Development of High-temperature Microsample Testing

by M. Zupan, M. J. Hayden, C. J. Boehlert and K. J. Hemker

ABSTRACT—Microsample tensile testing has been established as a means of evaluating the room temperature mechanical properties of specimens with gage sections that are tens to hundreds of microns thick and several hundred microns wide. The desire to characterize the mechanical response of materials at elevated temperatures has motivated the development of high-temperature microsample testing that is reported here. The design of specially insulated grips allows the microsamples to be resistively heated using approximately 2 V DC and currents ranging between 2 to 6 A. An optical pyrometer with nominal spot size of 290 μm and 12 μm diameter type K thermocouples was employed to measure and verify the temperature of the microsamples. The ability of the pyrometer to accurately measure temperature on microsamples of different thicknesses and with slightly different emissivities was established over a temperature range from 400°C to 1100°C. The temperature gradient along the length and thickness of the microsample was measured, and the temperature difference measured in the gage section used for strain measurements was found to be less than 6.5°C. Examples of elevated temperature tensile and creep tests are presented.

KEY WORDS—Microsample, high temperature, tensile testing, TiAl

Introduction

The microsample testing machine originally designed by Sharpe[1–3] has greatly facilitated the evaluation of room temperature tensile properties of microsamples with 25 to 500 μm thick and 200 to 300 μm wide nominal gage sections. The microsample test machine consists of the basic components of a typical load frame but has been scaled down to accommodate the small specimen size. A schematic diagram of the microsample testing system used in this study is shown in Figure 1. A piezoelectric drive is used to apply the load to the sample through a linear air bearing. The drive has a total travel of 180 μm and load capability of 225 N. Loads are measured using a miniature load cell with an overall load capacity of 90 N and a resolution of 0.02 N. Strain is measured directly in the gage section of the microsamples via a noncontact laser-based interferometric strain displacement gage (ISDG).[1,2] This optical technique allows us to measure strain in the microsample gage section without affecting the material's mechanical properties.

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The microsample testing technique has been used to evaluate a variety of materials since its inception, including but not limited to polysilicon thin films;[4,5] base metal, recast and heat-affected zones of steel weldments;[6,7] LIGA (Lithography, Galvanoforming and Abformung) nickel, copper and permalloy;[8,9] negative photoresist;[10] single-crystalline TiAl;[11,12] nanocrystalline metals;[13–15] and electron-irradiated 316 stainless steel and Fe-Cu-Mn alloys.[16] Although successful in testing a broad range of materials, to date microsample testing has been limited to ambient temperature tests. The current work was motivated by the desire to characterize the mechanical properties of materials at elevated temperatures.

The addition of high-temperature testing capability allows microsamples to be mechanically deformed in a variety of loading excursions and through a variety of temperature regimes. The initial successful experiments have been in tension and tension-creep, and future work will target elevated temperature fatigue and thermomechanical fatigue. This paper describes the development of high-temperature microsample testing, as well as its application to the testing of the intermetallic alloy TiAl and 316 stainless steel. Emphasis is placed on the use of a resistive heating system and our ability to accurately monitor temperature at different locations along the microsample. This work is intended to provide a detailed description of the development of the equipment and the techniques required to perform successful high-temperature experiments.

Experimental Procedures

Several novel experimental techniques have been employed to bring high-temperature microsample testing to fruition. Resistive heating of the microsamples was used in place of a traditional heating furnace for several reasons. The small size of the microsample machine and geometry restrictions imposed by the laser of the ISDG strain system make it difficult to surround the microsample with a conventional furnace. Because the microsamples are locally heated, only the gripping section of the load frame needs to be high-temperature capable. System components such as the air bearing, which is used for frictionless loading, and the load cell remain at room temperature, thus alleviating the need for water-cooling jackets on such parts. Finally, by stepping the current used to resistively heat the specimen, the microsample's temperature can be changed rapidly, permitting temperature change experiments to be executed.

