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Characterisation of Mechanical Properties, Damage Progression and Fracture Modes of Recycled Aluminium Alloys AA6061 Reinforced Alumina Oxide

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Abstract: Characterisation of the deformation behaviour of recycled aluminium alloy reinforced alumina oxide in terms of mechanical properties, damage progression and fracture mode is investigated via the Uniaxial Tensile Test in this manuscript. The tests are carried out at room temperature at strain rates of $6X10^{-3}s^{-1}$, $6X10^{-2}s^{-1}$, and $6X10^{-1}s^{-1}$. Stress-strain curves and microstructural analysis are used to examine tensile behaviour, damage progression, and fracture mechanism. The primary form of aluminium alloy AA6061 was compared to the stress-strain curve analysis. It has been observed that when the strain rate increases, the flow stress increases. Field-Emission Scanning Electron Microscopy (FESEM) is used to analyse the damage progression, including fracture mode and continued with a close review on voids quantity and size using Image J Tooling Software. The results show that increasing strain rate increases the number of voids. In general, the recycled AA6061 reinforced Alumina Oxide exhibits strain-rate dependency behaviour. In terms of the fracture mode, an apparent necking develops around the fracture area in primary specimen. Necking, on the other hand, is less apparent in the recycled specimen. Due to alumina characteristics (ceramic), which promote fracture when necking begins to occur. The ductility of the recycled specimen is degraded after going through the recycling operations. With an increase in strain rate, the flow stress and damage evolution become more pronounced. This work contributes valuable information that can be established in various engineering applications significantly to improve the existing recycling process.

Keywords: Reinforced recycled aluminium alloy; uniaxial tensile test; deformation behaviour; damage characteristic; fracture mode

1. Introduction

Aluminium is number three in the most abundant crust element in the earth after oxygen and silicon. Aluminium alloys AA6061 are widely used in various applications due to their availability, mechanical properties and effective corrosion resistance. However, the increasing demand for aluminium leads to a lack of production. The primary production of aluminium is produced from bauxite (ore), which is a high energy-consuming process and also contributes to pollution. Therefore, secondary production is one of the best solutions to this issue. Secondary production is a process by using recycled aluminium alloy and becomes a more crucial component of aluminium production. Producing recycle aluminium capable to save energy almost 95% compared to primary aluminium [1]. The primary production also generates solid waste such as red mud residue and mine waste. Thus, the increase in using recycled aluminium is very crucial from standpoint of ecology. The recycling of aluminium shows a positive trend as an awareness of the environment and becomes more important. In the European Union, most aluminium products are made from recycled

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raw material [2]. As stated by [3], recycled Aluminium is more economic and gives benefit to the environment compared to primary aluminium. The primary aluminium production requires ~45 kWh/kg of metal produced while secondary metal produced from recycled products requires only ~2.8 kWh/kg of metal produced. This translates about 1.72 × 1011kWh/y to an energy-saving for the year 2003. By using recycling aluminium, more energy can be saved and also reduce the use of natural resources [21].

Due to the increasing product demand and usage of recycled aluminium alloy every year, the manufacturer is required to introduce and explorer more advanced material capable of withstanding when subjected to specific force vectors and high temperature. In practice, metal matrix composite is a competent material globally and widely used in various industries due to its excellent mechanical properties [4,5,6]. Most users in the automotive and aerospace industries used metal matrix composite as a critical component. Metal matrix composites are made up of a lightweight metal alloy called a matrix that is reinforced with non-metallic particles (hard ceramics) such as Silica Carbide (SiC), Alumina Oxide (Al2O3) and Boron Carbide (B4C) to produce a good performance rather than the conventional alloy. Besides, metal matrix composite shows good mechanical and physical properties such as high specific modulus, strength, lightweight, good wear resistance and low thermal expansion.

Moreover, the quality of aluminium matrix composite was based on the varying constituent and fraction. The composite mentioned above has excellent combination qualities which cannot be challenged by others monolithic material [7]. The reinforcement Alumina Oxide (Al2O3) is mainly used as an excellent combination of aluminium alloy. Alumina was chemically inert and also can withstand a higher temperature compared to unreinforced aluminium alloy. The alumina particulates can be used to improve the mechanical properties of the composite [7,8,9]. Alumina showed a significantly strengthened composite. However, it might reduce the ductility and elongation due to its properties (ceramic), which promote fracture specimens during necking.

There are two main methods in recycling aluminium alloys that have been used and developed by many researchers which are conventional recycling and direct conversion recycling method. Figure 1 shows comparison between the conventional recycling and direct conversion recycling methods. As can be seen in this figure, the conventional method had many steps and produce a high amount of new scrap. During the re-melting process, the yield rate only achieved around 55% and the mechanical properties of this material were decreased due to the purity of the recycled ingots decreased [10]. Generally, direct conversion methods as shown in this figure are also recognized as solid-state recycling methods. This method is turning scrap or chip into new forms through severe plastic deformation (SPD) [10,11,12]. As can be seen in the figure, this method is relatively simple, small energy consumption produces a small amount of waste, hence, environmentally friendly [13]. The metal loss could be prevented by 20% due to the re-melting process is not included in this method [14]. This method also has the potential to enhance the mechanical properties of the material [15].

