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A Submicrosecond Pulse Heating System for the Investigation of Thermophysical Properties of Metals at High Temperatures[1]

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A submicrosecond ohmic pulse heating technique is described for measurements of thermal properties of cylindrical metallic samples at high temperatures. Electrical and optical measurements for determination of thermophysical data such as enthalpy, specific heat, and electrical resistivity are presented. Effects that can falsify the results are discussed.

KEY WORDS: electrical resistivity; enthalpy; exploding wire; high temperature; liquid metals; pulse method; thermal expansion.

1. INTRODUCTION

At present there exists an increasing demand for thermophysical data of metals. This demand is not limited to the solid state; the liquid phase of metals is also of interest. In addition, knowledge about metastable states of matter should be improved.

For thermophysical measurements in the solid state there are many high-precision methods available. Measurements in the liquid and gaseous phases at high temperatures are subject to enormous problems. In order to avoid such problems, several methods were applied [1–4]. Investigations of liquid metals were performed in different temperature regions. A classification of these experiments can be made according to their time and temperature resolution [5].

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Our experiment tries to solve the above-mentioned problems by using an exploding wire technique. Early experiments by Lebedev [4] showed that high heating rates can be obtained in this way. The restrictions which exist for static measurements are reduced, and a broad range of thermal properties can be determined. Our discharge circuit is very similar to that used by Seydel et al. [1]. Below is a description of our high-speed diagnostic system and an estimation of the possible experimental uncertainties.

2. EXPERIMENTAL

The experiment is based on a fast ohmic heating of cylindrical samples having different dimensions. Heating rates of slightly more than $10^9\ \mathrm{K \cdot s^{-1}}$ are achieved. The principal layout of the experimental arrangement is shown in Fig. 1.

The heating of the wire sample (4) is accomplished by discharging a 5.4-μF capacitor bank (1) through it. Usually this bank is charged to a voltage between 4 and 15 kV and switched by a three-electrode spark gap (3). Another three-electrode gap (3′) can be used as a crowbar in order to stop heating after a certain time interval and to get a defined energy input into the sample.

The short circuit ringing period can be varied between 6 and 7 μs. The inductance of the coaxially built circuit is about 180 nH, and the resistance

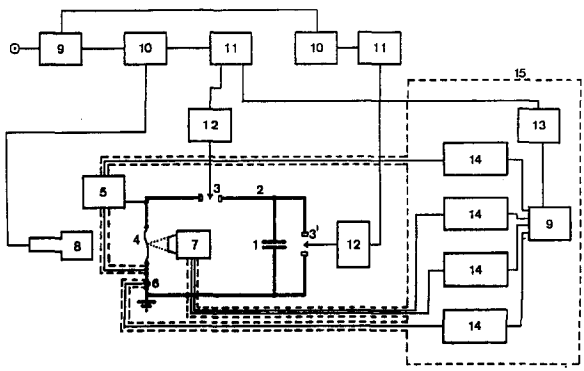

Fig. 1. Block diagram of electrical and optical arrangement of the measurement apparatus. 1, Capacitor bank; 2, discharge circuit; 3 and 3′, spark gaps; 4, wire sample; 5, voltage divider;6, induction coil; 7, pyrometer; 8, flashlight; 9, start pulse; 10, delay; 11, trigger; 12, thyratron stage; 13, photodetector; 14, 200-MHz oscilloscopes; 15, shielded measuring room.

about 23 mΩ. The short rise times of the discharge currents are responsible for strong electromagnetic fields, which can influence the diagnostics. For this reason a good shielding is required. All oscilloscopes (14) for the recording of signals are placed in a shielded cabin (15). The measurement lines consist of coaxial wires placed in copper tubes between the cabin and the apparatus. The entire system is grounded at one point only. The start of the discharge is initiated by a pulse generator (9). This signal reaches the grid of a thyratron stage (12), which after a certain delay in 10, gives a sharp pulse to the three-electrode spark gap. In a similar way, the back light for the expansion measurements is switched on. All oscilloscopes in the cabin are triggered simultaneously.

The specimens are clamped by two electrodes in a vessel filled with water in order to suppress electric surface discharges. Such discharges would falsify the results. For experiments up to pressures of 5000 bars, the vessel shown in Fig. 2 is used.

