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Investigation of Influences of Pressing Pressure and Sintering Temperature on the Mechanical Properties of Al-Al_4C_3 Composite Materials

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Abstract

In this study, Al powders containing 3% wt carbon black were mechanically alloyed in a high energy ball mill for 20 h. Mechanically alloyed powders were compacted at various pressures (400, 500, 600, and 700 MPa) to produce specimens with dimensions of 6.35 x 12.70 x 31.70 mm. Specimens were then sintered at 500, 600, 650, and 700°C for 5 h under argon atmosphere in a tube furnace. XRD analysis was carried out to see the Al_4C_3 transformation. SEM analysis was conducted to determine the sintering behavior of the powders. Transverse repute strength and hardness tests were carried out to fined the mechanical properties of specimens. The results showed that ideal compacting pressure was 600 MPa and the ideal sintering temperature was 650°C for good mechanical properties.

Key words: Mechanical alloying, Aluminum Carbide, Mechanical properties

Introduction

There are vast reserves of Al ores in the world, and it is the second most commonly used metal after iron. On account of its easy formability and light weight, it finds many applications in industry (Arık *et. al.*, 2000). Strengthening is generally done by dispersion of high temperature resistant fine particles of oxides such as Al_2O_3, ThO_2 or Y_2O_3 and carbides such as TiC or SiC, in the Al matrix (İbrahim *et al.*, 1991; Lu *et al.*, 1999; Bronsveld *et al.*, 1991). These fine particles are added to the matrix either in solid or in the liquid state. In the liquid methods, particles are added to liquid metal by stirring before casting, but the resulting distribution is sometimes inhomogeneous. In the solid state process, known as the powder metallurgy technique, oxide or carbide powders are added to metal powders, and then compacted and sintered to produce block materials (German, 1984). Another way of producing the fine and homogenous distribution of these hard particles is their production insitu, generally by reaction milling and annealing processes (Jangg *et. al.*, 1989). Mechanical alloying (MA), first used in the 1960s, is a simple and useful technique to synthesize both equilibrium phases of commercial materials starting from elemental powders (Benjamin and Volin, 1974). This method was developed during the late 1960s to produce high temperature materials (Singer *et al.* 1980, Suryanarayana, 1995). MA produces a homogeneous distribution of inert fine particles within the matrix and avoids many problems associated with melting and solidification (Fischer and Weber, 1990). In the application of MA, two or more elemental powders are mixed with each other and the blend is processed in a high energy ball mill. A synthesis between the processed powders occurs either during mechanical alloying or the sintering process. The resulting ox-

ide, carbide or nitride particles can occur in a lower temperature or time than those forming in their normal condition (Tweed, 1991). The aim of this study is to produce Al_4C_3 reinforced aluminum composite powders by reaction milling through MA of a 3% carbon black and aluminum powder mixture and to investigate the mechanical properties of that composite.

Experimental Procedure

Materials

Gas atomized Al powders were produced by Gazi University PM Lab. Powders with a maximum particle size of 150 μm (-100 mesh) were produced from 99% pure Al ingots supplied by ETİBANK (Turkish Aluminum Producing Co.). Carbon black of 99% purity and mean powder size (agglomerate size) of 2.4 μm was obtained from YARPET (Turkish Petrochemical Trade Co.). The properties of carbon black are given in Table 1.

Mechanical alloying

Al powders weighing 48.5 g, 1.5 g of carbon black and 300 g of steel balls with a diameter of 10 mm were placed into the 750 cm^3capacity tank of a high energy attritor. Afterwards, 3% stearic acid was added to the mixture to prevent the Al powders sticking to the balls and to the walls of the milling tank. In order to eliminate the oxidation of Al during MA, the process was conducted under argon atmosphere. Ar gas was purified from residual oxygen by passing it through Cu chips heated to 650°C. The tank of the attritor was water-cooled during MA, and the mixtures were milled for 20 h (Figure 1). The choice of MA condition, are given in Table 2.

