



# Article Mitigating Corrosion Effects of Ti-48Al-2Cr-2Nb Alloy Fabricated via Electron Beam Melting (EBM) Technique by Regulating the Immersion Conditions

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**Abstract:** The corrosion behavior of newly fabricated  $\gamma$ -TiAl alloy was studied using electrochemical impedance spectroscopy (EIS) and cyclic potentiodynamic polarization (CPP) techniques. The  $\gamma$ -TiAl alloy was produced from powder with compositions of Ti-48Al-2Cr-2Nb processed using electron beam melting (EBM) technique. The corrosion behavior of the bulk alloy was investigated in 1 M HCl solution for different immersion times and temperatures. The experimental results suggest that the fabricated alloy exhibits good resistance to corrosion in acid solution at room temperature. The results also indicate that with an increase in immersion time and solution temperature, the corrosion potential (Ecorr) shifts to a higher positive value, resulting in an increase in corrosion current (jcorr) and consequently a decrease in the corrosion resistance (Rp) of the alloy.

Keywords: electron beam melting; titanium alloys; corrosion behavior; polarization; EIS

## 1. Introduction

The need for increasing the performance and efficiency of engines has led to a huge demand for advanced materials in the aerospace and automobile industry. Gamma titanium aluminides ( $\gamma$ -TiAl) are intermetallic alloys with high specific strength and excellent high temperature properties as compared to other titanium- and nickel-based alloys [1]. They are being considered as alternatives to Ni-based super alloys for jet engine parts, especially low-pressure turbine blades and high-pressure compressor blades [2]. In addition,  $\gamma$ -TiAl has superior creep, oxidation and burn resistance to many other alloys. The presence of alloying elements such as Al, Cr Nb lead to the formation of protective oxide layers, which enhances the mechanical properties, as well as corrosion resistance, of these alloys [3].

 $\gamma$ -TiAl intermetallic is a difficult-to-cut material because of its low ductility at room temperature and the complexity of its processing [4]. Manufacturing of complex parts from  $\gamma$ -TiAl is a challenging task. Although a lot of progress has been made in extrusion and forging, manufacturing complex parts with these techniques is expensive [5]. Casting, on the other hand, is a cost-effective technique, but it has its own limitations [6].

The additive manufacturing route has great potential for the fabrication of complex parts directly from powder. Electron beam melting (EBM) is a powder bed additive manufacturing process that builds near net shape complex parts in a layer-by-layer manner. EBM build  $\gamma$ -TiAl parts have shown good mechanical properties and the potential to be used in aerospace and automotive applications [7].

Although over the last few years extensive research has being done on EBM of  $\gamma$ -TiAl alloy, there is little research available on its corrosion resistance. Delgado et al. [8] has reported the significant corrosion behavior of  $\gamma$ -TiAl in Ringer's solution, and it was found to have a very good corrosion resistance. In our previous work, we investigated the  $\gamma$ -TiAl in chloride solution and found that the formation of an oxide layer is responsible for the excellent corrosion behavior [9].



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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). The intent of this work is to investigate the corrosion morphology of  $\gamma$ -TiAl alloy fabricated using the EBM technique and then exposed to an acidic (1 M HCl) solution for different exposure times (0, 1, 4 and 24 h) and temperatures (30, 40, 50 and 60 °C). The present study was executed using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques in addition to the use of surface characterizations (SEM and EDX) of the  $\gamma$ -TiAl alloy. The results are reported and discussed.

#### 2. Materials and Methods

### 2.1. Electron Beam Melting of $\gamma$ -TiAl

Titanium-based fine powder was purchased from ARCAM company (Arcam AB, Designvägen, Sweden) with 98.5% purity and nominal composition of Ti-48Al-2Cr-2Nb (atomic percent). The particle size distribution of the purchased powder was measured by laser diffraction technique using MALVERN mastersizer 2000 particle size analyzer device (Bradford, UK) with Kernel firmware software version 4.01. The powder particle sizes were 40–110  $\mu$ m with average size of 65  $\mu$ m approximately, as shown in Figure 1. The morphology of the powder particles was shown in Figure 2. According to the FE-SEM image, it has a regular spherical shape with different homogeneous sizes.



Figure 1. Particle size distribution of the as-received powder by laser diffraction.



Figure 2. FE-SEM image of the the as-received powder particles.