In recent years, many researchers have explored the recycling aluminium alloy process to give better results and improved properties of such material, including combination with reinforcement [10,12,14,16,17,18,19]. However, there is still a lack of complete characterization mechanical behaviour of this material from quasi-static to high strain rate. Although the best setting is achieved, it only considered a few characteristics. For instance, it only focuses on the ultimate tensile strength and elongation to failure without response information related to damage characteristics under different strain rates. Therefore, this research investigates the deformation behaviour, damage characteristics, including the fracture mode of recycled aluminium alloy reinforced Alumina Oxide (Al2O3) at different strain rates.

In this paper, the specimen is produced from recycled aluminium alloy (AA6061) and reinforced with Alumina Oxide (Al2O3) by using the hot extrusion solid-state recycling technique proposed by [20]. The Uniaxial Tensile test is used at different strain rates to examine the tensile behaviour. The setting and optimum method recycling process was also established. The mechanical performance of hot-pressed recycled Aluminium Alloy AA6061 was proved in the study by [21].The microstructure analyses are conducted using Field-Emission Scanning Electron Microscopy (FESEM) to review the damage characteristics including fracture mode of recycled reinforced specimen undergoing finite strain deformation. The ImageJ software was used to quantify the number and size of voids.

2. Specimen Preparations

Figure 2 showed that specimen preparation consists of chips preparation, hot press forging (direct recycling method) and heat treatment. For the chip preparation to produce a recycled specimen, the plate of Aluminium Alloy AA6061 is milled using a vertical-centred Nexus 410A-II CNC machine from MAZAK. Table 1 presents the chips milling parameter 2017 and has been proven to a better tensile strength [22, 23]. Using Ultrasonic Bathtub, the chips are cleaned with acetone solution and drying at 600C through the thermal oven for 30 minutes. Next, the chips are mixed with a constituent of 2.0wt% alumina Oxide in an SYL 3-Dimensional mixer at 35rpm for 30 minutes. For the hot press forging process, the chip is compact and press to form ASTM-E8 dog bone shape using the moulding process setting used by [23,24]. ASTM-E8 is specifically standard for the specimen tested at room temperature, as shown in Figure 3 and the detail described in Table 2. After completed the hot press forging process, the specimen immediately undergoing the heat treatment process. Next, the specimen quenching at a rate of 1000C/s. Again, to complete the heat treatment process, the specimen is heated at 1750C for 120minutes

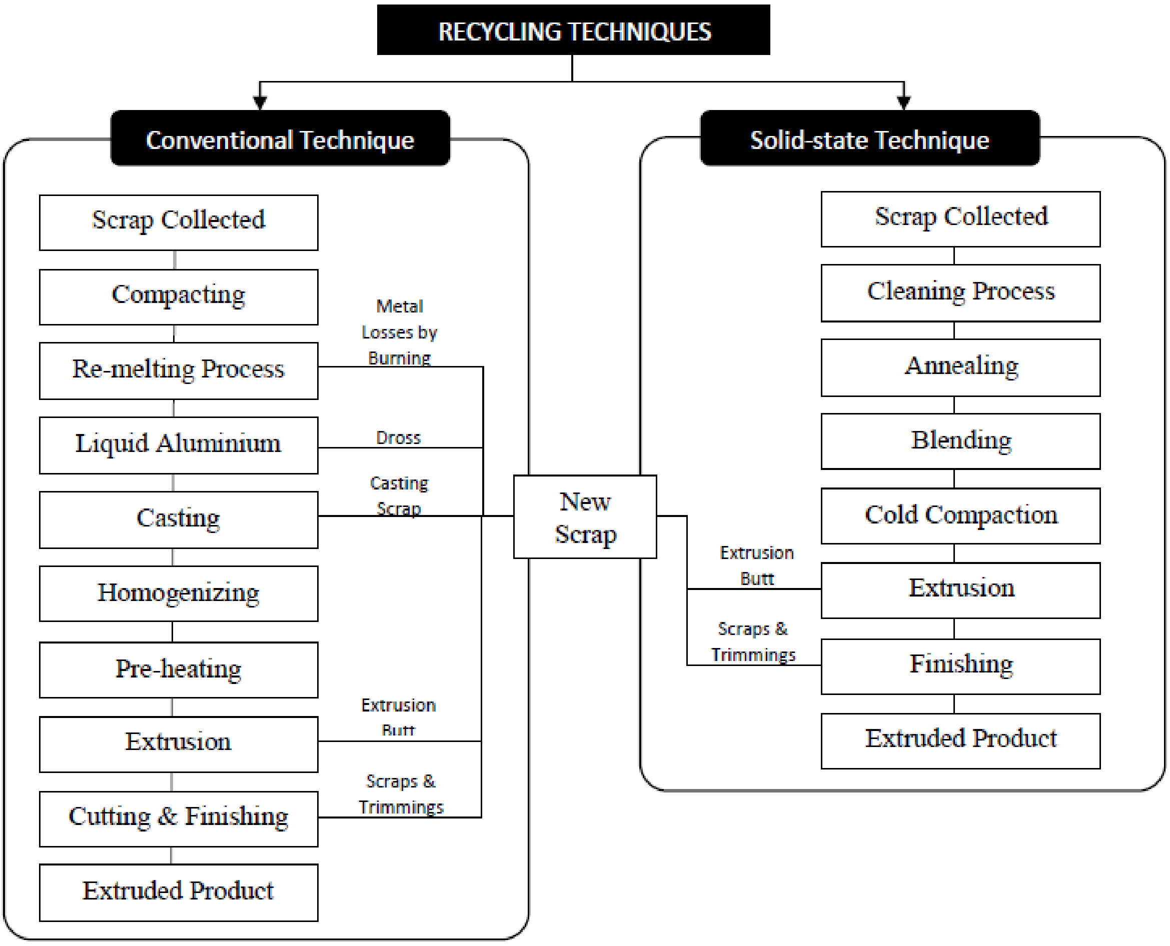


Fig. 1 - Process Conventional and direct conversion Recycling Method (Samuel, 2003)