Powder compaction and sintering

Mechanically alloyed powders were compacted at various pressures (400, 500, 600, and 700 MPa) to produce specimens of 6.35 x 12.7 x 31.7 mm in size according to MPIF 44 (Metal Powder Industries Federation Standard). After compacting, the green den-

Table 1. Properties of carbon black

Reflection (%, with toluene)	325 Mesh - sieve oversize (%)	Moisture (%)	Density (g/l)	Sulfur (%)
Min 80	Min -	Min -	Min 320	Min -
Max -	Max 0.1	Max 25	Max 380	Max -

Table 2. MA condition of powder mixture

Vessel volume (cm^3)	750	Rotor speed (rev min^{-1})	450
Mass of aluminum powder (g)	48.5	MA atmosphere	Argon
Mass of carbon black (g)	1.5	Cooling	Water
Charge ratio (mass of grinding balls : mass of powder in mill	6:1	Milling time (h)	20
Steel ball diameter (mm)	10	Stearic acid (%)	3

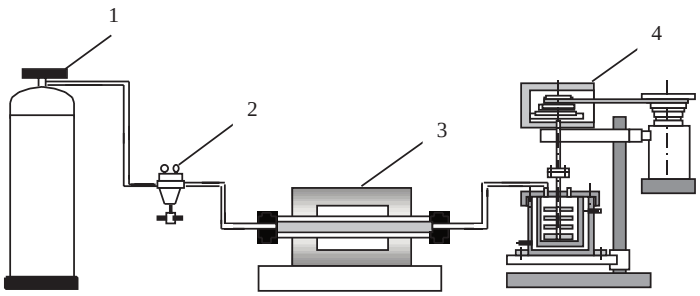


Figure 1. Mechanical alloying set-up: 1) Argon cylinder, 2) Manometer, 3) Tube furnace, 4) Attritor.

sity of all blocks was calculated depending on compaction pressures. Four specimens compacted at various pressures were placed in a tube furnace on graphite boats, and each series of blocks was sintered at 500, 600, 650, and 700°C for 5 h.

Mechanical tests

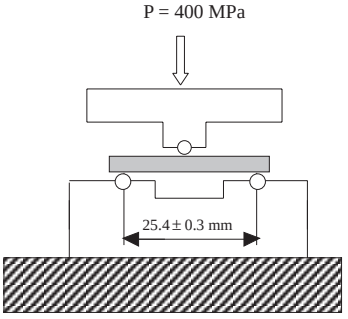
The surfaces of all sintered samples were lightly ground and polished to remove any irregularities or debris. The samples were then subjected to transverse rupture tests in a special device, designed and manufactured according to MPIF Standard 41(Determination of Transverse Rupture Strength (TRS) of Powder Metallurgy Materials) at Gazi University P/M Lab. (Figure 2). The hardness of all samples was measured on the Rockwell B scale. A 10 kg load with 1 mm ball diameter was applied and a mean of at least five readings was taken to represent the sample hardness.

Results and Discussion

Particle size distribution of the mechanically alloyed powders was determined by a Malver Master Sizer E Ver. 1.2b laser scattering device. The mean particle size of original and mechanically alloyed powders was about 150 mμ and 31.96 mμ respectively. A similar study had earlier been carried out by Rodriguez *et al.* (1997), who claimed that increasing the MA duration resulted in the formation of coarser particle size. This was attributed to the cold welding of the powder trapped between the colliding steel balls. However, in this study a decrease in powder size was observed with increased milling time. This was due to the adding of stearic acid and carbon black, which hindered the welding of the particles to each other and resulted in their plastic deformation. As a consequence of this, in the early stages of milling, powder formed in a flake shape and then fracturing and fragmentation started, resulting in the formation of fine particles. The mechanically alloyed powder was characterized by means of X-ray diffraction with Cu K_α radiation at 30 kV and 40 mA. XRD results of MA processed powders showed the characteristic peak of pure Al. This indicated that there was no chemical reaction between Al and carbon black during the MA process (Figure 3).

This is the expected result according to the literature (Wu *et al.*, 1997). It was found that the interaction between Al and carbon black was dependent on the amount of carbon black and Al_4C_3 formation occurring after 40 h MA. A broadening of the XRD peaks in mechanically alloyed powders was observed attributed to high deformation of the powders that resulted in the formation of an amorphous structure (Figure 3). MA alloyed powders were compacted at different pressures, and their green densities increased with increasing compaction pressure (Figure 4). However, this increase slowed down after 600 MPa pressure and stabilized after pressure of 700 MPa. This is probably due to work hardening of Al particle during the pressing process (John *et al.*, 1986).

The highest sintering density was found in the sample sintered at 600°C. Increasing the temperature resulted in a decrease in the density of the block (Figure 5). SEM study of the fractured surface of the samples sintered at 650°C and 700°C showed some delamination cracks (Figure 9). The formation of these delamination cracks may be due to residual carbon, stearic acid and irregular sintering behavior of the sample. This crack decreased the densities of



$$T.R.S = \frac{3.P.L}{2.t^2w} \text{ (MPa)}$$

P : Force required to rupture lbf (MPa)
L : Length of specimen span of fixture (25.4 ± 0.3 mm)
W : Width of specimen (12.7 mm)
t : Thickness of specimen (6.35 mm)
d : Supporting bars diameter (3.1 mm)

Figure 2. Transverse rupture test apparatus.

