CRYSTAL GROWTH MEASUREMENTS ON A SODA-LIME- SILICA GLASS SPHERE USING FEG-SEM

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An alternative route to glass-ceramics technology is sintering and crystallization of glass powders. However, many experiments must be carried out to know the best conditions to obtain a dense sintered body or one having controlled porosity. In ongoing research we propose and use a computational simulation technique to verify if a glass sinters before it crystallizes or vice-versa, before any sintering experiment is carried out. For this purpose two models that describe the sintering of glass - the Frenkel and Mackenzie-Schuttleworth model - and the Johnson-Mehl-Avrami (JMA) theory of crystallization were used. To test the simulation, soda-lime-silica glass spheres with average diameters of about 200 μm were used. The crystal growth rate (U) of spherulitic crystals developed on the surface of the spheres was obtained through FEG-SEM. This technique was important due the following reasons: i) the crystal nucleation rate on the surface spheres is high and the crystals are too small (1-10 μm diameter) to be measured with precision by optical microscopy, ii) examination at low voltage in a FEG-SEM suppressed the need for coating the sample, permitting to treat the same sample at different times, without interference of the coating on the crystal growth rates.

Rabinovich [1] reported that glasses that show low surface crystallization rates are suitable for sintering. If a glass presents volume crystallization and/or high surface crystallization rate it crystallizes so fast that the viscosity increases to near infinity and sintering (by viscous flow) stops [2]. However, it is common to find in the literature that many experiments have to be made to achieve the best conditions to obtain a dense sintered body; sometimes without success. Glass properties, which greatly affect sintering, such as viscosity, particle sizes and nucleation and crystallization rates, can be measured in the laboratory. Thus, using this data it is possible to predict by simulation, before any sintering experiment, if a glass system is favorable to produce a fully dense glass-ceramic body. In this communication attention was given to crystal-growth rate measurements using FEG-SEM.

The system chosen for this study was commercial soda-lime-silica glass spheres (Potters) since most sintering models are based on spherical particles. A sample was placed in a stabilized furnace at 680 °C, initially for 6 h. After this time it was removed from the furnace and analyzed, without coating in a XL-30 FEG –Philips using 20kV. After obtaining an image, the sample was replaced in the furnace for additional 2h. This procedure was repeatedly carried out until the impigement of the crystals.

Figure 1 shows two micrographs of the glass sphere heat-treated at 680 °C for 8h and 10 h, respectively. The same region was taken in order to measure the size of the same crystal, to obtain the crystal growth rate.

FEG-SEM is an important tool to measure the crystal growth rate of soda-lime-silica glass spheres and inferentially of other glasses. Although the sample was not coated with a conductive material the quality of the images were good for measuring this important parameter to be used in the simulation.

ACKNOWLEDMENTS

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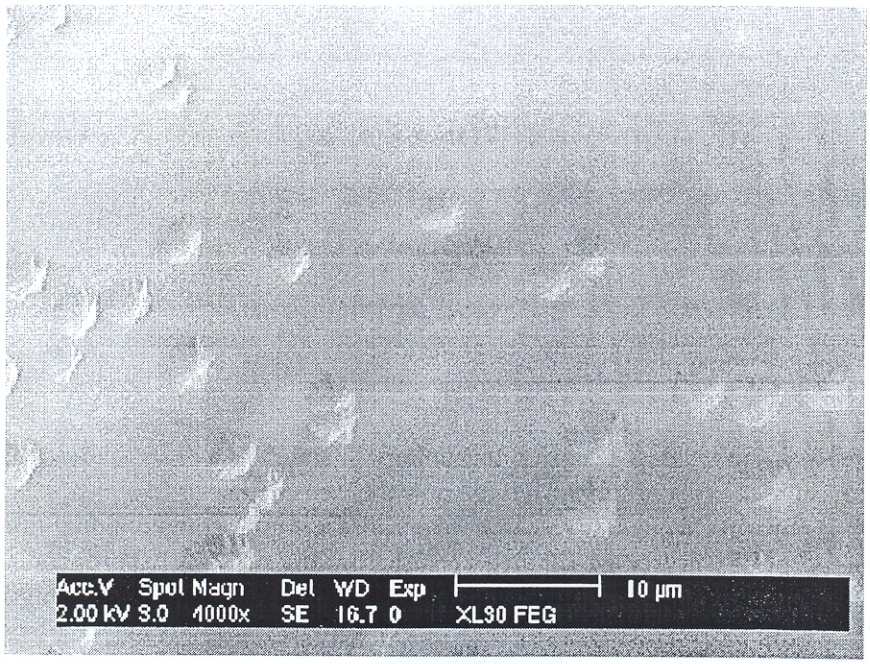
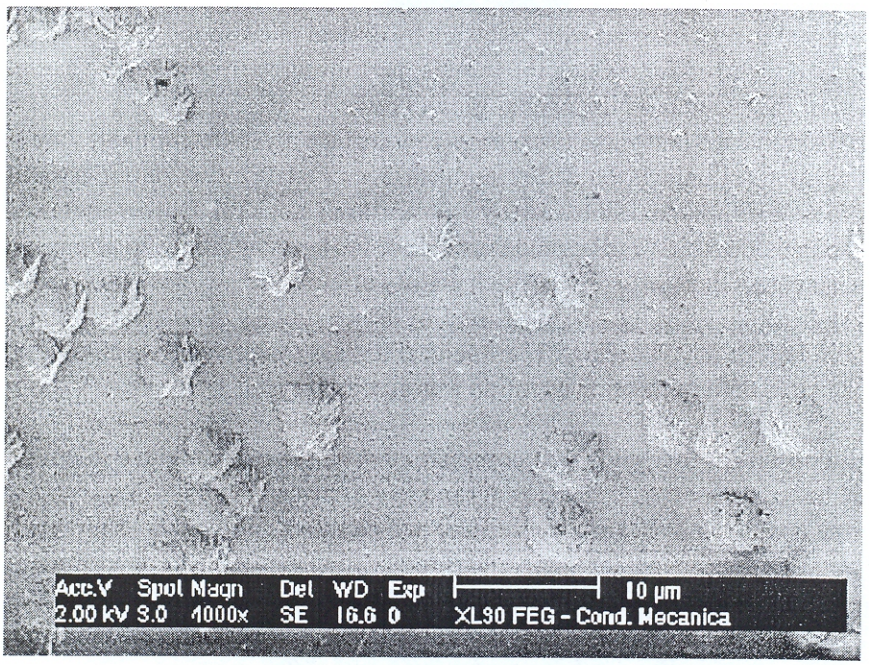
REFERENCE

1. RABINOVICH, E.M – *J. Mater. Sci.*, 20 (1985), 4259-97

2. KNICKERBOCKER, S., TUZZOLO, M.R.,LAWHORNE, S- *J. Am. Ceram. Soc.*, 72[10], 1989, 1873-79.



(a)



(b)

Figure 1. FEG-SEM micrograph of the glass sphere heat-treated at (a) 680°C/8h and (b) 680°C/10h.