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Volume 11, Preprint 5 Corrosion Inhibition of as Cast Al-4.5% Cu/15ZrSiO₄

Particulate Composite in Hydrochloric Acid Solution by **Lanthanide Chloride**

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Abstract

The corrosion characteristics of Al-4.5Cu/15%ZrSiO₄ particulate composite in 0.5molar hydrochloric acid without and with lanthanide chloride salt have been investigated. I5% ZrSiO4 was used for the production of the Al-4.5Cu matrix particulate composite using squeeze cast method. Addition of 15% zircon sand resulted to 5.04% decrease in density and 82.61% increase in hardness with respect to the unreinforced Al-4.5Cu matrix alloy. The time, temperature and concentration of inhibitor were varied in the range of 2-10hr at 2hr interval, 30°C-75°C at 15°C interval and 250-1000ppm at 250ppm interval respectively. Arrhenius kinetic model was used for the corrosion analysis. Results show that maximum inhibition was observed at 250ppm. XRF was used to determine the chemical analysis of the zircon sand. An inhibition efficiency of 73.58% was observed after 10 hours of exposure in the hydrochloric acid stimulating industrial atmosphere at 250ppm and 30°C.

Keywords: ZrSiO₄; Al-4.5Cu; reinforcement; hardness; density; XRF analysis; Corrosion; Inhibition efficiency; HCl, LaCl₃ Time; temperature and inhibitor concentration.

Introduction

Aluminium matrix composite (AMCs) have shown high mechanical properties such as high creep resistance, strength, stiffness and wear resistance and when compared to the unreinforced matrix alloy, which has lead to the use of aluminium matrix composite in the following; automobile industries (brake drum, crankshafts, values and suspension

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arms), recreational products (golf club shaft and head, skating shoe, bicycle frames and base ball shaft) and in construction company (truss structure) [1].

Aluminium matrix composite are generally reinforced either with short fibres or continuous fibres or ceramic particles or monofilament. Early research on metal matrix composite concentrated on continuous fibres but fabrication techniques have reduced their used in areas where the end justified the means. This had foster growth in the development of a low cost particle reinforced composite [2-4] The reinforcement of aluminium alloys with SiC, Al₂O₃, SiO₂, C, ZrSiO₄ particulates has led to a new generation of engineering materials, which offer some advantages over conventional materials such as Al-based alloys. [5-6].

Zircon sand particle have been used as reinforcement in Aluminium alloy [18]. Zircon sand possesses good hardness, brittleness, thermal and electrical resistance, strength and good obtrusion resistance. The particle size of the reinforcement depends on the applications and alloy types. However, the mean particles dimension normally lays in the range $2-20~\mu m$ [1, 7].

One of the main obstacles in the use of metal matrix composite (MMC) is the influence of reinforcement on corrosion resistance [8-19]. This is particularly important in aluminium alloy-based composites, where a protective oxide film imparts corrosion resistance. The addition of a reinforcing phase can increase the frequency of discontinuities in the film, thereby increasing the number of sites where corrosion can be initiated and rendering the composite liable to severe attack [8, 9]

Pitting attack has been reported to be the major form of corrosion in aluminium based MMCs [10-12]. Hence it affects the appearance, maintenance cost, value, safety and reliability of material. The use of inhibitors is one of the most practical methods for protection against corrosion. Studies have been carried out on the effect of inorganic inhibitors on the corrosion rate of composites [13-16]

Notice the processing plants using chromate will therefore have to search for new alternatives over the next few years.

Pardo et al [15] revealed that alternative corrosion inhibitors have been investigated in recent years to replace chromates in their different fields of application. For example efforts have focused on the search for new corrosion inhibitors and new formulation for both anodizing bath and conversion coating. According to Cohen in Pardo et al [15], the development of viable alternatives to chromate is still in its infancy or experimental stage.

