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Thermophysical Properties of Solid and Liquid 90Ti–6Al–4V in the Temperature Range from 1400 to 2300 K Measured by Millisecond and Microsecond Pulse-Heating Techniques[1]

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The heat capacity and electrical resistivity of 90Ti–6Al–4V were measured in the temperature range from 1400 to 2300 K by two pulse-heating systems, operating in the millisecond and microsecond time regimes. The millisecond-resolution technique is based on resistive self-heating of a tube-shaped specimen from room temperature to melting in less than 500 ms. In this technique, the current through the specimen, the voltage drop along a defined portion of the specimen, and the temperature of the specimen are measured every 0.5 ms. The microsecond-resolution technique is based on the same principle as the millisecond-resolution technique except for using a rod-shaped specimen, a faster heating rate (by a factor of 10,000), and faster data recording (every 0.5 μs). Due to the rapid heating with the microsecond system, the specimen keeps its shape even in the liquid phase while measurements are made up to approximately 300 K above the melting temperature. A comparison between the results obtained from the two systems with very different heating rates shows significant differences in phase transition and melting behavior. The very high heating rate of the microsecond system shifts the solid–solid phase transition from the $(\alpha+\beta)$ to the β phase to a higher temperature, and changes the behavior of melting from melting over a temperature range to melting at a constant temperature like an eutectic alloy or a pure metal.

KEY WORDS: electrical resistivity; high temperatures; pulse heating; specific heat capacity; titanium alloy (90Ti–6Al–4V); transient techniques.

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1. INTRODUCTION

In this paper, application of transient heating techniques (millisecond and microsecond) to measurements of selected thermophysical properties (specific heat capacity, electrical resistivity) of the alloy 90Ti–6Al–4V in the vicinity of the melting temperature is described. The measurement techniques are based on rapid resistive self-heating of the specimen from room temperature to high temperature in a short time by the passage of an electrical current pulse. Simultaneously, the specimen temperature, the current through the specimen, and the voltage drop across a defined portion of the specimen are measured.

2. EXPERIMENTAL

2.1. Millisecond Pulse-Heating System

The millisecond pulse-heating system uses heavy-duty batteries to supply the current. The current through the tubular specimen is determined by measuring the voltage across a standard resistor in series with the specimen. The voltage across the middle part of the specimen is measured between spring-loaded knife-edge probes. They are mounted to stationary clamps via a lever mechanism that allows the probes to move in the axial direction to follow the thermal expansion of the specimen during the experiment. The temperature is measured by means of a high-speed single wavelength pyrometer, targeted at a small sighting hole machined through the wall of the specimen, which approximates a blackbody cavity. The target of the pyrometer is a circular area 0.2 mm in diameter. The working wavelength of the pyrometer is 902 nm, which is selected by an interference filter with a bandwidth of 20 nm. The thermal radiation from the target is collected by a silicon PIN photodiode and converted to voltage by precision amplifiers. The pyrometer is calibrated by comparison with a tungsten-filament standard lamp, which has been calibrated by the Deutscher Kalibrierdienst (DKD). Small corrections are made for the deviation from blackbody conditions (approximately 1.6 K) and for scattered light effects (approximately 2.2 K) [1].

The experimental quantities are recorded simultaneously every 0.5 ms by a data acquisition system with sample-and-hold amplifiers and an analog/digital converter of 16-bit full-scale resolution. Details regarding construction of the system are given in Ref. 2. Measurements are taken in the temperature range from 1400 to 1900 K. In the millisecond pulse-heating system, the specimens usually collapse prior to the full melt due to electromagnetic and gravitational forces as well as the effects of surface tension.

2.2. Microsecond Pulse-Heating System

The microsecond pulse-heating system uses high-voltage capacitors to supply the current, and the current through the rod-shaped specimen is determined using a current transformer. The voltage across the specimen is determined by measuring the current through a high-resistance path connected in parallel to the specimen. The radiance temperature is measured with a high-speed pyrometer, targeted at the surface of the specimen. The working wavelength of the pyrometer is 651 nm, with a bandwidth of 30 nm. The true temperature was calculated from the radiance temperature assuming a constant normal spectral emissivity in the liquid phase equal to the value at the beginning of melting. The normal spectral emissivity at the beginning of melting was determined from the measured radiance temperature and the true melting temperature obtained in millisecond-resolution experiments [3]. The pyrometer is calibrated by comparison with a tungsten-filament standard lamp, which has been calibrated by the Optical Technology Division of the National Institute of Standards and Technology (NIST).