The grips of the microsample machine were designed so that the taper of the microsample fits directly into the matching wedge shape of the grip, providing a large mechanical and electrical contact surface (see Fig. 2). The grips of

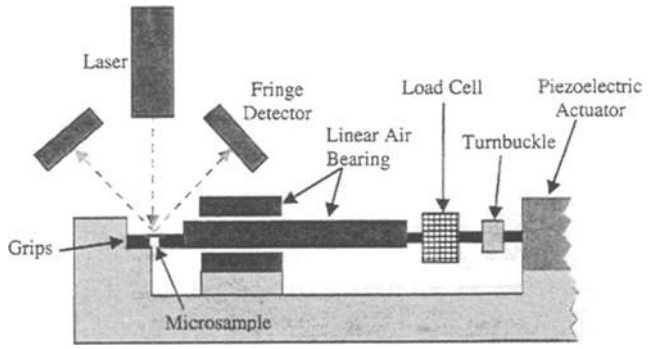

Fig. 1—A plan view schematic of the high-temperature microsample test frame

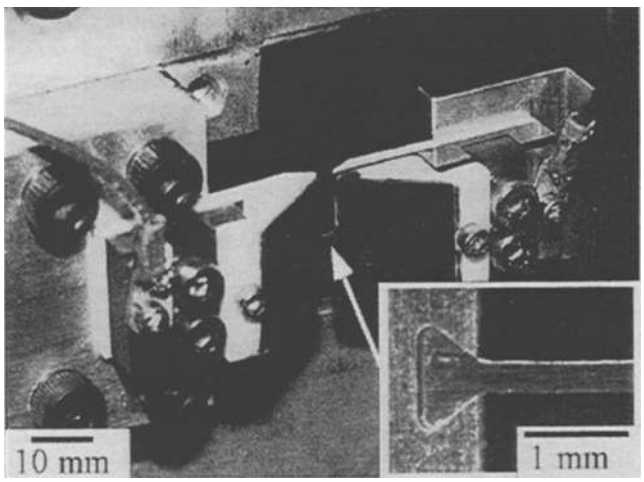

Fig. 2—An enlarged view of the grip section of the microsample test machine. The inset shows the large tapered loading surface that ensures good mechanical and electrical contact between the microsample and the grips

the machine were manufactured out of the nickel-base superalloy Rene N5 with special locations for attaching the electrical leads for passing current through the microsample. These superalloy grips were sandwiched between ceramic plates that thermally and electrically isolate the grips and microsample from the rest of the test system. This allowed the microsample to be heated locally, without heat or electricity conducting into the rest of the system.

The inset of Fig. 2 shows the large contact area between the microsample and the grips. To heat the sample, a small amount of preload was applied to establish good electrical contact. Once electrical contact was established, a programmable power supply with a full feedback loop was used to control the current delivered to the microsample. The voltage was allowed to fluctuate with changing resistance caused by varying microsample thermal and electrical contact due to loading conditions. Early experiments using AC voltage resulted in arcing within the small air gap between the two grips and from the grips to the microsample. For this reason, DC voltage was used to heat the microsamples. Because the cross-sectional area in the gage of the microsamples was substantially smaller than the gripping section, a low DC voltage ($\sim$ 2 V) and a current ranging between 2 to 6 A was found to sufficiently heat the microsamples in excess of 1500°C.

Although resistive heating makes it easy to achieve high microsample temperatures, this technique requires that the temperature gradients of these microsamples be accurately measured. In the current study, thermocouples and an optical pyrometer were used to measure the temperatures of the microsamples. The laser-based ISDG[2,3] requires that the surface of the microsample be clean and polished to a mirror finish. Mechanical tests cannot be performed with thermocouples attached to the microsamples. Moreover, the welding of thermocouple wires to the gage of a microsample is likely to alter the specimen's internal structure. There is always concern in microsample testing that any measurement device that contacts the microsample may affect the measurements. For these reasons, microsamples with attached thermocouples were used to calibrate an optical pyrometer, but the pyrometer was used to measure temperature during all tensile and creep experiments.

