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Ultrahigh precision measurements of optical heterogeneity of high quality fused silica

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We present ultrahigh precision measurements of the optical heterogeneity of high quality fused silica at the $\delta n = 10^{-8}$ index level. The measurements were made with a unique scanning heterodyne interferometer developed as part of the Gravity Probe-B Program. Striae of refractive index variation $\Delta n < 10^{-7}$ (optical path difference smaller than 5 nm) are clearly visible on an extended heterogeneity background of around $\Delta n = 6\times 10^{-7}$. Both striae and extended heterogeneities are repeatable to within a few parts in 10^8. These results show the nature and extent of the heterogeneities of high quality vitreous silica. © *1995 American Institute of Physics.*

The optical heterogeneity of high quality glass (including vitreous silica) has long been of importance to the manufacture of high precision optical components, glass manufacturers, and the modeling of the bulk properties of glass. Of the three major types of inhomogeneities, namely, microheterogeneities, striae, and extended inhomogeneities, only the latter type is thought to be significant for high quality glass at the $\Delta n = 10^{-7}$ index level after very careful preparation procedures.[1] Extended inhomogeneities appear as small refractive index gradients spreading smoothly over a relatively large region of the material, and depend on the melting process, density gradients due to thermodynamic imbalance, and permanent stress resulting from temperature gradients during the annealing stage.[1] Striae on the other hand, are long streaks of generally higher index gradients than those found in extended inhomogeneities, while microheterogeneities are best detected through scattering experiments where the sizes of the heterogeneities must be comparable to the wavelength of the probing radiation.[2,3] As such, much of the inhomogeneity seen in high quality glass results from the glass production procedure, and is a record of its history of formation which has been frozen into the glass itself.

The uniformity of the refractive index is a major factor in determining the performance of all optical imaging and telecommunication systems,[4] thus there is a constant effort by glass manufacturers to improve on homogeneity,[5] by material scientists to model its properties,[3–8] and by optical scientists to improve on heterogeneity measurement.[9–11] In some special circumstances, where the highest homogeneity is required, such as in the manufacture of extremely uniform gyroscopes for the Gravity Probe-B satellite test of general relativity,[12] new techniques must be developed to improve the measurement precision δn, nominally 10^{-7}, by a factor of at least 10. This letter reports ultrahigh precision measurements of the variation of refractive index in quality fused silica samples at the $\delta n = 10^{-8}$ level. The measurements reported here were made on a unique scanning interferometer designed to screen gyroscope blanks for the Gravity Probe-B experiment.[13] A significant advantage of our approach over almost all other interferometric methods is that we measure Δn, the refractive index difference directly over a regular grid, obtaining in the process, a true picture of the refractive index gradient (or tilt information); tilt information is often lost in other approaches, e.g., by arbitrary manipulation to improve fringe spacing in traditional interferometers.

The homogeneity instrument is a dedicated computerized narrow beam scanning, heterodyne interferometer. It is built around the Hewlett-Packard 5526A (helium–neon 632.8 nm) laser measurement system, and incorporates an HP 217 computer for data acquisition and stepper motor control. A schematic of the instrument is shown in Fig. 1. We have tapped signals from the laser head and fed these into a custom-built analog phase meter with an observed resolution of 0.2 nm. The thermal coefficient of refractive index of fused silica is around 10^{-5} K^{-1}, so in order to control thermal gradients and drifts it is necessary to maintain the immediate environment of the interferometer to within a few m °C of a preset (index matching) temperature. This has been achieved by building an active servocontrolled thermal enclosure around the interferometer.[13] Moreover, the reference arm has been carefully designed to compensate for thermal drifts in the measurement arm, further reducing sensitivity of the instrument to small temperature drifts. The surface features of the sample are removed by immersing it in 1,8-cineole, which matches vitreous silica in refractive index at about 17.49 °C. Scanning is achieved by moving the sample alone, in the form of a 50 mm sized cube, across the beam incident normally to the face of the cube, thus rendering the instrument insensitive to spatial variations in all other optical components. The sample and index matching liquid appear to be in full thermal equilibrium after 2 days within the ther-