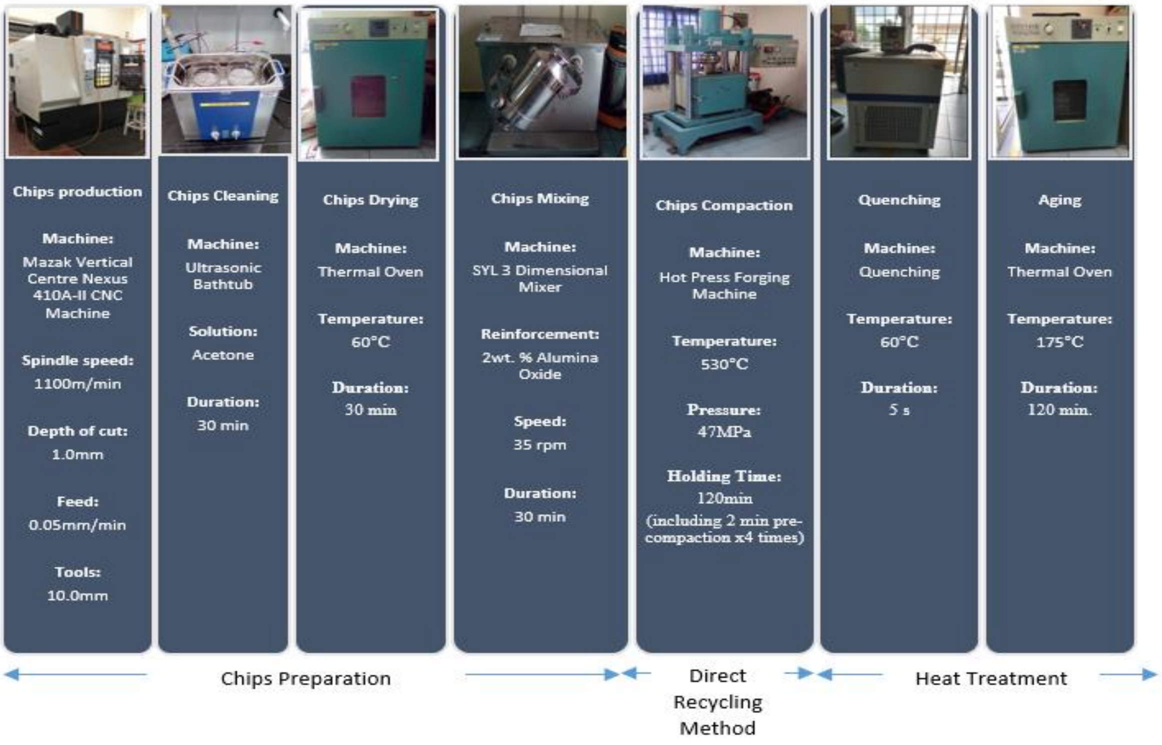
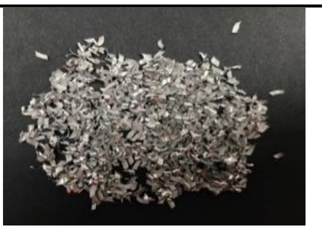


Fig. 2 - Specimen preparation process

Table 1 - Parameter of chip milling

Parameter	Value	Chips Geometry
Cutting speed, v	1100rpm	
Tool diameter	10mm	
Feed, f	0.05mm/tooth	
Depth of cut, DOC	1mm/min	

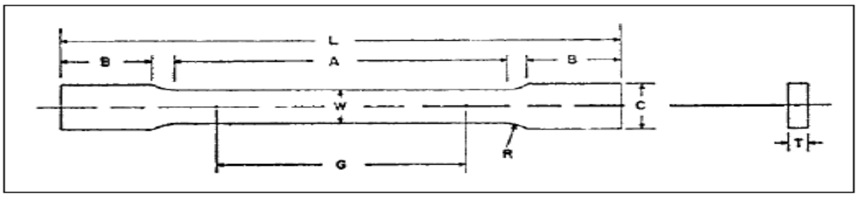


Fig. 3 - Cylindrical shaped specimen/ bullet

Table 2 - Detail dimension standard geometry of ASTM E8

Parameter	Size (mm)
Gauge length, (G)	25.00
Width, (W)	6.00
Thickness, (T)	1.00
Radius of fillet, (R)	6.00
Overall length, (L)	100.00
Length of reduced section, (A)	32.00
Length of the grip section, (B)	30.00
Width of the grip section, (C)	10.00

2.1 Experimental Implementation

The tensile test was carried out on a Zwick Roell Z030 Universal testing equipment. Table 3 shows the test matrix for this experiment. The settings are applied for the reinforced specimens tested at different strain rate $6X10^{-3}s^{-1}$, $6X10^{-2}s^{-1}$, and $6X10^{-1}s^{-1}$ and compared with the primary form of Aluminium Alloy AA6061. Each setting is tested four times to achieve a good result. As shown in Table 4, Energy-Dispersive X-ray Spectroscopy (EDS) is used to examine the chemical composition between the primary and recycled forms before beginning Tensile Test. Based on the findings, it shows that the recycled reinforced specimen has slightly less aluminium (Al), magnesium (Mg), and silicon (Si), but carbon (C) and oxygen (O) is higher than the primary form due to the appearance of damage such as micro-voids and micro-cracks. Strictly speaking, the study shows that the degradation of the recycled material corresponds to damage. As a result, the bonding behaviour of the chips is disrupted, resulting in a loss in strength and ductility.

