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HOT EXTRUSION FOLLOWED BY A HOT ECAP CONSOLIDATION COMBINED TECHNIQUE IN THE PRODUCTION OF BORON CARBIDE (B_4C) REINFORCED WITH ALUMINIUM CHIPS (AA6061) COMPOSITE

KOMBINIRANA TEHNIKA IZDELAVE KOMPOZITA NA OSNOVI BOR KARBIDA (B_4C), OJAČANEGA S KOSMIČI Al ZLITINE AA6061 S POSTOPKOMA VROČEGA IZTISKOVANJA IN ECAP

Sami Al-Alimi[1*], Mohd Amri Lajis[1], Shazarel Shamsudin[1], Nur Kamilah Yusuf[1], B. L. Chan[1], Didane Djamal Hissein[1], Mohammed H. Rady[2], Muntadher Sabah Msebawi[3], Huda Mohammed Sabbar[3]

[1]Sustainable Manufacturing and Recycling Technology, Advanced Manufacturing and Materials Center (SMART-AMMC), Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, BatuPahat, Johor Malaysia
[2]College of engineering, Wasit University, Kut, Iraq
[3]Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

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A new and promising MMC approach to the reduction of pollution, greenhouse effects, and emissions is to develop a technology related to materials composite forming. Hot extrusion followed by hot ECAP is a combination of solid-state recycling method (direct recycling) that consists of chip preparations, cold compaction, and hot extrusion, followed by the ECAP process. The developed process is used to consolidate the chips for direct chip recycling purposes without the remelting phase. In this study, finished or semi-finished products from B_4C-reinforced particles and AA6061 aluminium chips were produced. The samples made by hot extrusion were compared with samples obtained from hot extrusion followed by the hot ECAP process in terms of mechanical properties. Additional plastic deformation by hot ECAP after hot extrusion significantly increased the mechanical properties of the MMC compared with the samples obtained from the hot extrusion only. The density and microstructure of the samples were also determined.

Keywords: hot ECAP, hot extrusion (HE), metal matrix composites (MMCs), and solid-state recycling

Avtorji so izvedli obetajoče preizkuse izdelave kompozita s kovinsko matrico (MMC, angl.: Metal Matrix Composite), s katerim se zmanjšuje emisija toplogrednih plinov in onesnaževanje okolja. Razvili so nov postopek, ki se nanaša na oblikovanje kompozitnih materialov. Metoda direktnega recikliranja v trdnem stanju s kombinacijo vroče ekstruzije in vročega ECAP postopka (angl.: Equal Channel Angular Pressing) je bila sestavljena iz priprave kosmičev, hladnega stiskanja in ECAP postopka, ki je sledil vroči ekstruziji. Razviti postopek omogoča direktno recikliranje kosmičev iz Al zlitine s konsolidacijo brez vmesne faze pretaljevanja. Avtorji so izdelali pol-proizvode in končne izdelke iz kompozita na osnovi Al zlitine AA6061 ojačane z delci B_4C. Primerjali so mehanske lastnosti izdelanih vzorcev z vzorci, ki so bili izdelani samo s postopkom ekstruzije (brez ECAP). Dodatna plastična deformacija, izvedena s postopkom vročega ECAP, je pomembno izboljšala mehanske lastnosti kompozitov v primerjavi z vzorci, ki so bili samo vroče ekstrudirani. Določili so tudi gostoto in mikrostrukturo izdelanih kompozitov.

Ključne besede: postopek vročega pravokotnega enako kanalskega iztiskovanja (ECAP), vroča ekstruzija (HE), kompoziti s kovinsko matrico (MMCs), recikliranje v trdnem stanju

1 INTRODUCTION

Aluminium is one of the metals commonly used in engineering due to its desired mechanical and physical characteristics. Here, a method of recycling metal matrix composites (MMCs) by extrusion followed by Hot Equal Channel Angular Pressing (ECAP) is introduced. The research consolidated aluminium chips to be the best route from the point of investigations on the metal properties. Recycling light metal scraps such as aluminium reduces the number of wasted materials, prevents the needs for the mining process, and offers new materials for both home usage and manufacturing industries. To reduce CO_2 emissions and climate hazards, it is required to apply waste recycling and processing technology. The most used processes are conventional recycling (CR) and solid-state recycling (SSR).[1] Aluminium, compared with steel, possesses several advantages, such as good strength, low weight, good resistance to corrosion, and an easy material-forming process. A wide range of industrial structures around the globe are made using aluminium, such as offshore platforms, bridges, building frames, and automotive parts. As the need for MMC materials increases, it results in a huge quantity of waste el-

