

Measurement of thermal expansion coefficient of nonuniform temperature specimen

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A new technique is developed to measure the longitudinal thermal expansion coefficient of C/C composite material at high temperature. The measuring principle and components of the apparatus are described in detail. The calculation method is derived from the temperature dependence of the thermal expansion coefficient. The apparatus mainly consists of a high temperature environmental chamber, a power circuit of heating, two high-speed pyrometers, and a laser scanning system. A long solid specimen is resistively heated to a steady high-temperature state by a steady electrical current. The temperature profile of the specimen surface is not uniform because of the thermal conduction and radiation. The temperature profile and the total expansion are measured with a high-speed scanning pyrometer and a laser slit scanning measuring system, respectively. The thermal expansion coefficient in a wide temperature range (1000 – 3800 K) of the specimen can therefore be obtained. The perfect consistency between the present and previous results justifies the validity of this technique.

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In the past decades, most of the accurate measurements of thermal expansion coefficients at temperatures above 1100 K generally rely on the steady-state techniques such as push-rod dilatometry, X-ray diffractometry, and optical comparator methods. The measuring precision of the steady-state techniques mainly depends on the degree of keeping uniform temperature of the specimen, which is hard to realize because of thermal conduction at ends, especially at high temperature^[1]. The problem of nonuniform temperature profile restricts the application of those methods at ultra high temperature. With the rise of pulse heating method and the development of modern electronics, Righini *et al.* proposed a new idea to measure the thermophysical properties by an assumed function of thermalphysical properties and temperature according to the rules of thermalphysical varying with temperatures^[2]. Moreover, this method was applied to the measurement of thermal expansion coefficient of a specimen with a nonuniform temperature profile based on the apparatus of pulse experiment and a high-speed scanning pyrometer^[3]. However, the temperature profile of the specimen must be measured as quickly as possible in the transient-state technique in order to minimize the influence of temperature fluctuation. Based on the advantages of the steady-state method and the Righini's calculation theory of nonuniform temperature profile, the present paper describes an integration method for the measurement of thermal expansion coefficient up to ultra high temperature. The requirement of measuring the thermal expansion coefficient of a C/C composite material can be satisfied with this method.

A method for thermal expansion measurements of a specimen with nonuniform temperature profile was successfully developed^[4]. Righini assumed that the expansion function in the temperature range of interest may be represented by an n th order polynomial constrained by a reference temperature T_0

$$f(T) = \frac{\Delta L(T)}{L(T_0)} = a_1(T - T_0) + a_2(T - T_0)^2 + \dots + a_n(T - T_0)^n, \quad (1)$$

where $L(T_0)$ is the length of the specimen at T_0 , $\Delta L(T)$ is experimental total expansion associated with a particular temperature profile, and T is the experiment temperature of the specimen.

If the experimental temperatures are measured in positions z_1, z_2, \dots, z_M with equal intervals, the specimen may be considered as composed of M segments with equal length of S . These segments are marked as l_i ($i = 1, 2, \dots, M$) and the corresponding length at temperature T_0 may be any sizes and they are not necessarily equal. The relations of these quantities can be expressed with the following equations:

$$L(T_0) = \sum_{i=1}^M l_i, \quad L(T) = \sum_{i=1}^M S = M \cdot S. \quad (2)$$

The specimen may have an arbitrary temperature distribution. An overdetermined system may be got if p profiles are measured ($p > n$)

$$\left\{ \begin{array}{l} \Delta L_1 = a_1 \sum_{i=1}^{M_1} l_{1i}(T_0) (T_{1i}^* - T_0) + \dots \\ \quad + a_n \sum_{i=1}^{M_1} l_{1i}(T_0) (T_{1i}^* - T_0)^n \\ \quad \vdots \\ \Delta L_p = a_1 \sum_{i=1}^{M_1} l_{pi}(T_0) (T_{pi}^* - T_0) + \dots \\ \quad + a_n \sum_{i=1}^{M_1} l_{pi}(T_0) (T_{pi}^* - T_0)^n \end{array} \right., \quad (3)$$

where M_1 is the segments at the first experiment, ΔL_i is the difference of i th experimental total expansion associated with a particular temperature profile, and $i = 1, 2, \dots, p$.