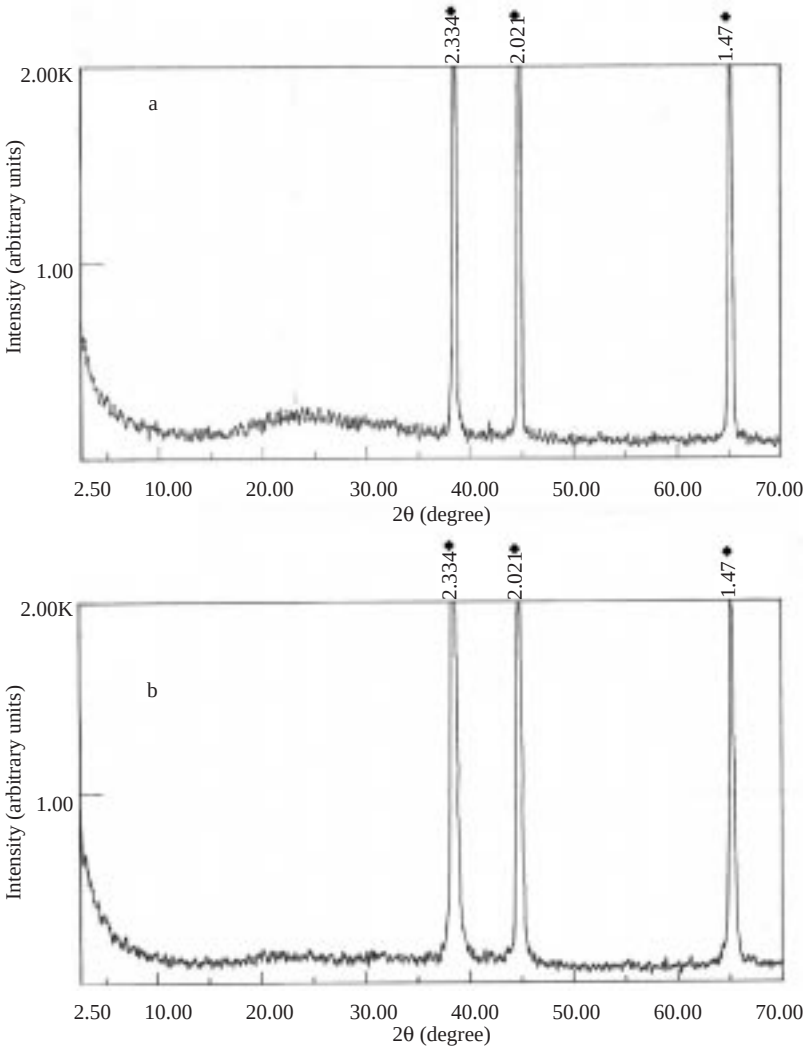


Figure 3. XRD result of the sample a) Before MA, b) After MA for 20 h.

the samples. The important decrease in the density of the samples sintered at 700°C is probably due to the local melting of samples during sintering. The local melting and formation of glassy phase were clearly seen on the surface of the block samples after sintering and also on the fracture surface of the original SEM pictures (Figure 10). Sintering temperature of the Al-Al_4C_3 composite sample was generally chosen below 650°C in similar studies (Besterci *et al.*, 1999; Besterci *et al.*, 2002; Jangg *et al.,* 1989).

A good relationship between transverse rupture strength and sintering temperature was observed. This indicates better sintering behavior of particles at high temperature. However, the rate of increase in transverse rupture strength with temperature slows down at higher temperatures (Figure 6).

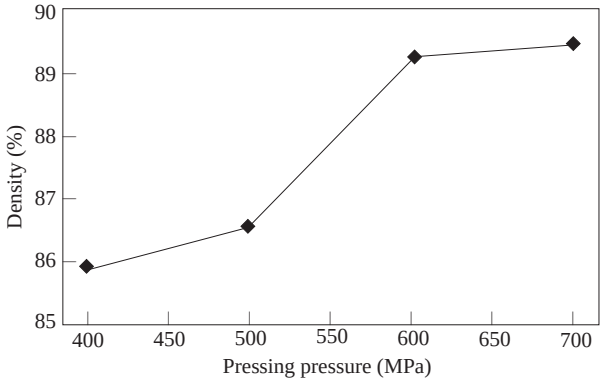


Figure 4. Relationship between compaction pressure and green density of samples.