3. Results and Discussion

The findings of the study are summarized in this section. This section consists of two parts, which are tensile behaviour and microstructural analysis. The first part of tensile behaviour is stress-strain analysis, where the mechanical properties of Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide were identified and compared with the primary form. For the next part, Field Emission Scanning Electron Microscopy (FESEM) is used to investigate damage progression and fracture mechanism. Next, Image J Tooling Software was used to continued analysis of damage progression with a close review on voids quantity and size.

Table 3 - Test matrix

Cross head Speed (mm/min)	Strain Rate (1/s)
0.15	$\mathbf{6 \times 10^{-3}}$
1.5	$\mathbf{6 \times 10^{-2}}$
15	$\mathbf{6 \times 10^{-1}}$

Table Error! No text of specified style in document. **- Chemical composition of primary aa6061and recycled aa6061 reinforced alumina oxide**

Element	Primary Aluminium Alloy AA6061		Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide	
	Weight (wt. %)	**Atomic (At. %)**	**Weight (wt. %)**	**Atomic (At. %)**
Al	86.60	76.64	80.37	66.42
Mg	0.83	0.81	0.63	0.58
Si	0.57	0.48	0.40	0.32
Fe	0.26	0.11	1.41	1.53
C	8.92	17.74	13.05	25.45
O	2.82	4.22	4.14	5.70
Total	100		100	

3.1 Mechanical Properties

The tensile result of Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide is compared with the primary form at different loading speed, and strain rates are summarised in Figure 4 and Table 5. In general, as seen in Figure 4, both materials are strain rate dependency. As the strain rate increase, the flow stress increases as well. As shown in Table 5, for the primary form, the young modulus, E; yield strength, and ultimate tensile strength are within the range of 64GPa - 66GPa, 246MPa - 252MPa and 305MPa- 308MPa, respectively. For the Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide, the young modulus, *E*; yield strength, and ultimate tensile strength are the ranges 62GPa - 64GPa, 225MPa – 233Mpa, and 235MPa – 254MPa, respectively. It can be observed that the value of these parameters increases for both materials due to the increment of strain rates. The Primary Aluminium Alloy AA6061 shows a higher value in all parameters from the comparison of both materials. The differences are not significant and almost identical in all parameters except the ultimate tensile strength due to the good distribution of reinforcement in the recycled specimen. Generally, a significant increase in dislocation density across the composite was obtained when reinforcement particles were introduced into Aluminium alloy AA6061. Besides that, the overall elongation for both materials may be seen in Figure 5. From the observation, the total elongation to failure of Recycle Aluminium Alloy Reinforced Alumina Oxide showed significant differences, compared to the Primary Aluminium Alloy AA6061. This condition related to ductility aspect of recycled material and due to reinforcement by alumina oxide, where alumina was a ceramic particle with brittle material properties. The weaker composite network also impacts the formability of specimens undergoing finite strain plastic deformation, resulting in a decrease in failure elongation. Alumina was a vital component in reducing the composite's flexibility and elongation. Mixing with the ceramic particles will increase the brittle of Aluminium and the rate of fracturing, which leads to a reduction in particle size. As a result, due to a poor plastic deformation compared to Primary Aluminium Alloy AA6061, the Recycle Aluminium Alloy Reinforced Alumina Oxide is not appropriate for absorbing energy applications.

Table 5 - Summary of Mechanical Properties

Materials	Loading Speed (mm/min)	Strain Rate	Modulus Young, E (GPa)	Yield Strength (MPa)	UTS (MPa)	Total Elongation (%)
Primary Aluminium Alloy AA6061	0.15	6×10^{-3}	63.73	245.51	304.94	18.01
	1.5	6×10^{-2}	64.99	248.54	306.74	18.83
	15	6×10^{-1}	65.77	251.20	307.21	19.11
Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide	0.15	6×10^{-3}	61.79	221.82	234.80	1.09
	1.5	6×10^{-2}	62.80	226.72	235.30	1.14
	15	6×10^{-1}	63.56	233.93	254.44	1.84

3.2 Microstructural Analysis

Field Emission Scanning Electron Microscopy (FESEM) is used to examine the microstructure. This analysis is conducted on pre and post specimens to investigate damage progression. From the observation in the previous section, Figure 5. The curve of Recycle Aluminium Alloy Reinforced Alumina Oxide is not fully identical as shown in brittle material. Even though the plastic hardening is too short, the curve predict a clear region of elastic and plastic part. While the primary Aluminium Alloy AA6061 is a ductile material. Therefore, it is important to examine the damage initiation and progression. Furthermore, from the microstructure observation (Figure 5,6,7), both materials experience localised plastic deformation during the tensile test due to the nucleation, growth, and coalescence of micro-voids, resulting in a crack.

3.2.1 Damage Initiation and Progression

To analyse the damage initiation and evolution, the tensile fracture surface of pre-test and post-test (Strain rate, $6x10^{-3}$) of the Recycle Aluminium Alloy Reinforced Alumina Oxide was observed by using Field-Emission Scanning Electron Microscopy (FESEM) as shown in Figure 5. According to the findings, the micro-voids that began in the pre-test specimen are growth and coalesced, resulting in the creation of further micro-voids when tensile loading is applied. Furthermore, a distinct chips bonding boundary may be seen on the fracture surface, particularly in specimens evaluated at low strain rates, indicating that the chip bonding is unstable and not totally bonded. The chip boundary displays the chip's solid-state welding performance. The smaller the gap between the chip's boundaries, the better the bonding behaviour of the recycled material [25,27].

