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Application of High-Speed Laser Polarimetry to Non-Contact Detection of Phase Transformations in Metals and Alloys at High Temperatures[1]

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ABSTRACT

A high-speed laser polarimetry technique, developed recently for the measurement of normal spectral emissivity of materials at high temperatures, was used to detect solid-solid and solid-liquid phase transformations in metals and alloys in millisecond-resolution pulse-heating experiments. Experiments were performed where normal spectral emissivity at 633 nm was measured simultaneously with surface radiance temperature, resistance and/or voltage drop across the specimen. It was observed that a phase transformation, as indicated either by an arrest in the specimen temperature or changes in the resistance and/or voltage drop, generally caused a change in normal spectral emissivity. Experiments were conducted on cobalt, iron, hafnium, titanium, and zirconium to detect solid-solid phase transformations. Similar experiments were also performed on niobium, titanium, and the alloy 85titanium-15molybdenum (mass%) to detect solid-liquid phase transformations (melting).

KEY WORDS: alloys; high temperatures; laser polarimetry; melting; normal spectral emissivity; metals; phase transformations; pulse heating.

1. INTRODUCTION

Recently, a high-speed laser polarimeter was developed for the measurement of normal spectral emissivity of a specimen pulse-heated to high temperatures in subsecond-duration experiments [1]. The main interest in measurement of emissivity was in connection with the determination of the true temperature of a specimen from radiometric measurements of its surface radiance temperature.

The objective of the present paper is to report and discuss the results of an experimental study on the application of the high-speed laser polarimetry technique to non-contact detection of solid-solid and solid-liquid phase transformations in metals and alloys in millisecond-resolution pulse-heating experiments.

2. MEASUREMENT METHOD

The method is based on rapid resistive self-heating of the specimen from room temperature to high temperatures (up to its melting point) in less than one second by the passage of an electrical current pulse through it; and on measuring, with millisecond resolution, current through the specimen, voltage drop across the specimen, normal spectral emissivity of the specimen, and radiance temperature of the specimen. The current through the specimen was determined from the measurement of the voltage drop across a standard resistor placed in series with the specimen. The voltage drop across the specimen was measured between the clamps at the ends of the specimen. The normal spectral emissivity (at 633 nm) of the specimen was measured with a high-speed laser polarimeter [1]. The surface radiance temperature of the specimen was measured at 651 nm with a high-speed pyrometer [2]. Data were recorded with a digital data

acquisition system (16-bit resolution) at the rate of 2 kHz for each experimental quantity. Details regarding construction and operation of the original measurement system, the methods of measuring experimental quantities, and other pertinent information are given in earlier publications [3, 4]. A recent significant modification to the system involving a computer-controlled solid-state switch for the control of the current through the specimen is described elsewhere [5]. For brevity, in the rest of the paper, 'emissivity' is used to mean 'normal spectral emissivity at 633 nm wavelength' unless otherwise noted.

3. MEASUREMENTS

3.1. Specimens

The purity, physical dimensions, and preheat conditions for the rod-shaped specimens used in the experiments are summarized in Table I. The specimens were polished to a smooth finish before the experiments to provide good reflectivity for the polarimeter laser beam. Whenever needed the specimens were preheated to remove surface contaminants and relieve internal stresses. Preheat was applied to a specimen either by a pulse current or by a brief steady-state current which maintained the specimen at a predetermined temperature for a short time, always below the transformation temperature for that material.

3.2. Experiments

For the study of solid-solid phase transformations, experiments were conducted on five metals: cobalt, iron, hafnium, titanium, and zirconium. Each specimen was heated from room temperature to above the transformation temperature

and experimental quantities: emissivity, radiance temperature, current, and the voltage drop across the specimen were recorded. For solid-liquid phase transformations, experiments were conducted on two metals, niobium and titanium, and the alloy 85titanium-15molybdenum (mass%). Each specimen was heated from room temperature to the melting temperature and experimental quantities emissivity and radiance temperature were recorded. All the experiments were conducted with the specimens in an argon (99.999% pure) environment at slightly above atmospheric pressure.

4. RESULTS

4.1. Presentation of Results

To study detection of phase transformations, emissivity of the specimen during the pulse heating experiment was plotted as a function of time. In addition, electrical resistance of the specimen (or voltage drop across the specimen), and radiance temperature of the specimen (if available) were also plotted in the same figure. Zero time indicates the start of the current pulse. The x-axis represents the elapsed time with reference to the start of the current pulse (zero time). Variations in emissivity as a function of time were studied to establish whether any change in emissivity corresponded to changes in the other quantities such as radiance temperature, resistance and/or voltage, as applicable.

