

HIGH TEMPERATURE THERMOPHYSICAL PROPERTIES OF ADVANCED MATERIALS FOR NUCLEAR DESIGN

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Abstract – Generation IV nuclear reactors will progressively replace a part of those currently in use. To improve the efficiency of these new reactors, they are required to operate at higher temperatures than the current generation. These high working temperatures induce metrological problems in relation with the selection of the most appropriate materials to safely construct reactors. This paper presents the technical program of the work package “Thermophysical Properties of advanced materials” related to the “MetroFission” project. This European joint research project aims to contribute to knowledge and technologies required for the development, construction and operation of the next generation of nuclear power plants, by offering to improve the metrology of temperatures, thermophysical properties of materials and ionizing radiation, for this type of application. The main objective of this work package (WP3) is to address the issues surrounding the high temperature characterization of thermal properties (thermal transport properties, specific heat, thermal expansion, radiative properties) of the materials required for reactors design. Reference metrological setups and methods will be implemented or improved for measurements up to 2000 °C. In addition suitable candidate materials to serve as “transfer reference materials” for high temperature thermal properties will be identified and characterised.

Keywords : Generation IV reactors, High temperature, Thermophysical properties.

1. INTRODUCTION

Around a third of the electricity and 15 % of the energy consumed in the EU comes from nuclear power plants. The nuclear power industry gives off low CO₂ emissions and is therefore an interesting option for the fight against climate change. At present, this sector is the largest source of low CO₂ emission energy in Europe. At the end of 2008, the EU-27 had 142 reactors in 15 Member States and the average age of these plants was around 25 years, with a lifespan of 40 years in general. Most European countries have decided consequently to launch the development of new nuclear power plants, based on new fission reactor concepts.

Generation IV nuclear reactors would operate at much higher temperature (i.e. above 1000 °C) than those of existing nuclear power plants. This will notably enable to increase the efficiency of power conversion and to create new applications beyond electricity generation, such as high

temperature process heat, desalinization or production of hydrogen (see figure 1). As part of the process of developing new fission reactors, a fundamental understanding of the thermal behaviour of the materials used at high temperature (structural and refractory materials, as well as irradiated and nuclear materials) must be established, in particular to define the critical performance limitations of these materials and possible design alternatives. The materials used can evolve when they are irradiated at high temperature, with appearance of creep, grain growth, pore formation, oxidation... This issue concerns a wide variety of materials which are used for in-core and out-of-core structures, pressure vessel, primary system, and components. These materials can be refractory materials, irradiated materials and fuels.

Moreover, the molten salt reactor (MSR) has been identified by the Generation-IV International Forum as one of the six nuclear energy systems that show potential for meeting the Generation-IV goals. As such, it is important to study the thermophysical properties of molten salts that are good candidates for either the primary coolant flowing through the reactor core of the MSR (and in which the nuclear fuel will be dissolved), or the secondary coolant flowing through the heat exchanger.

It is necessary to accurately determine the thermophysical properties (thermal transport properties, calorific quantities, radiative properties) of all these materials under temperature conditions encountered in fission reactors, that includes in some cases temperature conditions close to those of in-pile operation and accident conditions (up to several thousand degrees Celsius). The thermal characterizations of irradiated materials and nuclear fuels must be carried out under safe manipulation conditions (for example in glove boxes fitted with remote manipulators) by well experienced staff. For these reasons, some thermal properties of this kind of materials are already measured at high temperatures by nuclear research institutes (JRC-ITU, CEA...), which consist of dedicated facilities. The measurement methods, used by these institutes to study thermal quantities, require reference materials certified up to 2000 °C as a minimum, either for the validation of absolute measurement methods or for calibration of comparative ones. For a lot of thermophysical properties, there is currently no possibility of ensuring the traceability of these measurements to the SI above 800 °C, due to the temperature range limitation of the metrological reference facilities currently available in European National Metrology Institutes (NMIs) and the lack of suitable reference materials.

