

TEM / HREM observations of thin foils obtained by SEM-FIB for the study of the phase transitions in chromium slag vitrification

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A glass - ceramic obtained by controlled vitrification of slag with high levels of Cr⁺⁶ has been investigated under TEM transmission and analyzed in HREM (image high resolution mode). The partially crystallized areas were identified and other regions fully devitrified by nucleation and controlled crystalline growth. The mineralogical composition of the considered phases have shown, that these materials after their respective processing, contain pyroxenes in augite form, and crystallization of (Mg, Fe) (FeAlCr)₂O₄ spinels embedded into a residual vitreous phase that are produced from the composition of the vitrification within the region of MgO-CaO-Al₂O₃-SiO₂ composition. A sample has been selected according to its optimum characteristics from physical, mechanic and microstructural point of view, in order to elucidate in detail the vitrification aspects that have not been able to be detected with SEM/EDS[1].

The preparation of the samples were held at a double beam high resolution electronic scanning microscope (SEM/ FIB) Nova-200 Nanolab with a magnifying capacity from 30X up to 1 200 000X and with a resolution until 1.1 nm., which has a Focalized Ion Beam column (FIB) that allows selection and separation by ionic thinning of interest area within a range from micrometric to nanometric dimensions. Simultaneously, it is possible to perform the polishing (thinning of the sample) in a controlled manner until a thickness of less than 100 nm [2-4]. For the observations with TEM and HREM Philips equipment -TECNAI TYPE CM-30 operating at an acceleration voltage of 300 kV used with a Field Emission filament. This TEM / HREM has also an EDS (Energy Dispersive Spectrometer) with an ultra-fine window that allows the light detection (light weight) elements. The high resolution images (HREM) obtention, has been achieved using a contrast objective with an aperture of 20 μm, which corresponds point by point to a resolution of about 0.2 nm.

In the figure 1a has shown a dispersion that looks like immiscibility drops with different sizes and contrast of precipitate drops which are between the range of 5-20 nm diameter[5]. Now, when such “nanodrops” are observed in greater magnification, it is prove that they have some orderings, depicting atomic planes. Therefore, “nanodrops” are dispersed in the residual vitreous matrix that correspond to either nuclei or nanocrystal particles (Figure 1b).

The figure 2a shows areas without a visible order, that should correspond to residual glass (amorphous). In the figure 2b it can be observed a pseudo-rectangular crystal with 250 nm side, fringer image and a 8 nm separation between them (8nm would correspond to the projection of a spinel cubic crystal). We can also observe in the lower part of the micrography, contrast lines caused by the stress fields that this type of crystalline phase generates in the residual vitreous phase.

The figure 2c (higher magnification), clearly indicates the interphase between the disordered (vitreous) and the crystalline AB₂O₄ (Mg, Fe) (FeAlCr)₂O₄ spinel area “decorated” in the edge by high contrast

chromium atoms. In the figure 2d depicts loops of contrast emerging from corners due to the high stress fields produced between the spinel crystal and the residual glassy phase.

References

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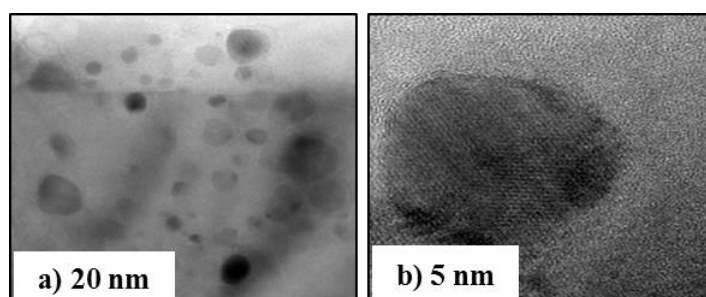


Figure 1. Micrographs obtained by TEM and HREM in a glass – ceramic from Cr⁺⁶ slag waste after thinning by SEM-FIB.

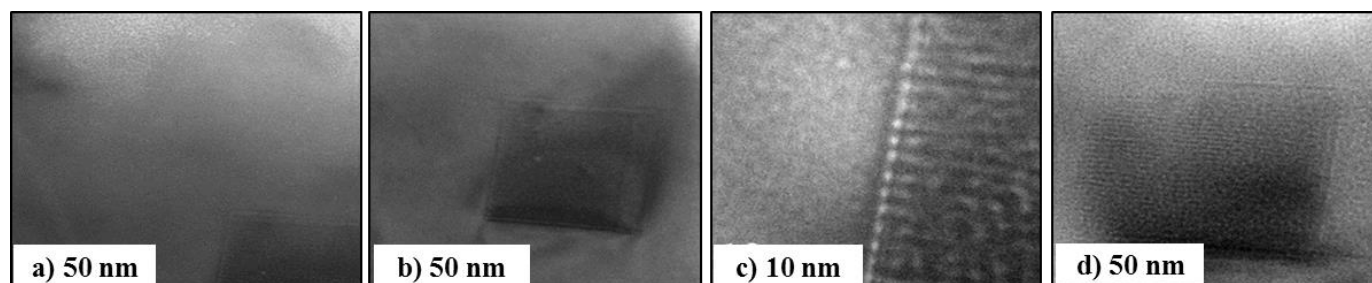


Figure 2. TEM/HREM observations in glass – ceramic obtained from a high chromium contrast metallurgical slag.