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Measurement of the Heat of Fusion of Titanium and a Titanium Alloy (90Ti-6Al-4V) by a Microsecond-Resolution Transient Technique[1]

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A microsecond-resolution pulse heating technique was used for the measurement of the heat of fusion of titanium and a titanium alloy (90Ti-6Al-4V). The method is based on rapid (50- to 100-μs) resistive self-heating of the specimen by a high-current pulse from a capacitor discharge system and measuring, as functions of time, current through the specimen, voltage across the specimen, and radiance of the specimen. Melting of the specimen is manifested by a plateau in the measured radiance. The time integral of the net power absorbed by the specimen during melting yields the heat of fusion. The values obtained for heat of fusion were 272 $J \cdot g^{-1}$ (13.0 $kJ \cdot mol^{-1}$) for titanium and 286 $J \cdot g^{-1}$ for the alloy 90Ti-6Al-4V, with an estimated maximum uncertainty of $\pm 6\%$ in each value.

KEY WORDS: heat of fusion; high temperatures; melting; pulse heating; titanium; titanium alloy (90Ti-6Al-4V); transient techniques.

1. INTRODUCTION

This paper describes measurements of the heat of fusion of titanium and a titanium alloy (90Ti-6Al-4V) performed using a microsecond-resolution transient technique developed earlier in our laboratory [1]. The method for measuring this property is based on rapid (50- to 100-μs) resistive self-heating of the specimen by a short-duration current pulse from a capacitor discharge system. While the specimen is heating, simultaneous measurements of current through the specimen, voltage across the

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specimen, and radiance of the specimen are made with microsecond resolution. Details regarding the construction and operation of the measurement system are given elsewhere [1–3].

2. MEASUREMENTS ON TITANIUM AND 90Ti-6Al-4V ALLOY

Measurements were made on seven titanium specimens and seven 90Ti-6Al-4V alloy specimens in the form of wires with the following nominal dimensions: diameter, 1.6 mm; and length, 63.5 mm. The specimen was clamped into the specimen chamber with approximately 38 mm of the specimen exposed between the clamps. Voltage probes, made of tantalum strips (6.4 mm wide and 0.25 mm thick) sharpened to a knife edge on one end, were placed on knife marks about 31 mm apart made on the middle portion of the specimen. The knife marks defined an "effective" specimen free of axial temperature gradients for the duration of the experiment. Each experiment was conducted with the specimen in an argon environment at slightly above atmospheric pressure.

The mass of each "effective" specimen was determined from the distance between the knife marks on the specimen and the mass per unit length of the material. The measured mass per unit length of the specimens was 0.08732 $g \cdot cm^{-1}$ for titanium and 0.09027 $g \cdot cm^{-1}$ for the titanium alloy. As reported by the manufacturer, the titanium material was 99.9% pure with the following major impurities: O, 700 ppm; Fe, 300 ppm; C, 100 ppm; N, 40 ppm; H, 35 ppm. The composition of the 90Ti-6Al-4V alloy material as reported by the manufacturer was: Ti, 89.4% by weight; Al, 6.3% by weight; V, 4.1% by weight; O, 600 ppm; Fe, 500 ppm; C, 200 ppm; N, 80 ppm; H, 37 ppm; and Y, <10 ppm.

In a typical experiment, the capacitor bank was charged to an initial voltage of about 4.4 kV and discharged in the crowbar mode of operation [1]. The resulting current pulse heated the specimen from room temperature through melting into the liquid phase in a time interval less than 100 μs. An oscilloscope trace photograph showing the time variation of current, voltage, and radiance for a typical experiment on titanium is shown in Fig. 1. The "notch" that occurs in the voltage trace just after its peak is due to the rapid resistance change associated with the $\alpha \rightarrow \beta$ phase transformation at 1166 K in titanium [4]. The peak current through the specimen was typically 24 to 26 kA and the peak voltage across the specimen was typically between 630 and 700 V. The plateau in the radiance curve indicates the melting of the specimen. In the liquid phase, the specimen rises approximately 200 K above the melting temperature before the specimen begins to disintegrate. The heating rate of the specimen below the melting temperature was estimated to be 5×10^7 $K \cdot s^{-1}$. Experiments

