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Effect of Imperfections on and Modelling of Conductivity and Hardness of High-Zn X7xxx Cast Al-Alloys Produced from Recycled Beverage Can

Abubakar Kazeem^{1,3}, Amkpa Job Ajala², Nur Azam Badarulzaman^{3*}, Wan Fahmin Faiz Wan Ali⁴

¹Department of Science Policy and Innovation Studies, National Centre for Technology Management (NACETEM), North Central Zonal Office, FCT-Abuja, NIGERIA

²Department of Foundry Engineering, Federal Polytechnic Idah, PMB 1037 Idah, Kogi State, NIGERIA

³Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia (UTHM), 86400 Parit Raja, Johor, MALAYSIA

⁴Faculty of Mechanical Engineering, Universiti Teknologi Malaysia (UTM), 81310, UTM-Skudai, Johor, MALAYSIA

*Corresponding author

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Abstract: In this paper, the effect of imperfections as a result of formation of new phases on electrical and macro hardness property of a novel experimental high-Zn Al-Zn-Mg-Cu alloys produced from recycled beverage can was investigated with the aim of correlating hardness with conductivity using ANOVA. Alloys were observed in the As Cast (AC), annealed (O), natural aged (T4) and artificial aged (T6) conditions. Heat treatments supported the formation of hardening precipitates like the $Al_{2.06}Fe_4$, $\alpha-AlFeSi$, Al_7Cu_2Fe , $\theta(Al_2Cu)$, $\beta-AlFeSi$ and $MgZn_2$ phases. Imperfections inform of voids and contaminants are by-products of recycled aluminium and casting route. Peak obtainable hardness of 122.94 HV in the T6 condition was observed in an alloy of Al-5.0 Zn-1.5 Mg- 0.35 Cu. The same alloy's conductivity is 3.676×10^7 S/m. The least hardness of 38.16 HV and conductivity of 3.533×10^7 S/m is credited to an alloy of Al- 5.0 Zn-1.00 Mg-0.35 Cu in the O condition. The relationship between hardness and conductivity is nonlinear. Models developed to predict the hardness of this experimental alloys fits all the variables and covers the AC, O, T4 and T6 respectively. The need for further investigation on the imperfections, optimization of mechanical properties and additional mechanical properties investigation is required.

Keywords: Recycled Beverage can, Imperfections, experimental alloy, conductivity, high-Zn

1. Introduction

The use of aluminium in the matured beverage can industry was dated back to decades. About 95% of the energy required in processing virgin Al was saved by recycling aluminium. On top of that, 8 Kg of bauxite, 4 Kg of chemical products and 14 Kwh of electricity that would have been consumed for each Kg of Al mined from new raw material are saved [1]. Globally, about 475 billion cans are produced and nearly 52 billion per year are used in the Europe. The beverage cans are totally recyclable. Dagwa & Adama [2] added pumice to aluminum beverage cans and reported an improved 11.08 HV in hardness and 28.39% on tensile strength. Abdulsada [3] attempted to recover aluminium alloy from beverage cans and reported that the alloy contained Mn as major alloying element with hardness of 50.59

*Corresponding author: azam@uthm.edu.my

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kg/mm² after homogenization. It was however difficult to clean recycled beverage cans from impurities and this made most scrap end up in casting alloys [1].

The potentials of adopting an aluminium alloy produced from recycled beverage can for the production of automobile bumpers frame is bright, hence the scarcity of aluminium scrap from cars deepens [4]. However, a good casting devoid of imperfections was germane to obtain high quality products. Still on imperfections, the microstructure of a material depicts the properties of the alloy and phase formations. The duo are products of material processing route and could be altered through heat treatments. In fact, misfit-stress fields, pores, dislocations were reported to have altered the properties of aluminium alloys [5,6]. The study conducted by Shi et al [7] concentrated on the relationship between solid solution temperature, water temperature and quenching time on the mechanical properties and conductivity of commercially available 7475 alloy. Similarly, AA 2219 alloy bars were forged and studied for the effect of aging time on the mechanical properties and electrical conductivity in the work of Prabhu (2017). In a recent study, Kazeem et al [9] suggested the correlation between hardness and electrical conductivity of the new X7475 alloy produced from recycled beverage cans.

