Effect of nickel oxide on crystallization and mechanical properties of $MgO-Al_2O_3-SiO_2-MoO_3$ glasses

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Introduction

Crystallization is a well-recognized practical means of improving the brittleness of glass. Many glass-ceramics have been investigated, and some have been incorporated into practical materials. Glass-ceramics in the $MgO–Al_2O_3–SiO_2$ (MAS) system have attracted much interest on account of their superior mechanical and thermal properties, i.e., high strength and stability at high temperatures.

To investigate the effect of nucleating agents, this study investigates the crystallization behavior of an $MgO–Al_2O_3–SiO_2$-MoO_3 glass with high % wt molybdenum oxide and low % wt nickel oxide addition. Under a not reducing condition, crystals formed in the bulk of the super cooled liquids, whereas melting in air yielded only surface crystallization. The purpose of using of Nickel oxide as nucleating to modify the mechanical properties, is based on the manipulation of the grain size during the solidification process. The first crystalline phase precipitated inside the glass was α-cordierite and β-cordierite in glasses doped with nickel oxides. Nickel oxide yielded a finer microstructure of the glass-ceramic.

Materials and Methods

Samples were prepared with 4 chemical compositions, the sample M7 (*31.83 % Al_2O_3, 12.09 % MgO, 55.42 % SiO_2*), M7-1A (*31.5117 % Al_2O_3, 11.9691 % MgO, 54.8658 % SiO_2, 1% NiO)*, M7-1B (*31.1934 % Al_2O_3, 11.7273 % MgO, 54.3116 % SiO_2, 2% NiO)* and M7-

1C (*30.8751 % Al_2O_3, 11.7273 % MgO, 53.7574 % SiO_2, 3% NiO)*. Figure 1 summarizes the experimental procedure that was followed for the development of this Project, in figure 2 we can observe the appearance of the vitreous samples before and after the thermal treatment at 1200 ° C.

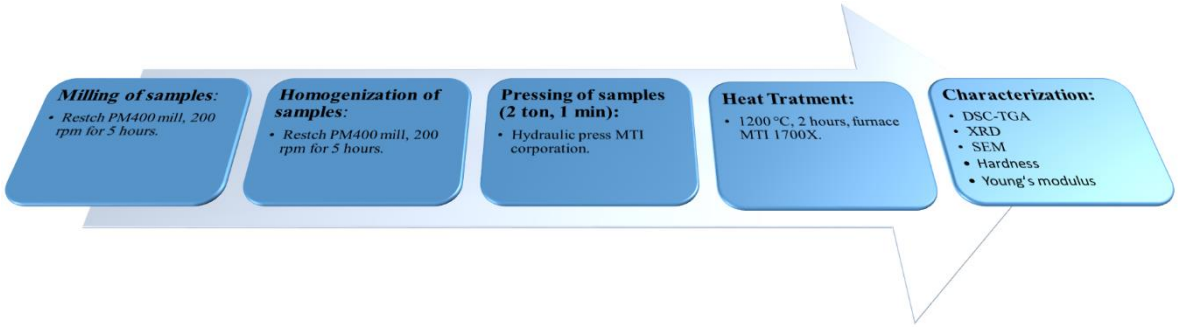

***Figure 1**. Experimental procedure.*

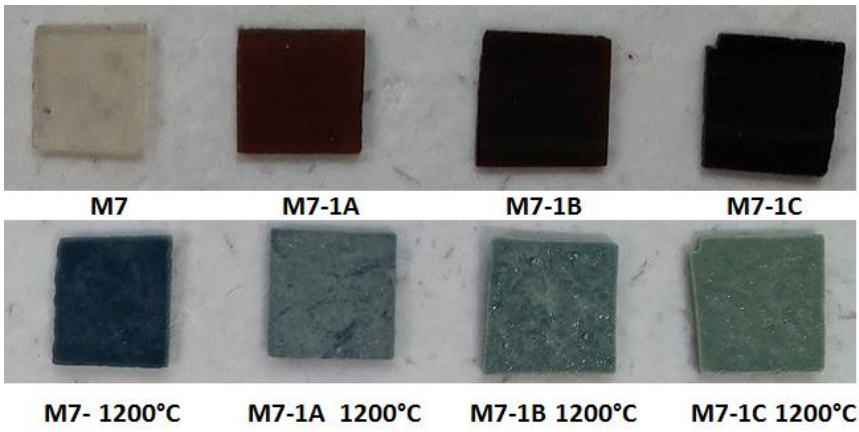

***Figure 2**. Samples before and after thermal treatment at 1200° C during 2 hours.*

Results

Figures 3 (a) and 3 (b) present the thermal analysis results TGA and DSC, respectively, for different samples in which the chemical composition was varied, in Figure 3 (a) it can be seen that the presence of the nickel increases the thermal stability of glass by 250°C. At a temperature of 1000°C the weight loss was 2% and was higher in samples with higher nickel content compared with the original sample. At a temperature of 1400°C the weight loss is greater (13%) in both, the original sample and that which contains the highest quantity of nickel. The abrupt weight loss occurs at temperatures very close to 1200°C due