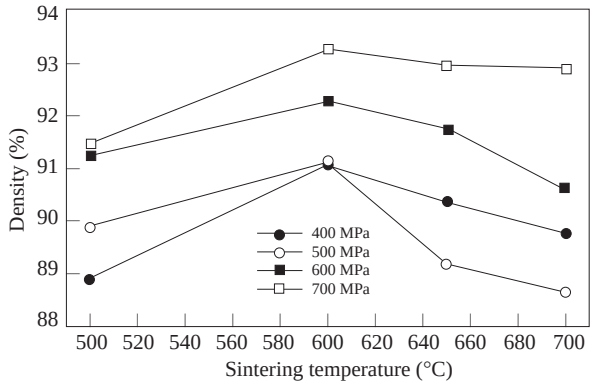


Figure 5. Relationship between sintering temperature and density of samples.

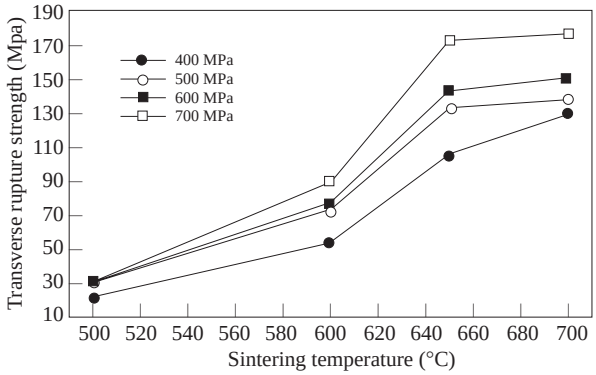


Figure 6. Relationship between sintering temperature and transverse rupture strength.

Among the samples sintered, the highest hardness value was obtained in the sample compacted at 600 MPa and sintered at 650°C (Figure 7). After this temperature a decrease in hardness was observed. This might be due to higher sintering temperature that resulted in the formation of local melted areas of blocks (Figure 10).

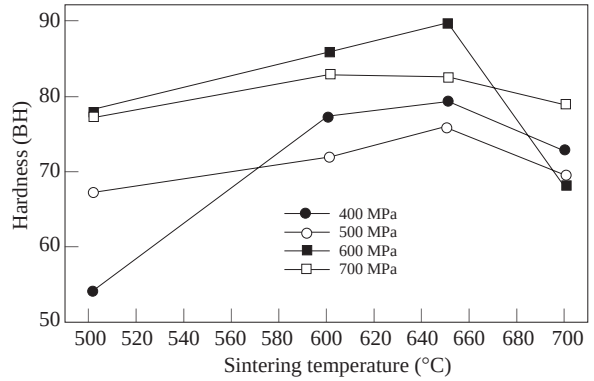


Figure 7. Effect of sintering temperature on hardness.

XRD analysis of the sintered blanks revealed that at least 600°C sintering temperature is necessary for the formation of Al_4C_3 particles. The most Al_4C_3formation was observed in the samples sintered at 650 and 700°C (Figure 8).

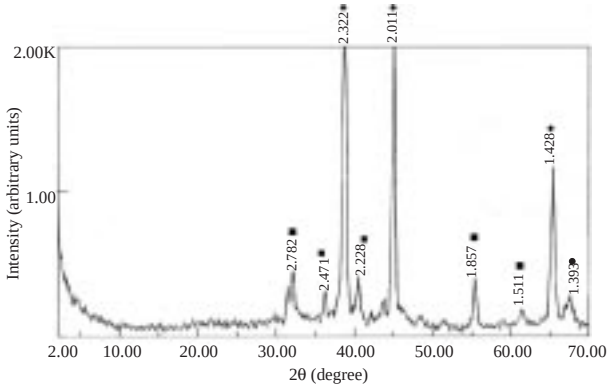


Figure 8. XRD result of the samples compacted at 600 MPa and sintered at 650°C. ♦ Al, ■ Al_4C_3, •Al_2O_3.

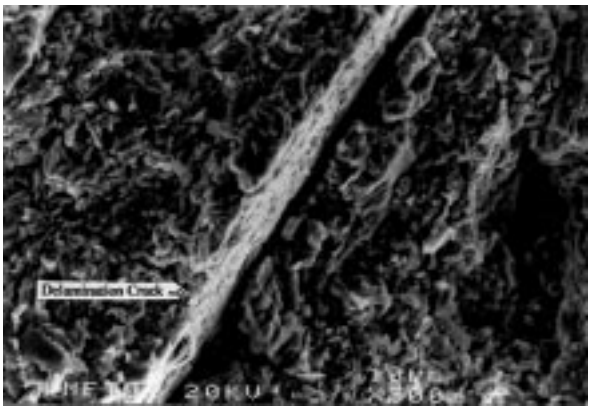


Figure 9. SEM micrograph of sample compacted at 600 MPa and sintered at 650°C for 5 h.