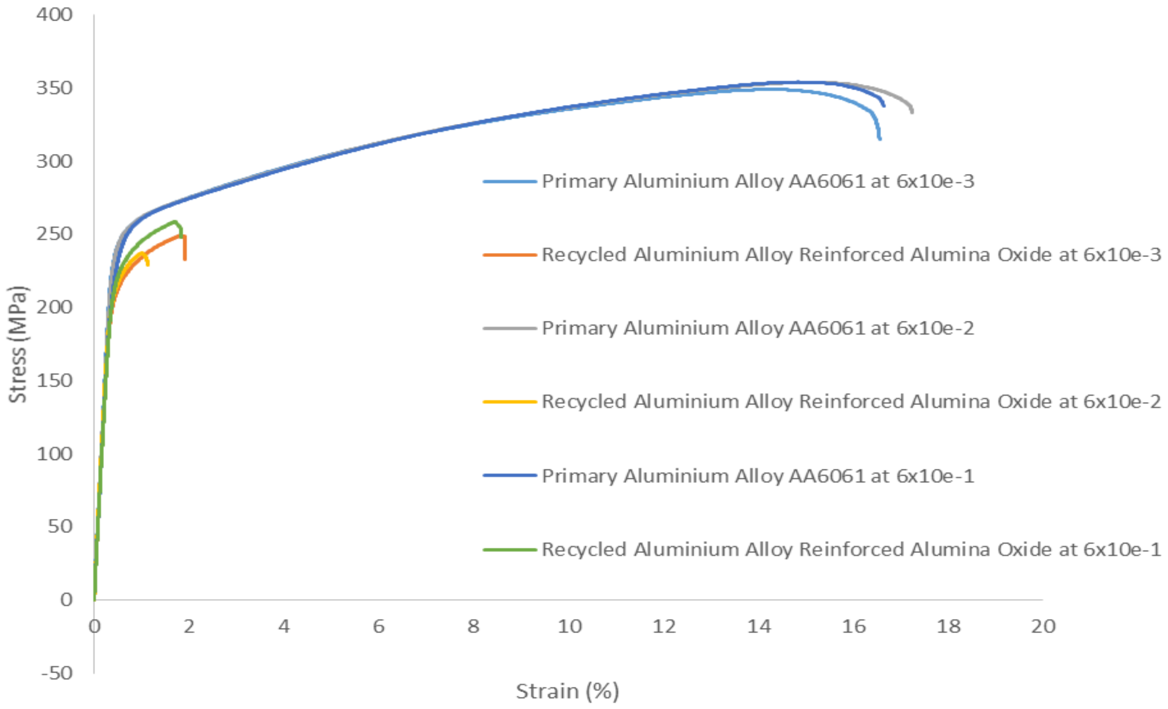
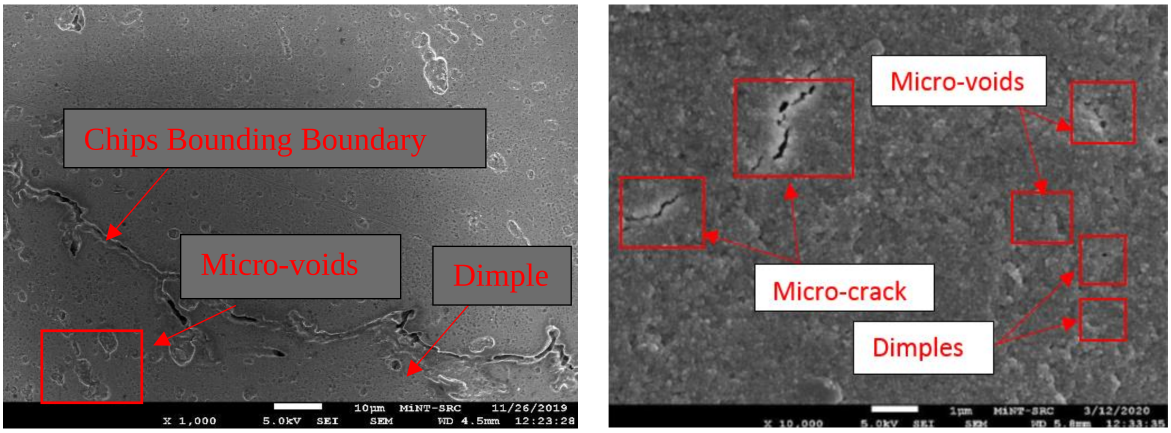


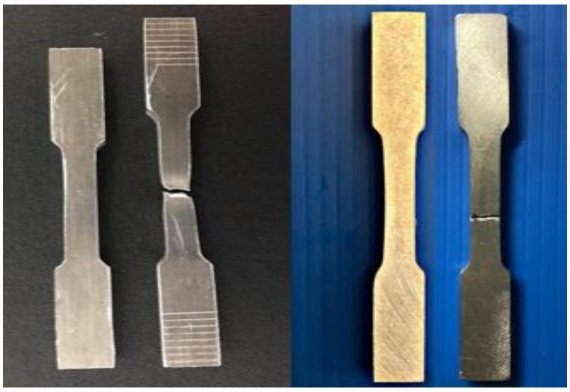
Fig. 4 - Stress-Strain curve of Primary Aluminium Alloy AA6061 and Recycle Aluminium Alloy Reinforced Alumina Oxide at different Strain Rates