to the thermal evaporation of MoO_3. In the same context, in Figure 3 (b) it can be seen that in the original sample only the peak related to the crystallization of the cordierite phase is visible, at a temperature of 920°C, while the presence of nickel increases the temperature at 950°C at which the transformation takes place. The nickel addition also causes the formation of the indialite phase which is greater with the increase in the amount of nickel.

On the other hand, Figure 4 presents the XRD diffraction patterns performed on the four samples of the study. As shown, XRD only detected the presence of cordierite with two different crystal structures, one orthorhombic (ICDD: 01-076-6037) and the other one hexagonal (ICDD: 01-084-1221). The presence of the indialite phase could not be identified by this technique because of the limits of detection. Figure 5 presents the microstructure of the materials obtained from scanning electron microscopy (SEM). As can be seen in Figure 5 (a), a well-defined crystalline zone and areas of vitreous phase were identified. The effect of the presence of nickel can be seen mainly in the morphology of the crystals formed after the heat treatment at 1200°C during 2 h (Figures 5(b)-5(d)), in which the crystallization is favored. The size of the crystal decreases as more nickel content is added, however in the sample with the highest nickel content (Figure 5 (d)), it also increases the percentage of porosity. The optimization in the relationship of the crystallographic orientation and the size and homogeneity of the crystal is presented in the sample with 2% e.p. addition of nickel.