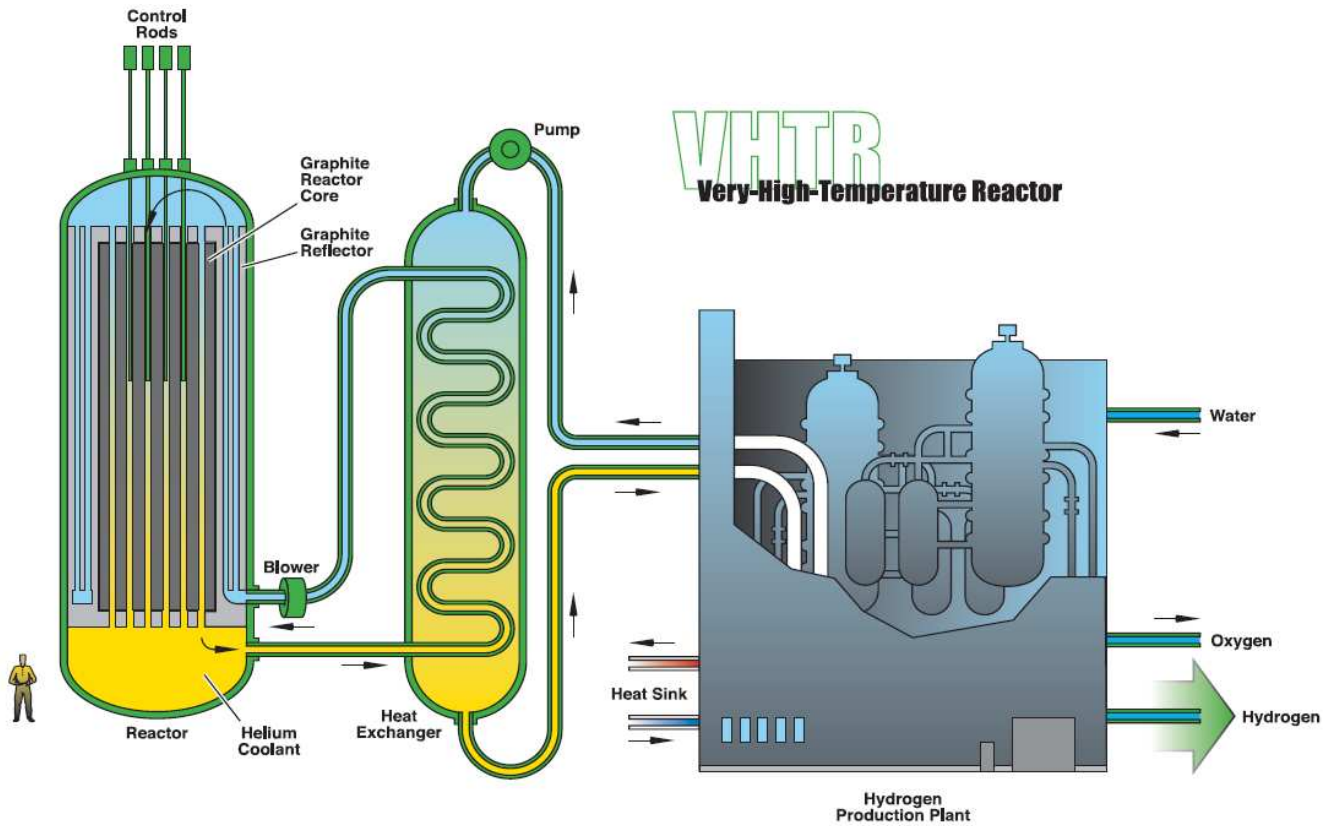


Fig. 1. Example of a Generation IV nuclear reactor concept [1]

To meet these needs, the work package “Thermophysical properties of advanced materials” of the European project “MetroFission” is devoted to the development and improvement of reference facilities for the high temperature characterisation of thermal properties of materials used in new fission reactors. Three National Metrology Institutes (LNE, PTB and NPL) and the JRC-ITU are involved in this work package (WP3). The joint research project “ENG08 MetroFission”, entitled “Metrology for new generation nuclear power plants”, is jointly funded by the European Commission and the participating countries within the EURAMET (European Association of National Metrology Institutes), in the framework of the European Metrology Research Program (EMRP). This project started in September 2010 and will last three years. This article gives a description of the different facilities and measurement methods, which will be implemented in the framework of this project, as well as the actions which will be undertaken in order to fulfil the assigned objectives.

