

EFFECT OF TiB_2 ON THE CORROSION RESISTANCE BEHAVIOR OF IN SITU Al COMPOSITES

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Abstract

This research focuses on a detailed investigation for understanding the corrosion behavior of A356 Al alloy-based metal matrix composites reinforced with in situ TiB_2 particles. The aforesaid newer composite samples were fabricated by adopting in situ technique using halide salts by stir casting route under various pouring temperatures of 750 °C, 780 °C and 810 °C maintained at a constant holding time of 20 min. The so-developed Al MMC samples were then subjected to microstructure and electrochemical studies. Surface morphology of samples showcases for the formation of TiB_2 particles over and above the surface of

fabricated composite specimens. Corrosion behavior of the samples were investigated exhaustively by using Tafel polarization and electrochemical impedance spectroscopy under 3.5 wt% NaCl solution. The attained results depict that whenever there is a gradual increase in pouring temperature, substantial improvement in the corrosion resistance of developed samples is also evidenced.

Keywords: Al, TiB_2 , casting route, Tafel polarization, impedance spectroscopy

Introduction

Automotive industry estimates that about 70% of fuel consumption was majorly related to the vehicle weight besides holding a known list of adverse effects. As a consequence, use of lightweight alloys in cars for end-to-end manufacturing has been drastically incorporated right from the last decade. Among the range of suitable materials availability, A356 aluminum alloy was widely used in the automotive field just because of its good mechanical properties and easy castability. However, its poor corrosion resistance restricts it from being recommended for a variety of applications effectively.¹⁻⁵ Headed for being acceptable without being simply submerged, these essentials are ratified through the adoption of new age technocrats, herein composite materials were considered as an extraordinary

choice of best alternative to assist in material selection. These so-called composite materials basically consist of two physically dissimilar phases, and hence they aptly disperse to deliver properties that are not easily attainable with either of the separate phases; the major highlight is that these dissimilar phases will join together, but it will not at all dissolve within each other.⁶⁻⁸

It is well acknowledged by universal researchers that the addition of reinforcement particles to the matrix materials will definitely help to deliver preferred properties of the developed composite is material concerned. Irrespective of the combination (other than the tailor-made developments), the newer composites exhibit better strength compared to the selected base matrix material. Any such metal matrix composite (MMC) will be manufactured by means of

orderly procedures rather than conservative metal alloying techniques, and these MMCs were fabricated by merging one or two constituents. The reinforcement particles added in any desired form were entrenched into monolithic material owing to the known fact of sollicitation of structures. Usually, the matrix material might be a lightweight metal such as magnesium or aluminum.⁹⁻¹⁵ In the recent past, MMC developments are mainly concentrated on fiber base reinforcement particles but, however, owing to high cost of fiber-based materials, certain easily adoptive manufacturing methods constrained their use to meet the preferred result. This concretes the tactic for novel investigations and adjudged fabrications thereof just by introducing low-cost reinforcement MMCs like that of particle-reinforced MMC in which hard ceramic particles like SiC, B₄C, TiC, ZrC, etc., and solid lubricant material, viz. graphite, MoS₂, Bn, etc., are used.¹⁶⁻²³ Among the generally used ceramic particles, TiB₂ attains higher attraction by researchers and the industrialist owing to its unique characteristic affirmations that too with better thermal stability, higher hardness and lower density.²⁴⁻²⁶ In the past two decades, researchers also strongly concentrate on fabrication of Al-TiB₂ by adopting in situ fabrication route, because many necessitated property enhancements along with various multi-property benefits can easily be attained when compared to ex situ development technique.

Researchers found that better interfacial bonding has been achieved for Al-TiB₂ composite by adopting these in situ fabrication routes. Even then, poor wettability of hard ceramics particles paves way for irregular dispersion that leads to lower mechanical properties and higher porosity. Among the available in situ fabrication techniques, usage of liquid state metallurgy processing was considered to be one of the predominant techniques for halide-based salts. There are also very few researches available in which TiB₂ particles are used mainly to improve the properties of Al alloys required as per the stated application.

