



Effect of Annealing and Artificial Ageing Parameters on The Ultimate Tensile Strength and Elongation of New Al-(4-5) Zn- Mg-Mn-Cu Alloys Fabricated using Recycled Beverage Cans

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Abstract: The transport industry is one of the highest consumers of aluminium alloys. However, the quest for new and cheaper aluminium with properties comparable to the contenders has remain in the research domain for sometimes. Even at that, the literature is dearth of clear ideas on conversion of the huge RBCs to X7475 experimental alloys. In furtherance to the fabrication of a new 7xxx (Al-4-5Zn-1.5Mg-1.0Mn-0.35Cu) alloy from Recycled Beverage Cans (RBCs), the effect of annealing temperature (350 °C, 380 °C and 413 °C) and artificial ageing time (6, 10.5 and 15 hours) on the ultimate tensile strength (UTS) and elongation (e) were reported. Nine samples (S1-S9) of experimental alloys were fabricated and subjected to tensile tests, SEM/EDX and XRD analysis. Secondary phases like MgZn₂, Cu₂Mg precipitates as observed in the XRD supported an improved UTS. The elemental analysis revealed the presence of alloying compositions. An alloy fabricated using Al-5Zn-1.5Mg-1.0Mn-0.35Cu (S9), artificially aged at 6 h and annealed at 413 °C had the highest UTS of 362.2 MPa and an elongation of 3.09 mm (15.45 %). The result demonstrated that a new X7475 alloy was fabricated from RBCs has comparable mechanical properties with alloys fabricated using pure aluminium.

Keywords: Recycled-beverage-cans, X7475, phases, tensile strength

1. Introduction

Nowadays, the demand for aluminium in the transport industry is on the increase. This is partly due to the quest for reduction in the weight of the automobile. Besides, the advent of electric vehicles and economic pressure on the automobile industry calls for reduction in the cost of production. More so, the dynamics and competitiveness in the transport industry are the major drivers for continuous development of cheaper, new and innovative materials. The

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conventional alloys used in the automobile industry include the AlMg (5xxx) and AlMgSi (6xxx) alloys. Conversely, the 7xxx alloys are gaining gradual attention as a competitive alternative in the transport industry [1].

Apparently, the use of aluminium by the beverage packaging industry was estimated to be about 2×10^{11} cans every year. By extension, assuming the weight of a can is 30×10^{-3} kg, thus 2×10^{11} cans translates to 6×10^9 kg waste per annum [1]. Global market size of aluminium beverage can was USD 40 billion in 2015, with compound annual growth rate (CAGR) of 4.6 % from 2016 to 2024. The demand for can usage was projected to witness over 5 % consistent increase within the same period, because demand in developing economies is increasing. This means there would be more cans to be recycled in the nearest future. Currently, beverage aluminium can recycle rate improved from 45 % in 2017 to 50 % in 2018. However, the can-to-can recycling is a closed product loop that has gained the attention of researchers more [2].

The direct and indirect challenges posed by this demand are environmental concerns of managing this huge resources and increase in energy demand to process cans from aluminium. On the other hand, secondary aluminium production through recycling consumes about 5-6 % (reduction of 95%) of the energy required in processing aluminium from the primary source [3,4]. Recycling is an essential activity required in sustaining the demand for aluminium in the automobile and aerospace industries. Concerted efforts are being made to reuse aluminium as more than a third of all the aluminium currently produced globally originates from old, traded and new scrap [5]. The attempt by Ochoa et al [6] showed the potentials in fabricating Al-Zn-Mg alloys containing up to 2.47 wt.% Zn using spent battery and beverage can. The addition of 2-8 wt. % pumice powder to RBCs reinforced the matrix as reported by Dagwa and Adama [7] showed a prospect for additional investigation. However, the focus of these researches was not to produce a 7 series alloy from recycled beverage can (RBCs). Sharma et al [8] showed that various properties are obtainable through heat treatments and alloying of aluminium. The addition of other constituents to and heat treatments of aluminium are reasons for improved mechanical properties [9-11].