4.2. Solid-Solid Phase Transformation

Figure 1, for cobalt, shows a sharp change in emissivity around 125 ms. This is accompanied by a sharp change in resistance (see the dashed line in Fig. 1). These changes are likely to be associated with the $\alpha \rightarrow \beta$ transformation in cobalt.

Figure 2, for iron, shows a sharp change in emissivity around 290 ms. A corresponding change is observed in the voltage signal. This is likely to be associated with the $\alpha \rightarrow \gamma$ transformation in iron. This transformation temperature is below the detection range of the pyrometer. A change in emissivity is also observed around 480 ms. The voltage signal does not show any corresponding change. Radiance temperature shows an arrest, signifying the beginning of the transformation and remains essentially constant until the end of the transformation. It may be noted that the drop in emissivity lags the temperature arrest (see the dashed line in Fig. 2). This change is likely to be associated with the $\gamma \rightarrow \delta$ transformation in iron. In addition to the above two distinct changes in emissivity, a change in the slope of emissivity is observed around 240 ms (Fig. 2). This change is likely to correspond to the ferromagnetic-to-paramagnetic transformation in iron.

Figure 3, for hafnium, shows an arrest in the radiance temperature between 410 and 460 ms, signifying the occurrence of the $\alpha \rightarrow \beta$ transformation. It may be noted that the resistance also undergoes a change in this region. The times corresponding to the changes in resistance at the beginning and the end of the transformation agree with the change in the radiance temperature (see dashed lines in Fig. 3). It may be noted that while the increase in emissivity is sharp, it lags the start of transformation, as indicated by the beginning of the temperature arrest. During

cooling, emissivity undergoes a change which is in agreement with the change in the slope of radiance temperature; this change is likely to correspond to the $\beta \rightarrow \alpha$ transformation.

Experiments were also performed on titanium and zirconium to detect the $\alpha \rightarrow \beta$ phase transformation in these metals. For both metals, emissivity and resistance, plotted versus time, showed existence of peaks, however, there were considerable disagreements in times at which these peaks occurred.

4.3. Solid-Liquid Phase Transformation

For the melting experiments, the radiance temperature versus time plot shows a region of rapid heating, followed by a temperature arrest during which the temperature remains essentially constant. The maximum point in the heating curve represents the onset of melting followed by reductions in both radiance temperature and emissivity. These reductions are due to the smoothing of the surface caused by the beginning of melting of the specimen.

The results of radiance temperature and emissivity for niobium, titanium, and the alloy, 85titanium-15molybdenum (mass%), are presented in Figs. 4, 5, and 6, respectively. It may be noted that a sharp change in emissivity corresponds to the change in radiance temperature upon beginning of melting of the specimen.

5. DISCUSSION AND CONCLUSION

The laser polarimetry technique was partially successful in detecting solid-solid phase transformations. In the case of the $\alpha \rightarrow \beta$ transformation in cobalt and in the $\alpha \rightarrow \gamma$ transformation in iron, a sharp change in emissivity was observed at the onset of

the transformations. In the case of the $\gamma \rightarrow \delta$ transformation in iron and the $\alpha \rightarrow \beta$ transformation in hafnium, a lag in the emissivity change was noted. A change in the slope of emissivity of iron was observed which likely corresponds to the magnetic transformation.

The laser polarimetry technique was quite successful in detecting solid-liquid phase transformations. A sharp change in emissivity of the specimen upon onset of melting was observed for all the studied materials.

Further work is required to more firmly establish the limits of applicability of the laser polarimetry technique to the detection of phase transformations, especially solid-solid, in metals and alloys.

REFERENCES

1. A. Cezairliyan, S. Krishnan, and J. L. McClure, Int. J. Thermophys. 17: 1455 (1996).

2. E. Kaschnitz and A. Cezairliyan, Int. J. Thermophys. 17: 1069 (1996).

3. A. Cezairliyan, M. S. Morse, H. A. Berman, and C. W. Beckett, J. Res. Natl. Bur. Stand. (U.S.) 74A: 65 (1970).

4. A. Cezairliyan, J. Res. Natl. Bur. Stand. (U.S.) 75C: 7 (1971).

5. T. Matsumoto and A. Cezairliyan, Int. J. Thermophys. 18: xxxx (1997).

Table I. Characteristics of the Specimens Used in the Experiments

Material	Purity (mass%)	Diameter (mm)	Length (mm)	Number of Specimens	Preheat Conditions
Cobalt	99.997	1	64	1	pulse to 1500 K
Iron	99.99+	1	64	2	pulse to 900 K
Hafnium[a]	97.0	1	64	4	steady-state at 1800 K for 1 s (3 times)
Niobium	99.9	1	64	4	steady-state at 2370 K for 1 s
Titanium	99.9+	1.6	64	1	none
Zirconium	99.85+	1	64	3	pulse to 550 K
85Ti-15Mo (mass%)	99.5	2	73	4	steady-state at 1800 K for 1 s

[a] Contained approximately 3 mass% zirconium.

