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A New Technique for the Measurement of Radiance Temperature at the Melting Point[1]

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Measurements of the radiance temperature of metals at their melting point received wide attention recently on account of controversial results obtained in different laboratories. During further investigations at IMGC, a new technique for radiance temperature measurements at the melting point was perfected, with numerous experiments performed at the melting point of niobium. The new technique consists in bringing the material to the melting point and interrupting the flow of current just before the specimen is destroyed. Using this method the melting plateau may be repeated several times using the same specimen. Repeatibility studies may be performed, and changes in the surface structure of the material may be evaluated. Typical results of this new technique are presented, along with a complete description of this new measurement method.

KEY WORDS: high-speed pyrometry; high temperature; melting; pulse heating; radiance temperature.

1. INTRODUCTION

The original development of the pulse technique for the measurement of radiance temperature of metals at their melting point is due to Cezairliyan [1]. During a 20-year period, measurements on many metals at different wavelengths were performed as a joint research effort between the National Institute of Standards and Technology (NIST, USA) and the Istituto di Metrologia "G. Colonnetti" (IMGC, Italy). A comprehensive review of this

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joint effort has been presented recently [2], summarizing the experimental results obtained both at NIST and at IMGC and suggesting the use of radiance temperature and of normal spectral emissivity of selected metals at their melting point as possible reference values [2].

Recent controversial results on the normal spectral emissivity of metals at their melting point obtained by a major research laboratory [3] prompted us to reevaluate the measurement technique and to consider some variants of it. The aim of this paper is to describe a new measurement technique for the radiance temperature of metals at their melting point. The new technique consists in bringing the material to the melting point and interrupting the flow of current just before the specimen is destroyed. Using this method, the melting plateau may be repeated several times using the same specimen, performing repeatibility studies and studying the changes in the surface properties of the material.

2. NEW MEASUREMENT TECHNIQUE

The original technique for the measurement of radiance temperature of metals at their melting point [1, 2] consisted in bringing the specimen to its melting point with a current pulse of subsecond duration. The radiance temperature of the specimen surface was measured with a high-speed pyrometer with submillisecond time resolution. The specimen was destroyed each time and repeated experiments on the same material were performed using different specimens: the repeatibility thus included small differences among the specimens (due to surface conditions, differing cross sections, pyrometer alignment, etc.) and was in the range ± 1 K for most materials. Occasionally the recorded melting plateau was poor: this was attributable to different causes such as specimen contamination, problems with the clamping arrangement, movements during melting, etc. Since the specimen was destroyed at the end of the experiment, no specific evaluation of the problem was possible.

For the reassessment of the measuring technique, the experimental apparatus used at IMGC was partially modified as follows.

(a) A new current switch was installed permanently in the main power circuit. The switch is of the electromechanical type and is rated with a breaking capacity of more than 5000 A at 500 V. The main drawback of the new switch is that, on the open command, the coil releases a big burst of electromagnetic interference that disturbs the measured signals. Careful shielding has somewhat reduced, but not eliminated, this interference problem. The main advantage of the new switch is its very accurate timing in

interrupting both small and large currents, with reproducibility better than 0.01 s.

(b) The specimen was observed simultaneously with two high-speed pyrometers that were focused on targets with different areas situated on opposite faces of the strip specimen.

(c) The voltage output of each pyrometer was sent in parallel to separate channels of two different data acquisition systems.

Figure 1 shows a schematic arrangement of the experimental set-up and Table I presents the main technical characteristics of the high-speed pyrometers and of the data acquisition systems used for the experiments. Additional technical details on these items may be found in a recent review [6]. The use of two high-speed pyrometers operating at the same wavelength, but with different target areas and with the possibility of connection to data acquisition systems with different time resolutions, was intended to check both for time-dependent and for surface-dependent effects.

