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Noncontact method of thermal linear expansion coefficient determination for various materials in temperature range 500 to 2500°C

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A noncontact method has been developed to determine the thermal linear expansion coefficient (TLEC) of carbon-graphite and ceramic materials and heat-resistant alloys in a temperature range of 500 to 2500°C. The setup and its functional potentialities are shown schematically. The TLEC values measured on primary standard specimens (electrolytic nickel and commercial APB graphite) differ by at most 5 % from literature reference data. Experimental temperature dependences of relative elongation and TLEC for the high-temperature Rene 80 alloy in the 500–1200°C interval are presented. A hysteresis is revealed in the $\Delta l/l(T)$ dependence, which is connected with the cooling-induced formation and heating-induced dissolution of the ordered γ-phase (Ni_3Al).

Разработан бесконтактный метод определения термического коэффициента линейного расширения (ТКЛР) углеграфитовых, керамических материалов и жаропрочных сплавов в интервале температур 500–2500°C. Приведена схема установки и показаны ее функциональные возможности. Измеренные значения ТКЛР эталонов (электролитического никеля и промышленного графита АРВ) отличаются от справочных литературных данных не более, чем на 5 %. Представлены экспериментальные данные по температурной зависимости относительного удлинения и термического коэффициента линейного расширения жаропрочного никелевого сплава Rene 80 в области температур 500–1200°C. На зависимости $\Delta l/l(T)$ выявлен гистерезис, связанный с образованием при охлаждении и растворением при нагреве упорядоченной γ-фазы (Ni_3Al).

To determine the TLEC of different heat-resistant alloys, carbon-graphite, and ceramic materials in the 500–2500°C range, a noncontact method was developed to measure the specimen length during heating and cooling. The setup for TLEC measuring (Fig. 1) is a cylindrical water-cooled vacuum chamber (*1*) equipped with a vacuum pumping system (*2*, *3*), a power unit (*4*, *5*), and a thermal assembly (*6*, *7*, *8*, *9*, *19*). The

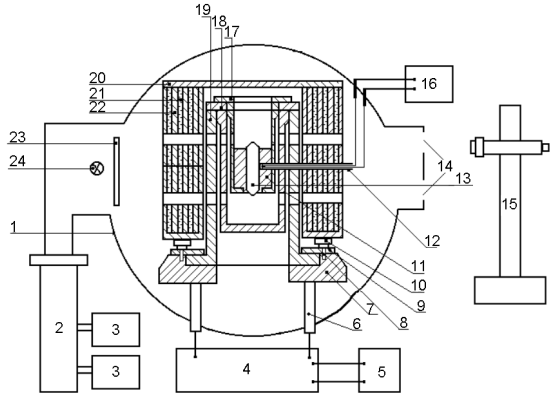

Fig. 1. The basic scheme of the setup for measuring TLEC: *1*, water-cooled vacuum chamber; *2*, N-630 diffusion pump; *3*, 2NVR-5DM vacuum pump; *4*, OSU 100/0,5D power transformer; *5*, RN630 voltage regulator; *6*, water-cooled lead-in wires; *7*, bulk graphite current bearing bars; *8*, graphite bolts; *9*, graphite plates; *10*, ceramic supports; *11*, mounting block for the specimen; *12*, corundum tube; *13*, specimen; *14*, looking windows; *15*, V-630 cathetometer; *16*, Shch 300 digital voltmeter; *17*, *18*, graphite containers; *19*, graphite heater; *20*, graphite cap; *21*, carbon felt; *22*, heat shields; *23*, frosted glass; *24*, light source.

pumping system includes two 2NVR-5DM roughing-down pumps (*3*) connected in parallel, an N-630 pump diffusion (*2*), and a vacuum-line system. The power unit of the setup includes three parallel-connected OSU 100/0,5D transformers (*4*) (the total power is 300 kW) and a RN630 voltage regulator (*5*). The thermal assembly consists of copper (*6*) and graphite (*7*) lead-in wires, heater (*19*), and graphite plates (*9*) fastening the heater to the lead-in wires by graphite bolts (*8*). The carbon-graphite and ceramic specimens (*13*) are shaped as 100 mm long cylinders of 15 mm in diameter. The cylinder butts are tapered off with 45° angle at the cone vertex. The specimen is fixed in a graphite supporting ring (*11*). The supporting ring with the specimen is mounted at the bottom of a graphite container (*17*) which in turn is placed within another container (*18*). The whole system is put inside a graphite heater (*19*).

A heat-resistant alloy specimen is a plate ($75\times2\times10$ mm^3) with a 4 mm diameter hole in the upper part. A corundum tubular rod is fed through the hole to suspend the specimen (the mounting block for metal specimen is detailed in Fig. 2).

