



# Investigation of the effects of SiC reinforcement ratio in iron-based composite materials on corrosion properties

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## Synopsis

The corrosion properties of iron-based composite materials containing graphite and silicon carbide (SiC) reinforcement were investigated. The effects of silicon carbide reinforcement were investigated by adding 0.5% graphite and 1%, 2%, and 4% SiC. A powder metallurgy method was used to produce the composite samples. Iron, graphite, and silicon carbide powders were blended for one hour with a three-axis mixer and then unidirectionally pressed under a pressure of 750 MPa. After pressing, the composite materials were sintered at 1100°C for one hour. The corrosion properties and microstructure, density, and hardness properties of the composite materials that can affect the corrosion properties were also investigated. It was determined that the pore ratio and hardness of the composite material increased, and corrosion resistance decreased, with increasing silicon carbide content.

## Keywords

iron, silicon carbide, SiC, composite, hardness, corrosion.

## Introduction

Composite materials have been produced and used for many years due to their advantageous properties. Composite materials are defined as materials that are formed to combine the best properties of two or more similar or different groups of materials in a new material. These materials are composed of reinforcing elements and surrounding matrix material. The main function of the matrix is to transmit load to the reinforcing elements, and that of the reinforcing elements in the composite structure is to carry the load. (Hull and Clyne, 1996; Balasubramanian, 2013; Saxena *et al.*, 2017).

Although iron metal has good ductility and toughness, its mechanical properties such as hardness, yield strength, tensile strength, abrasion resistance, and fatigue strength are quite poor. The low-strength properties of iron and low-carbon steels limit the use of these metals. By adding carbon and various alloying elements or hard ceramic particles to iron or cast iron, iron-based composite materials are obtained (Wu *et al.*, 2019; Majumdar *et al.*, 2008; Szklarz *et al.*, 2018). Composite materials containing a matrix metal are called metal matrix composites (MMCs) (Smith, Hashemi, and Prakash, 2014; Kataria and Mangal, 2018). Metallic matrix materials are generally Fe, Al, Cu, Ti, or Co metals or alloys (Chang *et al.*, 2018). While copper-based metal matrix composites constitute the majority of the market by mass, significant amounts of Fe and Ti composites are also produced (Malaya, Troy, and Sarat, 2019). Different ceramic reinforcements such as SiC, Cr<sub>3</sub>C<sub>2</sub>, TiC, Ti (C, N), WC, VC, CrB, and Cr<sub>2</sub>O<sub>3</sub> are used in the production of high-strength iron-based composite materials (Wu *et al.*, 2019; Majumdar *et al.*, 2008; Thawari, Sundararajan, and Joshi, 2003). One of the most powerful reinforcing elements in MMC production is SiC particles (Wu *et al.*, 2019). In SiC-reinforced MMCs, very high hardness, yield strength, abrasion resistance, high-temperature oxidation resistance, and creep resistance are obtained (Wu *et al.*, 2019; Majumdar *et al.*, 2008; Szklarz *et al.*, 2018; Thawari, Sundararajan, and Joshi, 2003; Abenojar *et al.*, 2002, 2003). In addition to these excellent physical properties, SiC reinforcing elements are also cost-effective (Chang *et al.*, 2018).

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As is known, the mechanical properties of the composites are improved by the presence of rigid reinforcing elements. Chang *et al.* (2018) reported that Ti- and Mo-coated SiC-reinforced iron-based composites had higher hardness and flexural strength than coated composites. There are few studies on the corrosion properties of SiC-reinforced iron-based composite materials. In some of these studies, SiC particles were added to the structure by a powder metallurgy method, while in others liquid production methods or SiC-containing composite surface coating methods were used (Wu *et al.*, 2019; Majumdar *et al.*, 2008; Szklarz *et al.*, 2018; Thawari, Sundararajan, and Joshi, 2003; Abenojar *et al.*, 2002, 2003). In a study by Wu *et al.* (2019), phase transformations, microstructure, hardness, and corrosion behaviour of SiC-reinforced 316 L stainless steel matrix composites were investigated. It was reported that the grain size of the solidification microstructure was reduced by the addition of SiC, and the hardness increased due to solid-solution and second-phase hardening. Solid-solution strengthening by C and Si originated from the decomposition of SiC. With the addition of SiC,  $\alpha$ -(FeCrNi) phases were precipitated out of the  $\gamma$ -(FeCrNi) phases and the inherent characteristic of  $\alpha$ -(FeCrNi) was hard. The corrosion resistance decreased with increasing SiC ratio.