2. DESCRIPTION OF THE WP3 “THERMOPHYSICAL PROPERTIES OF ADVANCED MATERIALS”

The work package “Thermophysical properties of advanced materials” addresses the following scientific and technological objectives:

- Implement reference metrological setups and methods for the measurement of thermal properties of advanced materials at temperatures up to 2000 °C,
- Perform a feasibility study of “transfer reference materials” for high temperature thermal properties measurements.

The different tasks which will be performed in this work package are described in the next paragraphs.

2.1. Measurement of thermal diffusivity of solid materials and molten salts up to 2000 °C

The measurement of thermal diffusivity will be performed by the four laboratories (LNE, NPL, PTB and JRC-ITU) by applying the laser flash method [2]. This method is by far the most frequently used method to measure the thermal diffusivity of solid homogeneous opaque materials. It enables rapid results on small specimens (a few millimetres thick) over a wide temperature range, from cryogenic temperatures up to several thousands degrees Celsius. It is applicable to a very wide range of materials with thermal diffusivities from $1 \cdot 10^{-7} \text{ m}^2\text{s}^{-1}$ to $1 \cdot 10^{-4} \text{ m}^2\text{s}^{-1}$. This is a primary absolute method, which allows performing measurements that are directly traceable to the primary SI units (such as temperature, time, length, voltage...) without calibration using reference materials.

2.1.1. Principle of measurement

A cylindrical specimen having a thickness L is submitted on its front face to a short energy pulse, and the induced transient temperature rise on the opposite face (rear face) is measured as a function of time. The thermal diffusivity a of the material is estimated by comparing the obtained experimental thermogram with a theoretical model, which is obtained by solving the heat conduction equation, usually in the case of an opaque homogeneous specimen.

A schematic diagram of a typical laser flash apparatus is shown in Figure 2. The energy pulse is supplied by a laser.

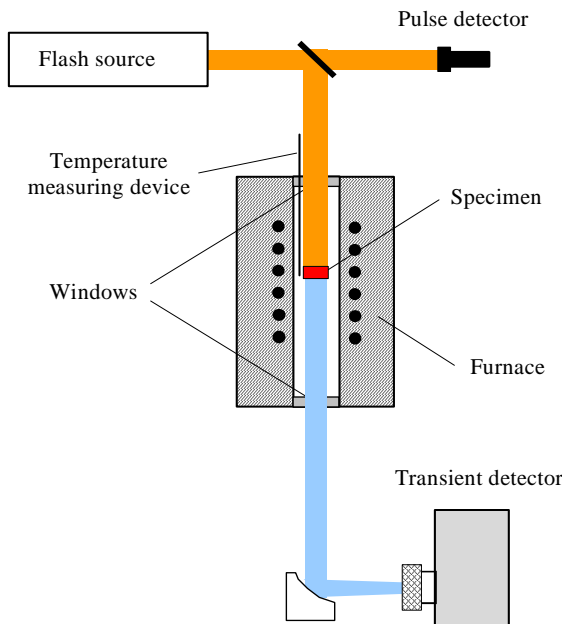


Fig. 2. Schematic diagram of a laser flash set-up

The duration of the pulse must be very short compared to the time needed to the rear face to reach its maximum temperature. The spatial energy distribution of the pulse heating must be as uniform as possible over the front face of the specimen. The transient temperature rise of the rear face of the specimen is monitored using an infrared detector. The specimen is kept at a constant temperature by means of a furnace. The test temperature is measured using a calibrated temperature measuring device (thermocouple or pyrometer).

