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DEVELOPMENT OF A NEW LASER INTERFEROMETRIC DILATOMETER AND ITS APPLICATION TO LOW EXPANSION MATERIALS

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ABSTRACT

The newly developed laser interferometric dilatometer permits measurement of absolute expansion of low expansion materials with high accuracy and high reproducibility, which has been difficult to measure by the conventional rod type dilatometer.

INTRODUCTION

Methods of measuring thermal expansion include push rod type dilatometry, optical interferometry, X-ray diffraction, micrometric telescopy, and others. Of these methods, optical interferometry is believed to provide the highest accuracy. However, the thermal dilatometer based on optical interferometry requires a high degree of skill and accuracy in forming the sample. A number of prototypes have been developed and commercialized, but it is not easy to form the sample and the instrument is very awkward to use. Consequently, the thermal dilatometer based on optical interferometry cannot be used in physical research and quality control except in a limited field of research. The authors have developed an ultrahigh-accuracy thermal dilatometer using stabilized laser beam and applied it to the measurement of a variety of low expansion materials.

PRINCIPLES OF OPERATION AND FEATURES OF INSTRUMENT

Figure 1 shows the block diagram of the laser interferometric thermal expansion meter (called "LIX" in this paper) developed by the authors.

The ultrahigh stability He-Ne laser beam emanating from the laser interferometer (1) enters the sample assembly (3) and is reflected at the two parallel mirrors between which the sample is sandwiched. The interference fringes obtained here enter the precision thermal expansion measurement circuit (2) and

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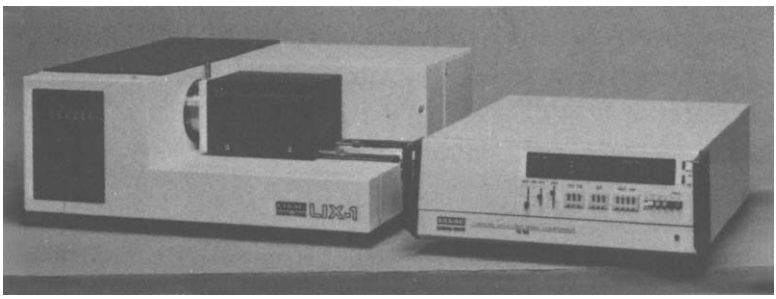

Photo 1. External view of the equipment (model LIX-1)

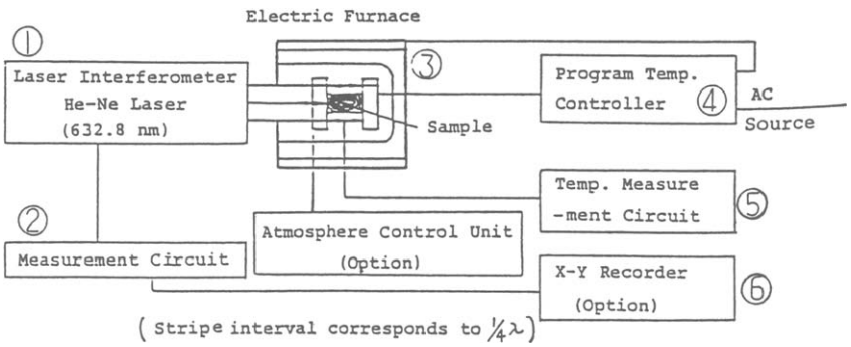

Fig.1 Block diagram of LIX-1

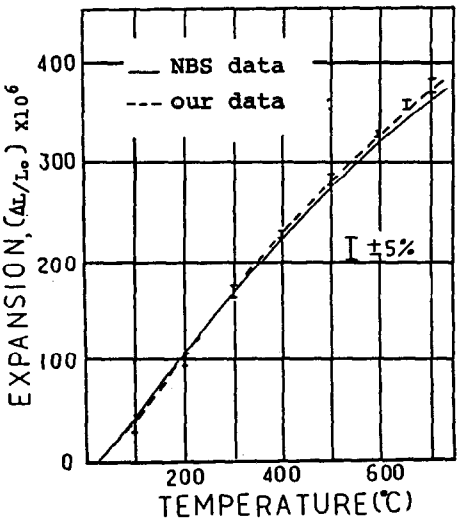

Fig.2 Measuring results of NBS fused quartz glass

are projected onto the image sensor. This image is counted once every 0.5 second, processed by a microcomputer, displayed in digital form in real time with an accuracy of 0.02 μm, and recorded on recorder (6) simultaneously. In this block diagram, numeral (4) represents the program temperature controller, (5) the sample temperature measuring circuit and (7) the atmosphere control unit. Measurement is made in vacuum (in He gas atmosphere in the case of the low temperature type). Specifications of this instrument are as follows.

(1) Temperature range : -150°C to 700°C
(2) Measurement method : dual beam laser interferometry
(3) Standard sample size : 6 to 7 mm OD, 1 to 15 mm long
(4) Measurement time interval : twice/sec
(5) Expansion measurement accuracy : ±3%, ±0.2 μm
(6) Detecting sensitivity : 0.02 μm
(7) Heating rate : 2°C/min, maximum 10°C/min
(8) Temperature measurement accuracy : ±0.5%, ±1°C

Photo 1 shows the external view of this instrument.

