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Research Article

## A research on effect of process parameters on mechanical properties of B<sub>4</sub>C reinforced aluminum matrix composites fabricated by mechanical milling and hot press sintering route

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### Abstract

The aim of this study is to investigate the effect of mechanical milling and hot pressing process parameters on the density and strength of Al-10%B<sub>4</sub>Cp composites. For this research, powder mixtures consisting of Al matrix and B<sub>4</sub>C reinforcement particles were prepared by mechanical milling. Powder mixtures were milled for different times (5, 10, 15 and 20 hours) in a high energy planetary ball mill. In the milling vials, 10wt% B<sub>4</sub>C particle reinforcements were added to aluminum matrix powders. Average particle size of B<sub>4</sub>C and Al powders were 77µm and 63µm respectively. Powder mixtures were compacted as cylindrical samples by uniaxial hot pressing at 30 MPa. Specimens were hot pressed in nitrogen atmosphere at temperatures of 500-550-600 °C for 15-30-45 minutes. Effects of milling and hot pressing variables on microstructure and mechanical properties were investigated by means of density and hardness measurements and compression tests. Microstructures of powders and compacts were investigated by microscopy techniques. Density measurements showed that compressibility of powders decreased with increasing milling times. Density, hardness and compressive yield strength values were increased with increasing hot pressing temperature and durations.

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## 1. Introduction

In recent years, the importance of aluminum based composites have increased in terms of manufacturing structural components over ferrous materials in transportation industries on account of unique properties including, low density, excellent mechanical properties and high wear resistance[1]. Aluminum MMC materials serves excellent properties compared to monolithic materials hence, they have been applied in different industries for high performance, economical benefit and environmental concerns. Lower carbon emission, less noise and lower fuel consumption are the utmost advantages of aluminum MMC in traffic engineering that environmental regulations and fuel economy concerns increases use of aluminum MMC in transportation industries[2]. Aluminum has several advantages including low density (2,71 g/cm<sup>3</sup>), excellent corrosion resistance, high toughness, high conductivity and low manufacturing cost that make aluminum one of the most appropriate matrix material[3, 4]. Therefore, there are many published studies on aluminum matrix composites with various reinforcement materials including alumina Al<sub>2</sub>O<sub>3</sub>[5, 6], silicon carbide(SiC)[7], magnesium oxide(MgO)[8, 9], carbon nano tube(CNT)[10] and boron carbide (B<sub>4</sub>C)[11, 12]. Relatively high hardness (9.5+ in Mohs

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scale), elevated wear and impact resistance, high melting temperature and low density (2.52 g/cm<sup>3</sup>) are the outstanding features of boron carbide (B<sub>4</sub>C) which make B<sub>4</sub>C a potential reinforcement for MMC applications[13, 14]. Showordi et al., compared three aluminum metal matrix composites containing reinforcing particles of B<sub>4</sub>C, SiC and Al<sub>2</sub>O<sub>3</sub> and showed that B<sub>4</sub>C exhibited better interfacial bonding than the others[15]. Pul, have also made a comparative study on aluminum matrix composite with SiC, B<sub>4</sub>C and mixture of SiC and B<sub>4</sub>C. It is reported that 20% wt. B<sub>4</sub>C samples displayed highest wear resistance[16]. B<sub>4</sub>C reinforced composites are being used in many applications including computer hard disk substrate, armor plate materials and structural neutron absorber an account of its superior properties[17].

