Recycling of Al chips and Al chips composites using high-pressure torsion

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Abstract

Pure Al chip, Al chip-20% Al₂O₃, and Al chip-20% SiC samples were recycled using high-pressure torsion (HPT). Influence of the HPT processing on the feasibility of the consolidation, the microstructure evolution, and the hardness of pure Al chip, Al chip-20% Al₂O₃, and Al chip-20% SiC samples was investigated and compared with those of the as-received Al and HPTed Al solid samples. The HPT processing successfully produces approximately fully dense ultrafine-grained (UFG) microstructure Al and Al composite samples with relative densities ranged from 99.7%–98.3%. Moreover, HPTed recycled UFG Al chip, Al chip-20% Al₂O₃, and Al chip-20% SiC samples cost was lower by 11%–2678% than that of Al and Al composites samples produced using micro and nanopowders processed by HPT or powder metallurgy (PM). The HPT processing of the Al chip and Al chip composites samples effectively refine and fragmented the Al matrix and reinforcement particles, and so the hardness obviously increases.

1. Introduction

With their excellent physical and mechanical properties, aluminum and its alloys are used extensively to produce many products, from food cans to transportation means and airplanes. Aluminum is produced from its ores or the melting of Al scraps [1]. However, the extraction of one ton from Al from its ores needs about ten times of power required to produce 1 ton of steel [2]. The recent world agreement about global warming, high-power consumption, and CO₂ emission limits the process of producing Al extraction from its ores. So, the production of Al was directed to depend more on Al scrap recycling [3]. Nowadays, 26% of aluminum world production is obtained by Al scrap recycling, and that amount will increase up to 50% soon [3].

Most of the recycled Al products are produced by traditional recycling techniques (melting of scrap and then casting into semi-finish and finished products). Unfortunately, the conventional recycling techniques have different disadvantages such as high power consumption, losing around 46%–48% of the Al scrap during the casting process [4, 5], and emission of harmful gases (mainly CO₂) to humans and the environment due to the burning of the fuel during the scrap re-melting process [6]. Therefore, the need for a new technique that can overcome the disadvantages of the conventional recycling techniques used in the recycling of the Al scrap was the motivation of different previous works [4–9]. A new nonconventional recycling technique of Al scrap was performed through cold compaction of the Al scrap or even Al composites followed by the hot extrusion and so-called solid-state recycling technique to obtain further energy saving [4–9]. Therefore, Al scrap can be recycled with a high degree of power saving and a low pollution level.

The metal cutting process is one of the Al and Al alloys scrap’s primary resources, which produces about 5% of the Al and Al alloys’ overall scrap amount in the world. The machining process produces scrap that so-called the chip that comes from removing the excess material to produce semi-finish and finished products. Interestingly, the machining chips have unique features relative to the other types of scrap types. The chip has nanometer or (UFG) size microstructures because of the shear strain imposed in the machining process [10–12]. Therefore, recycling of Al and Al alloys chips using the conventional (casting) or nonconventional (solid-state)
techniques under high temperature contributes to the grain growth of the recycled chips [7]. Therefore, the need for a new recycling technique that can save the chip nanometer or UFG grain size microstructures of the chip or even introduce a further refinement of the chip grain size is still required.

Severe plastic deformation (SPD) processes can be considered the most effective recycling metal chips method [6]. However, the equal channel angular pressing (ECAP) and high-pressure torsion (HPT) processes still the most popular processes among the various SPD methods [13, 14] used in the recycling of metal chips. Interestingly, most of the previous works related to the metals chip’s recycling process using ECAP have been performed under high temperature. The Al AA6060 alloy and pure Al chips were consolidated using ECAP and a combination of ECAP and extrusion under high temperature up to 590 °C and produce fully dense samples with superior mechanical properties [15, 16]. Nevertheless, the metal chips’ consolidation under high temperature produced large grain sizes [15].

On the other hand, metal chips recycling using the HPT process can be considered more effective in conserving the metal chips’ fine-grain microstructure due to the low recycling temperature. HPT processing of Cu and different Al alloys chips was performed under room temperature (RT) and a temperature that did not exceed 300 °C [12, 17]. More recent HPT was also used effectively in producing magnesium chip–alumina composite [18]. Therefore, using HPT processing can be considered a promising method in recycling metal chips and metal chip composite because of the significantly high strain of the HPT process relative to the ECAP [13, 14]. Therefore, the high strain imposed during the metal chips’ HPT processing under RT conserved the chip grain size or even contributed to a further decrease in recycled chip sample grain size [12, 17]. However, with the promising results of the chip and chip composites recycling using the HPT process, there is an apparent lack in previous works related to this topic. Therefore, a further investigation about the HPT processing efficiency in recycling pure Al chip and pure Al chip composites with comparable or superior properties relative to pure Al solid samples is still required.