The parameters a_1, a_2, \dots, a_n may be obtained by least squares techniques when $p > n$. Since thermal expansion functions are generally well represented by low degree polynomials, a few profiles are sufficient for an overdetermined system. The thermal expansion coefficients can be calculated by Eq. (1).

Based on Righini's method, a new measuring method of thermal expansion coefficient is developed to perform experiments as well as computations in situ. In the present work, the experimental process and the meaning of notations are same with Righini's, and a new expansion polynomials function about l_i and S with respect to the temperature T_i is defined as

$$Q(T_i) = \frac{S - l_i}{S} = a_1 (T_i - T_0) + a_2 (T_i - T_0)^2 + \dots + a_n (T_i - T_0)^n, \quad (4)$$

$i = 1, 2, \dots, M; \quad n = 1, 2, \dots,$

where T_i is the average temperature of the specimen segment between positions z_i and z_{i+1} .

Equation (4) may be expressed by

$$S = l_i + S \cdot Q(T_i). \quad (5)$$

Equation (5) is valid to any parts of the specimen, and the sum of all the segments gives the following expression:

$$\sum_{i=1}^M S = \sum_{i=1}^M l_i + \sum_{i=1}^M S \cdot Q(T_i). \quad (6)$$

If Eq. (2) are applied to Eq. (6), the equation may be generalized as

$$\frac{L(T) - L(T_0)}{S} = a_1 \sum_{i=1}^M (T_i - T_0) + a_2 \sum_{i=1}^M (T_i - T_0)^2 + \dots + a_n \sum_{i=1}^M (T_i - T_0)^n, \quad (7)$$

$n = 1, 2, \dots$

Since Eq. (7) is valid to any profiles, it may be rewritten when applied to the generic k th profile as

$$\frac{L_k(T) - L_k(T_0)}{S_k} = a_1 \sum_{i=1}^{M_k} (T_i - T_0) + a_2 \sum_{i=1}^{M_k} (T_i - T_0)^2 + \dots + a_n \sum_{i=1}^{M_k} (T_i - T_0)^n, \quad (8)$$

$n = 1, 2, \dots,$

where M_k is the segment at the k th experiment.

When p profiles (with $p > n$) are measured experimentally, a new overdetermined system of linear equation is

obtained

$$\begin{bmatrix} \frac{L_1(T) - L_1(T_0)}{S_1} \\ \vdots \\ \frac{L_p(T) - L_p(T_0)}{S_p} \end{bmatrix} = \begin{bmatrix} \sum_{i=1}^{M_1} (T_i - T_0) & \sum_{i=1}^{M_1} (T_i - T_0)^2 & \dots & \sum_{i=1}^{M_1} (T_i - T_0)^n \\ \vdots & \dots & \dots & \vdots \\ \sum_{i=1}^{M_p} (T_i - T_0) & \sum_{i=1}^{M_p} (T_i - T_0)^2 & \dots & \sum_{i=1}^{M_p} (T_i - T_0)^n \end{bmatrix} \times \begin{bmatrix} a_1 \\ a_2 \\ \vdots \\ a_n \end{bmatrix}, \quad (9)$$

where M_p is the segments at the p th experiment.

The parameter a_i can be obtained by least squares techniques when $p > n$. Consequently, the thermal expansion coefficient can be expressed by

$$\alpha(T_i) = \frac{S - l_i}{T_i \cdot l_i} = \frac{Q(T_i)}{T_i \cdot (1 - Q(T_i))}. \quad (10)$$

Measurements are performed with a thermal expansion apparatus^[5]. A block diagram of this apparatus is shown in Fig. 1. The apparatus mainly consists of the following five parts.

- 1) A central hot zone in which thermal expansion and steep temperature gradients towards the ends of the specimen are produced and confined.
- 2) A double-wall water-cooling environmental chamber for conducting experiments.
- 3) A twenty-wavelength pyrometer for central temperature measurement of the specimen and a high-speed scanning pyrometer for longitudinal temperature profile measurement.
- 4) A laser slit scanning measuring system for the measurements of thermal expansion.
- 5) A high-speed data acquisition system.