(a) (b)

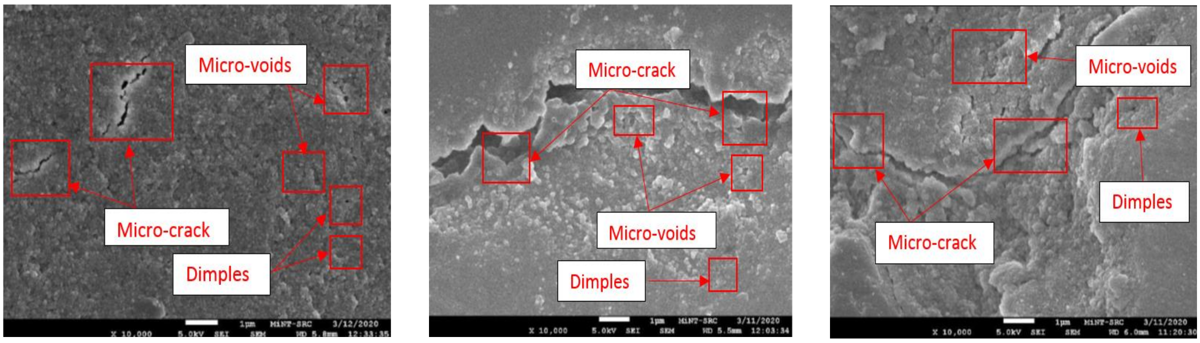
Fig. 5 - FESEM Microstructure of Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide at Strain rate, $6x10^{-3}$: (a) Pre-test and (b) Post-test

The sample of Primary Aluminium Alloy AA6061 and Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide pre- and post-test specimens are shown in Figure 6. On the primary specimen AA6061, there is noticeable necking around the fracture location, but this is less pronounced in the recycled reinforced specimen. After passing through the strengthening and recycling operations, the ductility of the recycled reinforced specimen is impaired due to Alumina characteristics (ceramic) that promote fracture during necking, reducing ductility and elongation. Even though both specimens appear to have a smooth and fine surface when viewed with the naked eye, the existence of micro-voids can still be seen under FESEM. Figures 7–8 show the creation and evolution of micro-voids underlying the fracture process. Figure 7 shows the fracture surface of the Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide at various strain rates as observed using Field-Emission Scanning Electron Microscopy (FESEM) at x10000 magnification. The increasing strain rates caused the severe micro-voids that developed into dimples and micro-cracks such as growth and coalescence due to damage evolution, according to the microstructure of the specimen. Furthermore, the dimples in Figures 7(a) and (b) are not densely distributed at low strain rates. The fracture, however, has a rough shape at high strain rates, as seen in Figure 7(c), with an increase in the number and size of dimples dispersed randomly and densely across the specimen's surface. Normally, dimples were created due to the dislocation mechanism, shifted towards the grain boundary during tensile deformation, increasing the dislocation density, and eventually resulting in intergranular fracture at higher strain rates.



(a) (b)

Fig. 6 - Sample of the undeformed and deformed tensile specimen: (a) primary aluminium alloy aa6061; (b) recycled aluminium alloy reinforced alumina oxide

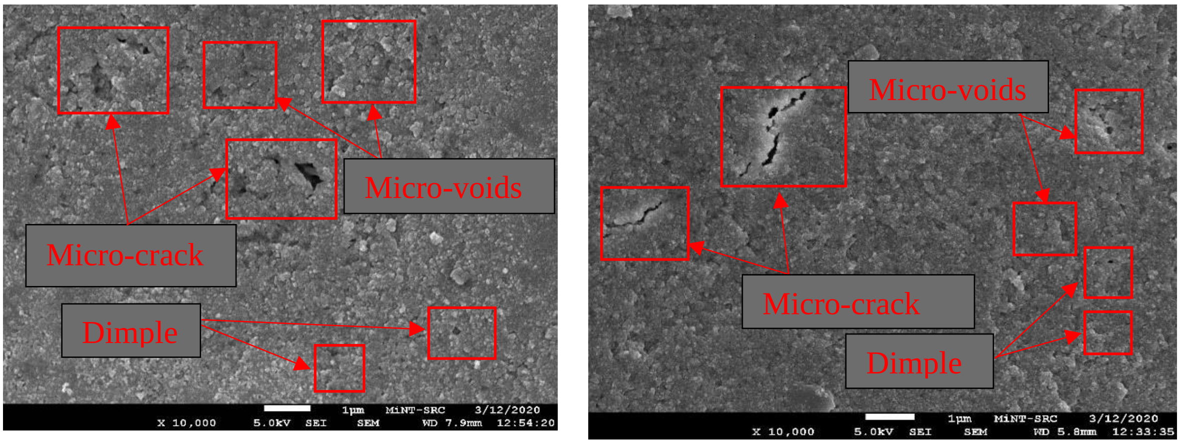


(a) (b) (c)

Fig. 7 - FESEM microstructure of post-test recycled aluminium alloy aa6061 reinforced alumina oxide at strain rate: (a) $6x10^{-3}$/s; (b) $6x10^{-2}$ /s, (c) $6x10^{-1}$ /s

Further, the results of the Recycled Reinforced specimen are compared to the Primary specimen as shown in Figure 8. Referring to both fracture surfaces of the materials, numerous dimples and micro-voids can be seen and the fracture mode is categorized as ductile fracture deformation. Figure 9 shows the macrostructural level of fracture surface on the primary and recycled reinforced specimen at different strain rates. The primary specimen shows necking on the fracture surface, indicating that the material is ductile due to a plastic strain energy (plastic deformation) driven by the ductile fracture mechanism of void initiation, growth, and coalescence. Due to the limitations of plastic deformation, the recycled reinforced specimen has a sharp, rough cup and cone shear fracture without apparent of necking. This behaviour is influenced by the reinforcement and contributes to reducing ductility. The ductile appearance may not be directly visible on a macro-scale.

