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EXCIMER LASER AND ELECTRON BEAM IRRADIATION EFFECTS IN $SB_{40}SE_{60}$ THIN FILMS

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Abstract

The selective wet-etching of laser and electron beam irradiated Sb-Se thin films was performed. The irradiation effects of the radiation from an excimer laser on Sb-Se thin films are compared with those from electron beam studies. Both types of irradiation were found to induce changes in the structure of Sb-Se thin film. Characterization of the structure and surface of studied films by AFM methods has been done and potential application is suggested.

We have realized direct, one-step e-beam recording of surface reliefs. An experimental study of the possibility to fabricate grey scale optical elements and 3D structures in resist was carried out.

Key words: *laser, electron beam, thin film, irradiation, relief*

1. INTRODUCTION

Chalcogenide glass possesses unique characteristics, such as photoinduced diffusion in multilayer systems (Debnath, Nusbar, & Fitzgerald, 2005), (Romero, 2004) changing their structure under the influence of light (Lyubin,Victor, 2009), photodarkening (TANAKA, KYOHYA, & ODAJIMA, 1984) photo-induced volume changes (KUGLER, HEGEDÜS, & LUKÁCS, 2007), remarkable sensitivity to the ionizing radiation (Mietzsch,K, n.d.), (ELSAYED, 2004), etc. These materials can be used in the photolithography, where they serve as high resolution registering media or inorganic photoresists. The utilization of chalcogenide thin films in lithography is based on different dissolution rates of exposed and unexposed parts of the film. The different etching rates occur as a consequence of photostructural changes induced by band- or sub-band gap energy exposure (Tanaka, 2003). Such media provide a resolution capability of the order of several nanometers due to their amorphous structure (Utsugi, 1993) and ability to produce surface relief after exposure and treatment. In a present work we will study the structures in chalcogenide glasses which are imaged and produced using He-Ne laser and scanning electron microscopy (SEM).

2. EXPERIMENTAL

Thin film was prepared by thermal evaporation technique from bulk glass samples of composition $Sb_{40}Se_{60}$ in vacuum 10^{-5} Torr onto BK-7 glass substrates at room temperature. The substrate was equipped with pre-deposited Ni layer of 100 nm thikness. Film thickness was controlled during evaporation by means of interference technique at wavelength of 650 nm, as it was shown in (Gerbreders, Teteris, Sledevskis, & Bulanovs, 2007). Thickness of prepared film was about 100 nm for electron irradiation and 130 nm for laser treatment. The composition and structure of the deposed layers have been analyzed using the INCA x-act detector, EBSD system and X-ray difractometer SmartLab Rigaku.

Characterisation experiments for $Sb_{40}Se_{60}$ thin films were carried out for both, photo and e-beam exposure. Local laser treatment of $Sb_{40}Se_{60}$ thin films was carried out using He-Ne laser, $\lambda = 633$ nm and output energy 16mW for 30 min. E-beam irradiation of the samples was performed by scanning electron microscopy on a SEM TESCAN VEGA LMU. The accelerating voltage was 30 kV, spot size - 210 nm, current of the electron beam - 0.23 nA and exposure time was 10 min.

Optical microscopy (confocal laser-scanning microscope Leica TCS-SPE) and scanning electron microscopy, SEM (TESCAN VEGA) were used to study the modification induced by e-beam irradiation of thin films. The topography of the structures drawn on the substrate was measured using Atomic Force Microscope (AFM) Veeco CP II in tapping mode.

Leica TCS-SPE was used for in-situ observation of thin film etching process as shown in (Shiman, Gerbreders, Sledevskis, Bulanovs, & Pashkevich Valfrids, 2011). Organic based solution e.g. amines was used for selective etching thin films. The etching solution were not stirred and kept at laboratory temperature during etching process.