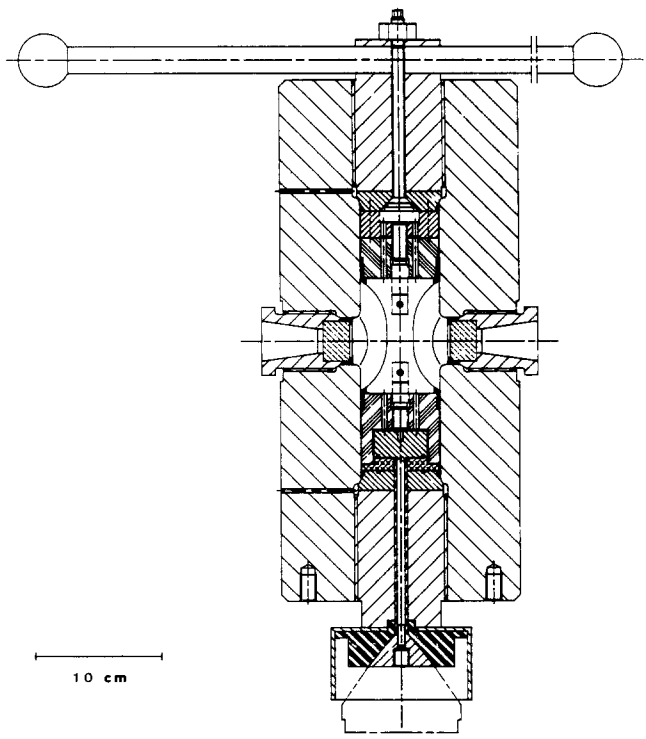

Fig. 2. High-pressure cell for measurements at up to 5000 bars in water (VEW Kapfenberg, Austria).

For the description of the diagnostics it is useful to discuss electrical and optical setups separately. The former includes the devices for the determination of current and voltage. Optical diagnostic elements are used to measure thermal expansion and temperature.

The measurement of current is performed by a small pickup coil, which is mounted inside of the coaxially built transmission line. The dI/dt signal is integrated by a RC circuit (time constant, 72.6 μs).

The voltage across the sample is measured by a resistive voltage divider developed by Fucke [6]. Inductive parts of the voltage signals are compensated by a numerical method [3].

With the data of current and voltage, the energy input and the specific enthalpy $H[H = H(t) - H_{298K}]$ can be calculated:

$$H = \frac{1}{m} \int_0^t I(t)\, U(t)\, dt \tag{1}$$

where I is the current, U is the voltage, t is the time, and m is the specimen mass. The ohmic resistivity ρ_0 is calculated by

$$\rho_0(t) = \frac{U(t)}{I(t)} \frac{\pi r^2}{l} \tag{2}$$

where r is the specimen radius and l is the specimen length. If the volume expansion is to be considered, one has to correct the resistivity value with the volume ratio V/V_0,

$$\rho(t) = \rho_0(t)\, V/V_0 \tag{3}$$

Usually, the determination of thermal expansion in fast pulse experiments is performed by optical streak or X-ray pulse pictures. Figure 3 demonstrates the basic idea used in our investigation. The sample (5) is illuminated by a flashlight (1) (with constant intensity during a time interval of about 200 μs). The edge of the sample is enlarged and focused onto the edge of a square slit-diaphragm (9). While the sample is expanding, a decrease in the intensity which reaches the detector occurs. The radiation of this light source is collected by an optical fiber (8) and led to the shielded detector. A characteristic signal of the decreasing light intensity during the expansion of a niobium sample is presented in Fig. 4a.

For this optical arrangement the kind of light source chosen is somewhat problematic, because intensities are usually too low to insert narrow band filters in a wavelength region where radiation of the sample itself would not affect the expansion measurement. Especially if the sample reaches higher temperatures, its radiation can falsify the intensity of the

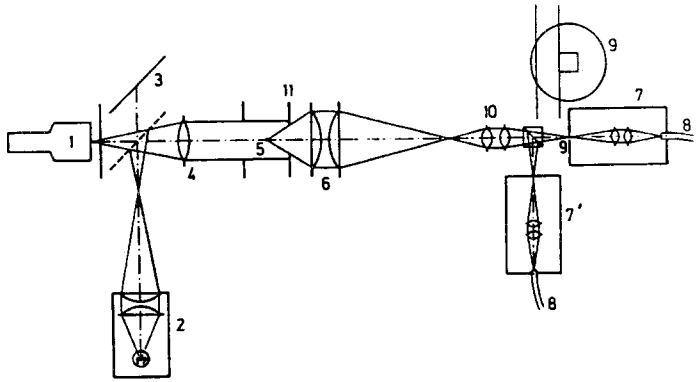

Fig. 3. Experimental arrangement for thermal expansion measurement. 1, Flashlight; 2, light for adjustment; 3, mirror; 5, wire; 7 and 7', detector; 8, fiber optics; 9, squared diaphragm with image of the enlarged wire; 11, diaphragm; 4, 6, and 10, lenses.

flashlight. Therefore, a correction of the measurements by signals without flashlight is necessary (Fig. 4b).

