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Structural, mechanical and optical studies on ultrafast laser inscribed chalcogenide glass waveguide

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Multi-scan waveguides have been inscribed in GeS_2 glass sample with different pulse energies and translation speeds. Mechanical and structural changes on GeS_2 binary glass in response to irradiation to 1047 nm femto-second laser pulses have been investigated. The optical characterization of these waveguides has been done at 1550 nm of laser wavelength and the material response to laser exposure is characterized by both nanoindentation studies and micro-Raman spectroscopy. Nanoindentation investigations show a decrease in hardness (H) and elastic modulus (E) upon laser irradiation. The change in E and H are found to be varying with the translational speed, pulse energy and hence the net-fluence at the sample. These changes are correlated with variations in the Raman response of photo-exposed glass which is interpreted in terms of structural modifications made by the laser inscriptions to the glassy network. The mechanical behavior and local structural changes on waveguide writing is found to be dependent on net-fluence and it is correlated with the preparation conditions like melt temperature and cooling rate.

1. Introduction

Chalcogenide glasses possess a good glass forming ability along with a relatively high glass transition temperature [1]. Their good infrared transmittance [2], high third-order optical nonlinearity [3,4] and low phonon energy [5] properties make them potential candidates for all-optical devices [6]. These glasses are photosensitive in nature and exhibit many photo-induced phenomena which include light induced change in local atomic configuration [7].

Photo-structural changes in chalcogenide glasses is an extensively investigated topic. Though the exact mechanism responsible for the photo-induced material modification in these glasses has not been completely understood, it is understood that the modifications are associated with changes in the fluidity of the glasses [8], density & refractive index [9], mass transfer [10] etc.

Laser writing using ultrafast lasers is a proven technique for making optical waveguides in transparent materials such as glasses and ceramics [11]. It reduces heat diffusion from the processed area to its surrounding regions, thereby enabling high quality micro/nano fabrication [12,13]. Direct laser writing allows the fabrication of integrated optical elements without the need for complex lithography and clean room facilities, making it cost effective. In direct laser writing, the multi-scan technique can provide waveguides with symmetric cross-section which is essential for a better performance [14]. The multi-scan technique builds up the waveguide cross-section over many scans, while keeping the peak intensity to a minimum. Hence, the number of scattering and absorbing defects induced in the modified material by the inscription process becomes less, thereby reducing the optical losses.

Earlier studies on the mechanical properties of single-scan chalcogenide glass waveguides reveal that the elastic modulus (E) and hardness (H) change under band-gap illumination and the changes are also position dependant [15,16]. Sulphur based chalcogenide glasses show two distinct regions with different values of E and H under bang-gap illumination [15,16]. A region is observed with lower values of E and H at the top of the inverted teardrop shaped waveguide, which has been attributed to annealing; while

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the rest of the waveguide shows *E* and *H* values of bulk glass, which has been attributed to quenching.

In the present work, mechanical behavior of the optical straight waveguides inscribed using multi-scan of femto-second laser pulses in GeS_2 glass is investigated by using the nanoindentation technique. The mechanical properties of GeS_2 glass are found to vary according to the net-fluence used in the waveguide inscription. The obtained mechanical behavior of the GeS_2 waveguide is correlated with the structural changes using micro-Raman spectroscopy and is found to be in good agreement.

2. Experiments

2.1. Glass synthesis

Bulk GeS_2 glasses have been prepared by vacuum-sealed melt quenching technique. Appropriate quantities of high purity (99.999%) constituent elements (Ge and S) are sealed in an evacuated quartz ampoule at 10^{-5} Torr and slowly heated in a rocking furnace, at a rate 100 °C/h. In order to ensure homogeneity of the melt, the ampoules are maintained 100 °C above the melting temperature of the constituents for 12 h rotating at 10 rpm. The ampoules are subsequently quenched in air to get bulk glassy samples. X-ray diffraction is used to confirm the amorphous nature of the quenched samples. These samples are further cut and polished to optical quality for waveguide inscription.

2.2. Waveguide fabrication

Optical straight waveguides have been inscribed in GeS_2 glass by multi-scan technique using a master oscillator power amplifier Yb-doped fiber laser (IMRA μJewel D400) of central wavelength of 1047 nm. The sample is mounted on computer controlled Aerotech x-y-z air bearing stages and translated perpendicular to the laser propagation direction to write a waveguide. The laser is focused to 100 μm below the substrate surface using a 0.67 NA objective lens. Individual waveguides are formed by 11 close scans, with an incremental movement of 0.4 μm relative to the previous scan, and each waveguide is separated by 50 μm. During inscription, the pulse repetition rate with circular polarization has been set at 1000 kHz, with a pulse duration of 350 fs. The pulse energies are varied from 100 to 9 nJ, and different scan speeds (25, 12.5, 6, and 2.5 mm/s) have been used to inscribe the waveguides.