The thermal conductivity λ ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) of medium and high conductive materials (ceramics, metals, alloys...) can be assessed by an indirect approach, from thermal diffusivity a ($\text{m}^2\cdot\text{s}^{-1}$), density ρ ($\text{kg}\cdot\text{m}^{-3}$) and specific heat c_p ($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$) measurements by means of the relationship (1).

$$\lambda = a \cdot \rho \cdot c_p \quad (1)$$

2.1.2. Current measurement capabilities of the partners

LNE performs thermal diffusivity measurements of homogeneous materials in the temperature range 23 °C to 1400 °C in argon or vacuum environments by using a homemade setup based on the traditional principle of the *rear-face* laser flash method [3]. In the case of a bulk homogeneous material, the thermal diffusivity is estimated according to the “partial time moments method” [4]. The relative expanded uncertainty ($k=2$) on thermal diffusivity measurements by the partial time moments method has been estimated to be between 3 % and 6 % [5], depending on the studied material and the temperature. A specific estimation procedure allowing thermal diffusivity measurements of coatings by *front-face* laser flash methods with the LNE setup has been developed [6].

NPL and PTB measure the thermal diffusivity of solid materials with a commercial apparatus: the LFA 427 laser flash system (Netzsch). This apparatus enables to measure thermal diffusivity from room temperature up to 1650 °C or up to 2000 °C, depending on the nature of the sample holder.

The thermal diffusivity is determined by the model of Cape-Lehman [7], which considers the cylindrical geometry of the specimen and simultaneously corrects for both finite pulse and heat losses. This approach is applied to measure the thermal diffusivity of single layer homogeneous materials. The reproducibility of the apparatus is better than 2 % and the uncertainty on thermal diffusivity measurement is estimated to be 4 % within the 95 % confidence level (coverage factor of 2).

Two laser-flash setups, called LAF and CLASH, were developed and built in-house at JRC-ITU. Their working temperature ranges are respectively [200 °C - 1300 °C] and [1300 °C - 3200 °C]. LAF was specifically designed for measurements on highly γ -active samples, such as reactor-irradiated nuclear fuel. It is, therefore, installed inside a standard hot cell, i.e. an α -tight glove box surrounded by lead shielding and equipped with remote manipulators. CLASH is designed for measurements on α -active samples at higher temperatures than is possible with LAF. Currently CLASH can be used with non-radioactive samples or those containing natural uranium only. However, once it is installed inside a glove box measurements on samples containing other actinides, for instance plutonium, will also become possible. Measurements in LAF are typically performed at about 10^{-2} mbar of nitrogen, which is the gas constantly purging the entire glove box, whereas a variety of atmospheres can be used with CLASH.

2.1.3. Improvement of the measurement methods and facilities in the frame of the ENG08 project

The LNE diffusivimeter will be modified in the framework of the JRP ENG08 in order to enable the measurement of thermal diffusivity of solid materials up to 2500 °C at minimum. This will involve in particular the development and integration of a new inductive furnace in the existing setup, and the adaptation of the associated IR detection systems.

Within the framework of the “MetroFission” project, JRC-ITU will perform a feasibility study on the applicability of the laser-flash technique to thermal diffusivity measurements on molten salts up to 1000 °C. JRC-ITU intends in particular to design and construct a specific container for measurements on molten salts of interest for the MSR.

