Effects of cold pressing and extrusion on the microstructures and mechanical properties of SiC and B₄C reinforced Alumix-231 alloys

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In this research, mechanical and microstructure properties of cold pressing (CP) and extrusion (EXTR) specimens produced via powder metallurgy were investigated and compared. For this comparison, EXTR specimens were hot extruded at 565°C with an extrusion ratio of 4:1. Likewise, CP specimens were compacted under a pressure of 600 MPa and sintered at 565°C. Then, the effects of volume fraction on SiC and B₄C particle reinforced Alumix–231 composites were investigated using optical microscopy, SEM and macro hardness testing. Compared to CP specimens, it was found that the hardness and mechanical strengths of Alumix–231 specimens without reinforcement produced using EXTR increased in the ratio of 140 and 190%, respectively. The test results showed that increase in uniformity of reinforcement dispersion and enhancement of interfacial bonding strength of the composites is the main reasons for the improvement of EXTR specimens. As a result, EXTR improves mechanical properties of the specimens by decreasing the porosity and thus increasing the density and interfacial bonding strength.

Key words: Cold pressing, extrusion, metal matrix composites, powder metallurgy, mechanical properties.

INTRODUCTION

Today, composite materials have been manufactured in order to improve the properties of metals that do not meet the requirements of specific applications. Composite materials, compared to other metal products, have some unique properties such as lightness and exceptional mechanical strength that makes them superior to metals. In addition to being light, with their high specific elastic modulus, strength, wear and corrosion resistance, and furthermore, the fact that these can easily be manufactured at low costs via conventional production methods (casting, extrusion, forging, wrought, powder metallurgy, etc.), the utilization of composite materials particularly in automotive, space and defense industry applications increase day by day (Lee et al., 1997). As a result of soaring interest in these materials, many experimental and theoretical studies are being carried out in order to determine their mechanical properties and behaviors (Yulong et al., 2004; Suraj, 2001; Ozdemir et al., 2000). In metal matrix composites (MMCs), aluminum and its alloys take the first place as matrix materials. Due to their low density, low fusion temperature, good mechanical and physical properties, inherent corrosion resistance in air atmosphere and other environments, wettability of many ceramic additive materials, these alloys are commonly preferred. Many techniques have been developed to fabricate these types of materials. One of these techniques is solid-phase processing (Huda et al., 1995; Srivatsan et al., 1995; Suryanarayana, 2001).

A solid-phase processing technique such as powder metallurgy offer uniform distribution of the reinforcements, fine grained structures and precise control of the microstructures (German, 1996; Lee et al., 1988; Lee et al., 1999). In general, consolidation of the particles and
matrix powder by vacuum hot pressing, followed by a secondary working such as extrusion, is an effective method for fabricating SiC reinforced aluminum alloy composites with enhanced mechanical properties (Lloyd, 1997; Zhangwei et al., 2010). When a material is fabricated via powder metallurgy, pores are generally formed in the matrix. These pores comprise the majority of micro-crack initiators during deformation. Thus, a fabrication process with proper parameters is very important to increase the interfacial bonding strength and decrease the number of pores, and so to improve the mechanical properties (Song et al., 2009). Aluminium-silicon P/M alloys have been of particular interest as these materials offer the advantage of high-wear resistance, high strength, good temperature resistance, and a low coefficient of thermal expansion. When producing hypereutectic aluminum-silicon alloys using traditional casting methods, the silicon present in the alloy can solidify into a large primary silicon phase that can have a detrimental effect on the mechanical properties. Therefore, the amount of silicon that can be added to aluminum is limited. The application of P/M processing to the manufacturing of aluminum-silicon alloys is of great interest as this route offers several advantages over traditional casting methods (Heard et al., 2009). Today, the following steps have been followed in the production of composites using powder metallurgy:

Blending, cold pressing (CP), vacuum degassing, hot pressing, can removal, extrusion. In this study; the authors have used a process. The steps of this process have been shown in Figure 1. The sequence is described as follows:

1. The aluminum alloy powders and B₄C reinforcement are blended;
2. The blended powder is CP’ed into ‘green forms’ with typically 90% density at this stage;
3. The ‘green forms’ are vacuum sintered to produce billets that are also typically 90% dense; no can removal is necessary.
4. The billets are extruded (Hu et al., 2001).