The hot-zone assembly contains a cylindrical specimen, two cylindrical graphite electrodes, two double-wall water-cooling brass electrodes, and a radiation shield. The typical dimension of the specimen is 8 mm in diameter and 100 mm in length. The specimen is screwed to two graphite electrodes; the upper graphite electrode is connected with a brass electrode through an

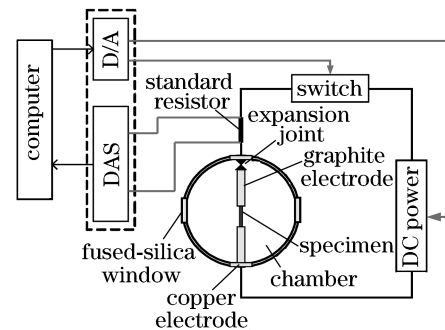


Fig. 1. Block diagram of the experimental apparatus. D/A: digital-to-analog converter; DAS: data acquisition system; DC: direct current.

expansion joint which is specifically for thermal expansion at high temperature experiments. The temperature profile of the specimen may be changed with different dimensions of graphite electrodes. The brass electrodes are fixed at the experimental chamber for electric connection and heat conduction. The temperature of the brass electrodes are very close to room temperature by water-cooling, which is helpful to reach a steady-state and provide steep temperature gradients towards the ends of the specimen. The specimen is encircled by the radiation shield, which is made of 30-mm-thick carbon felt and therefore can minimize the influence of reflected light and thermal radiation. The focusing of the pyrometer on the specimen is achieved through a fused-silica window.

The elements of the hot zone are vertically housed inside a special double-wall water-cooling bell chamber. The experiments of thermal expansion are performed by means of water-cooling to make sure that the heat from the hot zone is absorbed by the chamber walls at room temperature. The frame of the chamber is made of stainless-steel and is sealed at the front and back sides of the chamber by two double-wall water-cooling doors with windows for temperature measurement. There are also two fused-silica windows at the left and right sides of the chamber for thermal expansion measurement. The view angles of windows are 0° , 90° , 180° , and 270° , respectively. The chamber is designed for conducting experiments with the specimen in vacuum using a fore-pumping system, which is connected to the chamber by a high-vacuum valve. This is necessary to avoid the oxidation of the sample and graphite electrodes at high temperatures. By this way, the samples can be heated from room temperature up to the ultra high temperature.

The temperatures of the specimen are measured with a twenty-wavelength pyrometer^[6] and a scanning pyrometer^[7,8], which are designed and constructed specifically for surface measurements on the basis of the eight-wavelength pyrometer^[9]. The pyrometers cover a very wide temperature range (1000 – 3800 K) using an auto-ranging feature. The upper limit is determined by the possible maximum heating of the specimen. The lower limit of measuring temperature is determined by the conditions of the pyrometers and the radiative properties of the specimens. The response times are less than 10 μ s. The twenty-wavelength pyrometer (working wavelength 600 – 1000 nm) is placed at the front of the specimen and operates at the center of the specimen. The longitudinal temperature profiles of the specimen are obtained by a scanning pyrometer (working wavelength 656 – 900 nm), which is placed at the opposite position to the twenty-wavelength pyrometer. The schematic diagram of temperature measurements is shown in Fig. 2.

Thermal expansion of the specimen is measured by a laser slit scanning measuring system^[10–12]. A schematic diagram of the laser slit scanning measuring system is shown in Fig. 3. The details of the laser slit scanning measuring system may be found in earlier papers, and its measuring principle is presented here.

The laser beam from a semiconductor laser through an optical transferring system and a reflecting mirror is incident on the rotating mirror at angular speed ω . The

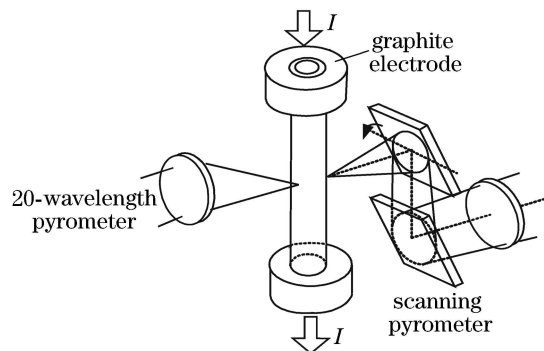


Fig. 2. Schematic diagram of temperature measurements.