The EBM setup used in this work is Arcam A2 model from Arcam (Mölndal, Sweden).  $\gamma$ -TiAl plates of dimension 25 mm  $\times$  25 mm  $\times$  13 mm were built on 10 mm thick stainless-steel substrate of dimension 100 mm  $\times$  100 mm. The parts that are to be built are first

saved in STL format (STereoLithography) in Magics software (Version 17; Materialise NV, Leuven, Belgium). After setting up the built layout, the STL file is then transferred to Build Assembler software (Version 3; Arcam AB, Mölndal, Sweden), which converts the STL file to machine-specific ABF file after slicing the geometry based upon the set layer thickness. The process parameters used for EBM of  $\gamma$ -TiAl are shown in Table 1.

Table 1. EBM process parameters.

Parameter	Level
Voltage	60 KV
Beam current	25 mA
Beam speed	2400 mm/s
Layer thickness	90 microns
Nominal Build temperature	1100 °C
Controlled vacuum	$\sim 2  imes 10^{-3}  ext{ mBar}$

After setting up the EBM process parameters, parts were then built in a layer-by-layer manner under controlled vacuum. The build envelope in powder-bed-based additive manufacturing systems is usually surrounded by powder, which also gets slightly sintered to the parts. Once the build is complete, the build envelope is taken to the powder recovery system to blast the powder sintered to the parts with the compressed air.

The samples of cubic dimensions 10 mm  $\times$  10 mm  $\times$  10 mm were cut from EBM produced  $\gamma$ -TiAl plates, and corrosion studies were carried out on the same. These samples were first analyzed under SEM. Figure 3 shows microstructure of the EBM-produced  $\gamma$ -TiAl alloy. SEM investigations revealed good consolidation of powder during EBM process.



Figure 3. Microstructure of EBM fabricated  $\gamma$ -TiAl alloy.

#### 2.2. Electrochemical Experiments

The corrosion studies were executed on an Autolab system by Metrohm (PGSTAT20). The spine specimen was used as working electrode (WE) for the electrochemical experiments with Ag/AgCl as a reference electrode (RE) and platinum foil as counter electrode (CE). One of the faces of WE was soldered by a copper wire, cold mounted in resin and left to dry in air for 24 h at room temperature. The other face was grounded with emery paper ranging from 100 to 1000 grit and then cloth-polished with alumina slurries. The polished surface of the electrodes was then washed with distilled water, degreased with acetone and finally dried with dry air. The electrolytic solution was prepared from analytical grade

hydrochloric acid and distilled water. The working electrodes were immersed in 1 M HCl solution for 1 h at open circuit potential ( $E_{OC}$ ) to confirm its stability with time. Electrochemical impedance spectroscopy (EIS) was measured at  $E_{OC}$  with applied 5 mV sinusoidal perturbations in the frequency range of 100 kHz~100 mHz with 10 steps per decade. The cyclic potentiodynamic polarization (CPP) was then conducted by stepping the potential at a scan rate of 1.67 mV/s in the range of -1.2 to 0.0 V against SCE at OCP followed by scanned in the reverse direction to complete the cycle test. For each experiment, three measurements were performed using freshly polished electrodes to ensure the reliability and reproducibility of the data.

## 2.3. Surface Characterization

In order to characterize the surface morphology of the fabricated samples, field emission scanning electron microscopy (FE-SEM) Model: JEOL JSM-7600F (Tokyo, Japan) was used. The chemical composition and the elemental analysis were obtained using energy-dispersive X-ray spectroscopy (EDS) from Oxford instruments, which were attached to the same FE-SEM microscope. Both SEM images and EDS analysis were performed before and after corrosion measurements in 1 M HCl solution.

## 3. Results and Discussion

#### 3.1. Electrochemical Measurements

The electrochemical studies of produced  $\gamma$ -TiAl alloy were performed in an aerated 1 M HCl solution at different periods of exposure using potentiodynamic polarization measurement. The potentiodynamic polarization curves show, in Figure 4, the  $\gamma$ -TiAl alloy after (1) 0 h, (2) 1 h, (3) 4 h and (4) 24 h immersion in 1 M HCl solution at room temperature. The corrosion parameters were determined by Tafel extrapolation method from the polarization curves and presented in Table 2. As previously reported in our earlier studies [10,11], the values of polarization resistance (R<sub>p</sub>), corrosion current density (j<sub>Corr</sub>), corrosion potential (E<sub>Corr</sub>), anodic Tafel ( $\beta_a$ ) slopes and cathodic Tafel ( $\beta_c$ ) slopes were obtained.