The objectives of the present work can be listed as follows:

1. Study the feasibility of using the HPT processing to consolidate pure Al chip, pure Al chip–Al2O3, and pure Al chip–SiC composites into fully dense samples.
2. Investigate HPT processing’s influence on the microstructure evolution of pure Al chips, pure Al chip–Al2O3, and pure Al chip–SiC samples (the grain size refinement and ceramic particles fragmentation).
3. Study the HPT processing influence on the hardness of the recycled pure Al chips, pure Al chip–Al2O3, and pure Al chip–SiC samples.

2. Experimental work

Pure aluminum AA1080 ingots provided by ALU Misr Company were melted and cast into cylindrical shape samples with a diameter of 3 cm and length of 25 cm were used in the present work. The AA1080 samples were dry turned (to avoid any contamination of the produced chip if lubrication used) by the center lathe machine, as shown in figure 1(a). The turning process performed using a single-point cutting tool with a rake, clearance, tool angles, and tool nose radius of 40 °, 6 °, 44 °, 0.9 mm, respectively. The cutting process was performed under a speed, feed, and depth of cut of 120 m min⁻¹, 0.21 mm/rev, and 5 mm. The cutting conditions used in the current study were selected based on those recommended by ASM standard of the turning of pure Al [19]. The turning process produced a continuous chip with a length ranges from 2–35 mm, as indicated in figure 1(b). The turning process with the selected cutting tool geometry occurs under a shear angle of 13° according to the following equation (1) [20].

\[
\tan \varphi = \left( \frac{r \cos \alpha}{1 - r \sin \alpha} \right)
\]

Where  and  are the rake angle, and the chip thickness ratio, the value of  can be obtained from equation (2) [20].

\[
r = \frac{t_0}{t_c}
\]

Where  and  are the chip thicknesses before and after the cutting process, respectively. In the current study, the values of chip thicknesses before and after the cutting process were 0.3 and 1.2 mm, respectively. So, the chip thickness ratio  value in the present work is 0.25. Therefore, based on the values of different variables of equation (3) [20], the turning process induces a shear strain of 3.83.
The machined continuous chip was cleaned by acetone in an ultrasonic bath for 1 h. The chip was then comminuted into fine particles with an average size of 85 μm and particle size range of 30.5–212.2 μm, as shown in Figure 1. Schematic diagram of the pure Al-chip production and commination process.

\[
\gamma = \tan(\varnothing - \alpha) + \cot \varnothing
\]  

(3)

The machined continuous chip was cleaned by acetone in an ultrasonic bath for 1 h. The chip was then comminuted into fine particles with an average size of 85 μm and particle size range of 30.5–212.2 μm, as shown
The comminuted chip was then mixed with flake-shaped Al₂O₃ and SiC powders with the average particle sizes of 41.4 and 25.5 μm and particle size range of 8.9–82.9 μm and 7.5–55.5 μm, as shown in figures 2(a), (b), and (c), respectively. The mixtures of the Al chip-Al₂O₃ and Al chip-SiC were further mixed using a roller mixer for 10 h to obtain Al chip–20% Al₂O₃ and Al chip–20% SiC (Volume% of Al₂O₃ and SiC) composites powders, as shown in figure 2(d) and (e). The Al chip–20% Al₂O₃ composition and Al chip–20% SiC composites powders were proved by the x-ray spectroscopy (EDS) analysis, as shown in figures 2(f) and (g).

The comminuted chip and different composites mixtures were cold compacted under a pressure of 500 MPa into disc-shaped samples with a 10 mm diameter and a thickness of 2.5 mm, as shown in figure 2(h). The cold compacted pure Al chip, chip composite, and pure solid Al alloy samples were carefully ground. Then the cold compacted pure Al chip, chip composite, and pure solid Al ground samples were HPTed for 20 revolutions under an applied pressure of 9 GPa, and a speed of 1 rpm at (RT) using a semi-constrained HPT die with 10 mm diameter [12, 14], as shown in figures 2(i) and (j).

The as-received Al, HPTed Al chip, and HPTed chip composites samples were ground and polished up to shiny surfaces. The experimental and relative densities of the different samples were measured. Moreover, the mirror-like surface samples were further polished using mixtures of colloidal silica and ethanol for one-hour to investigate the different samples’ microstructure. The microstructure observations of the as-received Al, HPTed as-received Al, HPTed Al chip, HPTed chip composites samples were performed using an FE-SEM with an electron backscatter diffraction (EBSD). The EBSD was used to characterize the Al matrix grains size and grain
boundaries misorientation in each sample. On the other hand, the tracing of the Al₂O₃ and SiC particles distribution and fragmentation after the HPT processing performed by field emission scanning electron microscope (FE-SEM; model JEOL JSM-6330F, JEOL, Japan). Therefore, the HPTed Al chip-20% Al₂O₃ and Al chip-20% SiC samples were prepared in the same way used in preparing the EBSD samples, then further etched using Keller’s reagent.

The Vickers microhardness of the different samples was measured using a Mitutoyo microhardness tester under an applied load of 100 gf and a dwell time of 15 s. The microhardness measurements were carried out along the sample diameter with a spacing of 0.5 mm between each two-measurement points. The microhardness measurements were obtained through 5 different diameters, and the average value of the five microhardness measurements in the same position was used and plotted. The standard deviation of the microhardness measurements was calculated to assess the deformation inhomogeneity index.