*Corresponding author's e-mail:
samialalimi@gmail.com (Sami Al-Alimi)

ements and unwanted materials, which cause harmful pollution to the planet. Hence, the processing technology of material formation needs to be developed for both powder form and recycling objects. The conventional recycling processes that involve remelting scraps have several disadvantages, such as a material loss, high energy consumption, high labour cost, and large operating steps. The material loss is related to the higher surface-area-to-volume ratio of the scraps.[2] The significance of implementing SSR is that it does not involve melting process, which results in higher cost of processing methods and avoiding material loses.[3,4] It is proposed that solid-state recycling forms fully dense metals and saves 95 % of the original materials, which can be reused.[5] To enhance the mechanical properties of the recycled materials for use in automotive engineering applications, further investigations are required for both processing techniques and the type of composite materials.[6–8]

MMC-processing techniques could be performed either in situ or ex situ. The ex-situ process involves the addition of reinforcement particles from outside the process, while the in-situ process involves exothermic reactions or chemical reactions.[9] The manufacturing processes produce MMCs materials that are suitable for both Al-based micro, nano or hybrid materials. Several researchers have proposed that the conventional processes such as casting, sintering, spark plasma, gas injection, and pressure-less infiltration have limitations on the composite's consolidation due to the scale of the grain growth during the forming process.[10,11] The critical impacts of including B_4C as reinforced particles to solidify the chips are recognizable from the mechanical and physical properties of the sample.

There is a need to develop methods such as hot extrusion and hot ECAP methods to process nano/micro reinforced light metals.[12,13] Some researchers investigated the related parameters and technology of hot extrusion such as chip size, ram speed, extrusion ratio pre-heating temperature, billet temperature, and die properties. Others investigated ECAP-related technology such as pressing force, pressing temperature, inner angle, outer angle, billet temperature, and type of reinforced particles. In addition, there are limitations to using hot extrusion to enhance the mechanical and physical properties of the processed material. Also, cold ECAP results in material failure, and there is a need to supply heat to the ECAP die. This research combined the processing techniques of both extrusion followed by hot ECAP for four passes to obtain properties as close as, or superior than, the as-received AA6061 aluminium. The research aimed to produce recycled screws, which could be marketable for industrial applications.

The conversion of the direct process of aluminium chips into a finished product by a hot-extrusion process was patented by Stern (1945). Also, the method to solve the problem of the metal loss during the remelting process and more cost saving to the energy usage during the production.[14] The extrusion technique could be used for both types of material, either powder form or scraps. Different researchers have worked on the related technology improvement and types of MMC. Güley et al.[10] investigated the effects of the processed die design, welding quality, and machining chips. A flat-faced die is typically used to form solid rectangular profiles of AA6060. The authors proposed that the porthole die resulted in better welding chips and obtained more than 80 % of the ductility than the flat-faced die. Cun-Zhu et al.[15] fabricated AA2024 aluminium reinforced with B_4C particulates by a hot-extrusion method. The elasticity modulus and strength of the materials improved with the addition of B_4C particles into the matrix alloys. At 20 $\varphi/\%$. B_4C/AA2014 Al composites, 626.7 MPa was reported as the highest tensile strength compared to 489 MPa for pure AA2024 aluminium alloy and strong interface bonding between the B_4C particles and AA2024/Al was achieved by hot extrusion. Jahedi et al.[16] studied the effects of ram speed on the final products of Al–SiC composites through the hot-extrusion method. They showed that the highest tensile strength achieved was 217 MPa, obtained at a speed rate of 2 $mm \cdot s^{-1}$. The microstructure showed good agreement to support the tensile strength of the material. Mani et al.[17] investigated and compared the mechanical and physical properties of both hot forward extrusion and hot ECAP forward extrusion. The highest Ultimate Tensile Strength (UTS) value was 140 MPa, and it was obtained at 10 $\varphi/\%$ of SiC produced by the hot ECAP forward extrusion process. In comparison, 136 MPa was obtained by forward extrusion. Also, the microstructure investigations showed better-consolidated samples and reinforcement particles distribution at 10 $\varphi/\%$ of SiC, which evidences the superior performance of the hot FE-ECAP process.