Karbalaee Akbari et al.¹⁸ adopted liquid casting method to develop Al/TiB₂ composite in two types, i.e., by TiB₂ particle with nano and microparticle size; the team investigated both the tensile and fracture behaviors of fabricated MMC. The observed results reveal that significant improvements could be witnessed in tensile strength (43%) and elongation (27%) which were attained in 1.5 vol. % of TiB₂ nanocomposite and further addition of TiB₂ nanoparticle content leads to deflation in the strength values and elongation of nanocomposites; further tensile strength of the MMC increased with increasing reinforcement content. However, MMC made up of micro-size TiB₂ particles showed nearly lower and also constant values of toughness compared to base alloy. Molten salt electrodeposition method was adopted by Huang et al.²⁷ to develop Al/TiB₂ composite and further used anodization technique to enhance their corrosion resistance. Polarization method was utilized by them to analyze the corrosion behavior of

developed samples. Results pulled out the fact that corrosion resistance has got greatly enhanced in TiB₂/A356 composites with an anodized Al coating than that of the anodized composites and also depict that this new treatment of metal electrodeposition in molten salts and following anodization is an effective method for anticorrosion. Huang et al.²⁸ adopted ionic liquid electrodeposition technique to fabricate Al/TiB₂ composite and investigated the corrosion behavior of developed samples using potentiodynamic polarization method. Also, they observed that the current efficiency decreased with increase in current density; on the other hand while increasing the temperature, the current efficiency increased slightly and also the research group addressed that the effect of deposition time on current efficiency is insignificant for composites. They further observed that corrosion resistance of the fabricated composite was greatly enhanced to an extent because of electrodeposition of Al in ionic liquid.

H.H.Sun et al.²⁹ developed Al MMC with varying wt. % of TiB₂ from 5 to 15 wt% using exothermic reaction process. They studied for the electrochemical corrosion behavior of developed composite using CHI660C electrochemical workstation under 3.5 wt% NaCl solutions. The research team observed that the corrosion resistance of the composites markedly decreases with increase in TiB₂ content. Morphologies of corroded samples showcase that corrosion primarily occurs at the inter-dendritic sites filled up with a large amount of TiB₂ particulates. Then, corrosion progresses and continues into the composite inners (along the regions of an Al dendrite). Poor corrosion resistance properties of the composites are considered primarily due to the galvanic corrosion between reinforcements and more active aluminum matrix, as well as the weak protection of the defective natural film over the composite. A well-defined literature review hauls out that only very limited work has been carried out so far in terms of characterizing corrosion behavior of TiB₂ particle-based composite processed by in situ reaction technique. Formulated to address the leftover consequence in this research, the in situ fabrication method was deeply adopted for MMC fabrications; wt% of TiB₂ was kept constant at 6% for all of the specimens developed for the breakthrough in the field of TiB₂ reinforcements. In order to ensure more vital applications, corrosion behavior of the developed composite was studied in depth by using Tafel polarization and electrochemical impedance spectroscopy under 3.5 wt% of NaCl solution.

Materials and Methods

In this research, A356 Al alloy is used as the base matrix material as because it is also been widely used in many trustful applications inclusive of aerospace. Chemical composition of the as-purchased base material is depicted in Table 1 for further cross-references. Titanium diboride

(TiB₂), which has unique properties like high melting point, thermal stability and chemical stability, is used as the main reinforcement for the sake of improving the basic and functional properties of the base matrix. From the underwent specific literature survey, usage of TiB₂ in the range of 4–8 vol% was said to exhibit better strength rather than any other percentage additions, and hence in this research, vol% of TiB₂ is kept constant as 6%.

As described, for the in situ method adopted to develop a TiB₂-reinforced Al MMC, the requirements for halide salts, viz. K₂TiF₆ and KBF₄, were found based on the stoichiometric calculations, and so as per the common procedural follow-ups, the salts were preheated at 250 °C. A pre-weight mixture of K₂TiF₆ and KBF₄ was added to the molten metal in order to produce TiB₂ reinforcement effectively, and then the melt was stirred continuously for at least 25-min duration by using a graphite rod (the slag was removed well before just to make the process easy). Ingots were cast for manipulated combinations of three different pouring temperatures, viz. 750 °C, 780 °C and 810 °C, for a constant holding time of 20 min. The developed samples were made into strips of 2 mm thickness uniformly by adhering state-of-the-art wire-cut EDM technology and further named as T1, T2 and T3, respectively, for easy reference in all future calculations.

