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THERMOPHYSICAL DEVICES FOR HIGH TEMPERATURE MEASUREMENTS

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Abstract

A complex of devices is described which consists of two thermal analysers (their working temperature ranges are 300–2500 K and 1000–3000 K) and a scanning calorimeter (its working temperature ranges from 300 to 2000 K) which are controlled by an automatic system based on IBM PC AT with the ADDA-14 card.

Keywords: high-temperature devices, thermophysical properties

Introduction

For a long time we participate in a competition for the development of world-top high-temperature methods of physico-chemical analysis of refractory materials [1–12].

Now we produce a complex of devices which includes two thermal analysers with upper working temperature limits being 2500 K and 3000 K for TA-2500 and TA-3000 thermal analysers, respectively, and a scanning calorimeter (up to 2000 K), all of them are controlled by an automatic system based on IBM PC AT.

The complex permits us to investigate various refractory materials, such as metals, alloys, ceramics, minerals, composites, etc. in solid (powdered) and liquid states. The heating (cooling) is provided in an inert gas (argon, helium), the vacuum before gas inlet is $6.7 \cdot 10^{-3}$ Pa, the probe dimensions (diameter and height) are up to 3.5 mm. As probe-holder (crucible) materials oxides (HfO_2, CaO, MgO, ZrO_2, Al_2O_3) or metals (tungsten, molybdenum, tantalum) are used. Temperature scale is calibrated according to critical points of standard materials.

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Results and discussion

TA-2500 apparatus

The lower limit of the operating temperatures of this apparatus is about 300 K. The W/W+20% Re thermocouple is used as a sensor. The thermocouple construction without any ceramic insulator in a high-temperature zone [3] allows to reach the highest operating temperatures which are featured for the thermoelectric sensors. Up to 2500 K the device operates regularly (up to about 100 heatings if the compatibility of probe and crucible materials at operating temperatures is provided). With temperature rise its service life decreases and at 3000 K is limited to 1–2 heatings. The heating (cooling) rate is 10–200 deg·min^{-1}.

TA-3000 apparatus

This apparatus uses photodiodes as sensors. The device according to [9] ensures constancy of the emmissivity factor when the probe (or its state) is changed.

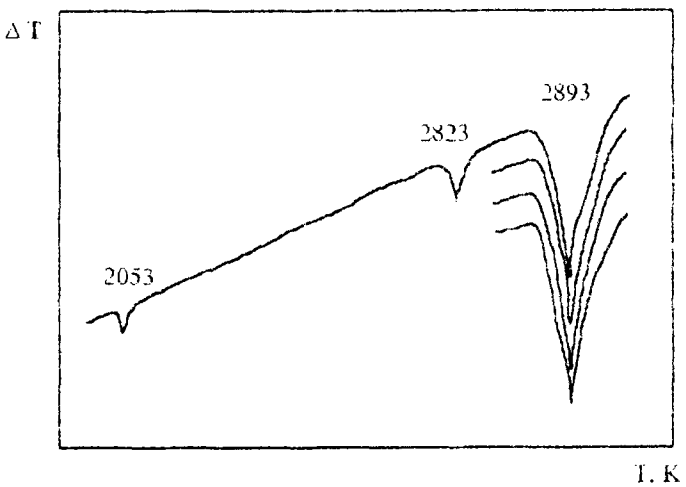

Fig. 1 Thermoanalytical curve of a Mo-melting

The operating temperatures range from 1000 to 3000 K. The upper limit is determined by the crucible material (HfO_2) melting point, and can be increased by using ThO_2-crucibles. An example of a Mo-thermoanalytical curves are shown in Fig. 1 (crucible HfO_2).

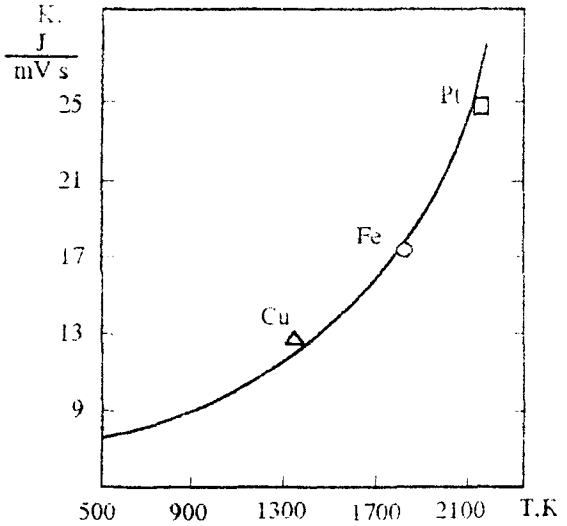

Fig. 2 Temperature dependence of calorimeter proportionality, factor *K*

Scanning calorimeter DSC-2000

The instrument is designed to measure specific heat capacity, temperature range and enthalpy of phase transformations and chemical reactions in solid and liquids in the temperature range 300–2000 K.