Processing light-metal-based bulk materials with coarse grains normally lowers the cost and minimises the manufacturing time. Amongst the common processing techniques that produce Ultrafine-Grained (UFG) materials are Severe Plastic Deformation SPD methods, ECAP, Conform, torsion straining, accumulative roll bonding, rolling, high-pressure torsion, and multiaxial deformation. They break the coarse grains, impose high strain and dislocations of the density and thus, form new grain boundaries.[18] ECAP is one of the most effective processing methods. It imposes high strain to the billets without changing the specimen dimensions. It is the method to process small sizes of billets in labs or large billets in industrial applications. Fang et al.[6] reported that the grains of two alloys, Al/0.63 w/% Cu and Al/3.9 w/% Cu, that were subjected to the ECAP process were refined to submicron level after four passes. The tensile fracture strength increased as the ECAP pass was increased for both alloys. Miyazaki et al.[19] studied the effect of preheating temperature treatment ranging from 260 °C to 560 °C of the AC4CH aluminium alloys by a cold ECAP process. The hardness increased due to the ECAP passes, which caused material strain hardening. Also, based on the observation of the reported process, the preheat should not be more than 470 °C or lower than 410 °C. Cracks would occur near the inner section of the ECAP channel of the samples and cracks could be overcome by the repetitive process of the ECAP passes. Jafarlou et al.[20] processed the aluminium alloy AA6061 by the modified ECAP of tubular components. The mechanical and physical investigations revealed notable increases in the tensile strength and hardness, along with a reduction in grain size and ductility compared to the as-received workpieces.

Recently, researchers have enhanced the mechanical and physical properties of the materials by developing processing methods to produce MMC, whether recycled or in the powder or chip form. The combined processing methods improved the strength of the MMC by imposing higher strain and longer contacts between the dies and billets. The methods also improved the UFG of the material.[3] Yoshimura et al.[4] conducted solid-state recycling of Al–Si through cold extrusion followed by a cold-rolling method. They showed that extrusion with a rolling process minimized the material's voids and the insufficient plastic strain, which resulted in higher strength of the recycled specimens compared to the samples produced by a cold-extrusion process. Fogagnolo et al.[21] investigated the method of recycling aluminium scraps by hot pressing followed by hot extrusion. The process could satisfactorily consolidate the scraps and prove that the process has higher mechanical strength.

Among the advanced developed materials, materials produced by hot extrusion followed by multiple passes of the hot ECAP process show superior mechanical and microstructural properties when compared with those made using the extrusion process and the as-received Al-AA6061 sample. The deformation processes change the shape of the initial solid metals from a billet to an extruded bar and is then plastically deformed between dies to obtain the final desired geometries. A sequence of extrusion and hot ECAP methods were progressively used to form the extruded hot billets into the used engineering products.[22] A novel process of hot extrusion combined with hot ECAP to directly recycle aluminium chips has been developed in this study. The method is used to consolidate the chips for direct chip recycling purposes without remelting. The method guides the production of finished or semi-finished products from a boron-carbide-reinforced aluminium chip composite. The end products can be achieved by performing finishing processes on the extrudates processed by this research.

Processing MMC materials by extrusion or ECAP is currently being conducted for the manufacture of ultrafine-grained structures for different engineering materials. High-strength, semi-finished products produced from aluminium powder or scraps that have undergone ECAP could be used in the automotive, power, or aerospace industrial applications. These products include fasteners such as screws, screw rivets used in the assembly of aluminium components for aircraft, and other engineering structures or elements for aircraft components (stringers, skin plates, etc.).[22] The results were validated to the as-received samples of T6-AA6061. **Figure 1** shows the screws produced by SMART-AMMC at UTHM. **Figure 1** shows the gears and screws of the experimental process of hot extrusion followed by ECAP.

Figure 1: Fabricated gears and screws of 5 φ/% B_4C/AA6061 chips at SMART-AMMC, UTHM

3 MATERIALS AND METHOD

Samples of recycled MMC were obtained in several steps after the combination of AA6061 aluminium chips and B_4C particles. The combined materials contained two different groups of material or more. The process was aimed to enhance the mechanical and physical properties of the products. B_4C powder was selected as the reinforcement particles due to its high hardness, lower density, high strength, high wear, high impact resistance, high melting point, low coefficient of thermal expansion, and good chemical stability.

The AA6061 scrap size is 1 mm and the chemical composition of both AA6061 matrix and B_4C particles is shown in the following **Tables 1** and **2**.