In this study, the alloy Alumix-231, one of the novel...
hypereutectic aluminum-silicon alloys and been produced by Ecka Granules with a nominal composition of Al–15Si–2.5Cu–0.5Mg, reinforced with SiC and B4C particles has been used in the production of the specimens by using CP and EXTR in the powder metallurgy method in order to improve the properties of composites. The reason for using Alumix-231 is that hypereutectic Aluminum–silicon PM alloys offer the advantage of high-wear resistance, high strength, creep resistance and a low coefficient of thermal expansion. The effects of CP and EXTR on the mechanical properties and microstructures of the composites, which are made of SiC and B4C particles reinforced Alumix–231, have been studied.

**MATERIALS AND METHODS**

The chemical compositions (in weight percent) of the matrix used in the present study were given in Table 1. The Alumix–231 powders were produced by inert gas atomization, and have an average size of about 162 µm. The SiC and B4C reinforcements were in the form of particulates with an average size of about 32 µm and 110 µm, respectively. The volume fractions of the SiC and B4C particles in the composites were 5, 10 and 20 wt. %, respectively. Alumix–231/SiC and Alumix–231/B4C powders were mixed homogenously for 30 min using a milling machine (Turbula-Model T2F, Willy A. Bachofen AG Maschinenfabrik, Switzerland).

In the manufacturing process of these mixed powders; two different methods were used. The first one was CP and the other one was EXTR. In the first method CP; the mixed powders were compacted under a pressure of 600 MPa at room temperature by using a hydraulic press of 150 ton. All compacted tensile specimens were sintered in a horizontal tube furnace (that was used for sintering process that break up the oxide layer and decrease the number of pores. As the applied external pressure increases, the porosity of the specimens decreases. As seen in the figure, porosity in CP specimens with added SiC and B4C particles is higher than that of the EXTR specimens. The porosity of specimens having SiC is lower in EXTR. This indicates that EXTR improves the density of the specimens. Comparing the microstructures of specimens produced by CP and EXTR, it can also be seen from Figure 3 that the pores were generally co-existed with the SiC and B4C particles and around them, and so indicating that the interfacial bonding between both SiC and B4C particles and the matrix is relatively weak. Figure 3 shows the optical micrographs of the specimens containing 20 wt. % SiC and B4C particles produced by CP and EXTR. It can be seen from Figures 3 (a) and (b) that the SiC and B4C particles in specimens produced by CP distribute quasi-uniformly in the matrix without much aggregation while in specimens produced by EXTR the distribution

<table>
<thead>
<tr>
<th>Nominal target</th>
<th>Al</th>
<th>Si</th>
<th>Cu</th>
<th>Mg</th>
<th>Fe</th>
</tr>
</thead>
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<tr>
<td>Bal.</td>
<td>14 to 16</td>
<td>2.4 to 2.8</td>
<td>0.5 to 0.8</td>
<td>N/A</td>
<td></td>
</tr>
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The porosity is calculated as where \( \rho_t \) is the theoretical density measured from rule of mixture and \( \rho_e \) is the theoretical density measured from rule of mixture and \( \rho_e \) is the density measured by water displacement method:

\[
 \text{Porosity(\%)} = \frac{\rho_e - \rho_T}{\rho_T} \times 100
\]

By using a Vickers hardness tester (HSV–30, Shimadzu, Japan), the HV5 hardness measurements were performed on all specimens produced by CP and EXTR in order to observe the evolution of hardness as a function of volume fraction. The tensile strength of the composites under different volume fractions were measured by uniaxial tensile testing. At room temperature, tensile test specimens were tested at a constant cross-head velocity of 0.5 mm/min on a servo-hydraulic test frame (AG-50 kNG, Shimadzu, Japan) with video extensometer. Average tensile data was collected by performing the tests at least 3 times for each specimen type. The microstructures of the specimens produced by CP and EXTR were inspected by using optical microscopy (Eclipse LV150, Nikon, Japan) while the fracture surfaces of the tensile specimens were inspected using scanning electron microscope (SEM; JSM–6060, JEOL, Japan).

