TECHNICAL NOTE

THE STUDY OF THERMODYNAMIC SURFACE AND CONTACT CHARACTERISTICS BY A NEW METHOD OF SIMULTANEOUS THERMOOPTICAL ANALYSIS

V. M. Perevertailo, A.V . Zolotukhin, A. V. Gorbach, O. B. Loginova and L. Y. Ostrovskaya

V. N. Bakul Institute for Superhard Materials of the Ukrainian National Academy of Sciences
2, Avtozavodskaya St., Kiev, 254074, Ukraine

Abstract A device is described for studying thermodynamic and surface characteristics of metals as well as for differential thermal analysis of materials. A promise its shown is application for a multiple examination of high-temperature capillarity in a number of technological processes involving the formation or contribution of a liquid phase.

Key Words Thermodynamic, Surface, Metal, DTA, Thermooptical, High-Temperature Capillary

چکیده وسیله ای برای بررسی ترمودینامیکی، خواص سطحی و تجزیه تفاوت حرارتی DTA فلزات تشریح شده است. کاربرد موفقیت آمیز وسیله در آزمایشهای مکرر موئینگی در دمای زیاد و تعدادی از فرآیندهای صنعتی مستلزم فاز مایع به اثبات رسیده است.

INTRODUCTION

Data on adhesion, wettability, contact melting and interaction at the melt-solid interface are of great importance for the further progresses in physic-chemistry of surface phenomena and practical needs, especially, for the development of processes occuring with the involvement of a contact between a solid and a liquid phases. We have developed, manufactured and promoted into operation a device for studying thermodynamic and surface characteristics of melts as well as for differential thermal analysis of materials. The device hardware and software which include some verified design features, have been described elswhere [1-5].

GENERAL DESCRIPTION

Functionally, the device consists of a working vacuum chamber, systems for automation, visualization, heating, temperature control and registration as of a vacuum and a water cooling systems.

The main technical specifications of the device are as follows: working temperature range is from 320 to 2320K; height and width of the furnace space observed were 16 and 20 mm, respectively; a crucible diameter of 20 mm; the chamber inner diameter of 160 mm; the number of alloying component samples being charged is 30; the number of operation positions of a rotation table is 8; temperature sensors are thermocouples of W/W-Re20, Mo/W-Re20 types; the pressure in the chamber makes 6.7×10^{-3} Pa; the

working medium is a vacuum, Ar, or He; the power input is 3 kWA; the circuit voltage is 380/220 V.

The device is available in two versions. When made without a professional personal computer (PPC), the visualization system incorporates an illuminator, a vacuum chamber with a specimen, an Electronika A-50 video camera with a Jupiter-8 objective and a telemonitor (Figure 1a). Heating and cooling curves obtained with the help of a block of electronics, when performing DTA, are recorded by an N-307 self-recording two-coordinated device.

A version incorporating a PPC of AT/386 type (Figure 1b) with an AVER-2000 interface plate for digitizing a videoimage and an ADDA-14 plate for analog-digital conversion of a signal from temperature transmitters is of a higher grade. In this case the display of PPC will show an image from the videocamera or DTA curves; the computer takes over

