AFM Study of Ni- and Cu-Doped Li₂B₄O₇ Glass Surface

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Abstract. The results of atomic force microscopy (AFM) studies of surface of the lithium tetraborate glasses doped with Cu, Ni and annealed in the hydrogen (H_2) atmosphere are presented.

Key words: AFM, borate glasses, surface.

I. INTRODUCTION

Borate compounds represent a wide class of perspective materials in the form of single crystals, polycrystals or glasses. The tetraborates of alkali and alkaline earth elements are of particular importance important among them due to their potential for nonlinear optics, laser engineering, thermoluminescent dosimetry and radiation detection.

Surface has a significant influence on the parameters of optical devices based on borate glasses. Thus, the studies of the surface and the effect of various factors on the surface topology are very important. This paper reports the results of the AFM studies of Cu and Ni doped and annealed in H_2 atmosphere Li₂B₄O₇ glass surface.

II. EXPERIMENTAL

The glasses have been prepared using the normal melt quenching technique. Reagent-grade Li2CO3 and boric acid H₃BO₃ were used as raw materials. The Cu and Ni impurities were added into the $Li_2B_4O_7$ composition in the form of CuO and NiO oxide compounds in amount 1.0 mol. %. A 6 g batch of reagents, which corresponded to the chemical formula $Li_2B_4O_7$ of appropriate glass were mixed together by grinding the mixture repeatedly to obtain a fine powder. The mixture was melted in a Al₂O₃ ceramic crucible in an furnace under conventional atmospheric conditions at temperature 1270 K for Li₂B₄O₇ and homogenize the melt 2 h. The glasses obtained from the fast melt quenching to room temperature. After being annealed at glass transition temperature 730 K for 3 h, the glasses samples were then grinded and optically polished to have the dimensions $10 \times 7 \times 1$ mm³ for the measurements. To check the noncrystallinity of the glass samples, X-ray measurements were performed. The results showed that XRD patterns of the glasses did not reveal any discrete or sharp peaks, but broad bands characteristic of amorphous materials. The samples of glass were also annealed in a hydrogen atmosphere at 730 K during 8 hours.

The AFM studies were conducted using a NT-MDT Solver Pro instrument. The measurements were carried out in semi-contact mode with a scan size $1 \times 1 \mu m$, $5 \times 5 \mu m$ and $10 \times 10 \mu m$.

III. RESULT AND DISCUSSION

AFM images of the investigated samples are shown in Fig. 1-5. As can be seen in Fig. 1, there are relatively deep (up to 40 nm) pits on the polished surface of the sample of $Li_2B_4O_7$ glass before annealing.

Edges of the pit as well as other surface irregularities are smooth. Glass surface of the Cu doped $Li_2B_4O_7$ (Fig. 2) shows similar features.

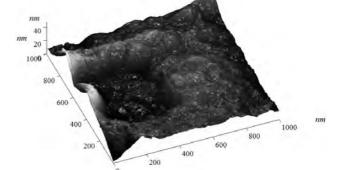


Fig. 1. AFM image of the surface of Li₂B₄O₇ glass before annealing.

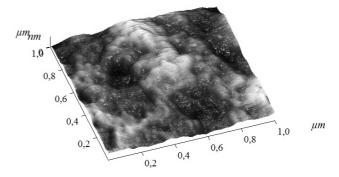


Fig. 2. AFM image of the surface of glass Li₂B₄O₇:Cu before annealing.

One observes a completely different topology in case of Cu doped $\text{Li}_2\text{B}_4\text{O}_7$ glass surface subjected to a long-term annealing of the sample in the hydrogen atmosphere at 730 K (Fig. 3). There are clearly seen sharp peaks up to 150 nm backgrounded by a relatively flat surface. The nature of these peaks is yet to be investigated. One can also notice a deep and wide depression area, which is due to the scratching of the surface during sample polishing procedure. Edges of the depression region are sharp as compared to the edges of the pits in Fig. 1. Therefore, it can be argued that the surface of the samples of lithium tetraborate glass is a subject to change during the annealing at temperatures in the

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region of the glass transition temperature.

A somewhat different situation is observed with samples of Ni doped $Li_2B_4O_7$ glass. Surface topology of $Li_2B_4O_7$:Ni sample before annealing (Fig. 4) resemble that of the $Li_2B_4O_7$: Cu sample before annealing (Fig. 2). However, the

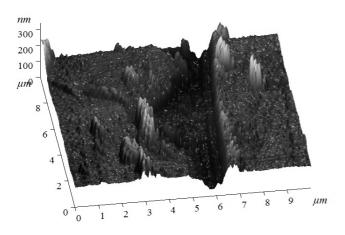


Fig. 3. AFM image of the surface of $Li_2B_4O_7$:Cu glass annealed in H_2 .

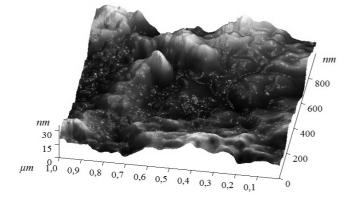


Fig. 4. AFM image of the surface of $Li_2B_4O_7$:Ni before annealing.

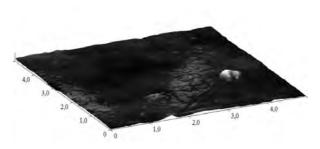


Fig. 5. AFM image of the surface of Li₂B₄O₇:Ni glass annealed in H₂.

surface of the annealed $Li_2B_4O_7$:Ni sample (Fig. 5) significantly differs.

First, it becomes much more smooth, and, secondly, the formations which are shaped like metallic nanoparticles appear on the surface.

As seen from Fig. 5, the concentration of metallic particles in the sample is small, but their number can be increased by increasing the contamination of NiO in the melt during the preparation of $Li_2B_4O_7$: Ni glass.

IV. CONCLUSIONS

In this paper we investigated the AFM topology of pure and Cu and Ni doped tetraborate glasses, both as-prepared and annealed in hydrogen atmosphere.

V. ACKNOWLEDGMENT

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