In the primary specimen, ductile fracture is generally associated with high-load ductile tearing. The initial ductility can be observed by the lateral strain at the crack tip. However, in this case, the size of the plastic zone may be micro-scale. The fracture surface of recycled aluminium is formed by ductile fracture of the base metal and cleavage fracture of hard and brittle structural parts [28]. The Mixed-mode ductile and brittle cracks can be observed in this material by the presence of an intimate mixture of cleavage and micro-voids bound at the micro-scale. The cracking process occurs by a ductile mechanism while a fracture may appear due to macroscopically brittle. However, the terms ductility and brittleness are applicable to fracture at a nano-scopic level. On the micro-scale, the ductile fracture due to micro-voids and coalescence of the ductile process is characterised by plastic deformation. In contrast, macro-scale brittle fracture by cleavage is characterised by rapid crack propagation, which consumes less energy than ductile fracture and has no macroscale evidence of plastic deformation.



(a) (b)

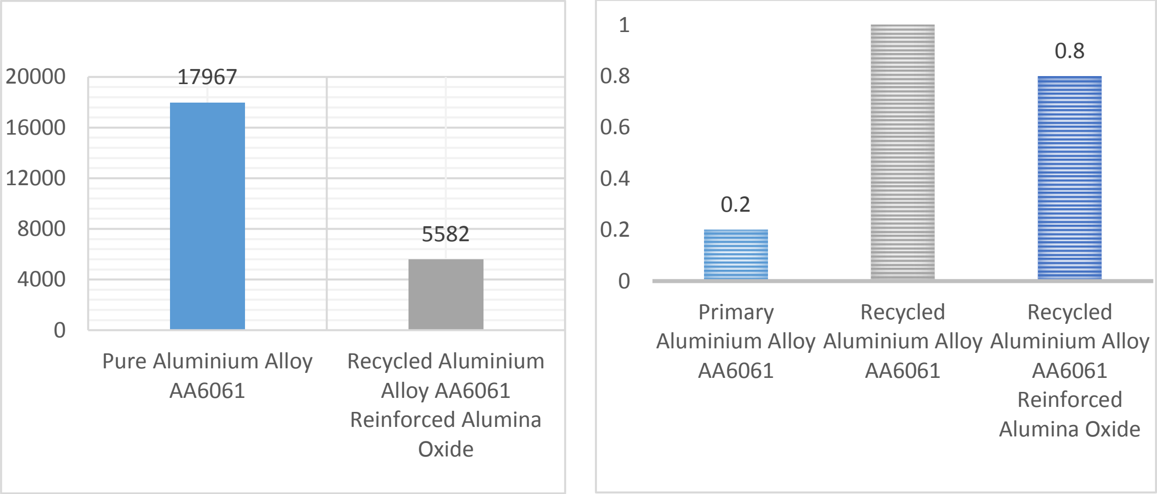
Fig. 8 - Comparison between microstructure of deformed at strain rate 6x10^{-3}/s: (a) Primary Aluminium Alloy AA6061 and (b) Recycled Aluminium Alloy AA6061 Reinforced Alumina oxide

Strain Rates	Primary Aluminium Alloy AA6061	Recycled Aluminium Alloy AA6061 Reinforced Alumina Oxide
6X10^{-3}		
6x10^{-2}		
6X10^{-1}		

Fig. 9 - Macrostructural level of the fractures surface on the primary aluminium alloy aa6061 and recycled aluminium alloy aa6061 reinforced alumina oxide at different strain rate

Analysis of damage progression is continued with a close review on voids quantity and size using Image J Tooling Software. Again, the analysis performed upon pre and post-test to compare the damage progression in the primary specimen and Recycled Reinforced specimen undergoing finite strain deformation. The result of ImageJ analysis is summarised in a chart shown in Figure 10 - Figure 11. Damage agents, such as micro-voids, are initiated in both pre-test specimens, as seen in Figure 10. Recycled reinforced specimen was lower and the average size of void was larger than primary form. This observation shows that the recycled material is a ductile fracture. This condition due to the reinforcement of recycled materials, where it reduce number of voids and the increased the size of voids which may result in ductility enhancement [29]. The larger the size of micro-voids, the greater the material's ductility and strength [30].

Referring to the Figure 11, the number of void for both materials increased at increasing the strain rates. At the low strain rate condition (0.006/s and 0.06/s), the number of voids for both materials almost identical. However, at high strain rate conditions (0.6/s), the number of voids for Recycled Reinforced specimen is lower than Primary specimen. The average size of void for recycled reinforced specimen is higher than the primary specimen and revealing that the secondary resources fail at a high number size of the void. This observation verifies that the damage accumulation is higher for recycled aluminium alloy reinforced with alumina oxide based on its microstructure than the primary aluminium alloy for a particular strain rate. This condition due to hard and brittle alumina which influenced the ductility of the material. The ductility and strength of the material have a direct relationship with the damage characteristics. As a result, it is demonstrated that the flow stress and total elongation of the specimen tested at a high strain rate are greater than tested at a low strain rate.