It has been confirmed that lanthanide ions form insoluble hydroxides, therefore can be used as cathodic inhibitors [14-16] Lanthanides have low toxicity; their inhalation is not considered harmful to health, while the toxic effects of their oxides are similar to those produced by sodium chloride [15]. Furthermore, lanthanides could be economically competitive products since they are relatively abundant in nature [15-17].

Corrosion inhibition of 6061-SiC by rare earth chlorides was studied in 3.5% NaCl solution by Mishra et al, [14] It was observed that the polarization resistance increased after addition of Lanthanide chlorine and cerium chloride, with maximum increase noticed for 250ppm Lanthanide chlorine and 1,000ppm cerium chloride.

Microstructure and aging behaviour of Al₂O₃/ZrSiO₄ particulate reinforced Al-4.5wt%Cu matrix composite, produced by stir casting route, was investigated by Das et al, [18]. The authors observed that both composites showed acceleration in aging compared to the monolithic alloy. However, the Al₂O₃ reinforced composite showed more acceleration compared to the ZrSiO₄ reinforced composite. Many studies have proposed an optimum percentage of reinforcement to 15 in other for the developed composite to possess a

balance of mechanical properties [1, 18, 20]. Up till now, not much has been known of corrosion characteristics of Al-Cu/15ZrSiO₄ composite. Hence the aims of this research are:

- 1. To investigate the corrosion rate of as cast Al-4.5Cu/15ZrSiO₄ in hydrochloric acid stimulating industrial atmosphere.
- 2. To investigate the inhibition efficiency of LaCl₃ in 0.5M hydrochloric acid solution.
- 3. To further justify the use for metal matrix composite in wear resistance applications, the microstructure, density and hardness of the as cast Al-4.5Cu/15ZrSiO₄ particulate composite were examined.

Experimental methods

Materials and Methods

The experimental steps to be followed in this research work are outlined as follows:

Materials

The materials that would be used in this study are:

Zircon sand with mean particle size of $8\mu m$, high purity copper, high purity aluminium electrical wire which will be obtained from Northern cable company NOCACO Kaduna, squeezed casting machine, hydrochloric acid, tripod stand, burette, lanthanide chloride and beakers.

Table 1: XRF analysis of zircon sand particles (wt %)

ZrO ₂	SiO ₂	CaO	TiO ₂	Cr ₂ O ₃	MnO	Fe ₂ O ₃	PbO	Rb ₂ O	Y_2O_3	Ag ₂ O
65.6	21.3	.27	.076	.075	.045	1.97	.167	.059	.73	4.73

CeO ₂	Yb ₂ O ₃	HfO ₂	Bi ₂ O ₃	U_3O_8
.098	.41	2.53	.37	. 57

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The equipments that were used in this study are as follows: metallurgical microscope with built in camera, analytical weighing balance, electrical resistance furnace, mechanical stirrer and crucible.

Methods

The samples were produced by keeping the percentage of copper constant while varying the percentage of aluminium and zircon sand. The 15% zircon sand was used as reinforcement. The high purity electrical wires free from contamination were charged in a graphite crucible kept in an electrical resistance furnace and 0.01%NaNO₃ powder was used as a cover for melting the alloy. The NaNO₃ addition was aim at reducing oxidation of aluminium and creating a protective atmosphere inside the furnace. At the beginning of melting of pure aluminium, the furnace temperature was raised to 750°C. 4.5 percent copper was introduced into the molten pure aluminium using hardener (50%Al50%Cu). The reason was to facilitate easy melting of the charged copper. With progressive melting the furnace temperature was raised to 800°C and the melt was held at this temperature for 12minutes. Then, skimming was done to remove oxides and impurities.