3. RESULTS AND DISCUSSION

It is well known that an electron-beam bombardment can induce structural changes and generate micrometer and nanometer patterns in many materials, including chalcogenide glasses where its effect is rather similar to the light-induced changes (NEILSON, KOVALSKIY, VLCEK, JAIN, & MILLER, 2007). Fig. 1 shows the surface pattern produced by the electron beam and laser. For the tests presented here, dot patterns were exposed. It is seen that e-beam irradiated surface is modified while laser treated area has uniform surface. Laser-treated unetched sample (Fig. 1a): the space between the crystallites is probably filled with amorphous Sb40Se60.

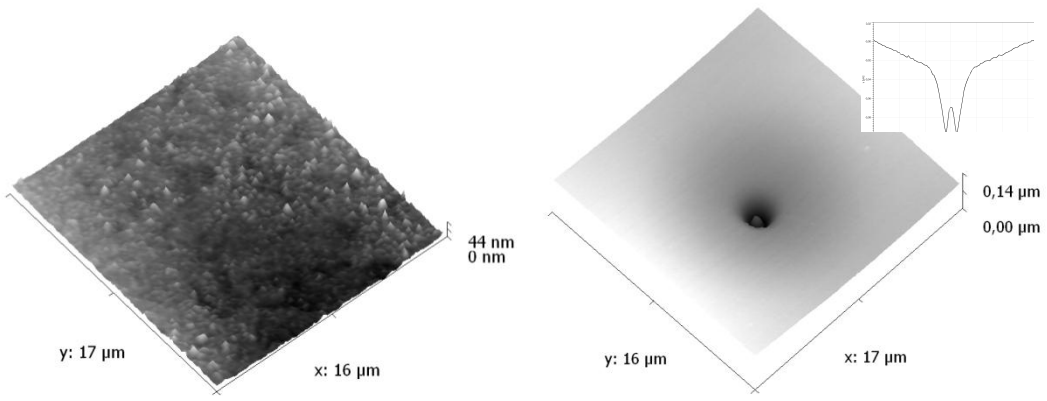

Fig. 1. AFM image of the surface topography in 3D for the $Sb_{20}Se_{80}$ photocrystallized (a) and e-beam treated (b) non-etched thin films.

Figure 2 shows the reflected signal versus etch time for $Sb_{40}Se_{60}$ thin films. Very high etch selectivity was detected in the samples: non-irradiated regions are completely dissolved, whilst locally laser or electron beam treated areas remain intact: intensity of the reflected signal remains practically unchanged as it is schematically depicted on Fig. 2. The etching rate of amorphous phase is 1.8 nm/s. High etch selectivity is connected with changing of the structure upon the irradiation.

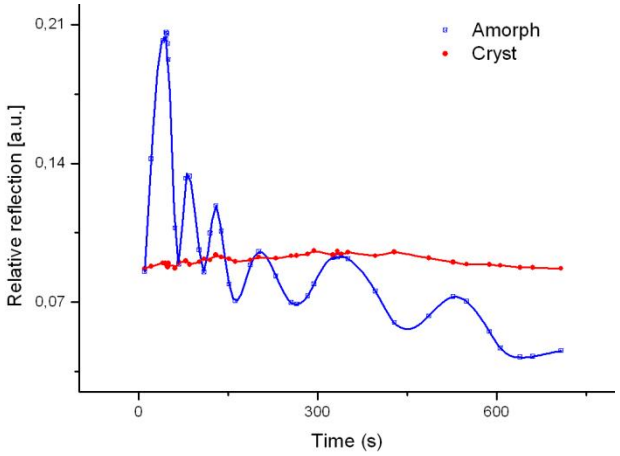

Fig. 2. Kinetics of dissolution of crystallized and non-crystallized areas of $Sb_{40}Se_{60}$ in organic etcher (room temperature).