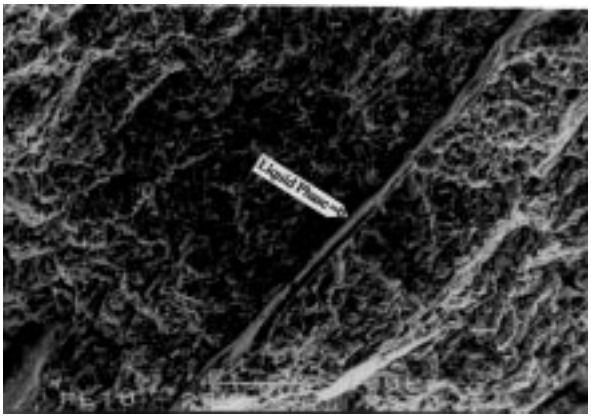


Figure 10. SEM micrograph of sample compacted at 600 MPa and sintered at 700°C for 5 h.

Transverse rupture strength and hardness values of about 7% Al_4C_3 containing Al based composite showed the ideal compacting pressure and sintering temperature of the blocks to be 600 MPa and 650°C, respectively.

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References

Arık, H., Türker, M., and Sarıtaş, S., "Investigation of the Mechanical Properties of Al_4C_3 Reinforced Aluminum Based Composites by Mechanical Alloying Technique", Proceedings of Powder Metallurgy World Congress, Page 1713-1716, 15 October, Japan, 2000.

Benjamin, J.S., and Volin, T.E., "The Mechanism of Mechanical Alloying", Metallurgical Transactions, 5, 1929-1933, 1974.

Besterci, M., Kohutek, I., and Sülleiova, K., "Analysis of Spatial Arrangement of Particles in Thin Foil of Al-Al_4C_3 Material", Journal of Materials Science, 34, 1055-1060, 1999.

Besterci, M., Velgosova, O., and Kovac, L., "Superplastic Deformation of Al-Al_4C_3 Composites Prepared by Powder Metallurgy", Materials Letters, 54, 124-130, 2002.

Bronsveld, P.M., and Bruinsma, P., "Microstructural Analysis of Hot Isostatically Pressed Al-SiC", J. Th. De. Hosson: Materials Science and Engineering, A135, 77-81, 1991.

Davidson, J.E., "Compressibility of Metal Powders", Metals Handbook, 7, 286-287, 1986.

Fischer, J.J., and Weber, J.H., "Mechanical Alloying", Advanced Materials & Processes, 10, 43-50, 1990.

German, R.M., Powder Metallurgy Science, Metal Powder Industries Federation, 1984.

İbrahim, I.A., Mohamed, F.A., and Lavernia, E.J. "Particulate Reinforced Metal Matrix Composite – A Review", Journal of Materials Science, 26, 1137-1156, 1991.

Jangg, G., Slesar, M., and Besterci, M., "Influence of Heat Treatment During Manufacturing of Al-Al_4C_3 Materials on Microstructure and Properties", 21, 25-30, 1989.

Lu, Y.X., Meng, X.M., and Lee, C.S., "Microstructure and Mechanical Behavior of a SiC Particles Reinforced Al-5Cu Composite under Dynamic Loading", Journal of Materials Processing Technology, 94, 175-178, 1999.

Rodriguez, J.A., Gallardo, J.M., and Herrera, E.J., "Structure and Properties of Attrition-Milled Aluminum Powder", Journal of Materials Science, 32, 3535-3539, 1997.

Singer, R.F., Oliver, W.C., and Nix, W.D., "Identification of Dispersoid Phases Created in Aluminum during Mechanical Alloying", Metalulurgical Transactions A, 11A, 1895-1901, 1980.

Suryanarayana, C., Bibliography on Mechanical Alloying and Milling, Cambridge International Science Publishing, 1995.

Tweed, J.H., "Manufacture of 2014 Aluminium Reinforced with SiC Particulate by Vacuum Hot Pressing", Materials Science and Engineering, A135, 73-76, 1991.

Wu, N.Q., Wu, J.M., and Wang, G.X., "Amorphization in the Al-C System by Mechanical Alloying", Journal of Alloys and Compound, 260, 121-126, 1997.