3. Results and discussion

3.1. Density and cost of the recycled samples

3.1.1. Experimental and relative density results

Figure 3 shows the experimental and relative densities of as-received Al, HPTed Al solid, HPTed chip Al, HPTed Al chip-20% Al₂O₃, and HPTed Al chip-20% SiC samples. The Al as-received and HPTed Al solid samples experimental and relative densities are equal with relative densities of 100%, as indicates in figure 3. On the other hand, the HPTed Al chip sample has a relative density of 99.7%, according to equation (4). Considering the theoretical and experimental densities of HPTed Al chip sample are 2.7 and 2.68 g cm⁻³.

\[
\rho_{\text{rel}} = \left( \frac{\rho_{\text{exp}}}{\rho_{\text{theo}}} \right) \times 100
\]  

Where \(\rho_{\text{exp}}\) and \(\rho_{\text{theo}}\) are the values of the sample’s relative, experimental, and theoretical densities. The HPT Al chip sample’s relative density indicated that HPT processing effectively consolidated Al chip into approximately fully dense samples with void content, not more than 0.3%. The HPTed Al sample’s relative density was near or even higher than those of Al powder consolidated by HPT of 99.5%–99.99% under a pressure of 1.5–6 GPa for 6–50 revolutions [21–23]. Although different, other solid-state recycling methods can provide approximately fully dense Al, and Al alloys recycled samples using cold compaction followed by sintering, hot extrusion, and ECAP [8, 15, 16]. Nevertheless, it must consider that recycling of the AlMg₂, AA6060, and commercially pure Al samples was performed under high temperature ranged from 200 °C–550 °C [8, 15, 16]. Therefore, the HPT of the Al pure chip effectively produces fully dense samples without any needing for heating.
The HPTed Al chip sample density increased after the addition of the Al$_2$O$_3$ and SiC particles. The HPTed Al chip sample experimental density increased from 2.68 up to 2.9 and 2.77 g cm$^{-3}$ in the case of the HPTed Al chip-20% Al$_2$O$_3$ and HPTed Al chip-20% SiC samples. On the other hand, the Al chip-20% Al$_2$O$_3$ and HPTed Al chip-20% SiC samples’ theoretical densities were calculated depending on the rule of the mixture (5) [24].

$$\rho_{\text{theo composite}} = \sum (\rho_{\text{matrix}} V_{\text{matrix}} + \rho_{\text{reinforcement}} V_{\text{reinforcement}})$$  \hspace{1cm} (5)

Where $\rho_{\text{matrix}}$, $\rho_{\text{reinforcement}}$, $V_{\text{matrix}}$, and $V_{\text{reinforcement}}$ are the density and volume fractions of the Al matrix and the Al$_2$O$_3$ or SiC reinforcement.

Considering the Al matrix, Al$_2$O$_3$ and SiC densities are 2.7, 3.95, and 3.21 g cm$^{-3}$, respectively. Moreover, the Al matrix, Al$_2$O$_3$, and SiC volume fractions are 80, 20, and 20%, respectively. Depending on the values of the theoretical densities of the Al chip-20% Al$_2$O$_3$ and HPTed Al chip-20% SiC samples of 2.95 and 2.80 g cm$^{-3}$, it can be noted that the theoretical densities of the Al chip-20% Al$_2$O$_3$ and HPTed Al chip-20% SiC samples are 98.3 and 98.7%, as shown in figure 3.

The relative density of the HPTed Al chip-20% Al$_2$O$_3$ and HPTed Al chip-20% SiC samples in the present work of 98.3 and 98.7% were near, or even higher than those of HPTed Al powder-carbon nanotubes (CNT) and Al powder-10, 20 and 30% nano Al$_2$O$_3$ composites of 96%–98.4% [21–23]. Therefore, the HPT recycling of chip composite can be useful in producing approximately fully dense samples. Through comparing the present results with the relative density of HPTed Al-nano Al$_2$O$_3$ composites with volume fractions from 10 to 30% and Al$_2$O$_3$ particle size of 30 nm. It can be noted that the chip recycled composite samples reinforced with micro-size particles can be more effective in reducing the agglomeration of the reinforcement particles [22, 23] that contribute to improving the sample density. Interestingly this observation was also noted by comparing the relative density of the HPTed AlSiCu-5% SiC, Ti-18% Al$_2$O$_3$, and the density of Al-nano Al$_2$O$_3$ composites [22, 23, 25, 26]. The relative density of HPTed AlSiCu-5% SiC, Ti-18% Al$_2$O$_3$, reinforced with an initial particle size of SiC and Al$_2$O$_3$ of 53 and 1 μm of 99.4 and 100% [25, 26] were also higher than those of the HPTed Al-nano Al$_2$O$_3$ composites [22, 23].