For each melting 250 grams of charge material was used to produce the alloy. ZrSiO₄ particulate of particle size in the range of 2-15µm was preheated to a temperature of 1200°C so as to ensure good interfacial bonding between the alloy matrix and the reinforcement. Then, the molten aluminium alloy is mixed with the preheated zircon sand and the mixture was stirred thoroughly with a mechanical stirrer for 15minutes at an average stirring rate of 180rpm. In the final stirring stage the furnace temperature was controlled between 760°C and 780°C and pouring was controlled to a temperature of about 750°C. A squeeze casting machine with a press capacity 10MPa, approach speed of 50m/s, return force of 5MPa, pressing speed of between 1-10mm/s, die length of 18cm and die diameter of 2cm was used to prepare the test samples.

Examination of Microstructure

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The metallographic specimens of the as cast samples was obtained by cutting with the aid of a hacksaw and distilled water as coolant. The cut samples was mounted on bakelite and mechanically ground progressively on grades of SiC impregnated emery paper (60 – 600) grits) sizes using water as the coolant. During grounding the specimen was turned through 90° between papers. Polishing was done in two steps. First, the rough polishing was done using a 1.0- micron size alumina polishing powder suspended in distilled water, while the second fine polishing was done using 0.5 – micron alumina powder on a rotating disc of proprietary nap cloths. Following the polishing operation was etching of the polished sample using Keller reagent and finally the microstructure is recorded using an optical microscope with an inbuilt camera.

Determination of Hardness

Hardness values of the samples were determined using the B scale of Rockwell hardness testing machine. The indenter used was a 1.56mm steel ball. Minor load of 10kg and major load of 100kg was applied. Hardness of 101.2HRB standard block was used.

Determination of Density

The masses of the used samples were obtained by weighing the cut samples using a digital weighing balance and obtaining their volumes from the volume of water displaced when each samples was totally immersed. Hence the densities of the unreinforce and reinforce cast composition were obtained from the formula given above.

Corrosion Analysis

The as cast specimens was sectioned into sizes of $10 \text{mm} \times 10 \text{mm}$ (average geometrical area of $10.0 \times 10^{-3} \text{m}^2$) and was abraded with a series of emery papers (30-600 grades). They were then washed with distilled water, followed by acetone and dried using a steam of air. Weighing was done using an analytical balance. They were stored in a desiccator. Corrosion tests were carried out in freely aerated .5molar hydrochloric solution. Lanthanum chlorides were added at concentrations of 250, 500, 750 and 1,000ppm. Freshly prepared hydrochloric acid solution was used in all the experiments. All the

experiments were conducted at 30°C, 45°C, 60°C and 75°C. The total immersion time was 10 hours while the corrosion rate was examined every 2hours, to ascertain the effect of time on the corrosion rate. The weight loss method was adopted to determine the corrosion rate. Then the rate of corrosion, inhibition efficiency and activation energy were determined.

Corrosion Rate

The standard expression for measurement of Corrosion rate in mils per year (mpy) was used, which is expressed in the equation below.

$$MPY = \frac{534W}{\rho AT}$$

where W is the weight loss, ρ is the density of the material (g/cm³), T is the time of exposure (hrs) and A is the total surface area (in²).

Inhibitor Efficiency

The

inhibitor efficiency was computed using the relationship in equation below.

$$Inhibitor Efficiency = \frac{Ro - R}{Ro} * 100\%$$

where R and Ro are the corrosion rates with and without the inhibitor respectively.

Film Attractive Power

The degree of surface coverage was calculated from the equation below.

$$\theta = \frac{Ro - R}{Ro}$$

From the result of the degree of surface coverage at a given inhibitor concentration and temperature, the film attractive power was computed using the expression below [21].

$$c/\theta = 1/k + c$$

Where c is the inhibitor concentration (mol/dm²), k is the film attractive power and θ is the surface coverage

Thermodynamic Analysis

The corrosion inhibition of lanthanide chloride for Al-Cu/ZrSiO4 composite was examined using a thermodynamic model. Hence the film attractive free energy (ΔG^o) was calculated to elucidate the phenomenon for the inhibition action of lanthanide chloride. These were achieved using the following equation.

$$k = (1/55.5) \exp(-\Delta G^{\circ}/RT)$$

where T is the absolute temperature and R is the molar gas constant.

