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Solid State Transition from γ to α-Al_2O_3 Induced by SPEX Mechanical Milling

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The high energy ball milling is an easy and low cost technique that has been highly used in the processing of powder materials. This technique has demonstrated to be effective in the synthesis of new compounds, formation of solid solutions, production of nanostructured materials, activation of powders and homogenization of phases [1]. In this study we report the use of the SPEX mechanical milling to synthesize α-Al_2O_3 from γ-Al_2O_3. The γ-Al_2O_3 was obtained by the calcination of pseudoboehmite at 500 ºC for 2 h. The pseudoboehmite was synthesized from aluminum sulfate hydrated ($Al_2(SO_4)_3$• xH_2O) and anhydrous ammonia (NH_3). The powders of γ-Al_2O_3 were poured into a steel stainless jar in batches of 10 gr and milled for times of 5 and 10 h. No control agent or any other additional component was added during the milling process. The mechanical treatment was performed at room temperature and under atmospheric conditions. The as milled powders were analyzed by XRD and SEM. Figure 1(a, b and c) shows the effect of the mechanical milling in the particle size and distribution. Figure 1a displays the raw powders of γ-Al_2O_3 that are composed by big particles of irregular shape. The particles after 5 h (1b) adopted a round shape and a light decrease of particle size in comparison to the raw powder. Short times of milling demonstrated to be effective in the reduction and homogenization of the particle size. The Figure 2 shows the XRD analysis of the powders before and after milling treatment. These analyses show that the SPEX mechanical milling supplies enough energy to achieve the beginning of the transformation from γ to α-Al_2O_3. After 10 h of milling, new reflections were detected and assigned to the reflections (-216), (-224), (-213), (-114) and (-112) in good agreement to the XRD pattern COD 1000032. No evidence of contamination generated during the mechanical milling was observed. Moreover, the presence of the intermediate phases δ and θ was not detected. Therefore, it appears that the transformation from γ to α-Al_2O_3 occurred with the absence of these transitional changes as showed previously by A. Tonejc [2]. Recently, other researchers have shown similar results in the milling of commercial powders of γ-Al_2O_3 and boehmite by means of planetary ball milling [3][4]. The results reported in our work show that SPEX mechanical milling is highly effective in the reduction and homogenization of particle size. Moreover, it has been demonstrated that SPEX milling is successful to achieve the transformation from γ-Al_2O_3 to α-Al_2O_3 at room temperature without producing any significant level of contamination.

References:

[1] T. Prasad Yadav *et al*, Nanosci. Nanotechnol. **2** (2012), p. 22.
[2] A. Tonejc *et al*, Mater. Sci. Eng. A **181–182** (1994), p. 1227.
[3] T. Kozawa and M. Naito, Adv. Powder Technol. **27** (2015), p. 935.
[4] S. R. Chauruka *et al*, Chem. Eng. Sci. **134** (2015), p. 774.

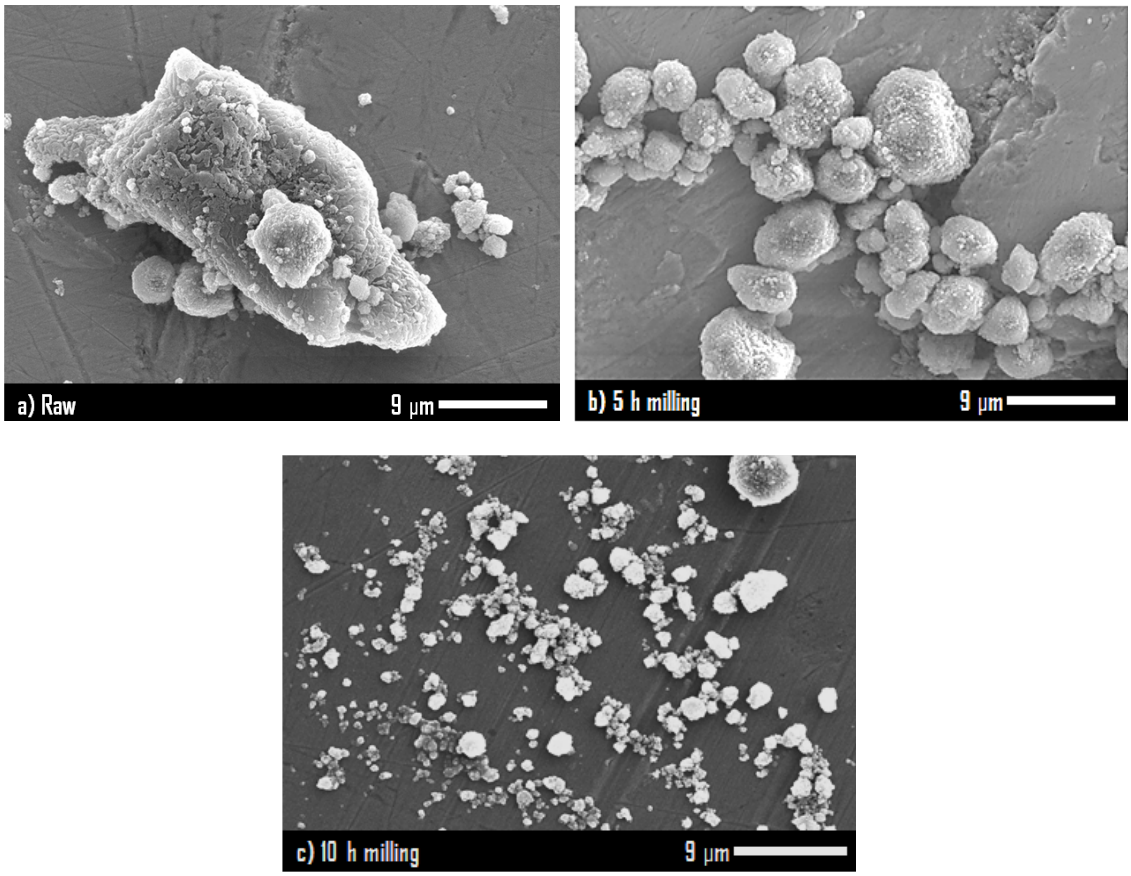

Figure 1. Powders of γ-Al_2O_3 before and after milling treatment.

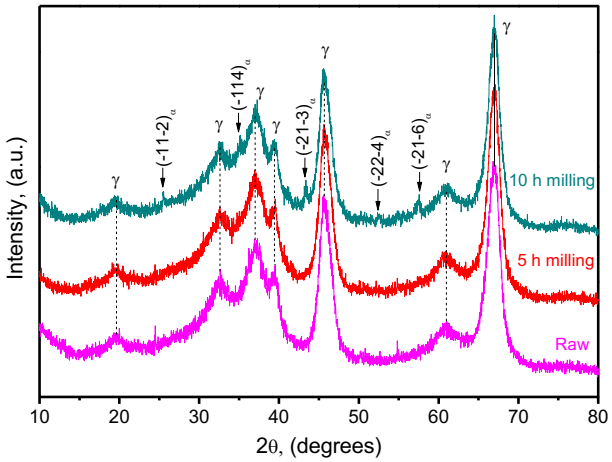

Figure 2. XRD of γ-Al_2O_3 powders before (Raw) and after 5 and 10 h of milling.

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