The HPT processing’s effectiveness in recycling Al chip and Al chip composites into approximately fully dense samples was also confirmed through the cold compacted samples’ microstructure observation. The cold compacted Al chip, Al chip-20% Al$_2$O$_3$, and Al chip-20% SiC samples microstructure are shown in figures 4(a)–(c). The cold compacted Al chip and Al chip composites’ microstructure indicate large-sized pores between the chip particles or between chip particles and the reinforcement particles, as indicated by the arrows in figures 4(a)–(c). Therefore, the cold compaction under 500 MPa is not sufficient to obtain fully dense samples. This observation was consistent with that previously observed of Al scrap’s cold compaction with different sizes from 0.5–2 μm using a pressure of 360 MPa, which produces samples with a relative density of 90% [5]. Therefore, improving the density of the recycled Al chips and powders or their composites was obtained by applying sintering and sintering followed by hot extrusion under temperatures up to 650 °C [7, 8, 27–29]. Therefore, HPT recycling of the Al chip and Al chip composite at (RT) without heating is an effective and promising solid-state recycling to produce fully dense recycled samples.

### 3.1.2. Cost of the recycled samples

Although the conventional and solid-state recycling techniques consume overall energy with a cost of US$ 0.36 and 0.018 per Kg of recycled Al scrap [5, 29], that is much lower than that of US$ 375 needed in the HPT recycling the 1 Kg of Al scrap. This difference can be explained by the small size of the HPT sample with an overall weight of 0.5 g that requires the machine with an operating time of 20 min for one sample and a total of 670 h to process 1 kg of Al scrap. However, it must consider the type of energy and the way by which it is applied. As the heating during the conventional and solid-state recycling techniques contributed to the direct emission of gases that increase environmental pollution. On the other hand, recycling using the HPT machine is performed at RT using electrical energy, which considers clean energy. Therefore, recycling using HPT processing can consider an environmentally friendly method more than conventional recycling (casting) and solid-state recycling (cold compaction followed by hot extrusion) methods.

In addition to the recycling method’s processing cost, the cost of labor and other tool and materials needed in each recycling method is required to obtain the total cost of each recycling method. The HPT machine needs only one operator, but in the conventional recycling method, a total of 26 laborers are needed to produce a final product [5]. On the other hand, the solid-state recycling method needs 10 laborers to perform the recycling, chemical cleaning, and commination processes (with daily salary not less than US$ 50 in all recycling methods) are needed [5]. Therefore, the HPT process’s labor cost is much lower than those required in the conventional and solid-state recycling methods. Moreover, the different steps in the conventional and solid-state recycling techniques cost around US$ 4.29 and 1.14 [5], but the HPT process did not need any other expense, just the chip commination that costs US$ 0.38 per 1 Kg of Al chip [5].
The cost of the recycled material in the conventional, solid-state recycling techniques and HPT processing includes the Al scrap cost that can be considered US$ 1.8 for all cases. However, in the conventional and solid-state recycling techniques, there are other materials and tools such as lubrication, cutting tools, abrasive papers, and grinding wheels are needed for trimming and finishing processes with a cost of US$ 300–400. And so on, the approximate overall cost of recycling 1 Kg of Al scrap by conventional, solid-state, and HPT methods was calculated depending on the experimental work performed in the current research and previous studies [5, 29]. However, the cost of the mass production of the recycling of the Al chip using the conventional, solid-state, and HPT methods will be provided soon in future work.

The formation of a 1 Kg Al–20% Al2O3 and Al — 20% SiC composite samples needs the weights from the Al, Al2O3, and SiC indicated in table 2. Moreover, depending on the composite matrix and reinforcement materials, the cost of the formed composites will be varied from one case to another one. Considering the price of 1 Kg of the Al chip, Al micro, Al2O3 micro, and SiC micro, Al2O3 nano, and SiC nanopowders of US$ 1.8, 50, 1170, 225,
Table 2. Weight of the Al, Al2O3, and SiC powders needed in the fabrication of 1 Kg sample of Al-20% Al2O3 and Al-20% SiC composites.

| Type of composite | Weight of Al(g) | Weight of Al2O3 (g) | Weight of SiC (g) |
|-------------------|----------------|---------------------|------------------|
| Al-20% Al2O3      | 732            | 268                 | —                |
| Al-20% SiC        | 771            | —                   | 229              |

Table 3. Cost of the materials used in fabrication of 1 Kg of different Al, Al-20% Al2O3, and Al-20% SiC samples using PM and HPT methods.

| The samples type | PM (cold compaction followed by sintering or sintering combined with hot extrusion) (US$) | HPT recycling (US$) |
|------------------|------------------------------------------------------------------------------------------------|---------------------|
| Recycled Al chip | —                                                                                          | 1.8                 |
| Recycled Al chip-20% Al2O3 | —                                                        | 314.9                 |
| Recycled Al chip-20% SiC | —                                                        | 52.9                 |
| Micro Al          | 50                                                        | 50                   |
| Micro Al-20% Micro Al2O3 | 350.2                                                    | 350.2                 |
| Micro Al-20% Micro SiC | 90.1                                                    | 90.1                 |
| Micro Al-20% nano Al2O3 | 1954.2                                                   | 1954.2                 |
| Micro Al-20% nano SiC | 1447.9                                                   | 1447.9                 |