Szklarz *et al.* (2018) added SiC particles to Fe Co Cr Mn Ni alloy powders by mechanical alloying to investigate the effects of SiC reinforcement on the electrochemical properties of the composites. It was reported that with the addition of SiC, the mechanical properties of the composite were improved and the corrosion properties deteriorated (Szklarz *et al.*, 2018). Abenojar *et al.* (2002, 2003) investigated SiC reinforcement in iron-based 316L stainless steel composite and the effects of sintering media during production. They reported that with increasing SiC content the hardness and abrasion resistance of the composite increased and corrosion resistance decreased. Ramesh, Srinivas, and Channabasappa (2009) produced iron-based SiC-reinforced composites by laser sintering. They observed an increase in micro-hardness and a decrease in density with increasing SiC content. Volumetric wear rate decreased with increasing SiC content.

Fe-based composites are used for many applications. Some examples in the literature include composites for biodegradable implant applications (Malgorzata, 2018), AC magnetic properties (Kollár *et al.*, 2010), anti-friction composites (Babets, Vasil'ev, and Ismailov, 2012), composites with very good abrasion resistance (Hulin, Haiping, and Jianhong, 2019), intermetallic-ceramic composites combining the properties of ceramics and metals (Kopeck, Joźwiak, and Kowalewski, 2021), increasing the strength of metal materials (Zemtsova *et al.*, 2020), and use as anode in lithium-ion batteries (Uzunov *et al.*, 2007).

Most of the works on SiC-reinforced iron-based composites are related to the mechanical properties. There are few studies on the corrosion properties of these materials. Stainless steel matrix composites are generally used in a limited number of studies on corrosion of SiC-reinforced iron-based composite materials. In this study, we report on the hardness properties of iron-based composite materials with different amounts of SiC ceramic reinforcement that were produced by a powder metallurgy method.

## Experimental studies

### Materials and method

Iron-based composite materials containing 0.5% graphite and 1%,

2%, and 4% SiC were produced. The average grain size of the SiC powders used as reinforcing elements was 8  $\mu\text{m}$ . The Graphite-UF4 powders had a particle size below 20  $\mu\text{m}$ , and the average grain size of the iron powders used as a matrix material was 100  $\mu\text{m}$ . Zn stearate was used as a lubricant in all mixtures. Table I shows the chemical composition of the iron-based composite materials used in the experimental studies.

Although SiC reinforcement powders improve the hardness and wear resistance of the composite material, the electrochemical behaviour of SiC differs from that of the iron matrix material. Therefore, a galvanic couple is formed between iron matrix and SiC reinforcement, and the corrosion behaviour of the composite material is greatly affected.

The powders were weighed to 0.0001 g with a RADWAG AS-60-220 C/2 precision scale in the ratios given in Table I. The weighed powders were mixed with a three-axis mixer without a ball for one hour. The homogeneously blended powders were compacted at a pressure of 750 MPa to form cylindrical specimens 32 mm in diameter and 10 mm thick. Sintering of the samples was carried out in a 90% nitrogen 10% hydrogen atmosphere at 1100°C for one hour. The density of the composites was determined using the Archimedes method and the hardness tests were carried out by applying a 1 kg load using a Shimadzu microhardness device. Hardness measurements were taken from five points for each sample and the average value accepted as the hardness result.