Material Characterization

Surface morphology of the developed samples was analyzed in detail by using JOEL SEM (scanning electron microscopy). Necessarily, the research-oriented hardness values were measured using a Brinell hardness tester for an applied load of 100 kgf maintained statically for 20 s. CHI604C, USA-made electrochemical workstation, was used for studying the corrosion behavior of developed Al-TiB₂ MMC. It constitutes a three-electrode setup as specified below: saturated calomel electrode (AgCl) as the reference electrode, platinum wire as the counter electrode and MMC strip as the working electrode. The Tafel polarization was carried out for a range between – 1.5 and – 0.5 V at a scan rate of 0.01 V/s mandatorily. For the purpose of fulfillment, all of the experiments were repeated for at least three times in order to further confirm the exactness of the results attained. Electrochemical impedance spectroscopy (EIS) was carried out under the frequency range of 0.01–100000 Hz with amplitude of

0.005 V; corrosion behavior of the developed samples was studied in a detailed manner under 3.5 wt% NaCl electrolyte.

Results and Discussion

Microstructure Characterization

The dendritic growth of the as-received A356 Al alloy is shown in Figure 1a. Formation of reinforcement particles is clearly visualized in Figure 1b, d, and it could highly be witnessed that the grain size of particles increases normally with respect to the pouring temperature. Occurrence of small micro voids (size 0.5–1 μm) and finer and equally distributed TiB₂-reinforced particles (size 0.2–1 μm) could be visualized at a pouring temperature of 750 °C; the reason for any such happenings are supposed to be faster cooling and the slower grain solidification which in turn minimizes certain other attainments such as the grain coarsening. As since it is a well-known artifacts that Al alloy contains silicon particles as one of the major alloying elements, it might habitually possess the tendency to get segregated to form the grain boundaries. Likewise, formation of dendrite has not been notified in case of the developed MMC and this might be due to the reinforcement particles, viz. TiB₂, which constrain the dendrites growth by means of nucleating the grains as α-Al grains. While increasing the pouring temperature to 780 °C, the grain refinement will get reduced and so the size of TiB₂ particles (size 1–1.5 μm) also gets increased apart from the notified happenings; decrease in the cooling rate against increasing solidification rate minimizes the presence of voids and porosity, thereby increasing grain coarsening.²²

As per the specified hypothesis of this research, while further increasing the pouring temperature to 810 °C, it could be distinguished that size of the reinforcement particles is much higher (size 1.5–3 μm) being mainly accounted due to slower cooling rate of the melt. Because the cooling rate is minimum and solidification rate is high, the grain refinement was observed to be minimum which in turn leads to the growth of equally and evenly distributed coarser grains majorly in and around the molten zone; uniform dispersion of particles could also be identified from surface morphology. Herein, the general phenomenon of dispersion of reinforcements besides the known limitation of dendrites formation is supposed to be the main

Table 1. Chemical Composition of A356 Al Alloy

Elements	Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
Wt%	6.5–7.5	0.2	0.2	0.1	0.25–0.45	0.1	0.1	Balance