A specially designed miniature spot welder was constructed for attaching type K thermocouples to the gage sections of microsamples. This device combines a Unitek dual-pulse 125DP stored energy power supply with an optical microscope. The optical microscope and a spring-loaded electrode allow for precise placement of the thermocouple wires. The Unitek power supply is ideally suited for welding the thermocouples, supplying energy pulses with a rise time of 0.65 ms and pulse width of 2.3 ms. These power conditions produce a sound weld with minimal alteration of the underlying microsample. With this apparatus, type K thermocouple wires (12 μm in diameter) can be routinely placed on the gage section of microsamples (e.g., see the stainless steel microsample with five thermocouples in Fig. 3).

Because the thermocouple is attached both mechanically and electrically to the microsample when the heating power is applied, the voltage reading from the thermocouple is the sum of a fraction of the voltage applied to the microsample and the voltage produced by the Seebeck effect. As illustrated schematically in Fig. 4, when the heating power is applied, the thermocouple temperature will be shifted to a higher or lower value depending on the bias of the applied voltage. It is necessary to separate these two voltages if the true microsample temperature is to be measured, but exact positioning of the thermocouple wires on the microsamples is difficult to control and the precise potential across the thermocouple is impossible to predict. Moreover, the voltage applied to the microsample for heating is noisy and several orders of magnitude larger than the voltage produced by the thermocouple due to temperature. For these reasons, the applied voltage must be removed in order to measure the voltage produced by the Seebeck effect and the true microsample temperature. Rapidly removing the heating voltage and then reapplying it

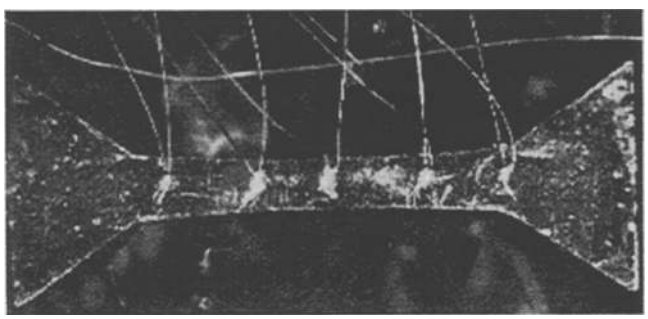

Fig. 3—A stainless steel microsample with five type-K thermocouples placed across its gage section. Thermocouple wires are 12 μm in diameter

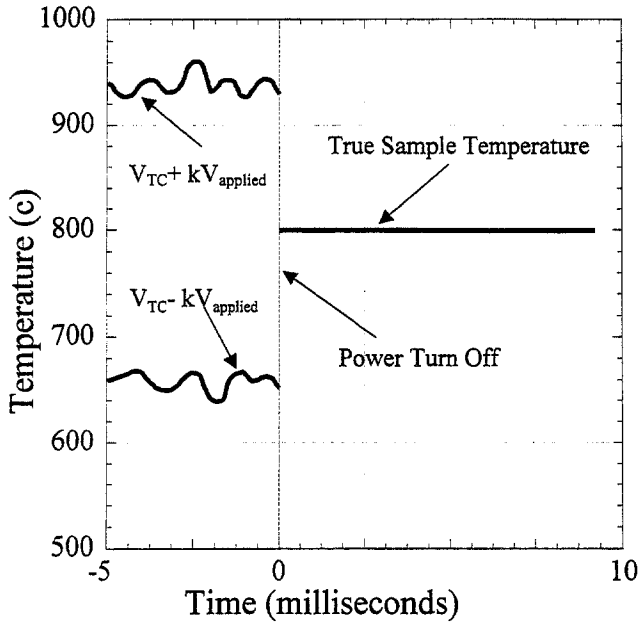

Fig. 4—A schematic graph of the effect of the heating voltage on the thermocouple temperature measurements

within 0.01 seconds allows the microsample temperature to be measured before a significant amount of cooling occurs.