Table 1: Composition of Al and B_4C used as matrix material (w/%)[3]

Element	Mg	Si	Cu	Fe	Mn	Cr	Balance
AA6061	1.01	1.07	0.35	0.25	0.05	0.12	

Table 2: Composition of B_4C particles used as reinforced material (w/%)[15]

Element	B+C	B	C	B_2O_3	Fe	Si
B_4C	94–98.5	74–79	17–24	0.1– 1.0	0.2– 0.5	0.1– 0.3

Thus, the process steps of the manufacturing procedure are summarized in the following points.

AA6061 aluminium alloy was machined by CNC machine and converted into chips with 1 mm size.

The scraps were degreased with acetone in an ultrasonic bath for 30 min. The process of cleaning removed the impurities of the coolant oil and other substances, in accordance with ASTM G131-96, which outlines the standard practice for cleaning materials by ultrasonic process.

- Drying: The drying oven was set at a 100 °C preheating temperature for 30 min to dry the chips.
- Mixing: The cleaned chips were mixed with 5 % B_4C powder by using the 3D-Mixer for 2 h at a speed of 35 min^{-1}.
- Billet Cold Compaction: The billet compaction was carried in a cylindrical die of 30 mm diameter and 80 mm length under a pressing force of 490.33 kN.

- After the extrusion process, the products were cut into several specimens and subjected to ECAP for four passes, which is the saturation level where no further increases of the material properties were observed.
- The hot ECAP process was completed with the use of cold press hydraulic machine, ECAP die, heaters (cartridge type), thermocouples, and a data-acquisition system. The capacity of the cold-press machine is 490.33 kN. The ECAP die consisted of two parts with a channel sized 12 mm × 12 mm. A K-type thermocouple with a diameter of 3 mm was used to measure the temperature. The ECAP die has an inner angle (φ) of 90° and an outer angle (Ψ) of 20°. Details of the setup are shown in **Figure 2**.
- The temperature of the ECAP die was set to 300 °C by four heating rods, fixed at different locations. Five K-type thermocouples fixed to specific locations on the ECAP die were used to record the temperature distribution within the die. The thermocouples were connected to a TC-08 data logger to provide temperature feedback for the controller. The ECAP die was encapsulated by a special insulator to retain the heat in the die. In this method, the pressing velocity was set to 7 mm/min.
- Before the ECAP process, each feedstock was annealed at 550 °C for 1 h to increase its ductility and deformability. A small amount of MOLYKOTE lubricant was applied in the inner die to reduce the friction. The feedstock was then inserted in the ECAP die and pushed by the ram. The hot ECAP process was carried out in four passes to obtain the optimum results. The part produced from the hot ECAP process could either be used as a final product or sent for the secondary processes to obtain net-shape products.
- The samples extruded from the hot ECAP die were tested for tensile and microhardness tests. For the tensile test, the parts extruded from the die were machined according to ASTM E8 – E8M standard to produce the dog-bone shaped samples. Then, the tensile test was carried out with an initial strain rate of 2.53×10^{-3} s^{-1} and pulled to failure at room temperature. The microhardness measurement was carried out using a Shimadzu Microhardness Tester with a Knoop indenter, 25 g load and indent time of 15 s. The standard utilized was ASTM E92 – 82.
- Performing the Atomic force microscopy (AFM) and Scanning Electron Microscopy (SEM) to investigate the reinforced particles distribution, chip boundaries and measuring the grain size of the samples.

