanium aluminides have received considerable attention in recent years and are considered to be strong candidates for use as intermediate-temperature materials [1,2]. Commercial alloys of TiAl will most likely have a lamellar two-phase structure, but the present study has focused on single phase γ with the expectation that a fundamental understanding of its mechanical properties will promote the development of the more advanced alloys. To date, the mechanical testing of single crystalline γ -TiAl has been limited by the fact that sizeable single crystals of γ -TiAl are very hard to obtain. Compression testing has been used to measure the compressive strength of TiAl at a variety of crystallographic orientations and temperatures, see for example [3–14]. These studies have reported an anomalous yield strength temperature dependence, similar to what has been observed in Ni₃Al, and a propensity for superdislocation activity. By comparison, the limited

crystal size and room temperature brittleness of TiAl have precluded macro scale tensile testing.

The microsample testing machine designed by Sharpe [15,16] has greatly facilitated the evaluation of room temperature tensile properties for microsamples that have overall dimensions of 1 × 3 mm and are on the order of 200 μ m thick and 200 μ m wide in the gage section. Microsample testing has been used to evaluate a wide variety of materials in recent years, examples include, polysilicon thin films [17,18]; base metal, recast and heat affected zones of steel weldments [19,20]; LIGA Ni and Permalloy [21,22]; negative photo-resist [23]; nanocrystalline metals [24–26]; and electron irradiated 316 stainless steel and Fe–Cu–Mn alloy [27]. Preliminary attempts to use microsample testing to measure the tensile properties of γ -TiAl [28–31] have been very encouraging. The work summarized in this paper describes the efforts undertaken to extend microsample testing to elevated temperatures and the use of elevated temperature microsample testing to make accurate measurements of the Young's modulus (E), the coefficient of thermal expansion (CTE) and tensile flow strength ($\sigma_{0.2\%}$) of γ -TiAl as a function of temperature and crystallographic orientation.

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2. Experimental procedures

Single crystals of Ti–55.5 at.% Al have been grown for this study using the optical float zone furnace at the University of Pennsylvania Laboratory for Research on the Structure of Matter. The 6–10 mm diameter and 50 mm long crystals were heated treated to 1300 °C for 24 h, furnace cooled to 1000 °C, held for an additional 100 h and furnace cooled to room temperature. This heat treatment was performed to remove excess point defects and compositional inhomogeneities. The crystals were oriented using selected area electron diffraction and sliced into 500 μm thick disks with a wire electrode discharge machine (EDM). The disks were oriented along the [100] disk normal containing the [001] and [010] crystal directions, and [110] normal containing the $[-110]$ crystallographic direction. Microsamples were then cut from these disk along the three directions of interest using a sinking EDM and graphite electrode. Before testing, the top and bottom faces of the microsamples were mechanically polished to a mirror finish. Photolithography and vapor deposition were used to place gold lines 250 μm apart on both the top and bottom surfaces. These lines serve as the reflecting gage markers for the non-contact strain/displacement gage that was used to measure strain [32].

The microsample tensile machine and ISDG used in the present study follows the designs of Sharpe et al. [15,16]. Superalloy Rene N5 grips for the microsample machine were machined so that the taper of the microsample fits directly into the matching wedge shape of the grip, which provides a large mechanical and electrical contact surface. These grips are sandwiched between ceramic plates, which electrically and thermally

isolate the grips and microsample from the rest of the test system and allow the microsample to be heated resistively. A programmable power supply is used to control the temperature during each test, and a low DC voltage (~ 2 V) and a current ranging between 2–6 A has been found to be sufficient to heat the microsamples in excess of 1200 °C.

Two independent techniques, thermocouples and an InGaAs optical pyrometer, have been employed to characterize the actual temperatures and temperature gradients of these microsamples during resistive heating. Calibration experiments conducted using both sensors in parallel indicated that the measured values of temperature are in very good agreement (± 5 °C) as long as the specimen thickness is 300 μm or greater, i.e. greater than the 290 μm nominal spot size of the pyrometer. The fact that the microsample is heated locally greatly simplifies design of the test machine and the running of the tests since only the gripping section of the machine needs be high temperature capable. However the presence of the relatively cool grips may be expected to introduce a temperature gradient across the microsample. The temperature profiles of TiAl microsamples heated to set temperatures of 725 and 901 °C are shown in Fig. 1. Close inspection of these curves indicate that the temperature drop across the entire gage section is significant (54 and 72 °C, respectively) but that the change in temperature across the ISDG gage section is much smaller (2 and 6.5 °C, respectively). These results are encouraging because they indicate that homogeneous high temperature microsample tests can be conducted as long as the ISDG is used to measure strain. The microsample tensile tests reported in this paper all employed the ISDG and were

