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A Focused-Beam Type Laser Interferometric Dilatometer

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Using a laser beam focused near interferometer plates, an improved interferometric dilatometer has been developed. It has a couple of features, i.e. it has a much greater latitude in parallelism adjustment of the interferometer plates than that of the conventional Fizeau-type ones and enables, on the other hand, automatic recording with a simple device just composed of a solar cell and a recorder to measure reflected beams. Moreover, collimation of the optical system is very easy. Therefore, it is applicable even to routine determination of dilatation for quality control in a factory. The dilatometer is capable of determining the thermal dilatation of materials with low and/or negative expansion as well as those with transition, extremum or inflection point in expansion curves. Experimental results on some metallic, ceramic, semiconductor and glassy materials are presented.

§1. Introduction

Hermetic and vacuum-tight sealing is of vital importance in electronic industry. Thermal expansion characteristics are the key to select the materials for seals. The Fizeau-type interferometric dilatometer[1,2] is suitable for the most precise measurement to evaluate dilatometric properties of materials for the following reasons: 1) it is applicable to small components, and 2) thermal expansion is measured very precisely referring to the wavelength of monochromatic light.

The dilatometer is based on the measurement of interference fringes of equal thickness formed by an interferometer, the two plates of which are held apart by a specimen or a set of specimens with the separation of 2 to 15 mm in most cases. The fringes move with dilatation of the specimen.

In the actual measurement, the movement of the fringes must be watched continuously by an operator. Automatic recording has required time consuming or extremely sensitive devices such as photographic films[3,4] or photomultipliers.[5] Even lasers have been used as light sources instead of conventional discharge lamps.

The authors have developed a new type of interferometric dilatometer using a focused laser beam. This improves conventional dilatometer in the following respects: 1) automatic recording is possible simply with a silicon solar cell and a recorder, 2) the latitude in the angle between two interferometer plates is greater, and 3) collimation of the optical system is very easy.

§2. Apparatus

The optical system is shown in Fig. 1. A specimen to be measured is sandwiched between two optically polished vitreous silica plates. The specimen is shaped by grinding, so that two surfaces in contact with the specimen are nearly parallel to each other. A ring-shaped specimen with three points contact is shown. The specimen S and a diagonal reflecting prism are placed at the focal points of a convex lens L. The beam B of a He–Ne gas laser is introduced into the dilatometer through the prism

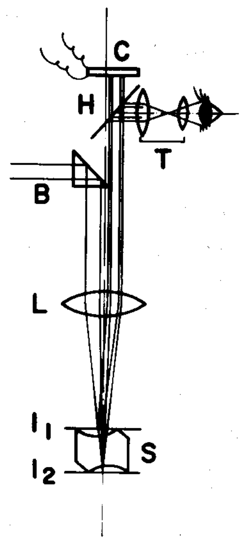

Fig. 1. Light path in the focused-beam type laser interferometric dilatometer.

and is focused by the lens L at an intermediate point between the interferometer plates. The beam B is reflected from the bottom surface I_1 of the upper plate and from the top surface I_2 of the lower one. These two reflected beams interfere with each other at the position where they are superposed and form a bright or dark fringe depending on the optical path difference between the two beams, or in other words, the dimension of the specimen at the position where the beam is focused.

These are again made into mutually parallel beams, respectively, by the lens L. They continue to interfere with each other in the region of superposition. The total luminosity of the beams, which changes with the optical path difference, is measured by a solar cell C. A small part of the beams is reflected from a thin glass plate H and is introduced into a telescope T. The telescope adjusted for infinity enables the visual observation of interference phenomenon between the reflected beams near the specimen S.

The methods of specimen preparation and measurement are almost identical with those in conventional Fizeau-type dilatometer.[6-8] They are not repeated here.

The reflected beams can interfere with each other as long as the angle between the two plates is less than 9 min when the focal length of L is 240 mm and the diameter of B is 1.2 mm. On the contrary, in conventional Fizeau-type dilatometer, the angle of 9 min gives the fringe spacing of 0.12 mm; the fine fringe spacing causes severe difficulty in both visual observation and automatic recording.

The incident and reflected laser beams in the dilatometer give tiny spots due to scattering on the prisms, lenses, interferometer plates, cover glasses of a furnace or a cryochamber and so on. This makes the collimation very easy. To confirm the completion of collimation, a light diffuser may be inserted temporarily in the path of incident beam adjacent to the diagonal prism to see if a fringe pattern similar to that of conventional Fizeau-type dilatometer is produced (see Fig. 2).