**RESULTS**

Microstructures and porosity evolution of the specimens produced by CP and EXTR

The porosity evolution of the composites as a function of volume fraction was shown in Figure 2. The specimens are of high density due to combined mechanical and thermal effects during sintering process that break up the oxide layer and decrease the number of pores. As the applied external pressure increases, the porosity of the specimens decreases. As seen in the figure, porosity in CP specimens with added SiC and B4C particles is higher than that of the EXTR specimens. The density of the specimens having SiC is lower in EXTR. This indicates that EXTR improves the density of the specimens. Comparing the microstructures of specimens produced by CP and EXTR, it can also be seen from Figure 3 that the pores were generally co-existed with the SiC and B4C particles and around them, and so indicating that the interfacial bonding between both SiC and B4C particles and the matrix is relatively weak. Figure 3 shows the optical micrographs of the specimens containing 20 wt. % SiC and B4C particles produced by CP and EXTR. It can be seen from Figures 3 (a) and (b) that the SiC and B4C particles in specimens produced by CP distribute quasi-uniformly in the matrix without much aggregation while in specimens produced by EXTR the distribution...
homogeneity of the SiC and B$_4$C particles in the matrix was improved, as shown in Figures 3(c) and (d), respectively.

Figures 3(c) and (d) also indicate the particle fractures in specimens with SiC and B$_4$C particles after EXTR because of the extensive plastic deformation of Alumix–231 matrix during EXTR, the load applied is transferred to the ceramic particles. It was observed from Figures 3(c) and (d) that the average sizes of the SiC and B$_4$C particles decreased up to 50% in EXTR because large sized particles were broken into smaller sized particles during EXTR.

**Hardness changes with volume fraction**

Figure 4 shows the hardness variations versus volume fraction and contents of the specimens. It can be seen from the figure that the hardness of the specimens increases with increasing the volume fraction of both SiC and B$_4$C particles. On the other hand, as seen from Figure 5, the tensile strength decreases with increasing the volume fraction of ceramic particles. This situation shows that particle cracking has no significant effect on the hardness of the specimens.

**Mechanical properties of the specimens produced by CP and EXTR**

The tensile properties of the specimens produced by CP and EXTR are showed in Figure 5. Compared to CP specimens, it was found for Alumix–231 specimens without reinforcement that the hardness of the EXTR specimens increased in the ratio of 140% as well as the tensile strength of the specimens increased in the ratio of 190%. Moreover, during EXTR, it was seen that while the hardness increased; the tensile strength decreased with increasing the volume fraction of the SiC and B$_4$C particles, due to being more brittle of the alloy. The reason for which the tensile strength decreased is the particle fracture. It was observed that the fracture mechanism in the matrix was ductile accompanied by the separation of the particles from the matrix and causing the surface to be uneven during CP or by the SiC and B$_4$C particles fracture during EXTR. Then the surrounding of the particles fractured was again, completely covered by matrix due to the larger deformation during the extrusion process. As a result, the EXTR improves the mechanical properties. The test results showed that the main reasons for the improvement of EXTR specimens are the increase in uniformity of particle dispersion (Figure 3) and enhancement in the interfacial bonding strength of the specimens. Figure 5 shows that the tensile strengths of the specimens produced by EXTR are higher than the ones produced by CP. It can be seen from Figures 3(c) and (d) that EXTR causes the interfacial bonding between the particles and matrix to be stronger, moreover; EXTR decreases the porosity and thus improves the density.

As shown in Figure 5, the tensile strength of EXTR
Figure 3. Optical microstructures of the specimens with the volume fraction of the SiC and B_4C particles of 20%.

Figure 4. The variation of the HV hardness versus volume fraction and contents of the specimens.
Alumix-231 specimens without reinforcements increased in the ratio of about 190% according to their CP specimens. When the tensile strength of the specimens containing SiC and B$_4$C particles has been compared, on the contrary of the expectations that the results of SiC and B$_4$C particles would be higher than Alumix-231, it was obtained that the strengths of the specimens with the additive ratios of 5, 10 and 20% particles are lower.

