Abstract. Severe plastic deformation (SPD) processes were used in mixing and consolidating aluminum based nanocomposites containing various amounts of C nanoparticles. A planetary ball milling machine was used to mix the powders, and back pressure equal channel angular pressing (BP-ECAP) for consolidation. For comparison, Al-Al$_2$O$_3$ nanocomposites were processed by the same processing method. TEM observations showed that the combination of the two SPD processes was effective in dispersing the C nanoparticles in the Al matrix, and better dispersion was achieved by increasing the number of ECAP passes. Especially in Al-5 wt% C after ECAP consolidation for 24 passes, the compressive plastic strain was significantly improved. The Al-Al$_2$O$_3$ materials appeared to have better particle distribution and ductility compared to the Al-C materials although their strengths were lower.

1. Introduction
Aluminum alloys and composites have attracted considerable attention in structural and functional applications due to their light weight and high specific strength and stiffness. However, precipitation hardened aluminum alloys have not attained the same level of high strength as that achieved in Fe-Fe$_3$C materials, and they are thermally unstable. Although aluminum matrix nanocomposites have great potential, the challenge remains how to uniformly distribute nanometer sized ceramic particles in the Al matrix, since these nanoparticles have high tendency to agglomerate due to their large surface to volume ratio. Thus, it is critical to solve this problem in order to achieve the desired mechanical properties.

Equal channel angular pressing (ECAP) is a unique SPD process which has found extensive uses in producing ultrafine grained (UFG) metallic materials [1]. The use of back pressure equal channel angular pressing (BP-ECAP) is effective in consolidating particles into bulk material due to the high amount of shear involved. In the present investigation, BP-ECAP is used to produce fully dense Al based nanocomposites reinforced with inexpensive ceramic particles (e.g. carbon black). Good dispersion and excellent mechanical properties were obtained in these nanocomposites by combining mechanical milling for dispersion of nanoparticles with ECAP for consolidation.

2. Experimental Materials and Procedures
A detailed description of experimental materials and methods can be found in [2]. The carbon black used in this work consisted of large clusters of an average size of ~500 nm although individual particles had an average size of ~30 nm with a density of 1.7-1.9 g/cm$^3$. The Al$_2$O$_3$ particles had an average size of < 50 nm with a density of 4 g/cm$^3$. Different amounts of ceramic particles (2, 5 and 10 wt%) were added to a pure Al powder and the
mixtures were processed for 30 hours each using a planetary ball milling machine. BP-ECAP was used to consolidate the mixed powder at 400°C for 8 and 24 passes, respectively, following the C route with the application of a back pressure of 50 MPa. The set up of BP-ECAP is similar to that described in [3].

Density measurements were conducted based on the Archimedes principle. The Vickers microhardness was measured by using a 50 g load for 15 seconds, and an average of 6 readings was taken from each material. Compressive specimens of 4 × 4 × 8 mm were taken along the longitudinal direction of the sample after ECAP and tested at room temperature. An FET Tecnai F20 was used for TEM observations at 200 kV.

3. Results and Discussion

3.1. Density and Hardness

The density and microhardness values are shown in Table 1. It is apparent that the materials produced were fully dense (the density value of 1.87 g/cm³ was used for C [4] in the calculation of the theoretical densities). There was a significant increase in hardness with an addition of 2 wt% C, although only a moderate further increase was observed in the Al-5 wt% C material. The hardness values of the Al-2 and 5 wt% Al₂O₃ materials were lower than the corresponding Al-C materials, although the effective difference was smaller since the Al-Al₂O₃ composites were lower in volume fractions.

These results showed that the BP-ECAP consolidation is capable of producing bulk metal matrix nanocomposites with full density at temperatures lower than those used in the conventional consolidation processes like HIP and sintering [5-9]. This is desirable since high temperature could cause excessive grain growth and damaging reactions between matrix and reinforcement, resulting in poorer mechanical properties. On the other hand, SPD processes such as ECAP can provide the amount of strain necessary to deform the particles, leading to the filling of pores and strong interfacial bonding without the need of high thermal energy. In addition, fully dense materials with homogeneous particle distribution were produced in this work without resorting to secondary processing such as rolling and extrusion, which are often required following HIP and sintering.

| Material                          | Measured Density (g/cm³) | Theoretical Density (g/cm³) | HV (kg/mm²) |
|----------------------------------|--------------------------|-----------------------------|-------------|
| Al (unmilled) (ECAP for 8 passes)| 2.7                      | 2.7                         | 33.9        |
| Al (milled) (ECAP for 8 passes)  | 2.7                      | 2.7                         | 37.1        |
| Al-2 wt% C (ECAP for 8 passes)   | 2.68                     | 2.68                        | 83.1        |
| Al-5 wt% C (ECAP for 8 passes)   | 2.65                     | 2.64                        | 96.5        |
| Al-5 wt% C (ECAP for 24 passes)  | 2.65                     | 2.64                        | 90.5        |
| Al-2 wt% Al₂O₃ (ECAP for 8 passes)| 2.72                   | 2.72                        | 61.9        |
| Al-5 wt% Al₂O₃ (ECAP for 8 passes)| 2.74                   | 2.74                        | 81          |
| Al-10 wt% Al₂O₃ (ECAP for 24 passes)| 2.79                 | 2.79                        | 83.5        |
3.2. Microstructure and Mechanical Properties

