

# Directly Photoinscribed Thick Bragg Gratings in Ohara WMS-15 Glass-Ceramic

Peter A. Krug, Rodica Matei Rogoian and Jacques Albert

Department of Electronics, Carleton University, 1125 Colonel By Drive, Ottawa, ON K1S 5B6, Canada  
[pkrug@doe.carleton.ca](mailto:pkrug@doe.carleton.ca)

**Abstract:** Volume gratings were UV inscribed in WMS-15 glass-ceramic at 193 and 248nm without additional processing. Weak, easily bleached gratings resulted from fluences below  $0.3\text{kJ}/\text{cm}^2$ . Stable gratings with  $\Delta n \sim 6 \times 10^{-5}$  were formed at higher fluences.

©2009 Optical Society of America

OCIS codes: (050.7330) Volume gratings; (160.5335) Photosensitive materials

## 1. Introduction

Glass-ceramics, which combine many useful properties including a wide range of values (negative, zero or positive) of both thermal expansion and thermo-optic coefficients, are obtained by the controlled nucleation and crystallization of glass [1]. Glass-ceramics that are sufficiently transparent to be used in transmissive optical components have recently been reported and have become available commercially [2,3]. Diffraction gratings have previously been photoinduced in glass-ceramics by UV exposure, followed by chemical etching [4], thermal treatment [5] or both [6]. Here we report, for the first time to our knowledge, the direct photoinscription of diffraction gratings in a glass-ceramic without the need for any subsequent processing.

## 2. UV Inscription of gratings

The composition of Ohara WMS-15 transparent glass-ceramic [7] is a trade secret, but the material is known to contain oxides of Si, Al, Zr, P, and possibly Zn and Sb. Polished 3mm thick samples were exposed through zero order nulled phase mask to spatially uniform beam with energy densities of  $226\text{mJ}/\text{cm}^2/\text{pulse}$  (25 pulses per second at 248nm) or  $36\text{mJ}/\text{cm}^2/\text{pulse}$  (100 pulses per second at 193nm). The grating depths were calculated from the angular dependence of diffraction efficiency [8] to be between  $127 \pm 20$  and  $254 \pm 20\mu\text{m}$  for the 248nm written gratings but only  $8.5 \pm 2\mu\text{m}$  for the 193nm written gratings. From the depths and the Bragg diffraction efficiencies measured using a collimated laser beam at 632.8nm, we determined the index modulation depth of gratings written with UV doses between 0.01 and  $30\text{kJ}/\text{cm}^2$ , as shown in Fig. 1.

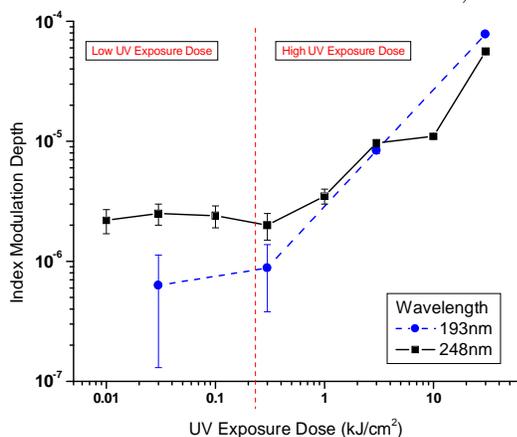


Fig. 1. Refractive index modulation depth as a function of dose.

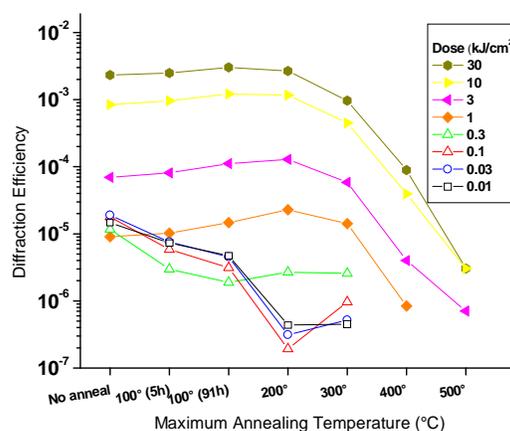


Fig. 2. Grating diffraction efficiency as a function of annealing.

We see that there are two very distinctly different regions of grating formation. For UV doses less than  $0.3\text{kJ}/\text{cm}^2$  we observe the rapid formation of weak gratings, having diffraction efficiency less than  $2 \times 10^{-5}$ . For 248nm inscription, these gratings reach maximum strength at the relatively low dose of  $0.03\text{kJ}/\text{cm}^2$ , after which their strength decreases with increasing dose up to  $0.3\text{kJ}/\text{cm}^2$ . For UV doses of  $1\text{kJ}/\text{cm}^2$  and above, much stronger grating growth is observed, with the gratings continuing to grow with increasing UV dose up to the maximum applied dose of  $30\text{kJ}/\text{cm}^2$ . The strongest grating in the high UV dose regime, written at  $30\text{kJ}/\text{cm}^2$ , has an index modulation depth of around  $6 \times 10^{-5}$ , which is around 25 times the modulation depth of the strongest grating in the low UV dose

regime. Fig. 2 shows the evolution of grating diffraction efficiency with annealing for the 248nm inscribed gratings. In the annealing behavior we again see a marked difference in the behavior of the low UV dose (hollow symbols) and high dose (solid symbols) gratings. Interestingly, there appears to be a modest *increase* in strength of the high exposure dose gratings for annealing temperatures up to 200°C.

