Micro-Raman mapping of micro-gratings in 'BACCARAT' glass directly written using femtosecond laser

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ABSTRACT

We present some of our results on the femtosecond laser direct writing and characterization of micro-gratings in Baccarat glass. Gratings were inscribed with amplified 800 nm, ~100 femtosecond pulses at 1 kHz repetition rate. The change in refractive index of the modified region was estimated from grating efficiency measurements and was found to be ~10^{-3}. Micro-Raman studies demonstrated an increase in the intensity of the band near 596 cm^{-1} in the laser irradiated region clearly indicating an increase in the refractive index. Micro-Raman mapping of the grating showed a periodic variation of the band intensity further confirming the formation of grating. Structures with sub wavelength dimensions (<800 nm) were achieved with shaping of the input pulses using a rectangular slit. Waveguides were inscribed by optimizing parameters like slit width, focusing conditions, translation speed etc. We shall present our results on the physical, spectroscopic and optical characterization of these structures.

Keywords: Laser Direct Writing, Micro-Raman, gratings

1. INTRODUCTION

Recent advances in femtosecond laser direct writing (LDW) demonstrate unprecedented prospects for miniaturization and integration of highly functional photonic and microfluidic devices directly inside transparent dielectric and polymeric materials.[1-12] Femtosecond pulses possess the unique ability to precisely deposit energy inside the material and significantly the ultrafast interaction does not require specially prepared or photosensitive materials unlike other fabrication techniques. This technique is attractive for fabrication of photonic structures in rare-earth activated glasses for creating gain elements and waveguide lasers[13] and structures for microfluidics.[14,15] We have recently established the efficacy of Er activated 'Baccarat' glass as a potential candidate for applications in telecommunications with good spectroscopic properties and high quantum efficiency, close to 80%.[16] Here, we present some of our results on the direct writing and characterization of micro-gratings and waveguides in Baccarat glass. Gratings were inscribed with amplified 800 nm, ~100 femtosecond pulses at 1 kHz repetition rate. Structures with sub wavelength dimensions (< 800 nm) were achieved with shaping of the input pulses using a rectangular slit. Waveguides were inscribed by optimizing parameters like slit width, focussing conditions, translation speed etc. The change in refractive index of the modified region was estimated to be ~10^{-3} from grating efficiency measurements. The modified structures were studied by micro-Raman spectroscopic technique.

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2. EXPERIMENTAL

The glass has been produced at Cristallerie Baccarat by a conventional melt-quenching technique with the following molar composition: 77.29 SiO_2: 11.86 K_2O: 10.37 PbO: 0.48 Sb_2O_3. The density of the samples has been measured by a gas pycnometer. The Er^{3+} concentrations have been determined to be 0.2 mol%, taking into account both the nominal composition and the measured density of the glass. Refractive index value at several wavelengths has been measured to be 1.5427 with an accuracy of ± 0.001 and resolution of ±0.0005, using a standard prism coupling method.[17]

2.1 Spectroscopic characterisation of glass:

Optical absorption experiments were performed at room temperature from the ultraviolet to the near infrared spectral range with a double beam spectrophotometer (UV-Vis-NIR Cary 5000 Varian). The Fourier Transform Infrared (FTIR) spectroscopy measurements were carried out by using a JASCO FTIR-660 plus spectrometer with a resolution set to 4 cm^{-1}. The polarized VV and depolarized HV Raman spectra were obtained by exciting the samples with the 488 nm line of an Ar^+-ion laser. The signal was selected by a double monochromator and analyzed by a photon-counting system. The 514.5 nm line of an Ar^+ -ion laser and the 980 nm line of a Ti:Sapphire laser were used as excitation sources for near infrared photoluminescence (PL) spectroscopy measurements. A Triax 320-mm-focal-length single-grating monochromator was used to disperse the luminescence light onto an InGaAs photodiode and the signal acquisition was performed using a standard lock-in technique. To measure the PL decay of the excited $^4I_{13/2}$ level, the continuous wave excitation laser was pulsed with a mechanical chopper and data were acquired with a digital oscilloscope.

