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A simple, reliable and cost-effective, high temperature dilatometer for bulk thermal expansion studies on solids

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ABSTRACT

A simplified, highly reliable, cost effective high temperature (RT-1373 K) vertical dilatometer system, based on linear variable differential transformer (LVDT) has been designed, fabricated and tested for measurement of linear thermal dilation of solid samples. Unique features of this fully automated PC-controlled system are: simple design, fabrication from locally available components, ease of operation and maintenance, good temperature resolution (1 K), wide measurement range (±1 mm, with 100 nm resolution), microcontroller-based data acquisition hardware and a simple windows-based software. The system has been tested for its performance using NIST sapphire reference and high purity metals including aluminum and nickel. Overall accuracy of the system for measurement of linear thermal dilation is within 3% of the reported values for NIST sapphire. The system is a good example of a simple experimental facility for routine thermal expansion characterization of solids. Combination of simplified design, automation and reliable performance without using sophisticated instrumentation/control systems makes it a novel cost effective thermo-analytical equipment, which can be put to widespread routine usage for researchers, academy and small/medium-scale industries.

1. Introduction

Dilatometry is a thermo-analytical technique for measuring the linear thermal dilation (expansion/shrinkage) of a solid material that is subjected to a programmed heating/cooling. From the dilation data, important physico-chemical properties of material can be inferred. For example, linear dilation characteristics of a highly sintered sample (length 'L_0' at ambient temperature), enables the evaluation of its linear thermal expansion behavior in terms of coefficient of thermal expansion 'α', given as:

True coefficient of linear thermal expansion:

$$\alpha_{\text{Linear}} = \lim_{\partial T \to 0} \frac{1}{L_0}\left(\frac{\partial L}{\partial T}\right) \tag{1}$$

Average coefficient of linear thermal expansion:

$$[\alpha_{\text{Linear}}]_{T1}^{T2} = \frac{1}{L_0}\left(\frac{\Delta L}{\Delta T}\right), \quad \Delta T = (T_2 - T_1) \tag{2}$$

Similarly, volume thermal expansion can also be evaluated for a sample. Thermal expansion information of a material is important in (i) basic research to develop new materials with known/tailored thermal expansion characteristics and (ii) applied engineering, where dimensional compatibility of two dissimilar materials crucially depends upon their thermal expansion characteristics. Dilatometry and high temperature X-ray diffraction (HT-XRD) are complementary techniques for studying dimensional changes in materials as a function of temperature. In conjunction with HT-XRD, dilatometry provides crucial information about defect concentration. Dilatometry is also an important tool for (i) studying the sintering behavior of powdered materials [1] and (ii) studying the phase transitions in solids that involve dimensional changes such as 1st order structural transitions, glass transition, etc. Dilatometry can also be used to study the chemical reactions at solid–gas interface (oxidation of metals) under controlled reaction atmosphere.

In nuclear technology, dilatometry finds a very important place. Thermo-chemical and thermo-physical scenario inside an operating nuclear reactor continuously evolve. It is known that the fuel pellets swell during high temperature operation and fission product buildup. Therefore, thermo-physical characterization of these fuels and fuel-fission product adducts is required to ensure their

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safe and optimum performance [2]. Dilatometry is widely used to characterize all these materials (fuel, clad, structural materials, etc.).

In summary, dilatometry is an important thermo-analytical technique for characterization of solids. It finds wide applications in several areas such as ceramic & glass industries, metals & alloys engineering, polymers, and powder metallurgy. While several commercial dilatometer systems are available worldwide, most of them are based on sophisticated design, use expensive components and controlled by complex integrated circuits. These attributes make such systems quite cost intensive and always require manufacturer's support for routine/incidental repair/maintenance. Having a widespread utility, as mentioned above, a simplified and cost effective design of such experimental facility without compromising on the accuracy and reliability of results for routine measurement of thermal dilation of solids is highly desirable. Keeping this in mind, we have developed a simplified, fully automated, PC-based, vertical dilatometer using locally available components. Highly reproducible and reliable performance of the equipment coupled with ease of fabrication and maintenance is the unique attribute of this system. In this paper, we report the design, fabrication and testing of developed system.