Kinetic Analysis

Kinetic models are useful tool to explain the mechanism of corrosion inhibition of lanthanide chloride. Arrhenius equation was used to determine the corrosion kinetic using the expression below

$$Ro = B_o e^{-\text{Ea/RT}}$$

The logarithm of Ro could be represented as a linear equation given below

$$LnRo = -Ea/RT + \ln Bo$$

RESULTS

Microstructure

The microstructure of the as cast Al-4.5%Cu alloy and Al-4.5%Cu/15ZrSiO₄, particulate composite are shown micrograph 1 and 2.



Micrograph 1. Microstructure of the unreinforced Al-4.5Cu Alloy(grey) magnification x 125



Micrograph.2. Microstructure of the reinforced Al-4.5Cu alloy $(15\%ZrSiO_4)$. Uniform distribution of the $ZrSiO_4$ (black) magnification X 125.

Hardness and Density

The chart 1 and 2 also shows the hardness and density result of the as cast test samples in the above test condition respectively.

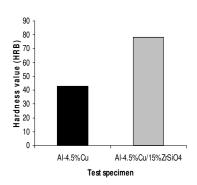


Chart .1. Showing the hardness values of the unreinforced and reinforced as cast materials

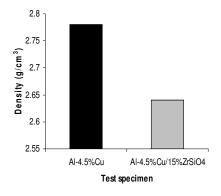


Chart .2. Showing the densities of the unreinforced and reinforced as cast materials

Corrosion

The variations of corrosion rate with inhibition concentration at all experimental test time are shown in figure 1-5.

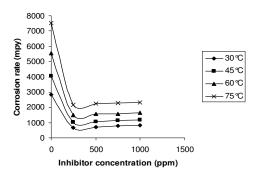


Fig.1. Variation of corrosion rate with inhibitor concentration after 2hrs exposure time.

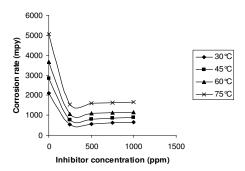


Fig 3. Variation of corrosion rate with inhibitor concentration after 6hrs exposure time.

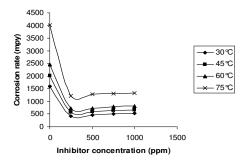


Fig.5. Variation of corrosion rate with inhibitor concentration after 10hrs exposure time.

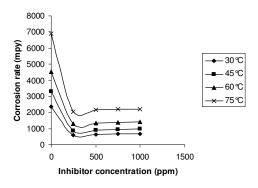


Fig. 2. Variation of corrosion rate with inhibitor concentration after 4hrs exposure time.

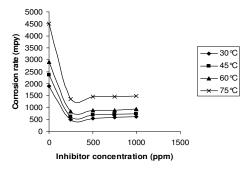


Fig 4. Variation of corrosion rate with inhibitor concentration after 8hrs exposure time.

The variation of inhibition efficiency in with inhibition concentration is shown at various test time in shown in fig. 6 - 10.

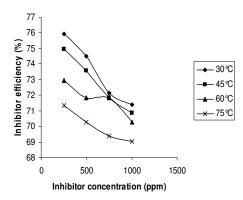


Fig..6. Showing the variation inhibition efficiencies with inhibitor concentration after 2hrs exposure time

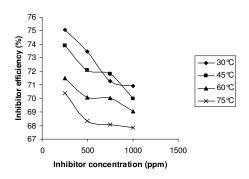


Fig.7. Showing the variation inhibition efficiencies with inhibitor concentration after 4hrs exposure time

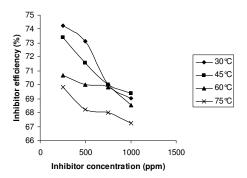


Fig. 8 Showing the variation inhibition efficiencies with inhibitor concentration after 6hrs exposure time.

