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Leucite Crystallization: Kinetics of Nucleation and Growth

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Abstract – Leucite feldspathic glass-ceramics is used for making dental prothesis. Leucite grains have been nucleated and grown from feldspathic glass frit powders. The kinetics of nucleation and growth of leucite crystals in the glass matrix was investigated under non-isothermal test conditions using differential thermal analysis (DTA) and characterization by using X-ray diffraction (XRD), and scanning electron microscopy (SEM). The activation energy of leucite crystallization was determined by the Kissinger method.

Leucite grains have been nucleated and grown from feldspathic glass frit powders which were produced by melting from a mixture composed [1] by 56.1wt% SiO_2, 19.9wt% Al_2O_3, 0.7wt% CeO_2, 11.2wt% K_2O and 10.5 wt% Na_2O, starting from Armil (Brazilian) Feldspar, and commercial grade Na_2CO_3, K_2CO_3, borax, CeO_2, Al_2O_3. The melting has been carried out at 1200°C for 3h, followed by iced-water quenching. The glass frit was ground in an automatic mortar and sieved using 325 Tyler-mesh, with the coarser particles returning to the grinder.

Figure 1 shows the X-ray diffraction pattern of the ground frit powder whereby it is seen its amorphous nature. Figure 2 presents the DTA curve of the glass frit powder heated up to 1100°C at a rate of 10°C/minute, where it can be seen that the crystallization of the leucite occurs simultaneously with 'glass-matrix melting', which is an endothermic process. In Figure 3 it is seen the round leucite crystals (SEM picture) while Figure 4 presents the XRD of the same final product generated in DTA test.

Figure 5 shows the DTA curve corresponding to leucite crystallization without the contribution from glass-melting appeared in Figure 2. DTA tests have been performed at different heating rates: 2.5, 5, 10 and 20°C/minute and their peak-temperatures have been used to calculate the activation energy of leucite crystallization by the Kissinger equation [2-4]:

$$\ln\left(\frac{\beta}{T_p^2}\right) = -\frac{E}{RT_p} + C$$

where β is the heating rate, E is the activation energy, T_p is the exothermic peak temperature and C is a constant. The plot of $\ln(\beta/T_p)$ vs $1/T_p$ is shown in Figure 6. Therefore, the value of E determined from the slope of this plot was 333 kJ/mol.

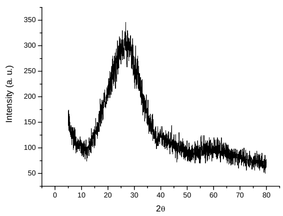

Figure 1: XRD of the glass frit powder.

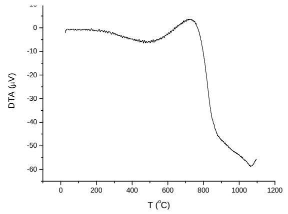

Figure 2: DTA curve of a glass-frit powder at a rate of 10°C/minute.

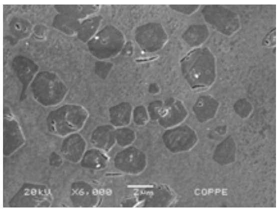

Figure 3: SEM picture of leucite crystals.

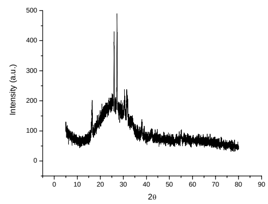

Figure 4: XRD of the final product generated in DTA at a rate of 10°C/minute.

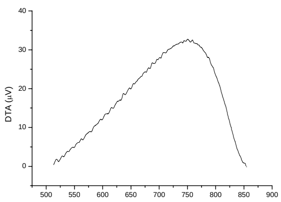

Figure 5: DTA curve without the contribution from glass-melting.

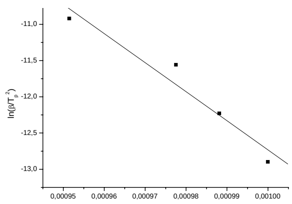

Figure 6: Kissinger plot of data obtained from DTA curves.

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