On heating or cooling specimen, the output of the solar cell gives ordinarily wave-like record with time on recording chart (Fig. 3); one cycle corresponds to a half wavelength of the laser light in dilatation (expansion or contraction) of the specimen or to one cycle in fringe motion in conventional Fizeau-type dilatometer.

Fig. 2. Interference fringes given by insertion of light diffuser in the path of incident beam.

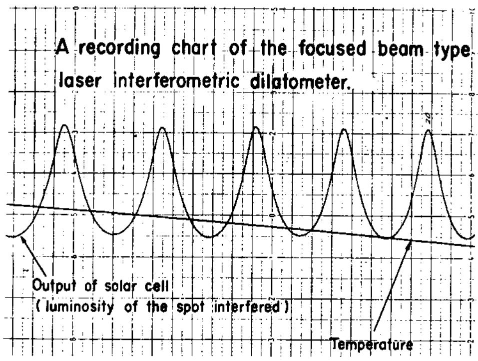

Fig. 3. A recording chart of the dilatometer.

The determination of the sign of dilatation is possible by the following means. Visual observation through the telescope T shows a bright spot with fine structure in luminosity distribution (Fig. 4). The pattern corresponds, as described before, to the interference phenomenon near the interferometer plates and the specimen. The fine structure changes cyclically with time during measurement with the appearance, passage and disappearance of dark areas. The direction of passage corresponds to the direction of fringe motion in conventional dilatometer. Continuous recording by the cell C and, if necessary, occasional observation of the pattern through T confirm the direction of fringe motion and the reversal in the sign of expansion coefficient.

Specimen and the interferometer plates are placed in a thick copper container to ensure temperature uniformity. They are placed in an electric furnace or in a vacuum cryochamber.

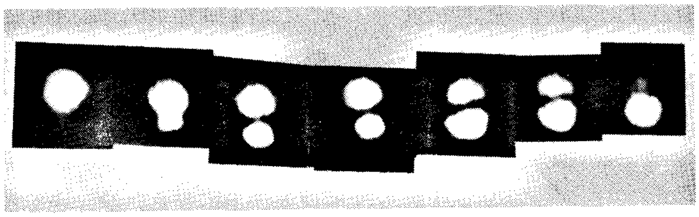

Fig. 4. One cycle of the change of the interference pattern in a spot seen by the telescope.

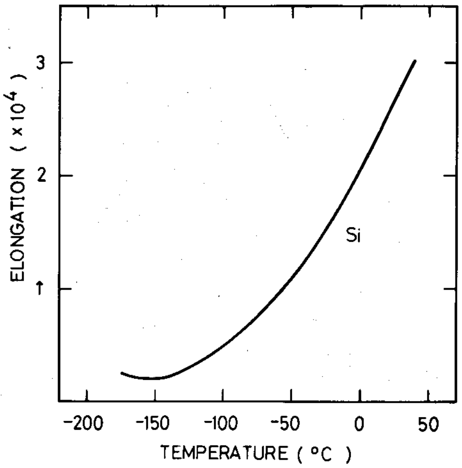

Fig. 5. Dilatometric curve of single crystal silicon along ⟨111⟩ direction.

Special precaution is paid in the construction of the chamber to prevent frosting on the interferometer plates.

Air correction, which is the correction term to compensate the effect of wavelength change with temperature in calculating dilatation, is applied by assuming that the refractive index of air is inversely proportional to absolute temperature. In the case of vacuum cryochamber, air correction is unnecessary.

§3. Experimental Results

3.1 *Silicon*

The dilatation along ⟨111⟩ direction of a silicon single crystal was measured (Fig. 5). The growth direction of the crystal was also ⟨111⟩ axis. The reversal in the sign of expansion coefficient occurs at about -150°C.

3.2 *Germanium*

The dilatation along ⟨111⟩ direction of a germanium single crystal is presented in Fig. 6, which shows the expansion curve in two parts measured separately in high and low temperature regions.

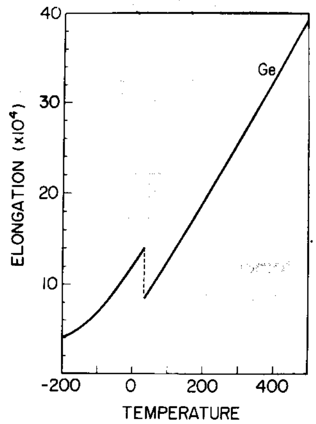

Fig. 6. Dilatometric curve of single crystal germanium along ⟨111⟩ direction, which was separately measured for high and low temperature regions.

