N94-31851

(NASA-CR-195911) ELASTIC MODULUS
MEASUREMENTS OF LDEF GLASSES AND
GLASS-CERAMICS USING A SPECKLE
TECHNIQUE (Vanderbilt Univ.) 7 p

Unclas

G3/27 0009435

NASA-CR-195911

NAG 8-156

N/S

IN-27-CR

9435

P-7

ELASTIC MODULUS MEASUREMENTS OF LDEF GLASSES AND GLASS-CERAMICS USING A SPECKLE TECHNIQUE

D. E. Wiedlocher and D. L. Kinser
Department of Applied Science and Engineering
Vanderbilt University
Nashville, TN. 37235

Abstract

Elastic moduli of five glass types and the glass-ceramic Zerodur[1], exposed to a near-earth orbit environment on the Long Duration Exposure Facility (LDEF), were compared to that of unexposed samples. A double exposure speckle photography technique utilizing 633 nm laser light was used in the production of the speckle pattern. Subsequent illumination of a double exposed negative using the same wavelength radiation produces Young's fringes from which the in-plane displacements are measured. Stresses imposed by compressive loading produced measurable strains in the glasses and glass-ceramic.

Introduction

The Long Duration Exposure Facility (LDEF)which orbited the Earth in a 400 km orbit carried 120 glass and glass-ceramic samples of compositions listed in Table I. Sixty eight of the 120 samples were exposed to an environment which received 9,600[1] hours of solar radiation.

Speckle effect refers to the grainy appearance of an object illuminated by a coherent light source[2]. A speckle pattern forms from the interference of reflected light from different depths on an objects surface. The pattern acts as a grid which may be used to produce a moire. A method introduced by Leendhertz[3] uses a double laser beam technique resulting in real time measurements of surface contours. The method depends on the beam angles and is sensitive to out-of-plane displacements. A method relatively insensitive to out-of-plane displacement introduced by Archbold[4] uses a single laser beam. The method utilizes the speckle ruling captured on a photographic plate produced from the interference of the reflected light. Deformation of this grid due to the displacement of the surface speckle superimposes on the undeformed grid to produce a moire. This maybe done by doubly exposing the deformed and undeformed speckle pattern to the same film. The moire constructed on the negative produces lines acting as slits, and when re-illuminated with coherent laser light produces Young's fringes in the diffraction halo of the laser beam.

Speckle techniques have the advantage of measuring displacements to 1 um[5] depending

[1]Schott Glass Technologies Inc.

on the technique. Measurements depend on the magnification and resolution of the photographic process. Differentiation of two speckle patterns is determined by speckle size on the photographic plate. Movements must be greater than the speckle diameter in order to distinguish between speckle patterns doubly exposed on the plate. The recorded speckle size may not be smaller than the grain size of the film and is therefore dependent on film resolution and magnification.

Experimental Procedure

Five glass samples and one glass-ceramic sample exposed on LDEF, and the corresponding control samples designated for mechanical properties studies, were used in this experiment. The samples, originally 32 mm x 5 mm discs, were centrally loaded to fracture as described by Wiedlocher[6] et. al. producing pie shaped fragments. Fragments from the samples were joined with a cyanoacrylate adhesive to produce a sample with a 10 mm thickness. Three control and three exposed samples of approximately 3 mm x 10 mm were extracted using a 3 mm water-cooled diamond-tip core drill. The samples required a gold surface coating to eliminate back surface interference, insuring the speckle pattern was due to reflection from the front surface of the sample.

