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EFFECTS OF FERRO–SILICON ADDITION, COLD ROLLING AND HEAT–TREATMENT ON POST–DEFORMATION QUALITY AND CORROSION BEHAVIOUR OF 6063 ALUMINIUM ALLOY

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Abstract: 6063 aluminium alloy is widely used in architectural applications such as window and door frames because of its nice appearance and acceptable strength. Its major alloying elements are magnesium and silicon. Up to now, very few studies focused on the effect of the alloy content, and cold rolling on post deformation quality and corrosion properties of the alloy have been published, considering the high impact of chemical composition changes on the said properties. Thus, this work investigates the influence of Ferro–Silicon addition, heat–treatment and cold rolling on post–deformation quality and corrosion behaviour of 6063 Aluminium Alloy. 5000 g of Aluminium 6063 alloyed with 500 g of ferrosilicon was produced. Cast samples from the alloy were homogenized at 350 ºC before cold rolling in the range of 10%, 15%, 20%, 25%, and 30% reduction. The corrosion resistance of the samples was investigated using a potentiodynamic polarization test in a 3.5wt % sodium chloride solution. The results show that the addition of FeSi content and cold rolling results in improved mechanical properties but have little effect on corrosion resistance.
Keywords: aluminium; ferro silicon; cold rolling; heat treatment; corrosion

1. INTRODUCTION

Aluminum is used widely in many sectors such as chemical plant, manufacturing lines, architectural design, and marine industries. Although it is easy to fabricate aluminium into any form, its application for engineering purposes is restricted due to its softness. Hence the need to strengthen this metal to improve its usefulness. Cold rolling and heat treatment are techniques used to improve mechanical properties of materials and enhance their performance on application but an important side effect of these techniques is that they may cause deterioration of corrosion resistance of the material (Stephen, 2003).

Appropriately alloyed and treated aluminum can withstand corrosion by water, salt, and other environmental factors (Davis, 2001). Intermetallic such as Al–Fe–Si and Al–Fe–Si–Mn have been reported to have significant influence on mechanical properties of 6XXX aluminium if properly controlled (Nowotnik 2012).

Rana, *et al*., (2012) has established that the low density of silicon helps to reduce the total weight of cast components while the solubility of silicon in aluminium increases the abrasion resistance. Majority of alloys are susceptible to intergranular corrosion when exposed to specific environment because grain boundaries are site for precipitation and segregation (Phull, 2003).

Milind et al., (2015) studied the impact of silicon content on mechanical properties of aluminium alloys. Al–Si alloys were prepared by melting pure aluminium with pure silicon and held at 720ºC to obtain homogenous mixture. They varied the silicon content from 5, 7, 9, 12.5 and 14% in five different aluminum alloys. It was observed that the silicon content in the alloy increased the ultimate tensile strength of the alloy to the maximum value of 175 MPa at 14 wt% silicon. However, the research did not consider the effect of cold rolling on the mechanical properties of the aluminium alloy.

Gupta et al., (2001) studied the effect of precipitation hardening in Al–Mg–Si alloys with and without excess Si. Magnesium and silicon were varied in different proportions to produce different alloys, homogenized at 560ºC and it was hot and cold rolled to 1mm thick sheet. The cold rolled samples were solutionized at 560ºC, rapidly cooled and naturally aged for three days. It was observed that excess of silicon enhanced the precipitation kinetics and improved the strength until the overall Mg/Si ratio in the alloy was close to 0.4. The hardness increased rapidly to the maximum at approximately 260ºC – 270ºC and then fall rapidly beyond this temperature. However, the corrosion behaviour of the alloy were not studied and mechanical test was limited to hardness test.

Gundu et al., (2016) studied the effect of cold rolling and annealing treatments on microstructure, impact and toughness and corrosion resistance of Cu–12Al–2Ni–5Fe alloy which was cold rolled to 5%, 10%, 15% and 20% reductions and annealed at temperature of 350ºC, 400ºC, 450ºC and 500ºC. It was simulated in seawater under flow condition using open circuit potential and potentio–dynamic polarization. It was observed that, as the degree of cold rolling and heat treatments of the alloy increased there was higher impact strength and reduction in corrosion rate.