Using the new experimental arrangement, the specimen can be brought to the melting point with a current pulse of programmed duration, and the current can be interrupted while the specimen is melting but before its destruction. The good time reproducibility of the switch makes this process repeatable several times and many different measurements of the melting plateau of the same specimen are possible.

The new measurement technique was tested using niobium strip specimens with the following nominal dimensions: length, 40 mm; width,

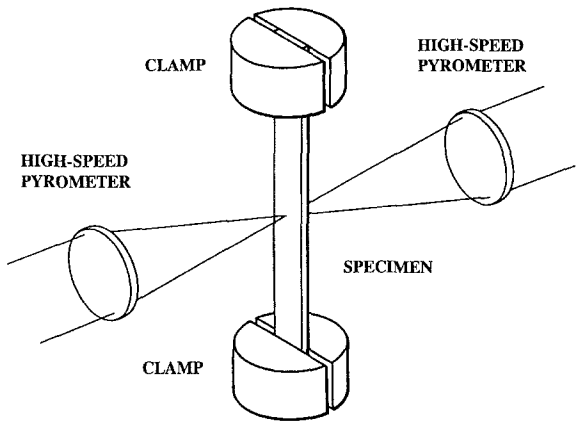

Fig. 1. Schematic representation of the experimental arrangement used to evaluate the new technique for the measurement of radiance temperature at the melting point.

Table I. Main Technical Characteristics of the High-Speed Pyrometers and of the Data Acquisition Systems

Pyrometer [Ref. No.]	Target area diameter (mm)	Nominal wavelength (nm)	Nominal bandwidth (nm)	Data acquisition system	Number of bits	Measurement time (μs)
Millisecond pyrometer [4]	0.3	900	82	Datel 149	14	900
Microsecond pyrometer [5]	0.8	900	82	Analogic 652	16	10

6.3 mm; and thickness, 0.13 and 0.5 mm. Both thin (0.13-mm) and thick (0.5-mm) specimens were used to check for any effect due to the amount of material being melted. The purity of all specimens was 99.9% or better and the thin material was obtained from two different sources.

The first melting of the niobium specimen causes permanent changes on the surface of the strip, as shown in Fig. 2. Figure 2a presents the history of radiance temperature versus time during the first melting. The normal spectral emissivity of the solid specimen is higher than that of the molten material as demonstrated by the temperature peak just before the melting plateau. The spike before melting is related to the roughness of the solid surface, as described in detail in Ref. 1. After the first melting, permanent changes in the surface structure of the specimen can be observed by visual

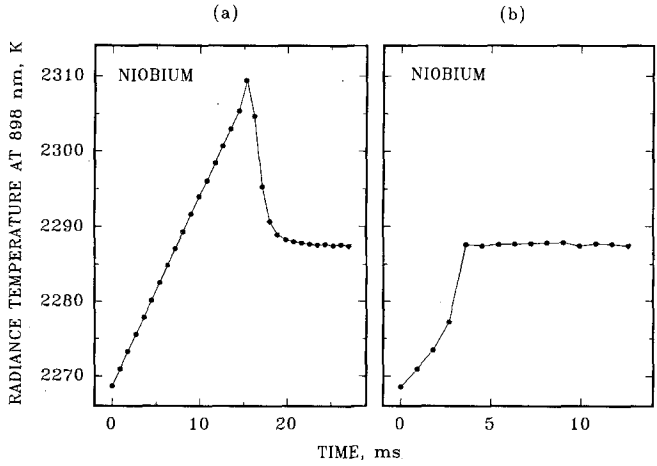

Fig. 2. Typical changes between the first melting (a) and the second melting (b) of a niobium specimen.

inspection: large grains appear and the surface is smooth and shiny, especially if observed with lateral illumination. The second melting (and all the following ones) show the behavior of Fig. 2b. The plateau occurs at the same temperature of the first melting, but no peak is present, with a sudden transition between the emissivity of the solid and that of the melting material, as indicated by the sharp change in slope in the first measurements in Fig. 2b. This peculiar phenomenon is shown in detail in Fig. 3. A sudden emissivity change is observed both heating and cooling, as shown by the very noticeable changes in slope occurring on the data (see the dashed line in Fig. 3). This reproducible phenomenon is most likely due to a structural change on the surface of the specimen, which occurs on the solid material a few kelvins below the melting point.