**Fracture mechanisms**

The fracture surfaces of the specimens with 10% reinforcement exhibit both brittle and ductile fracture features according to production process although fracture in the specimens with 5 and 20% reinforcements is ductile and brittle, respectively. Thus, SEM fracture surfaces after tensile testing were shown in Figure 6 for the specimens with 10% volume fractions of SiC and B$_4$C particles manufactured by CP and EXTR.

In fracture surfaces of the matrix, there are numerous dimples and decohesion of both SiC and B$_4$C particles from the matrix, as seen in Figures 6 (a) and (b). The dimples can be a result of the void nucleation and subsequent joining with strong shear deformation and fracture process on the shear plane, while the decohesion of the particles can be explained by “pull-out” of the ceramic phases caused by high stress concentration. That the ceramic particles exhibit typical brittle fracture surrounded by the ductile dimples in matrix after EXTR can be shown in Figures 6 (c) and (d) and Figure 7. As can be seen from Figures 6 and 7, the reason of brittle fracture is mainly the crushing during EXTR process. The main difference of fracture surfaces is that: the “pull-out” of the SiC and B$_4$C particles from the matrix can be clearly observed in CP, while the dominant fracture mechanism for the specimens in EXTR is particle fracture, and also the “pull-out” of the SiC and B$_4$C particles from the matrix can hardly be observed.

**DISCUSSION**

Generally, pores are observed in all specimens. The generation of pores may be attributed to oxide layer formation on the surfaces of Alumix–231 during the fabrication process. It was observed that the oxide layer can substantially degrade the solid-phase-sintering ability, thus inhibiting diffusion (Zhangwei et al., 2010; Song and He, 2009; Flumerfelt, 1998; Gutin et al., 1972). It is believed that extrusion can decrease the number of pores and improve the interfacial bonding strength between the SiC and B$_4$C particles and matrix, since high pressure can crack the oxide layer at the surface of the aluminium powders and enhance cohesion between the powders. It should be noted that extrusion generated severe plastic deformation of the matrix, which led to the rearrangement of SiC and B$_4$C particles, and thus,
improved the distribution uniformity of the SiC and B\textsubscript{4}C particles. It can be seen from Figures 3 (c) and (d) that, when the stress concentration is beyond the fracture strength of the ceramic particles, the hard and brittle ceramic particles fracture (Kiser et al., 1996). During deformation, bigger particles fracture generally more easily than smaller particles (Tham et al., 2002). Larger sized particles have larger interface area with the matrix and so face higher stress concentration. Then, the intrinsic flaws within the particle control the particle fracture strength. Since the size of a flaw is restricted by the size of the particle, larger particles are more prone to fracture when the containing a flaw, which is greater than a certain critical size (Zhao et al., 1994).

It was reported that the contradiction between the hardness and tensile strength of the alloys is due to the different types of loadings (Shen et al., 2000). During hardness test, the localized pressure is transferred to the underlying material directly under the indented surface to support most of the load. In this loading, the stress field directly below the indenter is predominantly compressive while the matrix under a severe triaxial pressure. This stress is completely different from deformation of sample under tensile testing. The pre-cracked particles cannot subject to the tensile stress and thus will separate when the specimen is under the tensile loading. When the tensile loading increases, this facilitates the crack propagation and the growth as well. During the hardness test, pre-cracked particles can still put up with the compressive traction across the cracking surfaces. Plastic flow of the matrix between the particles accommodates the deformation caused by the indentation pressure, and so the particles are "pushed" into the material (Shen et al., 2001). Therefore, the material response under indentation is not significantly effected by the extrusion induced fractured particles. As a result of this, the hardness of the specimens increases with increasing the volume fraction of both SiC and B\textsubscript{4}C particles, as seen in Figure 4. Another reason of the increase in hardness is that Si-phase in the materials with silicon addition treats.
like as-extruded state. When the mechanical properties were determined during EXTR, it can be observed from Figure 5 that the tensile strength decreased with increasing the volume fraction of the SiC and B₄C particles. In some other studies (Chawla and Shen, 2001; Chawla et al., 1998; Ganesh and Chawla, 2005), it has generally been seen that as the volume fraction of particles increases, the interfacial area between particles and matrix also increases, as a result, from the matrix to the hard ceramic particles more load can be moved. Moreover, the increase in the volume fraction of the SiC and B₄C particles causes dislocations in the matrix (Zhangwei et al., 2010). However, for a specimen subjected to elastic loading, the reinforcements are initially carried a significant fraction of the stress, and the specimen undergoes microplastic yielding.