Alexander *et al*., (2013) and Israfil (2018) investigated the effect of cold rolling on the microstructure and pitting resistance of the NBR Iso 5832–1 Austenitic stainless steel with deformation corresponding to 30%, 50% and

70% reduction by cold rolling using potentio–dynamic polarization. It was observed that 30% and 50% reduction decreased the corrosion resistance of stainless–steel causing inclusions and voids at the matrix–inclusion interface. However higher level of reduction 70% improved localized corrosion resistance, the void created by fragmentation and the matrix – inclusion interface was shielded with matrix material. The study did not consider the mechanical properties of the samples.

Sekunowo *et al*., (2015) investigated corrosion propensity of cold deformed 5052 Aluminium alloy in seawater. They concluded that a highly deformed sample is more prone to corrosion than the fairly deformed and undeformed samples. Adeosun *et al*., (2014) investigated combined effects of rolling and heat treatment on the mechanical properties of aluminium–titanium alloy. They established that rolling and heat treat treatment of Al–Ti alloy enhanced tensile strength by 24.7% and hardness by 20.5%. However, the work did not consider the effect of rolling and heat treatment on corrosion behaviour of the alloy.

Chuan–bo *et al*., (2016) investigated the effect of heat treatment on corrosion resistance of 6061 Aluminium alloy using 3.5% NaCl solution and varying heat treatment by slow strain rate equipment, polarization curves and impedance. It was established that the solution treated 6061 aluminium alloys had a higher hardness and lower corrosion resistance but a decrease in hardness improve the corrosion resistance of the alloys. However, the work did not look into the effect of rolling on corrosion behaviour.

Cevik *et al*., (2012) Isadare *et al*., (2015) studied the effect of peak aged heat treatment on corrosion behavior of the 6063 alloy containingAl_3Ti using 30gr/l NaCl + 10ml/l HCl solution at room temperature of 20 to 23ºC at various time interval for the corrosion test. It was observed that heat treatment improved corrosion resistance. Prabhu and Padmalatha, (2013) and Peter and Jan (2015) has established that, post mechanical working such as drawing and straightening impaired corrosion resistance of the metal.

Adeosun *et al*., (2010) studied the Effects of Heat Treatment on Strength and Ductility of Rolled and Forged Aluminum 6063 Alloy. The result obtained showed that combination of improved strength and elongation of 127MPa, 24% respectively can be obtained in rolled sample when solution heat treatment (SHT) was applied after deformation and cooling in water. Adeosun *et al*., (2011) has also established that cold working of 6063 Aluminium alloy increased its hardness.

It is known from the literature source that intermetallic such as Al–Fe–Si have significant influence on mechanical properties of 6XXX aluminium alloy if properly controlled. However, the effect of the alloy content and cold rolling on post deformation quality and corrosion properties of the alloy is rarely studied. Hence, considering the high impact of chemical composition changes on the said properties, it is of interest to study the influence of Ferro–Silicon addition, cold rolling and heat–treatment on post–deformation quality and corrosion behaviour of 6063 AlumINIUM ALLOY.

2. Experimental Methodology

Al–Si alloys were prepared by melting 5000 grams of 6063 aluminium with 500 grams of ferrosilicon in a crucible furnace. The melted 6063 aluminium alloy and the grinded ferrosilicon were stirred thoroughly to obtain a homogenous mixture and held at 720ºC. The molten metal was poured into cylindrical metal dies of diameter 16 mm and length/height 140 mm. Five samples were cast at a time.

Cast samples from the alloy were homogenized at 350 ºC before cold rolling in the range of 10%, 15%, 20%, 25%, and 30% reduction using a laboratory rolling mill. One of the samples not cold– rolled served as control. The machined samples for each of the reductions were heat treated using carbolite electric arc furnace. The samples were heated to 500°C and held for one hour and finally quenched in tap water.

Microhardness test was carried out using a test load of 490.3N and the dwell time for the test was 10s. The test was done at 3 different points on each of the samples. The average hardness was then calculated. Impact test was carried out using the Charpy impact testing machine, made in England, Serial no: 3915. The test was carried out on 10 mm x 60 mm test pieces with a V notched angle of 45o at 27°C while maintaining a uniform striking velocity. Tensile test was carried out using a Universal Instron Machine, model 3369.