2.2 LDW experimental set up :

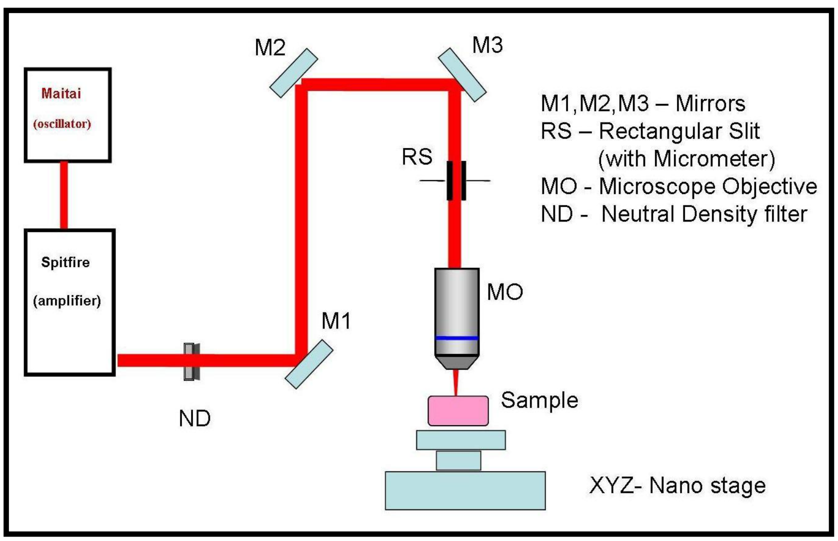


Figure 1 Schematic diagram of laser Direct writing setup

The sample was typically cut to a dimension of 1 cm X 2 cm X 0.5 cm (thickness)) for ease of use and all the 6 faces were optically polished. The sample was first cleaned using acetone and then with methanol to ensure the surface is devoid of any stains and dust. The cleaned sample was placed on computer controlled XYZ nanostages for better control over the structures to be written in them. Figure 1 shows the schematic of the experimental setup used for direct writing. The quality of the structures fabricated depended critically on the quality of the focused spot and the movement of the sample placed on XYZ stage. Any aberration in the optics or stage movement will affect the shape and size of the structures drastically. Typically 100 fsec pulses from a femtosecond amplifier were employed for direct writing at a wavelength of 800 nm with 1 KHz repetition rate. Neutral density filters were used to attenuate the energy of the input beam. The beam was steered such that it focuses downwards on to the sample after the microscope objective (typically 40X with 0.65 NA). A rectangular slit whose width could be varied from 500 μm to 2 mm was used to modify the spatial profile of the input beam for improved structures.

2.3 Inscription of grating:

The procedure followed for inscribing and characterizing the gratings is as follows. Initially simple straight line structures were inscribed into samples. Each line corresponded to a specific set of parameters (like the sample translation speed, input energy of the fsec pulses, and size of the aperture before the microscope objective). Later these structures were imaged using Leica scanning confocal microscope. Images were obtained both in transmission mode and reflection mode. The imaging essentially takes advantage of deferential transmission and reflection properties of the samples in the regions exposed to the femtosecond laser and the un-exposed regions. In the transmission mode the width of the structures was determined. In the reflection mode several snapshots of the structures were taken along the depth at definite intervals (typically the step size along the z is chosen to be ~1 μm). Each snapshot corresponded to a particular depth and all these snapshots were collated to obtain a 3-D (cross-sectional, depth profile) view of the structures written. In the transmission mode the width of the structures is determined. The lines/structure with desired width and depth profile were identified and with the corresponding writing parameters the structures/lines were written again and the images compared to ensure the reproducibility.