FIGURE CAPTIONS

Figure 1. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, and electrical resistance, R, of cobalt, as a function of time. The dashed line indicates the solid-solid transformation.

Figure 2. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, radiance temperature, T_r, and voltage across the specimen, V, of iron, as a function of time, showing three (dashed lines) solid-solid transformations: a magnetic transformation and two structural transformations.

Figure 3. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, radiance temperature, T_r, and specimen electrical resistance, R, of hafnium, as a function of time. The first two dashed lines indicate the beginning and end of a solid-solid transformation, respectively, during heating, and the third dashed line indicates the same transformation during cooling.

Figure 4. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, and radiance temperature, T_r, of niobium, as a function of time showing the onset of melting and the melting plateau. The dashed line indicates the onset of melting.

Figure 5. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, and radiance temperature, T_r, of titanium, as a function of time showing the onset of melting and the melting plateau. The dashed line indicates the onset of melting.

Figure 6. Normal spectral emissivity, $\varepsilon_{n,\lambda}$, and radiance temperature, T_r, of the alloy 85titanium-15molybdenum (mass%), as a function of time showing the onset of melting and the melting plateau. The dashed line indicates the approximate onset of melting.

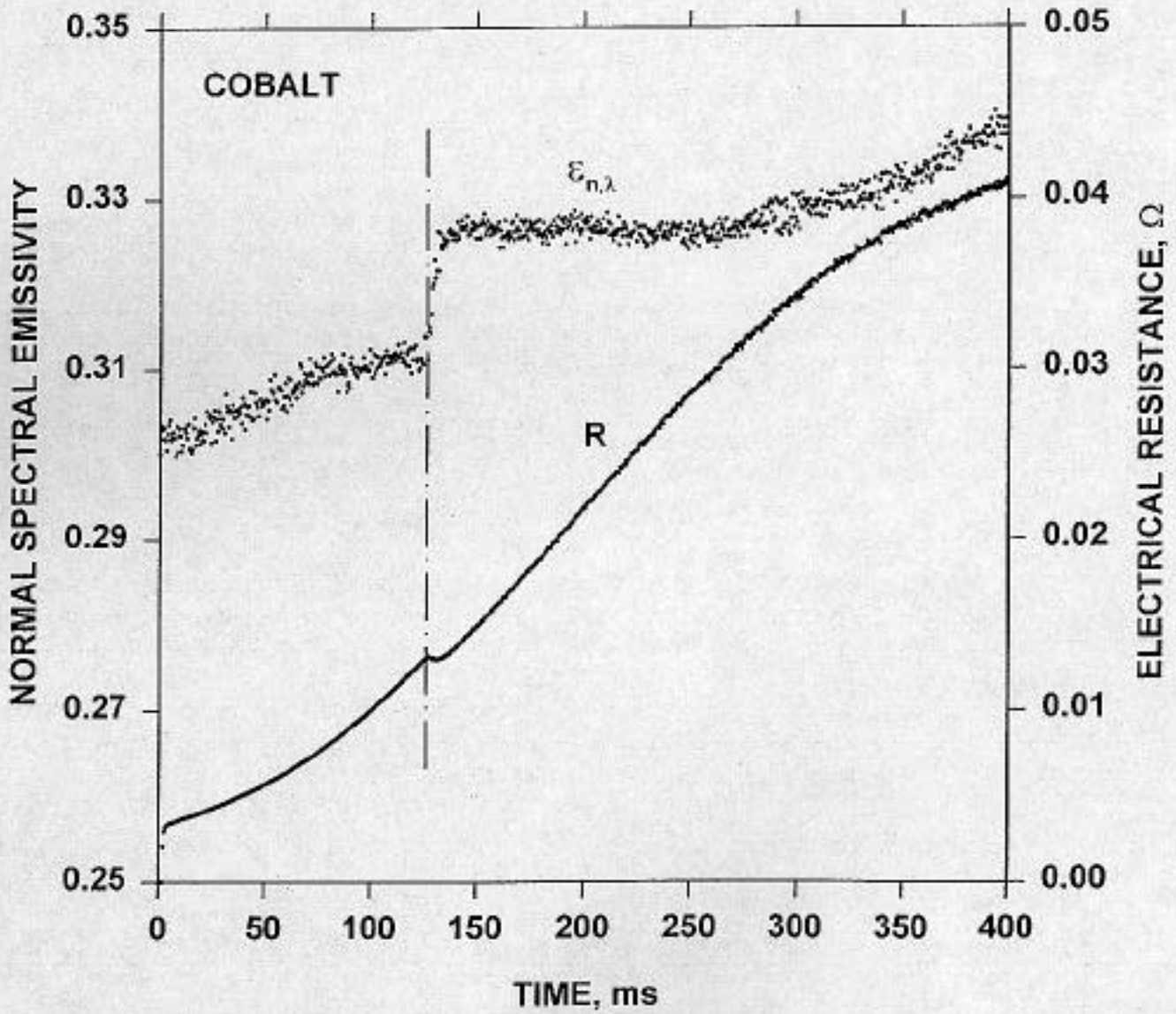
COBALT
$\varepsilon_{n,\lambda}$
R
NORMAL SPECTRAL EMISSIVITY
ELECTRICAL RESISTANCE, Ω
TIME, ms
0.35
0.33
0.31
0.29
0.27
0.25
0.05
0.04
0.03
0.02
0.01
0.00
0
50
100
150
200
250
300
350
400