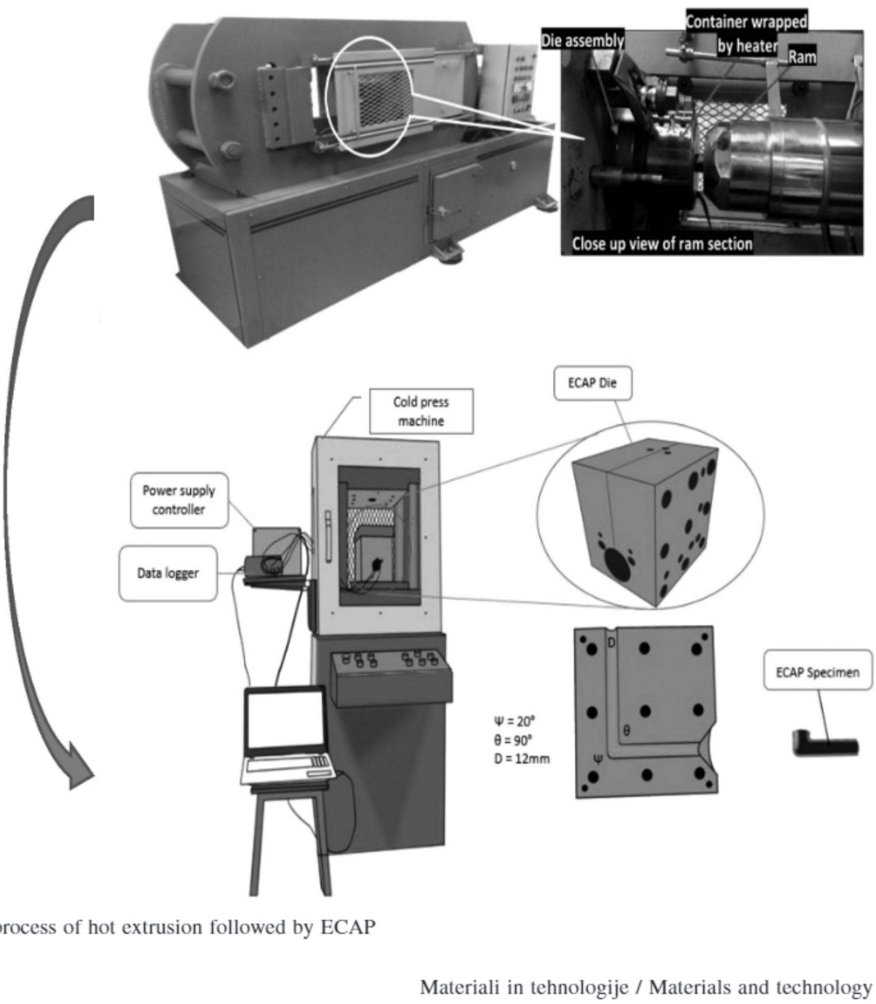

Figure 2: Combined process of hot extrusion followed by ECAP

4 RESULTS AND DISCUSSIONS

4.1 Tensile strength

The tensile strength of the Al chips and Al–B_4C composites made from both extrusion and extrusion followed by ECAP methods with multiple passes, the investigated findings verified the original materials of T6-AA6061. Seven samples were prepared of the extruded ECAP rods containing 5 φ/%. B_4C particles and 550 °C processing temperature. **Figure 3** shows increases of all the ECAP samples with an increasing number of passes as a result of work hardening.[6] The tensile strength values were 254.36 MPa after four passes, 291.087 MPa after four passes with heat treatment, 189.25 MPa after one pass, 222.25 after two passes, and 227.44 MPa after three passes. Amongst the samples were recycled Al chips and Al–B_4C composites that were subjected to the extrusion process, which had a tensile strength of 189.93 MPa and 219.86 MPa, respectively. This result proves the material enhancement of the MMC. The tensile strengths of the Al–B_4C samples are related to the contents of the particles, the diameter of the B_4C particles, and optimized processing parameters of both hot extrusion and hot ECAP.23 As well as the rapid hardening strain of the multiple numbers of passes. The strength of recycled MMC-based composites increased because of the work hardening and extensive strain softening of the ECAP passes. The UTS of ECAP sample strengthening effects could be attributed to two reasons: grain refinement and the broken phase's dispersion. The observations show that a significant increase of the 4th pass by 29 % compared to the 1st pass of the same specimen and reached up to 53 % for the heat-treated sample.

4.2 Effects of B_4C particles on hardness

Hardness is a variable dependent on the plasticity, elasticity, strength, and ductility of the samples. **Figure 4** shows that the hardness of samples made from extrusion followed by ECAP have higher values than the samples processed by hot extrusion only. The sample from the fourth pass presents the highest value with 94.78 HV compared to 68.65 HV for one pass, which proves that hardness progressively increases with a increasing number of passes.[22] In comparison, the hardness values of samples processed by hot extrusion for both recycled Al chips and Al–B_4C were 63.27 and 63.71 HV, respectively. The overall findings show that the pass four value is higher than all the tested samples, including the as-received Al. Hence, the passes were inversely proportional to the grain size and result in the hardness increment, the hot extrusion followed by ECAP samples have demonstrated to be significantly higher than those processed by hot extrusion only. The improvement showed the important increases of the 4th pass by 38 % compared to the 1st pass of the same sample and reached up to 51 % for the heat-treated sample.