On the above premise, the aim of this article was to investigate the effect of contaminants on the electrical and macro hardness property of experimental X7475 alloys produced from recycled beverage can for passenger car bumper beam applications. The paper further establishes the relationship between hardness and electrical conductivity using ANOVA and suitable linear models. In this work, 9 samples were produced with the variations in Zn (5.0, 4.5, 4.0 wt. %), Mg (1.50, 1.25, 1.00 wt. %) and Mn (0.075, 0.050, 0.025 wt. %). For instance S1 was an alloy of Al-5.0 Zn-1.5 Mg-0.075 Mn-0.35 Cu while S9 was made of Al-4.0 Zn-1.0Mg-0.025 Mn-0.35 Cu. In all the 9 alloys, Cu remain constant while Zn, Mg and Mn vary through S1-S9.

2. Methodology

More than 2 Kg of recycled beverage cans of the AA 3004 aluminium alloy series was collected from recycle center located nearby Universiti Tun Hussein Onn Malaysia to produce Al-ingots. Zn and MnO was recovered through physical dissection and separation from nine spent *Hawk* batteries of the GB/T 8897.2-2008 specifications. Cu was sourced from copper wire by unwinding the coil of a standing fan. MgO in powder form was as supplied. The copper wire coating was scratched off before cutting it to pieces. Beverage cans were smashed and fed into a gas-fired furnace to produce Al-ingots. Zn ingot was made from pieces of Zn recovered from the battery using a portable induction *Electric Melting Furnace, model JT0332*, made in China. An amalgam of 70 wt% Cu - 30 wt% Al was produced to reduce the melting point of Cu in Al. Constituents were measured per table 1, using the *Model: PL 303, Mettler Toledo* digital weighing machine.

Table 1 - constituents in wt% used in this experiment

Constituent	Zn	Mg	Mn	Cu	Al
Wt%	4-5	1-1.5	0.025 - 0.075	0.35	Balance

Casting commenced by preheating the furnace and graphite crucible to 200 °C. Al-ingot chips were charged, then Cu-Al alloy followed by Zn, MnO and MgO in that order. Manual stirring was achieved by using portable *Pentec TAC 1803* mixer set at high speed. Pouring of the molten alloy was done at 720 °C. A custom made permanent steel mould of Ø18mm × 160mm pipe was used in casting. Rods were allowed to age for 48 hrs before sectioning of characterization samples. The *Labotom-3 cutter* equipped with Buehler, 10-4150-010 cutting tool for aluminium was used in cutting the coupons following the removal of external solidification-hardened layer on the *Harrison Alpha A400 Lathe Machine*.