Figure 1

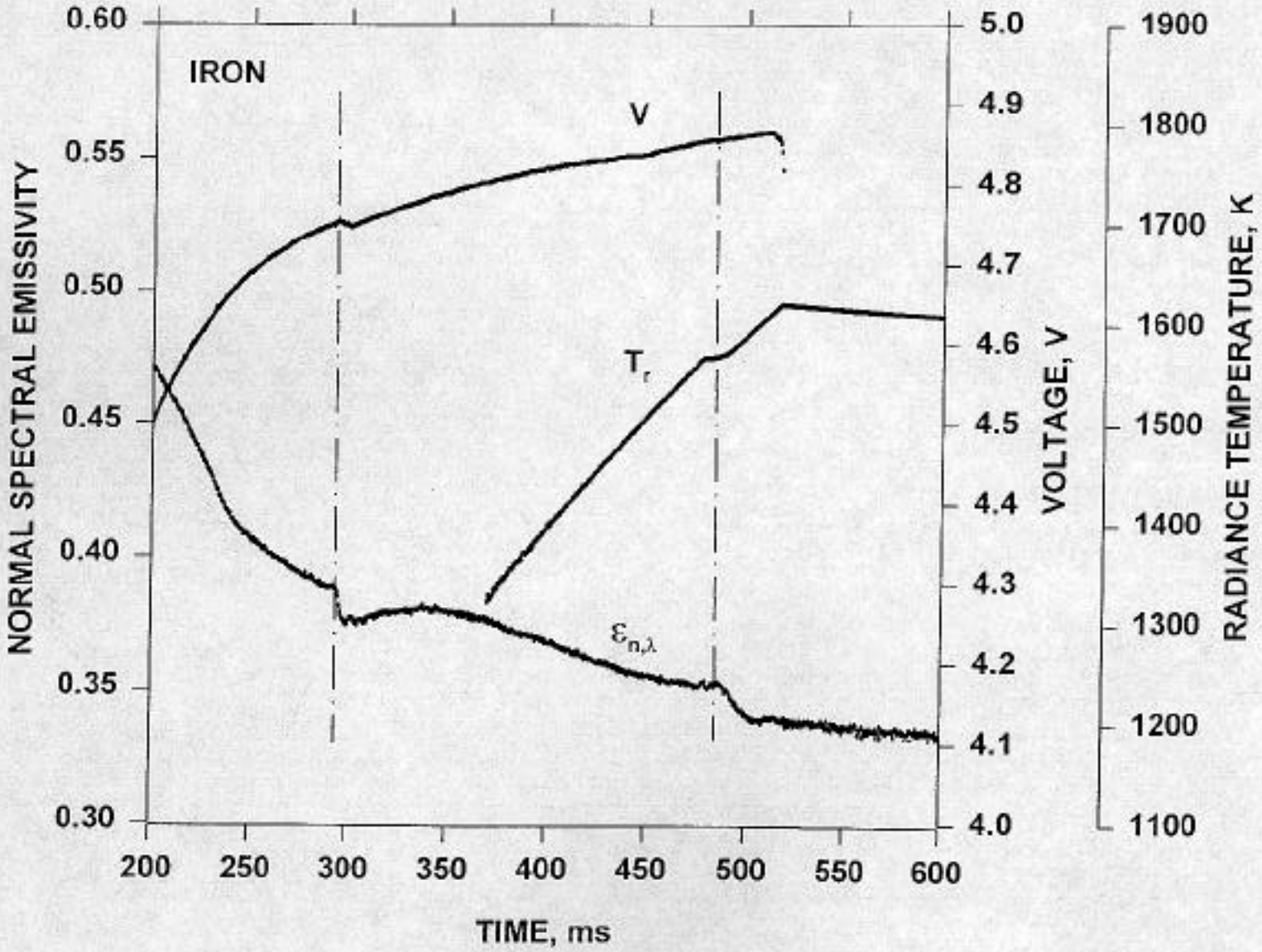
IRON
V
T_r
$\varepsilon_{n,\lambda}$
NORMAL SPECTRAL EMISSIVITY
0.60
0.55
0.50
0.45
0.40
0.35
0.30
VOLTAGE, V
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
RADIANCE TEMPERATURE, K
1900
1800
1700
1600
1500
1400
1300
1200
1100
200
250
300
350
400
450
500
550
600
TIME, ms