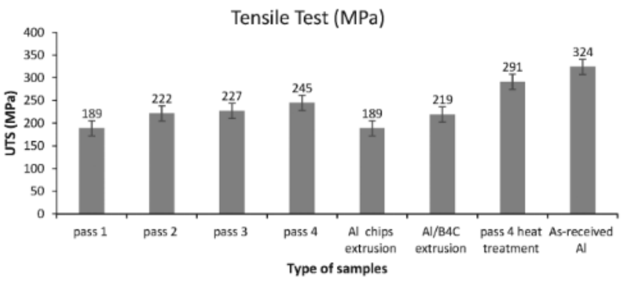

Figure 3: Tensile tests of recycled samples by hot extrusion and hot extrusion followed by ECAP

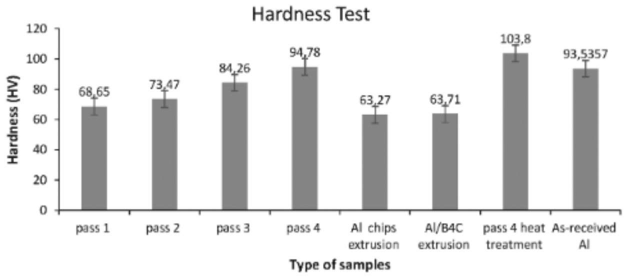

Figure 4: Hardness test of recycled samples by hot extrusion and hot extrusion followed by ECAP process

4.3 Effects of B_4C particles on density

The density of samples produced under different conditions were measured in the air and distilled water. The dimensions of each sample were (8 × 8 × 8) mm. The density was determined by using Archimedes' principle.[24]

$$\frac{1}{\rho_C}=\frac{w_f}{\rho_f}+\frac{w_m}{\rho_m} \tag{1}$$

where ρ is density, w is volume fraction, while m, f, and c are related to the composites and reinforcement. The percentage of pores was calculated from the difference between the theoretical and measured density values.[25]

Table 3: Density and pores of Al-5 φ/% B_4C composites

Materials	Theoretical Density (g/cm^3)	Experimental Density (g/cm^3)	Pores (%)
B_4C	2.52	–	–
As-receive Al	2.7	2.7	–
Al-5 φ/% B_4C	2.69	2.47	0.22

As shown in **Figure 5**, the density increased linearly with the number of passes. The sample from the first ECAP passes had a density of 2.64 kg/m^3 and increased to 2.71 kg/m^3 after the fourth pass. After pass four, no improvements were recorded. Al chips and Al–B_4C samples processed by hot extrusion had densities of 2.64 kg/m^3 and 2.63 kg/m^3, respectively. The sample from four passes is denser than the other samples due to the sample consolidation and the effects of cold compaction or hot pressings during the forming process on the materials bonding.[26] Furthermore, the samples from

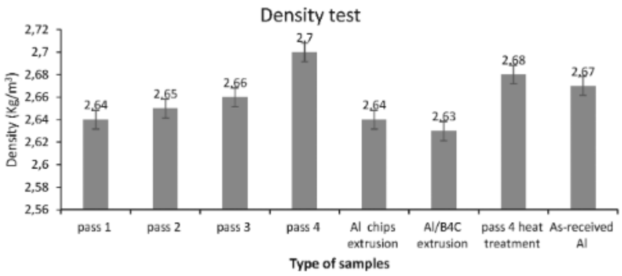

Figure 5: Density test of recycled samples by hot extrusion and hot extrusion followed by ECAP process

passes 1–4 became denser as a result of multiple passes of ECAP. Lokesh et al.[27] mentioned that the shear of plastic deformation of the hot ECAP process yields to a change of pores closure as the hydrostatic pressure is applied. The recycled samples underwent shear deformation because of the mechanical interlocking phenomenon and results in a density compaction that densify as close as to the theoretical density. Haghighi et al.[28] ensure that the porosity reduction of the hot ECAP sample undergoes as front plug shear deformation by imposing higher process level of strain and the overall 5 $\varphi/\%$. B_4C/Al justifications based recycled composites, which is harder due to reinforced powder particles.

4.4 Scanning Electron Microscopy (SEM)

The B_4C particles and damage mechanism for the prepared composites of the hot extruded samples and extrusion followed by hot ECAP samples with multiple passes were investigated and compared with the as-received AA6061.[12] **Figure 6** shows the microstructure of the reinforced samples with B_4C particles, revealing a uniform distribution of particles in the MMC. The ceramic phase is dark, whereas the white phase is for the AA6061 metal matrix. Also, the distribution of the composite is influenced by good interfacial bonding between the B_4C particles and matrix along the grain boundaries. However, voids and pores could be seen on chip boundaries, which resulted from oxidation during the forming process.[29] The specimens produced by the combination of hot extrusion and hot ECAP, such as the pass 4 sample, showed no voids or cracks. This is confirmed by the density measurement, in which the sample from extrusion followed by four passes of ECAP had a density of 2.71 kg/m³. This value is higher than that of the as-received Al. So, the density measurement had a strong determination of the materials possible porosities and strong correlation with the porosity of the material. By using the SEM, the produced samples were compared to the as-received sample.