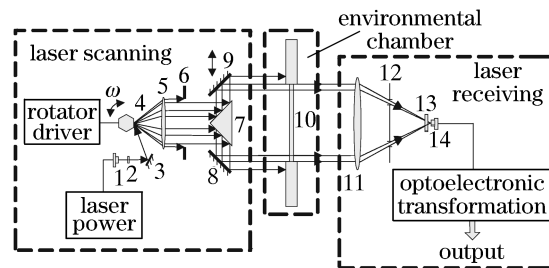


Fig. 3. Schematic of the laser slit scanning measuring system. 1: semiconductor laser; 2: optical transferring system; 3: reflecting mirror; 4: rotating mirror; 5: laser scanning optical system; 6: aperture; 7: corner cube prism; 8,9: mirrors; 10: specimen; 11: convex lens; 12: aperture; 13: filter; 14: receiver.

reflecting system produces a high-speed scanning beam which is parallel to the optical axis through the laser scanning optical system. Two laser-scanning beams are formed through the aperture and the corner cube prism, in which beams pass two mirrors respectively and scan the length of the specimen at a constant speed.

The scanning beams with the length information of the measured specimen enter the receiver through a convex lens, another aperture, and a filter, and then the light signals are transferred into electric signal through the optoelectronic transformation electronic system and processed by microcomputer. In this way, the gauge length of the specimen is obtained.

The data acquisition circuit (DAC) consists of a computer and a 16-bit data acquisition system. The function of the DAC is used for controlling the working state of the DC Power and the Switch. The electrical signals, such as outputs from the pyrometers and the laser slit scanning measuring system as well as the voltage across the standard resistor are recorded through the DAC. The typical data acquisition rate of all these voltages is 100 kHz.

Several measurements of longitudinal thermal expansion are performed on a C/C composite material. And its thermal expansion coefficient at high temperatures above 1273 K is measured in order to test our method. The laboratory is air-conditioned to keep constant room temperature. After five minutes, the chamber is evacuated down to 0.001 Pa and purged with argon. Repeat such procedures three times to reduce the influences of water vapor and oxygen in the air. Then the chamber is floated with argon up to 1.10 Pa and

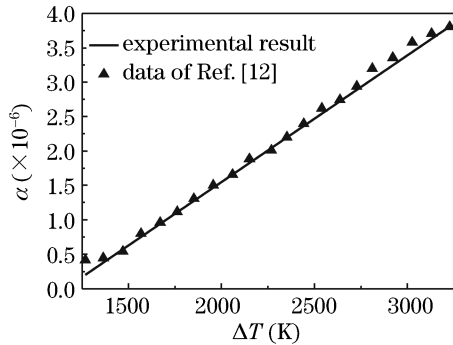


Fig. 4. Temperature dependence of the linear expansion coefficient α .

the sample is heated in steps of 300 – 400 K. The temperature is measured at equilibrium state, which can be judged from the outputs of the twenty-wavelength pyrometer. Several temperature profiles and corresponding thermal expansion are recorded to calculate the function of thermal expansion at high temperature with the method described above.

The results for thermal expansion of the specimen are illustrated in Fig. 4. The solid line represents the linear function fitting to the data by the least-square method. The fitted function of the thermal expansion coefficient α , which is available in the range from 1000 to 3200 K, is given by

$$\alpha = -2.13651 \times 10^{-6} + 1.84 \times 10^{-9} \Delta T. \quad (11)$$

The previously reported data in the same temperature range are shown for comparison^[12]. Because of the same compositions of the specimen, the perfect consistency between two experiments results justifies the validity of our method for measuring the thermal expansion coefficients at high temperature although there are few differences between the conditions of the present and previous experiments. The maximum error of thermal expansion coefficient occurs at low temperature. The small discrepancy at lower temperature range can be attributed to few overlapping profiles and the thermal expansion of

the C/C composite material is weak. The experiments of thermal expansion at even higher temperatures are limited for oxidation and evaporation of the specimen.

In summary, the construction of the apparatus and the measurement principle of thermal expansion coefficient of a specimen at nonuniform temperature are described. The experimental apparatus are constructed and measurements are performed to measure the thermal expansion coefficients of C/C composite material. The validity of this method is proved by experiments, and the reason for the measurement error is analyzed.

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