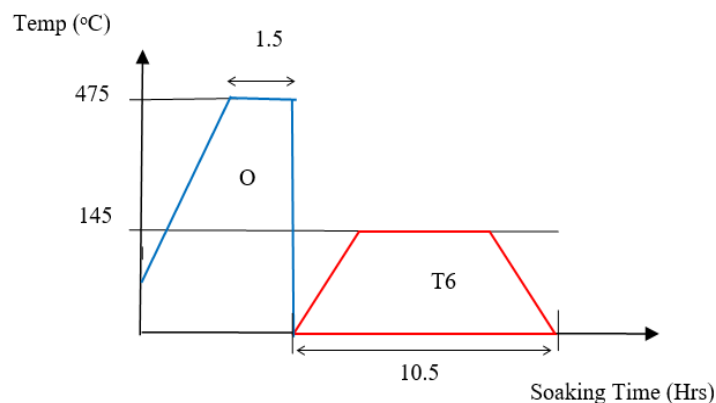


Fig. 1 – Heat treatment profile

Thereafter, heat treatment was done per figure 1 using the Carbolite HTF 1800 furnace. Alumina crucible plate was used to prevent contamination. Solution heat treatment was done at 475 °C for 1.5 hrs, quenched in clean water at a fast rate in a container placed at about 100 cm away from the furnace to “frozen in” precipitates within the α -Al matrix. This was followed by annealing (3 hrs, 350 °C), natural (4 hrs, 110 °C) and artificial aging respectively. Samples were taken through mechanical preparation processes of grinding and polishing for morphological and hardness investigations. Coupons of $\varnothing 18$ mm \times 6 \pm 0.5 mm height were prepared per ASTM E384-17 for Macro hardness test using the *Buehler machine, Model 1900-2005-250* installed with a WF 10 \times microscope. Indentation load was set at 5kgf for a loading time of 10 sec and loading speed of 50 μ m/sec.

Characterization was done using *Hitachi SU1510 VP Scanning Electron Microscope (SEM)* installed with high-performance electron optics of S-3700N in conjunction with Energy Dispersive X-Ray Spectroscopy (EDX) and Nikon Eclipse-LV150NL Optical microscope (OM) quipped with Solution DT software. A GWINSTEK LRC- 816 meter equipped with test leads of 105 °C, 6v 52386, LR 77177 CSA specifications was used in conducting 5-point probe of samples. Samples were polished to mirror like surface before the conductivity test. The calibration followed an accuracy of $\pm 1\%$ Ω while test was performed per ASTM E 1004 – 02 standard. The obtained resistance was converted to conductivity.

3. Results and Discussion

3.1 Characterization

During precipitation heat treatment, phases are formed and the speed of cooling rate may deliver fine or coarse grain size. The Al_2CuMg , $MgZn_2$ and other phases in the Al-Zn-Mg-Cu alloys are hardening phases. Vacancy conglomeration and interaction within the α -Al atoms play significant influences on the precipitation kinetics [7]. From the characterization in figure 2 (a-g) it was evident that the experimental alloy was a high-Zn 7xxx alloy which was capable of delivering good mechanical properties [10]. Figure 2 (a) revealed 18.17Zn-5.71Cu in α -Al with 3.12Si, 6.29C and voids.