The 7xxx alloys find application in the transportation (aerospace and automobile) industries and can be sourced from the RBCs. This is because the RBCs have about 0.2 years useful lives, hence it is not a long term economic engagement when the can-to-can is sustained, with only 25 % to 85 % currently recycled [4]. Whereas, the RBCs are capable of generating 400,000 - 500,000 tons of recycled aluminium with 98% yield [5] for engineering applications [4]. However, bulk of researches on the 7xxx alloys used commercial alloys [12-14], whereas RBC was suitable as a source of Al that can be converted to the 7xxx alloys by the addition of Zn, Mg, Mn and Cu. The effect of wt.% Zn addition of other grain refining elements, annealing and artificial ageing heat treatment parameters had been reported to affect the mechanical properties of 7xxx alloys [15-17]. The formation of secondary phases like the η (MgZn₂), T(Al₂CuMg), and S(Al₂CuMg) were affected by the aforementioned parameters [15,18] as a result of the increase in the solute content around the vacancies [19]. Our analysis of the transformation of phases within the Guinier–Preston (GP) zones [20] in presented in Figure 1. Characterizations of Al-Zn alloys done in previous works [9,10] were deficit in addressing the effect of annealing after solution treatments prior to the artificial ageing on the mechanical properties of the novel Al-Zn-Mg-Cu alloy fabricated from RBCs.

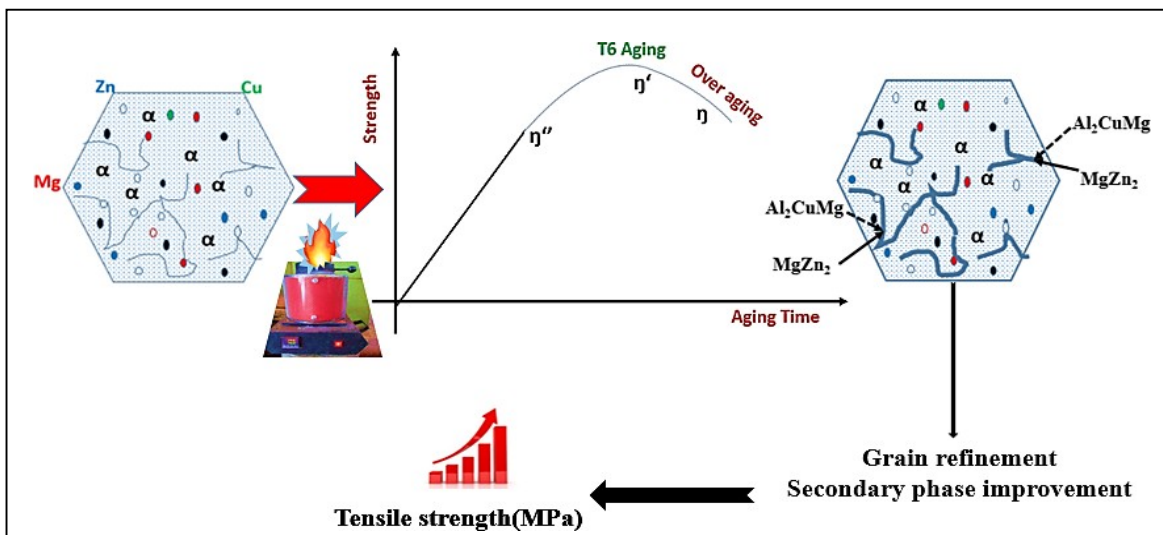


Fig. 1 - Effect of artificial ageing on phase transformation and strength of X7475 alloys

