doi:10.1017/S1431927614011416
Microsc. Microanal. 20 (Suppl 3), 2014

Nanodiffraction Characterization of Grain Boundary Structures in Nanocrystalline $MgAl_2O_4$ prepared by Electric Field Assisted Sintering

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Appling a contact or non-contact electric field during sintering, i.e., electric field assisted sintering (EFAS), can improve consolidation behavior and is commonly used in many sintering techniques, e.g., Spark Plasma Sintering (SPS)[1], Flash Sintering[2], and Pulsed Electric Current Activated Sintering (PECAS)[3][4]. Electric fields have been shown to densify ceramic powders within seconds as well as, depending on the experimental conditions, retarding or enhancing grain growth, however, the mechanisms which affect sample densification and grain growth remain unclear [1] [2][4]. To control and tailor microstructural aspects of sintered materials, better correlation is required of the applied non-contact electrical fields and current to the resultant microstructure. Due to the complexities of the sintering processes, we have chosen to isolate our investigation to the influence of non-contact electric fields on microstructural evolution.

Ultrafine magnesium aluminate, $MgAl_2O_4$, was synthesized and isothermally sintered at 1300°C in the presence and absence of an electrostatic field strength of 100kV/m [5]. The aim of the study is to probe the role of non-contact electric fields, i.e., in the absence of any current, on the grain growth and densification of nanometric $MgAl_2O_4$ ceramic powders. Density measurements of sintered material suggested the applied electric fields had no appreciable effect on final density. Imaging using an FEI 430 NanoSEM of polished pellet surfaces revealed a difference in average grain size and grain size distribution between the two types of samples (see Figure 1) in which coarser microstructure were obtained under an applied electric field, suggesting grain boundaries distributions may also be affected by the applied electric field.

Samples were thinned to electron transparency using the conventional method of mechanical dimpling and ion beam thinning using Ar ions in a Gatan PIPS at 4kV until perforation and finished at 500eV for surface cleaning. Nanodiffraction experiments, with the ASTAR system from NanoMegas, were conducted on a Phillips CM300 TEM operated at 300kV to study microstructural grain orientation and grain boundary character distrubitons. The benefits of the ASTAR technique is that resolution, in principle, is only limited by the size of the beam probe, which for these experiments was 2.2nm. Although, grain overlap and inclined boundaries can pose difficulties in orientation determination and spatial resolution in vicinity of a grain boundary. Additionally, for samples that do not provide strong kikuchi patterns (i.e. light elements and/or small grains) electron diffraction becomes a more reliable alternative to SEM based electron backscattered and transmission diffraction techniques. To improve indexing of the acquired patterns, the beam was precessed about the optical axis 0.5° to reduce dynamical diffraction effects. Figure 2a and 2b show representative STEM annular dark field (ADF) images and inverse pole figures (IPFs), respectively, obtained by this technique. Preliminary data suggests applied electric fields do not induce preferential texturing during annealing of the microstructure, however, a higher grain boundary length fraction of coincidence site lattice (CSL) type boundaries was observed for samples sintered in the presence of an electrical field [7].

It has been established that CSL boundaries trend toward corresponding to energetic minima and in some cases increased mobility which would assist coarsening [6].The type of fractures seen in pellet fracture surfaces qualitatively correlate with the grain boundary character distributions from ASTAR. Atomic resolution characterization by HAADF-STEM imaging and STEM-EELS of specific grain boundary structures is underway. Preliminary results will be discussed during the presentation. [8].

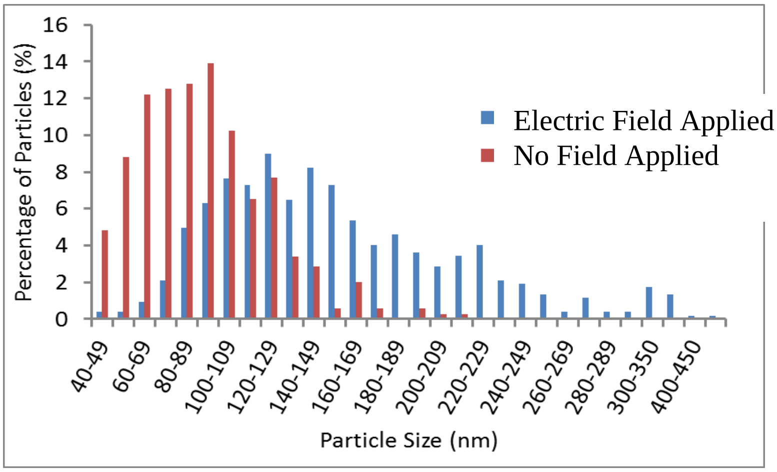

Figure 1: Grain size distribution of $MgAl_2O_4$ nanopowder sintered with (blue) and without (red) an electric field showing the larger average grain size coupled with a broader grain size distribution for the field sintered sample *([7] Manuscript in preparation to be submitted).*

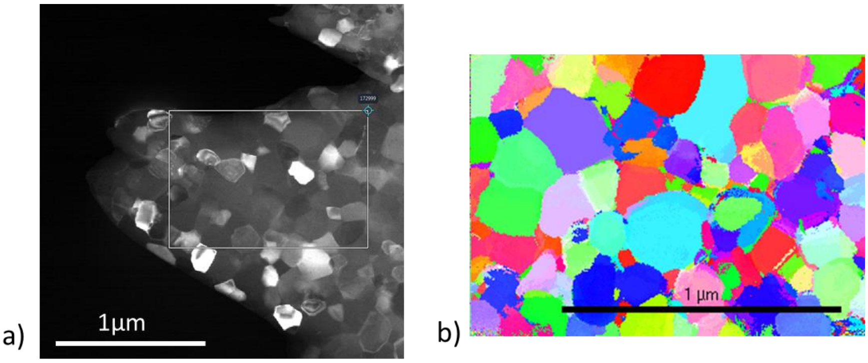

Figure 2: (a) STEM ADF image of sintered $MgAl_2O_4$ acquired during ASTAR operation showing the sample's polycrystalline nature and (b) a representative inverse pole figure generated from ASTAR data from which grain boundary character and distributions can be determined *([7] Manuscript in preparation to be submitted).*

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[8] Funding is acknowledged from UC Davis start-up funds, UC Laboratory Fee grant #12-LR-238313, US Department of Energy, Office of Basic Energy Sciences, Los Alamos National Laboratory Material Design Institute, and Office of Naval Research #N00014-11-10788