It has been revealed by researchers that mechanical performance of Al matrix with B<sub>4</sub>C reinforced composites were affected by many parameters. Raj and Thakur investigated the effect of particle size and volume fraction of B<sub>4</sub>C particle on microstructural and tensile properties of aluminum matrix composite. They have reported that both volume fraction and particle size have influences on activation of different strengthening mechanisms[18]. Karakoç et al., showed that increasing volume fraction of B<sub>4</sub>C particle reinforcement in the range of 0-20% improved the hardness, transverse rupture strength and tensile strength, on the other hand impact toughness was decreased[19]. Behm et al., have observed improved plasticity with both nano (40 nm) and submicron (500 nm) particle sized B<sub>4</sub>C reinforcement over micron sized counterparts at the cost of reduced yield strength[20]. However, accumulation tendency of nano particle reinforcement due to robust Van der Waals forces between the particles must be considered[21]. Production method is also another parameter which yields different results in terms of mechanical and microstructural properties. Al/B<sub>4</sub>C composites have been produced through a number of production technique such as stir casting[22], centrifugal casting[23] powder metallurgy[24], microwave sintering [25], spark plasma sintering[26], and shock compaction[27].

Liquid state methods and solid state methods are two main group of production route of metal matrix composites. Reaction between liquid aluminum and B<sub>4</sub>C generate undesirable reaction product including Al<sub>3</sub>BC, AlB<sub>24</sub>C<sub>4</sub> (AlB<sub>10</sub>), Al<sub>6</sub>B<sub>4</sub>C<sub>7</sub>, Al<sub>3</sub>B<sub>48</sub>C<sub>2</sub> (b-AlB<sub>12</sub>), AlB<sub>2</sub>, AlB<sub>12</sub>C<sub>2</sub>, AlB<sub>48</sub>C<sub>2</sub>, Al<sub>4</sub> C<sub>3</sub> and a-AlB<sub>12.7</sub>[28]. Powder metallurgy (PM) is a solid state processing technique which have several advantages over liquid state methods such as uniform distribution of reinforcement, precision forming and prevention from undesirable chemical reactions[29, 30]. Oxide layer on the surface of aluminum powders is a concern which must be taken into account in PM of aluminum matrix composites due to the fact that it behaves as a diffusion barrier during sintering and inhibit inter-particle diffusion[31]. One of the main methods to eliminate the unfavorable effect of the oxide layer is to apply high compaction pressure which breaks layers and promote metal-metal contact. The other methods are employing oxide reduction additives including Mg, Zn and Si and liquid phase sintering[32]. Mechanical milling (MM) is a PM technique which can overcome this problem by fracturing detrimental oxide layer on the surface of aluminum powders for sintering [33]. Uniform distribution of reinforcement particles into the metal matrix, which is critical issue in PM to reach high performance, can be accomplished by MM[34]. Meignanamoorthy and Ravichandran synthesized AA8079 aluminum matrix composites contain various weight percentages (0, 5, 10 and 15) of B<sub>4</sub>C through mechanical milling and powder metallurgy route. They have demonstrated that reinforcement phases were homogeneously distributed in the matrix powder without agglomeration subsequent to milling operation[35]. Carreño-Gallardo et al., fabricated 2024 aluminum alloy matrixed B<sub>4</sub>C reinforced composite via mechanical milling followed by cold compaction, sintering and T6 heat treatment. They have showed homogeneously dispersion of B<sub>4</sub>C particles by SEM images and attributed high microhardness and

compression yield strength values to distribution of the reinforcements[36]. Furthermore, MM refines grain size of matrix material occurs by severe plastic deformation due to collision of milling balls during the process that increases mechanical properties of composite materials[37]. Khakbiz and Akhlaghi, obtained 57 nm crystal size by carrying out 16 hours mechanical milling to Al 6061/B<sub>4</sub>C composite powders[38]. Alizadeh et al., also reported nanocrystalline aluminum matrix reinforced with B<sub>4</sub>C by mechanical milling and they have reached extremely high strength (1.1 GPa)[39].

Porosity is also a critical issue in PM technology which must be taken into account and reduced as low as possible in order to obtain good mechanical properties[40]. Porosity is a critical issue for mechanically milled powders especially by virtue of decreasing compressibility of powders during MM process. It is well known that powders undergo severe plastic deformation during MM which increases hardness of powder particles[41]. Karasoglu et al., reported low bulk densities for longer milling durations and they have attributed poor density results to decreased compressibility of milled powder due to excessive work hardening [42].