On the above premise, this letter aim at reporting the effect of annealing and artificial ageing parameters on the Ultimate tensile strength and elongation of new Al-(4-5) Zn-Mg-Mn-Cu alloys fabricated using RBCs. In addition, elemental analysis, and XRD were presented to report the observed compounds. During the fabrication, wt.% Zn was between 4.0 - 5.0 while Mg, Mn and Cu were held at 1.5, 1.0 and 0.35 wt. % respectively. A total of 9 samples were fabricated and identified as Samples 1-9 (S1-S9). Subsequent sections of this letter are dedicated to the methodology

used in converting the RBCs to experimental 7475 alloys within the green letter specifications [23]. Results, discussions, and followed by conclusions drawn from the findings of this study were presented in the third and fourth sections of the paper.

2. Materials and method

Bulk (80%) of the raw materials used in this study were sourced from secondary materials. Recycled Beverage Cans (RBCs) were collected from waste bins on the campus of Universiti Tun Hussein Onn Malaysia (UTHM) and re-melt to recover aluminium. Zinc (Zn) and Manganese (Mn) were recovered from type *DR 20, BG/T 8897.2 - 200 Hawk* dry cell battery. Copper (Cu) was recovered from *2-ARI 410* standard wire of standing fans. Characterization of raw materials using SEM/EDX showed that the RBC was an Al-Mg-Mn alloy while Zn and Mn were from the battery as presented in Figure 2. Hence, 97 wt. % Al, 98 wt. % Zn and 95.87 wt. % MnO were recovered from RBCs and *Hawk* battery respectively, while only Mg was as supplied. An amalgam of 70 wt. % Cu-30 wt. % Al was prepared to expedite the alloying of Cu in Al.

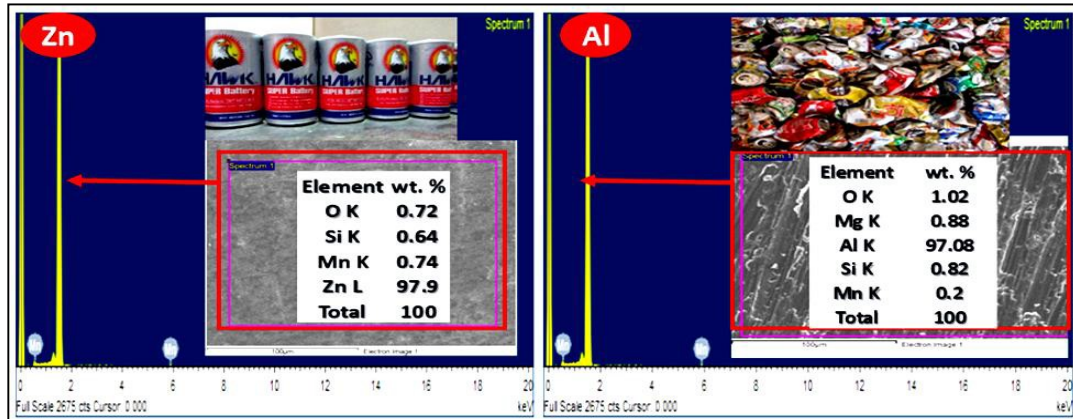


Fig. 2 - SEM/EDX elemental analysis of raw materials obtained from hawk battery and RBCs

Fabrication of alloys of Al-Zn-Mg-Mn-Cu was done in a *JT0332* model 2 Kg induction furnace using self-made zirconia crucible while a model *TAC 1803*-Pentec stirring machine was used to achieve mechanical mixing. A 200 °C pre-heating temperature was used to eradicate moisture from the crucible prior to casting. Al, Zn, 70% Cu- 30% Al, Mn and Mg were introduced at intervals, while temperature was adjusted gradually to 1200 °C. Following the attainment of homogenized alloy, pouring was done at 720 ± 10 °C. The composition of Mg, Mn and Cu were maintained at 1.5 wt. %, 1 wt.% and 0.35 wt.% respectively, while Zn was varied in accordance with the description in Table 1.