**Figure 4.** Potentio-dynamic polarization curves (PP) of  $\gamma$ -TiAl alloy after (1) 0 h, (2) 1 h, (3) 4 h and (4) 24 h immersion time in 1 M HCl solution at room temperature.

Exposure			Parameter		
Period/h	$\beta$ a/mV·dec <sup>-1</sup>	$\beta c/mV \cdot dec^{-1}$	ECorr/mV	jCorr/µA·cm <sup>−2</sup>	$Rp/k\Omega \cdot cm^2$
0	12.76	15.72	-674	0.857	3.568
1	13.69	15.51	-653	0.955	3.306
4	14.91	17.15	-634	1.456	2.378
24	15.62	17.34	-604	2.005	1.779

**Table 2.** Potentiodynamic polarization parameters obtained for  $\gamma$ -TiAl alloy in 1 M HCl solution at room temperature.

As presented in polarization curves, it is clearly seen that, with increasing the potential till the lowest current values, where the corrosion current density,  $J_{Corr}$ , becomes minimum, the cathodic current of the tested samples changes to a lower negative value. Then, the current increases in the anodic branch with the increase in the applied potential along with the aggressiveness of acid solution. It is also observed that with the increasing in immersion time, the values of  $J_{Corr}$  increases and the values of  $E_{Corr}$  shift towards the more positive direction. From Table 3 it is also confirmed that increasing immersion time decreases the polarization resistance ( $R_p$ ) and the dissolution rate due to corrosion increases because of the aggressive action of acid medium. More details about the dissolution of iron in acid medium mechanisms are discussed in previous work [12]. However, due to the tough and continuous acid attack on the steel surface, it does not allow for any oxide or corrosion product formation that can reduce corrosion rate. Further enhancment of the applied potential in the less negative direction enables steel corrosion, as shown by the increase in anodic currents with potential and immersion time.

Parameter Temperature/°C βa/mV·dec<sup>−1</sup> βc/mV·dec<sup>−1</sup> ECorr/mV jCorr/ $\mu$ A·cm<sup>-2</sup> Rp/k $\Omega$ ·cm<sup>2</sup> 30 13.17 16.02 -6030.926 3.389 40 16.17 0.985 3.238 13.46 -61150 15.74 17.22 -6251.364 2.618 16.48 17.94 -6381.925 1.937 60

**Table 3.** Potentiodynamic polarization (PP) parameters obtained for  $\gamma$ -TiAl alloy in 1 M HCl solutions after different temperatures.

Potentiodynamic polarization analysis was done on  $\gamma$ -TiAl alloy in 1 M HCL acidic solution in order to check the effect of increasing temperature on its corrosion. Figure 5 displays the obtained polarization curves for  $\gamma$ -TiAl alloy in 1 M HCl solution at (1) 30 °C, (2) 40 °C, (3) 50 °C and (4) 60 °C, respectively. The values of corrosion parameters  $\beta c$ ,  $\beta a$ , ECorr, jCorr and Rp obtained after polarization analysis are presented in Table 3. The corrosion of alloys increased with the increasing temperature via increment in anodiccathodic currents and j<sub>Corr</sub>, while the E<sub>Corr</sub> and Rp values decreased and shifted to lower negative. It is clear from the results presented in Table 3 that increase in temperature significantly increased the corrosion of alloys in the presence of acidic medium. This increase in corrosion is because of increase in alloy's surface activation with increasing temperature, which increased the rate of dissolution in acidic medium, which in turn increased its corrosion [13,14].



**Figure 5.** Potentiodynamic polarization (PP) behavior for  $\gamma$ -TiAl alloy in 1 M HCl solution at (1) 30 °C, (2) 40 °C, (3) 50 °C and (4) 60 °C, respectively.

#### 3.2. Electrochemical Impedance Spectroscopy (EIS)

To analyze the corrosion and passivation phenomena of different types of alloys and metals electrochemical impedance spectroscopy (EIS) is the well-known method for analysis in various types of corrosive environments [15–20]. Corrosion analysis of  $\gamma$ -TiAl alloy was carried out in 1 M HCl solution. The analysis was performed at different intervals of time at room temperature; the nyquist plots obtained after different intervals of time are presented in Figure 6. A similar type of analysis was also performed on alloys with increasing temperature of testing solution with temperatures at 30, 40, 50 and 60 °C; the analysis curves obtained for these tests are shown in Figure 7. The results were extracted by fitting the obtained curves of both the results shown in Figures 6 and 7, with an equivalent circuit model shown in Figure 8.

