



Influence of Potential Scan Rate on Corrosion Behaviour of Heat Treated AA 7075 Alloy in Sulphuric Acid Solution

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Abstract

In this paper, the effect of scan rate on the corrosion parameters and electrochemical behaviour of Retrogression and Re-aging (RRA) heat treatment of 7075 aluminium alloy has been investigated by Potentiodynamic polarization (PD) and electrochemical impedance spectroscopy (EIS) methods. The Potentiodynamic polarization (PD) curves were obtained at various scan rates for aged aluminium alloys in 0.5 M H₂SO₄ solution. The results show that at high scan rates shows unpredicted fluctuation of charging current, whereas at lower scan rates fewer disturbances are observed. The aged samples were chemically and mechanically characterized. It was observed that the RRA at 0.7 h shown improved corrosion property as well as mechanical properties compared to T6 temper. The corrosion properties were confirmed by EIS and polarization experiments.



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Introduction

Aluminium alloys of 7075 kind containing Zn, Mg and Cu as major alloying elements have shown potential applications in automobile and aerospace applications¹. They possess good corrosion and mechanical properties than other Al alloys². These alloys show specific behaviour in Retrogression and Re-aging (RRA) heat treatments³. The most important feature of the aluminium alloy is to form oxide on surface which act as a barrier⁴. This barrier

oxide breaks due to aggressive environments⁵ and thus study of the corrosion parameters of any alloy is required to understand to estimate the current and its reaction with a metal surface⁶. The electrochemical processes consist of anodic and cathodic reactions in addition have current to voltage relationships that follow Butler–Volmer kinetics⁷⁻⁸, which is applicable for a corroding system and is shown in Eq. 1.

$$I_{app} = I_{corr} \left[\left(e^{\frac{2.303(E-E_{corr})}{\beta_a}} - e^{\frac{-2.303(E-E_{corr})}{\beta_c}} \right) \right] \quad \dots(1)$$

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Where $(E - E_{corr})$ is the over potential; E and i_{corr} are the applied corrosion potential and corrosion current density; β_a is the anodic Tafel slope and β_c is the cathodic Tafel slope. Eq.1 indicates that unless the over potentials are small compared to the Tafel slopes, a large potentially damaging currents can be generated on the material. When E is distant from corrosion potential, there can be a similar relationship between the corrosion current and corrosion potential as per the Tafel equation⁹

$$E = \beta_a \pm \beta_c \log |i| \quad \dots(2)$$

Eq. 2 shows that the logarithm current is directly proportional to the applied potential at the high value of over potential and helpful for determining the corrosion current. The electrode in contact with aqueous solution tends to develop a potential difference across the interface. When metal dissolves to form ions, the metal becomes positively charged with respect to the solution. The positive ions in the solution form a layer of negative charges on the metallic surface. It means that this concept is analogous to the electrical behaviour of a capacitor. The charging current across the capacitor is directly proportional to the product of time derivative of the potential field (Scan rate) and interfacial capacitance. Thus, the effect of scan rate in the potentiodynamic experiment can be visualized. At very low scan rates, the charging current through the capacitor becomes small and at high scan rates, the fraction of charging current flows through the capacitor will be increased¹⁰. This effect will cause an underestimation of corrosion rate and may be incorrect. In this case, it is very important to know the correct polarization data without any kind of disturbance of charging current. When studying the corrosion behaviour the effect of potential scan rates are one's concern as it is crucial to opt a potential scan rate value. Various studies have performed to check the effect of the

scan rate on the corrosion behaviour of alloys¹¹. But not much work is carried out on the Al 7075 alloy. This paper discusses the effect of scan rates on the corrosion and electrochemical behaviour of RRA treated AA 7075 alloys in acidic environment.

Materials and Experimental Methods

Five specimens each of AA 7075 alloy in cylindrical shape of 14 mm (exposed surface area of 0.785 cm²) in diameter have been taken for study. All the samples were given suitable heat treatment to produce desired microstructures. Details of the heat treatment are mentioned in Table 1. All the samples were prepared using conventional metallography up to 0.25 micron diamond polishing. Etching was carried to reveal the microstructures using Keller's reagent (95 vol.% H₂O, 2.5 vol.% HNO₃, 1.5 vol.% HCl and 1.0 vol. % HF).