Table 1 - Experimental parameters

Parameters/Samples	S1	S2	S3	S4	S5	S6	S7	S8	S9
Zn (wt.%)	4	4	4	4.5	4.5	5	5	5	5
T6-Time(Hrs)	15	6	15	10.5	15	6	15	6	6
Annealing Temp(°C)	413	413	350	380	413	350	350	350	413

Tensile test samples were machined per ASTM E8/E8M-11 with dimensions shown in Figure 3. Solution heat treatment was done at 475 °C, while artificial ageing was at 100 °C using *Nabertherm B180 MB2* furnace. Artificial ageing time was varied at 6, 10.5 and 15 h, while annealing was varied at 350 °C, 380 °C and 413 °C respectively. The aim of conducting annealing (350 – 413 °C) was to relief the residual stress due to machining and quenching in the alloy. This was done to obtain a trade-off in strength-elongation as requisite for selected applications such as bumper beam. The artificial ageing time of 6-15 hrs was sufficient to allow the formation of secondary phases required to improve on the strength of the alloys after annealing. The tensile test was done following the ASTM B 557M – 02a using *Gotech AI-7000 LA5* Servo controlled machine. Load resolution and test speed were 1/200,000 and 0.5 mm/minute. Ultimate tensile strength (UTS) and elongation were determined from the tensile strength report. Fractographic investigation, SEM/EDX analysis were performed on *Hitachi VP-SEM, SU1510* machine. The phase identification of the As-Cast and heat treated alloys were done using a Bruker D8 advance machine. The pattern was scanned in series of angles of diffraction from $10^0 - 90^0$ (2θ) in steps of 0.034 designed for metals using copper (CuK α) with a wavelength of 1.5406Å as X-ray source.

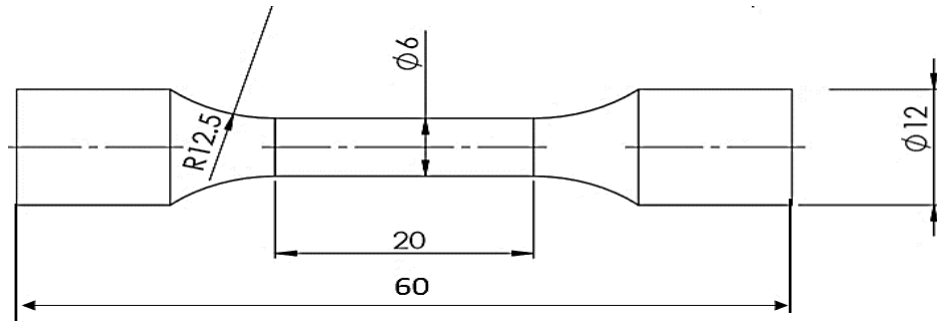


Fig. 3 - Schema of tensile test sample, all dimensions are in mm

3. Results and Discussion

3.1 XRD Phase Analysis and Fractograph

The prominent phases as shown in Figure 4 are crystalline of Al, FCC, with $hkl = 111$ (ICDD 004-0787). There were multiple phases where Zn was entrenched on a phase containing aluminium and identified with $hkl = 101$ (ICDD 004-0831). The dual peak indicates that the constituents were homogenized and confirmed the formation of an Al-Zn alloy. Similar phases like the ICDD 004-0787 were reported to contain bulk aluminium in the findings of Higashino et al [24] and Fernández et al [25]. Heat treatment was responsible for the phase transformation [17] as observed in as cast and heat treated samples. The study conducted by Michael and David [26] showed that the stability of FCC solid solution was associated with higher valence electron with concentration ≥ 8 .

It followed that the crystallinity of $MgZn_2$ reduced to a considerable extent with artificial ageing. That was an indication that heat treatment supported the dispersal of hardening precipitates. For instance, $MgZn_2$ represented with ICDD = 100-034-0457 at $2\theta = 57.480^\circ$ had Miller indices $hkl = 105$.