Three different circuits were built to remove the applied voltage by cycling the heating power off and back on. A Kepco model number ABC 10-10DM programmable power supply was programmed to cycle off and back on after 0.01 seconds, but the experimental performance was largely unsatisfactory. The internal control on the power supply caused a very slow decay in voltage and significant overshooting of the set point [see Fig. 5(a)]. Inspection of Fig. 5(a) shows that nearly 0.05 seconds is needed for the power supply to reach an output voltage of zero. Figure 5(b) displays the results produced with a mechanical switch, which provides no overshoot but displays a long off-cycle time that was still experimentally unacceptable. An electromechanical relay was also employed and found to produce an off-cycle time of approximately 5×10^{-5} seconds with no overshoot [see Fig. 5(c)]. The superiority of the electromechanical relay in producing a rapid controlled off-cycle can be seen by comparing Figs. 5(a) through 5(c); note that the sensitivity of the time scale is increased by an order of magnitude between each figure. The electromechanical relay was employed in this study and found to be suitable for removing the applied voltage rapidly enough to allow the temperature of the microsamples to be read before any appreciable amount of cooling occurred.

The thermocouple readings were sampled into a data acquisition system with a built-in cold junction sensor and linearized. All voltage data were converted to temperature using the National Institute of Standards and Technology Monograph No. 175 polynomials[17] for the appropriate temperature ranges. Thermocouple readings were sampled at 50,000 samples per second, allowing for the heating voltage turn-off and microsample temperature to be fully acquired before the specimen was significantly cooled.

An InGaAs pyrometer with a spectral response of 1.6 μm and a nominal spot size of 290 μm was also used to measure temperature on the top or side surfaces of the microsamples. The pyrometer is accurate to $\pm\{0.3\%T_{measured} + 1°C\}$ and operates over a temperature range of 350°C to 2000°C. The pyrometer reading is unaffected by the applied voltage, and when the heating voltage is removed, as described earlier, the temperature measurement of the thermocouple should converge to the pyrometer's measurement if proper calibration has been achieved.

In the current study, microsamples with thermocouples attached to the gage section were used to ensure the proper calibration of the pyrometer. TiAl microsamples with thicknesses of 385 μm and 500 μm and stainless steel microsamples 238 μm and 318 μm thick were used to explore the role of microsample geometry on temperature readings (e.g., to determine the minimum sample size and the amount of background radiation for which the pyrometer measurement is still valid). The use of these microsamples in the as-polished and oxidized conditions also allowed us to test optical pyrometer's ability to properly read temperature on materials with slightly different emissivities. Profiles of temperature gradient along the length and across the thickness of the microsample also were determined to ensure the microsample was evenly heated during testing.

Experimental Results and Discussion

Figure 6 shows the results from a typical heating power-off/power-on cycle. Prior to power-off, the optical pyrometer is able to measure the microsample temperature unaffected while the thermocouple's reading is skewed by some fraction of the applied voltage. When the heating power is removed, time = 0, and the thermocouple temperature converges to that of the pyrometer. Inspection of Fig. 6 shows that for the time frame of interest, there is only a small decay in the thermocouple temperature, which confirms that the heating power is cycled rapidly enough that cooling of the microsample is insubstantial. In comparison, the temperature reading of the pyrometer is not affected, since its output is a running average over 0.2 seconds.

The previously described heating power-off/power-on cycle was used to calibrate the pyrometer over a temperature range from 400°C to 1100°C. A plot of thermocouple temperature versus pyrometer temperature is displayed in Fig. 7 for the 318 μm and 500 μm thick test microsamples. The line drawn on the plot represents a 1:1 correspondence between the two temperature measurements. Inspection of the plot shows that there is good agreement between the two measurement techniques over the entire temperature range for both microsample thicknesses. Accurate measurement of microsample temperature on the 318 μm microsample is encouraging because this thickness is near the pyrometer's nominal spot size of 290 μm. The 318 μm microsample was held at 760°C for 72 hours and then retested. A thin oxide coating was formed under these conditions, but it is encouraging to note that the formation of this oxide did not have an appreciable effect on the temperature measured with the pyrometer, and measurements taken with the pyrometer after the 72-hour oxidation treatment are in good agreement with the thermocouple. For the 30 experiments conducted, the average difference in pyrometer and thermocouple measurements was found to be 4.71°C.