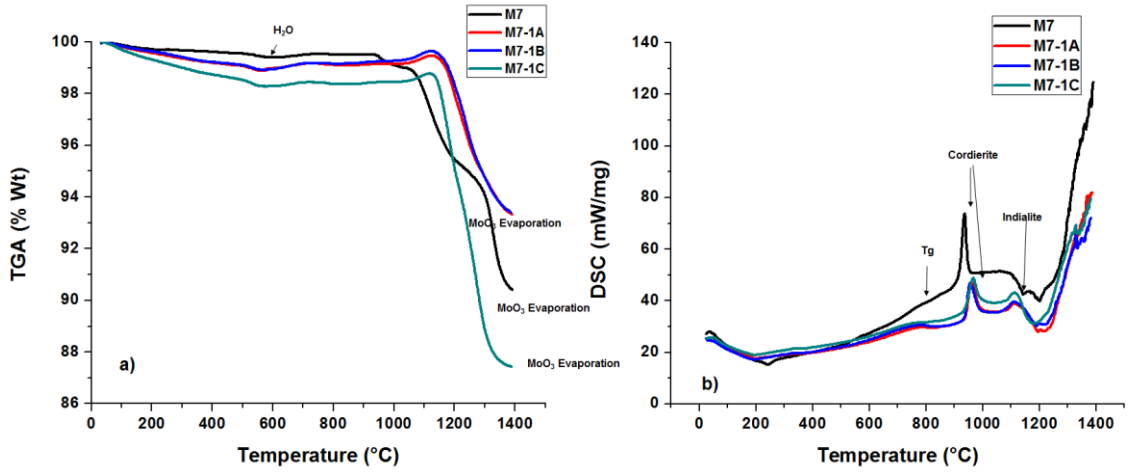

Figure 3. *a) TGA and b) DSC of samples M7, M7-1A, M7-1B y M7-1C.*

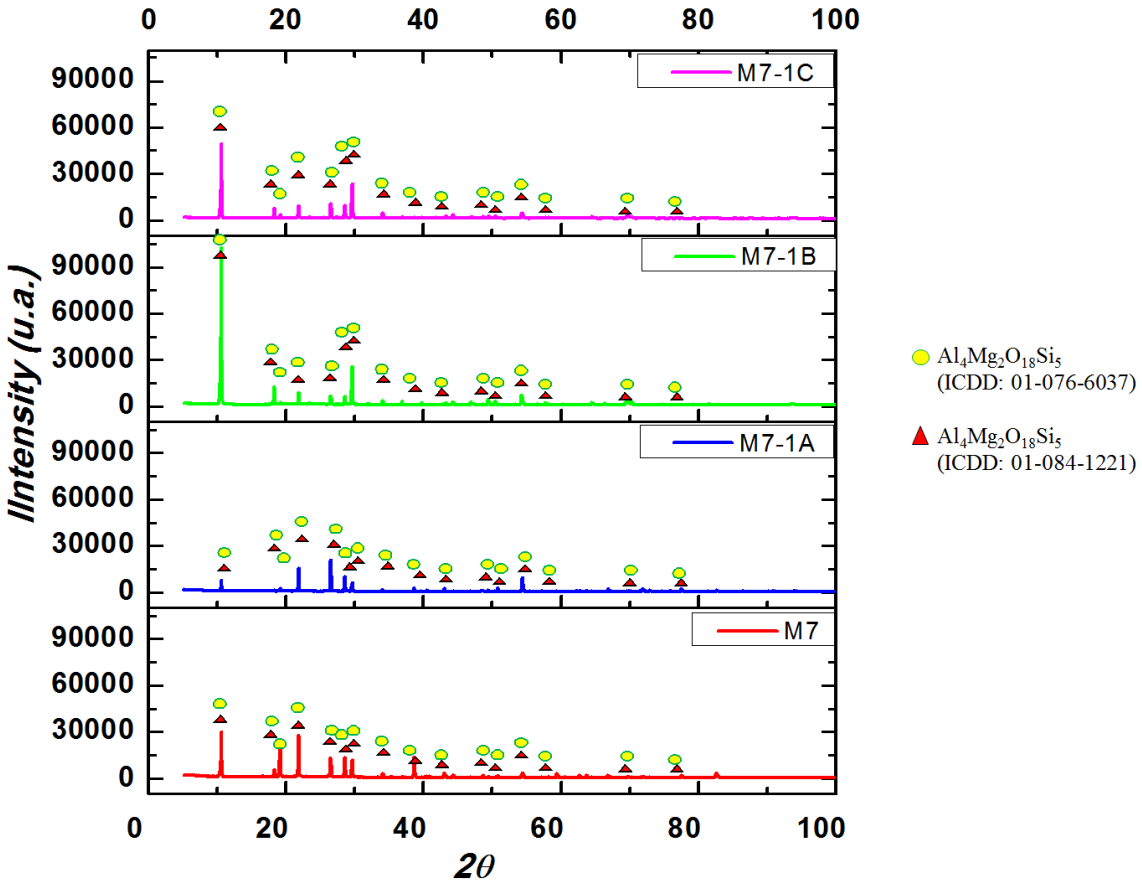

Figure 4. *X-Ray Diffraction, Structural evolution depending on temperature, M7, M7-1A, M7-1B y M7-1C.*

Figure 6 presents the comparative results of the mechanical evaluation measured by the Nanoindentation technique of the original sample and the samples with the addition of nickel. As can be observed, in the original sample the crystalline phase presents a low Young modulus, whereas this property increases in the vitreous phase. As can be seen, the greater Young's modulus is obtained in the two samples with the greater amount of nickel. Evidently the variability is greater in the original sample due to the heterogeneity in the percentages of the present phases. The largest Young's modulus was obtained in the sample with 2% nickel addition, while the highest nano-hardness was obtained in the vitreous area of the original sample. However, said zone is minimal with respect to the size of the crystalline zone. Of the samples to which the nickel was added, the highest nano-hardness was obtained, again in the sample with addition of 2% e.p. of nickel. Therefore, both the distribution and the size of the phases and crystals formed, have an impact on the

mechanical performance of the bulk material, as can be seen in Figure 6 (c). The highest Vickers microhardness was obtained for the samples with addition between 1 and 2 Wt% of nickel, the difference in the value differs minimally by the homogeneity due to the orientation and the amount of crystalline phases present in the overall microstructure of the material.