The Potentiodynamic Polarization (PD) and Electrochemical Impedance Spectroscopy (EIS) are measured using electrochemical characterization techniques (PGSTAT203N AUTOLAB). The three electrodes are immersed into electrolyte for electrochemical measurements. The saturated calomel electrode (SCE) as reference electrode, platinum electrode used as the counter electrode, and the aged sample served as the working in 0.5 M H₂SO₄ solution. An optical microscope (ZESIS inbuilt AXIO VISION Rel.4.8 software) used to capture the microstructure of aged samples. Hardness is measured by Vickers microhardness tester (Shimadzu-HMV-2E) with the applied load of 1 kgf.

Results and Discussions

Metallography Examination

The optical micrographs of the various aged samples are shown in Fig.1. Microstructure of the aged aluminium alloys shows, elongated grains in a particular direction, which is because of rolling

Table 1: Details of Heat treatment on aluminium alloy samples.

S. No	Sample	H Heat Treatment Processes
1	T6	470°C /1h+ Water Quenching+ 120°C /6 h
2	SHT	470°C/2h
3	RRA .5 h	470°C/2h+ Water Quenching+ 120°C/24 h+ 200°C/0.5h+ 120°C /24 h
4	RRA.7 h	470°C/2h+ Water Quenching+ 120°C/24 h+ 200°C/0.7h+ 120°C /24 h
5	RRA .9h	470°C/2h+ Water Quenching+ 120°C/24 h+ 200°C/0.9h+ 120°C /24 h

condition and shows wide and tiny grains. The wide grains may be un-recrystallized and small grains related to recrystallized, which look like pancake structure¹². The microstructure shows dark and light regions, which is attributed to the presence of intermetallic particles (dark) uniformly distributed in the aluminium matrix (bright)¹³.

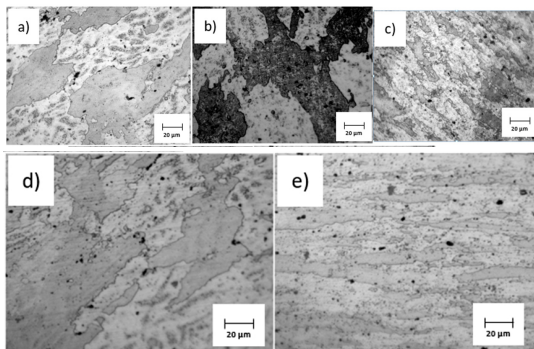


Fig. 1: Optical micrographs of aged samples (a) T6 (b) Solution Heat treatment (c) RRA 0.5 h (d) RRA 0.7 h (e) RRA 0.9 h.

Hardness Measurements

Fig. 2 shows the micro hardness measurements of Aged 7075 Aluminium alloy as a function of the various tempering process. It can be depicted from the figure, that the RRA at 0.7 h shown highest hardness¹⁴ than other samples. The reason behind the increase of hardness is due to formation of η (coherent) and η' (semi-coherent intermediate) phases in the aluminium matrix¹⁵. However, the rapid decrease of hardness at RRA 0.9h is due to the partially transformation of η' Phases to stable η phases in the alloys. It can be understood that the decrease of hardness further RRA0.7 h is due to over aging¹⁶.

Electrochemical Measurements

Fig.3 shows a plot against applied potential (mV) and logarithm plot of corrosion current density ($\mu\text{A}/\text{cm}^2$) obtained for 7075-T6 aluminium alloy. The polarization experiments were carried out at varied potential scan rates from 30mV/m into 240mV/min. The charging current across the capacitor is directly proportional to the product of time derivative of the potential field (Scan rate) and interfacial capacitance. Thus, the effect of scan rate in the Potentiodynamic experiment can be visualized.

From the potentio dynamic curve, it can be clearly seen that, T6-60, T6-120, and T6 -180 specimens showed a rapid decrease in corrosion current with respect to potential. It is attributed to the formation of protective oxide film on the metal surface. On the other side, T6-30 follows the lowest corrosion rate due to the formation of uniform passive layer and show a sudden decrease in the current density. The cathodic branches for T6 -120, T6 -180 and T6 -240 exhibits a linear trend in polarization curve.