Corrosion test was carried out using electrochemical process. Electrochemical measurements tests were performed using a Gamry's conventional three electrode cell system (model PC14/750 Potentiostat/ Galvanostat/ZRA), in 0.6M NaCl solution of pH 5.5 according to ASTM G69–12, aerated for 60 minutes by magnetic mixer, before start. The cell was assembled with the 6063 aluminium alloy sample as working electrode (WE). A saturated calomel electrode was used as a reference (RE) and a graphite rod was used as an auxiliary electrode (AE). Exposed areas for working and auxiliary electrode were 0.785 cm^2. The open–circuit potential (OCP) was recorded for 6000 seconds to investigate how long the OCP value of the samples reaches a steady state. Polarization curves were recorded in a single sweep starting from –0.5 V versus OCP and goes as high as 0.5 V versus OCP. The scan rate was 0.20 mV/s. The measurements were conducted at room temperature

(25°C). From this experiment, icorr and Ecorr values were measured using Tafel plot and the corrosion rate was determined.

Specimens for microstructural analysis were prepared by grinding with silicon carbide emery paper and thereafter polishing the samples with Grinding and Polishing Machine QPOL 250 M2 followed by etching with diluted hydrofluoric acid while the microstructural analysis was performed using scanning electron microscope.

3. RESULTS AND DISCUSSION

— Chemical Composition of the 6063 Aluminium Alloy

Chemical composition of the investigated alloys in wt %, measured via optical emission spectrometry (SPECTROMAXx from SPECTRO, Kleve, Germany) before and after alloying with ferrosilicon is given in Table 1. Figures 1 and 2 shows the XRD Results of the as received and alloyed Al 6063 respectively.

Table 1: Chemical composition of 6063 aluminium alloy

Elements	Composition (wt %)	
	6063 aluminium	6063 aluminum alloyed with ferrosilicon
Si	0.45	2.34
Fe	0.22	0.65
Mn	0.01	0.01
Mg	0.54	0.48
Zn	0.01	0.01
Na	0.03	0.02
Ti	0.01	0.02
Others	0.22	0.01
Al	98.70	96.14

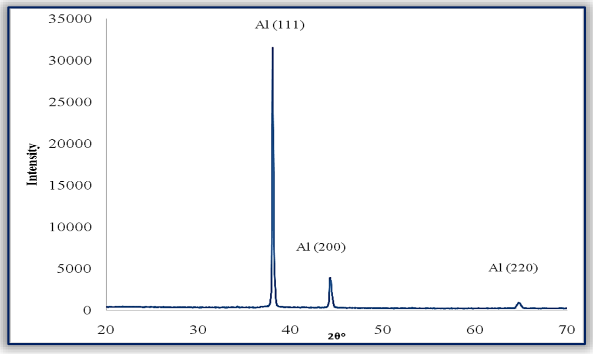

Figure 1. XRD pattern of as received Al 6063

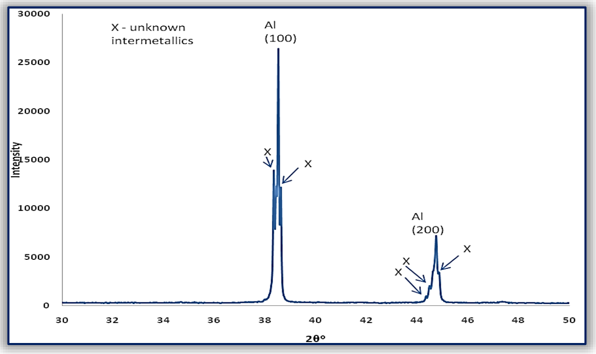

Figure 2. XRD pattern of Al 6063 alloyed with ferrosilicon

From Figures 1 and 2, the Al–Si–Fe phase present at about angle 38° is in consonant with that reported by (Birol 2012, and kumar *et al*., 2012). It would be observed that the concentration of Si and Fe increased indicating that alloying with ferrosilicon was successful. The presence of tin and copper may indicate the presence of impurity from the ferrosilicon. Also, there was reduction in the magnesium which may be due to loss of some element during homogenous mixing. The concentration of other elements such as Mn and Zn did not change.