2.2. Measurement of emissivity of solid materials up to 2000 °C

Two complementary set-ups based on two different metrological approaches will be developed for the measurement of emissivity of solid materials at high temperature (up to 2000 °C). The directional spectral emissivity $\varepsilon_\lambda(\lambda)$ is a dimensionless quantity and is for a thermally radiating specimen defined as the ratio of the spectral radiance $L_{\lambda,\text{Specimen}}$ of the specimen in a particular direction to the spectral radiance of an ideal blackbody $L_{\lambda,\text{BB}}$ at the same temperature:

$$\varepsilon_\lambda(\lambda) = \frac{L_{\lambda,\text{Specimen}}(T, \lambda)}{L_{\lambda,\text{BB}}(T, \lambda)} \quad (2)$$

2.2.1. Current measurement capabilities of the partners

LNE can measure currently directional spectral emissivity of solid materials from room temperature up to 800 °C in the spectral range 1 μm to 14 μm and in the angular range 12 ° to 60 °. The technique for measuring directional spectral emissivity at room temperature is described in [8] and the one for measuring quasi-normal spectral emissivity from 150 °C to 800 °C is presented in [9].

The method applied by PTB to measure the spectral emissivity [10, 11] is described hereafter. The emissivity measurements are performed as a spectrally resolved comparison between the specimen under test and a reference blackbody by using a Fourier-transform spectrometer. The measurement principle follows the definition of the emissivity in equation (2). The emissivity of a sample is determined by measuring the spectral radiance of the sample with respect to the spectral radiance of a reference blackbody.

2.2.2 Measurement methods developed for the measurement of emissivity at high temperature

LNE will develop a new optical facility for measuring the directional spectral emissivity of solid materials in the temperature range 800 °C to 1500 °C. The general principle of the technique is directly based on the definition of directional spectral emissivity (cf. equation 2). The facility will be composed of one or two reference blackbodies working in the temperature range 800 °C to 1500 °C, a heating system allowing the specimen to be heated up to 1500 °C in an inert atmosphere with a background radiation that is controlled, an optical system, a Fourier Transform Spectrometer to measure the spectral radiance of the source (sample or blackbody) and a blackbody at room temperature for the correction of background radiation. The spectral radiances are measured with the Infrared Fourier Transform Spectrometer in the wavelength range 0.85 μm to 14 μm. The temperature of the blackbody is measured with a platinum type thermocouple or with a pyrometer. The surface temperature of the specimen is a critical parameter for the direct technique of measurement by comparison to a blackbody. The technique for measuring this temperature is not yet selected. Different techniques are envisaged: monochromatic pyrometry, bichromatic pyrometry, "pyroreflectometric" technique and measurement with contact temperature sensors. For emissivity measurement with the direct technique at high temperatures, one of the technical problems is heating the sample in an inert atmosphere with the front surface of the sample radiating freely toward surfaces at room temperature. The radiation flux density lost by the front surface is so high (up to 56 W/cm² at 1500 °C) that it is very difficult to supply enough heat with a classical electrical resistor. The solution that LNE intends to use is a lamp image furnace where heat is produced by a set of lamps and is focused on the rear side of the sample with ellipsoidal mirrors.

PTB is developing a measurement facility for measuring the spectral emissivity of solid materials above 1000 °C [12]. For this, PTB adapted the Laser-Flash-Technique described in paragraph 2.1, to enable a Dynamic Emissivity Measurement (ADeM). The measurement principle is given by:

$$\varepsilon_{\lambda}(\lambda, T) E_{\lambda} = m \cdot c_p(T) \cdot \Delta T(T) \quad (3)$$

The left side of equation (3) represents the absorbed optical power, while the right is the increase in internal energy of a specimen with mass m and specific heat $c_p(T)$ after a step change in temperature of ΔT . It is evident from equation (3) that a knowledge of the optical flash energy E and the temperature step ΔT allows a measurement of ε if $c_p(T)$ and the mass m of the specimen are known. Figure 3 shows the principle design of the laser-flash apparatus used for the dynamic emissivity measurement.