A 238 μm microsample was used to explore the effect of microsample size on the temperature reading. This microsample was known to be smaller than the pyrometer's nominal spot size, and results of this test are shown in

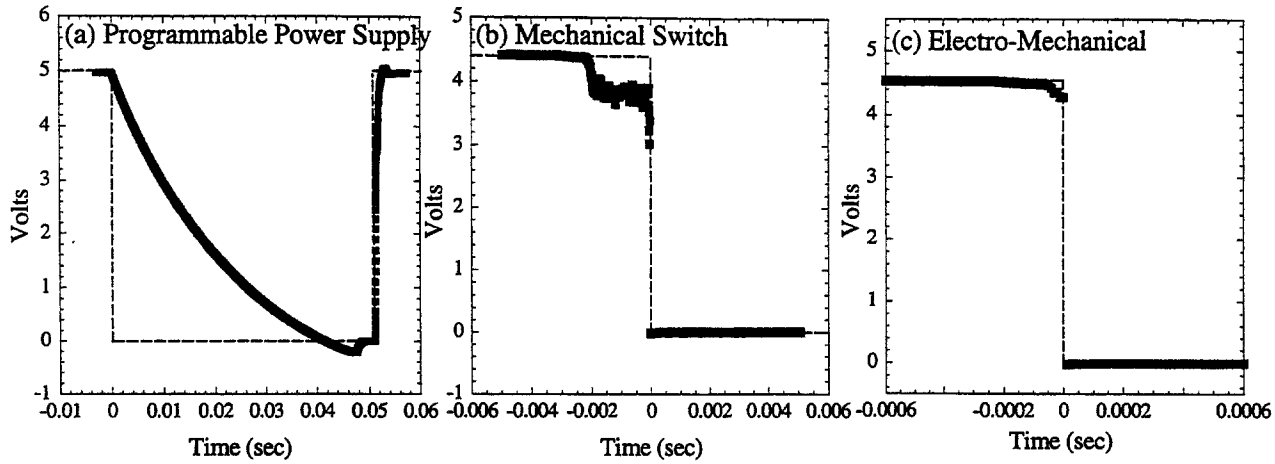

Fig. 5—Plots displaying the off-cycle response of the heating power: (a) turning the power off with the programmable controller produced a slow voltage decay and a large overshoot, (b) a mechanical switch gave little or no overshoot but a large delay in off-cycle time, (c) an electromechanical relay yielded an off-cycle time of $\sim 5 \times 10^{-5}$ seconds with no overshoot

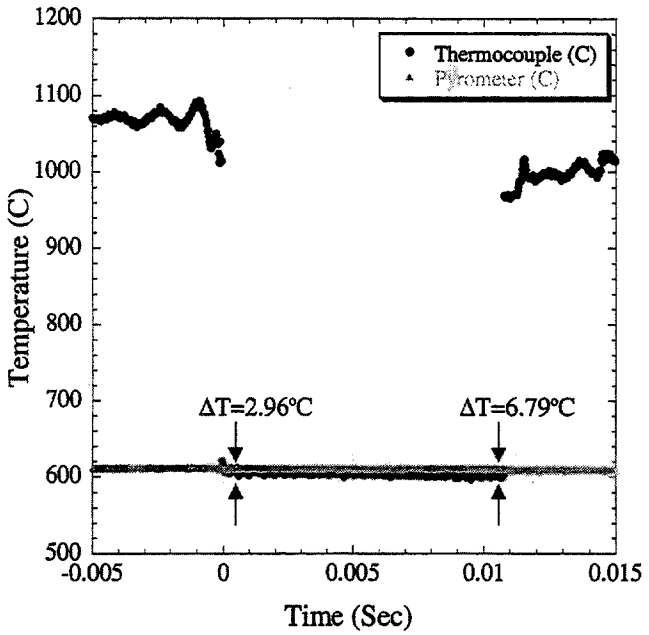

Fig. 6—Comparison of thermocouple output and optical pyrometer measurements during an on-cycle/off-cycle/on-cycle excursion