Hot pressing is a powder consolidation technique which makes possible achieving nearly full density in a wide range of materials by applying powder pressing and sintering at the same time during consolidation. This method is convenient for consolidation of material with poor sintering characteristic[43]. Mohammad Sharifi et al., consolidated nano particle B<sub>4</sub>C incorporated nanocrystalline aluminum powders by hot press technique under 300 MPa at 450 °C for 30 min., subsequent to mechanical milling. The relative density value of the hot pressed samples was measured to be about 98%[44]. Zhang et al., have also achieved nearly full dense Al/B<sub>4</sub>C composite samples (>98.5%) via vacuum hot pressing[45].

In this study, boron carbide reinforced aluminum matrix composite fabricated by MM and hot pressing processes and influences of milling time and hot press parameters including, temperature, time and pressure were investigated.

## **2. Experimental Procedure**

Pure aluminum powder with an average particle size of 63 μm was selected as matrix material and 10 wt% B<sub>4</sub>C with an average particle size of 77 μm opted as reinforcement. Mixture of matrix and reinforcement powders was blended in a tubular mixer for 2h. Prior to blending, 2 wt.% zinc stearate was added to the powder mixture as a process control agent (PCA) in order to reduce cold welding propensity of ductile aluminum powders during MM process. After blending, MM was performed in a planetary ball milling machine for durations of 5, 10, 15 and 20 hours under argon atmosphere. Ball to powder weight ratio and milling speed were adjusted to 10:1 and 200 rpm, respectively.

Subsequent to milling, powders were consolidated through hot press sintering route. In order to determine optimum sintering parameters, sintering temperature and time were set at 500-550-600 °C and 15-30-45 min., respectively. All sintering processes were carried out in a graphite mold at 30 MPa uniaxial sintering pressure. Density of the sintered samples was measured geometrically. Morphology of powders and microstructures of bulk samples were investigated with an optical microscope (OM) and a scanning electron microscope (SEM). Hardness of sintered samples was measured under a test load of 500 gf by using a Vickers indenter. Compression tests were performed at a strain rate of 0.05 mm/min.

### 3. Results

#### 3.1. Microscopy

Microstructural examination of milled powders obtained by a microscopy analysis is given in Figure 1. A predominantly flake-like morphology is observed in all milled powders (5-20h). Complete transformation from flake-like to equiaxial particle morphology could not be obtained even after 20 hours of milling time. The thickness of flakes decreased with increasing milling time (See also Fig.3). Rather than fracture, deformation and cold welding seem to be the dominant mechanisms. Very ductile nature of pure aluminum matrix, insufficient PCA, or relatively low milling speed (200 rpm) might be the reasons for the morphology of milled powders.

It has also been observed that various reinforcing particles are embedded in the soft matrix particles (Fig.2), which is one of the major aims of the MM process of ductile-brittle systems. However, steady state with equiaxial particles containing homogenous distribution of refined embedded reinforcement particles could not be obtained.