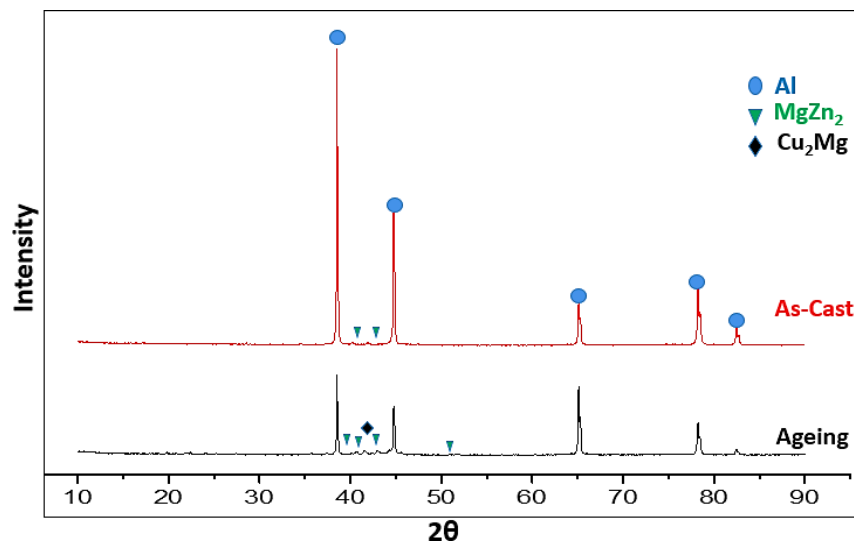


Fig. 4 - XRD plot of as-cast and artificial ageing alloys

This formation was a weak one and differ from the $MgZn_2$ with 00-034-0457 ICDD. The implication was that when T6 treatment was done, more of the precipitates dissolved, hence the change in the direction of the Miller parameter was partially the reason for the difference in elongation and UTS observed in the samples. This result is comparable to the findings in Fang et al [27]. Growth of phases and hardening precipitates were suggested to be formed with the heat treatments in the vicinity of the EDX analysis as shown in the fractographic analysis in Figure 5. The presence of fibrous particles (Figure 5 a and b) and micro dimples were observed on the rough fracture surface. The fracture planes revealed fine particles frequently associated with improved strength and representing areas of extensive deformation that preceded the ductile fracture in the matrix due to the tensile load under test. The ductile fracture was responsible for the formation of cup and cone fracture in Figure 5 (e). The addition of Cu was suggested to have formed the Cu_2Al and Al_4Cu_3Zn phases. The duo were hardening precipitate, with fewer larger particles [28] and improved the strength of Al alloys [29] as characterized by dimples. In addition, the EDX analysis of elements presented in the fractography (Figure 5c) had 5.72

wt. % O, 1.07 wt. % Mg, 1.66 wt. % Si, 0.2 wt. % Mn, 1.41 wt. % Cu and 5.18 wt. % Zn. On the other hand, 4.91 wt. % O, 0.97 wt. % Mg, 1.06 wt. % Si, 0.39 wt. % Cu, 4.51 wt. % Zn in 5(d) were confirmations that a new experimental alloy was formed from RBCs.