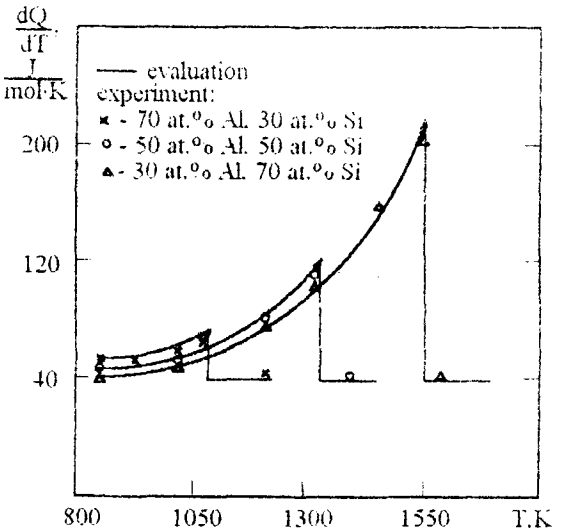

Fig. 3 Temperature distribution of melting enthalpy of Al–Si alloys

The system combines two instruments:

1) a diathermal calorimeter [10] which determines the difference between heat capacities of a specimen and of a primary standard from the difference between their temperatures in heating at a given rate;

2) a calorimeter of a constant heat flow (like one of [14]) for measurement of enthalpy changes during transformation.

Heating control is carried out to a given program: either at a given rate or when maintaining a given temperature difference between a specimen being heated and heating medium (a block).

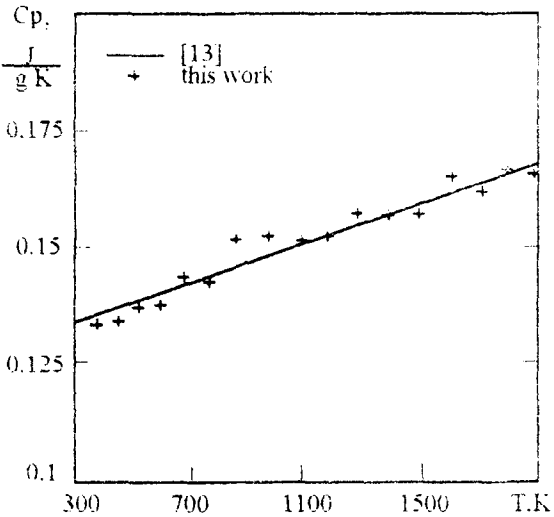

Fig. 4 Temperature dependence of specific heat capacity of tungsten

Recording of values being measured is accomplished by a recorder and in computer memory. Automatic system is based on IBM PC AT with the ADDA-14 card and provides control of experiments, reading and recording of experimental data, calculation and display of results.

Table 1 The main technical characteristics of the instruments

Characteristics	TA-2500	TA-3000	DSC-2000
Temp. measurement error / %	1	1.5	0.5
Temperature sensors	Thermocouple	Photodiode	Thermocouple
Temperature sensor type	W/W+20% Re		W+5%Re/W+20%Re
Heating rate / deg·min^{-1}	10–200	10–480	10–50
Time of an experiment / min	100–300	100–300	100–600
Power consumption / kW	2.5	3.0	2.0

Figure 2 shows the temperature dependence of proportionality factor, K, between the heat flow and temperature difference. The continuous curve was obtained using a Mo-standard specimen of thermophysical properties, the points were obtained from experiments on melting of Cu, Fe and Pt. The last point was obtained at 2043 K.

Figure 3 shows temperature distribution of melting enthalpy of Al–Si alloys with Al-content 70%, 50% and 30%.

Figure 4 shows temperature dependence of heat capacity of tungsten obtained using Al_2O_3-standard specimen of thermodynamic properties in the temperature range 1000–2000 K.

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Zusammenfassung — Es wird ein Gerätekomplex, bestehend aus zwei Thermoanalysatoren (Arbeitstemperaturbereich 300–2500 K und 1000–3000 K) und einem Scanning-Kalorimeter (Arbeitstemperaturbereich 300–2000 K), beschrieben, der von einer Automatik auf der Grundlage eines IBM PC AT mit einer ADDA-14-Karte kontrolliert wird.