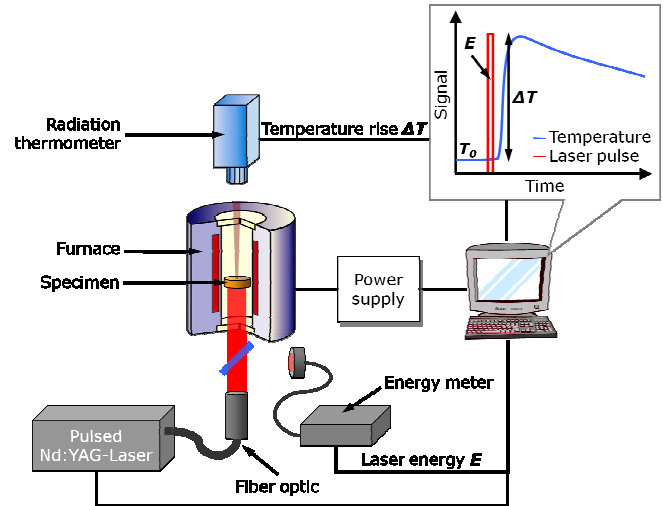


Fig. 3. Schematic of the dynamic emissivity measurement with a laser-flash apparatus

It basically consists of a high-energy pulsed laser, a high-temperature furnace which contains the specimen, and a radiation thermometer to measure the temperature rise of the specimen. In contrast to a conventional laser-flash apparatus used for thermal diffusivity measurements, the absolute laser energy will be measured via a beam splitter and a laser-energy-meter. Also the relative measuring infrared detector is replaced by a calibrated radiation thermometer for measuring the absolute value of the temperature rise.

2.3. Measurement of specific heat of solid materials up to 1500 °C

The measurements of specific heat of solid materials at high temperature will be performed by using a combination of drop calorimeters and Differential Scanning Calorimeters (DSC).

2.3.1. Current measurement capabilities

NPL and LNE use commercial Differential Scanning Calorimeters to measure specific heat from -100 °C to 800 °C by comparison with reference materials, with an estimated measurement uncertainty of 3 % to 4 % ($k = 2$) over the total range. In addition, LNE disposes of a drop calorimeter (Setaram HT1000) enabling to measure specific heat and enthalpy of fusion from 100 °C to 1000 °C.

Specific heat measurements are performed up to 1500 °C at JRC-ITU with a Setaram MHTC-96 multi-detector high-temperature calorimeter equipped with a graphite resistor furnace and operating in drop mode. The calorimeter was modified for installation of all necessary components inside an α -shielding glove box to allow measurements with actinide-containing radioactive materials, while keeping the

control unit and the power supply outside the glove box (see figure 4). A second identical calorimeter, without the above-mentioned custom modifications, can be used outside a glove box for measurements up to 1300 °C on non-radioactive samples. Measurements are performed under a flow of air or inert gas on the sample material.



Fig. 4. The drop calorimeter installed inside a glove box

2.3.2. Principle of specific heat measurement by drop calorimetry

Enthalpy measurements by drop calorimetry consist of maintaining a specimen of mass m at a constant temperature T_0 and dropping it into a heat-flow calorimeter (heated at temperature $T > T_0$) placed below. The energy absorbed by the specimen during its heating inside the calorimeter is measured by a thermopile which provides a signal proportional to the temperature difference between the specimen and the calorimeter. The enthalpy variation $\Delta H_{T_0}^T$ is obtained by integration of the signal delivered by the calorimeter over the whole duration of cooling. Measurements of the enthalpy variation are performed for various temperatures T in order to obtain the enthalpy variation as a function of temperature. The specific heat $c_p(T)$, expressed in $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$, is then calculated by differentiating the obtained function with respect to temperature according to equation (4).

$$c_p(T) = \frac{1}{m} \left(\frac{\partial \Delta H_{T_0}^T(T)}{\partial T} \right)_p \quad (4)$$

2.3.3. Improvement of the measurement capabilities of specific heat in the frame of the ENG08 project

NPL and LNE plan to extend their measurement capabilities of specific heat up to 1500 °C by using a calorimeter similar to that of JRC-ITU in DSC mode.

LNE is also improving the calibration procedure of its drop calorimeter with the aim to measure specific heat and enthalpy of fusion from 100 °C to 1000 °C with a relative uncertainty less than $\pm 0.5\%$. A new calibration system allowing to calibrate the calorimeter by electrical substitution (Joule effect calibration) is presently under study.