Figure 2

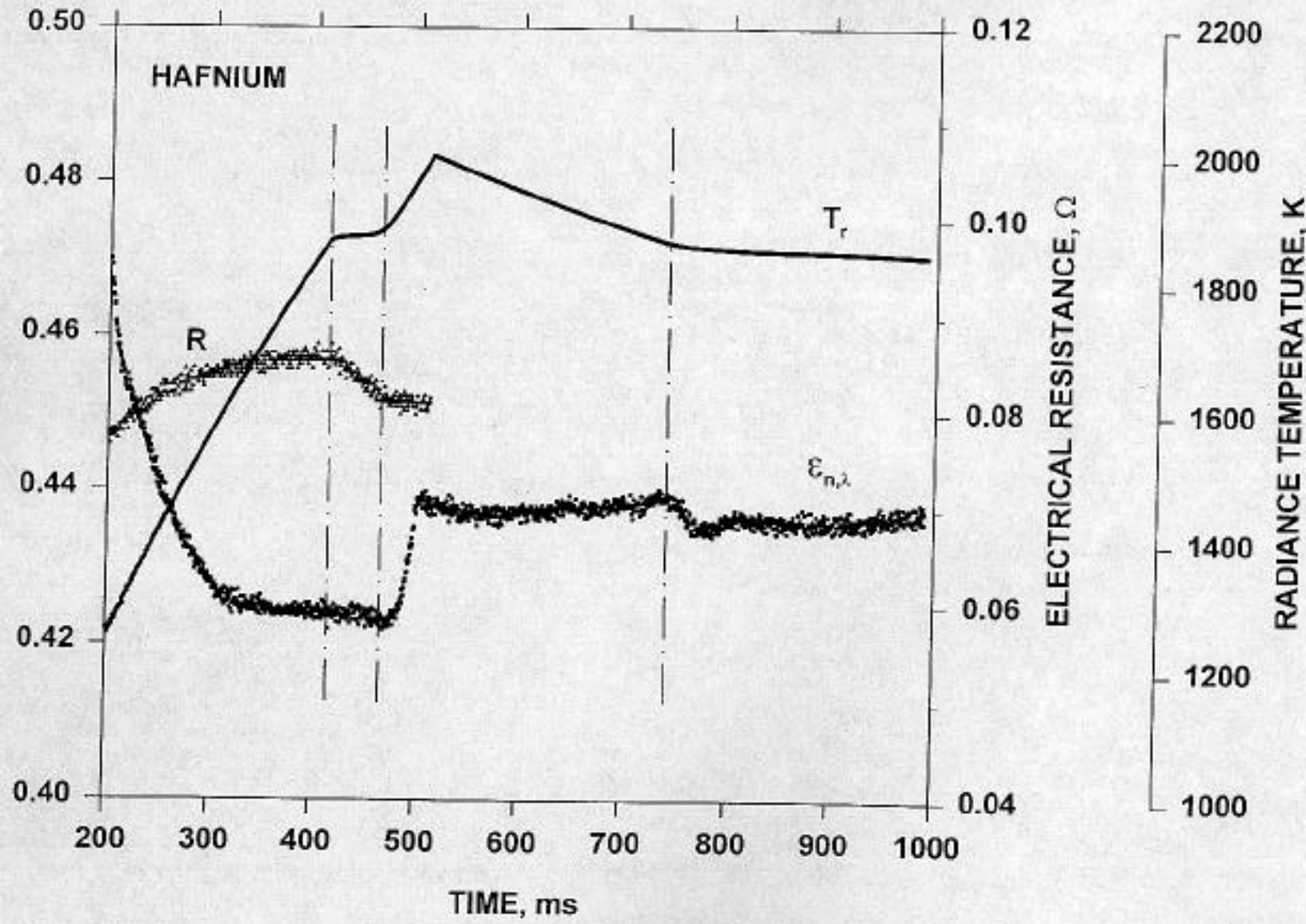
HAFNIUM
0.50
0.48
0.46
0.44
0.42
0.40
0.12
0.10
0.08
0.06
0.04
2200
2000
1800
1600
1400
1200
1000
200
300
400
500
600
700
800
900
1000
TIME, ms
ELECTRICAL RESISTANCE, Ω
RADIANCE TEMPERATURE, K
R
T_r
$\varepsilon_{n,\lambda}$

