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doi:10.1017/S143192762200321X

Microsc. Microanal. 28 (Suppl 1), 2022

Microstructure and Oxygen Distribution of Mechanically Milled Al Particles Sintered by the Induction Heating, Spark Plasma and Conventional Methods

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Mechanical milling is an effective technique to obtain nanostructured particles (NP) through severe plastic deformation by the highly-energized milling media. There are many techniques to consolidate the NP powders; however, the conventional methods involve the mandatory use of high temperature with longer processing times, which promotes the grain growth, reducing the mechanical response of sintered samples [1]. Spark plasma sintering (SPS) and induction heating (IH) are used to maintain the highly refined microstructure after sintering achieving a notable increase in the sample mechanical performance. NPs have attracted attention due to their great potential for enhancing the mechanical properties of pure elements [2]. The objective of the present work was the consolidation and sintering of pure aluminum (Al) powder samples milled for 2 hours in a SPEX 8000M high-energy mill, using three different sintering routes: the Conventional Method (CM), IH and SPS. In CM, the prepared Al NP were cold compacted at 900 MPa and subsequently sintered in a furnace at 550ºC for 3 hours under an Argon atmosphere.

Meanwhile, by IH, the NP were compacted at 450 MPa and simultaneously sintered at 450ºC for 3 minutes after sintering the die was cooled down under a forced flow of air and the sample was extracted at room temperature. The SPS process also involves the compaction in a steel die under 450 MPa and simultaneous sintering, using a heating a slope of 58°C/min at 350°C, a heating slope change to 25°C/min until 450°C and it was held for 3 min in the chamber under vacuum. When the sintering process finished, the die was cooled down as IH. For microstructural and oxygen distribution studies, the cold compacted and sintered samples were prepared through the focused ion beam technique to be analyzed using transmission electron microscopy (TEM) and energy-dispersive X-ray spectroscopy (EDS).

After milling, a laminar microstructure is formed by the impact forces of milling media is presented in the compacted sample. After CM, this feature was almost eliminated due to high temperature and prolonged sintering time; bigger grains growth as expenses of the small ones by diffusion. The refined microstructure is still noticeable in the SPS sample; however, the IH sample presents a close microstructure similar to cold compacted, maintaining the unique nanostructure with small and elongated grains (Fig. 1). In EDS elemental mappings for Al and O, a small signal for O is observed in the cold compacted sample; this is attributed to the presence of this element physically adsorbed in the surface. Also, the O concentration in the IH sample is lower than CM and SPS specimens; here, the high O concentration is because of the high oxidation rate due to the exposition time at high temperature compared to the IH sample (Fig. 2). Based on the above results IH method was confirmed as an efficient technique to sinter NPs, keeping their refined microstructure avoiding excessive sample oxidation.

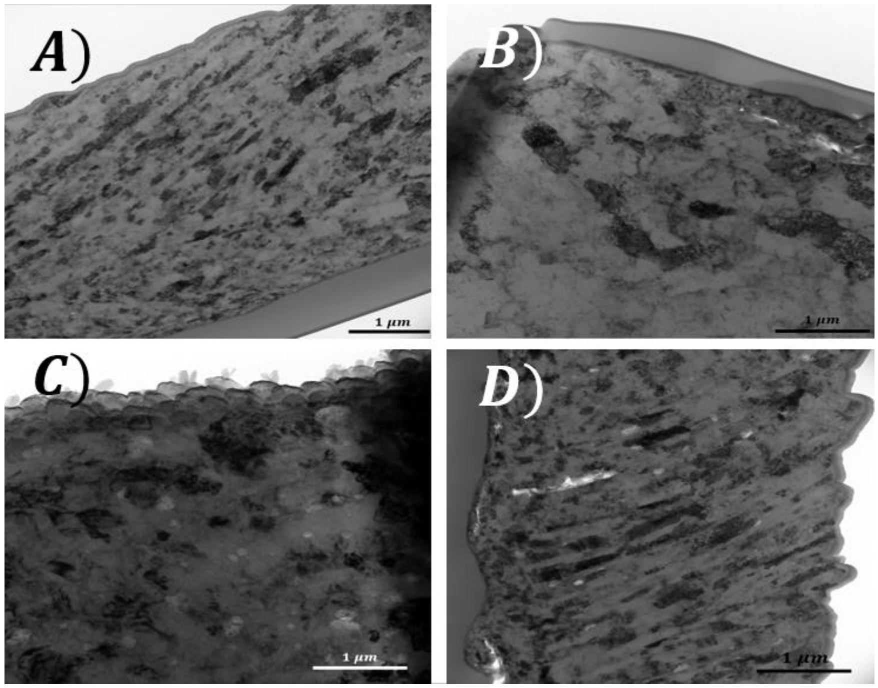

Figure 1. TEM micrographs of cold compacted (A) and CM (B), SPS (C) and IH(D) sintered samples.

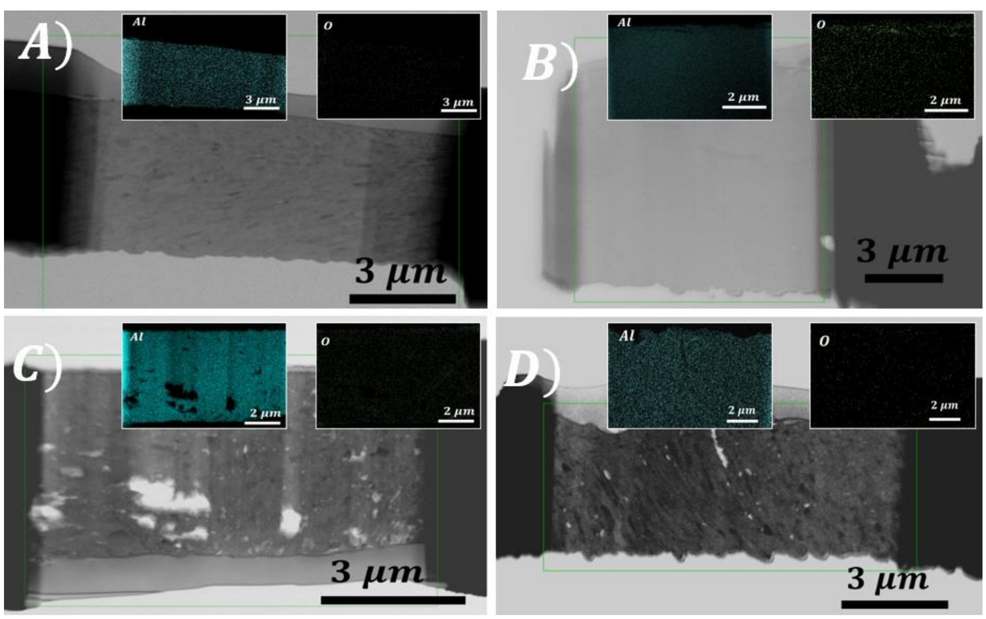

Figure 2. TEM micrographs and elemental mappings (Al and O) of cold compacted (A) and CM (B), SPS (C) and IH (D) sintered samples.

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https://doi.org/10.1017/S143192762200321X Published online by Cambridge University Press

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https://doi.org/10.1017/S143192762200321X Published online by Cambridge University Press