After inscription, waveguide facets are ground and polished to remove any tapering. After completing a set of optical loss measurements on the above-mentioned waveguides, optimum parameters are found and another set of waveguide are inscribed on the same sample with pulse energy ranging from 20 to 10 nJ at different translation speeds (60, 40, 25, 12.5, 6, 2.5, and 1 mm/s) keeping rest of parameters constant. Fig. 1 shows a schematic diagram of multi-scan technique used to inscribe the waveguides in GeS_2 substrate.

2.3. Optical characterization

Insertion loss measurements have been carried out at 1550 nm of wavelength. Initially, a single-mode fiber (SMF-28) is fusion spliced to source (amplified spontaneous emission source) and detector (OSA - Advantest Q8384) for the reference measurement. Once the reference measurements have been recorded, the fiber is cleaved and butt-coupled to the waveguide facets using index matching gel and the measured output power is compared with the reference. For the mode field imaging of the waveguides, a 1550 nm light source is butt-coupled to the waveguides at one end and an Electrophysics-7290A IR Vidicon camera on the other end. This is detailed further in the results and discussion section.

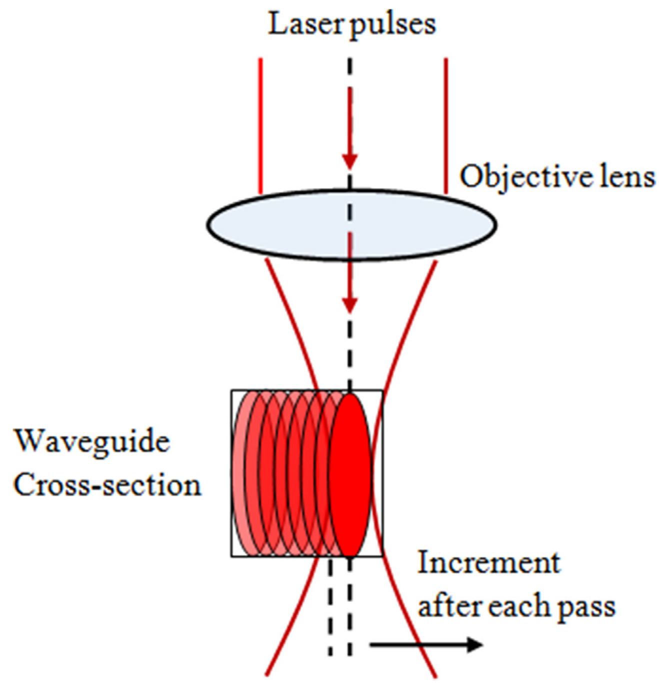

Fig. 1. Schematic diagram of multi-scan writing technique.

2.4. Nanoindentation studies

Nanoindentation studies have been performed on the samples using the Triboindenter (Hysitron, Minneapolis, USA) with in situ imaging capability. The machine continuously monitors the depth of penetration *h* of the indenter and the load *P* with resolutions of ~0.2 nm and ~1 nN, respectively. A Berkovich tip diamond indenter with a tip radius of ~100 nm has been employed for indentation. A peak load P_{max} of 5 mN with loading and unloading rates of 0.5 mN/s and a hold time (at peak load) of 10 s is used. The indentations have been performed on different parts of the waveguides as well as on the bulk of the sample for reference values. From the measured *P-h* responses, elastic modulus, *E*, and hardness, *H*, are extracted by employing the Oliver-Pharr method [17], which is a standard method employed for such analysis.

2.5. Micro-Raman spectroscopy

Micro-Raman spectroscopic measurements have been undertaken on the waveguides using a Horiba JobinYvon (LabRAM HR) Raman Spectrometer in the backscattering mode. The scattered light is detected with the aid of a triple monochromator and a CCD cooled to −70 °C. The sample is illuminated by the 532 nm argon-ion laser focused using 50× objective. All the spectra are recorded using 2 mW of laser power (by using neutral density filter D_1). The spectral resolution for the recorded Stokes-side Raman is ~0.6 cm^{-1}. The spot size of the laser used in the Raman setup is ~2 μm and the data acquisition time is 5 s.

3. Result and discussions

3.1. Optical studies

A representative optical micrograph of a multi-scan waveguide, which has been inscribed at 17 nJ of pulse energy and 12.5 mm/s