

## Controllable 3D crystallization in SiO<sub>2</sub>-based glasses by femtosecond laser

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The use of femtosecond laser (fs laser) to rationally manipulate micro-/nano-structures inside transparent materials is of great interest since it allows engineering the optical properties of the material via controlling distribution, size and spatial orientation of nano/micro-crystals in glass [1]. In present work, the direct photo-induced precipitation of crystals is taken in silica-based glasses by ultra-short laser irradiation via manipulation of applied pulse energy in 32.5Li<sub>2</sub>O-27.5Nb<sub>2</sub>O<sub>5</sub>-40SiO<sub>2</sub> glass. And the size and orientation of these crystals are characterized by Scanning Electron Microscope-Electron Backscattered Diffraction (SEM-EBSD) method. We also discuss the mechanism of controllable crystallization in glass using femtosecond laser in this paper.

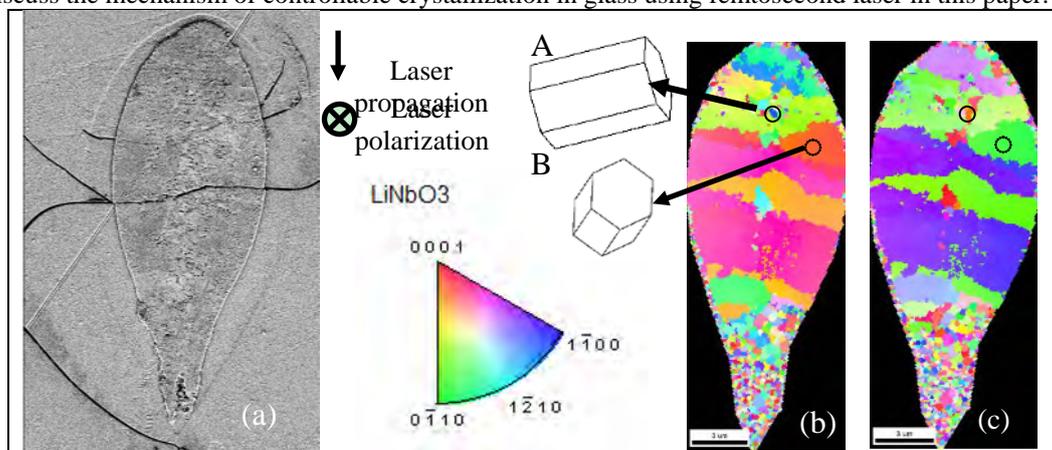


Fig.1. (a) SEM micrograph of etched cross-section of written line with pulse energy 1.5  $\mu\text{J}$  [2], EBSD scan image coding the crystal orientation (b) along the direction parallel to the laser polarization and (c) along the perpendicular both to the laser polarization and to the laser propagation direction. For example, the points A and B (in solid and dashed circle) are related to the blue (1-100) and red (0001) color in figure 1-(b) and to pink (1-101) and green (0-110) in figure 1-(c), respectively.

The lens-shaped laser trace can be well observed using SEM (secondary electrons) as shown in Fig. 1-(a) after HF etching [2]. With the help of indexed Kikuchi diffraction patterns, we obtained that the LiNbO<sub>3</sub>-like crystals with R3c space group have been successfully induced in the rough region and c-axis [0001] is polar-axis in this crystallographic hexagonal unit cell. The size and the uniformity of laser-induced crystals in the cross-section were probed by EBSD in figure 1-(b) and (c). The color in indexed sector is used to give the orientation of c-axis of crystals in the cross section. It is clear that the polar axis of crystals oriented obviously along the laser writing direction at the center of laser track is coded by red color. We can see that the disoriented crystals are located both at the head and at the tail of laser trace in figure 1-(b) [2]. As looking at blue point A in the cross-section, we saw that the polar c-axis of hexagonal unit oriented in the cross-section is nearly perpendicular to the laser polarization. Meantime, the hexagonal unit at green point B reveals that the polar-axis is almost perfectly along the laser polarization.

As a result of the evolution of photo-induced crystallization obtained by manipulating the pulse energy, which will be presented in the conference, it seems that the two main impact factors of the crystallization and the orientation of polar axis of crystals are the distribution of temperature field and the orientation of temperature gradient. Therefore, we are able to obtain a volume completely crystallized on contradistinction to ref [3] where partial crystallization occurs only at the head or at the tail of the laser track. The subsequent question, useful for the applications, is the mechanism of controllable orientation as we can see that large crystals have been achieved. We will discuss this point at the conference.

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[3] M. Zhong, Y. Du, H Ma, Y. Han, B. Lu, Y. Dai, X Zeng, "Crystalline phase distribution of Dy<sub>2</sub>(MoO<sub>4</sub>)<sub>3</sub> in glass induced by 250 kHz femtosecond laser irradiation", OME 2 1156-1164 (2012)