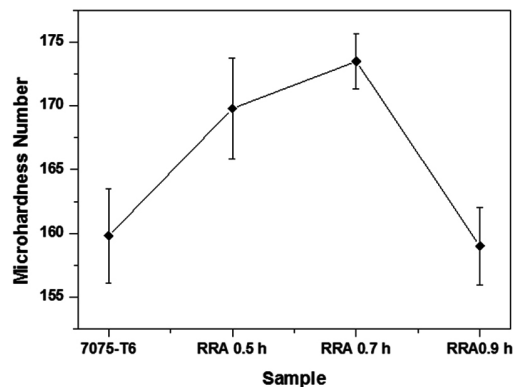


Fig. 2: Variation in the micro hardness of aged 7075 aluminium alloy under different temper process.

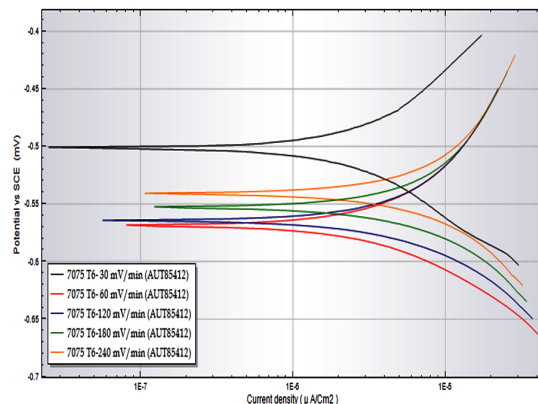


Fig. 3: Potentiodynamic curve of 7075-T6 temper on different potential scan rate in 0.5 M H_2SO_4 at room temperature.

Stern and Geary¹⁷ have simplified the kinetic expression to provide the charge transfer controlled reaction kinetics by Eq. 1 for the case of small over potentials with respect to E_{corr} . The Polarization Resistance (R_p) can be calculated from slopes at E_{corr} .

$$R_p = \frac{\Delta E}{\Delta I} = \frac{\beta_a \beta_c}{2.303 I_{corr} (\beta_a + \beta_c)} \quad \dots(3)$$

Rearranging of above equation, we get

$$I_{corr} = \frac{1}{2.303 R_p} \left(\frac{\beta_a \beta_c}{\beta_a + \beta_c} \right) \quad \dots(4)$$

Where all the electrode parameters have their usual significance, R_p (ohm-cm²) is the polarization resistance, B is proportionality constant, corrosion potential (E_{corr}) and I_{corr} is the corrosion current (A), EW is the equivalent weight of metal specimen (g), F is the Faraday constant (96500 C), d is the density of the metal (g/cm³). The polarization resistance values of various scan rates are determined from Stern-

Geary equation¹⁷⁻¹⁸ and the various parameters of corrosion for aged 7075 aluminium alloys are summarized in the below Table 2.

The data show that corrosion potential (E_{corr}) shifts to slightly higher negative values for low scan rate (30 mV/minutes) to high scan rate (240 mV/minutes). The change in the potential values could be possibly due to the crack of double layer on the interface¹¹. The difference between corrosion current and open circuit potential provides slight variation during the scan. The more difference between E_{corr} and E_{ocp} is due to the enrichment of disturbance of charging current⁶⁻¹⁹ which was observed at high scan rates. This charging current may affect both positive and negative direction. In the positive scan rate,

