A Complex of Methods for Examination on Thermophysical Properties of Irradiated Materials.

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Abstract

The report covers the complex of the techniques for investigations of thermal properties of irradiated reactor materials, which are necessary for calculations of temperature, thermal stresses and phase stability of the reactor core elements used at SSC RIAR.

In particular, the report describes the principal diagrams, operation principles, accuracy of the techniques, and the examples of the results obtained.

They are as follows:

- The technique for measurement of thermal diffusivity of irradiated powders by radial and linear-in-time heating of a cylindrical sample;
- The technique for determination of thermal conductivity and capacity of compact materials in the form of disks by monotonic heating of a sample;
- The technique for determination of thermal conductivity and capacity of structural materials by the "flash" method;
- The technique for measurement of thermal linear expansion coefficient of irradiated materials with by the quartz dilatometer DKV-5AM-01;

The technique for high temperature differential thermal analysis (VDTA-8), allowing for investigation of the stability of the irradiated materials within a wide temperature range.

Keywords: Irradiated materials; Thermophysical properties

1. Introduction

One of the main tasks of the reactor material science is to study thermophysical properties of core materials of nuclear reactors as well as changes of these properties after in-core irradiation. It is important

to know thermophysical properties so as to develop calculation codes on product serviceability, chose and determine the right temperature conditions that can change during the irradiation.

The most important thermophysical properties of core elements are thermal conductivity, thermal capacity, thermal-diffusivity, and thermal expansion factor, melting temperature and phase transition. The paper presents the description of schematic flow sheets, operation principle, temperature range and uncertainty of methods used at SSC RF RIAR.

2. Methods for thermal capacity measurements

Thermal capacity measurements are carried out at the ИТ-С-400 rig intended for examination of the temperature dependence on the specific thermal capacity.

The facility operation is based on the dynamic C-calorimeter that has adiabatic cladding and contains a thermo flow meter. Thermal diagram of the measurement technique is given in fig.1

The principle of thermal capacity measurement is the following: thermal flux passing through the middle cross-section of the thermometer heats the tested specimen and capsule. The value of the thermal flux passing through the thermometer is evaluated according to the value of the temperature drop on the thermometer as well as its thermal conductivity determined in the course of independent calibration experiments using a copper specimen. The temperature range is up to 400°C. The method error is no greater than 6%.

For the first time the data are obtained on the thermal capacity of the promising absorber that is dysprosium titanate. It was done by means of this rig.

3. Method for phase transition temperatures determination by means of differential-thermal technique.

The essence of the differential-thermal technique is the following: if a specimen is placed in the medium, the temperature of which changes fluently, the specimen temperature will deviate from that of the medium. It is the result of the processes that occur in the specimen under study and that are related to thermal energy absorption or release (phase transformations, chemical reactions). By fixing these deviations a fact of endothermal as well as exothermal processes occurrence can be revealed and the temperature range of the their occurrence can be determined. In addition, in the course of one experiment all the transformations are revealed that the specimen undergoes at the temperature range under study both in the solid state and on the transition into liquid or gaseous one. The temperature range of material examination is up to 1800°C. The error of phase transition temperatures determination is 5°C at a temperature less than 1000°C and it is 12°C and higher at a temperature higher than 1000°C. The diagram of the differential sensor is given in fig.3. This method enables to determine material thermal capacity at the whole temperature range with an error of 10%.

Thermograms of heating and cooling of curium-aluminum alloy containing intermetallic $CmAl_4$ in the roentgenoamorphous state are given in fig.4.

Two thermal peaks are observed on the alloy thermogram (fig.4). One peak is intensive and corresponds to the eutectic ($CmAl_4$+Al) melting temperature of ≈650°C and the other is of extremely weak intensity at a temperature of ≈722°C. Based on the fact that the aluminum peak of the Cm-Al state diagram is similar to that of U-Al state diagram this peak has to correspond to ***Liquid***+$CmAl_4$→***Liquid*** transition.

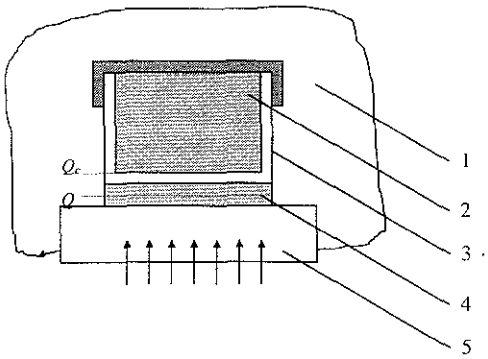

Figure 1. Diagram of thermal capacity measurement technique. 1-adiabatic cladding; 2-tested specimen; 3-capsule; 4-thermo flow meter; 5-basement