7155.3, and 6154.2, respectively (depending on the prices of the different materials on Sigma-Aldrich company site on the internet). The cost of the material used in the formation of 1 Kg of Al, Al − 20% Al2O3, and Al − 20% SiC samples using HPT through the chip recycling and HPT or PM using micro and nanopowders can be summarized in table 3. It can be observe that the cost of the consolidated Al chip and Al chip composites using the processed by HPT is cheaper by 11%−2678% and 521%−2678% than that of Al and Al composites process using Al, Al2O3, and SiC micro and nanopowders processed by HPT and PM. Therefore, the HPT processing can be considered an effective recycling technique in producing approximately fully dense Al and Al composites with a much lower cost than those processed based on Al, Al2O3, and SiC powder [22, 23, 27, 28].

3.2. Microstructure evolution

The EBSD color-coded orientation and grain boundaries maps images (high angle >15° and low angle <15° shown by blue line and red colors) of as-received Al are shown in figures 3(a) and (b). The as-received Al sample has equiaxed grains with an average grain size of 414 μm with high angle grain boundaries. Moreover, energy dispersive x-ray spectroscopy (EDS) shown in figure 5(c) did not indicate the presence of any oxygen.

The grains of the as-received Al sample were refined after the turning process, as shown in figure 6(a). The chip sample has an elongated grain with a grain size range of 3.9−0.13 μm and an average grain size of 0.97 μm with approximately 70% of high angle grain boundaries (HAGBs), as shown in figures 6(a) and (b) and previously noted in the case of the chip of the AlSi8Cu3 alloy and pure iron [12, 30]. The Al chip grain size decrease after the turning process is due to the high strain imposed during the machining. The imposed strain during the turning process of 2.2 calculated based on the shear strain obtained from equation (3) of 3.8 [20]. The imposed strain on the turned Al sample is equal to that imposed through 2 passes of ECAP, according to Y. Iwahashi’s equation [31]. Similar results of the formation of UFG microstructures were noted after the machining of different aluminum alloys [10–12]. Reverse to that observed in the as-received Al sample oxygen, with a percentage of 2.8%, was found in the chip sample’s EDS analysis, as shown in figure 6(c). The presence of oxygen in the chip sample can be explained by the oxidation that occurred during the machining process due to the high temperature generating in the turning process.

The microstructure of HPTed Al solid, Al chip, Al chip–20% Al2O3, and Al chip–20% SiC samples are shown in figures 7 and 8. The HPTed Al solid, Al chip, Al chip–20% Al2O3, and Al chip–20% SiC samples consist of approximately equiaxed grain. Therefore the HPT processing of the Al chip, Al chip–20% Al2O3, and HPTed Al chip–20% SiC samples effectively transformed the microstructure of the initial chip with elongated grains into equiaxed grain microstructure. The Al solid as-received sample average grain size of 414 μm decreased down to 0.61 μm and a grain size range of 1.25−0.05 μm after HPT processing, as shown in figure 7(a). Moreover, the HPTed Al solid sample has a percent of 79.5% of HAGBs and an average grain boundary misorientation angle of 32.57°, as shown in figure 7(b). The microstructure features and grain size of the HPTed Al solid sample were close to that of solid Al-1080, and Al 99.7HPTed samples with average grain sizes of 0.58 and 0.8 μm [32, 33].
The average grain size of the chip sample decrease to 0.54 μm with a grain size range of 2.39–0.07 μm after the HPT processing, as shown in figure 7(c). The HPTed Al chip sample has a percent of 80.9% of HAGBs and an average grain boundary misorientation angle of 33.42°, as shown in figure 7(d). The average grain size of the HPTed chip samples was smaller than those of different HPTed pure solid Al samples in the present and previous works [32, 33]. Moreover, the grain size of the HPTed Al chip was very near that of 0.5 μm of the HPTed Al powder disc shape samples [21, 23]. However, the combination of ball milling and HPT processing of Al powered is capable of producing an Al sample with a grain size of 0.16 μm [22].

Through the microstructure observations of the HPTed processed solid, powder, and chip pure Al samples in the current and previous works [21–23, 33]. It can be noted that the HPT of Al powders and chips can produce microstructures with smaller grain sizes and higher percentages of HAGBs than the HPTed solid samples. The smaller grain size with a high percentage of HAGBs in the case of the HPTed Al powder and chip is due to the presence of the oxide layer in the surface of the processed Al powders and chips, as indicated by the EDS analysis shown in figure 6(f) and previously noted [21]. The oxide particles in the form of alumina particles are good sites that can hinder the dislocation motion, and so the dislocations are accumulated in those sites. The dislocation accumulation then contributed to the formation of subgrain boundaries. Furthermore, due to the high strain imposed in the HPT process, enormous numbers of dislocations are generated, and so the combined interaction
of dislocations becomes enhanced, and the subgrain boundaries further evolve into grain boundaries with HAGBs.

