

A study of the effect of holding time on the crystallization of ZnO-Al₂O₃-SiO₂ glass

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Abstract— Glass ceramics materials were prepared from a ZnO-Al₂O₃-SiO₂ glass, these glasses were heat treated at 890°C during 2,4 and 8 hours. An optical study using a Scanning Electron Microscopy, and Spectro-colorimetry were carried in order to find the effect of the holding time on the crystallization of this glass.

Keywords— Glass-ceramic, Crystallization, SEM, Colorimetry

I. INTRODUCTION

Glass-ceramics are polycrystalline materials formed by controlled crystallization of the glass with a designed schedule of heat-treatment, exhibiting a number of outstanding characteristics in comparison with the traditional ceramics and glasses and a wide range of exciting potential applications [1-2].

In this work the effects of the holding time on the crystallization behavior of ZnO-Al₂O₃-SiO₂ glasses were investigated with a scanning electron microscopy, and Spectro-colorimetry.

II. Experimental

Three samples of ZnO-Al₂O₃-SiO₂-CaO- Na₂O glass have been heated at 890°C for 2,4 and 8 hours in an electric oven, with the rates of heating and cooling from room temperature to the holding temperature and conversely, in each heat treatment performed were 2 °C min⁻¹ and 5 °C min⁻¹ respectively.

The crystallization behavior of glasses was studied using a Hitachi S-4700 Field Emission Scanning Electron Microscope (FE-SEM). Also, the L * a * b* measurements of these samples were made with a Konica Minolta CM-2600 Spectro-colorimeter.

III. Results and Discussion

L*, a*, b* coordinates of each sample (A, A2, A4, and A8 which are respectively the original glass and the glasses heated for 2,4, and 8 hours) which indicates color differences, are shown in **Table 1**. Samples tended to be white and the A8 sample is the whitest.

TABLE I. L*, a*, b* data of the samples.

Sample	A	A2	A4	A8
L*	24.225	32.285	33.99	74.285
a*	0.65	-0.635	-0.635	-3.83
b*	10.52	5.43	5.845	3.41

SEM micrographs of the three heated samples are presented in **Figure 1**, **Figure 2**, and **Figure 3** with different magnification for each one. On the first glass A2 (**Figure 1**) the crystallization reached only the surface layer of the sample. On the other hand, in sample A4 (**Figure 2**) a dense and homogeneous crystallization with smaller crystals. when in **Figure 3** it shows that the A8 glass has also a dense and homogeneous crystallization but in this one with a large size of crystals. the crystals that appear in the three treated glasses are of the Wollastonite [3] type.

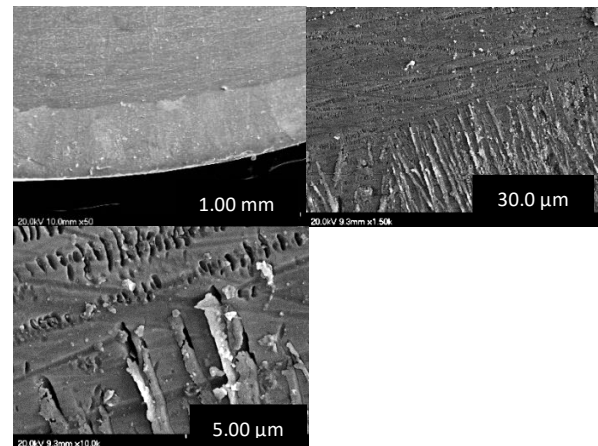


FIGURE 1. SEM images of the glass-ceramic heated for 2 hours (A2)

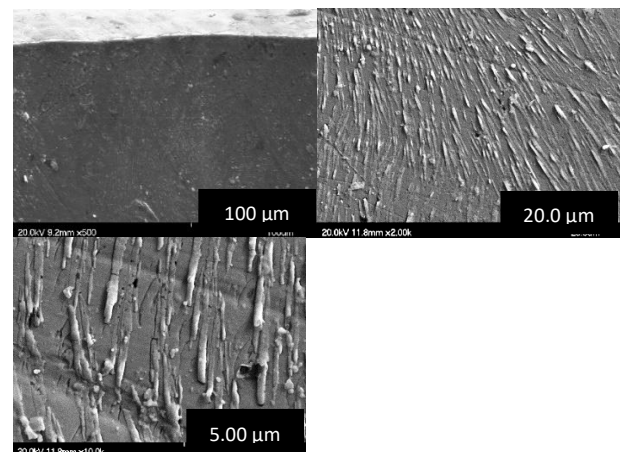


FIGURE 2. SEM images of the glass-ceramic heated for 4 hours (A4)

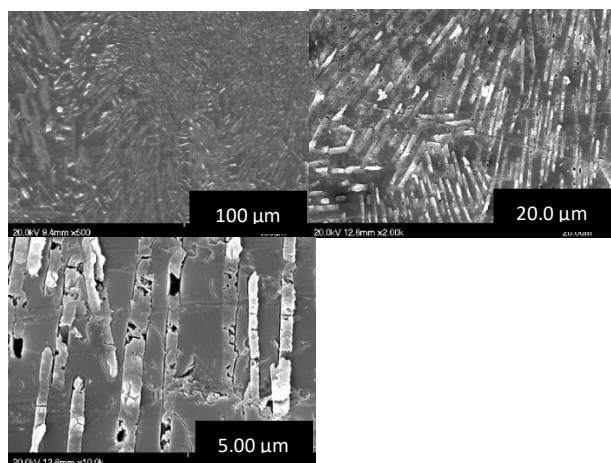


FIGURE 3. SEM images of the glass-ceramic heated for 8 hours (A8)

IV. Conclusion

In this work, a white glass ceramic was found. As well as a description of the crystallization behavior of the glasses and the impact of the holding time on this phenomenon was made, in order to give the best performance in terms of glass ceramic production.

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