The Rs in the equivalent circuit model describes the solution resistance, Rp is the polarization resistance that can also be defined as charge transfer resistance and Q is the content phase element. The obtained values of parameters on the analysis performed at room temperature with different time intervals of exposure are presented in Table 4, whereas the obtained parameter values for the test conducted at different temperatures are shown in Table 5.

As can be seen from Figure 6, only one depressed semicircle was obtained for all the samples even after prolonged exposure up to 24 h; also, the diameter of semicircle decreased. The frequency dispersion because of non-homogeneities is the main cause of path deviation from ideal semicircle [21,22]. It can also be seen from Table 4 that the values of Rs did not vary much, while the values of Rp were found to decrease. On the other hand, the values of Q increased with the immersion time. The CPE with their "n" value less than one, according to Macdonald et al. [23], represent impure double layer capacitance to reduce the effects due to surface irregularities.



**Figure 6.** Nyquist plots for  $\gamma$ -TiAl alloy after (1) 0.0 h, (2) 1.0 h, (3) 4.0 h and (4) 24 h immersion in 1 M HCl solution at room temperature.



**Figure 7.** Nyquist plots for  $\gamma$ -TiAl alloy in 1 M HCl solution at (1) 30 °C, (2) 40 °C, (3) 50 °C and (4) 60 °C, respectively.



Figure 8. Equivalent circuit model that fits the EIS experimental data.

Table 4. EIS	parameters of	btained for <sup>-</sup>	γ-TiAl allc	y in 1 M	HCl so	lutions at ro	om temperature.
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Exposure Period/h	P /O?	Q	Q		
	K <sub>S</sub> /Ω·cm <sup>-</sup>	CPE (mMho)	n	$K_{ct}/\Omega \cdot cm^{-1}$	
0	3.22	0.534	0.946	880	
1	3.83	0.712	0.992	520	
4	3.19	0.901	0.892	460	
24	3.43	1.294	0.821	230	

**Table 5.** EIS parameters obtained for  $\gamma$ -TiAl alloy in 1 M HCl solutions after different temperatures.

Temperature/°C	P (0?	Q	<b>P</b> ( <b>O</b> and <sup>2</sup>	
	$K_S/M^2 cm^2$	CPE (mMho)	n	$K_{ct}/12 \cdot cm^{-1}$
30	3.48	0.594	0.918	764
40	3.69	0.662	0.903	676
50	3.42	0.786	0.885	512
60	3.35	0.998	0.847	446

The effect of increasing temperature on the corrosion of alloys exposed to 1 M HCL solution was also studied with EIS and obtained curves at different temperature interval is shown in Figure 7. The nyquist curves were obtained at the temperatures of 30 °C, 40 °C, 50 °C and 60 °C, while the data fitting was done with the help of same circuit shown in Figure 8. With varying temperature, the diameter of the depressed semicircle in nyquist plot was also found to be decreasing. With decreasing diameter of the semicircle, the Rp values were also found to decrease similar to time-exposure experiment. The values of CPE were found to increase with their corresponding "n" values close to 1.

In order to obtain a clear image of time and temperature exposure, bode plot was obtained for both time exposure and increasing temperature experiment. The bode plot obtained for 0 h to 24 h exposure is shown in Figure 9, whereas the bode plot obtained for alloys exposure to 30 °C, 40 °C, 50 °C and 60 °C are shown in Figure 10. It can be seen in the Figures 9 and 10 that the impedance values in bode plot for both the time and the increasing temperature exposure decreased throughout the complete applied frequency sweep. It can be concluded from the experiments that both the increased exposure time and temperature of the solution caused the decrease in corrosion resistance of the alloys in acidic medium.

The results obtained were conclusive and verified the EIS experiments where the Rp in both the cases decreased with prolonged exposure time as well as increased temperature; a similar type of trend was also observed with the polarization experiments, where the polarization resistance was found to decrease with exposure time and increased temperature.



**Figure 9.** Bode (**a**) impedance of the interface and (**b**) the phase angle plots for  $\gamma$ -TiAl alloy after (1) 0 h, (2) 1 h, (3) 4 h and (4) 24 h immersion in 1 M HCl solution at room temperature.