Microplasticity in the specimens occurs at stress concentrations at the poles or at sharp corners of the reinforcements in the matrix (Williams et al., 2002; Corbin and Wilkinson, 1994; Chawla et al., 1998). The stress on the particles depends on the applied strain and it increases with rising strain. As the particles are compared to the matrix, they will usually begin to fracture before reaching the ultimate tensile strength of the specimen because of low strain-to-failure of the SiC and B₄C particles. In some cases, the onset of particle fracture has occurred at stresses slightly below the yield strength of the specimens and increases with the size and volume fraction of the particles (Finot et al., 1994). From Figures 3 (c) and (d), it is observed that EXTR procedure causes a large number of the particle cracking and the cracking fraction of the particles increases with rising the volume fraction of the SiC and B₄C particles. The cracked particles in the specimens can not transfer any load since the stress is released along the cracking surfaces. In addition, the cracked particles will act as the microcrack initiators during deformation, leading to the decrease in the strength of the specimen, as seen in Figure 5. Since the interfacial reaction between Alumix-231 and ceramic particles depends on some fabrication conditions (fabrication method, temperature, time, atmosphere, and chemical composition of both the Alumix-231 and the particles), due to the difficulty in bonding of Alumix-231 and B₄C having larger particles compared to SiC particles, voids at the particle/matrix interface, fraction of B₄C particles after EXTR and thus weakness of the interfacial bonding strength, it is observed that the tensile strength of the specimens having B₄C is lower than the tensile strength of the specimens having SiC.

The critical criterion to determine the fracture mode of the specimens during deformation is the relationship between the particle strength and particle/matrix interfacial bonding strength (Shen et al., 2001). When the particle/matrix interfacial bonding strength is high, particle fracture usually happens during deformation, as can be seen in Figure 7. Moreover, decohesion between the SiC and B₄C particles and the matrix takes place prior to the particle fracture when the particle/matrix interfacial bonding strength is weak. It can be seen in Figures 3 (c) and (d) that EXTR process can help to decrease the number of pores and improve interfacial bonding strength of the specimens. Another important feature is that EXTR causes the fracture of a large number of the SiC and B₄C particles. The above mentioned situation results in the fracture mode changing from particle “pull-out” from the matrix CP to particle fracture EXTR. It is seen from Figure 6 that CP specimens have coarse and shallow dimples whereas EXTR specimens have fine ductile dimples, except for B₄C EXTR specimen. As a result of this, it is observed that the strengths of the specimens with SiC after EXTR processes are higher than the strengths of the specimens having B₄C after both CP and EXTR processes.
Conclusion

Effects of extrusion and reinforcement volume fraction on mechanical properties of SiC and B4C particles reinforced by Alumix-231 composites have been investigated in this paper. The test results showed that the increase in uniformity of particle dispersion and enhancement in the interfacial bonding strength of the composites are the main reasons for the improvement of EXTR specimens. Compared to CP specimens, it was found for Alumix–231 specimens without reinforcement that the hardness of the EXTR specimens increased in the ratio of 140% as well as the tensile strength of the specimens increased in the ratio of 190%. Moreover during EXTR, it was seen that while the hardness increased; the tensile strength decreased with increasing the volume fraction of the SiC and B4C particles. The reason for which the tensile strength decreased is the particle fracture. It was observed that the fracture mechanism in the matrix was ductile accompanied by the separation of the particles from the matrix and causing the surface to be uneven during CP or by the SiC and B4C particle fracture during EXTR. As a result, the EXTR improves the mechanical properties of the specimens by decreasing the porosity and thus increasing the density and interfacial bonding strength.

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