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SEM and TEM Analysis of Cryomilled Nanocrystalline Al Powder

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Cryomilling involves the ball milling of metal powders in a liquid nitrogen medium. It has been used to produce bulk nanocrystalline materials with high thermal stability [1]. The benefits of milling at cryogenic temperatures include accelerated grain refinement, reduced oxygen contamination from the atmosphere, and minimized heat generated during milling. This mechanical attrition process induces severe repetitive deformation in powders. During milling, the powder particles are repeatedly sheared, fractured and cold-welded, and severe plastic deformation effects the formation of nanostructures [2]. Cryomilled powders exhibit typical grain sizes of 20–60 nm [3].

Gas-atomized Al 5083 (Al–4.4Mg–0.7Mn–0.15Cr wt.%) powder (Valimet Inc.) was cryomilled in a 20 kg batch (by DWA Aluminum Composites) for 8 hours. The cryomilling was performed using a non-cleaned attritor that had been previously used to mill Al–7.5%Mg alloy. Consequently, 2.6 kg commercial pure aluminum powder was blended in with Al 5083 powder to compensate for the increased in Mg content from the residual Al–7.5%Mg on the attritor, impeller, and milling balls. Before cryomilling, ~0.2 wt. % of stearic acid ($C_{18}H_{36}O_2$) was added in the milling chamber as a process control agent (PCA). PCA can prevent excessive agglomeration and mediate the cold welding of powders. Stainless steel balls were used with a ball-to-powder weight ratio of 32:1.

Cryomilled powder was mixed with epoxy (G-1, Gatan Inc.), pressed, and cured to produce a pellet. The pellet was cross-sectioned using a JEOL cross-section polisher (CP) [4]. The section was examined in an SEM (JEOL 7000F FEG) to determine grain structure and morphology and to perform EDS and EBSD analysis. The CP sample preparation procedure utilizes a low voltage Ar ion beam (5KV) to polish and remove material (Fig. 1), eliminating the need for mechanical polishing and/or embedding. The ion-polished surface enables unambiguous SEM observation of microstructure, defects, and voids which are not obscured by artifacts typically present in metallographically polished specimens. The sample preparation procedure also introduces minimal strain to the specimen, enhancing channeling contrast and high-quality EBSD analysis.

The CP sample clearly reveals the grain structure in cryomilled powder particles. Both nanocrystalline and coarse-grain regions are evident, along with voids and microvoids (normally obscured in conventionally polished sections) as shown in Fig. 2. The exceptionally strong channeling contrast in back-scattered images is attributed to the relatively strain-free surfaces produced by CP polishing. This feature also enhanced EBSD analysis of crystal structure, texture, and grain orientation. Grain size distribution and microvoid content were correlated with powder particle size/shape. EDS was used to identify intermetallic phases derived from dispersoids and constituent particles. The evolution of oxides, intermetallic phases, microvoids, and grain structure was studied as a function of cryomilling time.

References

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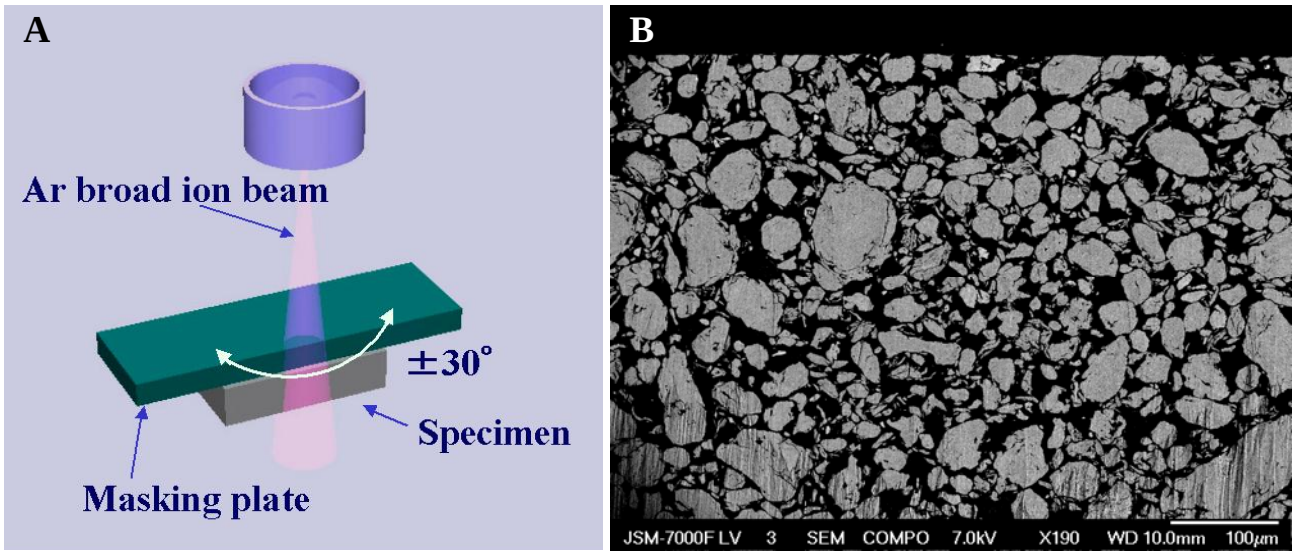