Corrosion tests were carried out using a potentiodynamic (electrochemical) method. A 3.5% NaCl solution was used as the corrosion medium. In the corrosion cell, SiC-reinforced iron-based composite samples were used as the working electrode (anode), platinum plate as the counter-electrode (cathode), and a saturated calomel electrode (SCE) as the reference electrode. The composite working electrodes were cold-moulded with resin on the surfaces so that a surface area of 1  $\text{cm}^2$  was exposed and in contact with the corrosion environment. Before each electrochemical measurement, the exposed surfaces of the samples were cleaned by sanding and all measurements were performed on the surfaces with the same properties. Electrochemical corrosion tests were carried out in two stages. In the first stage, polarization curves were obtained between -1.5 V and -0.2 V, *i.e.* from the cathodic region to the anodic region and back to the cathodic region at a scan rate of 0.1 V/s. The purpose was to examine the general corrosion behaviour of the composite material, the immunity, and passivation and corrosion zones. In the second stage, Tafel polarization curves were obtained through scanning potentials between -1.5 V and -0.4 V at 0.002 V/s scan rate. The corrosion potential ( $E_{\text{corr}}$ ), corrosion current density ( $I_{\text{corr}}$ ), and corrosion rates of composite samples were determined from these curves.

Table I

### Chemical composition of composite samples (mass fraction)

	Graphite	SiC	Fe
Composite 1	0.5	1	Balance
Composite 2	0.5	2	Balance
Composite 3	0.5	4	Balance

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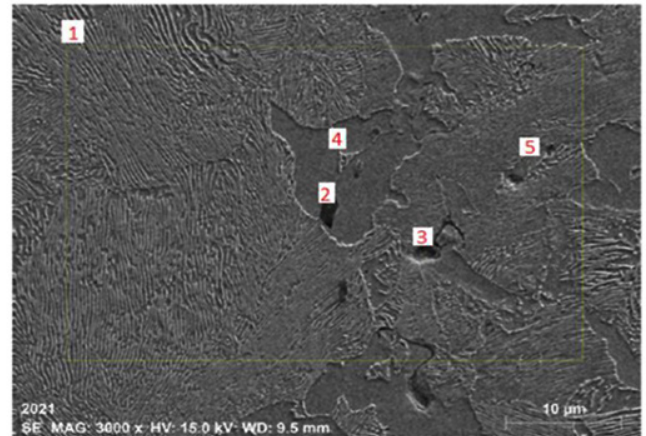
## Results and discussion

The microstructure and analyses of the phases of the iron-based composite sample are shown in Figure 1. The structure consisted of ferrite and perlite phases and SiC particles. In the SEM micrograph, the lamellar grains are the perlite phase, the flat grains are the ferrite phase, and the dark particle is the SiC reinforcing element. The EDS results also confirm this view. In the micrograph spectrum 2 is a SiC reinforcement particle, and spectrum 3 is a pore. It is thought that the high iron content at point 2 is due to the contact of the spectrum with the interface.

Table II shows the density and hardness values of the samples after sintering. Theoretical densities of the mixed powders were calculated using the mixing rule. The density of each component making up the composite was calculated separately and summed. It is known that in powder metallurgy production, after sintering, as a result of the combination of powders by diffusion the density of the material increases. This increase depends on the original density, compaction pressure, and other factors. There can be a significant increase in density if there is significant amount of porosity prior to sintering. Although the densities of the samples after sintering were close to each other, it was observed that the density of the composite material decreased with an increase in SiC reinforcement content. The density of the iron-based composite material containing 1% SiC reinforcement was 7.3468 g/cm<sup>3</sup>, decreasing to 7.1727 g/cm<sup>3</sup> with a SiC content of 4%. The relative density of the composite material decreased from 94.36% to 93.80% when the SiC reinforcement ratio was increased from 1% to 4%. As the SiC content increased, the pore ratio increased and accordingly the density of the composite material decreased slightly. It is believed that the increase in the pore ratio is a result of the increase in the hardness of the composite mixture powders with the increase in the ratio of hard and high-strength SiC reinforcing elements and consequently the weakening of the compressibility of the mixing powders during pressing.