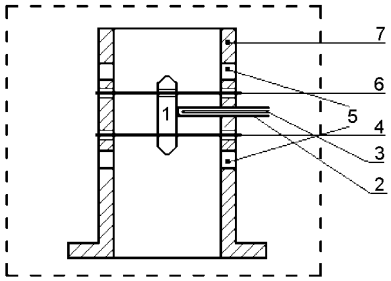

Fig. 2. Mounting block for heat-resistant alloy specimens. *1*, specimen; *2*, corundum tube; *3*, thermocouple; *4*, ceramic rod for fixing the specimen; *5*, looking windows; *6*, ceramic rod for suspending the specimen; *7*, heater.

With this construction of the thermal assembly, the specimen can be set in a vertical position. Graphite shields (*22*) with carbon felt (*21*) and a carbon-carbon composite cap (*20*) are used to thermostate the working zone of the furnace with the specimen (Fig. 1). The graphite shields were insulated from the lead-in wires with ceramic plates (*10*). Below 1000°C, the temperature was measured with a differential W–Re thermocouple fixed at the middle of the specimen using a Shch 300 digital voltmeter (*16*). During heating and cooling, the temperature was recorded using a KSP-4 potentiometer conditions. An Al_2O_3 casing was used to prevent the thermocouple carburizing. Above 1000°C, the temperature at the lower and upper ends of the specimen was additionally controlled through the viewing windows (*14*) using a Promin‘ pyrometer. The specimen length was measured by a V630 cathetometer (*15*) through the chamber windows and the slots cut in the shields, the heater and the containers.

The measurements were made in 10^{-4} Torr vacuum. The initial specimen length at room temperature was measured using a micrometer and a cathetometer. The difference between their readings was at most 0.5 %. The cathetometer objective was at 1500 mm distance from the specimen. The specimen initial temperature was found from the thermocouple readings and controlled with a laboratory thermometer. The specimen was heated and kept at the measurement temperature using a high-precision VRT-3 controller. The heating rate was about 5°C/min. The specimen length was measured at steady-state heat flow at intervals of 20–25°C. The exposure at each temperature was about 2 min. The temperature was maintained to within ±3 deg. One measure-

ment run took about 30 s and was made twice for each temperature. The specimen length at the measurement temperature was found as the arithmetical mean of two measurements, the difference between those being at most 1 %. As mentioned above, the temperature over 1000 °C was measured additionally by a pyrometer. The temperature difference between the lower and upper ends of the specimen did not exceed 10 °C and practically disappeared above 1500 °C. The total expansion of the system can be neglected in this method.

Because of the random measurement error, the experimental temperature dependences of the relative elongation exhibit a certain data scatter. The estimation of the TLEC directly from these results, i.e. differentiation of the relative elongation with respect to temperature, would further increase the scatter and make it difficult to clear up the regularities of the TLEC variation with temperature. To avoid this problem, the experimental results on the relative elongation were approximated with a third-degree polynomial (Microsoft Excel Program). The TLEC was then estimated from the resulting smoothed curve.

On heating, the TLEC was found as

$$\alpha = \Delta L / L_0 (T_2 - T_1), \qquad (1)$$

where α is the average TLEC value in the T_2–T_1 interval; L_0, the initial specimen length; ΔL, the specimen elongation at heating from T_1 to T_2. The TLEC measurement error was at most 10 %.

Electrolytic nickel was chosen as a primary standard specimen for checking the dilatometer. As comparison shows, the discrepancy between the measured TLEC values and the corresponding literature data [1] does not exceed 5 %. To test the reliability of TLEC measurement at higher temperatures, the TLEC of commercial APB graphite was measured along the compacting axis. The results obtained were compared with literature data [2]. So the largest deviation of commercial APB graphite TLEC from the tabulated data at temperatures up to 2500°C has been shown to be within 5 %.

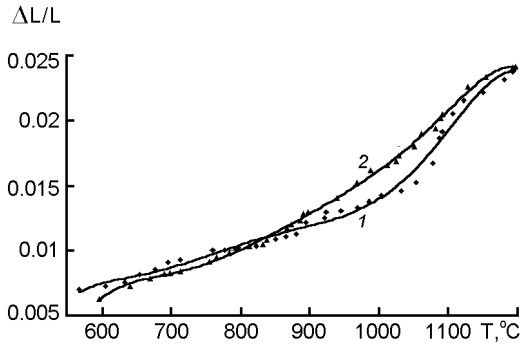

Fig. 3. Relative elongation of Rene 80 alloy versus temperature: *1* – heating, *2* – cooling.