The HPTed Al chip-20% Al₂O₃ and Al chip-20% SiC composites microstructures are shown in figure 8(a)–(d). The microstructure of the Al matrix of both composites was a mixture of micro, UFG, and nano grain sizes as that observed in the HPTed Al solid and chip samples. The HPTed Al chip-20% Al₂O₃ and Al chip-20% SiC composites samples have average grain sizes of 0.44 and 0.24 μm and grain size range of 2.18–0.09 μm and 1.51–0.06 μm, respectively. The trimodal microstructure samples’ formation with a combination of different grain sizes (a mixture of micro, UFG and nano grain sizes) conserving a high ductility degree with reasonable high strength and hardness, as noted previously in HPT processing AA5083-B4C and Cu-SiC composites [34, 35]. The formation of trimodal microstructure occurred due to the dislocation density’s difference through the deformed material combined with the non-homogenized deformation during HPT processing. The good bonding between the Al matrix and both the Al₂O₃ and SiC after the HPT processing contributes to hampering the dislocation’s motion. Therefore, localized deformation and dislocation activities occur, and the grain size is refined to the nano or UFG sizes in regions of high dislocations density and remains with coarse sizes in the regions of low dislocation activities.

Figure 6. (a) Color-coded orientation map images, (b) Color-coded grain boundaries map images (high angle ≥15° blue line and low angle <15° red line), and (c) EDS analysis of Al chip sample.
Interestingly, the Al matrix average grain sizes and grain size ranges of the HPTed Al chip–20% Al₂O₃ and Al chip–20% SiC samples were smaller than those of the HPTed Al solid and chip samples. The smaller Al matrix average grain sizes and grain size range of the composites are due to the presence of Al₂O₃, SiC, and oxide (Al₂O₃ noted in the case of the chip) particles. Moreover, the presence of such hard particles increases the number of sites, those obstacles the dislocation motion. So the dislocations accumulated in those sites become more apparent. Therefore, the dislocation accumulation becomes higher, so the formation of subgrain boundaries that evolved into grain boundaries with HAGBs becomes faster than that in the case of the solid Al and Al chip.

Figure 7. Color-coded orientation map images and color-coded grain boundaries map images (high angle $\geq 15^\circ$ blue line and low angle $< 15^\circ$ red line) of (a)–(b) HPTed Al as-received, (c)–(d) HPTed Al chip samples.

Interestingly, the Al matrix average grain sizes and grain size ranges of the HPTed Al chip–20% Al₂O₃ and Al chip–20% SiC samples were smaller than those of the HPTed Al solid and chip samples. The smaller Al matrix average grain sizes and grain size range of the composites are due to the presence of Al₂O₃, SiC, and oxide (Al₂O₃ noted in the case of the chip) particles. Moreover, the presence of such hard particles increases the number of sites, those obstacles the dislocation motion. So the dislocations accumulated in those sites become more apparent. Therefore, the dislocation accumulation becomes higher, so the formation of subgrain boundaries that evolved into grain boundaries with HAGBs becomes faster than that in the case of the solid Al and Al chip.
samples. The HPTed chip-20% Al$_2$O$_3$ and Al chip-20% SiC composites samples have a percentage of HAGBs of 81.7 and 82.9% with average grain boundaries misorientation angles of 34.22 and 35.1°, respectively, as shown in figures 8(b) and (d). Therefore, the percentage of HAGBs and average grain boundaries misorientation angles of the HPTed Al chip composites were higher than those of the HPTed Al and Al chip samples. Those observations of the higher percentage of HAGBs and average grain boundaries misorientation angles of the HPTed Al chip composites confirmers the effect of the addition of ceramic particles and their fragmentation on the evolving grain boundaries into HAGBs.
The smaller grain size of the HPT of metal matrix composites relative to those of HPTed solid or powder metal samples was also previously noted \[21–23, 25, 26, 35\]. The Al matrix grain size of the HPTed Al-CNT and Al-Al₂O₃ with the average grain size of 0.1 and 0.16–0.48 μm were smaller than those of 0.5 and 1.2 μm of HPTed Al powder and solid samples \[21–23, 34\]. Moreover the grain size of the HPTed Cu-20% SiC, Ti-Al₂O₃, and Al-15%Si-2.5%Cu-0.5%Mg-5%SiC composites of 0.3, 0.1 and 0.06–0.07 μm were smaller than those of the HPTed Cu, Ti, and Al-15%Si-2.5%Cu-0.5%Mg solid and powder samples of 0.95, 0.15, and 0.1–0.3 μm \[25, 26, 35\]. Therefore, the previous results of obtaining smaller matrix grain size in the HPTed composites \[21–23, 25, 26, 35\] supports the present work results.