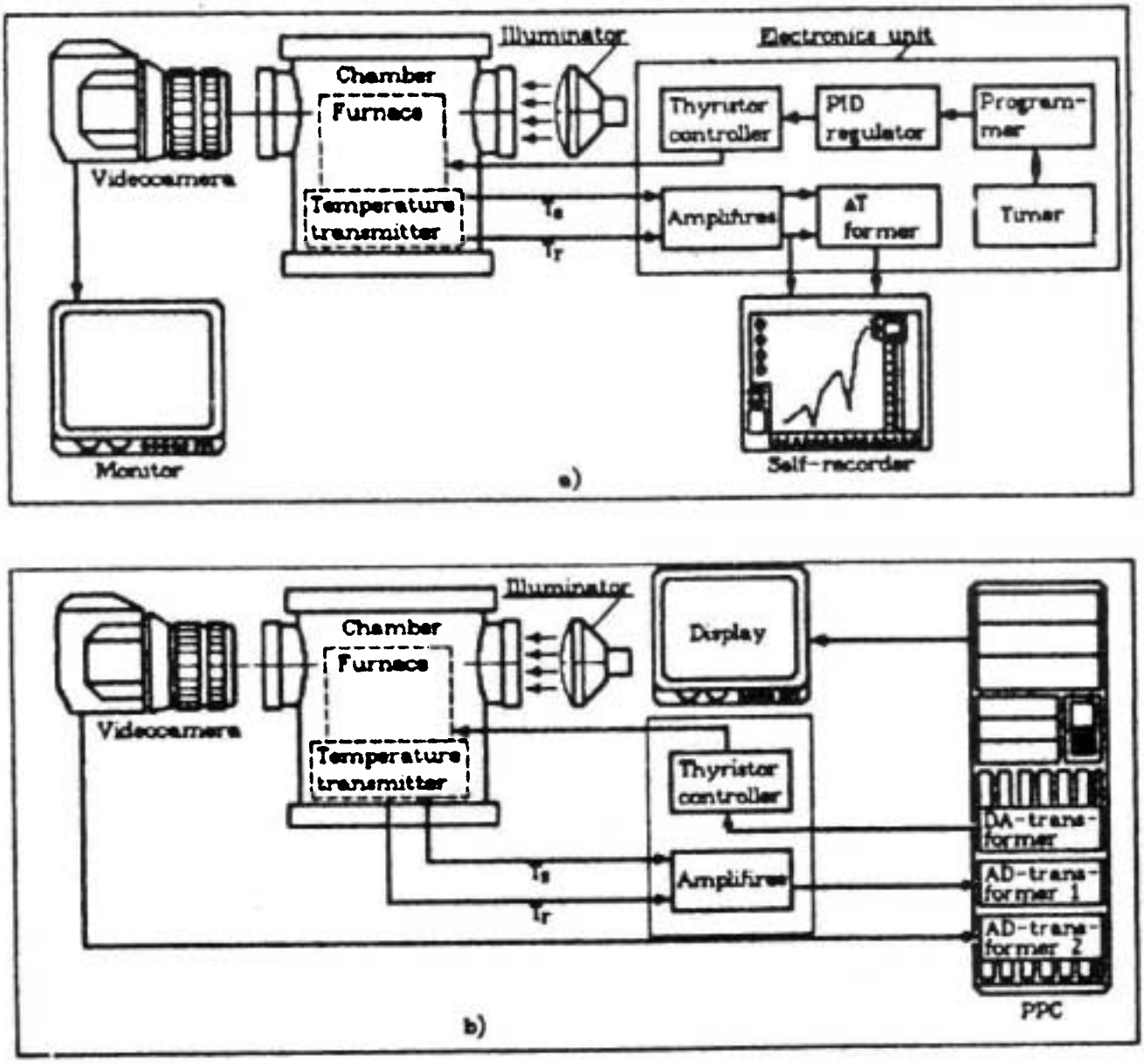

Figure 1. Structural schemes of the device without **(a)** and with **(b)** PPC.

the function of the electronics block too. The obtained image is recorded and kept in the computer memory and is processed mathematically using an appropriate soflware. Thus, the recording system permits a solid copy of the test results to be obtained.

A water-cooled working vacuum chamber is one of the main units of the device (Figure 2). It comprises a vacuum furnace having a heater and heat shields, vertical and horizontal sight holes, and an optical system for measuring parameters of a melt drop. The main unit is provided with an additional chamber, which is placed above the working one and separated from it by a water-cooled partition, the additional upper chamber has a cassette with samples of an