Figure 3

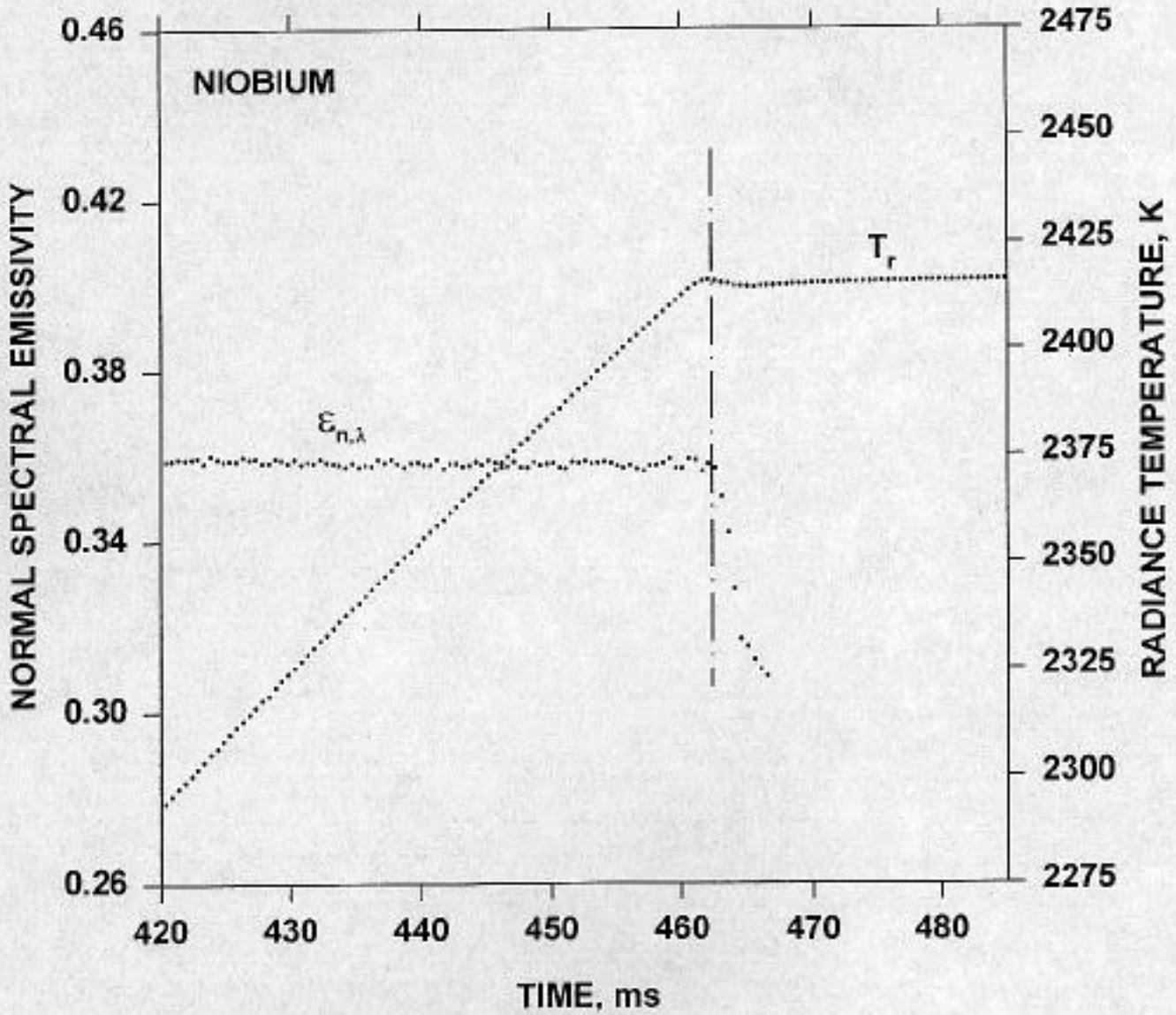
NIOBIUM
NORMAL SPECTRAL EMISSIVITY
0.46
0.42
0.38
0.34
0.30
0.26
RADIANCE TEMPERATURE, K
2475
2450
2425
2400
2375
2350
2325
2300
2275
T_r
$\varepsilon_{n,\lambda}$
420
430
440
450
460
470
480
TIME, ms