The experimental quantities are recorded simultaneously every 0.2 μs by a four-channel digital oscilloscope with a full-scale resolution of 12 bits. Details regarding construction of the system are given in Ref. 4. Due to the rapid heating with the microsecond system, the specimens keep their shape even in the liquid phase, which allows measurements to be made up to approximately 300 K above the melting temperature.

3. MEASUREMENTS

3.1. Specimens

Measurements of specific heat capacity and electrical resistivity in the solid range (millisecond experiments) were performed on tubular specimens. The nominal dimensions of the tubular specimens were as follows: length, 75 mm; outside diameter, 9 mm; and wall thickness, 1 mm. For the temperature measurements, a rectangular hole (0.5×1.3 mm) was machined through the wall. The departure from ideal blackbody conditions for the specimen was estimated to be less than 1% [5]. A total of 15 measurements on three specimens was made.

Measurements in the liquid range (microsecond experiments) were performed on five wire-shaped specimens with nominal dimensions of 50-mm

Table I. Chemical Composition of the 90Ti–6Al–4V Alloy

Material form	Ti (%)	Al (%)	V (%)	Fe (ppm)	C (ppm)	Cr (ppm)	Cu (ppm)	Ni (ppm)	Mn (ppm)	Ca (ppm)
Tube	Major	6.2	4.1	2200	130	150	18	91	19	4
Wire	Major	6.4	4.3	1900	130	49	30	43	4	3

length and 1.6-mm diameter. Although the tubes and wires were supplied by different manufactures, their chemical compositions show few differences as can be seen in Table I.

3.2. Procedure and Data Reduction

All experiments were performed with the specimen in an argon environment at atmospheric pressure. All temperatures reported in this paper are based on the International Temperature Scale of 1990 [6, 7].

The specific heat capacity in the solid range was computed from data taken during the heating period of the millisecond experiments. A correction for power loss due to thermal radiation of up to 3% at the highest temperatures was made using data measured during the cooling of the specimen. From the power balance for the specimen during heating and cooling periods, the specific heat capacity c_P as a function of temperature T can be expressed as

$$c_\mathrm{P}(T) = \frac{U(T)\,I(T)}{m\left[\left(\frac{dT}{dt}\right)_\mathrm{h}(T) - \left(\frac{dT}{dt}\right)_\mathrm{c}(T)\right]} \tag{1}$$

where U is the voltage, I is the current, m is the effective mass between the voltage probes, $(dT/dt)_\mathrm{h}$ is the heating rate, and $(dT/dt)_\mathrm{c}$ is the cooling rate. The heating and cooling rates are treated as independent variables. The heating rate is obtained by fitting a polynomial equation to the temperature-versus-time data for the heating period, while the cooling rate is obtained by fitting an exponential equation to the data for the cooling period.

The specific heat capacity in the liquid range was calculated from the slope of the enthalpy-versus-temperature function obtained from the microsecond experiments. The enthalpy as a function of time was computed using the mass of the effective specimen and the absorbed energy obtained by numerically integrating the product of current and voltage over time from the beginning of the heating period. Since temperature is measured with the same time base, enthalpy as a function of temperature was obtained by evaluating both at common times. Because of the high heating rate in the microsecond experiments, the heat loss from the specimen to the surroundings was negligible, and hence no correction for heat losses was made.

The electrical resistivity of the specimen was computed from the measured voltage and current and specimen cross-sectional area and effective length. All results obtained are based on room temperature dimensions of the specimens, and no correction for thermal expansion is applied. It should be noted that the specimen expands uniformly in all directions in the millisecond experiments, but in the microsecond experiments, the expansion occurs mostly in the radial direction due to the high heating rate (no bending of the specimen is observable).

4. RESULTS

4.1. Specific Heat Capacity

The variation of specific heat capacity as a function of temperature in the solid and liquid ranges is shown in Table II. All of the data in the solid range in Table II were obtained from the millisecond experiments. The reproducibility of measurements for an individual specimen is 0.6% (standard deviation), and that between specimens is 0.7%. The least-squares fit to the specific heat capacity versus temperature data, in the range $1400\ \mathrm{K} < T < 1900\ \mathrm{K}$, is

$$c_{\mathrm{P}} = 1547.3 - 1.1860T + 4.3040 \times 10^{-4} T^2 \tag{2}$$

where c_{P} is in $\mathrm{J \cdot kg^{-1} \cdot K^{-1}}$ and T is in K.