With increasing SiC content, the hardness values of the composite materials also increased. The hardness of the iron-based composite material containing 1% SiC reinforcement was 110 HV. The hardness values increased to 235 HV and 281 HV when the SiC reinforcement content was increased to 2% and 4% respectively. SiC is a ceramic-based material with high hardness and wear resistance, which when added to the composite structure improves the hardness of the material. Various studies of different iron-based SiC-containing composite materials produced by different manufacturing methods have shown enhancement of the mechanical properties such as hardness and wear resistance (Wu *et al.*, 2019; Szklarz *et al.*, 2018; Abenojar *et al.*, 2002, 2003; Ramesh, Srinivas, and Channabasappa, 2009; Pelleg, 1999).

Polarization curves and Tafel polarization curves of composite materials obtained in a 3.5% NaCl medium are shown in Figures 2 and 3. Corrosion data obtained from Tafel polarization curves



Mass percent (%)

Spectrum	C	Si	Fe
1	5.74	0.57	93.69
2	14.01	65.12	20.87
3	18.14	0.59	81.27
4	11.59	0.39	88.03
5	4.14	0.72	95.14
Mean value:	10.72	13.48	75.80
Sigma:	5.80	28.87	31.19
Sigma mean:	2.60	12.91	13.95

Figure 1—SEM image and EDS analysis of the SiC-reinforced iron-based composite material

is given in Table III, and surface SEM images of the samples after corrosion in Figure 4.

Figure 2 shows the behaviour of the cathodic and anodic potentials in the polarization curves for the iron-based composite materials containing different ratios of SiC. There are three regions in the polarization curves. Low potential ranges are the immunity zone. There was no corrosion of the composite material in this region. With increasing potential the curve enters the passivation zone. Although the potential increased in this region, the current density did not change. Corrosion resistance is imparted by the oxide film formed on the surface of the material in this region. The passivation zone in iron-based SiC-reinforced composite samples covers a very narrow range. This is attributed to the deterioration of the integrity of the passive film by the SiC reinforcement. The third region starts with the potential increasing to higher values. This region is the corrosion region, where the current density suddenly increases. At a SiC reinforcement content as low as 1%, the increase in current density took more positive potentials, such as -0.750 V, whereas where SiC content was higher, at 2% and 4%, the increase in

Table II

Density and hardness values for SiC-reinforced iron-based composite materials

	SiC ratio (wt.%)	Experimental density (g/cm <sup>3</sup> )	Theoretical density (g/cm <sup>3</sup> )	Relative density (%)	Hardness value (HV <sub>1</sub> )
Composite 1	1	7.3468	7.7856	94.36	110
Composite 2	2	7.2909	7.7392	94.20	235
Composite 3	4	7.1727	7.6464	93.80	281