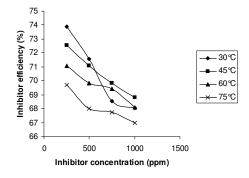


Fig. 9. Showing the variation inhibition efficiencies with inhibitor concentration after 8hrs exposure time.

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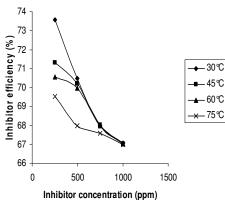


Fig. 10. Showing the variation inhibition efficiencies with inhibitor concentration after 10hrs exposure time.

Table 1 (**Appendix I**) is the film attractive power of the as Al-4.5%Cu/15ZrSiO4 composite. The change in free energy associated with film attraction is shown in table 2 (**Appendix I**). A plot of the logarithm of corrosion rate against the reciprocal of the temperature gave a linear graph with slope –Ea/R. Figure 11 -15 shows the linear regression of lnR and 1/T from without inhibitor to with inhibitor up to 1000ppm concentration of the inhibitor. Figure 16 show the plot of activation energy as a function of concentration.

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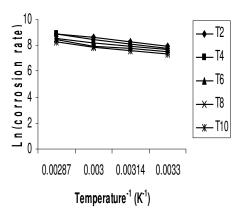
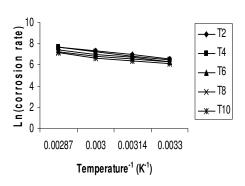


Fig. 11 Variation of Ln(corrosion rate) with reciprocal of absolute temperature in HCl without inhibitor.

Fig. 12 Variation of Ln(corrosion rate) with reciprocal of absolute temperature in HCl with 250ppm inhibitor.



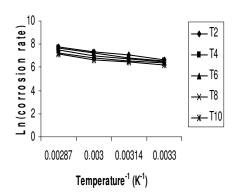


Fig. 13 Variation of Ln(corrosion rate) with reciprocal of absolute temperature in HCl with 500ppm inhibitor.

Fig. 14 Variation of Ln(corrosion rate) with reciprocal of absolute temperature in HCl with 750ppm inhibitor.

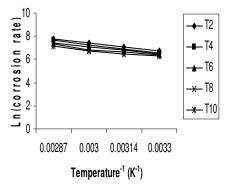


Fig. 15 Variation of Ln(corrosion rate) with reciprocal of absolute temperature in HCl with 1000ppm inhibitor.

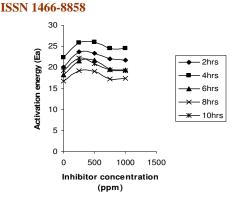


Fig. 16 Variation of activation energy (Ea) with inhibitor concentration (ppm)

Discussion

Microstructure

The microstructure of the unreinforced matrix alloy is shown in micrograph 1. The structure reveals uniform grain size separated by thin 70% black grain boundaries with even faint grayish precipitate of CuAl₂. Micrograph 2 shows the microstructure of the alloy reinforced with 15%ZrSiO₄ particles. The structure reveals the ceramics phase as dark phase, while the metal matrix alloy phase is grayish in colour. Micrograph 2 also shows a uniform distribution of zircon sand particles in the Al-4.5Cu matrix alloy. The high uniformity was an indication of good interfacial bonding attributed to the production method.

Hardness and Density

The hardness value increases with the addition of 15% ZrSiO₄ in the alloy as shown in chart1 from 42.85 to 78.25HRB. This was due to high proportion of the hard and brittle phase of the zircon sand in the alloy. The 15 percent zircon sand addition to the matrix alloy results to elastic and plastic incompatibility due to differences in the modulus of elasticity of the hard zircon sand reinforcer and soft matrix alloy, which enhance high dislocation density hence resulted to an increase in hardness value in the reinforced material.