Although the Al₂O₃ and SiC particles in the cold compacted sample are still not fragmented, as shown in figures 4(b) and (c). However, Al₂O₃ and SiC particles were fragmented and redistributed after the HPT processing of the Al chip composites, as shown in figure 9. The particle size and ranges of the Al₂O₃ and SiC were decreased to 3.2–0.15 and 2.97–0.19 μm. Moreover, the average particle sizes of the Al₂O₃ and SiC reinforcements were decreased to 0.92 and 0.47 μm, as shown in figure 9. The fragmentation and redistribution of Al₂O₃ and SiC particles during the HPT processing occurs as follows. First, the high-applied pressure and strain of HPT processing fragment the Al₂O₃ and SiC particles. Consequently, the rotation during the torsion process redistributes the fragmented Al₂O₃ and SiC particles in the Al matrix.

The low fracture toughness of the Al₂O₃ and SiC particles’ can also explain their fragmentation during the HPT processing. The HPT process applied pressure and strain used in the present work are sufficient or even higher than the pressure and strain values used in the fragmentation of the Al₂O₃ and SiC during the HPT of different metal matrix composites as previously noted \[25, 26, 35\]. As the HPT processing in the present work was performed under a pressure of 9 GPa, which is higher than the critical pressure value noted in the previous studies \[25, 26, 35–37\] of 5 GPa that needed to the fragmentation of the hard reinforcement particles to ultrafine and nano-size particles, as shown in figure 9. Generally, the HPT processing of the metal matrix composites reinforced with micro-size particles can effectively fragment the reinforcement particles down to UFG particle size \[25, 26, 35–37\] under pressure equal to 5 GPa or more. Therefore, the HPT pressure and the number of
reinforcement particles, with a more noticeable effect of the pressure.

Through the comparison between the previous works [25, 26, 35–37], it can be noted that the pressure has a more noticeable effect on the reinforcement particles fragmentation process. The HPT using pressure with a value of 5 GPa or less can fragment the Al2O3 and SiC particles but to a limited degree, as the particle size still in the micro size [26, 36, 37]. However, HPT under pressure higher than 5 GPa has successfully fragmented the Al2O3 and SiC particles to UFG and nanoparticle particle size [25, 35]. So the HPT processing of Al chip-20% Al2O3 and Al chip-20% SiC composites in the present study can be considered efficient in producing UFG Al composites.

The SEM photomicrographs indicate the HPT processing’s effectiveness in improving the reinforcement particles distribution with the chip composite’s consolidation without apparent content voids, as shown in figure 9. The Al2O3 and SiC particles have a homogeneous distribution across the Al matrix, as shown in figures 9(b) and (d). The HPT processing’s effectiveness in improving the reinforcement particles distribution in the Al chip composite samples can also confirm by comparing the optical microscope micrographs of the cold compacted Al chip composites samples shown in figures 4(b) and (c). The optical microscope micrographs of the cold compacted Al chip composites indicated the large voids with a high degree of the Al2O3 and SiC particles agglomeration. Therefore, the HPT processing can effectively produce approximately fully dense with a homogenized distribution of the reinforcement Al chip-20% Al2O3 and Al chip-20% SiC composites samples.

3.3. Microhardness results

Figure 10(a) shows hardness distribution curves along the diameter of the different samples’. The hardness distribution of the as-received sample was constant around its average hardness value of 27.6 Hv, as shown in figure 10(a). The Al as-received sample inhomogeneity index was 0.2 that confirms the constant distribution of the sample’s hardness in the form of a straight line, as shown in figure 10(b). Then hardness distribution becomes nonhomogeneous after the HPT processing, as hardness increased from the sample center to its edge along the sample diameter.

Hardness values increased from 61.5, 96, 151, and 175 Hv in the sample center to 71, 110, 179.1, and 203.8 Hv at the edge of the HPTed Al solid, Al chip Al chip-20% Al2O3, and Al chip-20% SiC samples, respectively, as shown in figure 10(a). The hardness distribution pattern of the HPTed sample results in a difference between the hardness values in the sample center and its edge of 9.5, 14, 28.1, and 28.8 Hv in the HPTed Al solid, Al chip Al chip-20% Al2O3, and Al chip-20% SiC samples, respectively. This kind of difference between hardness values produces a low hardness area in the sample center with a diameter of 0.5, 1, and 2 mm in the HPTed Al solid, Al chip, Al chip-20% Al2O3, and Al chip-20% SiC samples, respectively. The hardness increase from the HPTed sample’s center to the edge is due to the increase of the imposed strain from the center to the sample’s outer surface.