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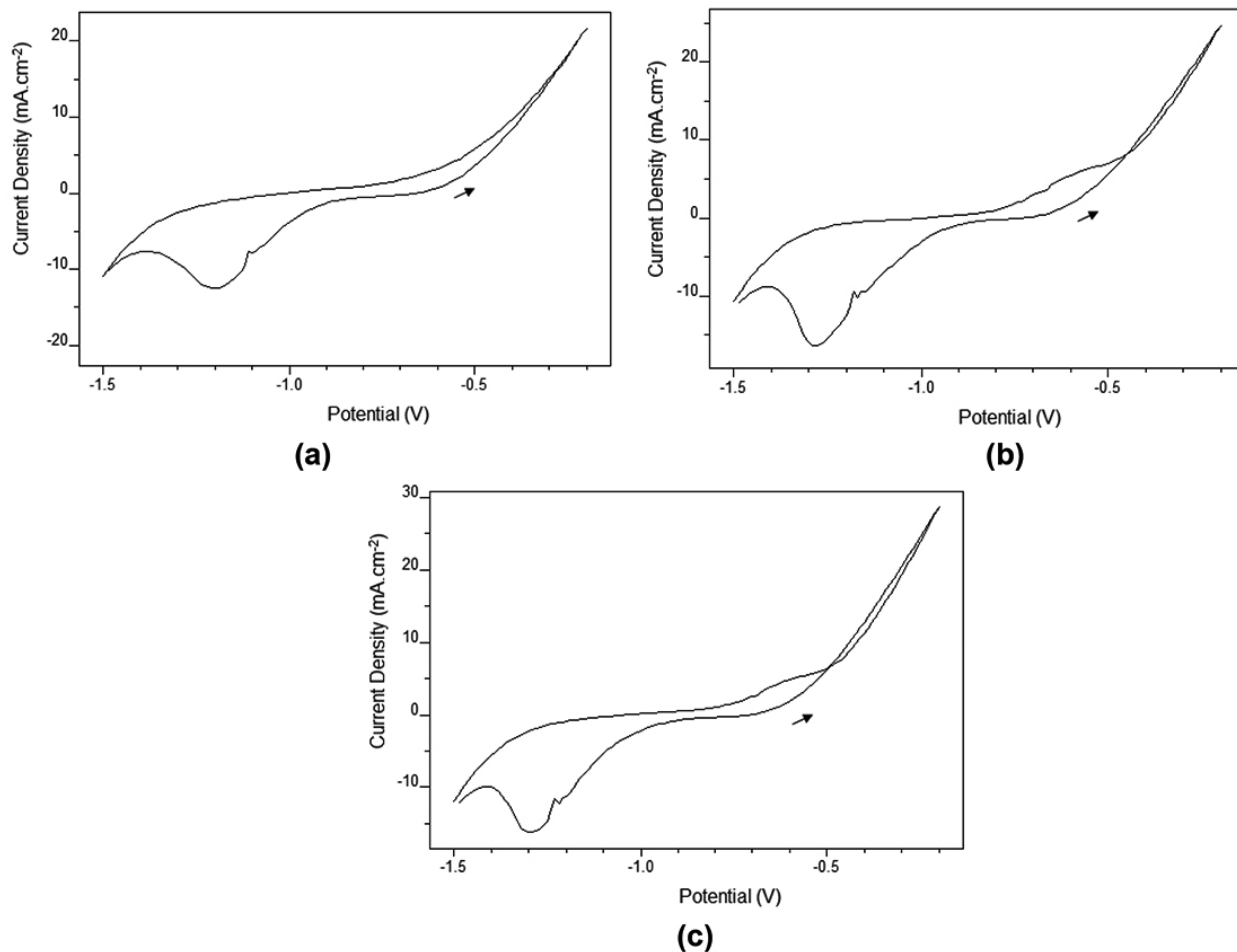


Figure 2—Polarization curves for (a) 1%, (b) 2%, and (c) 4% SiC-reinforced iron-based composite materials in a 3.5% NaCl medium

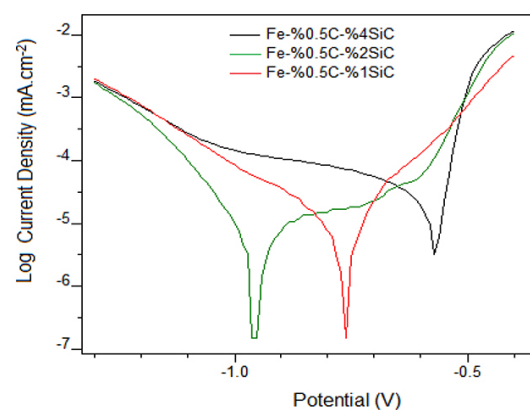


Figure 3—Tafel polarization curves for SiC-reinforced iron-based composite materials in a 3.5% NaCl medium

current density was in some negative values, around  $-0.850$  V. The fact that the current density increased to more positive potentials, and that metal dissolution started at more positive potential values, is an indication of the material's resistance to corrosion. In addition, at the highest potential value in the anodic direction,  $-0.2$  V, the current densities were approximately  $20 \text{ mA}\cdot\text{cm}^{-2}$  for the 1% SiC composite,  $25 \text{ mA}\cdot\text{cm}^{-2}$  for the 2% SiC composite, and  $30 \text{ mA}\cdot\text{cm}^{-2}$  for the 4% SiC composite. In other words, with increasing SiC content with the current density at the most anodic potential applied, metallic dissolution also increased.