Sample length and diameter was measured to the nearest tenth millimeter. The samples were compressively loaded between a fixed anvil and a pneumatically driven piston in an apparatus secured to an optics table. Total Pressure applied to the samples was measured within 6 MPa. A strain gage attached to the test fixture allowed variation of pressure between loading of the samples to less than 2.5 MPa. The sample chamber was illuminated by a divergent He-Ne laser beam produced by placing a 15 um aperture in the beam path. A 2x camera utilizing Polaroid type 55 positive/negative film was placed at an angle 30° to the beam direction. Each specimen was placed on the center of the anvil and preloaded to 5 MPa above the measurement load. The pressure was then reduced to 3 MPa. The speckle pattern of the sample surface was photographed using a 150 mm lens and f/8 aperture setting. The same piece of film was exposed to the speckle pattern produced with the sample under a strain of about 5 ppm. Illumination of the doubly exposed negative with a 1 mm He-Ne beam produced diffraction fringes which were measured on the image screen at a distance of 5 m. Measurements were taken with the negative illuminated at points near the end of the sample image corresponding to the area of maximum displacement. The image of the sample on the negative was measured to within one beam diameter of the end corresponding to a distance of about 0.5 mm. A minimum of four fringe spacings were measured to the nearest 2 mm at an image distance of 5 m. The average fringe spacing was calculated for each of the six samples and the modulus of the samples determined.

Results

The difference in modulus between the solar exposed samples and the control samples was compared. Table II lists the modulus of each sample and the standard deviation along with the average modulus of the sample set. The greatest variation between measured modulus and values found in the literature occurred for the Zerodur and soda-lime-silica samples. The accepted value for the elastic modulus of Zerodur at room temperature is 90.2[7] GPa, thus there is an error of 4.1% between the accepted value and the value measured for the control samples. The solar exposed samples were within 3.5% of the accepted value. Soda-lime-silica had the largest percent error of 7.6%; however, the measured modulus of both the Zerodur and the soda-lime-silica exposed samples was within 1% of the modulus measured for corresponding control samples. Moduli determined for the remaining glass types were within 2% of the accepted value.

Discussion

Literature concerning speckle techniques have described the ability of the method to measure displacements of 1um. This is dependent on the resolution of the film, quality of the camera lens, and the magnification. Film with a resolution of 150 lines/mm requires the displacement of a single speckle to be greater than 6 um, at unit magnification. The size of the speckle imaged on the negative is also a function of the aperture. The speckle size σ is a function of the aperture ratio F, magnification M, and wavelength λ by the equation given by Archbold and Ennos[8] as:

$$\sigma \approx 1.2\lambda F$$

The speckle diameter for 633 nm light transversing a f/8 aperture with magnification of 2 is about 6 um. Speckle intensity is a function of the aperture size, however; this is restricted by the resolution of the film. Archbold[3] suggests for He-Ne illumination that the aperture should be no larger than 1300/F lines per millimeter. An aperture size of f/8 produces speckle sizes of 160/mm comparable to the resolution of Polaroid type 55 film.

The area of the sample from which the measurement is taken is reduced as the image size increases. Magnification of the speckle increases the width of the grid lines produced on the negative. Intensity of higher order diffraction fringes is reduced due to the diffraction modulation term dependent on slit width, which in this case is the width of the grid lines. Reproducible 2nd order fringes were observed in this experiment for stresses of 400 MPa. This allows a single fringe spacing to be more accurately determined by averaging over 4 spacings. Strain in glasses with a modulus on the order of 80 MPa or less produced higher order fringes in the diffraction halo, which improved the accuracy of fringe spacing measurement.

The determination of the amount of strain depends on the measurable displacement and thus the resolution of the film used to produce the transparency. Since polaroid type 55 film is limited to 5 um resolution, strains in a 10 mm sample can be determined to +/- .25 um/mm. This corresponds to an error in modulus of +/- 4.7 GPa; hence, there is a 10% margin of error in the measurement. To be able to reduce this boundary a higher resolution film must be utilized.

Effects of radiation on the modulus of glasses and glass ceramics have been studied by Higby et. al[9] using 2 x 10^9 doses of 10 MeV electrons. Changes in elastic modulus were found to be at the most 0.5%, which is within the detection limit of the speckle technique. Studies[10] of the radiation environment on the trailing-edge of LDEF for the 5.8 year duration of orbit suggest that high energy particle radiation was in doses on the order of a few hundred rads. Jaffe and Rittenhouse[11] found ionization effects occurred in glasses for doses greater than 10^3 rads. This suggests that the trailing-edge environment of LDEF exposed the glasses to radiation doses less than that required to produce measurable changes in elastic modulus.