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The results of the density measurement of the matrix alloy and reinforced material reveals that the addition of 15 percent ZrSiO₄ particulate resulted to Al-4.5Cu decreased the density from 2.77g/cm³ at 0% ZrSiO₄ addition to 2.64g/cm³ at 15%ZrSiO₄ addition. This little change in density is due to faster cooling rate and good interfacial bonding between the reinforcement and the matrix alloy.

Corrosion

The results reveal that the 15% zircon sand particulate composite gave a higher corrosion rate as compared to the matrix alloy without reinforcement. This was due to higher cathodic site introduced by the reinforcement phase. Figure 1-5 indicates that the corrosion rate increases significantly with increasing temperature and inhibitor concentration above 250ppm. However as the exposure time increases the corrosion rate decreases, due to the formation of corrosion product on the test specimen, thereby, reducing the available site for further corrosion reaction to take place. Hence the temperature and inhibitor concentration are the most significant parameter for corrosion rate control.

Figure 6 –10 indicate that the inhibition efficiency decreased with increasing temperature, which suggest that the inhibition of LaCl₃ on the composite might be caused by electrostatic physical attraction and high temperature might cause the attractive force between the insoluble LaCl₃ to be broken, thereby, reducing the efficiency of the inhibitor. Further more, it is well established that LaCl₃ form insoluble LaOH₃ or LaO, which is used for the inhibition of the test material. The present study revealed a maximum inhibition at 250ppm and subsequent decrease in the inhibition efficiency as the lanthanide chloride is increased.

$$LaCl_3 + HCl + H_2O \Rightarrow La(OH)_3 + 4H^+ + 4Cl^-$$

The equation above shows the possible reaction taking place in the test medium. At 250ppm LaCl₃ concentration, the present the hydrogen positive ions and the chlorine negative ions resulted the inhibition efficiency less than 100. However few Cl⁻ ions at 250ppm led to the observed maximum inhibition. Subsequent increase in the LaCl₃

concentration above 250ppm at constant HCl and H₂O introduces additional mobile chlorine ions which migrate to the composite surface and thus pit the surface. Thereby, increasing the corrosion rate and decreases the inhibition efficiency. Table 1 (**Appendix** I) shows a decrease in the film attractive power as the temperature, time and inhibitor concentration is increased, which indicates a direct relationship with the inhibition efficiency.

Table 2 (**Appendix I**) shows an all negative values of ΔG , which suggest that the attraction of the surface film on the composite was spontaneous. Also table 2 (**Appendix I**) indicate an increase in the ΔG value as the temperature is increase which is an indication of higher tendency of the surface film of the inhibitor to be attracted to the composite. However attracted films are weakly bonded as the temperature were increased, hence constitute reduced protection as temperature is increased.

Fig. 11 –15 represents the linear regression of lnB and 1/T for the specimen immersed in 0.5M HCl with and without inhibitor. Some studies [7,21] have shown that compared with the activation energy (Ea) in the absence of inhibitor, higher values of Ea were found in the presence of inhibitors. Other studies [22,23] have shown that in the presence of inhibitor, the activation energy was lower than that in the absence of inhibitor. In the present study it could be found fig. 16 that the activation energy for the entire specimen exposed to corrosive reagent with inhibitor were higher than that of without inhibitors. This, suggest that the anti-corrosion reagent is an active inhibitor on the Al-4.5Cu/15%ZrSiO₄ particulate composite in a dilute 0.5 molar hydrochloric acid medium.

Conclusions

The experimental result show that when Al-4.5Cu/15%ZrSiO₄ particulate composite produced by squeeze casting was exposed in the as cast condition to 0.5 molar HCl solutions with and without anti-corrosion reagent at different concentration, the following were observed:

1. The squeeze cast production method enhances uniform distribution and good interfacial bond between zircon sand particle and the Al-4.5Cu matrix alloy.