Figure 4

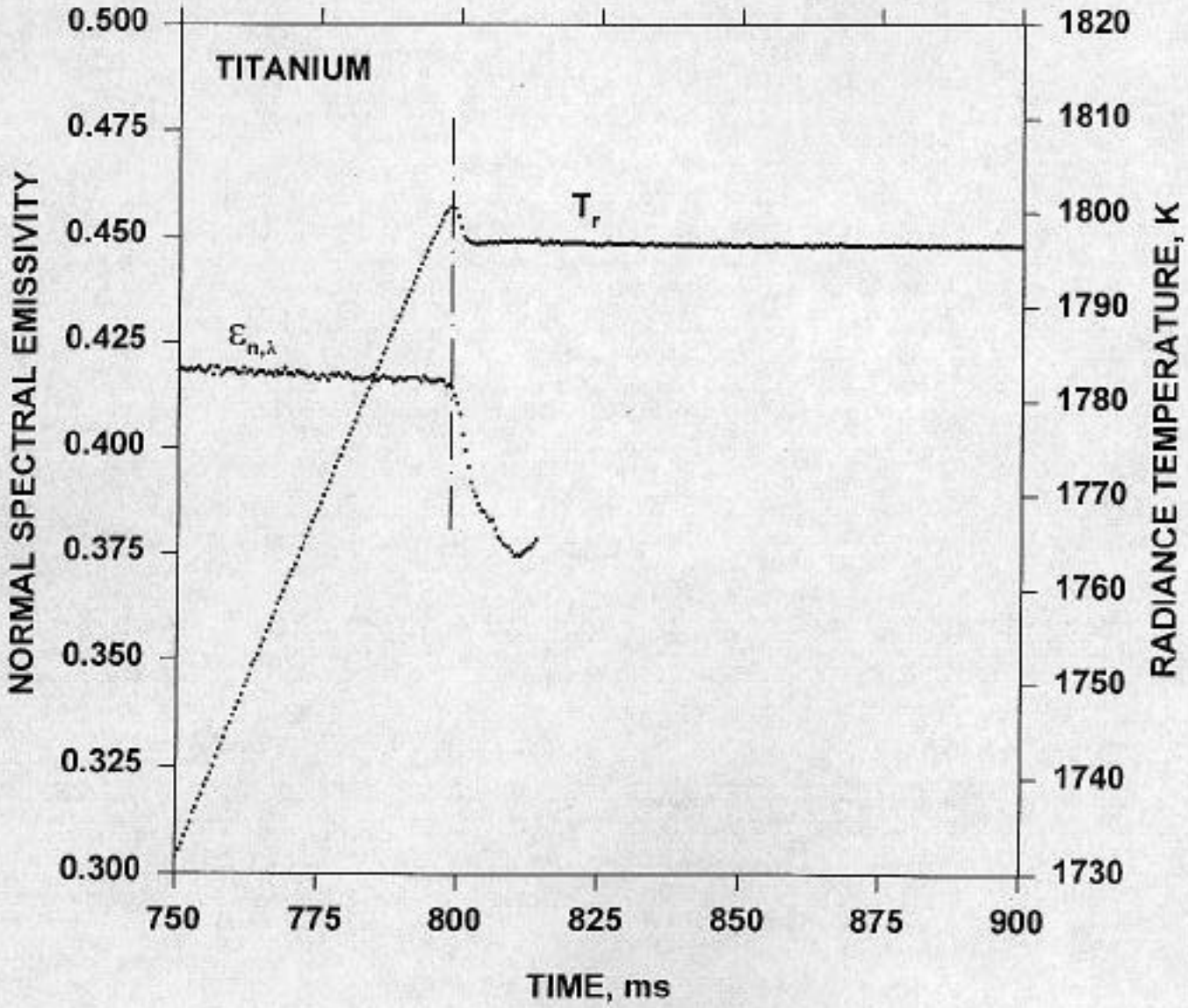
TITANIUM
NORMAL SPECTRAL EMISSIVITY
RADIANCE TEMPERATURE, K
TIME, ms
T_r
$\varepsilon_{n,\lambda}$
0.500
0.475
0.450
0.425
0.400
0.375
0.350
0.325
0.300
1820
1810
1800
1790
1780
1770
1760
1750
1740
1730
750
775
800
825
850
875
900