2.4. Determination of density of solid materials at 23 °C

Density at room temperature $\rho_{23^\circ\text{C}}$ is determined by NPL and LNE according to the Archimedean method. It is given by the following relationship:

$$\rho_{23^\circ\text{C}} = \frac{m_1 \cdot \rho_l}{(m_1 - m_2)} \quad (5)$$

where m_1 is the apparent mass of the specimen in air, m_2 is the apparent mass of the specimen in the immersion liquid (distilled water at 23 °C) and ρ_l is the density of the immersion liquid at 23 °C.

Density measurements at high temperatures are calculated from the room temperature density and the thermal expansion, assuming isotropic behaviour, using the equation (6):

$$\rho_T = \frac{\rho_{23^\circ\text{C}}}{\left(1 + \alpha_L \right]_{T_0}^T \cdot (T - T_0)}^3 \quad (6)$$

where $\alpha_L \right]_{T_0}^T$ is the mean linear thermal expansion coefficient between T_0 (23 °C) and T .

2.5. Measurement of the linear thermal expansion coefficient

2.5.1. Current measurement capabilities of the partners

The linear thermal expansion coefficient is determined at LNE, NPL and JRC-ITU with horizontally operating differential push rod dilatometers. The measurement method consists of measuring the changes in length between a reference specimen and the test specimen when they are heated at a constant rate in a furnace. The dimensional changes of the specimens are transmitted to an inductive linear displacement transducer via a fused silica or alumina push-rod. Temperature is measured by means of a thermocouple in the immediate vicinity of the specimen. The mean linear thermal expansion coefficient α_l between T and T_0 is given by:

$$\alpha_L \right]_{T_0}^T = \frac{1}{(T - T_0)} \cdot \frac{\Delta L \right]_{T_0}^T}{L_{T_0}} \quad (7)$$

Where $\Delta L \right]_{T_0}^T$ is the expansion measured between T_0 and T and L_{T_0} is the length of the specimen at room temperature T_0 .

The measurement accuracy is considered to be approximately $\pm 0.1 \cdot 10^{-6} \text{ K}^{-1}$ ($k=2$). The data obtained are also used for the thermal diffusivity measurements at high temperature, in order to calculate the corrections on the specimen thickness due to expansion.

The dilatometer of JRC-ITU is installed in a glove box for measurements on radioactive samples. It is equipped with an oven covering a temperature range from ambient up to 1550 °C. It can be operated under a flow of air, argon, argon/hydrogen, or nitrogen gas depending on the requirements.

2.5.2. Improvement of the measurement capabilities of thermal expansion

NPL and LNE will increase the temperature range of their dilatometers in the frame of ENG08, up to 1600 °C and up to 2000 °C, respectively.

2.6. Feasibility study of “transfer reference materials” for high temperature thermal properties measurements

The aim of this task is to perform a feasibility study concerning the thermal characterization of non active materials up to 2000 °C, which could be used by nuclear research laboratories as “transfer reference materials”. This feasibility study is an essential preliminary step for an eventual future certification process. The selection of these candidate materials will be based on the expertise of JRC-ITU, according to the needs of nuclear research laboratories. The measurement of their thermophysical properties (thermal diffusivity, specific heat and emissivity especially) will be done by using the metrological reference apparatuses which will be developed by LNE, NPL and PTB in the frame of this project, and the existing facilities of JRC-ITU.

3. CONCLUSION

A joint research project entitled “Metrology for new generation nuclear power plants”, funded by the European Union and EURAMET, is currently in progress. A work package of this project aims to implement a European metrological infrastructure in the field of thermo-physical properties in order to provide reliable and traceable high temperature measurements for materials having thermal properties similar to those used in fission reactors. The availability of accurate facilities and reference materials will improve the traceability of the nuclear research laboratories measurements.

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