**Figure 10.** Bode (**a**) impedance of the interface and (**b**) the phase angle plots for  $\gamma$ -TiAl alloy after (1) 30 °C, (2) 40 °C, (3) 50 °C and (4) 60 °C.

## 3.3. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) Analyses

In order to investigate the surface of  $\gamma$ -TiAl alloy before and after the exposure to 1 M HCl solution, FE-SEM and EDX analysis were performed on the surface to quantify the alloy elements before and after exposure. The SEM micrographs and EDX investigations were collected using a JEOL microscope (JEOL-JSM 7600F, Tokyo, Japan) with an EDX unit attached from OXFORD instruments; the scanning operated at 5 kV. EDX analysis was spotted on 300 µm square area for three different locations, and average values were taken. The surface analysis (SEM) and EDX of the  $\gamma$ -TiAl alloy before immersion into 1 M HCl solution are shown in Figure 11. A clear and homogenous surface can be observed for the unexposed sample, while the EDX elemental analysis of the alloy representing atomic



percentages found in the alloy's surface are described as following: 47.07% Al, 48.44% Ti, 2.46% Nb and 1.83% Cr.

**Figure 11.** SEM micrograph and EDX profile analysis for the  $\gamma$ -TiAl alloy before its immersion in 1 M HCl solution.

After immersing the  $\gamma$ -TiAl alloy for an hour in 1 M HCl solution at room temperature followed by forward directional potential scanning from -0.08 to 0.0 V (Ag/AgCl), SEM and EDX analysis were performed for the sample, and obtained image and EDX analysis are shown in Figure 12. It is clear from the obtained figure that immersing the sample in acidic medium changes the surface of sample—the surface is no longer smoother and is completely different from the unexposed sample. It is to be noted that the immersion of sample in 1 M HCl solution and applying potential resulted in development of shallow pits. The EDX analysis was also performed on this exposed alloy sample, and slight changes were recorded in the obtained atomic percentages, as follows: 40.18% Al, 46.69% Ti, 2.25% Nb, 1.65% Cr and 9.22% O. It can be noticed clearly that the percentage of Al decreased after exposure. This is because of the corrosion of alloy, which resulted in dissolution of Al under acidic environment and increased applied anodic potential. It also worth to notice that the appearance of oxygen in the EDX of exposed alloy is most likely because of the formation aluminum oxide layer, which also led to the corrosion protection of  $\gamma$ -TiAl alloy.



**Figure 12.** SEM micrograph and EDX profile analysis for the  $\gamma$ -TiAl alloy after its immersion 1 h in 1 M HCl solution followed by sweeping the potential from -0.8 V to 0.0 V vs. Ag/AgCl.

On the other hand, Nb and Cr atomic percentages are almost identical for unexposed and exposed samples, which also confirms that changing state of Al to its oxide is the main reason for protecting corrosion of the alloy.

## 4. Conclusions

Gamma Titanium based alloy (Ti-48Al-2Cr-2Nb) was successfully fabricated through the powder metallurgy route, then processed further via electron beam melting (EBM) method. The electrochemical corrosion performance of the fabricated samples was studied at different immersion times in 1 M HCl solution with different temperatures using both the EIS technique and the potentiodynamic polarization technique. The morphology of the exposed surfaces was characterized before and after immersion for corrosion tests using FE-SEM and EDX elemental analysis. The corrosion behavior of the EBM-produced titanium-based alloy was found to be excellent, as concluded from the high values of corrosion potential and polarization resistance results, which were confirmed by the low values of the corrosion rate/current.

It has been found that there is a proportional relationship between the corrosion resistance and the immersion time, i.e., the corrosion resistance of the fabricated alloy is increasing through decreasing both cathodic and anodic currents, which shifts the corrosion potential positively. On the other hand, it was found that there is an inverse relationship between the solution temperature and the corrosion resistance. Increasing the solution temperature from 30 to 60 °C leads to an increase in the corrosion parameters, which were measured by both impedance and polarization techniques. Surface morphology investigations before and after corrosion tests show and confirm the creation of precipitation of an oxide layer (dissolution of aluminum) covering the exposure surface and protecting it against corrosion as assured by FE-SEM and EDX analysis. Finally, the final percentages of other alloy elements (Cr and Nb) remained constant after corrosion tests, which proved the indirect protection due to the formation of AlO<sub>2</sub> layer.

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