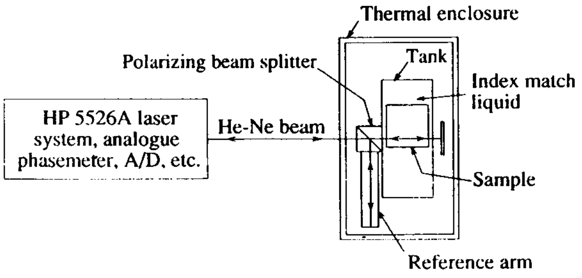

FIG. 1. Experimental setup.

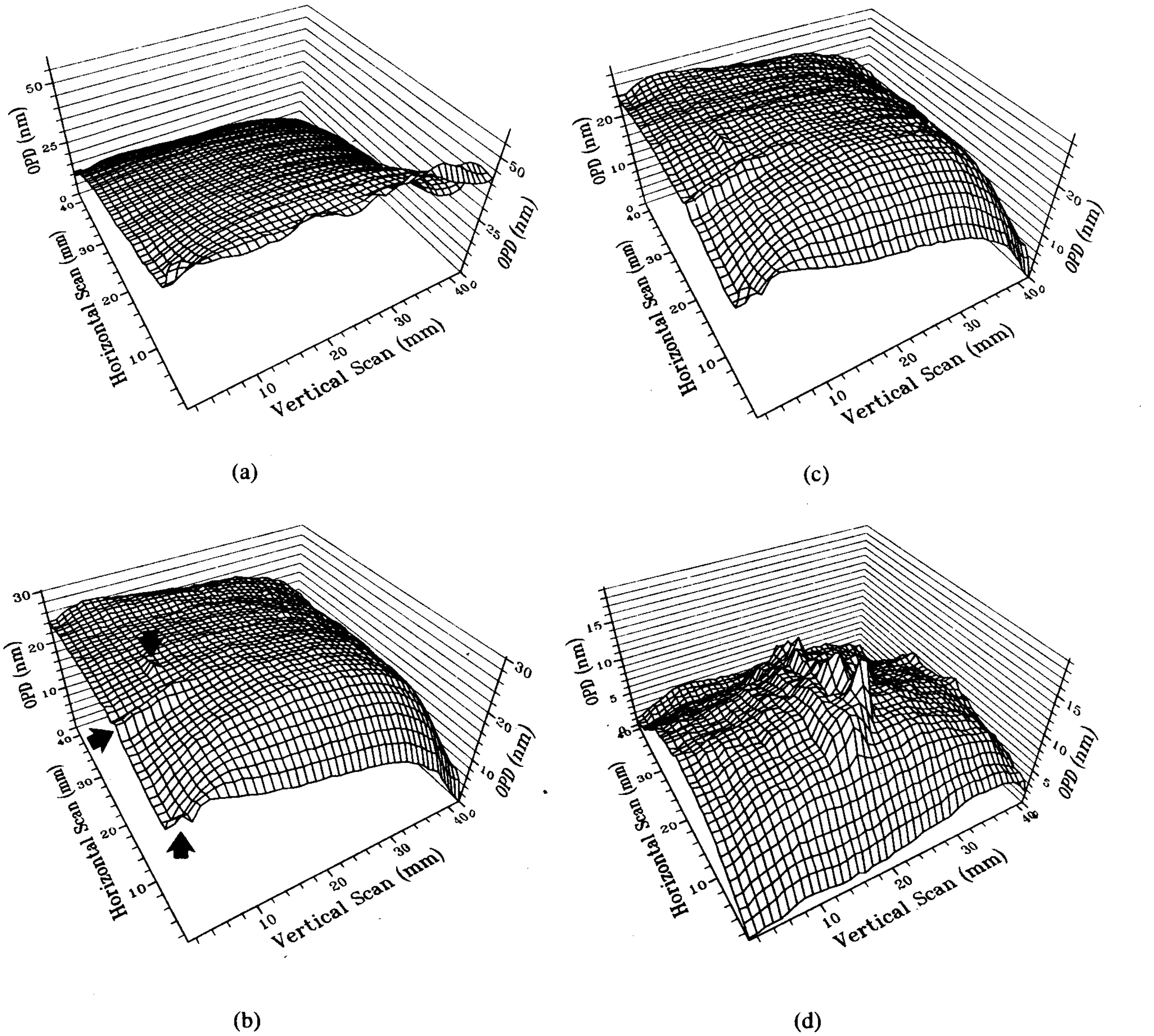

FIG. 2. Optical path difference of block 4. (a) Direction I, (b) direction II, (c) rescan of direction II showing repeatability of nanofeatures, (d) direction III. The origin of horizontal and vertical axes is at the top left-hand side corner of each scan. The right-hand side edge of direction I corresponds to the left-hand side edge of direction II. The top edge of direction II corresponds to the bottom edge of direction III, while the top edge of direction I is the left-hand side edge of direction III. The three directions "see" mutually orthogonal planes.

mal enclosure. The two-dimensional integrated refractive index variation $\Delta n(x,y)$ is given by

$$\Delta n(x,y)=\frac{1}{L}\int_0^L [n(x,y,z)-n_0]dz,$$

where L is the distance the beam travels through the cube in the z direction, n is the refractive index treated as a function of position in the block, and n_0 is the mean refractive index of the material.

High quality fused silica samples were obtained from Heraeus Quarzglas GmbH, Germany, with a Δn specification of 8×10^{-7}. Measurements on all samples were taken at around 17.5 °C with changes in ambient temperature and pressure of about 2 m °C and 2 mbar, respectively over one scan. Figures 2(a)–2(d) show scan results for one such sample. [To convert the optical path difference (OPD) in nm, shown in the figures, to refractive index difference Δn, multiply the OPD by 2×10^{-8}.] The first map in (a) obtained from direction I, shows a smooth variation in refractive index. The second map (b) (direction II) shows generally smooth features, but is punctuated by a ridge running along the vertical direction, starting near the center of the upper edge of the scan. Also notable are smaller 5 nm