3. RESULTS AND DISCUSSION

Raman and absorption measurements indicate the high transparency of the "Baccarat" glass. The water content of these glasses is very low and we estimate a C_{OH} concentration as low as 3.6×10^{18} cm^{-3}. Luminescence at 1.5 μm with a spectral width of ~18 nm was observed on both the glasses. No changes in the spectral width as a function of the excitation wavelength were observed and no NIR-to-visible up-conversion signal has been detected. The non-appearance of up-conversion in "Baccarat" glasses indicates that most of Er^{3+} ions are homogeneously distributed and that interaction clusters as well as chemical clusters are practically absent. The $^4I_{13/2}$ metastable state of the Er^{3+} ions decay curve present a single exponential profile, with a lifetime value of 14.2 ms for the sample doped with 0.2 mol % of erbium. Radiative lifetime were calculated with different models and compared with the experimental measured lifetimes. For the 0.2 mol % Er^{3+}-activated glass, quantum efficiency reaches very high values, up to 79.8 % in the case of a calculation based on the Judd-Ofelt theory. Such high quantum efficiency has already been observed in Er^{3+}-doped tellurite glasses and remains among the highest quantum efficiencies estimated in pure or modified-silica host glasses. The optical and spectroscopic properties of this modified-silica glass make it well adapted as host medium for the light propagation and a good candidate for many applications in telecommunication systems.[16]

3.1 Efficiency measurements and Micro-Raman mapping of the gratings

Figure 2 (a) shows the confocal microscopic image of the grating structure inscribed in the glass. A continuous wave He-Ne laser was diffracted through the grating and the internal diffraction pattern was captured on a screen (see figure inset). The intensity of the diffraction orders (0, 1) were obtained and the diffraction efficiency was calculated to be ~17%. From the diffraction efficiency measurements the estimated change in the refractive index was ~2.2×10^{-3} using the relation $\mathbf{\Delta n = \lambda \cos(\theta) \tanh^{-1}(\sqrt{\eta})/\pi d}$, where η is the diffraction efficiency fraction of first order to the zeroth order; d is the depth or the grating thickness; θ = 90 degrees; λ is the wavelength. The periodicity of the gratings was 6 μm. The periodicity and the grating width could be controlled through the input energy, scan speed, and the focal conditions. We could achieve grating periods (and spacing) ranging from 4 μm to 10 μm easily. The modified region was studied by micro-Raman spectroscopy for an insight into the physical changes which occurred at the focus due to fsec pulses interaction. Two main differences between irradiated and not irradiated lines were observed 1) an increase of the sharp defect band at 596 cm^{-1} and 2) an increase of intensity and line width of the broad band at about 470 cm^{-1}. This corresponds to a hardening of the structure by irradiation, which can be associated to a positive change of the refractive index.

The micro-Raman mapping of the grating structure was done across three lines of the grating. The rectangular area highlighted in the figure 3 (a) was the region of the sample mapped and 3(b) shows the results of the mapping of Raman intensity. The periodic variation of Raman intensity further proved the grating structure formation and also reflects the periodic refractive index variation across the grating structure.