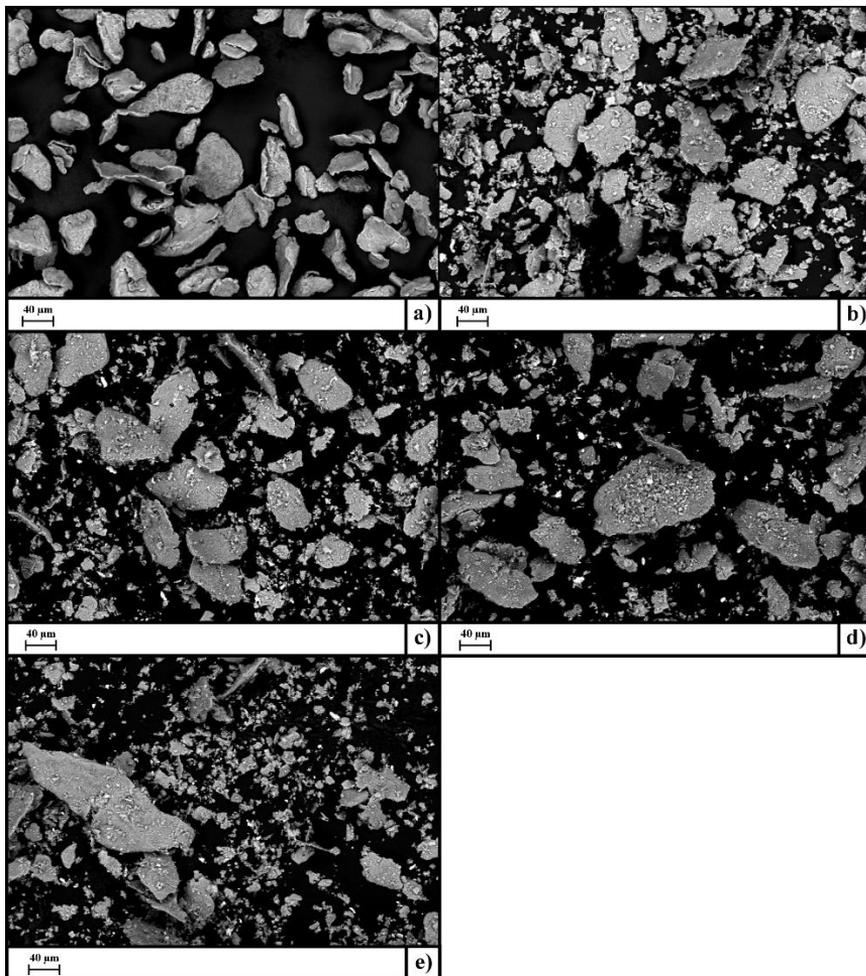


Fig. 1 Morphology of samples a) un-milled, b) 5h milled, c)10h milled, d)15h milled, e) 20h milled

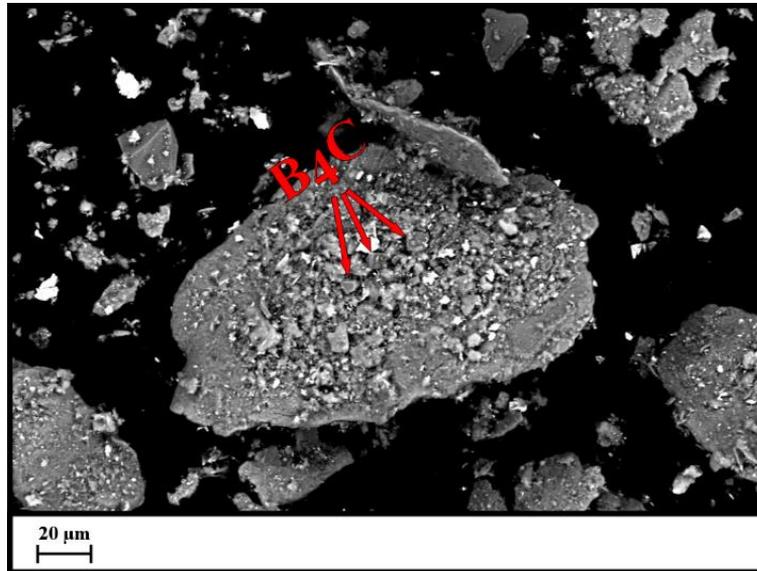


Fig. 2 B<sub>4</sub>C reinforcement particles embedded in Al matrix powder

Optical micrographs of some hot pressed samples are given in Figure 3. A predominance of deformed flake-like particles is seen in milled samples. It is also seen that; the average particle size of the reinforcing particles decreases with increasing milling time. At the same time the refined particles are more homogeneously distributed in the matrix.