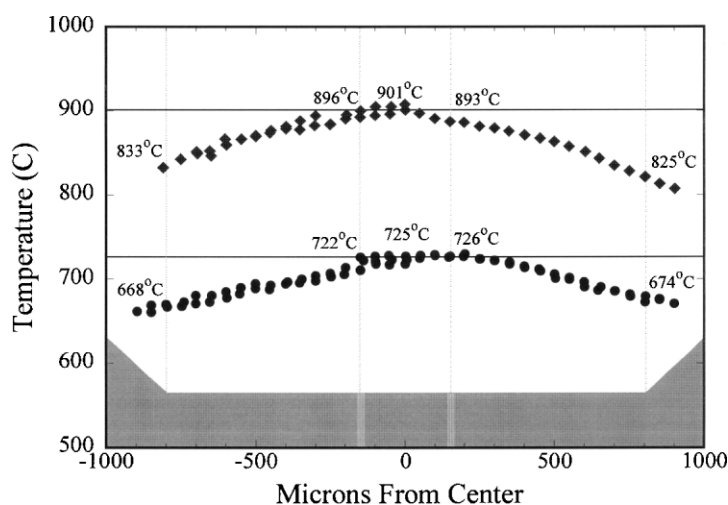


Fig. 1. The temperature drop along the length of resistively heated microsamples, that were heated to 725 and 901 °C. The schematic of the microsample drawn on the bottom of the plot displays the important parts of the sample, the ISDG gage section, the overall uniform gage length of the sample and the gripping section of the sample. The values included on the plot indicate the temperatures at the edge of the ISDG gage section and the uniform gage section. Note that a significant drop was observed across the entire gage section but that the temperature variation across the ISDG gage section was less than 6.5 °C.

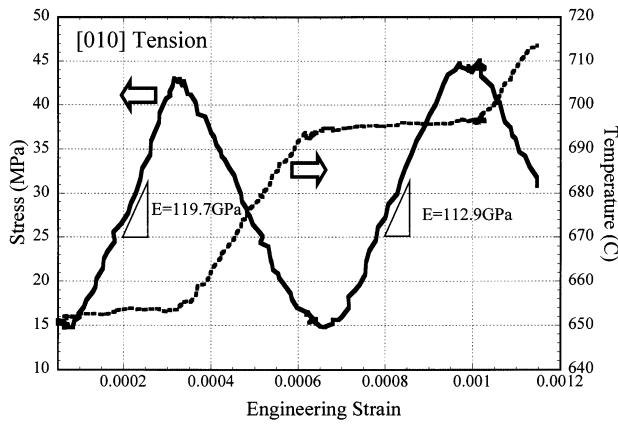


Fig. 2. The elastic loading and unloading of a microsample as its temperature is being raised to the final test temperature. The microsample is elastically loaded at a constant temperature allowing the Young's modulus to be measured as a function of temperature. The sample is then unloaded by stopping the load frame's and heating the sample. The coefficient of thermal expansion of the sample can be determined by monitoring the changes in stress, strain and temperature that occur during this unloading.

conducted by pulling the microsamples beyond the elastic region into the plastic deformation regime at a prescribed temperature and a constant strain rate of approximately 10^{-4} s^{-1} .

Prior to tensile testing, the microsamples were heated to the test temperature in a step-wise manner that allowed for extraction of the Young's modulus and CTE data at a number of temperatures. As is illustrated in Fig. 2, the microsamples were loaded to a stress that remained in the elastic regime, at this point the piezoelectric actuator was fixed and the temperature increased (which led to thermal expansion of the microsamples and a drop in the load), the microsamples were then reloaded while the temperature was held constant, and the whole process was repeated at four or five temperatures for each experiment. Monitoring temperature, stress and strain throughout each test made it possible to extract E and CTE for each temperature at which the microsamples were reloaded. The total strain measured by the ISDG is given by the equation:

$$\varepsilon_{\text{isdg}} = \alpha \Delta T + \frac{\sigma}{E}$$

The two terms in this equation represent the thermal and mechanical components of strain where α is the coefficient of thermal expansion (CTE) and ε , σ , E and T represent the strain, stress, Young's modulus and temperature. If the change in strain is measured with change in stress at constant temperature the first term, or thermal strain term, goes to zero allowing the elastic modulus to be measured directly. Fixing the piezoelectric actuator and increasing the temperature of the sample will result in the following change in strain:

$$\frac{\partial \varepsilon_{\text{isdg}}}{\partial T} = \alpha + T \frac{\partial \alpha}{\partial T} + \frac{1}{E} \frac{\partial \sigma}{\partial T} - \frac{\sigma}{E^2} \frac{\partial E}{\partial T}$$

It is important to note that fixing the piezoelectric actuator does not assure that the change in measured strain will be zero nor that the stress will remain constant. Both of these quantities change with temperature, but α can still be determined because both stress and strain are monitored throughout the test. Rearranging the above equation results in:

$$\alpha = \frac{\partial \varepsilon_{\text{isdg}}}{\partial T} - \frac{1}{E} \frac{\partial \sigma}{\partial T} + \frac{\sigma}{E^2} \frac{\partial E}{\partial T} - T \frac{\partial \sigma}{\partial T}$$

In the above equation the first two terms are measured directly and the last two terms are not used in evaluating α because their values fall several orders of magnitude below the first two terms.