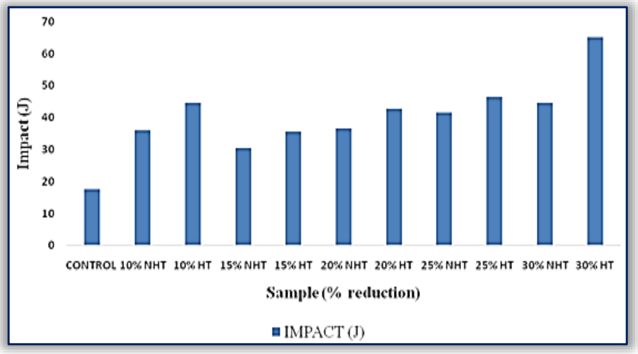

Figure 3. Impact Stress of Heat treated and non–heat treated cold rolled samples

— Mechanical characterization

To evaluate the mechanical properties of the alloys, impact, hardness and tensile tests were carried out. The results are presented in figures 3, 4 and 5 respectively.

The experimental results obtained from Vickers hardness measurements indicate that a higher value of hardness has been observed for alloy with FeSi addition in comparison to the as–received alloy. A rise in hardness shows that FeSi has acted as a grain refiner since alloyed samples has a significantly smaller grain size in comparison with non–alloyed samples.

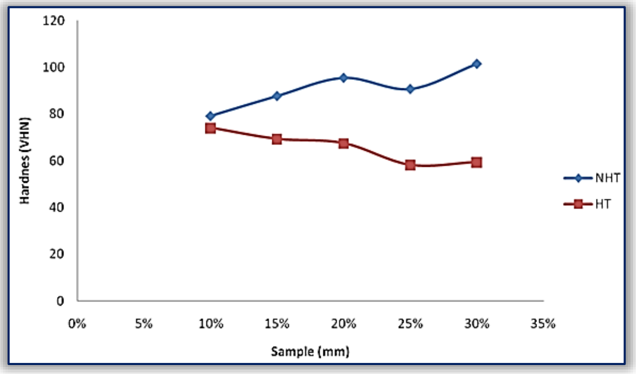

Figure 4: Comparison of heat–treated and non–heat–treated cold rolled Samples

From Figure 5, it was observed that the cold–rolled plus heat–treated samples had higher maximum tensile stress compared to the non–heat–treated samples with the exception of the 30 % cold rolled sample which may be due to presence of the metastable ζ–phase of leboite.

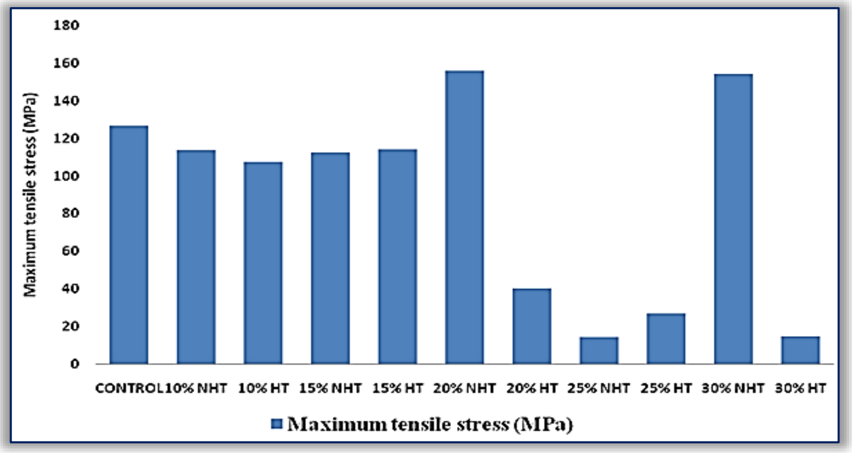

Figure 5: Maximum Tensile Stress of Cold Rolled and Heat–treated samples

The alloys with added FeSi show higher strength levels for all degrees of cold–rolling and heat treatment, while alloys without FeSi addition exhibits lower strength. Additions of the dispersoid FeSi content results in improved mechanical properties.

— Corrosion Characterization

Figure 3 presents the results of the Tafel Polarization curves of aluminium alloy, cold rolled at 10%, 20%, and 30% reductions in 3.5wt % NaCl (synthetic saline environment) while Figure 4 shows the Polarization curves of cold rolled and heat treated aluminium alloys. A summary of the experimental data obtained from potentiodynamic corrosion tests is summarized in Table 3.