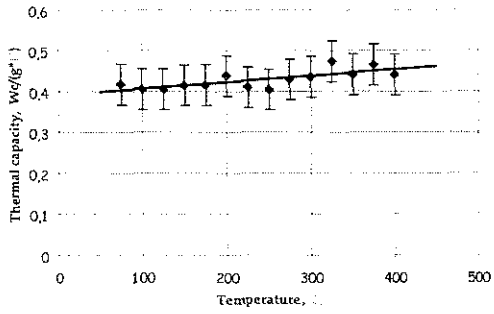

Figure 2 Dependence of dysprosium titanate thermal capacity on temperature

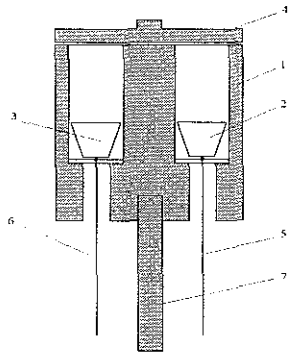

Figure 3. Differential sensor: 1 - tungsten block, 2 – crucible with reference specimen, 3-crucible with specimen, 4 - tungsten lid, 5, 6 – thermocouples, 7 - tungsten support bar

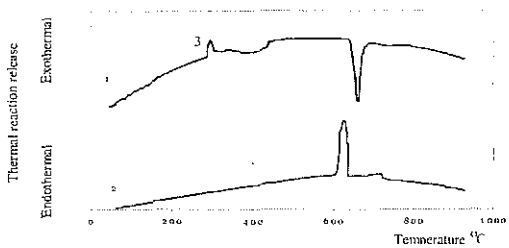

Figure 4. Thermograms of heating: 1) and cooling, 2) of curium-aluminum alloy in roentgenoamorphous state. 3) – thermal effect of crystallinity reduction

4. Measurement of powder fillings thermal - diffusivity.

To measure the thermal-diffusivity of specimens a technique of linear regular mode is used. Here the specimen is heated monotonously and its temperature changes depending on time in accordance with the linear law. In this case the temperature in the specimen center changes according to the similar law with a time delay, the value of which is determined by the specimen thermal properties: thermal-diffusivity, thermal conductivity and thermal capacity).

To measure powder thermal-diffusivity using the technique of linear regular mode it is necessary to determine the temperature on the surface as well as in the center of the cylindrical specimen in equal time periods. The geometric dimensions and thermal-diffusivity of the thermometric block cladding are known.

The rig enables to determine the temperature dependence of thermal-diffusivity in case of different

filling density, pressure and type of gaseous filling in the temperature range from 50 up to 1000°C. The measurement error is 15%.

The rig diagram is given in fig.5. This rig was used to determine the temperature conductivity of dysprosium titanate powders. The data on its thermal conductivity were obtained using the results of the thermal capacity examination (fig.6).

5. Flash method for thermal - diffusivity measurement

Flash method used for definition of thermal-diffusivity coefficient is the basis of measurements. The essence of the method is the following: a short light pulse (duration is 1.3 m/s) is directed to the frontal surface of the flat sample. The temperature response from the back surface is registered by a thermocouple. The rig diagram is given in fig.7.

The temperature response is amplified and transmitted to the digital storage oscillograph. The digitized response is transmitted to the computer where it is processed numerically to obtain the values of specimen thermal-diffusivity.

Maximum heating temperature per one light pulse runs from 0.5 to 3 ^{0}C. It is determined by the optic properties, thermal capacity and height of the specimen. The temperature range of the examination is from 20 to 1100 ^{0}C. The error of the thermal-diffusivity definition does not exceed 5 %.

6. Rig for measurement of structural material thermal conductivity

The rig for thermal conductivity measurement is intended for examination on temperature dependence of thermal conductivity of solid, mechanically processed materials. The facility operation is based on the dynamic C-calorimeter that has adiabatic cladding and contains a thermometer. Thermal diagram of the measurement technique is given in fig.8

The principal of thermal capacity measurement is the following: thermal flux passes through the middle cross-section of plate 3 that partially absorbs it. Then the flux heats plate 2, tested specimen 1 and heat receiver bar. If the heat accumulated by the specimen and plate 2 is 10 or more times less than that absorbed by the heat receiver bar then the specimen temperature field shows a linear tendency. It enables to determine the material thermal conductivity under the linear heating rate with the accuracy of 10% and in the temperature range from 50 up to 400°C (if the standard specimens with the known thermal conductivity are used for the rig calibration).

7. Rig for measurement of thermal linear expansion coefficient.

To determine the thermal linear expansion coefficient of the irradiated materials a quartz dilatometer ДКВ-5АМ-01 of box type was developed. The dilatometer (fig.9) consists of a shaft electric furnace with a slight temperature gradient in the operating area where a support quartz pipe is located. A quartz pusher moves inside the support pipe. The tested specimen 40-mm long that has plane-parallel ends is installed in the support pipe and tightened by the pusher from above. When heated, the specimen elongation is registered by a measuring head tightened to the pusher.

Measurements of thermal linear expansion coefficient are carried out in the air in the temperature range of 20-800°C. The measurement error is $0.2*10^{-6}$ at a temperature up to 100°C and $0.1*10^{-6}$ at a temperature above 100°C.