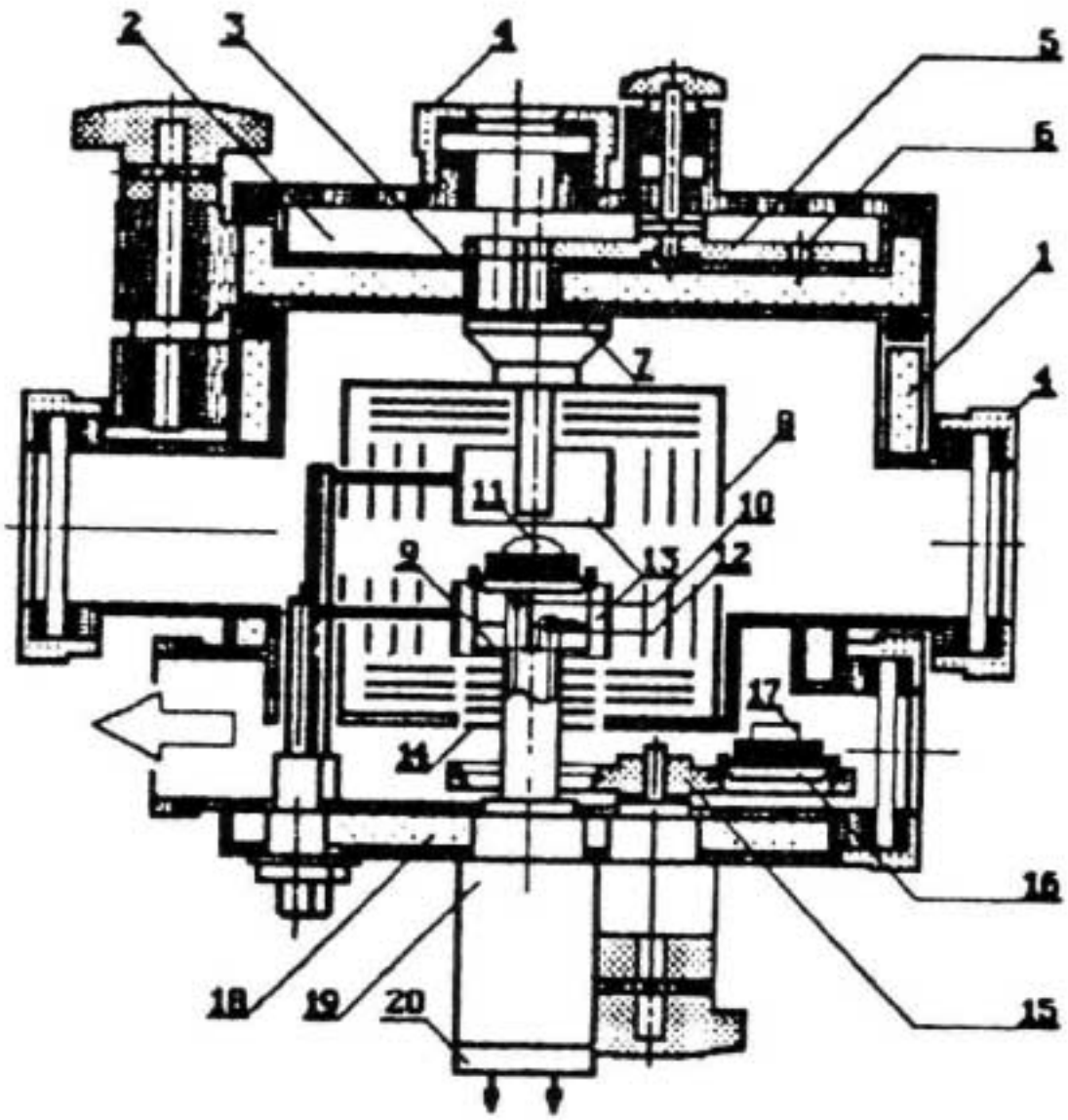

Figure 2. A Schematic Diagram of a Vacuum Chamber: 1-a working vacuum chamber, 2- an additional chamber, 3-a partition, 4- Vertical and horizontal sight holes, 5-a cassette, 6- a sample of an alloying component, 7-a hopper, 8- heat shields, 9- a platform for a crucible with a sample, 10, 12- thermocouples, 11,17 - the material under study, 13-a heater, 14-a plug unit, 15-a rotation table, 16-a crucible, 18- a bottom of the working chamber, 19-a feerder, 20- a joint.

alloying component used to change the concentration of the melt under study. Under the vacuum furnace, a rotation table is placed which ensures the setting of a crucible with a material under study into a loading position. On the flange of a water-cooled bottom of the working chamber, a feed unit is placed. This feed unit is used to take the crucible from the table, feed it into a working zone and return it back on the rotation table. The platform, on which a crucible with a sample is arranged, is a thermostat. It is fixed on a rod of the feed unit and is thermally insulated from it. Thermocouples are placed at the thermostat upper and lower planes to take the temperature of the sample (T_s) and that of a reference material (T_r), respectively. The electrodes of the thermocuples are brought out to a connector via the inner cavity of the feed unit rod. On the heat shields of the vacuum furnace, a hopper is placed to serve as a path for the alloying component to reach the crucible with the melt therein.