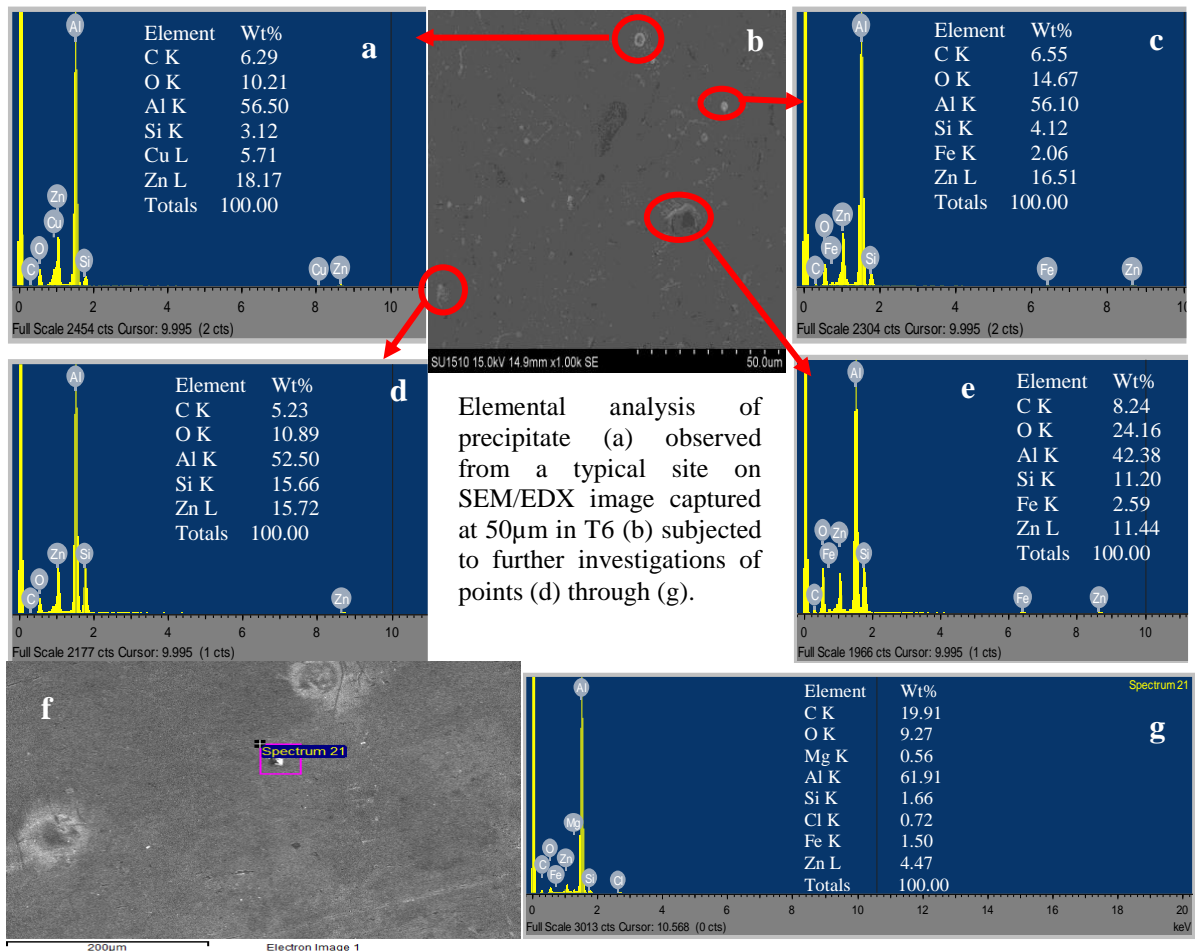


Fig. 2 - SEM/EDX characterization of typical imperfections observed during morphological investigation of an alloy of Al- 5 wt% Zn-1.75 wt% Mg after T4 treatment

