INVESTIGATION OF THE MICROHARDNESS OF SOME OXIDE GLASSES

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INTRODUCTION

It is well known that many properties of vitreous materials mostly depend on the nature, shape, and size distribution of submicroscopical crystalline or amorphous phase precipitations; for example such microphases play an important role in the production of glass-ceramics, microporous glasses, photochromatic glasses etc. Besides electron microscopy, mainly small-angle X-ray scattering studies have contributed to clarifying the mechanism and kinetic of microstructure forming processes in several glass systems [1]. The microhardness method has been proofed useful for both the mechanical behaviour and the physics of plastic deformation of various glasses [2]; in this report an attempt will be made to establish changes in the microstructure of some phase separating oxide glasses by this method.

EXPERIMENTAL

For our investigations three different glass systems were chosen. The samples belonging to system A have the composition (in wt. %): 12 BaO-26B_2O_3-27Al_2O_3-35SiO_2; while the systems B and C consist of 11MgO-32Al_2O_3-48SiO_2-7ZrO_2-2TiO_2, and 7Na_2O-23B_2O_3-70SiO_2, respectively. The melts of each composition were poured out into moulds and then thin slices were cut from the solidified glasses. In order to induce phase separation in these samples they were heat-treated for various periods of time at different temperatures. The temperatures of isothermal annealing were: for system A – 973 K, for B – 1093 K, and 903 K for system C. The specimens used for microhardness measurements, and for X-ray scattering studies too [3, 4], were surface-polished using the standard polishing technique for optical surfaces. The Vickers microhardness measurements were performed using a Zeiss (Jena, DDR) microhardness attachment and at least twenty indentations were performed on each sample.

RESULTS AND DISCUSSION

Fig. a) shows the dependence of microhardness on applied load obtained on sample b in Fig. b). It can be concluded that for load exeeding roughly 1 N, microhardness hardly depends on load. Thus, load of 1 N was used in subsequent experiments with the exception of the samples belonging to system C where a load of 0.2 N was used. Fig. b) shows the results obtained from the samples which were isothermally treated for various periods of time and temperatures given above. The difficulties which are accompanied with an exact determination of the microhardness of polished, viscoelastic glass samples are well known [5]. The vertical lines shown in Figs. a) and b) represent a measure for the certainty but not for the accuracy of our experiments! Consequently, the H_V values given in Figs. a) and b) are to consider only as hardness numbers relative to the microhardness of the respective reference sample. Similar microhardness behaviour can be observed for samples of the systems A and B, i.e. firstly, the microhardness decreases for the shorter annealing times while it increases for longer annealing. The drop in microhardness at the beginning might be connected with an annealing out of stresses introduced into the samples during cooling down the melt, while the increase of the microhardness for the samples annealed for the longer times is undoubtly caused by the decomposition and/or crystallization of the initial glass structure. Namely, it has been shown [4] that the untreated sample of system A contains a great number of small spherical particles with diameters of about 2 nm. During a heat treatment for 16 hours at 973 K or 8 hours at 1073 K (sample a) these particles crystallize to mullite and their size increases tenfold by Ostwald ripening. It is interesting to note that no further increase of microhardness is observed for sample b which is a complete glass-ceramic containing needle like mullite crystals more than 1 μm in length. During the isothermal annealing of the samples of system B nucleation and growth of one amorphous and two crystalline phases take place [3]. On the other hand, two interconnected amorphous phases exist in the samples of system C [1, 4]. During the heat treatment a coarsening process is running down and only a hardly visible maximum in microhardness appears.

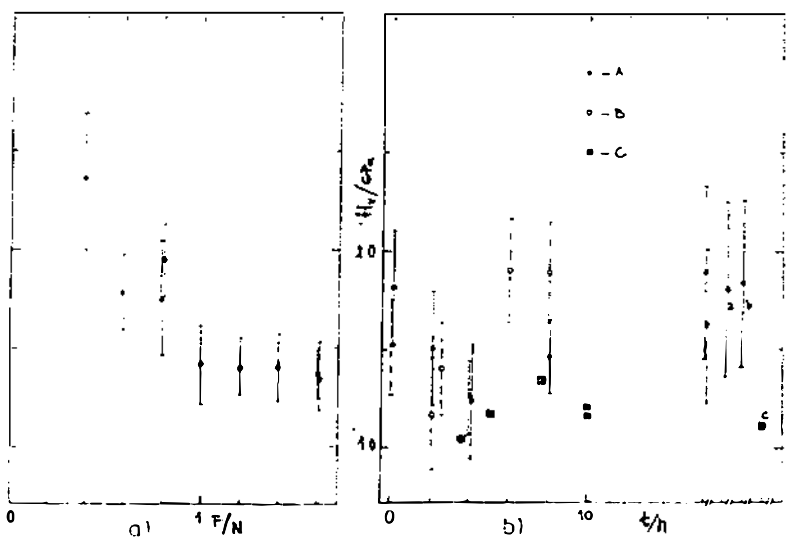

Fig. a) Dependence of microhardness H_V on indenter load F.

Fig. b) Dependence of microhardness H_V on the time t of isothermal annealing. Microhardness values indicated by a, b and c belong to samples annealed for 8 hours at 1073 K, for 16 hours at 973 K plus 1 hour at 1423 K, and 5 hours at 943 K, respectively.

CONCLUSIONS

It was established that the microhardness method can be useful in studying the development of microstructure of oxide glasses induced by decomposition or crystallization in the course of a heat treatment. For further investigations efforts should be made to avoid stresses that arise from quenching the glass melt.

REFERENCES

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