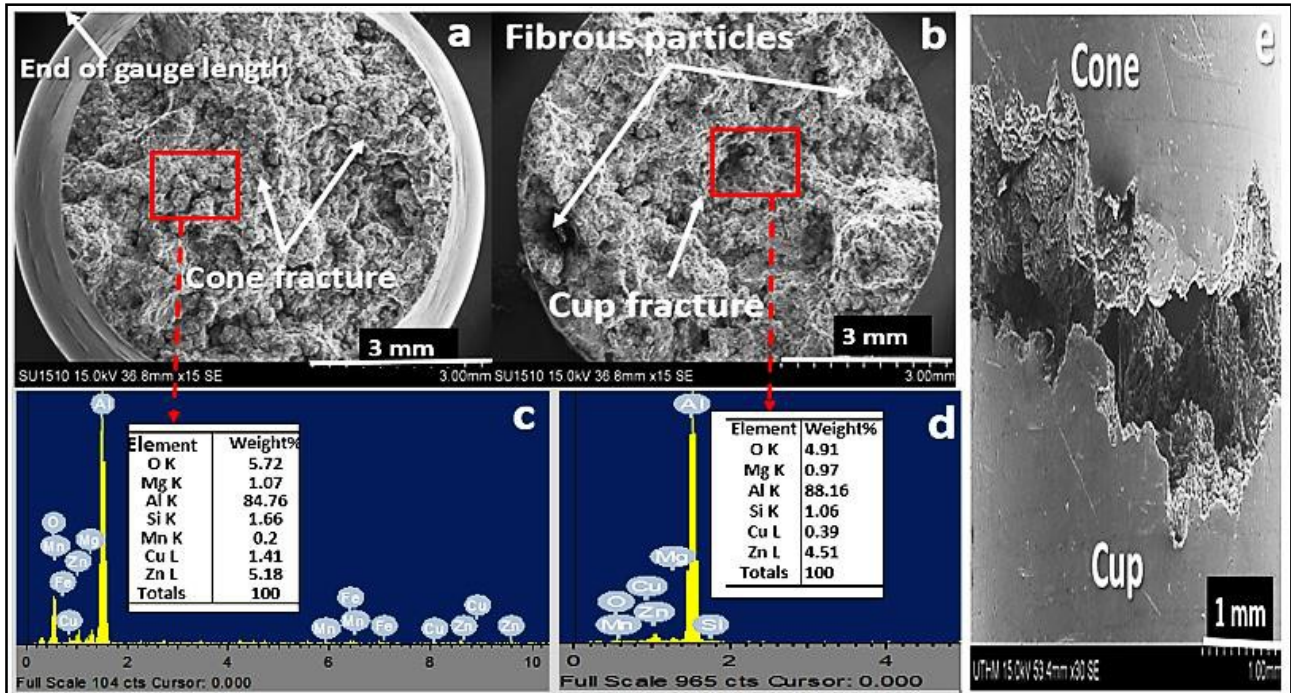


Fig. 5 - Cup and cone fracture with fibrous morphology and elemental analysis of Al-5Zn-1.5Mg-1Mn-0.35Cu obtained from the tensile test of alloy S8 after annealing at 350°C and artificial ageing at 100°C for 6 h

The phases observed in the XRD analysis were further strengthened with the presence of the aforementioned elements. The presence of Si was due to the crucible and the inherent contaminants from the recycled materials used in fabricating the alloys. The presence of O was an indication that the fractograph contain microvoids within the globular dendrites. The dendrites reduced with increase in heat treatment duration [30]. The dimples were indications of the ductile fracture observed in the Al-5Zn-1.5Mg-1Mn-0.35Cu experimental alloys. This was partly responsible for the 142.6 MPa observed in S4 as against the 167.6 MPa observed in S6. Fibrous particles with the cup and cone were observed in the ductile fracture as presented in Figure 5 (a) and (b). The elongation of 3.11 mm (15.55 %) observed in S5 suggest the formation of cup and cone fracture and improved properties.