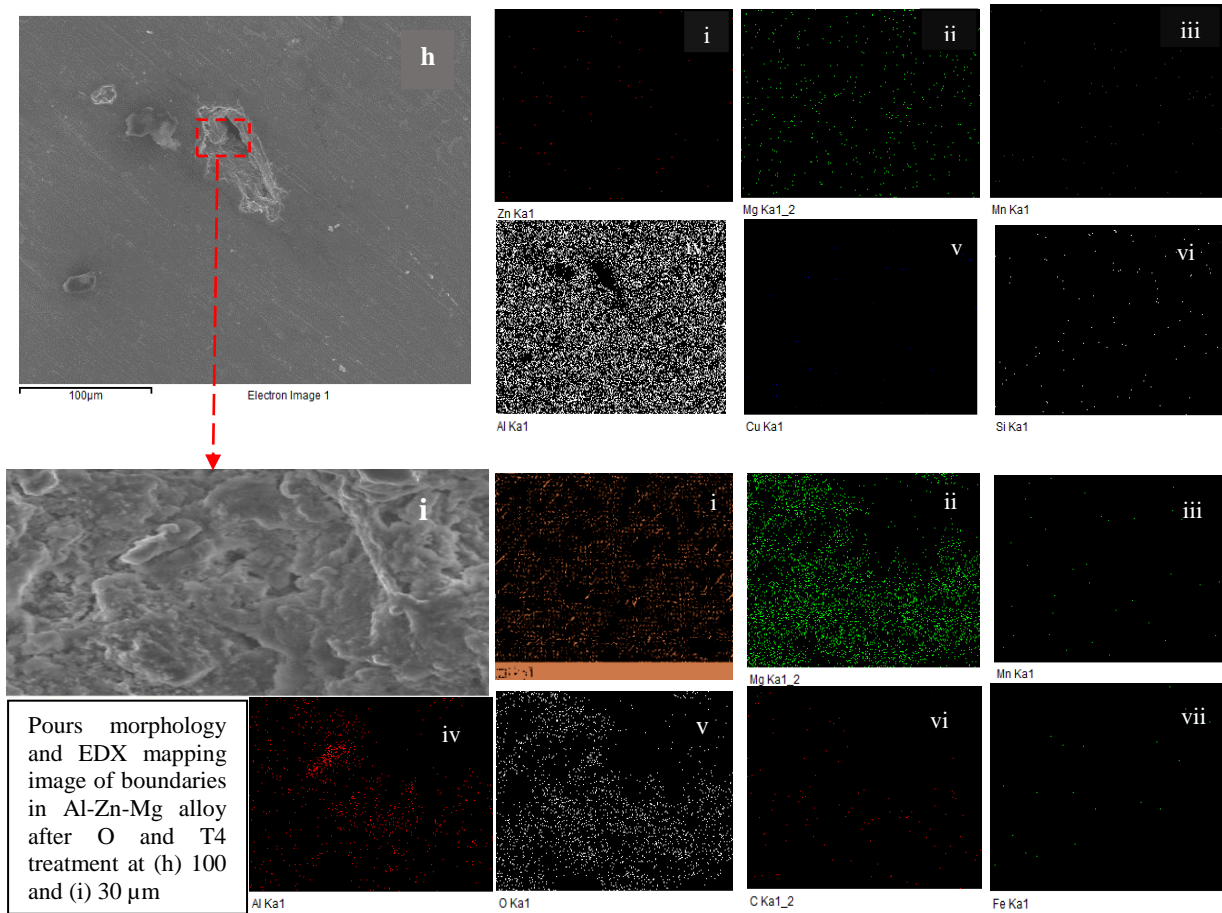


Fig. 3 - SEM/EDX characterization of typical imperfections observed during morphological investigation of an alloy of Al- 5 wt% Zn-1.75 wt% Mg after T4 treatment

Further, characterization (2c) was similar to (a) and supports the formation of more $\eta(\text{MgZn}_2)$ phases at the same heat treatment temperature. $\text{Al}_{2.06}\text{Fe}_4$, $\alpha\text{-AlFeSi}$, $\text{Al}_7\text{Cu}_2\text{Fe}$, $\theta(\text{Al}_2\text{Cu})$, and $\beta\text{-AlFeSi}$ phases are formable in the non-equilibrium solidification process. The 15.66 wt% of Si in (d) forms Mg_2Si [11]. The gradual dissolution of the phases during the heat treatment left a precipitate like (e) the morphology presented in (f) after polishing revealed the presence of Mg, Si, Cl and Fe-impurities, with C which may be from the graphite crucible and O in form of microvoids. In the presence of impurities, Wang et al., [12] reported the formation of $\text{S}(\text{Al}_2\text{CuMg})$, $\text{T}(\text{Al}_2\text{Mg}_3\text{Zn}_3)$ and $\theta(\text{Al}_2\text{Cu})$ with the addition of Zr-0.5Er to Al-Zn-Mg-Cu alloy under as-cast and homogenization conditions. These phases are functions of chemical composition of the alloy and non-equilibrium freezing process.

During solidification and homogenization processes, crystal structures, dislocations and microstructure defects which may include hydrogen evolution, air bubbles may surround the reinforcement particles [13]. The result of mapping in 3 h and i (i-vii) was a typical of such defects formed and that remains even after the O and T4 conditions. The mapping in h(i) shows Zn evenly dispersed within the $\alpha\text{-Al}$ and the micro void, whereas (ii), (iii) and (iv) represent Mg, Mn and Al matrix respectively. The presence of Cu and Si were shown in the mapping. Although the statistical analysis of wt% elemental investigation might not report the presence of Mn, the mapping did. Similar case was revealed in Cu, even as Si was partly dispersed in the $\alpha\text{-Al}$ matrix and the micro void.