According to the corrosion current density and corrosion rate values obtained from the Tafel polarization curves, corrosion current density and corrosion rate increased when the SiC content of the composite material increased. As seen in Table III, the corrosion rate was  $0.0944$  mm/a with 1% SiC reinforcement,  $0.1125$  mm/a with 2% SiC, and  $0.4302$  mm/a with 4% SiC reinforcement. When the SiC reinforcement content increased from 1% to 2%, the increase in corrosion rate was small, whereas when the reinforcement content increased to 4% a four-fold increase in corrosion rate was observed.

## Investigation of the effects of SiC reinforcement ratio in iron-based composite materials

Since SiC ceramic particles in the structure have high hardness, high strength, and high abrasion resistance, they improve weak properties of the composite material. However, the SiC particles have different electrochemical properties from iron matrix and disrupt the electrochemical homogeneity of the structure. SiC particles form cathodic regions in the composite material, while the iron matrix forms anodic regions. As the SiC ratio increases, the cathodic regions and cathode-anode interfaces increase in the composite material structure, while anodic regions decrease. The decrease of anodic zones and increase of cathodic zones (small anode area, large cathode area) accelerates dissolution in the anodic zones. As a result of the decrease in the anodic area, the current density at the anode increases. The reinforcement-matrix interfaces, which are the cathode-anode interfaces, are sensitive areas in the corrosion of composite materials because these interfaces have a more irregular atomic structure and the atomic defect density is high in these regions. This situation can be attributed to the fact that the iron matrix-SiC ceramic interface provided sites for active dissolution, leading to local weakness of the passive films. Similar results were obtained in a study on SiC-reinforced stainless steel composite materials (Wu *et al.*, 2019).

As a result of the increase in hardness with increasing SiC content, the compressibility of the composite powder mixture during pressing decreased. This led to an increase in the pore ratio and a decrease in the density of the material. Pore-type structural defects are main factor that reduces the corrosion resistance of the material. It is considered that the increase in hardness and pore ratio with increasing SiC content is another factor effective in decreasing corrosion resistance.

In the SEM images of the samples after corrosion in Figure 4, small pits, large pits, and fractures can be seen on the surfaces of the iron-based composite materials containing different ratios of SiC.

Another negative effect of the reinforcement in composite materials in terms of corrosion resistance is that it prevents the formation of a continuous passive film on the surface of the material (Wu *et al.*, 2019). In metallic materials, thin, stable, and compact structures on the surface of the material and continuous oxide metal films form without leaving defects on the surface, and slow down the corrosion rate. While the formation of a passive film is more protective in pure metals, and some alloys such as stainless steel, passive films generally do not form in the alloys to slow surface corrosion. Likewise, a continuous passive film cannot

Table III

Corrosion data for SiC-reinforced iron-based composite samples

SiC content wt.%)	Corrosion potential ( $E_{\text{corr}}$ ) (mV)	Corrosion current density ( $I_{\text{corr}}$ ) ( $\text{A}\cdot\text{cm}^{-2}$ )	Corrosion rate (mm/a)
1	-761	$8.041\cdot 10^{-6}$	0.0944
2	-953	$9.576\cdot 10^{-6}$	0.1125
4	-587	$3.663\cdot 10^{-5}$	0.4302