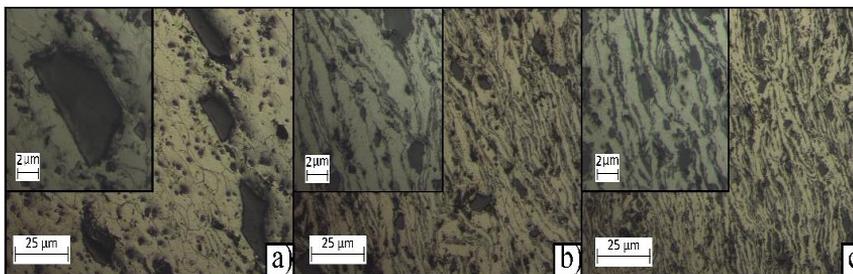


Fig. 3 a) reference sample b) 5h milled, sintered at 500 °C and 45 min c) 20h milled, sintered at 600 °C and 45 min.

### 3.2 Porosity

Figure 4 shows porosity levels of hot pressed samples. It indicates that mechanically milled samples have higher porosity levels than un-milled reference samples. This is because the hardness of milled powders is higher than as-received powders due to severe plastic deformation. Predominantly flake-like structure of the milled powders is another reason for low compressibility. Dispersion strengthening effect of reinforcing B<sub>4</sub>C particles, which were refined and embedded into Al matrix, also makes the compressibility of the milled powders difficult.

Thus, milled powders have less compressibility which can be attributed to high porosity levels of sintered samples. Furthermore, porosity results depicted that increasing milling time decreased densities. Therefore, it was argued that reduced compressibility due to enhanced deformation hardening in the particles with increased milling times, may have adversely affected the consolidation rate, thus leading to relatively low densities after sintering.

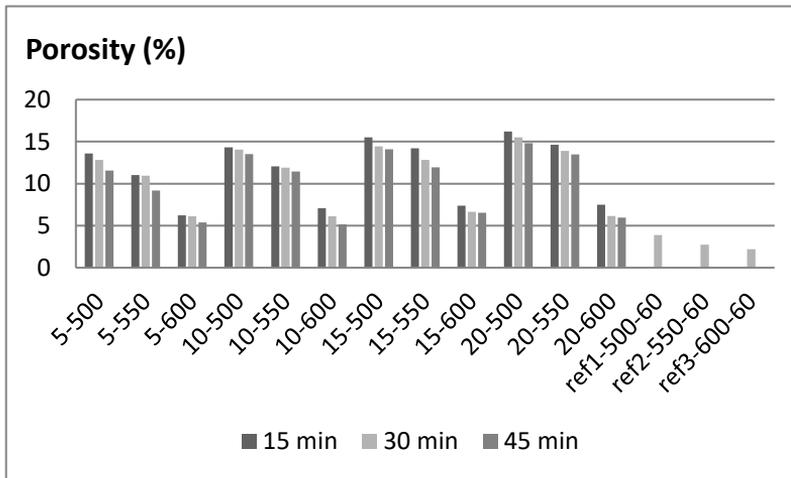


Fig. 4 Porosity of hot pressed samples

It can be concluded from Figure 4 that increasing hot pressing temperature led to formation of denser structure as a result of higher diffusion rates. Thus, the sintering temperature plays a major role as an important parameter in sintering process. Equation (1) explains this phenomenon[46] ;

$$D = D_0 \exp\left(\frac{-Q}{RT}\right) \quad (1)$$

where D is the diffusion coefficient,  $D_0$  is constant, Q is the activation energy, R is Boltzman's constant and T is the temperature. As expected, the relative density also increased with the sintering time prolonged which is expressed in equation 2[46];

$$r = 2.4\sqrt{Dt} \quad (2)$$

where r is radial distance, D is the diffusion coefficient and t is the sintering time. It appears that the lower sintering time did not allow for the formation of adequate inter-particle bonds required to consolidate the parts.