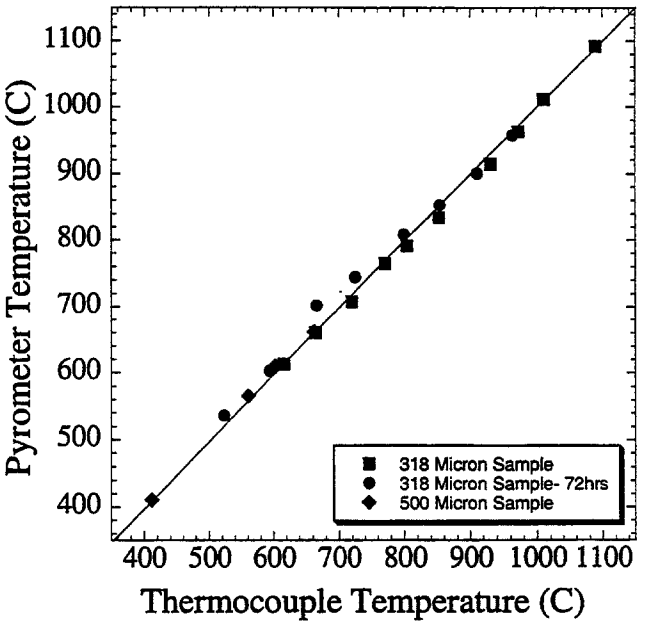

Fig. 7—A comparison of temperatures measured using thermocouples and the optical pyrometer for 318 μm thick stainless steel and 500 μm thick TiAl microsamples. The two methods were found to be in good agreement, within 5°C

Fig. 8. Inspection of the graph shows that the pyrometer measurement undershoots the actual microsample temperature and that the error increases with temperature, presumably because it is averaging the background temperature into the measurement. This test illustrates the need to keep the microsample thickness larger than the spot size of the pyrometer.

Once the pyrometer's ability to accurately measure the microsample temperature was established, it was used to map the temperature profile of the microsamples at various temperatures. Figure 9 shows a longitudinal temperature profile of a 385 μm thick microsample at two different set points, 725°C and 901°C. The origin of the horizontal axis represents the center of the microsample. The figure contains several points of interest, namely, the ISDG length, the overall uniform gage section and the shoulders of the microsample. Inspection of these points shows that for set points of 725°C and 901°C, the temperature drop across the entire uniform gage length is 54°C and 72°C, respectively. These relatively large gradients are undoubtedly due to the fact that the grips at the ends of the microsample serve as enormous heat sinks. It is, however, encouraging to note that the temperature drop in the ISDG is only 2°C at 725°C and 6.5°C at 901°C. This small temperature difference makes it possible to use resistive heating for high-temperature microsample testing if strain is measured using the ISDG.

Figure 10 shows a temperature profile across the thickness of a microsample. The thickness of the microsample is 318 μm and the nominal spot size for the pyrometer is 290 μm. Background radiation might be expected to influence the pyrometer readings if the center of the pyrometer spot is moved more than 14 μm from the center of the microsample. The data points in Fig. 10 are plotted as a function of