The specific heat capacity in the liquid range up to approximately 300 K above the melting temperature was obtained from the microsecond experiments. Because the measured enthalpy-versus-temperature function was linear in the liquid range, a constant specific heat capacity was calculated. The average value of the specific heat capacity in the liquid range is $931\ \mathrm{J \cdot kg^{-1} \cdot K^{-1}}$, with a standard deviation of $26\ \mathrm{J \cdot kg^{-1} \cdot K^{-1}}$ (2.8%).

4.2. Electrical Resistivity

The variation of electrical resistivity (based on room temperature dimensions of the specimen) as a function of temperature in the solid and

Table II. Specific Heat Capacity and Electrical Resistivity[a] of 90Ti–6Al–4V Alloy, Measured with a Millisecond Technique (Solid Range) and a Microsecond Technique (Liquid Range)

T (K)	c_P ($\mathrm{J \cdot kg^{-1} \cdot K^{-1}}$)	ρ ($\mu\Omega \cdot \mathrm{m}$)
1400	730	1.72
1500	737	1.72
1600	752	1.73
1700	775	1.74
1800	807	1.75
1900	848	1.76
Melting		
2000	931	1.66
2100	931	1.65
2200	931	1.64
2300	931	1.63

[a] Based on room-temperature dimensions of the specimen.

liquid range is shown in Table II. All of the data in the solid range in Table II were obtained from the millisecond experiments. The reproducibility of measurements for an individual specimen is 0.2% (standard deviation), and that between specimens is 0.3%. The least-squares fit to the electrical resistivity versus temperature data, in the range 1400 K $< T <$ 1900 K, is

$$\rho = 1.6096 + 7.6884 \times 10^{-5} T \tag{3}$$

where ρ is in $\mu\Omega \cdot \mathrm{m}$, and T is in K.

The electrical resistivity in the liquid range was obtained from microsecond experiments. The reproducibility of measurements between specimens is 0.4%. The least-squares fit to the electrical resistivity versus temperature data, in the range 2000 K $< T <$ 2300 K, is

$$\rho = 1.8919 - 1.1606 \times 10^{-4} T \tag{4}$$

where ρ is in $\mu\Omega \cdot \mathrm{m}$, and T is in K.

4.3. Estimates of Uncertainties

Detailed analyses of sources and magnitudes of standard uncertainties in specific heat capacity and electrical resistivity measurements with the millisecond technique and the microsecond technique are given in Refs. 1 and 2 and 4, respectively.

According to the earlier analysis of uncertainties, the expanded uncertainties (at a 2-standard deviation level) of the measured specific heat capacity in the solid phase for the millisecond and microsecond techniques are ±3 and ±5%, respectively, and that for the liquid phase is ±7% (microsecond). For the measured electrical resistivity, the uncertainties in the solid and liquid ranges are ±1.5% (millisecond) and ±3% (microsecond), respectively.

5. DISCUSSION

The only other measurements of specific heat capacity and electrical resistivity of solid 90Ti–6Al–4V near its melting range were performed by Cezairliyan et al. [3] using a similar millisecond pulse-heating technique. The average and maximum deviations of our results from the earlier results [3] in the range from 1450 to 1900 K are 0.14 and 0.52% for specific heat capacity and 0.12 and 0.31% for electrical resistivity, respectively. The present results for specific heat capacity and electrical resistivity agree very well with the earlier results.

The values obtained for the specific heat capacity in the solid state from microsecond experiments agree with the results obtained by the more accurate millisecond technique within their combined uncertainties. Taking into account the difference in thermal expansion as discussed in Section 3.2 (leading to an estimated difference of up to approximately 2% close to melting), the values obtained for the electrical resistivity in the solid state from microsecond experiments agree well with the results obtained by the millisecond technique.

The difference in measured electrical resistivity between the millisecond and the microsecond experiments became larger than the estimated uncertainties in the vicinity of the $(\alpha+\beta)$-to-β phase transition. This is probably due to the very high heating rate in the microsecond experiments, in which experimental conditions are not at thermal equilibrium. The heating rate is approximately 3000 $\mathrm{K \cdot s^{-1}}$ in millisecond experiments and $3 \times 10^7\ \mathrm{K \cdot s^{-1}}$ in microsecond experiments. Figure 1 shows the electrical resistivity as a function of the specific enthalpy (relative to 298 K), which generally corresponds to the temperature. The difference in electrical resistivity at higher enthalpies is due to the difference in the thermal expansion behavior mentioned in Section 3.2. In addition, under a higher heating rate in the microsecond experiments, the phase transition point from the $(\alpha+\beta)$ to the β phase, which is indicated by the arrow in Fig. 1, shifted to a higher enthalpy.