Table 2: Experimental corrosion data obtained for the heat treated samples

$I_{corr}(\mu A)$	$E_{corr}(mV)$	$E_{OCP}(mV)$	$E_{corr}-E_{ocp}(mV)$	CR (mm/yr)	7075-T6
2.41 X e ⁻⁷	-0.50		0.05	1.11	
8.08X e ⁻⁸	-0.56		-0.01	0.01	
5.64X e ⁻⁷	-0.56	-0.55	-0.01	0.02	
1.25X e ⁻⁷	-0.57		-0.02	0.58	
1.06X e ⁻⁷	-0.58		-0.03	0.49	
$I_{corr}(\mu A)$	$E_{corr}(mV)$	$E_{OCP}(mV)$	$E_{corr}-E_{ocp}(mV)$	CR (mm/yr)	SHT
6.4X e ⁻⁷	-0.58		-0.03	2.96	
1.5X e ⁻⁸	-0.62		-0.01	0.01	
3.2 X e ⁻⁸	-0.63	-0.61	-0.02	0.01	
2.4X e ⁻⁷	-0.62		-0.01	1.11	
2.2X e ⁻⁷	-0.64		-0.03	1.02	
$I_{corr}(\mu A)$	$E_{corr}(mV)$	$E_{OCP}(mV)$	$E_{corr}-E_{ocp}(mV)$	CR (mm/yr)	RRA0.5 h
1.74X e ⁻⁷	-0.57		0.01	0.80	
9.8X e ⁻⁸	-0.59		-0.01	0.01	
1.03X e ⁻⁷	-0.60	-0.58	-0.02	0.48	
8.75X e ⁻⁷	-0.60		-0.02	4.04	
1.82X e ⁻⁷	-0.61		-0.03	0.84	
$I_{corr}(\mu A)$	$E_{corr}(mV)$	$E_{OCP}(mV)$	$E_{corr}-E_{ocp}(mV)$	CR (mm/yr)	RRA0.7 h
6.4X e ⁻⁷	-0.55		-0.01	2.96	
5.69X e ⁻⁸	-0.55		-0.01	0.01	
1.53X e ⁻⁷	-0.56	-0.54	-0.02	0.71	
2.29X e ⁻⁷	-0.58		-0.04	1.06	
1.19X e ⁻⁷	-0.60		-0.06	0.55	
$I_{corr}(\mu A)$	$E_{corr}(mV)$	$E_{OCP}(mV)$	$E_{corr}-E_{ocp}(mV)$	CR (mm/yr)	RRA 0.9h
1.86X e ⁻⁷	-0.59		0.02	0.86	
6.98X e ⁻⁸	-0.62		-0.01	0.01	
2.27X e ⁻⁷	-0.63	-0.61	-0.02	1.05	
1.29X e ⁻⁷	-0.63		-0.02	0.60	
1.65X e ⁻⁷	-0.64		-0.03	0.76	

the charging current density increase by virtue of electron flow from electrode and current will move towards anodic faradic current. The polarization will act as cathodic and similarly the direction of charging current will be considered towards cathodic faradic due to the constant flow of electrons into aged sample. It means that corrosion potential shows positive open circuit potential values at lower scan rates. The discharging current density follow the same trend at higher scan rate (180 to 240 mV/minute) but slight decrease was observed at low scan rate (30 mV/min). It has been reported in the literature that the difference between corrosion potential and open circuit potential may reflect to high disturbance of the charging current. The same was observed by Poursaei *et al.*¹¹

The Potentiodynamic curve does not provide the appropriate measurement at rapid scan rate, which are representative of the system in the steady state²⁰. With increasing scan rate, current density slightly deviates from the steady state¹¹. The value of current density at 60 mV/minute gives good agreement as compared to other results obtained at other scan rates. It can be understood by Table 2, that the electrochemical system does not achieve steady state at a high scan rate and also with very low scan rate.

The data of T6 temper has been summarized from Potentiodynamic curve shown in Fig. 3. The corrosion potential, E_{corr} is almost the same for T6-60, T6-120, T6-180, and T6-240. Comparatively,

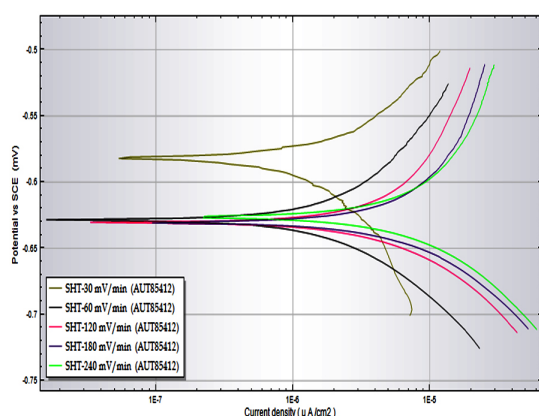


Fig. 4: Potentiodynamic curve of Solution Heat Treatment (SHT) on different potential scan rate in 0.5 M H₂SO₄ at room temperature.