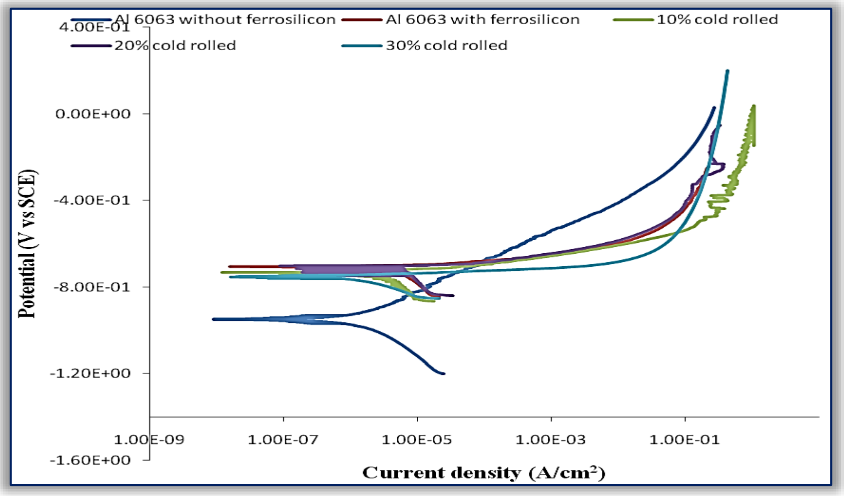

Figure 6: Potentiodynamic polarization curve of cold rolled aluminium alloys in 3.5wt % NaCl solution

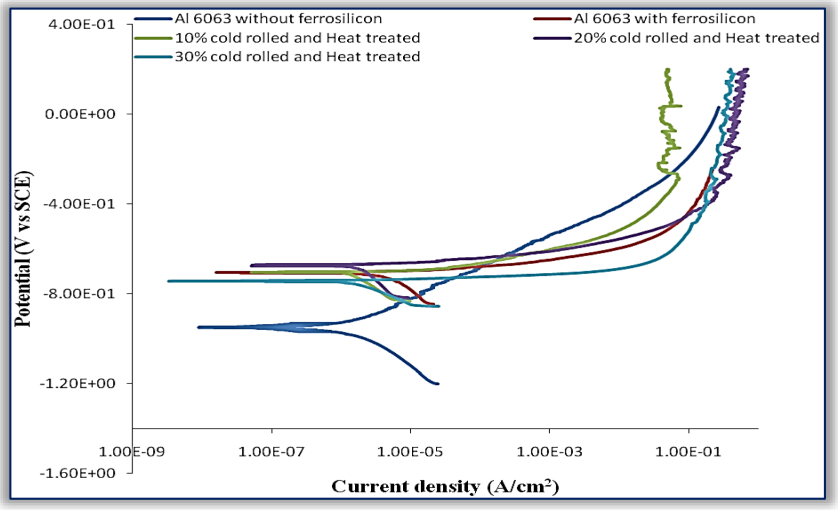

Figure 7: Potentiodynamic polarization curve of cold rolled and heat treated aluminium alloys with various degrees of cold rolling

From table 3, it can be seen that the as–received aluminium 6063 sample displayed the highest resistance to corrosion compared with the alloyed and cold rolled sample. Among the cold rolled samples, the 20 % reduction samples exhibited the highest corrosion resistance. This conforms to Sekunowo *et al*., (2015) who concluded that deformed sample due to cold rolling is more prone to corrosion than undeformed sample and this is attributed to initiation of corrosion sites. However, heat treatment improved the corrosion resistance of the samples.

Table 3: Summary of the experimental data derived from the potentiodynamic polarization curves of the Tafel method

	Cold Rolled		Cold Rolled and Heat treated	
Samples	Ecorr (v)	i_{corr} (A/cm^2)	Ecorr (v)	i_{corr} (A/cm^2)
As received Al 6063	−0.951	1.6×10^{-6}	−0.951	1.6×10^{-6}
Al 6063 alloyed with ferrosilicon	−0.704	3.93×10^{-6}	−0.704	3.93×10^{-6}
10 % cold rolled	−0.731	6.43×10^{-6}	−0.703	2.56×10^{-6}
20 % cold rolled	−0.715	9.63×10^{-6}	−0.676	3.22×10^{-6}
30 % cold rolled	−0.753	4.87×10^{-6}	−0.742	2.94×10^{-6}

— Microstructural analysis

Plates 1–7 shows SEM Micrographs of the cold rolled aluminium 6063 alloy and heat treated cold rolled alloy.