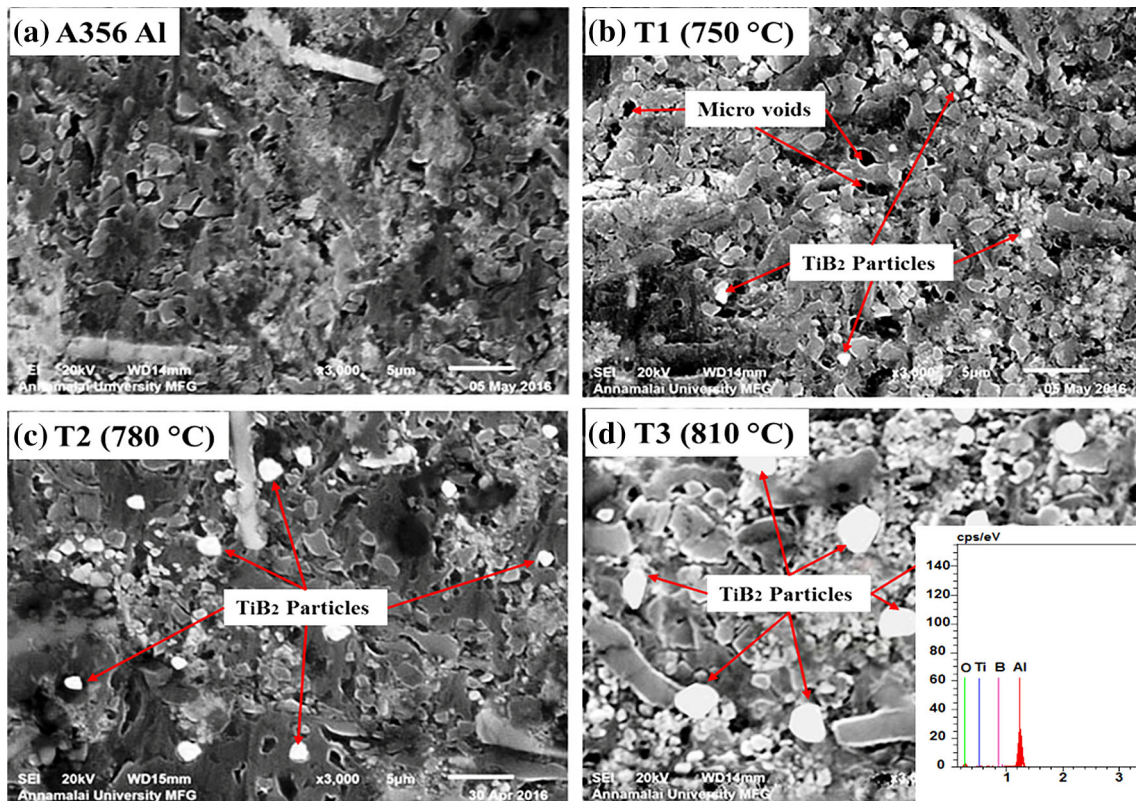


Figure 1. SEM of (a) as-received A356 Al, (b) Al-TiB₂ MMC at 750 °C, (c) Al-TiB₂ MMC at 780 °C, (d) Al-TiB₂ MMC at 810 °C(EDS).

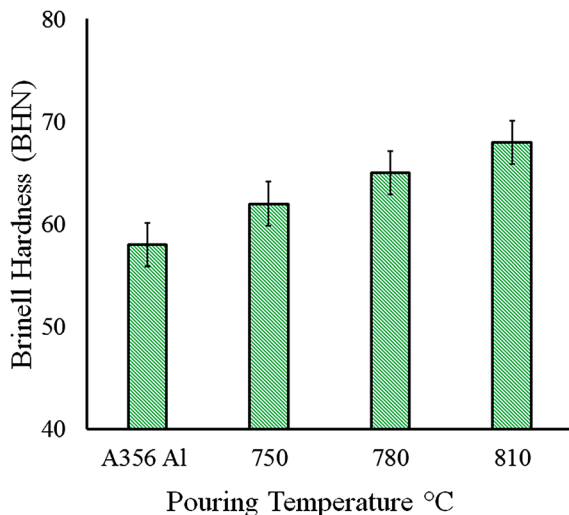


Figure 2. Variation of hardness with respect to pouring temperatures.

strengthening mechanisms that happen in case of the newly fabricated Al-based MMC. The presence of elements, viz. Ti, Al, B, O, notified from Figure 1d by the way of EDS hails in to deepen the understanding of importance and aftereffects of the individual elements towards the major research finding.