### 3.3 Compression and Hardness Tests

Compressive yield strength (CYS) and hardness values of hot pressed samples are demonstrated in Figure 5 and Figure 6 respectively. As seen in the figures, hardness and compressive strength values are very compatible with each other. It can obviously be seen that mechanically milled samples have higher CYS and hardness values compared to samples produced with as-received powders.

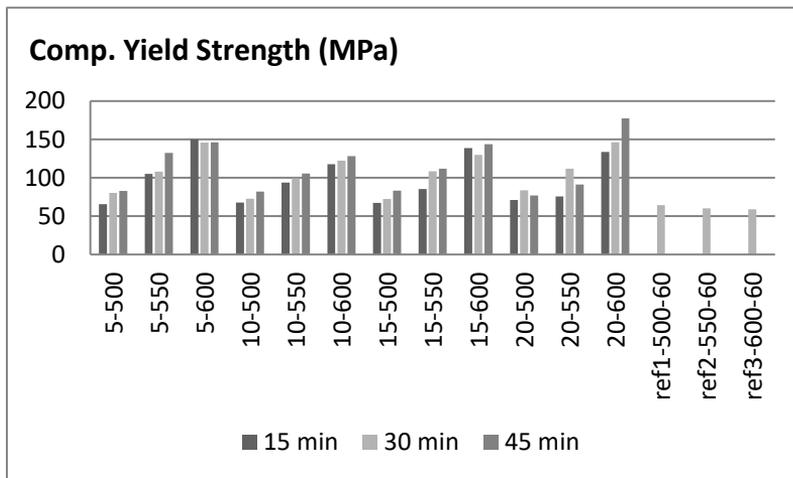


Fig. 5 Compressive yield strength of hot pressed samples

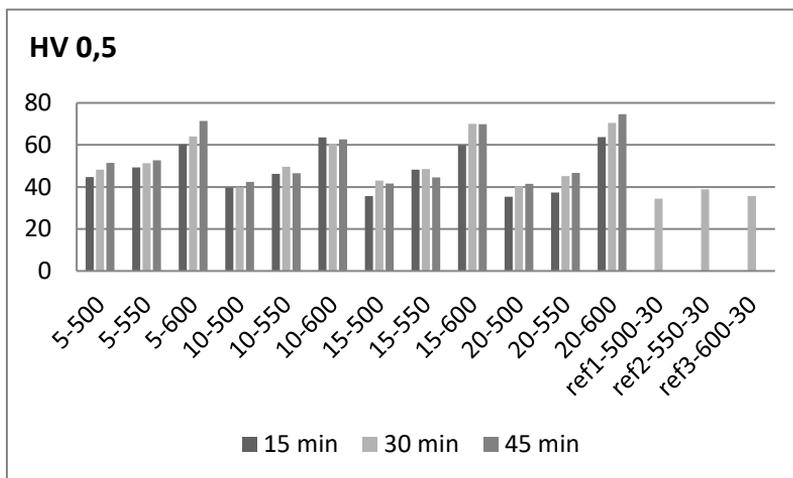


Fig. 6 Hardness of hot pressed samples

Higher CYS and hardness values can be attributed to finer grain size of the aluminum matrix of milled samples. It is well known that MM operation begets to grain refinement of metals by severe plastic deformation [14]. It can be seen from the microstructure of the samples that (Figure 3) while MM'ed samples have morphologically flattened matrix particles indicating heavily deformation, matrix particles of reference samples are equiaxed and free of deformation. Although milled samples have lower densities, grain refinement of matrix material and dispersion of finer reinforcement effects compensate the loss of density in terms of CYS and hardness. The relation between matrix grain size and the increase in strength can be explained by the Hall-Petch equation which is given by[47];

$$\sigma_y = \sigma_0 + kd^{-\frac{1}{2}} \quad (3)$$

where  $\sigma_0$  is the friction stress,  $k$  is a constant of yielding and  $d$  is the average grain size of the matrix. In Figure 3., finer reinforcement particles in MM'ed samples can be seen clearly. Particle size of reinforcement have pronounced effect on mechanical properties of composites. It is reported that reducing the particle size of reinforcement, greatly improves the strength of material[48].