Repeated measurements at the melting point are possible both on the thin and on the thick specimens with identical results, but repeated thermal and mechanical stresses at very high temperatures cannot be substained indefinitely. Short melting plateaus prolong the life of a specimen, and thick specimens are definitely stronger and substain better the repeated melting-solidification cycles. Ten to fifteen melting plateaus have always been possible on the same specimen (both thin and thick ones); afterward the surface quality becomes poorer, and the melting plateau looses its flat shape. For experiments where the plateau quality was less important (heating rate studies), we were able to perform more than 40 meltings on the same sample both on thin and on thick specimens.

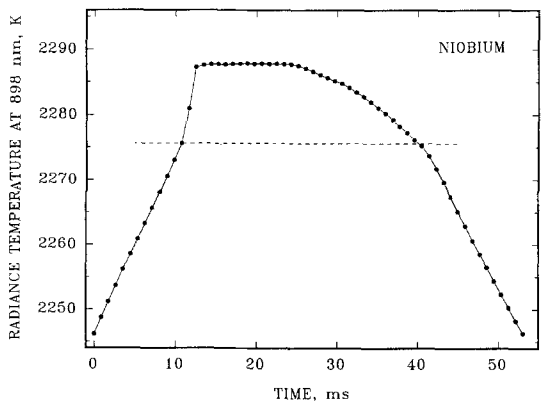

Fig. 3. Typical premelting and postmelting behavior of a niobium specimen. The dashed line indicates sudden changes that are noticeable both during heating and cooling. These are interpreted as surface structure (and emissivity) changes of the specimen, a few kelvins below the melting plateau.

3. RESULTS

Good melting plateaus of considerable duration are possible with this new technique: a typical example is shown in Fig. 4. In this experiment, measurements were taken with the microsecond resolution pyrometer, with data collected every 10 μs. For viewing purposes, only 1 of 20 data is plotted. The high-speed pyrometer noise band is clearly visible and is of the order of 0.1 K. The data gap on the plateau (at approximately 82 ms) indicates where spurious measurements due to electromagnetic interference from the switch coil were eliminated. From Fig. 4, it can be seen that the time of the mechanical opening of the switch is of the order of 20 ms.

Numerous experiments of the radiance temperature of niobium at its melting point checked for significant differences due to the following.

(a) The initial surface roughness of the specimen. None were found, even though this measurement technique permanently changes the surface conditions of the specimen after the first melting.

(b) The target area viewed by the high-speed pyrometers. Circular targets with diameters 0.3 and 0.8 mm gave similar results, except for cases when there were problems in the way the specimen melted (details explained later when discussing results as a function of heating rate).

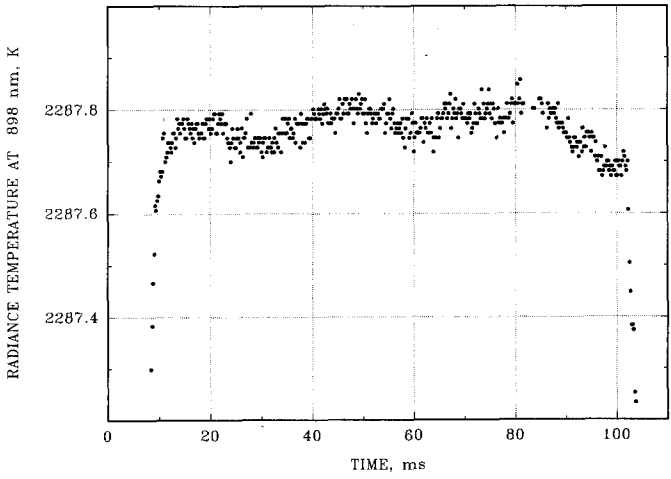

Fig. 4. Typical melting plateau of niobium measured with a microsecond resolution pyrometer. Data were collected every 10 μs; 1 temperature of 20 is plotted.