Hardness Analysis

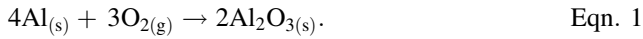
Figure 2 clearly illustrates the variation of hardness with respect to the adhered pouring temperatures. The figure also notifies that hardness of developed MMC increases with increment in pouring temperature. This behavior is well explained by the way of progressive increase in grain size of the fabricated composite with eventual increase in temperature, which deliberately produces greater homogeneity and compactness of the grains profounding for mutual increase in hardness. Further, these cast products are brought concerning to the faster cooling rates, thus giving rise to grain refinement; these usually trends in for elimination of porosity to a greater extent.

Corrosion Behavior

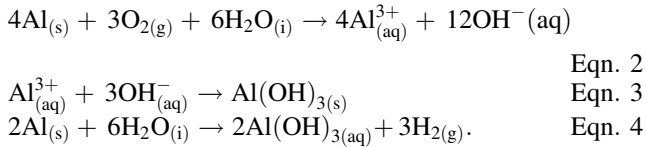
Tafel Polarization

Aluminum and such other low-density metals have always showcased themselves only for nominal corrosion resistance that too because thermodynamically positioning a lower place in galvanic series, which is the sequence representing standards in reduction potentials of several metals and non-metal ions that are in relation to the hydrogen ion. Remarkably, a lower place in this series was made known to defiantly make a metal to react in redox reactions and that of the anode to be called as loosing mass or ion

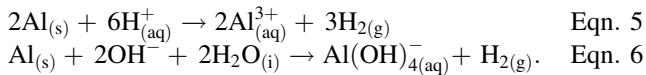
formation. Conversely, the reaction of aluminum with oxygen in the presence of air formulates a very thin film of aluminum oxide or alumina, which in turn tends to protect aluminum and as such make it to become more resistant against the corrosion environment as depicted in Eqn. 1.



The formation of oxide layer during corrosion reaction not only refuges aluminum from auxiliary oxidation, but still helps in protecting the aluminum from more damages that might occur. The reactions of aluminum under a defined environment (water) are exemplified here as Eqns. 2-4.³⁰



Aluminum oxide layer is very thin, and hence it would easily be degraded by the use of any sorted out chemical or physical approaches, whereas if physical approaches are of major concerns it could be shortened by obscure cleaning. For aircraft and/or allied applications, chemical degradation by conservational effects was always challenging to mitigate. Further, pH level was also an important factor, since oxide layers are steady only between the pH ranges from 4.0 to 9.0 (just before it becomes soluble slightly than solid). There are also various other well-known chemical reactions of aluminum that are highly reliable with acids and bases medium, eventually making these conditions even more detrimental.³¹



During corrosion reaction, formation of Al_2O_3 or $\text{Al}(\text{OH})_4$ occurs under cathodic reaction, which in turn controllably acts as an inhibiting barrier against the corrosive electrolyte. Likewise, formation of aluminum-based salts, viz. aluminum chloride, takes place during anodic reaction which might increase the pH values of the electrolyte and further be liable to increase the corrosion rate. During anodic reaction, metal dissolution or ion formation takes

place and as denotified end effect accelerates free Al ions into the aqueous electrolyte. These free Al ions react with chlorine ions present in the electrolytes and out of the same form Al-based salts.

In this research, corrosion behavior of all the developed samples were investigated under 3.5 wt% NaCl solution, and the obtained results are tabulated in Table 2. From Table 2, it could be taken for consideration that, when T1 sample is being dipped in electrolyte, its corrosion potential moves toward the anodic region as noticed through Figure 3. Again, from Table 2 it could easily be illustrated that β_a values are higher than β_c values; this implies that there is a chance for occurrence of anodic reaction or metal dissolution. During anodic reaction, conversion of metal to metal ions takes place; the same initiates for the flow of ions into the corrosive solution which in turn results in increased I_{corr} value. In general, it has always been well said that I_{corr} is proportional to CR of the material. In this research, T1 samples have showcased a CR value of 5.03×10^{-5} (mpy). The polarization resistance of the material is defined as the resistance offered by a material against dissolution; and as so, in par with the definition, T1