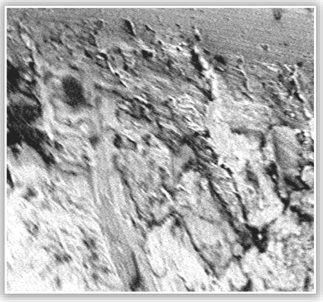
Plate 1: 10 % cold rolled sample

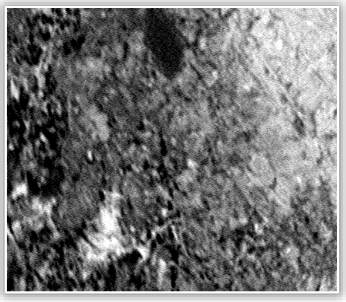
Plate 2: 10 % cold rolled and heat–treated sample

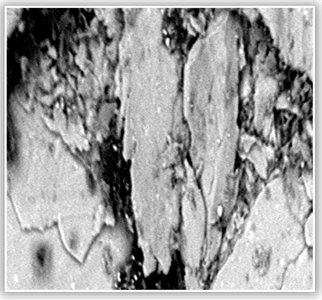
Plate 3: 20 % cold rolled sample


Plate 4: 20 % cold rolled and heat treated


Plate 5: 30 % cold rolled sample


Plate 6: 30% cold rolled and heat treated


Plate 7: as received

Plates 1–7: SEM micrographs of cold rolled and heat–treated samples sample (200μm)

The 30% cold rolled plus heat–treatment alloy yields grains with uneven grain boundaries and broad grain size distribution, illustrated in Plate 6.

The microstructure after heat–treatment show spherical grains with straight grain boundaries (See Plates 3 and 4). The grain size variation is considerably reduced after heat–treatment. 20% cold rolled alloy exhibit relatively large grains, while grains in 30% cold rolled alloys are comparatively small.

The microstructure of the alloy contains constituents of type Al_xFeSi_y, and metastable ζ–phase of leboite having the composition FeSi 2, 3 or $Fe_3 Si_7$ which are formed during solidification. Their presence is important not only for grain size control during plastic deformation and heat treatment, but also for the creation of mobile dislocations.

4. CONCLUSIONS

In this work, the effects of Ferro–Silicon addition, cold rolling and heat–treatment on post deformation quality and Corrosion behaviour of 6063 aluminium alloy has been presented.

- SEM images and XRD results have shown the presence of constituents of type Al_xFeSi_y, and metastable ζ–phase of leboite having the composition $FeSi_{2,3}$ or $Fe_3\ Si_7$.
- Alloys with FeSi addition show higher strength levels for all degrees of cold–rolling and heat treatment while alloys without FeSi exhibits lower strength.
- As the degree of cold rolling and heat–treatment of the alloy increased, there was higher impact strength and reduction in corrosion resistance
- Microhardness tests have corroborated that the sample composed of FeSi presents better mechanical properties with a higher value of hardness and tensile stress which is associated to grain size reduction.
- Addition of FeSi has not caused significant difference in corrosion behaviour of the alloy. However, tearing of the samples during cold rolling at high degree of deformation was observed.