As an example, we show here the temperature dependences of the relative elongation and the thermal linear expansion coefficient for the Rene 80 heat-resistant alloy. The dependences were taken using the noncontact method described in this study. The chemical composition of the alloy is presented in Table.

The alloy heat treatment process involved heating to 1200°C and keeping the specimen at this temperature for 30 min. The alloy was then cooled to 750 °C at a rate of 2.4°C/min and kept at 850°C for 50 h. This heat treatment resulted in formation of a two-phase ($\gamma + \gamma'$) structure: the γ phase has a FCC lattice where the atoms of each alloy component can occupy any position, i.e. the phase is disordered; while the γ' phase also has a FCC structure but with Ni and Al atoms occupying strictly specific sites in the crystal lattice, i.e. the phase is ordered.

The temperature dependence of the relative elongation for the Rene 80 alloy is shown in Fig. 3.

The temperature dependence of the thermal linear expansion coefficient for the Rene 80 alloy is shown in Fig. 4.

Heat-resistant Ni-based alloys were developed for applications in gas-turbine and turbojet engines. These alloys are two-phase ($\gamma + \gamma'$) systems in a certain temperature interval.

Table. Composition of Rene 80 Ni-based

Alloy	Mass fraction, per cent										
	C	Co	Mo	Al	Ti	Nb	Ta	Cr	W	P	S
Rene 80	0.16	9.34	3.94	3.00	5.00	0.01	0.02	13.84	4.00	<0.005	0.0006
Ni-based											

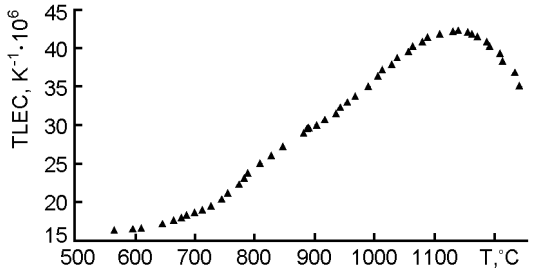

Fig. 4. Temperature dependence of TLEC for Rene 80 alloy.

The disperse crystals of the γ' phase strengthen the alloy, since the motion of dislocations therein is connected with the antiphase boundary formation. As a result, the γ' phase crystals are strained by paired dislocations. If the dislocations cannot cross the γ' crystals or carbides of refractory metals, the straining proceeds by the Orovan mechanism, also resulting in the alloy hardening. To raise the working temperatures (>1000 °C), Co is introduced into the Ni base. To harden the alloy, W and Mo are added, which react with carbon and form carbides of these refractory metals [3]. The Rene 80 alloy studied here belongs to this group.

The temperature dependence of the relative elongation of the Rene 80 alloy with initial two-phase structure containing about 50 % of γ' (Fig. 3) exhibits a pronounced hysteresis which is due to the γ' phase dissolution at heating and the formation thereof at cooling. The TLEC temperature dependence shows a distinct temperature interval where the γ' phase dissolution occurs at heating (860 to 1200)°C (Fig. 4). It is interesting that the alloy TLEC increases sharply as the γ' phase is dissolved. This is because the lattice parameter of the γ' phase is smaller than that of the γ one. As a result, during the $\gamma' \rightarrow \gamma$ phase transformation, the specimen volume increase due to the formation of the γ phase is superimposed on its thermal expansion. Above 1100°C, the TLEC decreases, presumably due to the γ' phase dissolution at the stage of the process completion is slowed down.

Thus, in the study, the thermal linear expansion coefficients of Rene 80 alloy in the specified temperature interval has been evaluated. The data obtained can be used in computation of welding-caused stresses and strains.

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Безконтактний метод визначення термічного коефіцінта лінійного розширення різних матеріалів в інтервалі температур 500–2500°C

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Розроблено безконтактний метод визначення термічного коефіцієнта лінійного розширення (ТКЛР) вуглеграфітових, керамічних матеріалів і жаростійких сплавів в інтервалі температур 500–2500°C. Наведено схему установки і показано її функціональні можливості. Виміряні значення ТКЛР еталонів (електролітичного нікелю і промислового графіту АРВ) відрізняються від довідкових літературних даних не більш, чим на 5 %. Наведено експериментальні дані з температурної залежності відносного подовження і термічного коефіцієнта лінійного розширення жаростійкого нікелевого сплаву Rene 80 в області температур 500–1200°C. На залежності $\Delta l/l(T)$ виявлений гістерезис, який зв'язаний з утворенням при охолодженні і розчиненням при нагріванні впорядкованої γ'-фази (Ni_3Al).