The chamber is provided with an additional block in order to measure a wetting angle for solid surfaces being wetted with melts by the "meniscus" method and to heat separately the solid and the liquid phases under study and to bring them into contact at the temperature of the experiment. The above mentioned block consists of a metal plug, which is placed instead of a glass of a vertical sight unit. A vertical rod with a fixed substrate of a solid phase under study goes through the metal plug. By moving the rod, the substrate is brought into contact with a melt within a working zone of the furnace.

The vacuum chamber is evacuated down to 6.7×10^{-3} Pa using a preevacustion and an oil-diffusion pumps. The device is equipped with a system for an inert gas inlet and a water-cooled trap of the oil-diffusion pump.

It is known that DTA is the method for recording the temperature difference (ΔT) between the material being studied and a reference (standard) substance ($\Delta T=T_s-T_r$) as a function of time $\Delta T=f(t)$ or temperature $\Delta T=f(T)$, when the temperature of the medium varies according to a preset program. A material of the thermostat serves as a reference in our device. In the case of a classic thermostat with cells for a sample, a reference substance and a dosing unit, the device can be used as a mixing estimating calorimeter similar to (6) by obtaining a DTA-signal and its subsequent integration.

The electronics block (see Figure 1) allows the heating and DTA to be made with a fixed rate of 0, 20, 50, and 100 K/min. between 320 and 2320 K as a preset holding time to be adjusted up to 60 min at the constant temperature in the furnace. For the version with a PPC, the heating rate is fixed at 0-100 K/min.

RESULTS

As an example, Figure 3 shows concentrational dependencies of wettability of different grades of graphite with Ni-Co and Ni-Fe melts at 1550° C in a vacuum of 6.7×10^{-3} Pa. Isotherms have been plotted for a separate heating, i. e. when a solid phase (MPG-6 graphite) and a liquid metal melt have been brought into contact at the temperature of the experiment. DTA has been performed and after holding for 15 min., the wettability angles have been measured at 1550 °C for Ni-15 at % C, Ni-58 at % Fe, and Ni-83 at % Fe being in contact with graphite of MGOSCH and "Le Carbone" grades in coheating (100 K/min). Eventually in the course of the experiment, the temperature of the contact melting (the initial liquid phase formation temperature at a preset heating rate) in the graphite-metal melt system was defined by DTA. For alloys of the Ni-Co-graphite and Ni-Fe-C systems, it turned out to approximate the temperature of Ni-C and Co-C eutectics (1330°C) and that of Fe-C eutectics (1250°C), respectively.

DISCUSSION

As is seen from the results of the investigation, the values of angles of graphite wettability with metal melts are higher under the coheating that those under the separate heating conditions. This fact is obviously due to the process of metal alloy carburization occurring back at the stage, when the alloy is in a solid. The process has been studied and reported elsewere[7,8]. The process of a metal alloy carburization is more intensive when MGOSCh graphite is used as a solid phase and less intensive with "Le Carbone" graphite; therefore, MGOSCh graphite wettability angles are higher.

CONCLUSIONS

The possibility to perform a DTA when studying the solid phase wettability with metal melts, especially, in the case of a partial dissolution of a solid phase material in a lipuid in contact, opens up new experimental opportunities of using the above mentioned device, which is very important for studying high-temperature capillarity in a number of technological processes, for the development of compositions of new solders, etc.

The integration of a PPC into the device allows not only the automatic control and regulation of the experiment parameters, but also the insurance of a qualitatively new level of storage, processing and interpretation of the information obtained in the run.

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