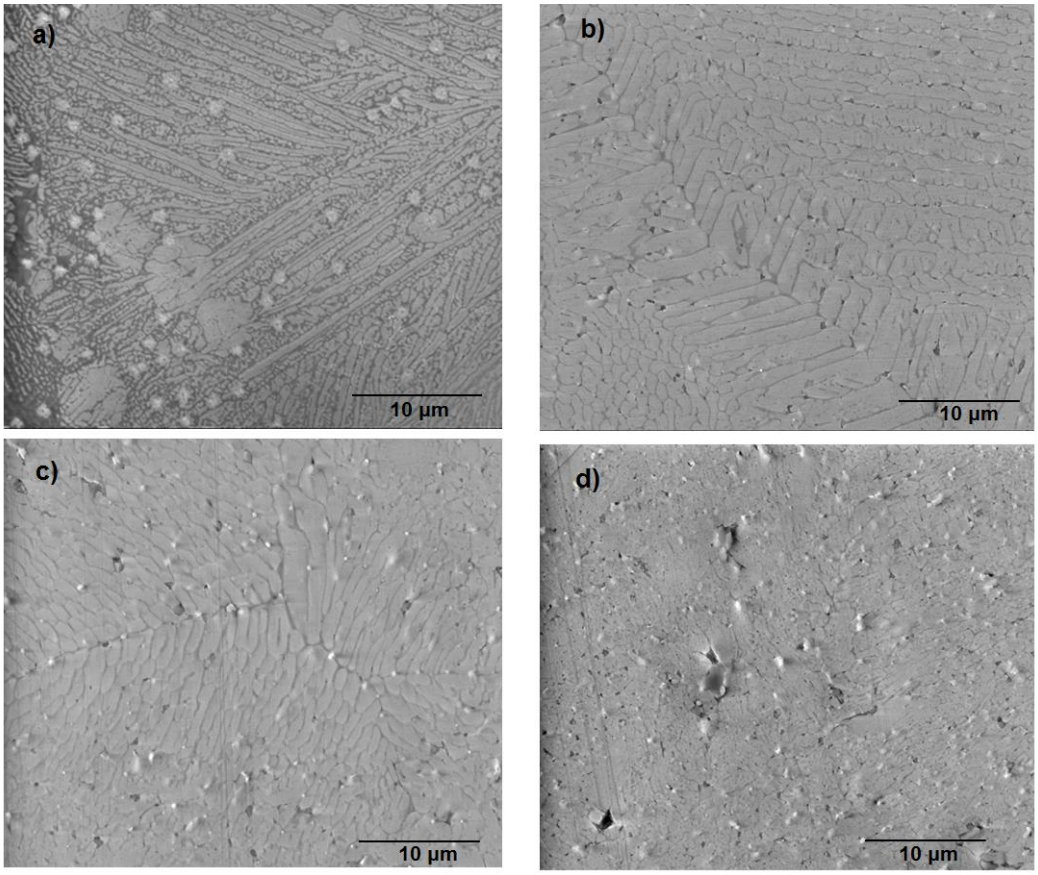

***Figure 5**. SEM a) M7, b) M7-1A, c) M7-1B and d) M7-1C, heat treatment at 1200°C during 2 hours.*

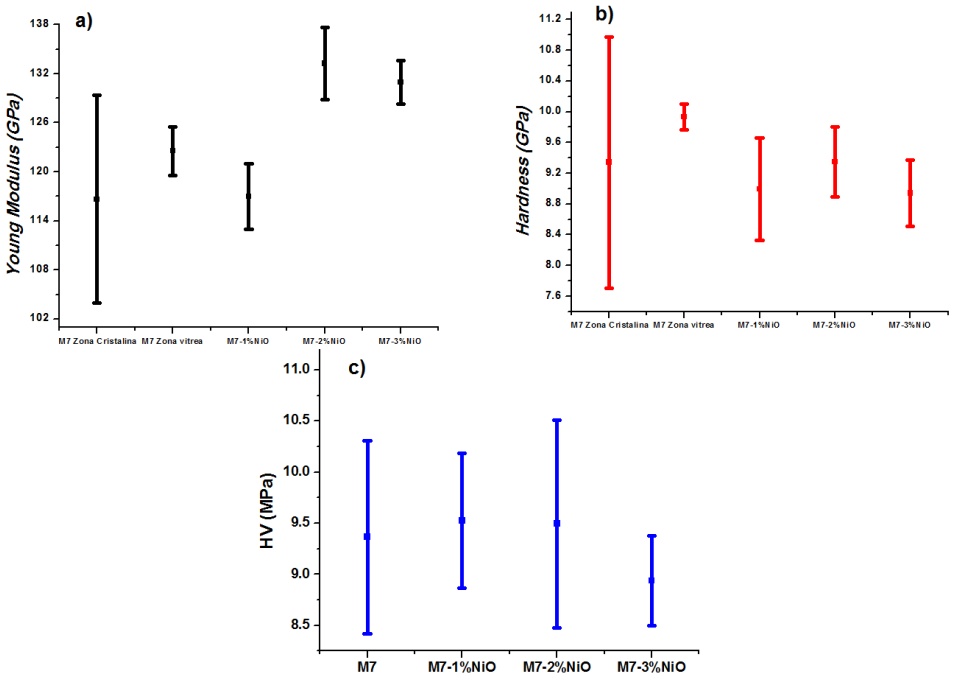

Figure 6*. Mechanical properties a) Young Modulus, b) Hardness and c) Hardness Vickers.*

Conclusions

The results obtained with the development of this work demonstrate that the addition of NiO to near stoichiometric crystals of cordierite nucleated with MoO_3 influence the degree of crystallization and the mechanical properties of the material. The NiO addition modifies the crystallinity of the cordierite as seen in the results of XRD, and also improves the Young´s Modulus.

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199- AMC
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