An in-depth characterization of the void was presented in (i) from 100 µm to 500 µm. Such typical voids in casting was similar to the one reported in the study by [14] and was suggested to be due to various reasons. The mapping revealed the presence of Cu, Mg, Mn, Al, O, C and Fe elements. Pores within the Al-matrix was common in aluminium alloy cast. Apparently, the higher levels of Mg depicting lower Zn/Mg ratio may support the formation of T phase ($\text{Mg}_3\text{Zn}_3\text{Al}_2$ or $\text{Mg}_{32}(\text{Zn}, \text{Al})_{49}$) [15].

3.2 Macrohardness and Electrical Conductivity

The result of macro hardness and conductivity presented in figure 4 revealed almost an inverse relationship between electrical conductivity and macro hardness. Sample S1 was an alloy of 5 wt. % Zn recorded 3.67647×10^7 S/m conductivity and 72.38 HV in as cast condition.

When annealed, the alloy experienced a decrease in hardness, dropping to 50.30 HV while conductivity remained almost unchanged. The difference of 22 HV in hardness may be linked to the microvoids and pores in the casting route, hence conductivity was almost equal. In the T4 condition, an increase in hardness was observed to 119.76 HV, whereas the T6 condition recorded an improvement to 122.94 HV. During the annealing, the alloy expectantly recovered from the machining and cutting effects which might lead to recrystallization and grain growth [16]. From the characterization in Figure 2 and mapping in Figure 3, coarse interdendritic eutectic compound are possible and the heat treatment dissolved them back to the α -Al matrix during homogenization. This improved the mechanical properties of the alloys. For instance $MgZn_2$ when dissolved and evenly dispersed in Al matrix improves hardness [12].

The variation in electrical conductivity observed between S1 and S2 may be as a result of the impurities and the occurrence of microshrinkage during the casting process [17]. Sample S2 in AC recorded an electrical conductivity of 3.55872×10^7 S/m and hardness of 87.36 HV. A slight drop in conductivity was observed between AC and O conditions with a corresponding decrease in hardness. The T4 and T6 conditions recorded an improvement in mechanical property in contrast to the conductivity. A trace of the curve in Figure 4 revealed a conductivity of 3.55872×10^7 S/m and 82.92 HV was linked to T4 whereas 3.55872×10^7 S/m was associated with 96.46 HV in the T6 condition. The result depict a nonlinear relationship and aligns with the report of strength and electrical conductivity for AA 7075 in [18].