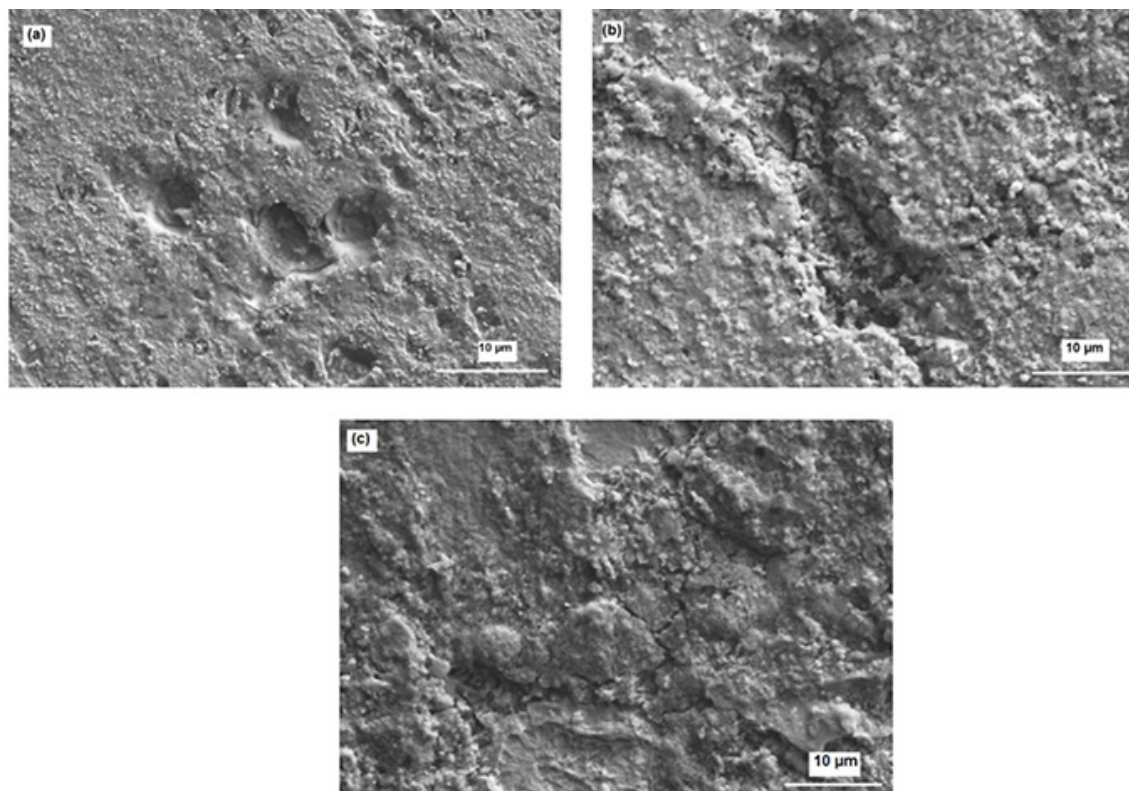


Figure 4—Surface SEM images of (a) 1%, (b) 2%, and (c) 4% SiC-reinforced iron-based composite materials after corrosion

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be formed in composite materials with different reinforcements, since the passive film formed by the matrix material combining with oxygen is interrupted in the areas which are reinforced on the surface. In these regions, where a passive film cannot form, corrosion accelerates due to the interaction of the matrix material with the medium.

## Conclusions

The effects of the addition of SiC in different ratios on the corrosion properties of iron-based composite materials were investigated. The main results can be summarized as follows

1. With increasing SiC reinforcement additions, the density of the composite material decreased and the pore ratio increased. The pore ratio increase was due to the increase in the reinforcement content and weakening of the compaction properties of the powder mixture during production.
2. With increasing SiC reinforcement, the hardness of the composite material increased linearly. The highest hardness was obtained in the 4% SiC-reinforced composite material. The increase in hardness is due to the superior mechanical properties of the SiC reinforcing material.
3. The addition of SiC up to 2% slightly increased the corrosion rate of the composite material, while the addition of 4% SiC caused a large increase in the corrosion rate. This is attributed to an increase of the cathodic regions and decrease of the anodic regions, an increase in the anode-cathode interfaces, increase in the porosity, and the discontinuity of the passive film with increasing reinforcement.

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