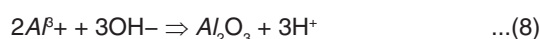
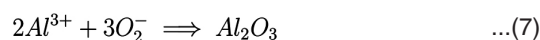
T6-240 is observed to have higher potential value than other temper samples. Thus exhibiting a bit of nobility nature same as T6-30. Corrosion rates for all the samples at different scan rates (SR) follow the order: T6-30 < T6-180 < T6-240 < T6-120 < T6-60. The lowest corrosion rate is observed for AA T6-30 (0.01 mm/year). It means that T6-30 corrodes at slower rate at minimum scan rates and this may be because promotion of oxide film on the surface.

Fig.4 shows the corrosion potential variation with different scan rates for a solution heat treated sample. Corrosion potential of the SHT decreases from -0.58 mV to -0.62V, as scan rate increases. Similarly, the corrosion rate of SHT-60 has lower tendency to corrode at low scan rate. It is indicated from corresponding corrosion data which clearly shows that corrosion current of film formation over the surface has significantly improved by varying scan rate. The observed sequence of corrosion rate (CR) according to the above discussion is as follows: SHT-30 > SHT-180 > SHT-240 > SHT-120 > SHT-60. In this regard, the reaction mechanism would be helpful for the formation of oxide film in presence of the sulphuric acid.

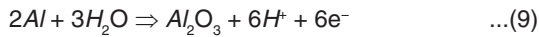
When the potential applied to the electrochemical cell, the following sequence of reaction is supposed to occur on the cathode. Sulphuric acid initiate to decompose and the hydrogen ions move to the cathode, where hydrogen gas is reduced:



Similarly, the negatively charged anions such as hydroxide, sulphate, and some other oxide ions goes to the anode side, which acts as working electrode in the cell. The dissolution of aluminium in form of positively charged aluminium ions (Al³⁺) moves toward the cathode. On the anode surface, they react with the oxide or hydroxide ions to form a high oxide barrier of Al₂O₃. The reaction on anode is mentioned below:



Then, the aluminium hydroxide converts into aluminium oxide:



Apart from the Aluminium oxide, the sulphate ions also play a vital role by forming an oxide coating that contains very few percentages of sulphate ions. It is believed that the sulphate ions assist the movement of hydrogen ions and thus reducing the corrosion potential. Secondly, in the presence of acid solution, a dense even layer of oxide film is formed on the metal surface. It is the passivity that break downs when kept in the aggressive medium⁵⁻¹⁸.

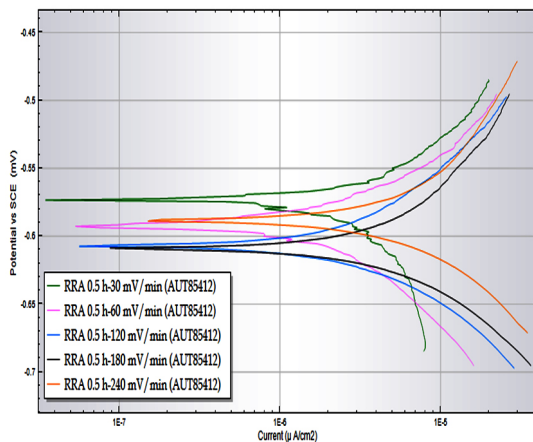


Fig. 5: Potentiodynamic curve of RRA 0.5 h on different potential scan rate in 0.5 M H₂SO₄ at room temperature

It can be observed from Fig.5 and as well as from the corrosion data (Table 2), the corrosion of RRA-0.5h-180 is more than the RRA-0.5h-30 and RRA-0.5h-240 alloys in acidic environment. This can be confirmed by the increase of the corrosion parameters with scan rates. The values of polarization resistance obtained for aged aluminium alloys have shown an increasing trend from RRA 0.5-60 to RRA0.5h- 180 with respect to scan rate. Further, the less negative shift of corrosion potential as can be seen from Fig.5 and also the corrosion rate (CR) increased in the specific scan rate. It is important to mention that the corrosion current increases nonlinearly during scan rate from 60 mV/min. to 240 mV/min. The reason behind increasing current is due to the breakdown of protective oxide film.