Features of this instrument include the following:

(1) Use of an ultrahigh stability gas laser and optical system permits reading the dimensional change of the sample with an absolute accuracy of approximately 200 Å, which is equivalent to 1/32 of the wavelength of the gas laser beam used.
(2) Employment of the mirror parallel movement mechanism with zero static friction has eliminated the need of precision machining of the sample. All needed for sample preparation is just to round its end surfaces by machining so that they contact the mirrors at a point. The number of sample needed is just one.
(3) The standard instrument permits measurement in a constant rate heating and cooling cycle at a maximum rate of 10°C/min from room temperature to 700°C and the change in length can be displayed digitally in real time.
(4) The absolute percent of expansion of low expansion materials and thin sheet materials can be measured, which cannot be measured accurately by the conventional technique.
(5) High resolution measurement can be made by receiving the image of interference fringes by an image sensor and processing the signal by a microcomputer.
(6) The polarity whether change in length represents expansion or shrinkage can be decided in real time.
(7) Elongation is automatically calibrated in real time by using the laser

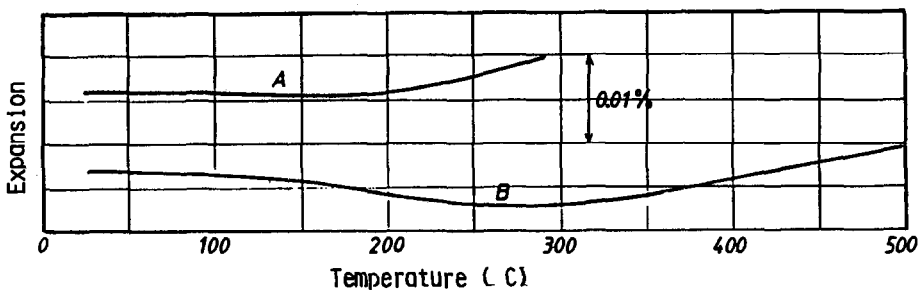

Fig.3 Thermal expansion behaviors of "ZERODUR" glass
A: as-received B: slow cooling after 500°C x 1 hr heating

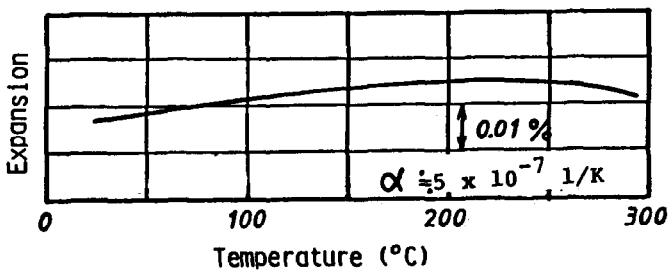

Fig.4-A

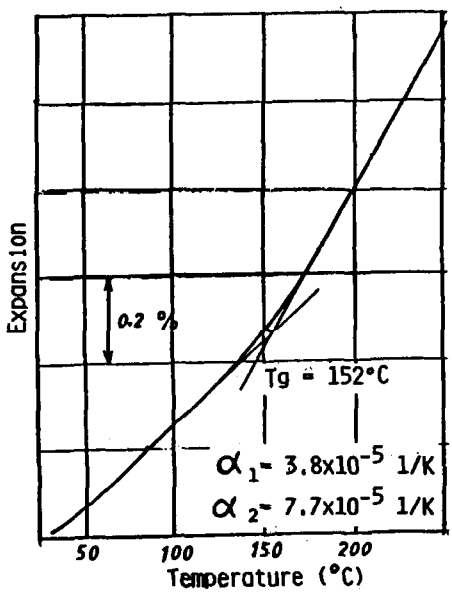

Fig.4-B

Fig.4 Thermal expansion of unidirectional carbon fiber reinforced PEEK.

Fig.4-A: fiber direction
Fig.4-B; perpendicular to fiber direction

wavelength as the reference. Unlike the push rod type dilatometer, this instrument requires no calibration by a micrometer.

EXPANSION MEASUREMENT BY "LIX"

The characteristics of "LIX" can be fully exhibited in the measurement of materials with small percent of thermal expansion, such as quartz glass, low expansion glass, carbon material, amber, and others. Figure 2 shows the measurement result of NBS fused quartz glass standard sample obtained from room temperature to 700°C and the NBS data for comparison. This figure indicates that this instrument can determine the absolute value of thermal expansion coefficient of a very low expansion sample such as quartz glass or others with an accuracy of ±5% and a reproducibility of ±3% in a vacuum. The size of the sample used in this measurement is 10.36mm long by 6.3mm in diameter and the heating rate is 10°C/min.

Figure 3 shows the thermal expansion behaviors of low expansion glass (partially crystallized glass made by Schott, trade name "Zerodur"). In this figure, A represents the as-received sample and B the as-received sample which was held at 500°C in air for one hour and then slowly cooled. The mean thermal expansion coefficient of sample A from room temperature to 150°C is approximately 1×10^{-8}/°C. When this glass is heated to above 200°C, the crystallized portion is unbalanced and the glass exhibits behaviors like those of B.

Figure 4 shows the thermal expansion behaviors of PEEK resin (volume fraction of fiber 70%) reinforced with unidirectional carbon fiber, in the direction of fiber (Fig. 4-A) and in the direction perpendicular to it (Fig. 4-B).

The thermal expansion coefficient in the direction perpendicular to fiber is approximately 60% that of PEEK resin alone. On the other hand, the thermal expansion coefficient in the direction of fiber is two digits smaller than that and its mean thermal expansion coefficient from room temperature to 200°C is approximately 5×10^{-7}/°C.