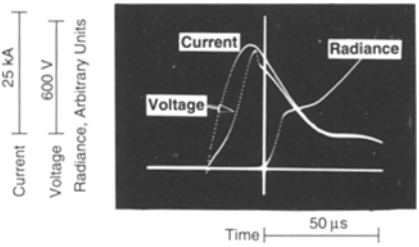

Fig. 1. Oscilloscope trace photograph showing current, voltage, and radiance waveforms during a typical experiment on titanium.

on 90Ti-6Al-4V alloy specimens yielded similar current, voltage and radiance results including a slightly broader "notch" in the voltage trace.

Actual radiance temperatures could not be determined from measured radiance because the interference filter (650 nm) of the high-speed pyrometer was removed in order to increase pyrometer sensitivity. Radiance temperature values were computed for each specimen as if the 650-nm filter were still in the pyrometer and then normalized to unity at the beginning of each melting plateau. This procedure yielded "effective" radiance temperature ratios which were used, as described in the next section, to determine more precisely the beginning and end of the melting plateau. These "effective" radiance temperature ratios varied from 0.80 to 1.15 in the vicinity of the melting region.

3. RESULTS

The heat of fusion of each specimen was determined from the energy absorbed by the specimen during the melting period. From the measured data for current and voltage, the absorbed power for each individual point was computed. The energy absorbed by the specimen above an initial "effective" radiance temperature ratio (0.80) was determined by integrating power point-by-point over time to the end of the experiment (an "effective" radiance temperature ratio of about 1.15). The measured mass per unit length of each specimen and the room temperature length of the effective specimen were used to express absorbed energy in units of $J \cdot g^{-1}$. Because of the speed of the experiments, corrections for heat losses due to thermal radiation or conduction were not required. The result for a typical experiment on titanium is illustrated in Fig. 2, which shows the variation of "effective" radiance temperature ratio as a function of absorbed energy. A similar variation was obtained for experiments on the alloy 90Ti-6Al-4V.

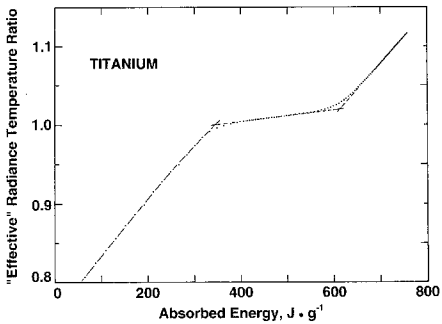

Fig. 2. Variation of "effective" radiance temperature ratio (ratio of "effective" radiance temperature to that at the beginning of the melting plateau) as a function of absorbed energy for a typical experiment on titanium.

The "effective" radiance temperature ratio-vs-energy data, as illustrated in Fig. 2, were fitted by the least-squares method to a quadratic function for the premelting region and linear functions for the melting (plateau) region and the postmelting region. Because of the lack of sharp discontinuities at the beginning and at the end of the melting plateau, those data points which lie in the transition between premelting and the plateau and postmelting and the plateau were excluded from the data fits. The dashed lines in Fig. 2 represent the least-squares fits to the data in each region. The percentage standard deviation of the fits in each region for the titanium specimens ranged between the following minimum and maximum

Table I. Experimental Results on the Heat of Fusion of Titanium

Specimen No.	Heating time to start of melting (μs)	Duration of melting plateau (μs)	Heat of fusion ($J \cdot g^{-1}$)
1	38.8	13.9	273
2	42.6	28.0	270
3	36.8	11.2	275
4	39.3	16.7	276
5	37.9	12.1	267
6	38.8	13.1	279
7	40.2	18.3	267
Average			.272