Fig. 1. (A) Schematic of general operation of a CP. (B) Backscattered electron image (190x) of CP prepared powders.

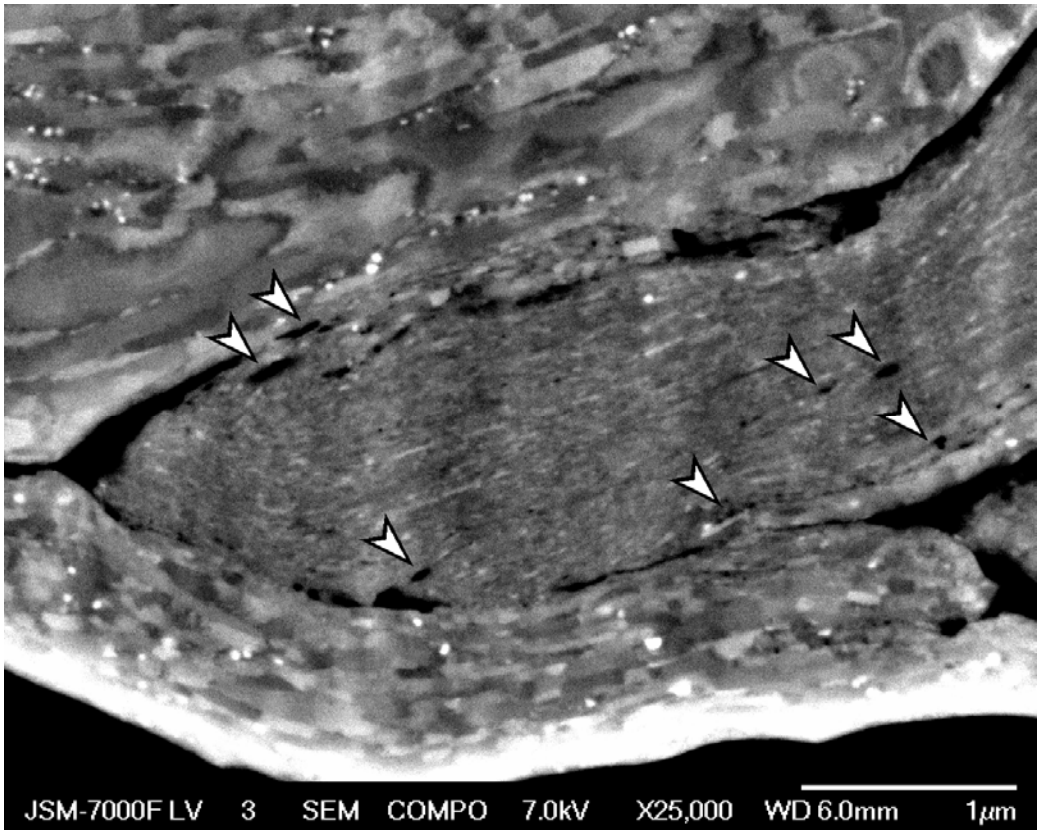

Fig. 2. Backscattered electron image (25,000x) of cryomilled Al powder cross-sectioned by the CP showing both nanocrystalline and coarse-grain structures with arrows indicating submicron voids.