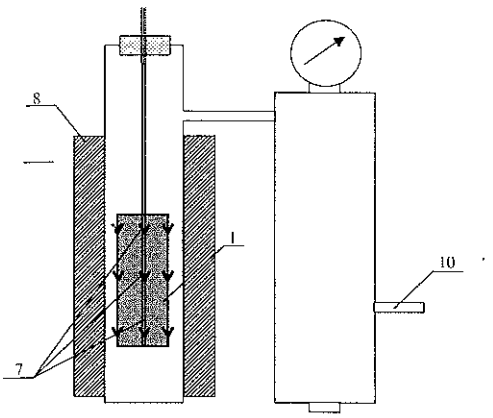

Figure 5. Rig diagram for measurement of powder material thermal-diffusivity. 1 – cylindrical specimen with vibropacked powder; 2 – free volume to maintain pressure ; 3 – outlet from thermocouples to amplifier and computer; 4 – pressure seal; 5 – joint for specimen loading; 6 – exterior thermocouple on specimen; 7 – central multijunction thermocouple, 8 – heater; 9 – vacuum manometer; 10 – outlet to vacuum-gas line.

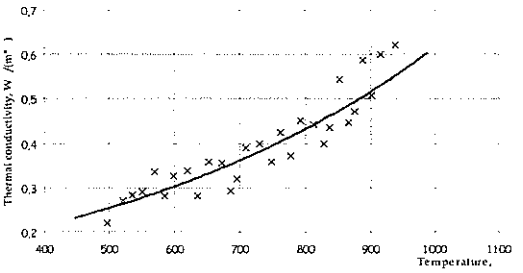

Figure 6. Dependence of dysprosium titanate thermal - diffusivity coefficient (powder filling density is 4.8 g/sm^3; helium pressure is 0.1 MPa) on temperature

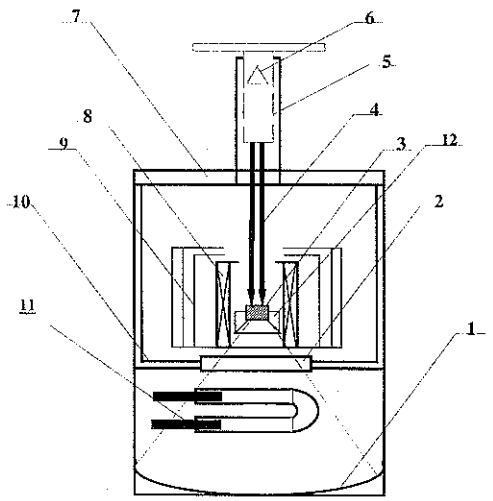

Figure 7. Flash rig diagram for thermal-diffusivity measurement: 1 - reflector, 2 – quartz glass, 3 - specimen, 4 - thermocouple, 5 – clamping device, 6 - first amplifier stage, 7 - lid with water jacket, 8 - heater, 9 - shields, 10 - vessel with water jacket, 11 – flash lamp, 12 – specimen holder

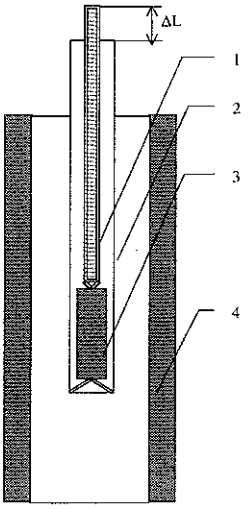

Figure 8 Diagram of quartz dilatometer. 1- quartz pusher, 2 – quartz support pipe, 3- specimen, 4 – heater, ΔL – measures specimen elongation

8. Conclusion

The paper presents a complex of material science methods available at SSC RF RIAR. The methods are intended for examinations on thermophysical properties of the irradiated materials. These properties are necessary for the calculation of temperature, thermal stresses and temperature-phase stability of nuclear reactor core elements.

The methods described allow for examinations of thermophysical properties of all the reactor materials including powders (except oxide fuel) before and after irradiation (including specimens fabricated from the spent standard devises).

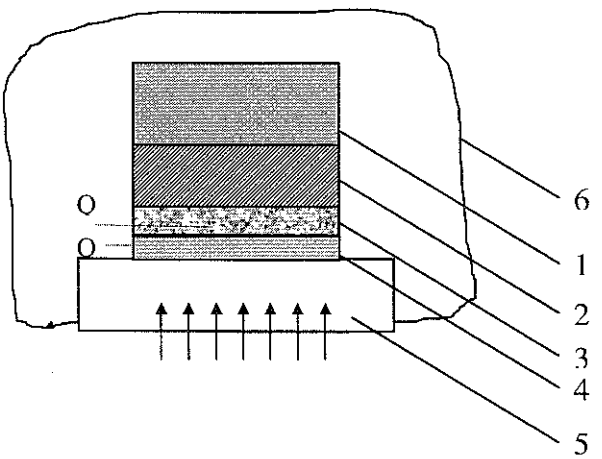

Figure 9 Rig diagram of for measurement of structural materials thermal conductivity: 1- heat receiver bar; 2- tested specimen; 3- plate; 4- thermometric plate; 5- basement; 6- adiabatic cladding