The resolution of the present system depends upon the chosen optical amplification (usually about 1:40) and is approximately 0.001 mm. However, such a resolution could be reached only in temperature regions where little self-radiation exists.

To obtain expansion data from measured signals, a calibration is necessary. For this purpose the slit-diaphragm is shifted toward the edge of the sample shadow with the help of a micrometer screw, diminishing the "free" area of the slit in a defined way. Using this method one can obtain a relationship between an increasing diameter and a decreasing light inten-

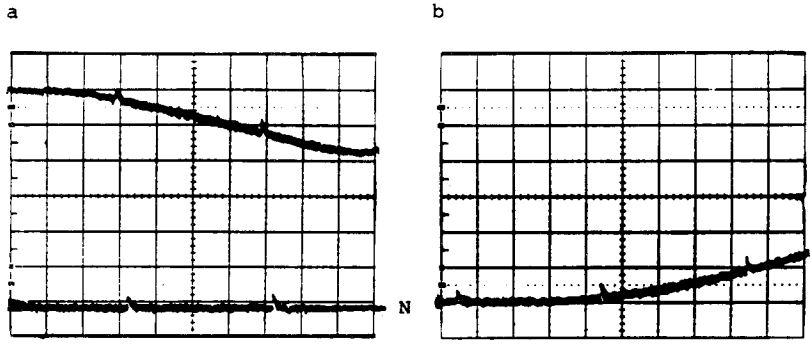

Fig. 4. (a) Signal of decreasing light intensity during expansion (N is the zero line). (b) Signal of wire radiation without flashlight. 0.5 μs per division (time mark distance, 2 μs).

sity. It was proved with different methods (e.g., with streak and pulse pictures) that no measurable sideways motion and no deviation from a cylindrical geometry take place during the time interval of interest. Figure 5 shows an example for an expansion measurement on niobium presented in Ref. 7.

Much importance has to be attached to an accurate determination of surface temperature. Two principal methods were applied, measurements at one and at two wavelengths. Usually, both methods depend upon the knowledge of spectral emissivity and its variation with temperature.

The pyrometer presented in Fig. 6 was used to measure the brightness temperature of the sample. A lens (3) produces a magnified image of the wire at a slit (4). Only a central part of the wire is viewed by the pyrometer. For calibration, the radiation from a tungsten strip lamp can be used. The radiation is divided by a beam splitter and the selection of wavelengths is done by using two interference filters (5). Optical fibers and photodiodes with a maximum spectral sensitivity near 850 nm were inserted for the detection of radiation. The temperature range to be measured lies between 2000 and 9000 K, and therefore the use of logarithmic amplifiers would be favorable. However, the short rise times did not allow the use of such amplifiers.

Temperature measurements were performed using the known melting

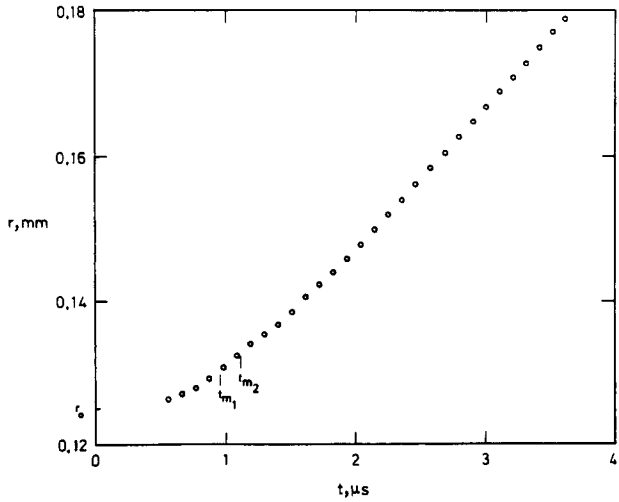

Fig. 5. Radius of wire versus time of a 0.25-mm-diameter niobium sample. The locations marked by t_{m_1} and t_{m_2} indicate the beginning and ending of the melting transition.