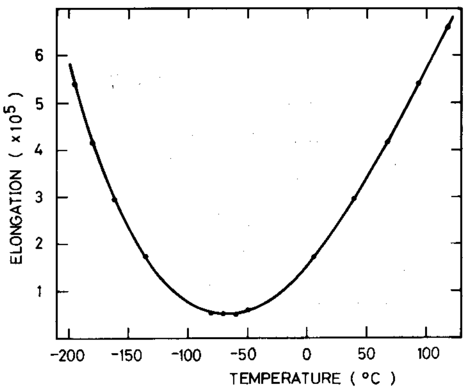

Fig. 7. Dilatometric curve of vitreous silica (fused quartz).

The results on the silicon and germanium single crystals are consistent with those reported by Gibbons.[9]

3.3 *Vitreous silica* (fused quartz)

The dilatation of a fused quartz specimen is shown in Fig. 7. The reversal in the sign of expansion coefficient occurs near -70°C.

3.4 *Miscellaneous*

Figure 8 shows the results on Pyrex-type

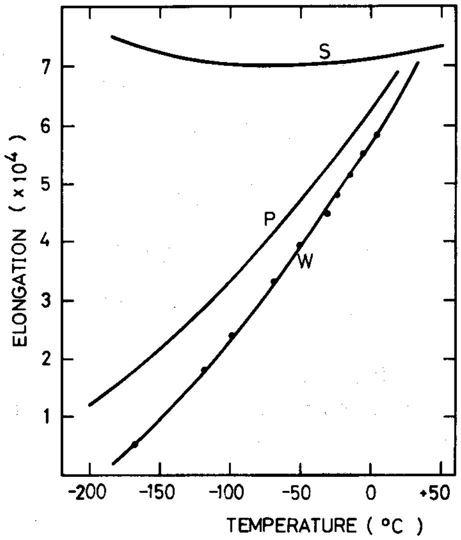

Fig. 8. Dilatometric curve of fused quartz(S), borosilicate glass(P) and lead borosilicate glass(W).

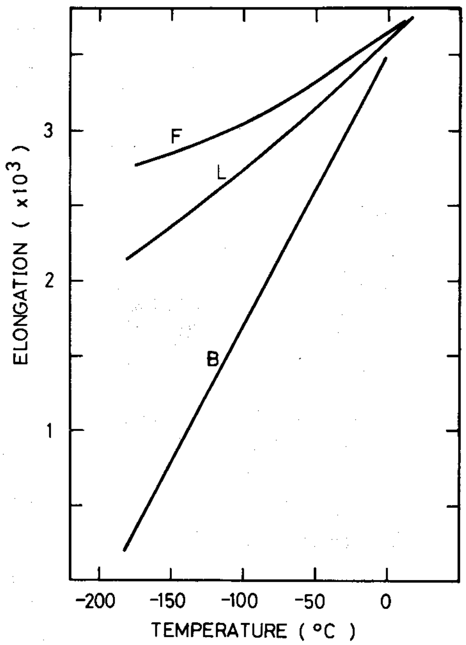

Fig. 9. Dilatometric curve of ferrite(F), lead potash silicate glass(L) and brass(B).

borosilicate glass (P) and tungsten-sealing lead borosilicate glass (W).

Figure 9 shows the results on sintered ferrite ceramic (F) used for the deflecting yoke of a television picture tube, dumet-sealing soft lead potash glass (L) and brass (B). They were measured to evaluate mechanical stress in the picture tube yoke system caused by the differential dilatation of the components in low temperature regions.

Average thermal expansion coefficients of the materials are given in Table I.

§4. Conclusion

The focused-beam type laser interferometric dilatometer is suitable for the characterization of materials. It permits a greater latitude in parallelism when specimens are prepared. Therefore, it is applicable not only to precise laboratory measurement but also to routine measurement for quality control in a factory. It can also be used for materials with low expansion and for those with transition, extremum or inflection point in their thermal expansion curve.

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Table I. Average thermal expansion coefficients (in $10^{-7}/°C$) of metal and ceramic materials. Minus sign means contraction with increasing temperature.

Specimens	Temperature range (°C)				Note*	
	−190∼0	−150∼0	−100∼0	−50∼0		
Si ⟨111⟩	—	12.3	14.6	18.1		
Ge ⟨111⟩	42.1	47.5	52.1	55.0		
Cu	140	161	174	183		
Brass	—	181	—	—		
Vitreous silica	−1.8	−0.53	0.76	1.9		
					TOSHIBA	CGW
Pyrex type borosilicate	25.7	27.3	29.5	30.8	#050,	7740
W-seal (nonex) glass	29.7	31.6	33.7	35.4	#070,	7720
Lead potash glass	79.4	82.0	85.0	86.0	#252,	0120

*Code number in Toshiba and the corresponding code number in Corning Glass Works.

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