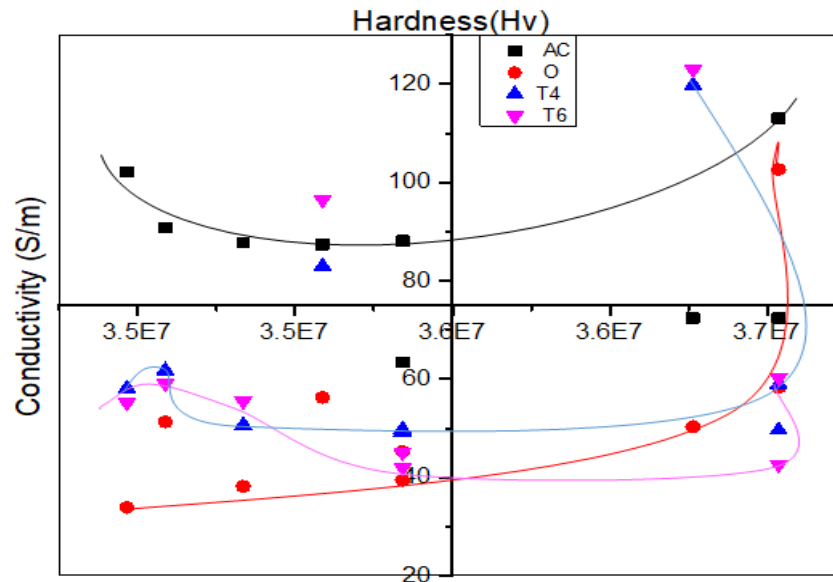


Fig. 4- Hardness and electrical conductivity curve of X7475 experimental alloy before (the black curve marked As-Cast indicate before heat treatment) and after heat treatment

Alloy composition, pouring speed, melting temperature and heat treatment type are key factors responsible for the dimension, distribution and the coherency or otherwise of the β precipitates formed in the α -Al matrix. However, in the case of S3, an alloy of 4 wt.% Zn, the effect of composition may not be prominent since the AC alloy recorded the obtainable peak hardness of 113.06 HV and 3.7037×10^7 S/m conductivity at 20 °C at the expense of the 102.60 HV observed in the O conditioned alloy. The drop in hardness from AC may be linked to the reversal of the hardening effect of machining and materials handling. Since surface microstructure alterations are in the form of plastic deformation, micro-cracks, phase transformation, microhardness, recrystallization, tears and residual stress may affect the micro structure and mechanical properties of aluminium, even responsible for phase composition in our multiphase alloy [19]. The T4 and T6 recorded 3.7037×10^7 S/m, 58.94 HV and 3.7037×10^7 S/m, 60.26 HV respectively. With the pores observed in figure 3, the indenter might punch a void which may be responsible for variation the mechanical properties.

In alloys S4, S5 and S6, Zn wt. % was held at 5 wt. % while Mg varied between 1.00-1.50 wt. % and Mn (0.025 - 0.075 wt. %). These alloys recorded close values in both hardness and electrical conductivity alongside heat treatments. Deducible here was that variation in wt.% of Zn and Mg results in the formation of similar second-phase may deliver comparable mechanical properties [9]. Noticeable in figure 4 was alloy S9 which recorded 3.4965×10^7 S/m conductivity and 102.10 HV. The alloy recorded the least hardness of 33.90 HV in O condition and 55.22 HV in T6 condition, whereas conductivity remains almost unchanged in the T4 and T6 conditions.

4. Hardness vs Conductivity Analysis And Modelling

Although a nonlinear relationship was obvious between hardness and conductivity. The curve fittings was done after sieving the non-significant terms from the data. The following analysis was a statistical description of the relationship between conductivity and macrohardness of our novel X7475 alloy produced from recycled beverage cans. From the analysis, it was clear that the relationship between HV and C selected was appropriate for the observed data. Linear model suffice in fitting all the data because $p > 0.05$ for all conditions.