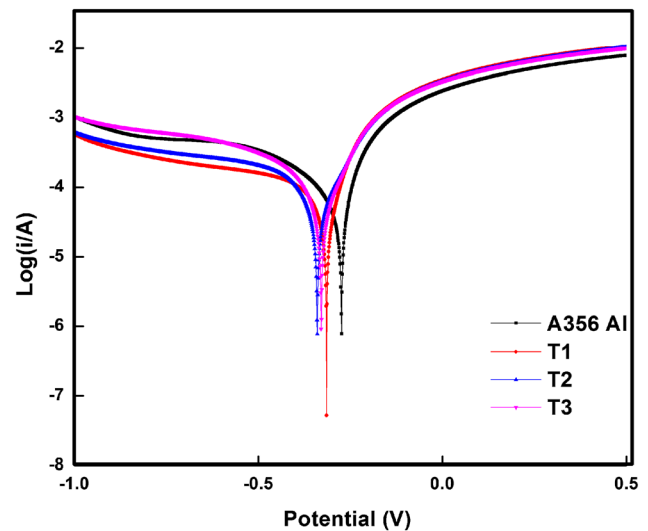


Figure 3. Tafel polarization curve for developed MMC.

Table 2. Corrosion Parameter Derived from Tafel Plot for 3.5 wt% NaCl Solution

Corrosion parameters	Cathode Tafel constant β_c , (1/V)	Anode Tafel constant, β_a , (1/V)	Corrosion current density $I_{\text{corr}} \times 10^{-5} \mu\text{Acm}^{-2}$	Polarization resistance R_p (Ωcm^2)	Corrosion rate, CR $\times 10^{-5}$ (mpy)
A356 Al	4.724	7.200	5.54	656	5.03
T1	2.974	9.220	4.51	789	4.09
T2	3.516	10.054	4.30	743	3.90
T3	4.510	10.410	4.10	708	3.72

samples have attained a lower R_p value of $656 \Omega \text{ cm}^2$. The reason for such outputs may be due to microporosity and certain other defects that occur over the surface of T1 samples as observed in casting process. These porosities initiate the galvanic coupling effect and further accomplish for any increase in the CR values.

When T2 samples were dipped in an electrolyte, its corrosion potentials get shifted toward anodic region as noticed from Table 2. β_a values, being higher, depict the occurrence of anodic reactions, but, however, it has a CR value of 4.09×10^{-5} (mpy) only. This might be attributed to the fact that, during thermochemical processing, TiB_2 particles are being distributed more homogeneously and thus pull out for a decrease in the corrosion rate. The oxidation reaction of Al MMC leads to the formation of a protective oxide layer over and above the surface of developed samples. These oxide layers protect the essential surface from further corrosion that generally takes place due to the involvement of an electrolyte. Conversely, the thickness and steadiness of shelter layer largely depend on the dispersion and percentage of TiB_2 content. T2 samples that showcase for higher R_p value of $789 \Omega \text{ cm}^2$ demonstrates that the developed MMC offers better resistance against metal dissolution too.

When T3 samples were dipped in corrosive solution, its potentials shifted toward anodic region, thus evidencing for the occurrence of anodic reaction. However, developed samples indicate a lower CR value of 3.72×10^{-5} (mpy). Thus, as shown, it could well be evolved that the variation in temperature also acts as an influential factor for defined alteration in corrosion rate. Further addition of TiB_2 has protected the MMC to a larger extent, owing to the fact of quality of the passive oxide layer. The higher corrosion resistance of TiB_2 could well be attributed to two major factors. First, the isolation of grain boundaries in TiB_2

(causes intergranular corrosion, viz. corrosion at grain boundaries) could be severe enough to drop grains over the MMC surface, which significantly accelerates the corrosion resistance.²⁴ Second, the TiB_2 , which gets deformed severely, could form passive surface layers more readily and further it is to be acknowledged that TiB_2 has high density of grain boundaries and dislocations as well inside the grains. If the passive films over the developed surfaces were nucleated over the surface of crystalline defects, TiB_2 would have a high density of nucleation sites for passive films, which in turn dominantly leads to high fraction of passive layers and lower range of CR value.

