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METHOD OF DETERMINING THE ELECTRICAL RESISTIVITY AT HIGH PRESSURES AND TEMPERATURES

D. B. Chernov and I. Ya. Shinyaev

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A method is described for measuring the electrical resistivity in solid-phase high-pressure installations. The high sensitivity of the method is ensured by using a heating current to determine the measurement of the electrical resistivity of the sample. The method is suitable for the investigation of phase transformations in metallic and semiconductor materials.

In investigating phase transformations in metals extensive use has been made of the electrical-resistivity method. Under conditions of high pressures and temperatures this method is used principally for the investigation of phase transformations in pure metals and compounds [1-3].

A scheme is proposed for measuring the electrical resistivity in a solid-phase installation, which allows temperature gradients in the sample to be minimized. The circuit is fairly simple to construct and ensures high measurement sensitivity. The positioning of the sample in the high-pressure chamber and the method of attaching the potential input leads and the thermocouple input leads to the sample are shown in Fig. 1a. The sample is placed between two cylindrical graphite inserts which play the role of a heater. An alternating or direct current, which is used to heat the sample and measure the electrical resistivity, is passed through the inserts and the sample.

Potential input leads for taking off the voltage drop ΔU across the sample which developed as a result of the heating current are attached to the ends of the sample. In order to determine the electrical resistivity it is necessary to measure the magnitude of the heating current and ΔU across the sample at each temperature. Since the resistance of the graphite inserts is considerably higher than the resistance of the metal samples, the heat is released predominantly in the inserts. Under these conditions the thermal flux is directed from the inserts to the sample, and this ensures the minimal temperature gradient in comparison with other heating schemes.

In investigating materials which react with carbon a washer made of a refractory metal (for example, Ta, Nb, or W) 0.1 to 0.5 mm thick is placed between the current leads and the sample. The thickness of the washer is chosen to be such that the carbon does not diffuse from the graphite into the sample during the experiment.

The temperature of the sample is measured by a Chromel–Alumel or tungsten–rhenium thermocouple. When the material of the thermocouple does not interact with the investigated sample, no special protection is required for the thermocouple. In those cases when such interaction is possible, especially when a Chromel–Alumel thermocouple is used, the thermocouple is protected by Al_2O_3 or BeO ceramic. Under these conditions the thermocouple is placed inside the sample for reliable temperature measurement.

The schematic of the unit is shown in Fig. 1b. The network consists of three circuits: 1) the heating circuit which contains the heating and cooling rate controller, a step-down transformer from which a low-voltage current is applied to the chamber dees, and a meter for recording the current; 2) the circuit for measuring ΔU, which contains an isolating transformer, a rectifier, a filter, and a two-coordinate PDS-021 potentiometer; 3) the thermocouple circuit whose emf is likewise applied to the PDS-021 potentiometer. In order to eliminate stray ac induction effects a filter consisting of a ~0.2 H inductance of two electrolytic capacitors of 100 μF each is connected at the input of the potentiometer in the thermocouple circuit. For a filter resistance of up to 10 Ω the filter introduces small errors into the measurement of the thermocouple emf, and these errors are easy to consider. In the case

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