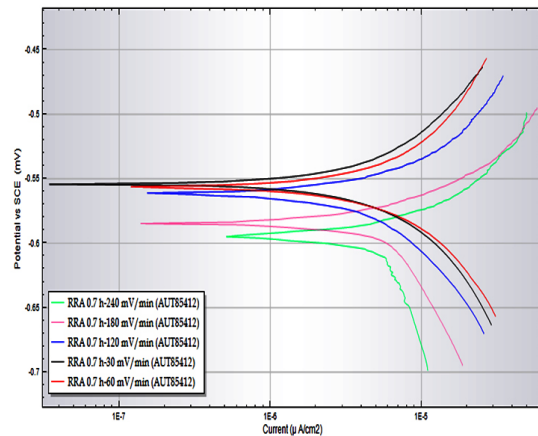


Fig. 6: Potentiodynamic curve of RRA 0.7 h on different potential scan rate in 0.5 M H₂SO₄ at room temperature

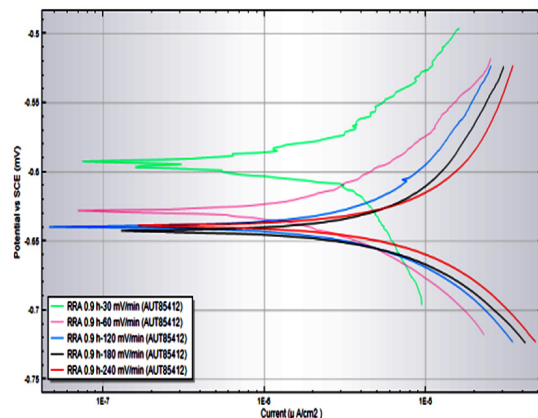


Fig. 7: Potentiodynamic curve of RRA 0.9 h on different potential scan rate in 0.5 M H₂SO₄ at room temperature

Fig.6 depicts that the cathodic and anodic current density show a decreasing trend within finite values of scan rate. This can be attributed to the formation of film on the metal surface and is observed to have noeffect on the higher side of scan rate. The Corrosion potential (E_{corr}) shifts to the less negative direction. The slightly variation in corrosion rate of the aged aluminium samples with increasing the potential scan rate are confirmed by the polarization parameters from Table 2.

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potential scan rate are confirmed by the polarization parameters from Table 2.

The Nyquist plots of aluminium alloy in 0.5 Molarity of H_2SO_4 solution with different ageing treatments are depicted in Fig.8. It is clearly seen in Fig.8 that only one semicircle for aged RRA 0.7h in the sulphuric acid solutions is observed. The variation from ideal semi-circular shape are due to frequency distribution of interfacial impedance which probably came into existence by formation of a porous oxide layer on metal surface. The diameter of the semicircle plotted with their centre under real axis for RRA 0.7h is recorded as the highest value which indicates that the corrosion resistance for aged aluminium at 0.7 h is more than the AA 7075-T6 temper treated sample. This reveals that with increase of aging time on the material, not only the corrosion resistance is increased, but also helps to identify the favourable heat treatment required for enhancement of the corrosion properties under acid environments²¹. It also discourses that there is a possibility of enhancing the corrosion properties due to distribution of precipitates in the matrix.

Fig.8(c) shows the Bode phase angle plots of various Aged 7075 alloy. The equivalent circuit is shown in below Fig. 9. The electrochemical parameters are defined as the R_{ct} charge transfer resistance, R_{sol} represents solution resistance, and $Q_p(CPE_p)$ is the constant phase elements of double oxide layer. The CPE_p is the function of frequency (Hz) and capacitance(C) which defined as the following equation.

$$Z_{CPE} = [C]\omega^{-n}]^{-1} \quad \dots(10)$$

where $j^2 = -1$ and n range 0 -1, when $n = 0$, Q_p will behave as resistor, when $n=1$, as purely capacitance with homogeneous surface²². The fitting values of aged alloys are shown in Table 3.