Another significant factor affecting physical and mechanical properties of composite is distribution of reinforcement particles in the matrix phase. Mechanical milling is a process producing uniform dispersion of reinforcement particles in the matrix by repeated welding–fracturing–welding of a mixture of powder particles[49]. More uniform distribution of reinforcement particles of milled samples can be seen in Figure 3-c obviously, which may contribute higher CYS and hardness despite of low densities. Strength values were increased with increasing hot press temperature and time which is consistent with density results. High densities on account of higher diffusion rates explained above, may have led to stronger interfacial bonding and resulted higher CYS levels. It is well known that if the interfacial bonding between the matrix and second phase is strong, the load transfer mechanism becomes active. Enhancement in yield strength by load transfer based on the modified shear lag model is expressed as[50];

$$\Delta\sigma_y = \frac{1}{2} \cdot f \cdot \sigma_m \quad (4)$$

where  $f$  is the volume fraction of the reinforcement and  $\sigma_m$  is the yield strength of the matrix.

In Figure 7, it can be seen that bright white colored second phase particles appeared in milled sample. These particles may be iron particles which could be formed from wearing of steel vial and balls during mechanical milling operation. These particles may have also behaved as reinforcements and led to an improvement in strength and hardness of the material.

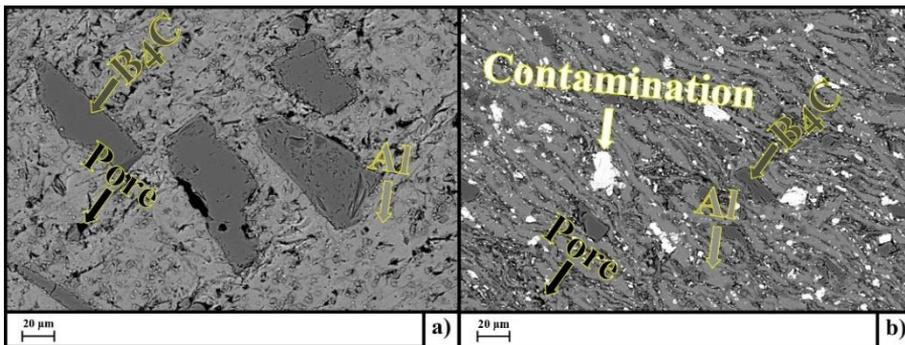


Fig. 7 SEM images of a) reference sample b) 20h milled, sintered at 600 °C and 45 min.

Although mechanical milling has a prominent effect on CYS and hardness, influence of milling time is not certain. Results did not follow expectation of increasing strength with increasing milling time. Instead, a fluctuation appeared in compression test measurement. This situation may be related to densities.

#### 4. Conclusion

The influence of the milling time, sintering temperature and sintering time on the mechanical properties of the Al matrix B<sub>4</sub>C reinforced composites produced through

mechanical milling and hot pressing route was investigated. The major findings of this study can be summarized as follows.

- A predominance of deformed flake-like particles was observed even in 20 h milled samples. Rather than fracture, deformation and cold welding were dominant mechanisms in mechanical milling processes.
- Strengthening of powders by work hardening, grain boundary hardening and dispersion hardening during mechanical milling decreased their compressibility. Porosity level of samples increased with increasing mechanical milling time.
- Density is one of the most important factors determining strength of the samples. High hardness and compressive yield strength require high density.
- Increasing sintering time and temperature increased the density of the samples which increased the compressive yield strength and hardness values.
- Despite their lower density, higher strength values were observed in the milled samples than in the un-milled samples. Much higher strength values can be achieved if higher densities can be obtained in milled powders. Since the pressure that can be applied in the graphite hot pressing molds is limited, a cold prepressing operation can be performed in the metal molds to increase the density.

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