3.2 Ultimate tensile strength and elongation

Samples S1, S2 and S3 were alloys fabricated with compositions of Al-4Zn-1.5Mg-1Mn-0.35Cu respectively. The annealing temperature of 413 °C was maintained in S1 and S2, while the temperature was reduced to 350 °C in S3 with the aim of studying the effect of the change in annealing temperature. An UTS of 50.3 MPa, 62.4 MPa and 95.7 MPa were recorded in these alloy, with elongation of 0.37 mm (1.85 %), 0.26 mm (1.30 %) and 0.37 mm (1.85 %) respectively. The result presented in Figure 6 showed that the reduction from 15 h holding time in S1 resulted in the low UTS and elongation observed. This result suggest that the prolonged ageing time increased the precipitation free zones (PFZs) that made S1 elongate for only 0.37 mm (1.85 %) before fracture. In an earlier study, it was reported that prolonged increase in the size of the grain boundary precipitate resulted in the formation of larger PFZs within the vicinity of grain boundaries and suggest vacancy due to solute depletion mechanism [31]. S2 had UTS that was 12.1 MPa greater than S1 and 33.3 MPa lower than S3. The reduction in the annealing temperature from 413 °C to 350 °C had supported the dispersal of the hardening precipitates prior to the T6 ageing at 15 h. Suffice to mention that all samples were aged at 100 °C, hence the differences in the mechanical properties being reported were as a result of the variations in annealing temperature and artificial ageing time.

In a further investigation, the additional 0.5 wt.% Zn in combination with 380 °C annealing temperature was responsible for the increase in the elongation from 0.37 mm to 1.61 mm (8.05 %) and UTS of 142.6 MPa recorded in S4. This was comparable to the findings of [32] where increase in wt.% Zn raised the mechanical properties of Al-Zn alloys. In the same vein, S5 was equally fabricated with Al-4.5Zn-1.5Mg-1Mn-0.35Cu like was the case in S4, the improved elongation from 1.61 mm (8.05 %) to 3.11 mm (15.55 %) and the UTS of 163.6 MPa was a reflection of the role of the

15 h T6-time. The result suggest that the 380 °C annealing temperature used in S4 felt short of effusively dispersing the precipitated phases as against the 413 °C in S5.

Further observed from Figure 6 was that the addition of 0.5 wt.% Zn had increased the mechanical properties of the Al-Zn alloys fabricated from RBCS. Take the elongations in S6 through S10 with 2.12 mm (10.60 %), 3.05 mm (15.25 %), 2.96 mm (14.80 %) and 3.09 mm (15.45 %) respectively as an instance.