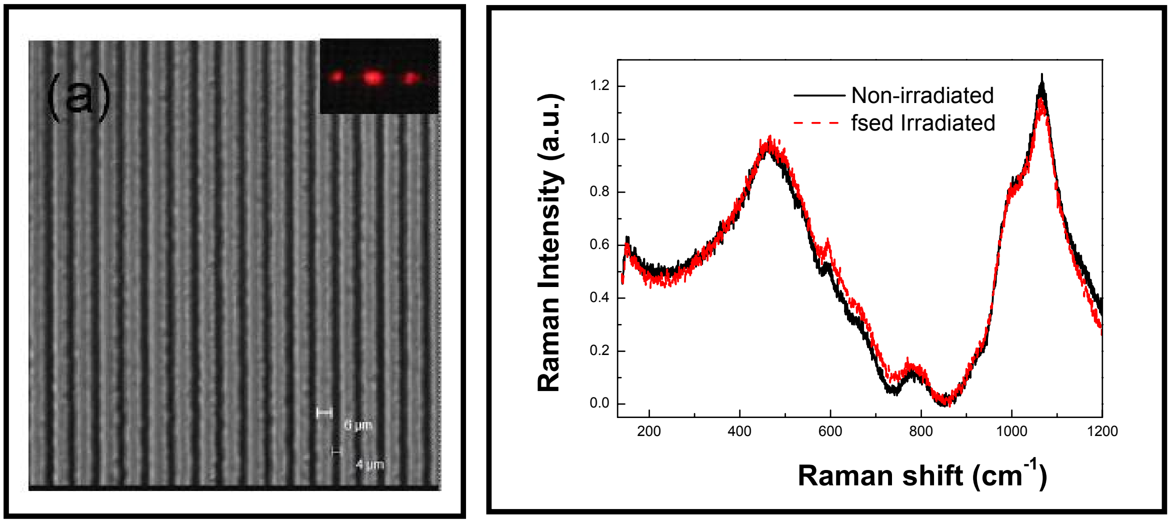


Figure 2 (a) Microscope image of the grating inscribed into baccarat glass; the inset shows the internal diffraction pattern. (b) The Raman spectrum from the laser modified region (red, dashed curve) and unmodified region (black, solid curve).

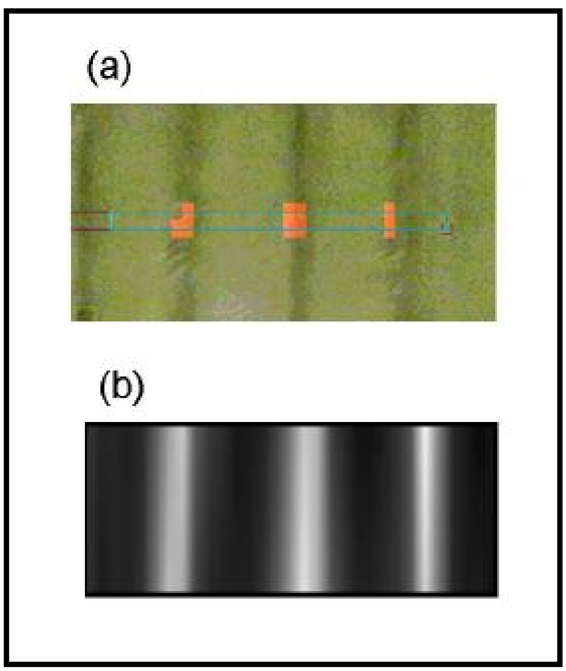


Figure 3 (a) Optical image of the grating inscribed into baccarat glass; the rectangular highlighted region was mapped for intensity (b) Raman intensity map of the rectangular region in (a) with bright region of the map indicating higher intensity

4. SLIT BEAM SHAPING FOR LDW TO FABRICATE MICRO STRUCTURES AND WAVEGUIDES

There are two principle ways of LDW a) Transverse configuration, where the sample translation is perpendicular to laser propagation and b) Longitudinal configuration, where the sample translation and the laser beam propagation are parallel. Longitudinal LDW creates cylindrical waveguides but the length of the waveguides is limited by the working distance of the focussing objectives.

The transverse writing geometry allows one to write waveguides and structures of arbitrary length and design. However, this method produces waveguides and structures with strong core asymmetry and significant losses (particularly in the case of waveguides).[18] This asymmetry can be explained as follows: perpendicularly to the beam propagation direction the waveguide size is given approximately by the beam focal diameter $2\omega_0$, while along the propagation direction, it is given by the confocal parameter $b = 2\pi\omega_0^2/\lambda$.[19] For focused diameters of the order of a few micrometers this results in a large difference in waveguide sizes in the two directions. This asymmetry becomes particularly severe when the waveguide size is increased, as required for wave guiding at the optical communication wavelength of 1.5 μm, thus greatly reducing the efficiency of fiber butt coupling in conventional telecommunications setups.[19]