Addition of 15% zircon sand resulted to 5.04% decrease in density and 82.61% increase in hardness, which indicate a possible application of Al-4.5Cu/15%ZrSiO₄ particulate composite in areas of wear resistance such as values and suspension arms, recreational products (golf club shaft and head, skating shoe, bicycle frames and base ball shaft) and in construction company (truss structure)

- 2. Increase in temperature above 30°C and inhibitor concentration above 250ppm led to higher electrochemical reaction and hence higher corrosion rate.
- 3. Maximum LaCl₃ inhibition was observed at 250ppm concentration in 0.5 molar hydrochloric acid solutions.
- 4. Inhibition efficiency of 73.59%, 71. 30%, 70.56% and 69.51% were observed in the medium with 250ppm LaCl₃ at 30°C, 45°C, 60°C and 75°C respectively after ten hours of exposure. This also indicates that the inhibition efficiency is temperature dependent.

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Table 1

Result of the film attractive power on Al-4.5Cu/15%ZrSiO $_4$ immersed in HCl at varied temperature and time.

Inhibitor		Time			
concentration (ppm)	Film a	(hr)			
	30°C	45°C	60°C	75°C	
250	4691.03	4444.46	4006.02	3701.15	
500	2171.21	2066.51	1895.86	1757.31	
750	1282.82	1262.00	1261.38	1121.98	2
1000	925.77	902.63	877.78	827.76	
250	4468.23	4203.72	3724.80	3531.24	
500	2055.92	1915.69	1739.76	1603.32	
750	1228.96	1261.38	1155.95	1054.66	4
1000	905.52	865.71	827.76	783.03	
250	4285.5	4098.92	3582.59	3434.87	
500	2046.41	1868.03	1733.97	1593.73	
750	1155.95	1155.95	1145.55	1052.72	6
1000	828.54	841.07	808.34	762.93	
250	4203.73	3927.98	3652.72	3418.63	
500	1869.87	1824.72	1720.83	1578.40	
750	1078.3	1147.19	1125.69	1040.25	8
1000	791.35	819.31	792.08	754.00	
250	4136.97	3690.31	3560.21	3386.45	
500	1771.97	1750.59	1729.02	1578.4	
750	1052.24	1052.72	1052.24	1031.72	10
1000	756.39	755.02	754.34	754.00	

Table 2 $Result of the change in free energy (\Delta G) \ associated \ with \ LaCl_3 \ inhibitor \ on \ Al-4.5Cu/15\%ZrSiO_4 \\ immersed in \ HCl \ at \ varied \ temperature \ and \ time.$

Inhibitor			Time		
concentration (ppm)		(hr)			
	30°C	45°C	60°C	75°C	
250	-31.41	-32.82	-34.08	-35.39	
500	-29.47	-30.80	-32.01	-33.23	
750	-28.14	-29.49	-30.88	-31.93	2
1000	-27.32	-28.61	-29.88	-31.05	
250	-31.29	-32.67	-33.88	-35.25	
500	-29.33	-30.60	-31.77	-32.97	
750	-28.03	-29.50	-30.64	-31.76	4
1000	-27.26	-28.50	-29.72	-30.89	
250	-31.19	-32.61	-33.77	-35.17	
500	-29.32	-30.53	-31.76	-32.95	
750	-27.88	-29.26	-30.6	-31.75	6
1000	-27.04	-28.42	-29.65	-30.82	
250	-31.14	-32.5	-33.83	-35.16	
500	-29.10	-30.47	-31.75	-32.93	
750	-27.71	-29.24	-30.57	-31.72	8
1000	-26.93	-28.35	-29.6	-30.79	
250	-31.09	-32.33	-33.75	-35.13	
500	-28.96	-30.36	-31.76	-32.92	
750	-27.64	-29.01	-30.38	-31.69	10
1000	-26.81	-28.13	-29.46	-30.78	



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