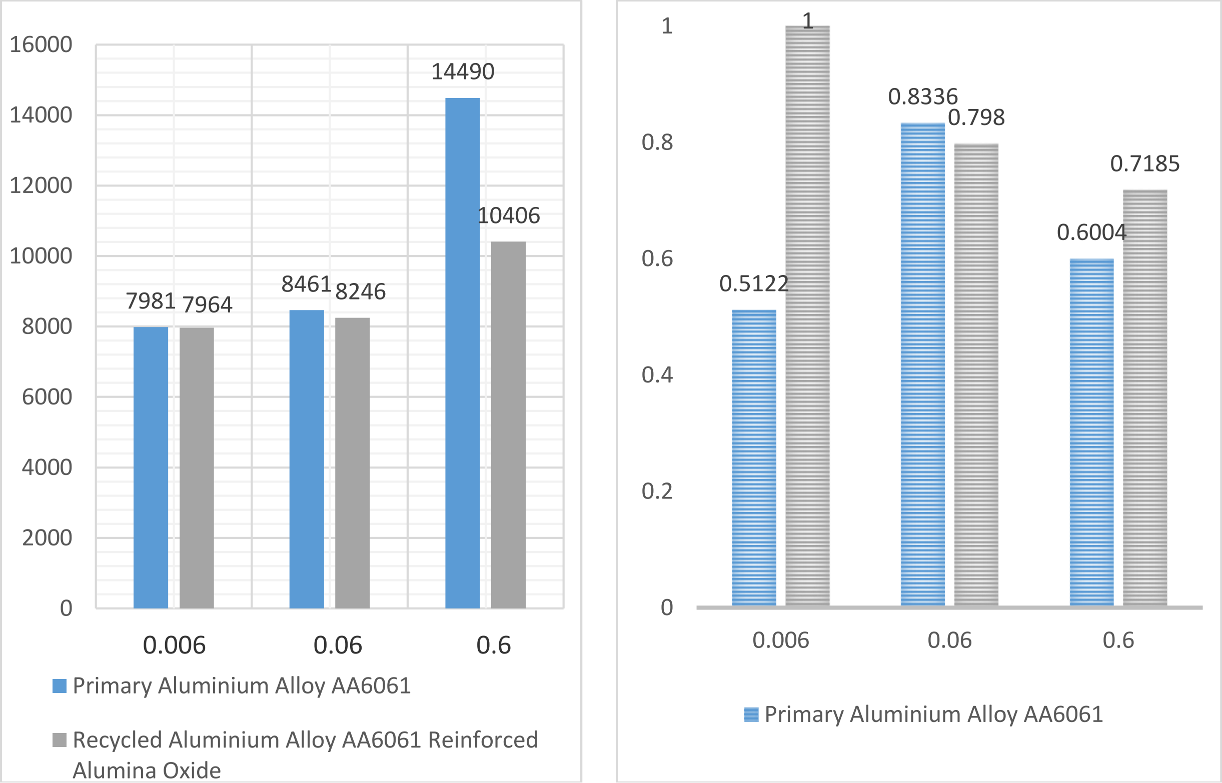


(a) (b)

Fig. 10 - Summary of ImageJ Analysis on Pre-test Specimen: (a) Number of Voids, and (b) Average Size of Void Count (nm^2)

4. Conclusion

This paper investigated the Mechanical Properties, Damage Progression and Fracture Modes of recycled aluminium alloy AA6061 reinforced Alumina Oxide at different strain rates. The result was analyses in terms of the stress-strain curve, microstructural of damage progression, including fracture mode and close review on voids quantity and size. The mechanical properties of recycled aluminium alloy reinforced alumina oxide show identical results to the primary aluminium alloy AA6061 except for ultimate tensile strength. The formability shows a weakness of this material by presented a low total elongation and also been described in microstructural behaviour. The reinforcement structures lead to reducing the capability to absorb energy and the ductility. The strain-rate dependency of recycled AA6061 reinforced Alumina Oxide was discovered by the investigation. Increasing the strain rate causes an increase in flow stress. The damage parameters begin to change as a result of the growth and coalescence of voids and cracks. Furthermore, it can be seen that the loading speed effects the damage evolution. With increased strain rate, the micro-voids are growth and coalesced, confirming a ductile fracture mechanism which leads in the production of dimples and micro-cracks. This mechanism is observable around a localised plastic deformation area, but it may not be prominent throughout the deformed specimen. Besides that, this material shows the Mixed-mode of ductile and brittle cracks by the presence of an intimate mixture of cleavage and micro-void bound at the micro-scale. The cracking process occurs by a ductile mechanism while a fracture may appear due to macroscopically brittle. The recycled reinforced specimen has a smaller number of voids and a larger average void size than the primary specimen in terms of quantity and size. The presence of a ductile fracture in the recycled material is indicated by this finding. In conclusion, significant effort remains to be done to improve the capability of recycled Aluminium Alloy AA6061 reinforced Alumina Oxide as fulfilled by the primary resource. The results of the method used are beneficial in gaining a better knowledge of the precise response of reinforced recycled materials when subjected to various strain rates.



(a) (b)

Fig. 11 - Summary of ImageJ Analysis on Post-test Specimen: (a) Number of Voids, and (b) Average Size of Void Count (nm^2)

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