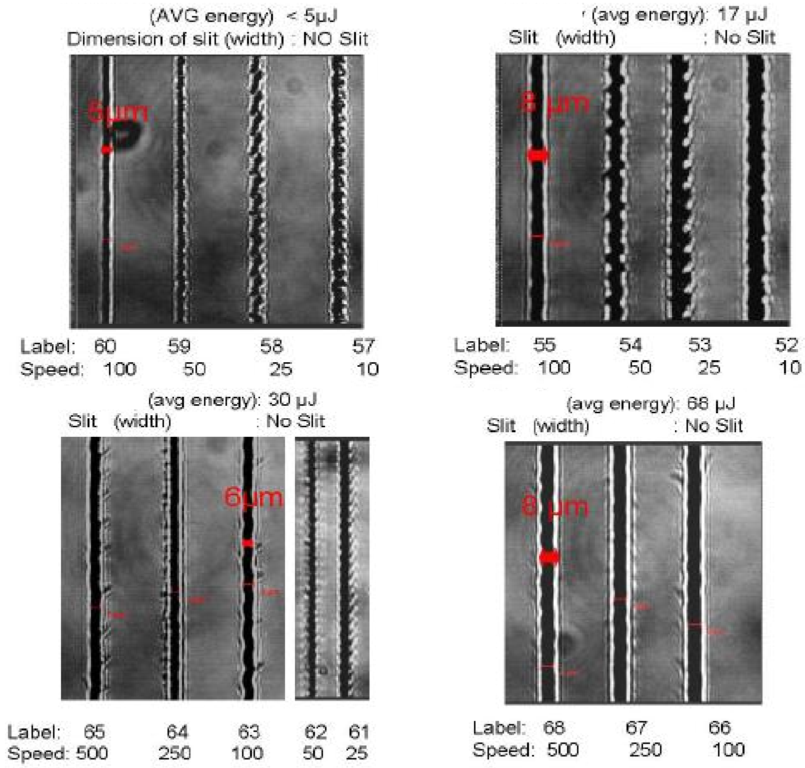


Figure 4 Confocal microscope images of micro-structures inscribed in Baccarat glass. The parameters of the LDW such as average energy of the incident laser pulse and the sample translation speeds (μm/sec) are given.

To circumvent this problem Osellame et al.[20] introduced a novel focusing geometry in which the femtosecond writing beam is astigmatically shaped by changing both the spot sizes in the tangential and sagittal planes and the relative positions of the beam waists. This shaping allowed the modification of the interaction volume in such a way that the waveguide cross section can be made circular and with arbitrary size.[21] Another simpler way is to use a rectangular slit oriented parallel to the writing direction.[22] In transverse LDW, by placing a rectangular slit close to the focussing objective acts as diffractive element and helps in redistribution of the laser intensity gradient around the focus. Various structures were written by placing a rectangular slit of different dimensions (2 mm, 1.5 mm, 0.5 mm) before the focussing microscope objective. For a particular slit width line-structures were inscribed by varying the incident laser intensity and the translation speed of the samples and the process was repeated for all other slit width's mentioned above. The incident laser energy shown in the figures was measured before the focussing by microscope objective and after the slit was placed (the typical values of average energy were varied from < 5 μJ to 75 μJ). The lower end intensity was estimated to be in the range 2-5 μ J taking into account the transmission efficiencies of the slits used and the filters placed. The higher end was fixed at around 80-90 μJ, as energies above this were observed to damage samples. The typical speeds of the sample translation were 10, 25, 50, 100, 250 and 500 μm/second. To avoid sample damage lower translation speeds (< 50 μ m /second) were not employed while inscribing micro-structures at higher incident laser intensities (> 40 μ J). Similarly higher translation speed were not used at very low incident laser intensities (< 20 μJ) as the modifications induced by laser inscription in the samples at such experimental conditions were minimal or negligible, and they could not be imaged by the confocal microscope even when operated at higher magnifications.