(c) Geometrical dimensions of the specimen (particularly thickness). The mechanical strength and larger mass of the thick specimens were an advantage after many melting plateaus; experimental results of the radiance temperature indicated no differences.

Measurements were performed both in vacuum (at approximately 10^{-3} Pa) and in an inert atmosphere (argon at approximately 10^5 Pa), with some significant differences occurring for slow experiments. The radiance temperature of three different niobium specimens as a function of heating rate is shown in Fig. 5. The data refer to two thin specimens, one measured in vacuum and the other one in argon, and to one thick specimen measured in vacuum. The radiance temperature at the melting point shows a very noticeable decrease with low heating rates (below $1000\ \mathrm{K \cdot s^{-1}}$) for vacuum experiments and a much lower dependence on heating rate for experiments in argon. For all experiments the heating rate is computed during the premelting phase at the same temperature (approximately 15 K below the melting plateau). The effect of window coating was checked during these experiments, but it was found to be negligible both in argon and in vacuum.

The main reason for the different behavior at low heating rates was traced to the way the specimen surface melts in vacuum or in argon. Figure 6 is a picture of two thin specimens that were taken to the melting point once using a slow heating rate. The upper specimen (melted in argon near atmospheric pressure) shows a small molten zone in the center of the specimen: if the high-speed pyrometer is focused on the melted area, it is clear that the result will be very similar to any other experiment (at a faster

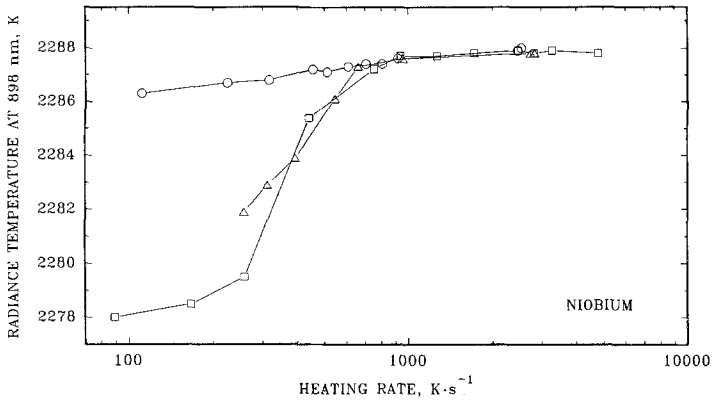

Fig. 5. Radiance temperature of niobium at its melting point as a function of heating rate for three specimens in different environments: ○, thin specimen in argon; □, thin specimen in vacuum; △, thick specimen in vacuum.