Laser-treated unetched sample has uniform surface without height discontinuity. In the process of etching the amorphous regions are dissolved, whilst homogeneously crystallized areas represent structures with clearly/sharply defined edges. It was confirmed that undissolved $Sb_{40}Se_{60}$ island crystallites were 100 nm in height (Fig.3). The height of the crystallites are equal to the film thickness.

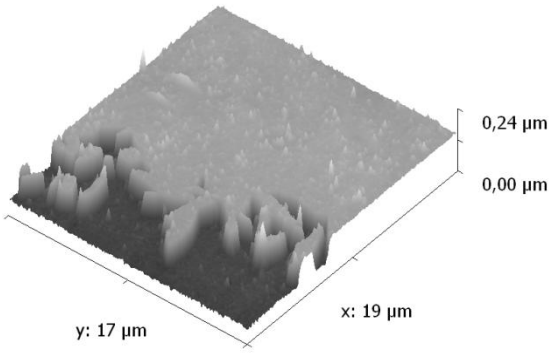

Fig. 3. AFM image of the surface topography in 3D for the $Sb_{40}Se_{60}$ laser crystallized etched thin films.

After the laser crystallization process $Sb_{40}Se_{60}$ thin film sample surface becomes more flat and smooth compared to the amorphous region. Fig. 3 shows typical AFM images of the surface topography in 3D for the stoichiometric compound thin films in crystalline phase: the small grains had coalesced, resulting in a uniform surface with some additional big grains observed on the film surface.

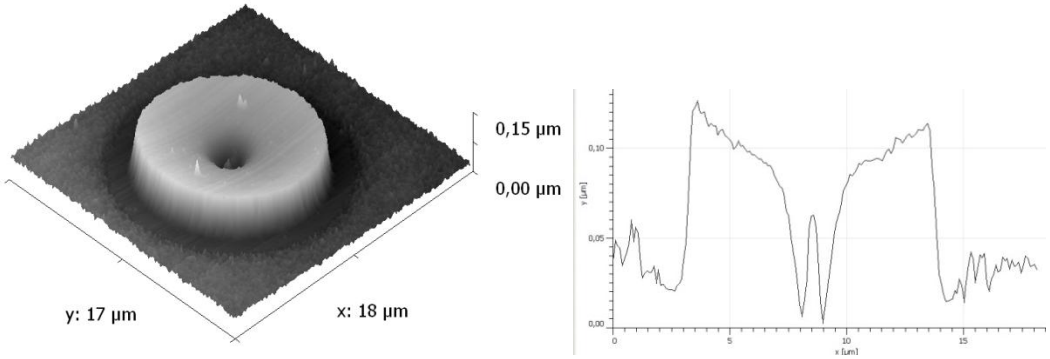

Fig. 4. AFM image of the surface topography in 3D and cross-section for the $Sb_{40}Se_{60}$ electron beam exposed etched thin films

In the beginning of electron beam (EB) irradiation process, a dimple is formed at the EB irradiation point, and the shrinking volume increases radially and in-depth with increasing exposition time. As exposition time increases further, a cone is produced at the center of the dimple. The height of the cone increases with radiation time. With increasing an exposure time, depth and width (spot size) of the dimple increases regardless of beam parameters. After 10 minutes exposure the dimple parameters reach their maximum value: 17 µm in width and 97 nm deep, whilst a total thickness of the chalcogenide layer is 100 nm (Fig. 4).

4. CONCLUSIONS

$Sb_{40}Se_{60}$ thin films have been exposed to the laser and electron beam irradiation with followed by etching in organic based solution e.g. amines. We showed the etching possibilities between local irradiated/non-irradiated $Sb_{40}Se_{60}$ thin films with regard to the surface quality. In all cases the irradiated area is more resist to organic etching solution in comparison with the non-irradiated area. We confirmed that, similar to exposure by suitable photons, exposure of Sb-Se films by electron beam significantly enhances their chemical resistance to alkaline amine based solutions. Thus smooth, shaped microstructures can be easily fabricated in these films. The direct surface relief formation during the exposure process by e-beam was obtained. It is very promising for practical application enabling the possibility to simplify technology of the surface patterning. Rather smooth and homogeneous surface of the samples after the photo- stimulation and after etching process as well as fine crystalline structure makes the $Sb_{40}Se_{60}$ composition attractive and promising for micro- and even nano-scale lithography.