References

[1] Adeosun S. O., Balogun S.A., Sekunowo O.I. and Usman M.A. (2010) "Effects of Heat Treatment on Strength and Ductility of Rolled and Forged Aluminum 6063 Alloy", Journal of Minerals & Materials Characterization & Engineering, Vol 9 (8), 763–773.
[2] Adeosun S.O., Sekunowo O. I. and Talabi S.I. (2014) "Combined effect of cold rolling and heat treatment on the mechanical properties of Al–Ti alloy", World Academy of Science, Engineering and Technology International Journal of Materials and Metallurgical Engineering, Vol 8 (3), 235–240.
[3] Adesola, A.O., Odeshi, A.G., and Lanke. U.D. (2013) "The effects of ageing treatment and strain rates on damage evolution in AA 6061 aluminium alloy in compression", Materials and Design. Vol 45, pp 212–221.
[4] Adeosun S.O., Sekunowo O.I., Balogun S.R. Osoba L.O. (2011) "Effect of deformation on the mechanical properties of Aluminium– Magnesium Alloy", Journals of minerals and materials characterization and engineering, Vol 10 (6), 553–560.
[5] Alexander H.R., Cristiaann H.R., Isolda C. (2013) "Cold rolling effect on the microstructure and pitting resistance of the NBR ISO 5832–1 Austenitic Stainless steel", International Journal of Electrochemical Science, Vol 8, pp 12810–12815
[6] Cevik E., Sun Y., Ahlatei H. 2012 Effect of peak–aged heat treatment on corrosion behaviour of the 6063 alloys containing Al3Ti, Archives of metallurgy and materials Vol 57, pp 469–477.
[7] Chuan–bo Z., Xi C., Chun–ling L., Xiao–Lan S., Ke C. 2016 International Journal of Electrochemical Science Vol 11, pp 7254 –7261, DOI: 10.20964/2016.08.35
[8] Davis J.R2001 "Aluminium and Aluminium alloys", ASM International pp.351–352
[9] Davis J.R 2001 "Aluminium and Aluminium alloys", ASM, Metals Handbook Vol 13 Corrosion Fundamentals and protection.
[10] Gundu D.T., Tile J.M. Folrunsho D.O. 2016 "Effect of cold rolling and annealing treatments on microstructure, impact, toughness and corrosion resistance of Cu–12 Al–2Ni–5Fe NaB alloy", American journal of engineering research, Vol 5 (10), 307–310.
[11] Haque, M.M., Limon S.A., Moniruzzaman M.d., Ali Bepari M.d. (2014) "Corrosion comparison of galvanized steel and aluminium in aqueous environments" International Journal of Automotive and Mechanical Engineering, Vol 9, pp 1758–1767.
[12] Isadare D.A., Adeoye M.O., Adetunji A.R., Oluwasegun K.M., Akinluwade K.J. 2015 "The influence of homogenization treatment on the aging response of 6063 aluminium alloy", Advance in research international, Vol 5 (3), 1–11.
[13] Israfil K. (2018) "Effect of cold rolling reduction rate on corrosion behaviour of twin–roll cast 8006 Al alloys", Cumhuriyet Science Journal, Vol 39 (1), 233–242.
[14] Milind G.K., Pradeep M.D., (2015) "Impact of Silicon Content on Mechanical Properties of Aluminum Alloys" International Journal of Science and Research, Vol 4 (6), 2319–7064.
[15] Nowotnik G.M (2010) "Influence of chemical composition variation and heat treatment on microstructure and mechanical properties of 6xxx alloys", Archives of Materials Science and Engineering, Vol. 46 (2), 98–107.
[16] Omotoyinbo, J. A. and Oladele, I. O. (2010). "The effect of plastic deformation and magnesium content on the mechanical properties of 6063 Aluminium alloys", Journal of mineral and materials characterization and engineering, Vol. 9 (6).
[17] Philip A. and Schweitzer, P.E. 2003, "Metallic Materials, Physical, Mechanical and corrosion properties, Marcel Dekker, Incorporation pp571
[18] Phull B. (2003), Evaluating Intergranular Corrosion, Corrosion: Fundamentals, Testing, and Protection, ASM Handbook, ASM International, Vol 13A, 568–571
[19] Rana R.S., Rajesh P. and Das S. (2012) "Review on the Influence of alloying element on the microstructure and mechanical properties of Aluminium Alloy's", International Journal of Scientific and Research Publication Vol 2 (6).
[20] Sekunowo O.I., Durowaye S.I. and Anozie E. C. (2016) "Corrosion Propensity of Cold Deformed 5052 Aluminium Alloy in Seawater", British Journal of Applied Science & Technology Vol 8(1) 46–52.
[21] Stephen, D.C. and Bernard S.C. (2003), Corrosion: Fundamentals, Testing, and Protection, ASM International, Vol (13A), 1783.

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