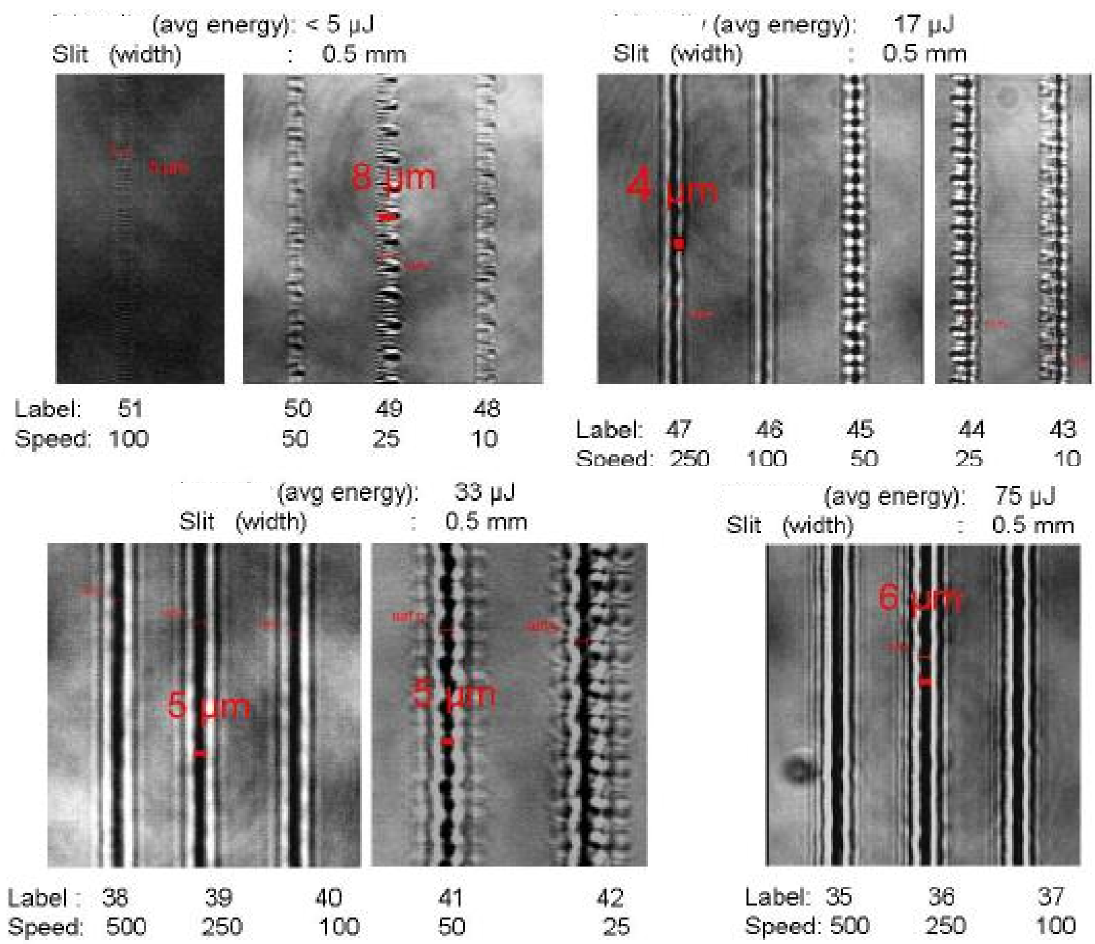


Figure 5 Confocal microscope images of micro-structures inscribed in Baccarat glass. The parameters of the LDW such as average energy of the incident laser pulse and the sample translation speeds (μm/sec) are given. A slit of 0.5 mm was used for beam shaping

The confocal microscope images of the structures written without a slit are shown in figure 4 while the structure written with a slit of 500 μ m placed before the focussing objective are shown in the figure 5. We feel that the wobbly features seen in some of the structures can be avoided by using a vibration isolation optical table for mounting the nano-positioner. In every figure, it can be seen that the incident femtosecond laser (average) energy and the width of the slit used are mentioned on the top, while at the bottom the line structure label number and the corresponding speeds (in units of μm/second) at which the sample was translated is mentioned. As seen from the figures the width of the structures written at lower speed are broader than that written at higher speed. When the sample was moved at lower speeds a particular region is exposed for more number of laser shots, hence the area modified is more. The structures written using a slit at moderate laser intensities (20 to 30 μJ) and at speeds of (250, 500 μm/second) appear more smooth on visual inspection through the microscope as compared to structures written without using slit. For faster speeds of translation of the sample the structures were smooth irrespective of the usage of slit. The width of the structures could be varied from 4 μm to 10 μm with varying speeds and input energy. Some of the structures had interesting features, both in perpendicular and parallel direction, with dimensions of the order of <1 μm and the reason for the appearance of these structures is being investigated.