Potential application of selective etching could be expected in the field of micro optical elements fabrication i.e. grids, waveguides, microlenses, highlighting phase change type recording memories.

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REFERENCES

Debnath, R., Nusbar, N., & Fitzgerald, a. (2005). Electron beam induced chemical modification of amorphous chalcogenide–metal bilayers and its application. Applied Surface Science, 243(1-4), 228-231. doi:10.1016/j.apsusc.2004.09.067

ELSAYED, S. (2004). Electron beam and gamma irradiation effects on amorphous chalcogenide SbSe2.5 films. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 225(4), 535-543. doi:10.1016/j.nimb.2004.05.033

Gerbreders, V, Teteris, J., Sledevskis, E., & Bulanovs, A. (2007). Photoinduced changes of optical reflectivity in As2S3 -Al system. Journal of Optoelectronics and Advanced Materials, 9(10), 3153 - 3156.

KUGLER, S., HEGEDÜS, J., & LUKÁCS, R. (2007). On photoinduced volume changes in chalcogenide glasses. Journal of Optoelectronics and Advanced Materials. Retrieved November 11, 2011, from http://newton.phy.bme.hu/~kugler/cikkek_kugler/JOAM07.pdf

Lyubin,Victor. (2009). Chalcogenide glassy photoresists: History of development, properties, and applications. physica status solidi (b), 246(8), 1758 - 1767. Retrieved from

http://resolver.scholarsportal.info/resolve/03701972/v246i0008/1758_cgphodpaa.xml

Mietzsch,K. (n.d.). Electron-beam-induced patterning of thin film arsenic-based chalcogenides. Applied Surface Science, 162-163, 464 - 468. Retrieved from

http://resolver.scholarsportal.info/resolve/01694332/v162-163inone/464_epotfac.xml

NEILSON, J., KOVALSKIY, A., VLCEK, M., JAIN, H., & MILLER, F. (2007). Fabrication of nano-gratings in arsenic sulphide films. Journal of Non-Crystalline Solids, 353(13-15), 1427-1430. doi:10.1016/j.jnoncrysol.2006.10.076

Romero, J. (2004). A transmission electron microscope study of metal/chalcogenide amorphous thin films. Applied Surface Science, 234(1-4), 369-373. doi:10.1016/j.apsusc.2004.05.039

Shiman, O., Gerbreders, V., Sledevskis, E., Bulanovs, A., & Pashkevich Valfrids. (2011). Selective Wet-Etching of Amorphous/Crystallized Sb20Se80 Thin Films . World Academy of Science, Engineering and Technology 75 2011, 75, 820-823. Retrieved from

http://www.waset.org/journals/waset/v75/v75-147.pdf

TANAKA, K., KYOHYA, S., & ODAJIMA, A. (1984). Anomaly of the thickness dependence of photodarkening in amorphous chalcogenide films. Thin Solid Films, 111(3), 195-200. doi:10.1016/0040-6090(84)90141-X

Tanaka, K. (2003). Nanostructured chalcogenide glasses. Journal of Non-Crystalline Solids, 326-327, 21-28. doi:10.1016/S0022-3093(03)00371-5

Utsugi, Y. (1993). Chemical modification for nanolithography using scanning tunneling microscopyUtsugi, Y. Nanotechnology - International Workshop on Atoms and Clusters (WAC-92); 1992 Jan 8; Atami, Japan. 1992 Oct. 161-3. Precision Engineering, 15(3), 201-201. doi:10.1016/0141-6359(93)90034-8