No pores in the nanocomposites produced were found in TEM observations, and this confirms the achievement of fully dense materials. The C clusters in the as-received powder had been dispersed in the Al matrix as individual nanoparticles (< ~100 nm in size), as shown in Figure 1 for the Al-5 wt% C material. The microstructure of pure Al without the addition of reinforcement was totally different, as shown in Figure 2a, having elongated grains with many subgrain boundaries; in contrast, an equiaxed grain structure was observed in Al-2 wt% C as shown in Figure 2b. The nanosized particles in the composites had prevented grain growth and refined the structure during the ECAP process. With increasing number of passes from 8 to 24, as shown in Figure 1 for Al-5 wt% C, more grain refinement was achieved (grain size reduced from ~1 μm after 8 passes to ~300 nm after 24 passes). This phenomenon was also observed by others [10]. On the other hand, the spherical Al₂O₃ nanoparticles (< 50 nm in size) were observed in the Al-Al₂O₃ materials to be homogeneously distributed, as shown in Figure 3.

It is therefore apparent that the combination of mechanical milling and ECAP was effective in dispersing the initially clustered C nanoparticles. A uniform distribution of individual C particles (~50 nm in size) was achieved, and it is believed that most of the dispersion occurred during the mechanical milling process, as also observed by others [11-13]. Following mechanical milling, ECAP ensured that the mixed powder was completely consolidated with full density which was not achieved by conventional compacting and sintering [11, 12]. In addition, continued ECAP led to further structural refinement and more homogeneous dispersion of ceramic nanoparticles in the Al matrix, and this would benefit the mechanical properties of the nanocomposites (see the next paragraph).

![Figure 1. TEM microstructures in Al-5 wt% C after BP-ECAP for a) 8 and b) 24 passes at 400°C.](image-url)
Figure 2. TEM microstructures of (a) pure Al with an elongated grain structure with many subgrain boundaries, and (b) Al-2 wt% C with an equiaxed grain structure, after BP-ECAP for 8 passes at 400°C.

Figure 3. TEM microstructure of the Al-10 wt% Al₂O₃ nanocomposite material, showing the homogeneous dispersion of Al₂O₃ nanoparticles (<50 nm in sizes) in the Al matrix.

The compressive strength and strain of the nanocomposite materials produced showed significant enhancement. The Al-2 wt% C material consolidated for 8 passes showed a 0.2% proof stress (σ₀.₂) of 225 MPa and a maximum strength (σₘₐₓ) of 302 MPa, significantly higher than those of the pure Al after the same processing (σ₀.₂ = 58 MPa and σₘₐₓ = 157 MPa). By the addition of 5 wt% C, these strengths were further increased to 260 and 352 MPa, respectively. Considerable compressive strain (tests stopped at ~35% without failure) was achieved in Al-2 wt% C after consolidation for 8 passes. However, the Al-5 wt% material consolidated for 8 passes fractured at ~10% true strain. The ductility was improved to 20% by increasing the number of ECAP passes to 24, while the compressive strength was almost unchanged. The ductility of the Al-Al₂O₃ materials appeared to be much better, showing true strains of 40-80% without fracture. However, the proof stress in Al-Al₂O₃ materials was lower. For example, σ₀.₂ in Al-5 wt% Al₂O₃ processed for 8 passes was ~153 MPa, compared to 225 MPa in Al-2 wt% C at the same conditions (the two having an equivalent volume fraction of ~3 vol%).

The high strength achieved is thought to be resulting from combined contributions from dispersion hardening and grain refinement. The Orowan mechanism becomes more effective with decreasing
particle size and increasing homogeneity in particle distribution. On the other hand, the fine and homogeneous dispersion of C nanoparticles had pinned grain boundaries, limiting recrystallization and grain growth and leading to a much refined, equiaxed grain structure, in contrast to the coarse and elongated grain structure in pure Al owing to the dynamic recovery process which occurs at ~400°C [14].

4. Conclusion

Fully dense monolithic Al as well as Al-C and Al-Al₂O₃ nanocomposites were successfully produced by combining two severe plastic deformation processes: mechanical milling to mix the particles and back pressure equal channel angular pressing (BP-ECAP) to consolidate the mixed powder. The BP-ECAP consolidation was carried out at 400°C for up to 24 passes. The clusters (~500 nm in size) in the as-received carbon black powder were broken and dispersed homogenously in the Al matrix following the mechanical milling and ECAP processes, with individual C particles of <100 nm in size.

The nanocomposites displayed significantly higher hardness which increased with increasing reinforcement content. Significantly higher compressive strengths were achieved in the nanocomposites although the Al-C materials appeared to exhibit greater strength and lower ductility than the Al-Al₂O₃ materials. The enhanced mechanical properties were attributed to the fine and homogeneous distribution of the ceramic nanoparticles which provide dispersion strengthening and cause the formation of a finer grain structure. The combination of mechanical milling and ECAP offers a promising method for producing strong metal matrix nanocomposites.

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