Similar observations of the hardness increase from the sample center to its edge were also observed after the HPT processing of solid, chip, powder, and composites samples of different metals [12, 18, 21–23, 25, 32, 33, 35]. Interestingly similar to that noted in the current study, the HPT processed solid samples have a smaller low hardness area in the sample center than that noted in the HPTed composites samples [18, 21–23, 25, 26, 35]. This observation can be explained by the hard reinforcement particles that hinder the deformation process with the lower imposed strain in the center relative to that at the sample edge. This observation can be proved by assessing the deformation inhomogeneity index in the form of the stander deviation of the hardness values in each case. The hardness inhomogeneity index was increased from 2.2 in the HPTed Al solid samples to 3.1, 9.5, and 9.6 in the HPTed Al chip, Al chip-20% Al2O3, and Al chip-20% SiC samples, respectively, as shown in figure 10(b). Therefore, the presence of the Al2O3 and the SiC particles decreases the deformation homogeneity of the samples. However, it can be noted that the composite samples in the present work have smaller low hardness areas in the sample centers relative to those noted in the previous works [18, 21–23]. The smaller low hardness area observed in the present work is due to the high applied pressure of 9 GPa for 20 revolutions used in the present study. Applying the current conditions contributes to reaching the grain refinement and particle fragmentation with smaller sizes than observed previously and so a higher degree of hardness distribution homogeneity is acquired.

The values of the average hardness of the different samples are shown in figure 10(b). The Al solid sample’s average hardness was increased from 27.6 to 67.8 Hv after the HPT. The hardness of the HPTed solid Al sample in the current study was very closed to those of the commercially pure Al 1080, Al 1070, and Al 1050 of 62.5, 61, and 65 Hv under the number of revolutions of 8–5 and applied pressure of 1–8 GPa [32, 33]. The increase of the hardness in solid Al sample after the HPT processing results from the grain refinement, as shown in the microstructure section through figure 7. According to the Hall–Petche relation shown in equation (6) [38, 39], the hardness of a material H is generally related to the grain size.
Where $d$ is the grain size, and $H_0$ and $K_H$ are constants. Moreover, the increase of the dislocation density also contribute to the increase of hardness $H$ and strength $\sigma$ after HPT processing according to the Taylor equation (7) [40, 41].

\[
\sigma = \sigma_0 + \frac{\alpha M G b \rho^2}{2}
\]

Where $\alpha$ is a constant, $G$ is the shear modulus, $b$ is the length of the Burgers vector of dislocation, $M$ is the Taylor factor, and $\rho$ is a dislocation density.

On the other hand, the HPTed Al chip, Al chip Al chip-20% $\text{Al}_2\text{O}_3$, and Al chip-20% SiC samples average hardness were higher by 55, 149.1, and 187.2% of the HPTed Al solid, respectively. The higher hardness of the HPTed Al chip sample relative to the HPTed Al solid sample can be explained by the presence of the $\text{Al}_2\text{O}_3$ particles, as shown in figure 6(c). Interestingly, the addition of the $\text{Al}_2\text{O}_3$ or SiC particles contributes to a further increase in the hardness of the HPTed Al chip-20% $\text{Al}_2\text{O}_3$, and Al chip-20% SiC composites over that of the
HPTed Al solid and chip samples. The higher hardness of the Al chip–20% Al2O3 and Al chip–20% SiC samples over that of the HPTed Al solid sample can be explained by the refinement and fragmentation of the Al, Al2O3, and SiC grains and particles, (as shown in figures 8 and 9), the high dislocation density, and the contribution of the fine Al2O3 and SiC particles in the obstruction of dislocation motion.

4. Conclusions

In the present work of Al pure chip and Al pure chip composites recycling, the following conclusions were obtained.

1. The HPT recycling of the pure Al chip, Al chip–20% Al2O3, and Al chip–20% SiC effectively produced approximately fully dense samples with relative densities ranging from 99.7%–98.3%.

2. The cost of the SPD processing of Al chip and Al chip composites using HPT can be considered comparable or even cheaper than the recycling cost using conventional and nonconventional methods. The HPT recycled UFG size Al, Al chip–20% Al2O3, and Al chip–20% SiC samples cost was less expensive by 11%–2678% than Al, and Al composites samples produced using micro and nanopowders processed by HPT or any other method.

3. The HPT processing of the Al chip and Al chip composites conserve the UFG microstructure of the chip with further refinement of the chip grain size and fragmentation of the Al2O3 and SiC particles. Therefore, HPT processing effectively produces UFG recycled bulk Al chip and Al chip composites with trimodal microstructure.

4. The HPT of Al chip–20% Al2O3 and Al chip–20% SiC composites effectively produced UFG Al composites with Al2O3 and SiC average particle sizes of 0.92 and 0.47 μm with Al matrix average grain sizes of 0.44 and 0.24 μm.

5. The HPTed Al solid, Al chip, Al chip–20% Al2O3, and Al chip–20% SiC samples have similar hardness distribution patterns with the increase of hardness from the sample center to its edge. Deformation inhomogeneity index based on the hardness distribution increased from 2.2 in the HPTed Al solid samples to 3.1, 9.5, and 9.6 in the case of the HPTed Al chip, Al chip–20% Al2O3, and Al chip–20% SiC samples.

6. The HPT recycling of the Al chip, Al chip–20% Al2O3, and Al chip–20% SiC increases the hardness by 55, 149, and 187.2% over that of the HPTed Al solid sample due to the Al grain refinement and Al2O3 and SiC particles fragmentation.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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