The samples from hot extrusion followed by ECAP had less agglomeration of particles and less grains size as shown by the AFM method than those processed by the extrusion method. Also, the grains were as close as or closer than the as-received AA6061. **Figures 6c** and **6f** show the chip boundaries in the samples from the extrusion process, while samples from extrusion followed

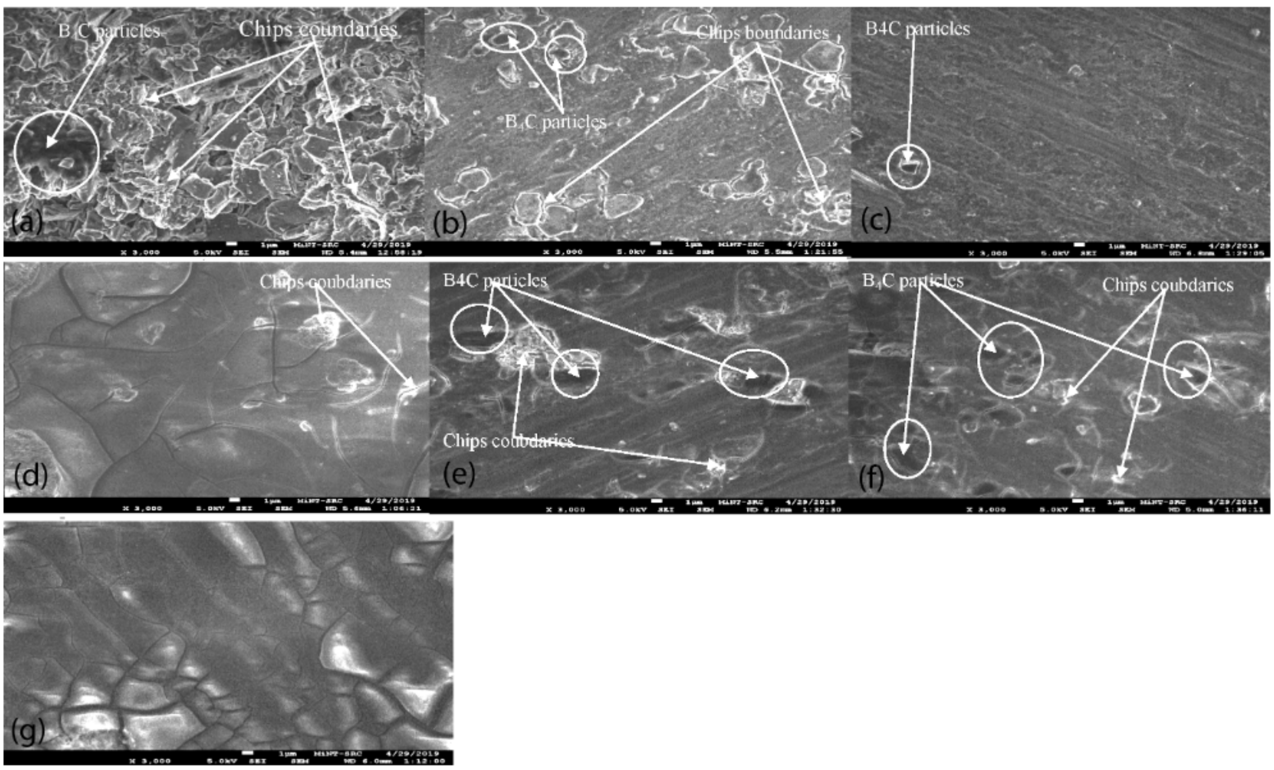

Figure 6: SEM for 5 $\varphi/\%$ B_4C/Al MMCs samples: a) pass 1, b) pass 2, c) pass 3, and d) pass 4, e) Al-chips, f) Al-B_4C and g) As-received T6-AA6061