2. System description

Fig. 1 shows the block diagram of dilatometer developed in our lab. It consists of (a) a high temperature furnace, (b) programmable temperature controller, (c) sample holder & push rod assembly, (d) linear displacement transducer (LVDT), (e) furnace lifting mechanism and (f) data acquisition system. The Description of individual components is provided in following sub-sections.

2.1. High temperature furnace

A high temperature resistive tube furnace was fabricated by winding kanthal wire over an open ended re-crystallized alumina tube. The winding was carried out to obtain a uniform temperature zone in the middle of the tube. A Pt–Pt13%Rh thermocouple (R-type) was fixed along the furnace tube to control the temperature. The heater tube was enclosed in a cylindrical aluminum shell with circular end-plates (made of asbestos) and insulated by low conductivity bubble alumina. An aluminum jacket for water circulation was fixed on the top side of furnace to minimize heat transfer to LVDT.

2.2. Programmable temperature controller

A compact; low-cost, multi-segment auto-tune PID temperature controller (PR 502, M/s Selec Controls Pvt. Ltd., Mumbai) was used for controlling the furnace temperature. The controller; with a temperature resolution of 1 K, can be operated in heating/cooling/isothermal modes. The control thermocouple is R-type, as mentioned above.

2.3. Sample holder and push rod assembly

Fig. 2 shows the schematic of sample holder and push rod assembly used in the dilatometer. It consists of a flat-ended re-crystallized alumina (RCA) sample holder tube, a hemispherical-ended RCA push rod ($\phi \sim 6$ mm), a one-end closed RCA protective cover tube, sample thermocouple (R-type) and top clamping assembly. A rectangular cutout was made near the closed end of sample holder tube for sample mounting and thermocouple positioning. For sample mounting, a highly polished RCA disc ($\phi \sim 8$ mm) was fixed at the closed end of this tube using alumina cement. Fixing of this disc ensured minimum baseline drift over repeated experiments. The other end of sample holder tube was fixed with an aluminum support, as shown in the schematic. This support was connected to LVDT protective shell with threading arrangement for vertical movement of sample holder tube. The push rod used to transmit the dilation signal to LVDT was placed in the sample holder tube so that its one end touched the LVDT plunger while the other end rested on sample surface. The sample thermocouple was fixed along the support tube and it's junction kept flexible using alumina beads in order to position it near to the sample. The protective cover tube was used to enclose the sample holder assembly after the sample is loaded. Both, the protective tube and LVDT were fixed at the clamping assembly made of aluminum.

2.4. Linear displacement transducer

A commercially available displacement sensor and indicator (SI-706 & SI-70T, M/s Syscon Instruments Pvt. Ltd., Bangalore) were used in the present system. The spring-loaded configuration LVDT has a displacement range of ±1 mm with a resolution of 100 nm. The analog output signal (±5 V DC for full range) of LVDT is fed to the data acquisition system in order to obtain temperature/time dependent dilation behavior.

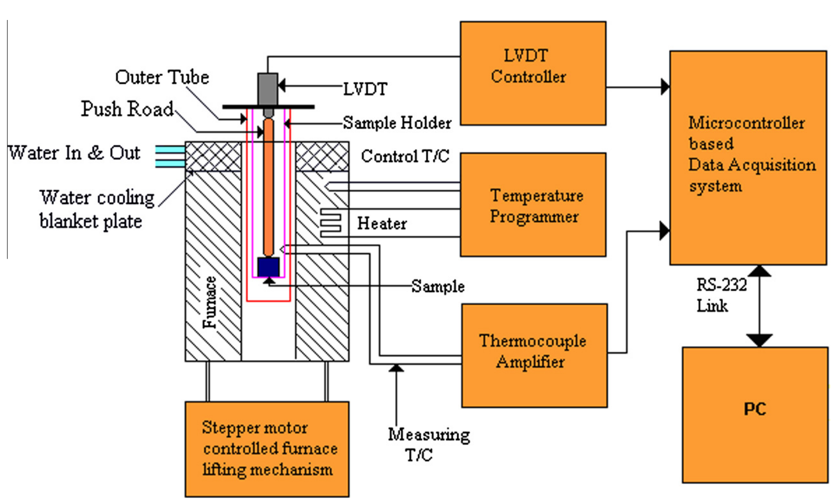

Fig. 1. Block diagram of developed dilatometer.