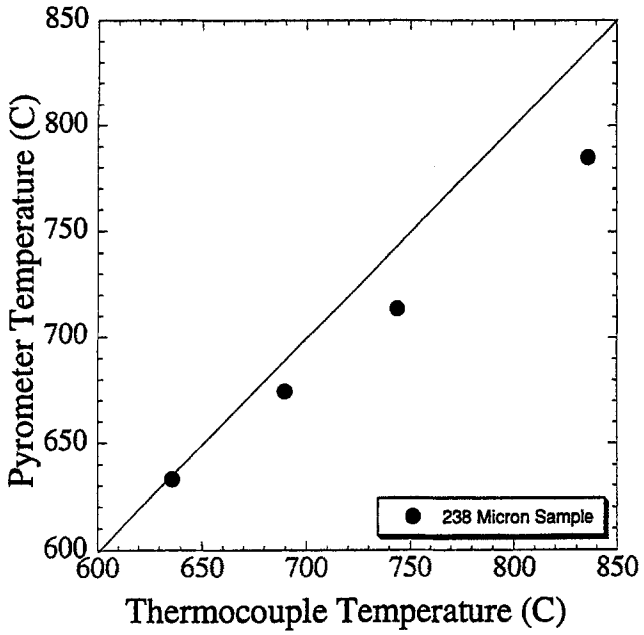

Fig. 8—Temperature measured with a thermocouple plotted as a function of the measured pyrometer temperature for a 238 μm stainless steel specimen

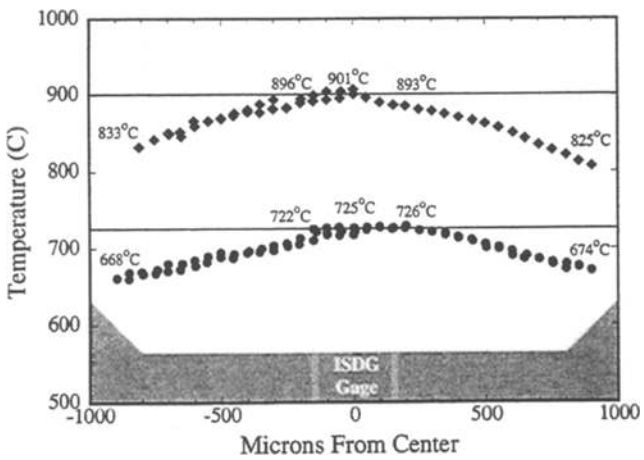

Fig. 9—Temperature drop along the length of resistively heated microsamples that have been heated to 725°C and 901°C. A schematic of a microsample is drawn at the bottom of the plot

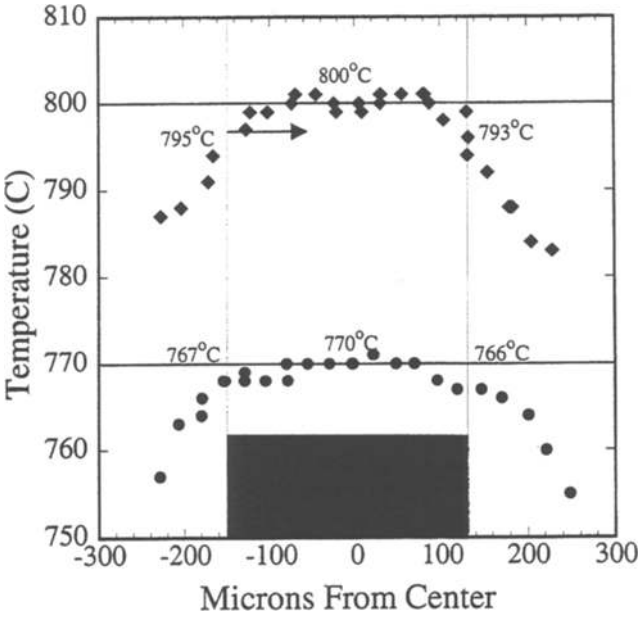

Fig. 10—Temperature profile across the thickness of a microsample heated to two different temperatures