### 3. Thermal annealing experiments

To better understand the physical changes in the glass-ceramic caused by the UV exposure, we measured the optical absorption spectra of uniformly irradiated samples, exposed at 248nm to total doses between 0.01 and 30kJ/cm<sup>2</sup>. Fig. 3 shows the absorption spectrum of the glass-ceramic before exposure.

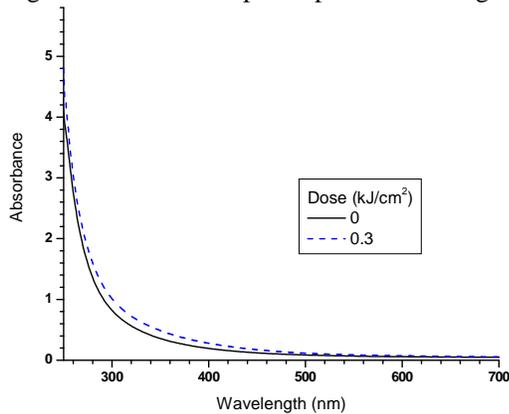


Fig. 3. Absorption spectra of unexposed and weakly exposed samples.

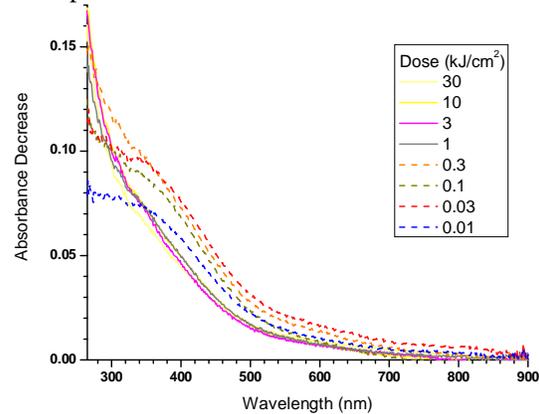


Fig. 4. Absorption decrease caused by thermal annealing at 200°C

The only readily apparent feature is the steady increase in absorption as wavelength decreases. Subsequent UV exposure increased the optical absorbance by about 0.1 at UV wavelengths, but by only around 0.02 at visible wavelengths. For low dose gratings, the UV exposure-induced increase in visible absorption was greater than for the high exposure gratings. When these exposed samples were annealed at 200°C, nearly all the increased absorption was reversed. Fig. 4 shows the decrease in absorption due to annealing of the exposed samples. For the low dose samples (dashed curves) there is a broad spectral shoulder in the difference spectra between 320 and 400nm, which resembles the thermally induced spectral feature observed by Efimov et al [9] in metal ion doped glass-ceramics. They ascribed the feature to the clustering of silver particles induced by annealing [10]. The Ohara WMS-15 glass-ceramic does not contain silver, but a similar mechanism, involving one or more of the metal ion species present in the material may be involved. Further studies, possibly involving surface profiling and various microprobe techniques may clarify the physical processes involved in the photoinduced refractive index changes.

### 4. Conclusions

We have demonstrated for the first time, to our knowledge, the UV inscription, without subsequent etching or thermal development, of thick diffraction gratings in a glass-ceramic material. Grating strength, thermal annealing and optical absorption measurements support the hypothesis that two different physical mechanisms are involved in the photoinduced refractive index change, according to whether the total exposure is less or greater than about 0.3kJ/cm<sup>2</sup>.

### 5. References

- [1] W. Höland and G. Beall, *Glass Ceramic Technology* (American Ceramic Society, 2002).
- [2] H. Minamikawa, K. Ohara and N. Goto, *Low Expansion Transparent Glass-Ceramics, Glass-Ceramic Substrate and Optical Waveguide Element*, US patent 7,148,164 (2006).
- [3] See, for example, Schott Robax: [www.us.schott.com/hometech/english/products/robax](http://www.us.schott.com/hometech/english/products/robax).
- [4] F.E. Livingston and H. Helvajian, *Variable UV Laser Exposure Processing of Photosensitive Glass-Ceramics: Maskless Micro- to Meso-Scale Structure Fabrication*, *Appl. Phys. A* **81**, 1569-1581 (2005).
- [5] M. Kösters, H.-T. Hsieh, D. Psaltis and K. Buse, *Holography in Commercially Available Photoetchable Glasses*, *Appl. Optics* **44**, 3399-3402 (2005).
- [6] Y. Cheng, K. Sugioka, M. Masuda, K. Toyoda, M. Kawachi, K. Shihoyama and K. Midorikawa, *3D Microstructuring Inside Foturan Glass by Femtosecond Laser*, *Riken Review* no. 50, 101-106 (2002).
- [7] MSDS for Ohara WMS-15 glass-ceramic. Provided by Ohara Corporation (private communication).
- [8] H. Kogelnik, *Coupled Wave Theory for Thick Hologram Gratings*, *Bell Syst. Tech. J.*, **48**, 2909-2947 (1969).
- [9] O.M. Efimov, L.G. Glebov, L.N. Glebova, K.C. Richardson and V.I. Smirnov, *High Efficiency Bragg Gratings in Photothermorefractive Glass*, *Applied Opt.*, **38**, 619-627 (1999).
- [10] S.D. Stookey, G.H. Beall, and J.E. Pierson, *Full-Color Photosensitive Glass*, *J. Appl. Phys.* **49**, 5114-5123 (1978).