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THERMAL ANALYSIS AT HIGH TEMPERATURES;

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ABSTRACT

Thermal analysis at the constant temperature difference with the use of high-temperature thermoelectrical detectors of special design and with the primary processing of data in their numerical form makes it possible to determine the temperature of phase transformation onset and completion with high accuracy in the range 100-2000°C.

INTRODUCTION

At the study of phase equilibriums it becomes often necessary to define temperature intervals where the phase transformation occurs (e.g. melting of multicomponent alloys). Common methods of thermal analysis (TA), differential (DTA) and derivative (DrTA), at high temperatures permit to define accurately enough only the temperature of the transformation onset. The present work deals with the reasons of this specific drawback in the methods of high-temperature TA and proposes the way to overcome it.

COMMON HIGH-TEMPERATURE TA METHOD

In traditional TA methods the heating and cooling of the sample (reference) is provided by the heating device (furnace), where the temperature varies by the linear law. The temperature is controlled through the primary parameter - the furnace temperature. Fig.1 shows the variation of furnace and sample temperatures with heating in the course of isothermal transformation, and also the variation of temperature difference between the furnace and the sample. During the transformation, which is accompanied by the heat absorption, this temperature difference is increased. As a result we have: 1) the increase in the heat supply into the sample; transformation is accelerated; the temperature stopping due to transformation decreases.
2) the sample temperature detector gets additional heating; there

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appears the transformation imaginary interval.

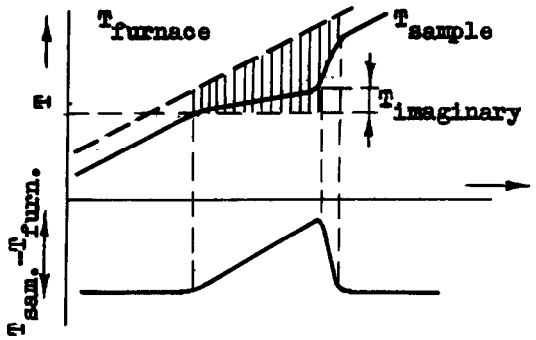

Fig.1. The common TA method.

Both the above drawbacks of common TA methods are most pronounced at high (above 1000°C) temperatures, where the heat exchange, obeying the Stefan-Boltzman law, begins to dominate:

$$Q = \alpha (T_f^4 - T_s^4) \approx 4 \cdot \alpha \cdot T^3 \cdot \Delta T \qquad (I)$$

here $T_{furnace} = T_{sample} = T$ and $\Delta T = T_f - T_s$

The use of DTA allows to overcome partly these problems but they remain at high temperatures. E.g. with the use of DTA devices 3,4 the melting imaginary interval makes up (10 ÷ 30)°C.

TA METHOD WITH A CONSTANT TEMPERATURE DIFFERENCE.

In a little known article by a russian author Kumanin (1) the TA method with a constant temperature difference has been proposed as the means to overcome the above drawbacks of common TA methods. According to his method the heating or cooling is realized by way of stabilization of temperature difference between the heating element and the sample. In this case the rate of furnace heating depends on the heating or cooling rate of the sample. The heat current into sample is determined by the magnitude of a given difference, if one doesn't account for the change in the heat conductivity with the temperature, even at the isothermal transformation. Kumanin's method works well at the temperatures up to 800°C. At atill higher temperatures the heat exchange is to higher degree determined by the formula (I), and the heating is accelerated.

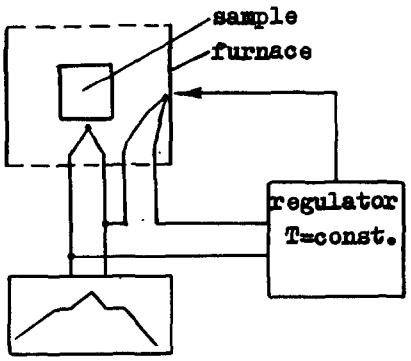

Fig.2. Scheme of TA with a constant temperature difference I

To solve this problem the thick-wall block,made of the material possessing low heat conductivity (e,g. porous W or Mo), was placed between the sample and the heater of the furnace. In the temperature range 100-2000°C the probe string construction has been used 3,4 shown in Fig.3a.

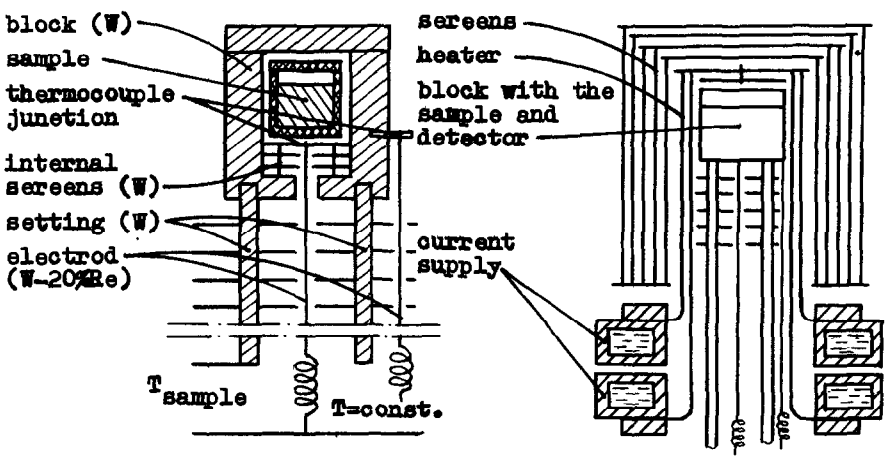

Fig.3. a)the detector block b) the high-temperature furnace for T A

Components made of W (block, internal screens and setting) serve as a common electrode of the differential thermocouple, which measures the temperature difference between the sample and the heater and one of the electrodes of the sample temperature probe. Two other electrodes in the high-temperature zone are elongated to the string form and made from the wire W + 20% Re.

TA with the constant temperature difference requires a homogeneous temperature field around the detector block. This is ensures by the high-temperature resistance furnace, which containes the double tubular heater made of Mo or W (see the construction in Fig.3b).

The TA method described above permits to determine the temperatures of transformation onset and completion as accurately as ± 1°C in the range upto 2000°C at the heating rate 10-30°C/min and the sample mass 1 - 2g.

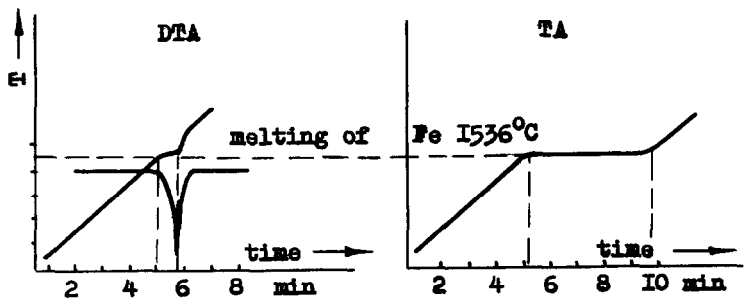

Fig.4. Examples of thermal melting curves for Fe, (sample mass 1g, heating rate 20°C/min) a) DTA method (W as the reference); b) TA method with the constant.

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