Electrochemical Impedance Spectroscopy

Electrochemical impedance spectroscopy (EIS) was investigated, in order to confirm the formation of passive inhibiting layer over the surface of the fabricated Al MMC samples. The Nyquist plot (Figure 4) comprises capacitive resistance loop at high-to-medium frequency and an inductance loop at low-frequency range. These interactions are known to create metastable Al ions, which are further represented by the way of inductive loop in impedance curve; hence, it is believed that the inductive loop of Al alloys in low-frequency range reveals dissolution of protective alumina layer over-fabricated MMC. Therefore, the absence of an inductive loop indicates that a more perfect protective film was formed on the developed MMC surface while immersed in corrosive solution (omitting titanium and boron composite networks, which always have better corrosion resistance than the considered base Al alloy).

In this fact, higher value of charge transfer resistance (R_{ct}) denotes the formation of oxide layer. As-received A356 Al alloy samples have lower R_{ct} of $\sim 69 \Omega \text{ cm}^2$, and this might be due to the lower R_p values of developed samples which are supposed to have only a lower resistance against metal dissolution. Due to such identifications, peeling of oxide layer obviously occurs and this further results in lower R_{ct} value of developed Al MMC. T2 samples have higher R_{ct} value of $\sim 88 \Omega \text{ cm}^2$. This result turns in as a proof for the formation of oxide layer over the developed samples. Yet again, when the obtained results were verified based on the R_p values, among the developed samples, T2 samples have higher resistance against the metal dissolution or ion formation. T3 samples indicating R_{ct} of $\sim 92 \Omega \text{ cm}^2$ signify efficiency of formed oxide layers; however, T3 samples have better corrosion resistance for the reason being selection of imperative process parameters.

Conclusion

$\text{TiB}_2/\text{A356}$ in situ composites were successfully synthesized by liquid metallurgy route, and corrosion behavior of developed samples were investigated in detail. The end

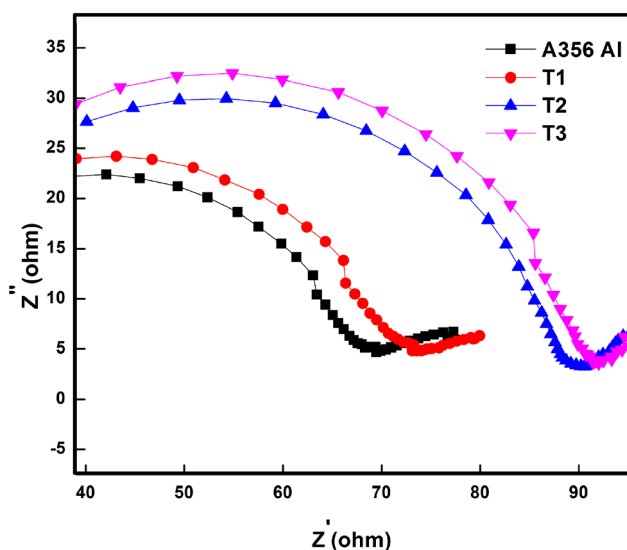


Figure 4. EIS analysis of developed MMC.

conclusions of the carried over novel research have been made as follows:

- (1) The adopted in situ method eliminates the adverse effects of TiB_2 particles; when there is a significant increase in the pouring temperature, improvement in the grain refinement was noticeable.
- (2) The amount of formation of TiB_2 particles was proportionate to the pouring temperature and holding time that too within the limits of 750 °C to 810 °C and 20 min, respectively.
- (3) The number of TiB_2 particles increases due to the fragmentation, and thus, the hardness gets increased. When pouring temperature increases, TiB_2 formation will also be a bit more, further resulting in higher hardness.
- (4) Hardness of the samples increases up to 69 BHN with respect to pouring temperature modulations.
- (5) Increase in pouring temperature decreases the corrosion rate of the sample up to 3.72×10^{-5} mpy for the developed Al MMC.
- (6) Larger semicircle loop obtained for T3 (810 °C) sample clearly depicts the formation of oxide layer as well better inhibition efficiency in saline environments.

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