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USE OF PULSE METHOD FOR STUDY OF STRUCTURAL CHANGES OF MATERIALS

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ABSTRACT

A dynamic pulse method for study thermophysical quantities of materials has been worked out. This method enables to estimate specific heat, thermal diffusivity and thermal conductivity within a single measurement. There were developed two types of measuring techniques for bulk materials and for thin metallic foils. The méthod is suitable for study of structural changes of materials.

MEASURING METHOD

The present contribution discusses the pulse method for measuring thermophysical quantities. The principle of method is simple. In a suitable place on the sample a response to heat pulse is measured. From the parameters of the response and the heat pulse, specific heat, thermal diffusivity and thermal conductivity can be calculated. Measurements can be performed in isothermal regime as well as in nonisothermal one. Choice of heating rate depends on required statistics of measurements. Because a time interval for stabilization of sample temperature response is required between two consecutive measurements, temperature difference between these consecutive measurements will increase with increased heating rate. Both regimes allow to obtain a lot of measured data. The heating rate can be chosen from 0.01 to 5 K/min.

Measuring process is fully automatized [1]. CAMAC system with calculator ensures the following functions:

- experiment control and measuring of the parameters needed for calculation of thermophysical quantities
- local data processing and data sorting in the time of stabilization of sample temperature response
- data acquisition

The main part of pilot programme is the measuring cycle. Service routine of measuring cycle consists of all instructions related to realization of one measurement and calculation of thermophysical quantities. The pilot programme consists of measuring cycle, error

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subroutines, subroutines of data acquisition and test subroutines of experiment control. Because during the measuring cycle a lot of information unnecessary for the final representation of measurement is obtained, the reduction of data must be performed. Such reduced set of data with complete information about parameters of measured sample is stored on magnetic tape to build database.

Methodics of measurement of bulk materials with planar heat source and thin metallic foils with line heat source was worked out and yields absolute measurements and relative ones as well.

One of the greatest problems in measuring thermophysical quantities is the sample condition analysis.

The influence of disturbing factors on measurement is included in correction factor in relations for ideal heat conduction. The ideal heat conduction means that no disturbing factor acts in measuring process of bulk materials (infinite media) or thin metallic foil (thin infinite foil). The form of correction factors is given by solution of heat equation with boundary conditions determining the influence of disturbing factor. The analysis of resulting relations yields the conditions for minimalization of influence of disturbing factor. In such a way there were analysed the influence of thermal losses from the sample surface, nonideal heat source and nonsteady sample temperature [2,3] . The influence of connecting wires to the heat source was analysed experimentally.

The apparatus for measuring thermophysical quantities of bulk materials and thin metallic foils can be used in temperature region of 100 - 1300 K and 300 - 1100 K in both regimes, respectively. All measurements are made in vacuum.

Typical parameters of measurements are:

0.1 - 100 sec	- width of thermal pulse
0.1 - 3 K	- maximum of temperature response to thermal pulse
1 - 300 sec	- time of maximum of temperature response
0.2 - 10 mm	- distance between heat source and thermoelements for measuring of temperature response

RESULTS AND DISCUSSION

There were chosen two systems to demonstrate the possibility of the utility of the pulse method, i.e. monocrystal of Hg_2Cl_2 [4] and metallic glassy system $Fe_{85}B_{15}$ [3].

Monocrystal Hg_2Cl_2 udergoes a structural phase transition at 185 K from ferro- to para-phase.

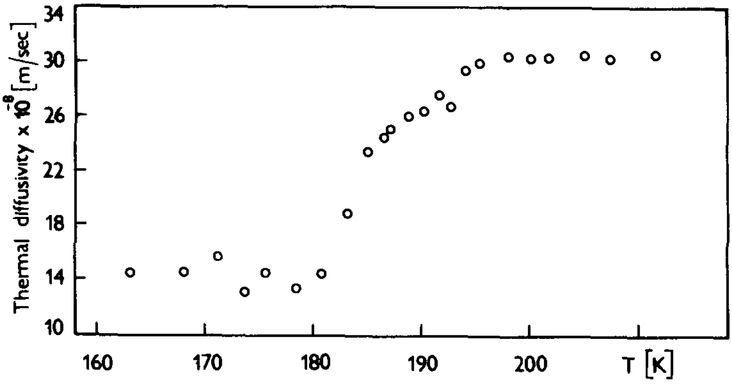

Fig. 1. Thermal diffusivity of Hg_2Cl_2

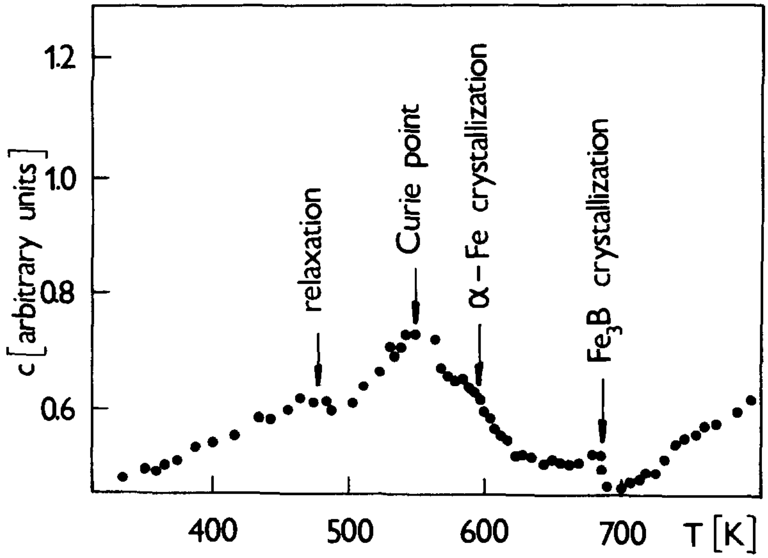

Fig. 2. Specific heat of glassy system $Fe_{85}B_{15}$

The temperature dependence of thermal diffusivity is shown on Fig. 1. In the transformation temperature region thermal diffusivity falls down due to scattering of phonons on domain walls.

Metallic glassy system $Fe_{85}B_{15}$ in temperature region from 300 K to 700 K exhibits different types of structural changes. They are the following:

- relaxation of amorphous structure above 470 K
- Curie point of amorphous phase at 550 K
- partial α - Fe crystallization from amorphous phase at 590 K
- Fe_3B crystallization of the rest of amorphous phase at 685 K.

Specific heat of glassy system $Fe_{85}B_{15}$ as a function of temperature is shown in Fig. 2.

CONCLUSION

To compare pulse method with some other methods one needs to consider temperature region and type of method used. By using pulse method three different informations, specific heat (thermodynamical quantitiy), thermal diffusivity and thermal conductivity (transport parameters) can be obtained within a single measurement. Additionally, for low rate transformations by using formulae of kinetic analysis, the method of measurement allows to evaluate enthalpy, activation energy or Avrami coefficient of transformation. In comparison to DSC technique the pulse method has higher sensitivity. It is manifested by measurements on glassy metal system

$Fe_{85}B_{15}$ where anomalies corresponding to structural relaxation and Curie point are more pronounced than by using DSC technique.

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