Table 3: Electrochemical Fitting parameters for aged 7075 aluminium alloy in 0.5 M H_2SO_4

Temper	R_{sol}	R_{ct}	$Q_n(CPE)$	n
RRA 0.5h	8.4915	1417.6	$5.869E^{-06}$	0.87
RRA 0.7h	12.031	1814	$1.369E^{-05}$	0.76
RRA 0.9h	6.433	933.04	$7.656E^{-06}$	0.88

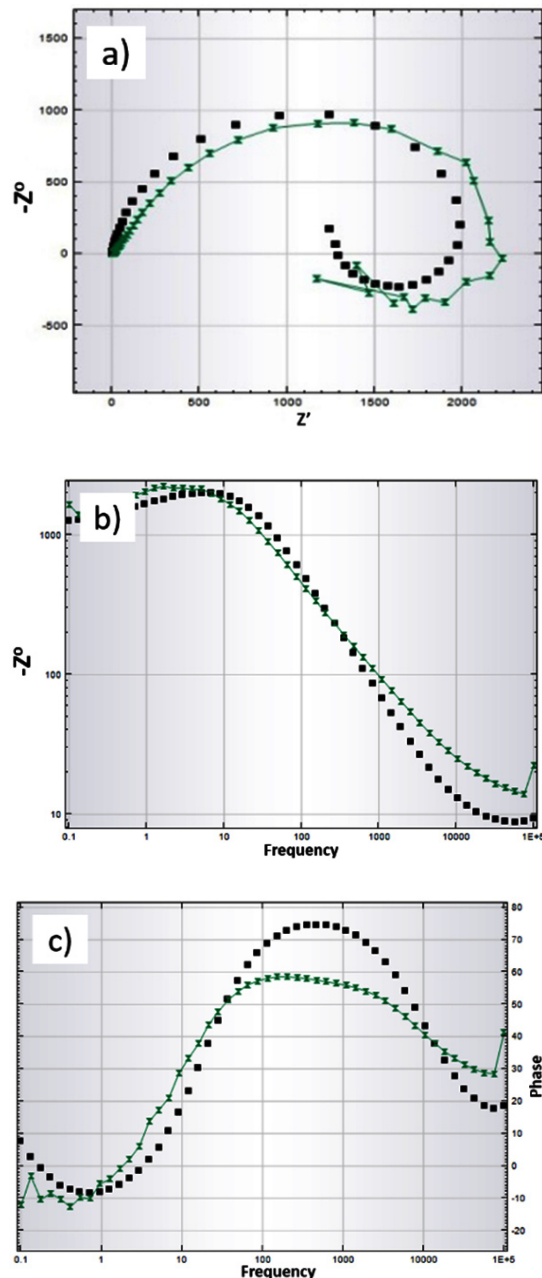


Fig. 8: (a) Experimental Nyquist (b) Bode frequency and (c) Bode phase angle plots for T6 and RRA 0.7Aged alloys in 0.5 Molar H_2SO_4 solutions at room temperature.

The obtained values of n from Eq.10 are observed to be less than 1 that means it is behaving like a resistor. It can also be understood that there can be a possibility of corroding surface and the same has been reflected in the R_{ct} values.

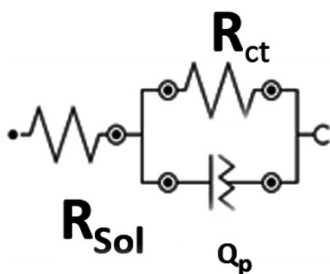


Fig. 9: Equivalent Circuit for Aged aluminium alloys

Conclusions

The effect of potential scan rates on various corrosion parameters of heat-treated AA 7075 alloy are studied in 0.5M H_2SO_4 solution. Samples were subjected to Retrogression and Re-aging (RRA) treatment and Solution heat treatments. Potentiodynamic test plays a vital role in the electrochemical test, the absolute scan rate is an essential parameter to determine the corrosion rate. It affects the difference between corrosion potential and potential at an open circuit. It has been observed from the experiments that the corrosion rate is always higher at high scan rate due to more discharging current on the surface. On the other

hand, if scan rate is slow, the system may deviate from correct value during the measurement and can indicate unsteady state condition. The RRA 0.7 h displays the characteristic age hardening process and attains maximum hardness value compared to the T6 temper sample. EIS measurements show that the corrosion resistance of RRA 0.7 h is more compare to T6 heat treatment process. With increase in aging time, the corrosion resistance is observed to be increased. This study clearly demonstrates that potentiodynamic studies can be used to disclose the electrochemical interaction of an alloy in an acidic environment.

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Conflict of Interest

The author(s) declare(s) that there is no conflict of interests regarding the publication of this article.

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