3. Results and discussion

Measurements of the Young's modulus, that were made at a variety of temperatures on a number of independent microsamples, are reported in Fig. 3. This plot shows that at elevated temperatures the modulus is comparable along the [001] and [010] orientations but significantly stiffer in the $[-110]$ direction. All three sets of data were observed to fall off gradually with increasing temperature. The lines in Fig. 3 were taken from the work of He et al. [33] and are based on measurements made using resonant ultrasound spectroscopy. The absolute values and temperature dependence of the modulus measurements that were made in the current study were found to be in very good agreement with the earlier reported values. This observation and comparison serves to corroborate both sets of data

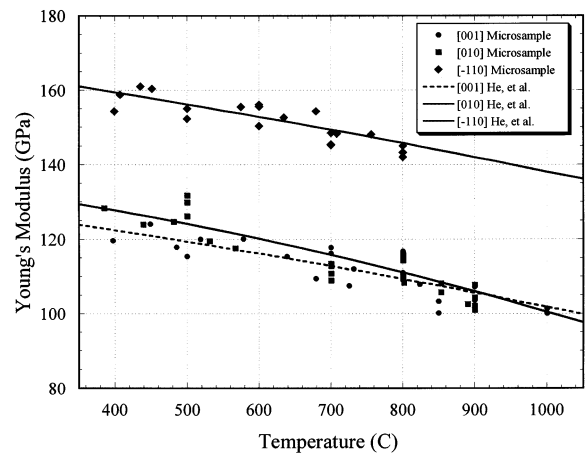


Fig. 3. Microsample measurements of the Young's modulus for γ -Ti-55.5 at.% Al single crystals at three different orientations and as a function of temperature. The lines drawn through the data are based on measurements made using resonant ultrasound spectroscopy by He et al. [33].

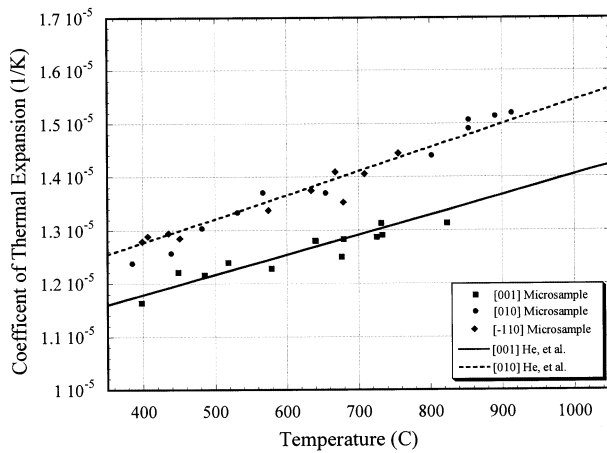


Fig. 4. Microsample measurements of the coefficient of thermal expansion for the [001], [010] and [−110] crystallographic orientations as a function of temperature. The data of He et al. [33] determined by capacitance dilatometer measurements are represented by the lines, and both measurement are in good agreement. CTE measurements for the [−110] orientation fall near those of the [010], which is expected due to the crystal symmetry exhibited by TiAl.

and to provide a reliable model of the temperature dependence of the stiffness matrix of γ -TiAl.

The measured values of the CTE are given in Fig. 4. In general, the CTE data was found to rise with temperature in the range of 400–900 °C. The CTE was measured to be the same for the [010] and [−110] orientations but found to be significantly lower along the [001] crystallographic direction. The difference between the [001] and [010] values are in agreement with the capacitance dilatometer measurements of He et al. [33], compare the data points obtained in the present study with the lines in Fig. 4. Moreover, the finding that the CTE data taken along the [−110] direction mimics the data for the [010] direction can be explained by considering the following transformation:

$$\alpha_{i'j'} = a_{ik} a_{i'l} \alpha_{kl}$$

where for the Cartesian coordinates of the $L1_0$ tetragonal crystal structure $\alpha_{11} = \alpha_{22} \neq \alpha_{33}$ and all other components of $\alpha_{kl} = 0$ [34]. Setting $i' = 1/\sqrt{2}$ [−110] gives:

$$\alpha_{[-110]} = \alpha_{11} = \alpha_{[100]}$$

The stress–strain curves used to measure the yield strength ($\sigma_{0.2\%}$) as a function of temperature and orientation are given in Fig. 5. For each orientation, the yield strength can be seen to increase with temperature to a peak temperature, after which it falls precipitously with further increases in temperature. The magnitude of the peak in the yield strength and the temperature at which this peak occurs has been found to be orientation dependent. This orientation dependence cannot be explained in terms of Schmid's law. Comparing this tensile data with the compressive data available in the literature [12] unveils a tension/compression asymmetry

that is orientation dependent. For the [010] and [−110] tensile axes the tensile strength of the crystals appears to be greater than the compressive strength, but for the [001] orientation the tensile strength was measured to be significantly less than the compressive strength. A model that is based on the asymmetric dissociation and

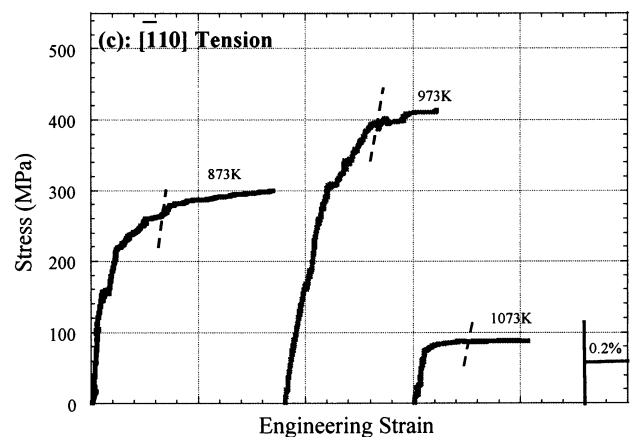
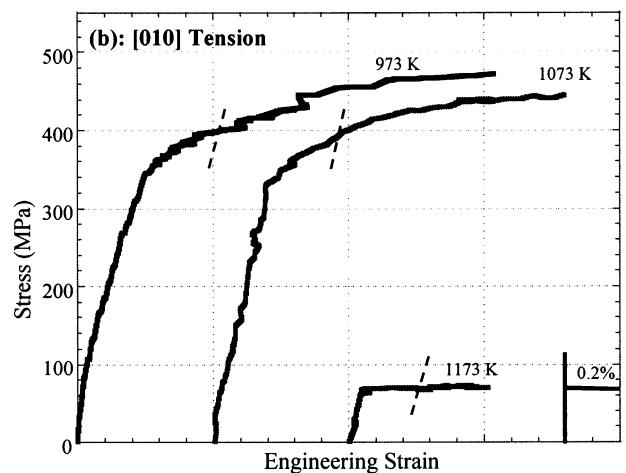
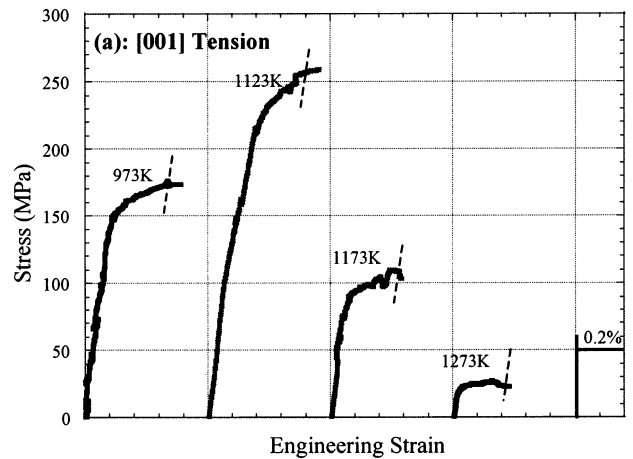


Fig. 5. Microsample tensile tests performed on single crystalline γ -Ti–55.5 at.% Al near the [001], [010] and [−110] crystallographic orientations for a variety of temperatures.

motion of $L1_0$ superdislocations has been shown to be consistent with the measured tension compression asymmetry [32], but further work is needed to clarify the role of superdislocation core structures in determining the macroscopic strength of γ -TiAl.

4. Summary

The recent developments in the area of high temperature microsample tensile testing have been outlined and used to characterize the elevated temperature physical and mechanical properties of γ -TiAl. Young's modulus and CTE data have been collected as a function of temperature and crystallographic orientation and shown to be in very good agreement with the earlier measurements of He et al. [33]. The high temperature stress–strain curves that were reported in this study have been used to measure the 0.2% yield strength as a function of orientation and temperature. Evidence of a Schmid's law violation and the existence of a tension/compression asymmetry have been highlighted.

Acknowledgements

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