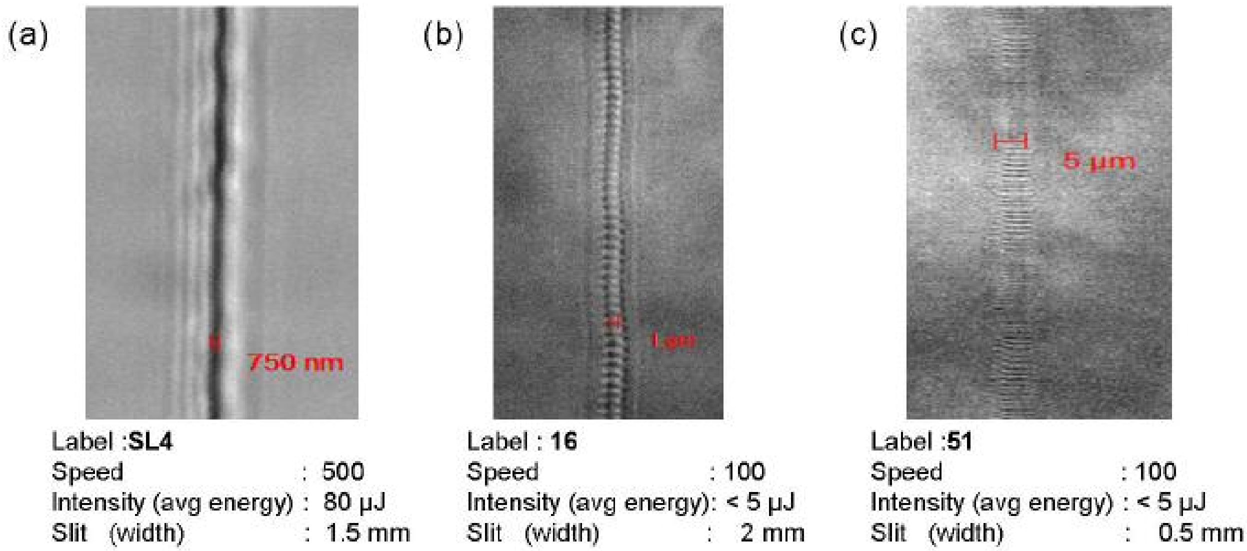
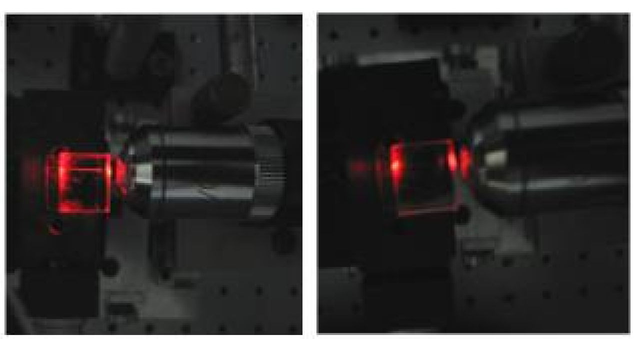


Figure 6 Microscope images of the structures inscribed in the baccarat glass

With the effects due to repetitive exposure of a specific spot and the translation speed some interesting structures were inscribed in Baccarat glass. Some of the interesting structures produced are depicted in figure 6(a) where the width of the structure was 750 nm. Such sub-micron structures when created in periodic fashion in three dimensions can lead to photonics crystals. Selective etching, if possible, of these regions will result in nanochannels. The contiguous pearl [fig 6 (b)] shaped micro-structure can be used as waveguide attenuators[7] as these structures scatter light. The grating like structures seen in figure [fig 6 (c)] are parallel to the scale bar, and are periodic with sub-micron dimensions. These modified planes are perpendicular to the written line structure are similar to the Bragg gratings. With a better control over dimension and periodicity of the structure, Bragg gratings can be inscribed directly by LDW while simultaneously inscribing the waveguides. We are investigating the effect of diffraction from the slit used in the variety of structures produced.