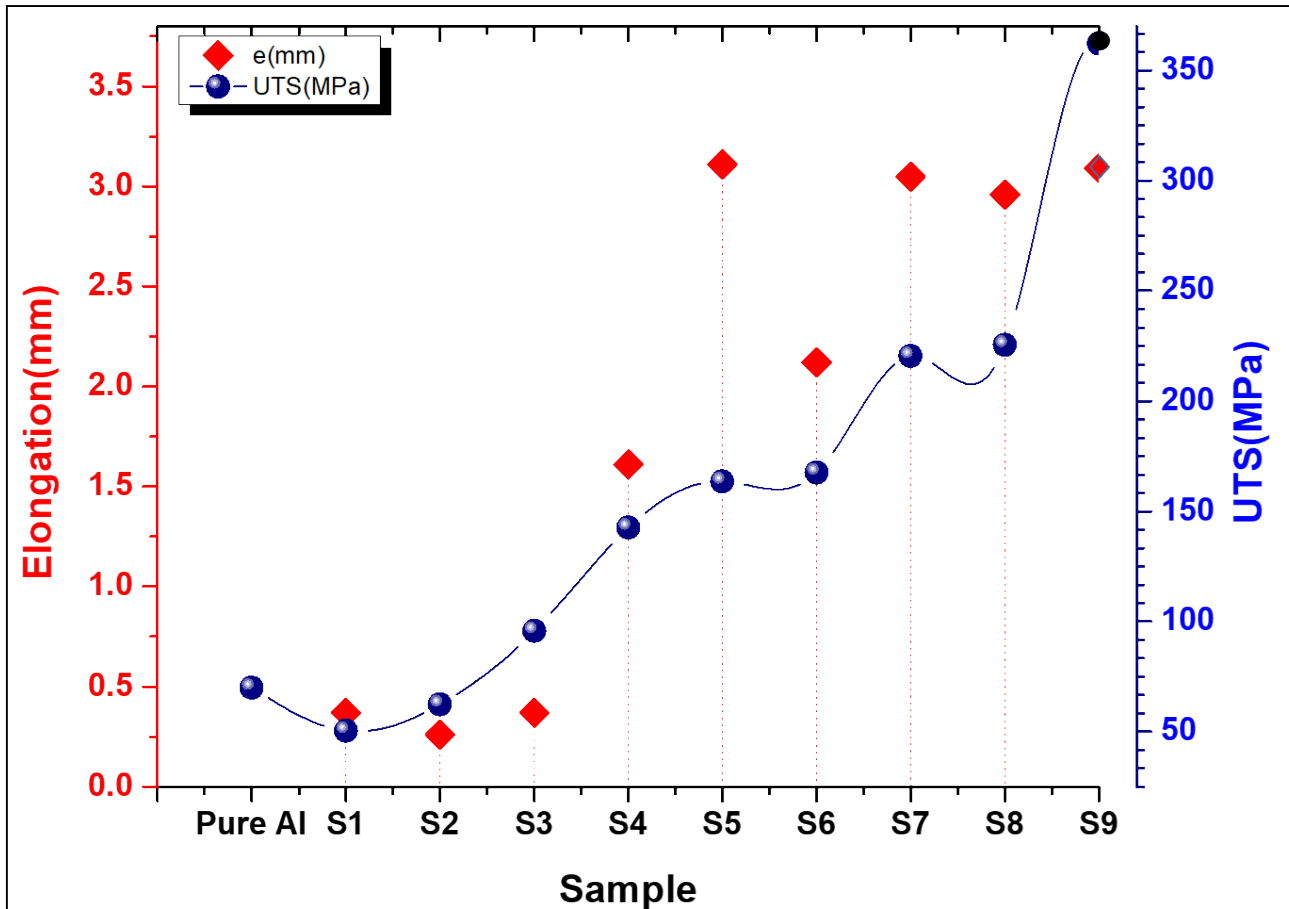


Fig. 6 - Effect of different heat treatment parameters on UTS and elongation of X7475 alloys

The improved elongation was due to the formation of the η phase in the aluminium alloy following the heat treatment like was demonstrated in Figure 1. Previous findings observed that this phase was with fewer large particles present within the Al-Cu phase and supported the 25 % improved elongation [28]. The alloys with 5 wt. % Zn had higher elongation and improved UTS with 167.6 MPa, 220.5 MPa, 225.5 MPa and 362.2 MPa observed in S6, S7, S8 and S9 respectively. For S9, the alloy was artificially aged at 6 h and had the highest UTS. The result show that the highest wt.% Zn and annealing temperature of 413 °C combined well with the lowest T6-time (6 h) to deliver the highest UTS of 362.2 MPa and an elongation of 3.09 mm (15.45 %).

4. Conclusions

The RBCs was explored resulting in the fabrication of a novel X7475 experimental alloy with 80% of raw materials from recycled sources. Following quenching, annealing was conducted to relief the stress in the alloy before artificial ageing. The alloys fabricated were characterized and the following conclusions were drawn;

- i. Fractographic, SEM/EDX and XRD analysis showed that a new X7475 alloy was fabricated from RBCs.
- ii. Increase in wt.% Zn produced improved mechanical properties with the highest UTS of 362.2 MPa and elongation of 3.09 mm (15.45 %). This was due to the annealing temperature of 413 °C and artificial ageing that supported the formation of hardening precipitates like the $MgZn_2$ and Cu_2Mg . These phases dissolved in the aluminium alloy with artificial ageing and the result suggest that the hardening precipitates resulted in a good UTS-elongation compromise.
- iii. The maximum elongation of 3.11 mm (15.55 %) was obtained in an alloy fabricated with 4.5 wt. % Zn, annealed at 380 °C and T6-Time of 10.5 h.

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