repeatable features—one being at the corner close to the origin, and the other just beside the ridge in the scan (these are indicated by arrows). Map (c) is a repeat scan of the sample from direction II, and shows the repeatable nanoscale features. Part (d) shows the third direction (III) with a repeatable structure within the central portion of the map; this structure has sharp spikes along one of its sides. Of the samples examined from the same batch, this is the only sample to show such striae; levels of extended heterogeneity are similar throughout.

However, striae will only be visible if fairly well-aligned with one of the directions of view, and will otherwise merge in with the extended heterogeneity. The existence of striae depends on a number of factors broadly covering the method of glass manufacture and impurity doping. In particular, for glasses formed from melt (rather than CVD), striae may arise from buoyancy convection during the melting process, such as those observed for fluoride glasses by West and Sen.[14] We cannot offer any immediate explanation for the striae observed in our samples, though the birefringent properties of the glass may provide further clues to the structures observed.

In summary, features at the 5 nm level of OPD ($\Delta n = 10^{-7}$ level) are clearly visible, and repeat at the $\delta n \simeq 10^{-8}$ level. Repeatability of spatially extended inhomogeneity is somewhat poorer, at δn a few times 10^{-8}. The operating principle of the scanning instrument makes it inherently insensitive to system errors, other than thermal drift, and in particular it gives a direct measure of index "wedge." At the levels of reproducibility demonstrated here, drift is consistent with residual changes of temperature of a few m °C over the sample.

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