values: premelting region, 0.06–0.09%; plateau region, 0.02–0.03%; and postmelting region, 0.01–0.03%. The percentage standard deviation of the fits for the 90Ti-6Al-4V alloy had the following ranges: premelting region, 0.1–0.2%; plateau region, 0.02–0.04%; and postmelting region, 0.02–0.04%. The beginning and end of the melting plateau were defined by the intersections of the fitted curves for each region. Heat of fusion values for each experiment were obtained from the difference between the value of absorbed energy at the end of the melting plateau and absorbed energy at the beginning of the plateau. The experimental results for the heat of fusion of titanium and 90Ti-6Al-4V alloy are given in Tables I and II, respectively. The average value of the heat of fusion for titanium is $272\ J \cdot g^{-1}$ ($13.0\ kJ \cdot mol^{-1}$), with an average absolute deviation of $4\ J \cdot g^{-1}$ and a maximum absolute deviation of $7\ J \cdot g^{-1}$. The average value of the heat of fusion for 90Ti-6Al-4V alloy is $286\ J \cdot g^{-1}$, with an average absolute deviation of $3\ J \cdot g^{-1}$ and a maximum absolute deviation of $4\ J \cdot g^{-1}$.

4. ESTIMATE OF ERRORS

The uncertainty in the determination of the melting duration is the single largest contributor to the uncertainty in the value of heat of fusion. In the present experiments, this uncertainty was estimated to be not more than $\pm 1\ \mu s$, which corresponds to a maximum uncertainty in the heat of fusion of about $\pm 5\%$. Uncertainties in other quantities, current, voltage, mass, etc., are 1% or less each. The maximum uncertainty from all sources in the reported values of the heat of fusion of titanium and 90Ti-6Al-4V

Table II. Experimental Results on the Heat of Fusion of 90Ti-6Al-4V Alloy

Specimen No.	Heating time to start of melting (μs)	Duration of melting plateau (μs)	Heat of fusion ($J \cdot g^{-1}$)
1	35.7	12.7	290
2	35.4	12.0	288
3	36.7	14.5	282
4	34.9	10.8	288
5	36.4	13.7	283
6	36.0	12.3	284
7	35.9	13.5	287
Average			286

alloy is estimated to be $\pm 6\%$. Details regarding the sources and estimates of error using the present measurement system are given in an earlier publication [1].

5. DISCUSSION

The measurement of the heat of fusion of titanium presented in this work is compared with the values reported in the literature [7–10] in Table III. The values reported by Maurakh et al. [7] and Martynyuk and Tsapkov [10] are more than 30% greater than the present value. The values of Treverton and Margrave [8] and Berezin et al. [9], both obtained by levitation calorimetry, are about 1.5 and 8.5%, respectively, higher than the present value.

No measurements of the heat of fusion of 90Ti-6Al-4V alloy were found in the literature. However, an estimated value can be computed from values of the heat of fusion of the metals in the alloy assuming that each metal contributes to the total heat of fusion in proportion to its percent weight in the alloy. Using the value for heat of fusion of titanium as reported here, 272 $J \cdot g^{-1}$, and heat of fusion of aluminum and vanadium as reported in the literature, 397 and 448 $J \cdot g^{-1}$ [11], respectively, a value of 287 $J \cdot g^{-1}$ was computed for the 90Ti-6Al-4V alloy. The measured value (286 $J \cdot g^{-1}$) agrees well with this estimate.

The observed melting plateaus of the titanium and 90Ti-6Al-4V alloy specimens show a much larger positive slope (Figs. 1 and 2) than that observed for experiments on other materials [1, 12]. It was determined that this slope is due largely to the removal of the interference filter from the pyrometer in the present experiments. Experiments on other materials (i.e., niobium) using the pyrometer with the interference filter removed produced radiance melting plateaus with much larger slopes than that observed in experiments using the 650-nm interference filter. These results indicate that there may be differential changes in the normal spectral emissivity of the material as it melts.

Table III. Heat of Fusion of Titanium Reported in the Literature

Investigator(s)	Ref. No.	Year	Heat of fusion ($kJ \cdot mol^{-1}$)	Technique
Maurakh et al.	7	1967	17.2	Drop calorimetry
Treverton and Margrave	8	1971	13.2	Levitation calorimetry
Berezin et al.	9	1974	14.1	Levitation calorimetry
Martynyuk and Tsapkov	10	1974	18.0	Pulse heating
Present work			13.0	Pulse heating

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