Conclusions

The elastic moduli of the six glass types and glass-ceramic exposed to a low earth-orbit solar radiation environment are affected less than 10% of the accepted value.
The speckle technique may be used to measure moduli of centimeter sized samples with moduli on the order of 100 GPa.
The limiting factor in the ability of the speckle technique to determine modulus is the resolution of the photographic plate used to record the speckle image.
The environment of the trailing-edge of LDEF exposed the glasses and glass-ceramic to radiation doses below that needed to produce measurable changes in elastic modulus.

Acknowledgements

The financial support of NASA for preparation and flight of samples under contracts NAS-8-32695, NAG-8-156 and analysis under L-17761D is gratefully acknowledged.

Table I
Nominal Compositions of Glasses in LDEF Experiment

Type	Comment	SiO_2	Na_2O	CaO	B_2O_3	Al_2O_3	Other
Fused Silica	Fused Natural Quartz	100	-	-	-	-	-
BK-7	Optical Crown	70	5.5	2.5	7.5	-	$15K_2O$
Pyrex	Corning 7740	81	4	-	13	2	-
Vycor	Corning 7913	96.5	-	-		0.5	
Soda-lime	ASG Low Fe	71	12	3	-	1	<0.05Fe
Zerodur	Schott Low α	57	0.7	2.0	-	2.5	$3TiO_2$

Table II
Elastic Modulus of LDEF Glasses Determined from Speckle Techniques

Sample	Accepted Modulus (GPa)	Experimental Modulus (GPa)	Standard Deviation (GPa)	Percent Error
Zerodur				
Control	90.2	86.5	0.4	4.1
Exposed		87.0	0.6	3.5
Pyrex				
Control	65.5	66.5	0.4	1.5
Exposed		65.2	0.6	0.5
Fused Silica				
Control	73.1	74.0	0.8	1.3
Exposed		73.5	1.3	0.6
Vycor				
Control	46.2	46.5	0.5	0.7
Exposed		46.7	0.3	1.1
BK-7				
Control	80.7	81.5	1.9	1.0
Exposed		82.1	1.0	1.8
Soda-lime Silica				
Control	73.8	68.7	0.7	6.9
Exposed		68.2	0.7	7.6

References

1. LDEF Spaceflight Environmental Effects Newsletter, Summary Issue, Vol. II, No. 3, Code 720, NASA, GSFC, Greenbelt, Md., June 15, 1991.

2. R. K. Erf, Speckle Metrology, Academic Press, New York (1978).

3. J. A. Leendhertz, "Interferometric Displacement measurement on Scattering Surfaces Utilizing the Speckle Effect",Journal of Physics E: Scientific Instruments, Vol. 3, Great Britain (1970).

4. E. Archbold, J. M. Burch, and A. E. Ennos, "Recording of In-Plane Surface Displacement by Double-Exposure Speckle Photography", Optica Acta, Vol. 17, No.12, 883-898 (1970).

5. S. P. Mukherjee and D. Suryanarauana, "Sol Gel processing in electrical packaging materials, "Jnl. Non-Xtal Solids., 147/8, 783 (1992).

6. D. E. Wiedlocher and D. L. Kinser,"Low Earth-Orbit effects on strength of Glasses", J. Am. Ceram. Soc., 75, Nov. (1992).

7. Schott Glass technical bulletin

8. E. Archbold and A. E. Ennos, "Displacement Measurement from Double Exposure Laser Photographs", Optica Acta Vol. 19(4), 253-271 (1972).

9. P. L. Higby, E. J. Frieble, C. M. Shaw, M. Rajaram, E. K. Graham, D. L. Kinser, and E. G. Wolff, "Radiation Effects on the Physical Properties of Low-Expansion-Coefficient Glasses and Ceramics", J. Am. Ceram. Soc., 71 [9] 796-802 (1988).

10. J. Watts, Marshall Spaceflight Center, Private Communication, Aug. 1991.

11. L. D. Jaffe and J. B. Rittenhouse, Am. Rocket Society., 32, 320 (1962), Cited from R. A. Becker, "Optical Material Problems of Interplanetary Space", Applied Physics, 6 [5] 2561-2565 (1962).