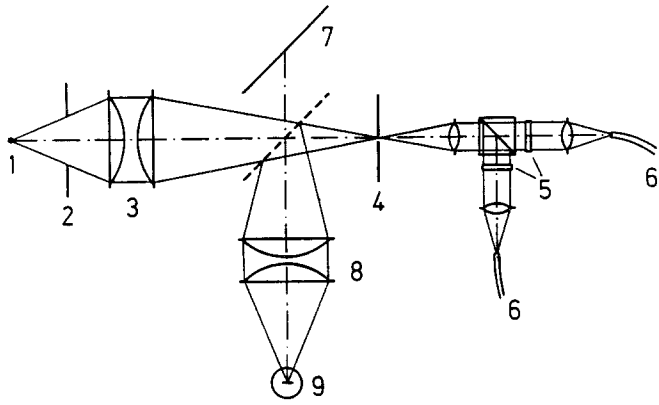

Fig. 6. The pyrometer. 1, Wire sample; 2, diaphragm; 4, pyrometer slit; 5, interference filters (700, 800 nm); 6, fiber optics; 7, mirror; 9, tungsten strip lamp; 3 and 8, lenses.

temperature of the specimen as a calibration point. The temperature was determined by the equation

$$T = c \Big/ \lambda \ln \left\{ 1 + \frac{\varepsilon(T)}{\varepsilon(T_m)} \frac{L_m}{L} [\exp(c/\lambda T_m) - 1] \right\} \tag{4}$$

where T is the temperature, λ is the wavelength, ε is the spectral emissivity, T_m is the melting temperature, L is the measured intensity, and c is the second radiation constant. For all temperature regions higher than the melting point, $\varepsilon(T)$ was assumed to be constant because of the lack of available data $[\varepsilon(T)/\varepsilon(T_m) = 1]$.

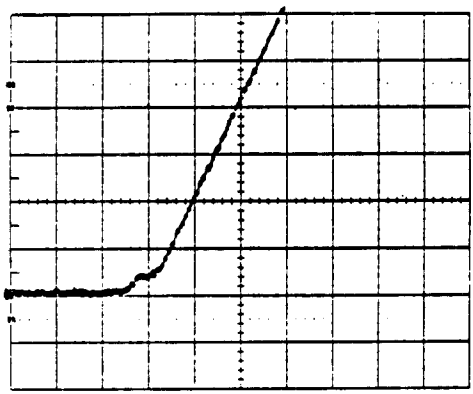

Fig. 7. A typical temperature signal. Horizontal scale, 0.5 μs per division. Vertical scale, arbitrary units.

After an accurate test of the time and temperature resolution of the pyrometer described was made, a rise time of less than 10 ns and a minimum measurable temperature of about 2200 K were found. A typical temperature signal is shown in Fig. 7.

3. ERROR ANALYSIS

An extensive error analysis for the described experiment cannot be done, because of the large number of parameters that have influence on the measurements. However, much attention was paid to a detailed condideration of the principal errors.

The main assumption for the determination of enthalpy by measuring current and voltage is the constant pressure condition. For this reason, a detailed study was done to calculate the surrounding pressure of the wire with the help of the "Charakteristiken-Verfahren", proven by measurements of the velocity of the melting shock wave [8]. The pressure varies greatly in the solid state of the metal, reaching a peak value at the melting transition. In the liquid region it is found to be nearly constant. In addition, these calculations show that the values of pressure are too low (range, up to 300 bars) to have much effect on measured data.

As the rise time of the discharge current is relatively low, the homogeneity of temperature could be affected by the skin effect. Calculations [8, 9] showed that for the heating rates used in our experiment, such effects decrease after a few hundred nanoseconds. As the measurements of expansion and temperature start after that time interval, these influences can be neglected. An analogous result could be found regarding the energy losses by heat transfer.

On the occurence of MHD instabilities in fast pulse-heated wires, several investigations and estimations have been made up to now [1, 8, 10, 11]. The results indicate that deviations from cylindrical geometry in the wire cannot be detected earlier than about 4 μs after the start of the heating, when conditions are chosen similar to those used here. Our measurements are finished within this time interval, and so this effect can be neglected too.

The error in our expansion measurements is approximately 10% in the vicinity of the melting point. For regions where the influence of self-radiation of the sample becomes stronger, the uncertainty obviously would increase. However, expansion data of several metals investigated so far are in good agreement with those of other authors.

A maximum error of 4% for resistivity and nearly 5% for enthalpy is assumed. Calculations indicate that temperature measurements using the

one-wavelength pyrometer yield errors of nearly 5% in the low-temperature regions, which increase to about 10% at higher temperatures.

A set of data for liquid rhenium and a flow chart showing how they were obtained are presented in the following paper [12].

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