(a) **(b)**

Figure 7 Typical photographs of the wave guides with He-Ne laser coupled in (a) and no coupling seen in (b)

The optical properties of the structures were tested with the help of a 1560 nm cw laser. The laser light was butt-coupled into some of these waveguides and near-field mode profiles from the exit end were captured with the help of a microscope objective and CCD camera. These initial results show that there is indeed a positive refractive index change sufficient enough for light to be confined and guided through them. Figure 7 shows the light coupling into a typical waveguide using 633 nm laser from a He-Ne laser. Figure 7(a) clearly indicates the streak of light guided along the waveguide whereas a small detuning of the laser beam path shows the streak vanishing indicating the light is now not coupled demonstrating the absence of any waveguide [figure 7(b)]. Figure 8(a) shows typical mode profile of the output from a fiber. Figure 8(b) shows image of the near field mode profile of a typical waveguide written using a slit of ~1.5 mm while scanning the sample at a speed of ~500 μm/sec and an input energy of 30 μJ. The effect of the slit is clearly seen in the figure 8(b) as the mode profile is fairly circular and comparable to fiber mode though it is bigger in size. The mode profile obtained with waveguide written without the slit was indeed elliptical in nature. The profile of the waveguide output clearly indicates single mode propagation. The loss measurements are in progress to determine the parameters which favor the light propagation through these waveguides with minimum attenuation. The waveguides having circular cross section are expected to possess lower losses as compared to the ones with elliptical cross sections. There have been several recent studies[23-40] investigating the fabrication of waveguide lasers, microfluidic structures, and Fiber Bragg gratings in variety of glasses. Baccarat glass is a potential glass in telecom spectral range and the possibility of making passive photonic structures in these glasses along with the capability of LDW to fabricate photonic devices in passive/active glasses augers well for the telecommunication applications. That the femtosecond laser pulses are able to join two similar/dissimilar glasses[41] increases the opportunity of writing structures separately in passive and active glasses and join them for a fully functional photonic device. Our future investigation will focus on the optimization of writing conditions for enhanced waveguide performance and demonstration of amplification in these waveguide structures.

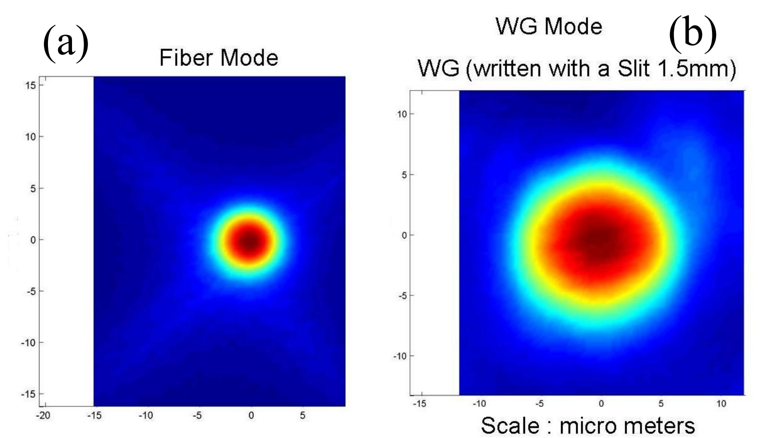


Figure 8 Near field mode profile of the waveguides inscribed in baccarat (b and c) as compared to the fiber mode profile (a)

5. CONCLUSIONS

Gratings and waveguides were inscribed in Baccarat glass using the technique of femtosecond laser direct writing. The change in refractive index was estimated to be of the order of 10^{-3}. Micro-Raman measurements confirmed that there was an increase in refractive index in the laser modified regions. By placing an appropriate rectangular slit before the focusing microscope objective and by varying the sample translation speed and femtosecond laser energy waveguides with circular cross-section were achieved. Measurements of the propagation losses are in progress and will be reported else were. Baccarat glass is a material promising for fabrications of waveguides and other devices by LDW, especially in the spectral region of telecommunications.

6. ACKNOWLEDGEMENTS

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