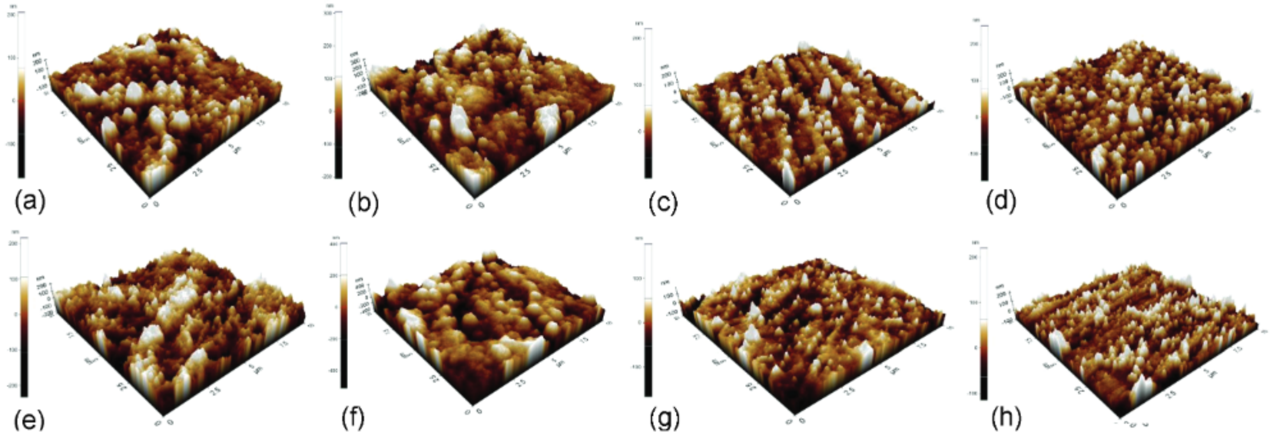

Figure 7: AFM topography images of the investigated samples: a) Pass 1, b) Pass 2, c) Pass 3, d) Pass 4, e) Al- chips, f) Al – B_4C g) Pass 4 – heat treated AL, h) as-received

by ECAP had good and homogenous microstructure with a good improvement in the B_4C particle distribution (**Figure 6** (Pass 4)). Pass 3 and Pass 4 illustrate the distribution of particles after hot ECAP, which had changes of the distributed orientations. The SEM analysis confirms that the ECAP process did not cause fractures in the samples.

AFM is a method that measures the morphology of the micron to nanoparticles of thin-film surfaces. The thin-film surfaces study the polycrystalline structure and grain size. However, the shapes are determining the materials grains radius and length.[30] The reported grains in **Table 4** are showing minimised values after each pass from pass 1 to pass 4. Islamgaliev et al.[31] proposed that the ECAP process led to improved materials strength properties as the results of gain refinement by hot extrusion followed by hot ECAP process. The **Table 4** below shows the measured mean grains of the investigated samples. The effects of the forming process on the materials characteristics, shape, surface structure let to have more soft and tin materials surfaces as mentioned by Attila et al.[32]

Table 4: Mean grain size

Samples	Mean grain size (µm)
Pass 1	0.29
Pass 2	0.27
Pass 3	0.26
Pass 4	0.25
Al-Chips	2.27
Al-B_4C	2.26
Pass 4 heat treatment	0.25
As-receive	0.25

5 CONCLUSION

In summary, solid-state recyclings of AA6061 chips reinforced with B_4C particles were successfully carried out by hot extrusion followed by the hot ECAP process. The samples have good mechanical properties and microstructure. No remelting steps were employed, which is an advantage, as it contributes towards energy and material savings. The following conclusions can be drawn:

- The continuous temperature of hot ECAP process improved the specimen deformation mechanism and microstructure.
- Samples of the Al chips made with extrusion process have a lower UTS compared to Al reinforced with 5 φ/% B_4C particles.
- Further enhancement of samples made from extrusion followed by hot ECAP is related to the rapid hardening strain resulting from the multiple numbers of passes.
- The samples from extrusion followed by four passes of hot ECAP and heat treatment had the highest UTS of 291 MPa, compared to 324 MPa for the as-received AA6061 sample.
- The heat-treated samples were stronger, harder and denser than all samples, except for the density value of the 4th pass.
- The properties of the material produced with extrusion followed by ECAP could be enhanced by optimizing the experimental design parameters.
- The best mechanical and metallographic properties of the selected sample was 5 % B_4C/Al MMCs and produced by hot extrusion followed by four passes of hot ECAP heat treated.
- Further investigations of the heat treatment and performing fatigue tests are recommended by forming in the combined techniques of hot extrusion followed by hot ECAP of the recycled Al/B_4C composites.

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