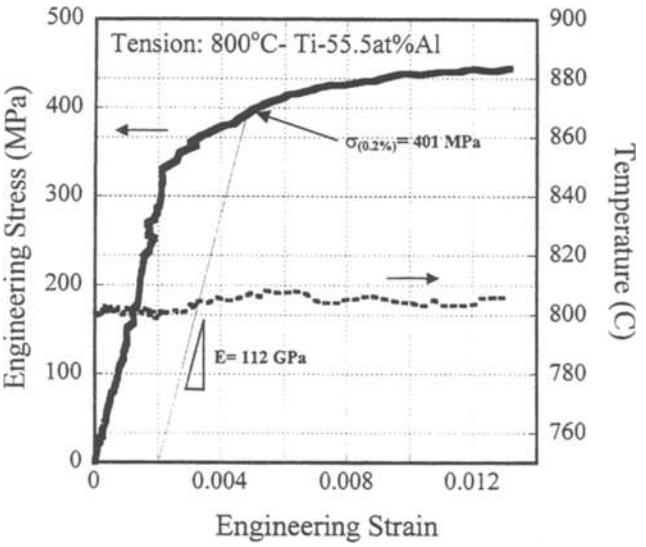

Fig. 11—A typical tensile stress versus strain curve for TiAl at 800°C. The linear elastic region of the curve was used to measure Young's modulus at 112 GPa, and the flow strength was found to be 401 MPa at 0.2-percent offset

the spot center position, and inspection of this curve indicates that temperature measurements are robust. Dramatic drops in temperature were seen at the microsample edges when the pyrometer's spot was moved off of the microsample, but the shape of the temperature profiles across the thickness of the microsample are flatter than expected. Radiation of the microsample appears to create a target cross section for the pyrometer that is greater than the thickness of the microsample, with the added benefit that the temperature remains constant across the thickness of the microsample and is more easily measured with the pyrometer. A temperature variation of only 3.5°C was measured across the thickness for a microsample temperature set point of 770°C, whereas a difference of 6°C was measured for a set point of 800°C.

Microsamples of single-crystalline TiAl were tested at elevated temperatures in both tension and compression, and the result of this study was used to characterize the critical resolved flow stress of γ-TiAl as a function of temperature, orientation and sense of the applied stress.[18] The stress-strain curve shown in Fig. 11 is representative of the curves that were obtained in this study. This particular microsample was preheated to 800°C, pulled at a constant strain rate of 10^{-4} s^{-1} and observed to fail after 1.4-percent plastic strain. The deformed specimen was inspected after testing to ensure plastic deformation had occurred. An example of a constant-load creep test that was conducted on a microsample of fully lamellar TiAl at 760°C and 180 MPa is shown in Fig. 12. The creep tests conducted using the fully lamellar TiAl alloys demonstrate our ability to both conduct controlled monotonic creep experiments and to perform meaningful temperature change experiments.[19]

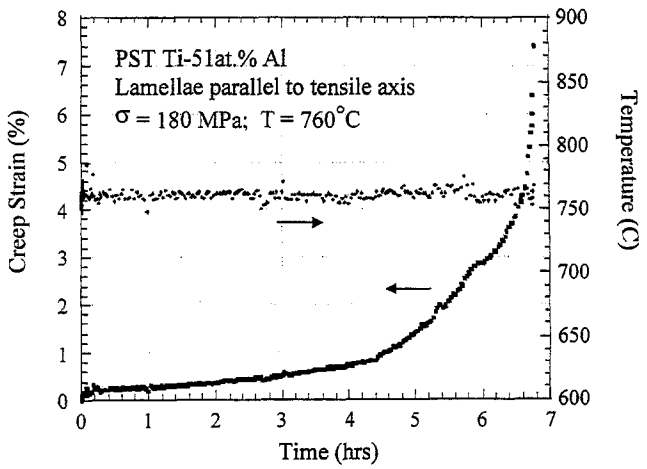

Fig. 12—Creep curve for a fully lamellar TiAl microsample tested in tension at 760°C and 160 MPa

Summary of Results

Microsample testing has established itself as a viable means by which the mechanical properties of a wide array of materials can be measured. With resistive heating, the capabilities of microsample testing have been expanded to include high-temperature tensile testing at temperatures in excess of 1500°C. Accurate in situ noncontact temperature measurements of the microsample were realized using an optical pyrometer. Although a significant temperature gradient is produced across the entire length of the microsample, a relatively small variation in temperature was measured within the gage section of the ISDG strain measurement system. Real-time control of the heating power delivered to the microsample allows microsamples be tested at constant elevated temperatures, and rapid variations of the applied power can be used to conduct temperature change experiments. Examples of successful elevated temperature tensile and creep experiments were provided.

Acknowledgments

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