Table 2 - ANOVA and linear models

Analysis of Variance for As Cast									
Source	DF	Adj SS	Adj MS	F-Value	P-Value	Regression equation			
C	6	772.0	128.7	0.23		$HV=86.96 + 15.1 C_{3.496} \times 10^7 + 3.8 C_{3.508} \times 10^7 + 0.7 C_{3.533} \times 10^7 + 0.4 C_{3.558} \times 10^7 - 11.2 C_{3.584} \times 10^7 - 14.6 C_{3.676} \times 10^7 + 5.7 C_{3.703} \times 10^7$			
Error	2	1132.3							
Total	8	1904.3							
Analysis of Variance for O									
C	6	2339.5	389.9	0.78	0.656	$HV1=50.37 - 16.5 C_{3.496} \times 10^7 + 0.9 C_{3.508} \times 10^7 - 12.2 C_{3.533} \times 10^7 + 5.9 C_{3.558} \times 10^7 - 8.0 C_{3.584} \times 10^7 - 0.1 C_{3.676} \times 10^7 + 30.1 C_{3.703} \times 10^7$			
Error	2	999.2	499.6						
Total	8	3338.7							
Analysis of Variance for T4									
C	6	4293.45	715.57	32.98	0.030	$HV2=68.14 - 10.02 C_{3.496} \times 10^7 - 6.36 C_{3.508} \times 10^7 - 17.50 C_{3.533} \times 10^7 + 14.78 C_{3.558} \times 10^7 - 18.67 C_{3.584} \times 10^7 + 51.62 C_{3.676} \times 10^7 - 13.85 C_{3.703} \times 10^7$			
Error	2	43.39	21.70						
Total	8	4336.84							
Analysis of Variance for T6									
C	6	5854.2	975.70	12.09	0.078	$HV3=69.16 - 13.94 C_{3.496} \times 10^7 - 10.12 C_{3.508} \times 10^7 - 13.66 C_{3.533} \times 10^7 + 27.30 C_{3.558} \times 10^7 - 25.62 C_{3.584} \times 10^7 + 53.78 C_{3.676} \times 10^7 - 17.74 C_{3.703} \times 10^7$			
Error	2	161.4	80.71						
Total	8	6015.6							
Coefficients for As Cast					Coefficients for Solution Treated (O)				
Term	Coef	SE Coef	T-Value	P-Value	Term	Coef	SE Coef	T-Value	P-Value
Constant	86.96	8.33	10.44	0.009	Constant	50.37	7.82	6.44	0.023
34965000	15.1	21.8	0.70	0.559	34965000	-16.5	20.4	-0.81	0.505
35087700	3.8	21.8	0.17	0.878	35087700	0.9	20.4	0.04	0.970
35335700	0.7	21.8	0.03	0.976	35335700	-12.2	20.4	-0.60	0.611
35587200	0.4	21.8	0.02	0.987	35587200	5.9	20.4	0.29	0.802
35842300	-11.2	16.5	-0.68	0.566	35842300	-8.0	15.5	-0.52	0.655
36764700	-14.6	21.8	-0.67	0.572	36764700	-0.1	20.4	-0.00	0.998
Coefficients for T4 heat treated					Coefficients for T6 heat treated				
Constant	68.14	1.63	41.81	0.001	Constant	69.16	3.14	22.00	0.002
C					C				
34965000	-10.02	4.26	-2.35	0.143	34965000	-13.94	8.22	-1.70	0.232
35087700	-6.36	4.26	-1.49	0.274	35087700	-10.12	8.22	-1.23	0.343
35335700	-17.50	4.26	-4.11	0.054	35335700	-13.66	8.22	-1.66	0.238
35587200	14.78	4.26	3.47	0.074	35587200	27.30	8.22	3.32	0.080
35842300	-18.67	3.23	-5.79	0.029	35842300	-25.62	6.22	-4.12	0.054
36764700	51.62	4.26	12.12	0.007	36764700	53.78	8.22	6.54	0.023

5. Conclusion

Imperfections and formation of new phases are inherent in the experimental alloys owing to the recycled source of raw materials and the stir casting process adopted in this study. Elemental analysis and mapping depicts expected hardening phases are formed and affects the macro hardness and electrical conductivity of the novel material. However, the relationship between hardness and conductivity was nonlinear and it was a pointer that conductivity in this alloy may be difficult to be predicted using electrical conductivity. 5 wt. % Zn recorded 3.67647×10^7 S/m conductivity and 72.38 HV in as cast condition. The T4 and T6 recorded 3.7037×10^7 S/m, 58.94 HV and 3.7037×10^7 S/m, 60.26 HV respectively. The linear model so developed was sufficient in predicting the macro hardness of the alloy in the as-cast, annealed, natural aging and artificial aging conditions respectively. The alloy may find application in the production of automobile bumper frame. Future research may focus on eliminating the variations in Mg and Mn. The need for further investigation of the imperfections, optimization of mechanical properties and additional mechanical properties investigation are required.

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