Figure 5

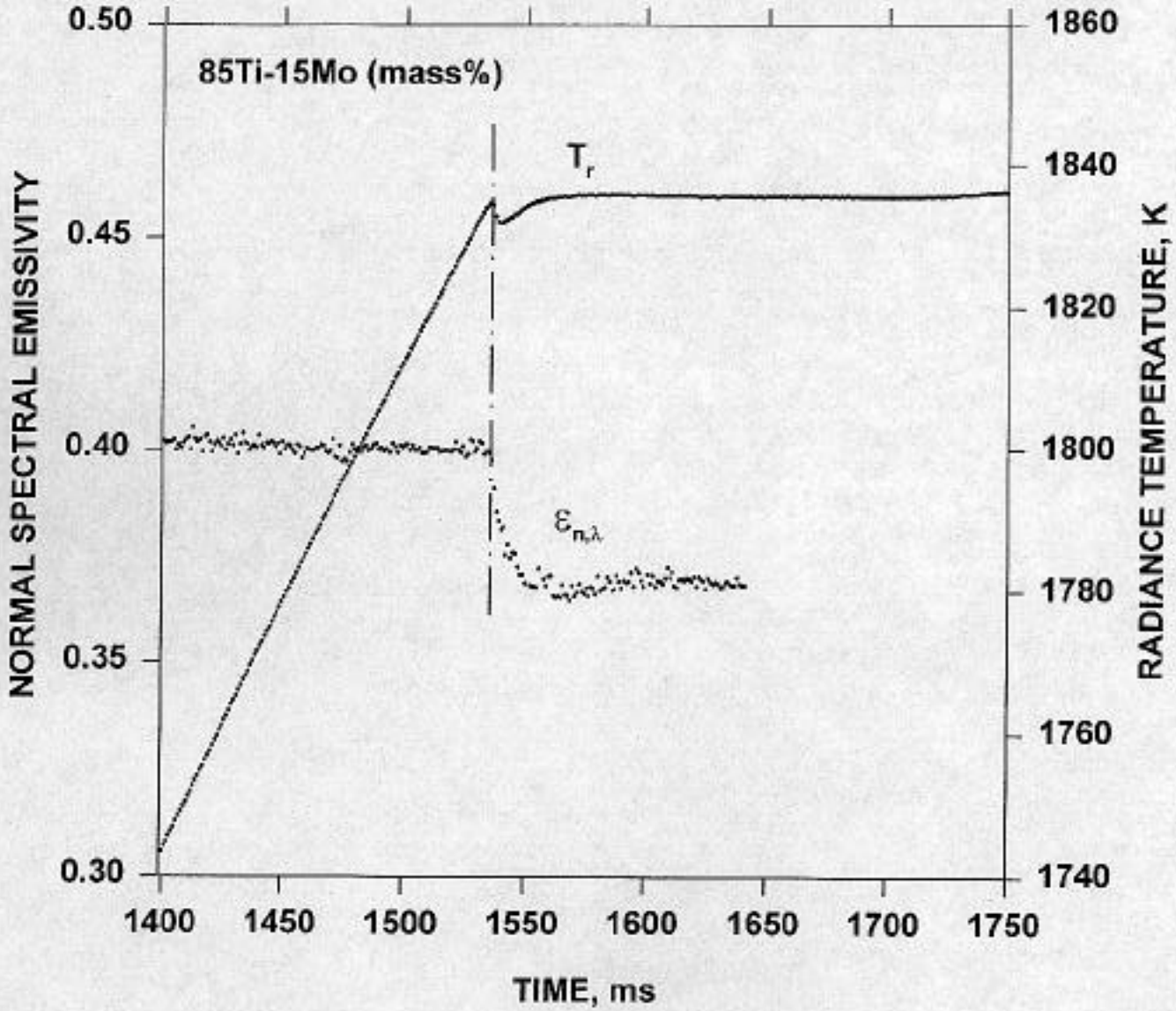
85Ti-15Mo (mass%)
T_r
$\varepsilon_{n,\lambda}$
NORMAL SPECTRAL EMISSIVITY
RADIANCE TEMPERATURE, K
TIME, ms
0.50
0.45
0.40
0.35
0.30
1860
1840
1820
1800
1780
1760
1740
1400
1450
1500
1550
1600
1650
1700
1750

Figure 6