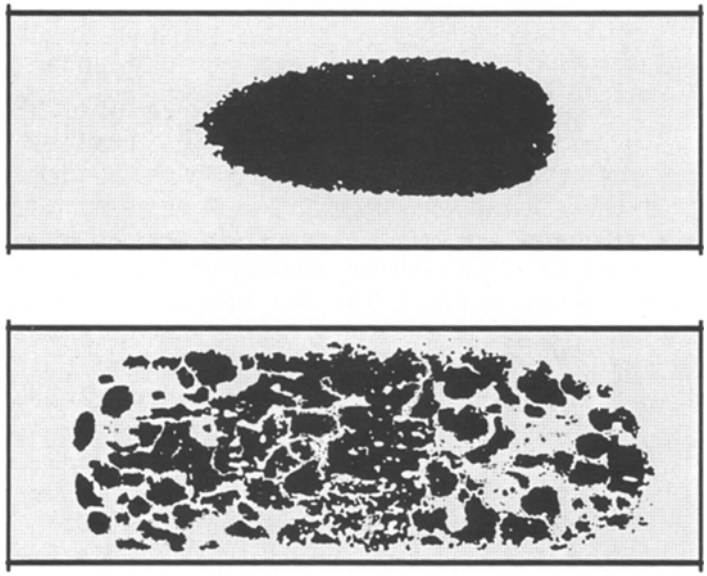

Fig. 6. Surface structure of two thin niobium specimens taken once to the melting point with a show heating rate: the upper specimen was melted in argon near atmospheric pressure; the lower specimen was melted in vacuum.

heating rate) when a larger portion of the specimen will be melted. A completely different behavior is shown by the lower specimen that was taken to the melting point in vacuum with a similar slow heating rate. A much larger area shows signs of melting, but the melting is incomplete, and patches of molten material are randomly mixed with patches of material that did not reach the melting point. The high-speed pyrometer output is an integral over the target area and the measured signal (and consequently the temperature) will be lower in this case because the radiation is collected from an area only partially molten.

When the input power is low (and consequently the heating rate is slow) the behavior of the surface of the specimen during melting is completely different in vacuum and in argon (see Fig. 6): indirect evidence of this is also shown in Fig. 7, which presents the shape of the melting plateau of two thin specimens in experiments with a heating rate around $300\ \mathrm{K} \cdot \mathrm{s}^{-1}$. Figure 7a refers to an experiment in argon and the sudden emissivity change from the solid to the molten material is clearly visible. Figure 7b refers to an experiment in vacuum, and in this case there is a smooth transition from the solid to the apparent melting, but the plateau is several kelvins lower because only parts of the surface have been melted, as shown in Fig. 6.

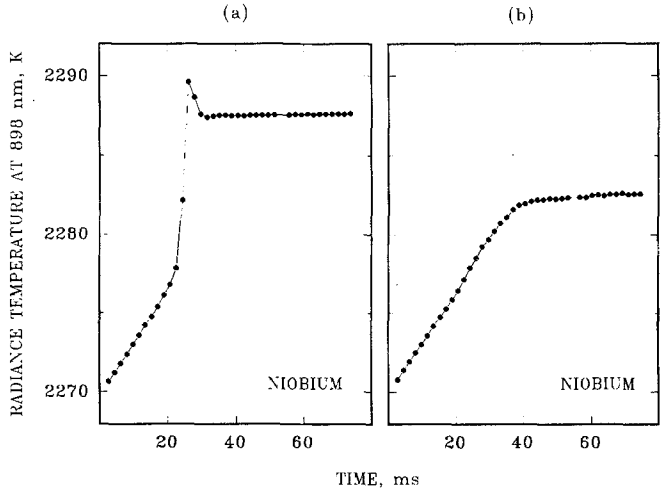

Fig. 7. Different behavior of two thin niobium specimens taken to the melting point with a slow heating rate: (a) experiment in argon; (b) experiment in vacuum.

4. CONCLUSIONS

The new technique for the measurement of the radiance temperature at the melting point has been applied to niobium. Technical details and experimental results are reported in another publication [7].

The applicability of this technique to conventional pyrometry was one of the original aims of this investigation. This is not possible unless the pyrometer can perform at least 100 measurements/s. As described previously, the slowing-down of the experiment (up to several seconds) creates problems related to the way in which the specimen surface undergoes melting. In the case of niobium, measurements in argon at slow heating rates (below 500 $K \cdot s^{-1}$) are still possible, while the same measurements in vacuum are not possible. The behavior of other metals when the melting point is reached with slow heating rates should be further investigated.

The new technique for the measurement of the radiance temperature at the melting point provides the following advantages:

- the possibility of repeated melting plateaus using the same specimen;
- measurements on a well-stabilized surface, established on the specimen after the first melting; and
- simplification of the measurement procedure and elimination of variations due to the use of different specimens.

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