A similar heating-rate effect is noticed at melting. Figure 2 shows the specific enthalpy as a function of the radiance temperature at 651 nm in the

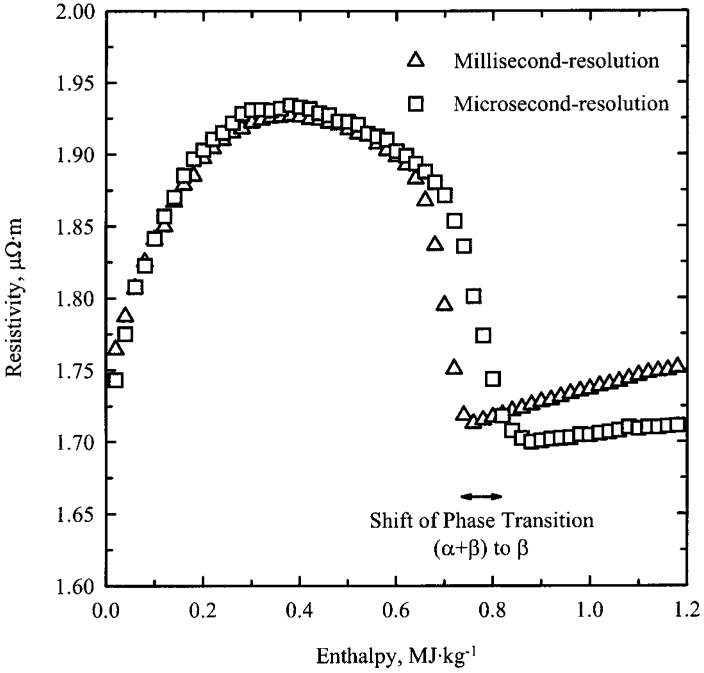

Fig. 1. Variation of electrical resistivity as a function of specific enthalpy for a typical millisecond experiment (heating rate of approximately 3000 $K \cdot s^{-1}$) and a microsecond experiment (heating faster by a factor of 10^4) on 90Ti–6Al–4V specimens in the $(\alpha + \beta)$ and β phases.

vicinity of melting, measured on wire-shaped specimens with the same pyrometer using microsecond and millisecond pulse heating (approximately 10^7 and 10^3 $K \cdot s^{-1}$, respectively). Under a microsecond heating rate, the specimen melts at a constant temperature (like a eutectic alloy or a pure metal) instead of over a temperature range as observed with a lower heating rate. This constant melting temperature is shifted toward the liquidus temperature. An analysis of the impact of heating rate on the shape and temperature of the melting plateau of alloys, based on solute segregation during melting, is given by Basak et al. [8].

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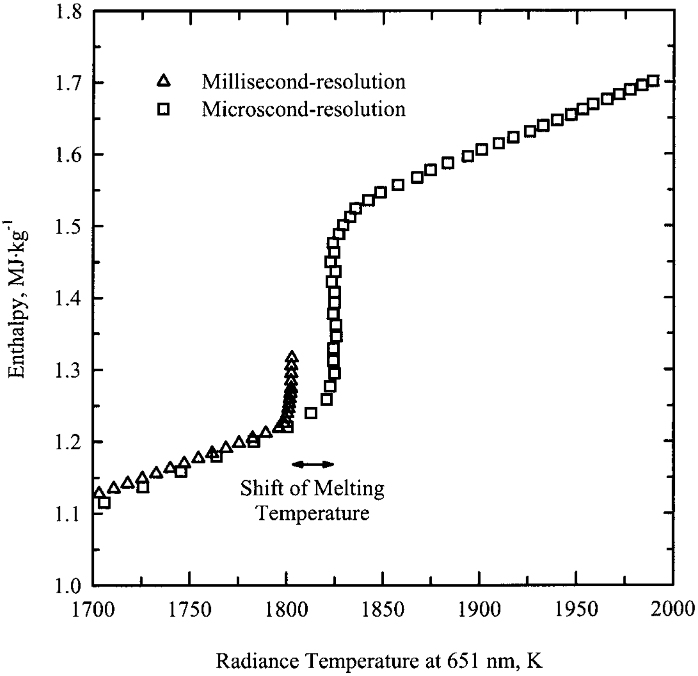

Fig. 2. Variation of specific enthalpy as a function of radiance temperature for a typical millisecond experiment (heating rate of approximately 3000 $K \cdot s^{-1}$) and a microsecond experiment (heating faster by a factor of 10^4) on 90Ti–6Al–4V specimens in the vicinity of the melting temperature.

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