Fabrication of Cu-adhered silicon carbide particles and its effect on properties of Fe-based matrix composites

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Abstract

In present study, copper powders (Cu) and silicon carbide (SiC) particles were used to produce Cu-adhered SiC particles (Cu/SiC), which were used to reinforce Fe-based matrix materials to modify properties. The orthogonal experimental design was used to investigate the relationship between ball mill parameters and particle size. Next, the mixed powders were pressed at 500 MPa using a hydraulic press. Then, the green compacts were wrapped in graphite powders, and sintered using a resistance furnace. Metallurgical microscopy, scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDS) and x-ray diffraction were employed to investigate the microstructures, element distribution, and phase of SiC-reinforced Fe-based matrix composites. The properties of the SiC-reinforced metal matrix composites were determined using the Microhardness test and Charpy pendulum impact test. The orthogonal experimental results indicated that the influence degree of milling parameters on particle size was the powder to ball ratio > milling time > milling speed. The milling parameters to obtain the smallest Cu/SiC particle were powder to ball ratio of 20:1, milling time of 15 h, and milling speed of 300 r min⁻¹. However, the adhesive effect was bad. The properties test results indicated that Cu/SiC particles reinforced with Fe matrix composites had better properties. Furthermore, the hardness and impact toughness improved up to 239.97 HV0.5 and 12.1 KJ·m⁻², respectively. Moreover, compared to raw SiC particles, the hardness and impact toughness increased by 12% and 15%, respectively. The improvement in properties was attributed to the Cu adhesion to the SiC surface, which effectively alleviated the difference in thermal expansion coefficient between SiC and Fe, and formed a chemical bond at the interface to improve the interfacial binding

1. Introduction

In powder technology, large-scale production equipment, such as ball mills, roller presses and jaw crushers, have serious wear problems during production. Especially in complex working conditions of impact and wear, the loss of metal materials is very huge. According to statistics [1], Chinese iron ore dressing plants consume more than 80,000 tons of liners every year, which causes a huge loss in metal materials and economy. Therefore, it is of great significance to choose suitable wear-resistant materials to reduce material loss. Fe-based wear-resistant materials, such as high manganese steel, cast iron and alloy steel, have been widely used in manufacturing wear-resistant products, depending on the well tribological properties [2–4]. However, more severe working conditions have put forward higher requirements on the properties of present Fe-based materials. Fe-based materials are required to have both high hardness and toughness as a whole. However, if the hardness of Fe-based materials are increased, the toughness of the material would decrease. Thus, the application of Fe-based materials is limited, and it remains challenging for traditional Fe-based wear-resistant
materials to be widely used in actual production, with large impact loads, high speeds and high temperatures. As a consequence, Fe-based materials needs to be modified to adapt to new working conditions.

Recently, a number of studies have reported that adding hard ceramic particles (TiC, silicon carbide [SiC], Al₂O₃, WC, etc) into the Fe matrix is an effective solution to improve the properties of Fe-based materials [5–8]. By combining the plasticity of the matrix and strength of reinforcements, these composites are expected to be used in impact wear conditions due to its high toughness and good wear resistance. Among the number of ceramic particles, SiC has been deemed as an ideal reinforcement due to its outstanding physical properties, such as high strength, high hardness, high wear resistance, corrosion resistance, and high temperature stability. In addition, SiC is inexpensive and easily available. The variety of advantages make SiC a promising reinforcement for use in manufacturing particle-reinforced metal matrix composites, and this has been widely used to strengthen various metal materials [9–12].

However, studies on SiC-reinforced Fe-based matrix composites have remained inconclusive. The reason was due to the difference in physical and chemical properties between SiC particles and the Fe matrix. On one hand, the Fe matrix and SiC particles have a large difference in thermal expansion coefficient. This means that when fabrication methods that involve high temperatures, such as powder metallurgy, casting and laser cladding, are used, residual stress occurs in the interface, between the matrix and reinforcement. This causes the growth of microscopic defects at the interface to accelerate, reducing the properties of the composites. On the other hand, the interface between SiC and Fe is thermodynamically unstable [13]. As reported by the literature, although SiC has high temperature stability, this would decompose into Si and C, and chemically react with the Fe matrix at a low temperature (over 900 °C) when mixed with Fe. There are various compounds in the Fe-SiC system, such as FeSi, Fe₃Si, Fe₅Si₃, Fe₇Si₉, and FeSi₂ [14]. These compounds all have large negative formation enthalpy. Hence, at high temperatures, there will be various reactions between SiC particles and the Fe matrix. As it is known, the interfacial reaction between SiC and Fe is the main factor that affects the properties of metal matrix composites [15]. Furthermore, the phase composition and the degree of reaction at the interface would affect the final performance of composites. Moreover, the manufacturing of SiC-reinforced Fe matrix composites is limited by the reaction between SiC and Fe. In summary, physical and chemical incompatibility limits the application of SiC-reinforced Fe-based matrix composites.

At present, forming a metallic coating on the surface of SiC particles has been considered as an effective method to improve the interfacial bonding between SiC particles and the Fe matrix. The conventional methods applied to obtain metal-coated SiC particles include electro-co-deposition, chemical vapor deposition (CVD), physical vapor deposition (PVD), and high-energy ball milling [16–21]. Various reports have confirmed that SiC surface metallization can effectively improve the interface bonding and properties of composites. Chang et al. [22] successfully prepared Ti-coated and Mo-coated SiC particles using spark plasma sintering, and fabricated metal-coated SiC particles-reinforced Fe-based matrix composites. The results revealed that both the Ti-coating and Mo-coating chemically bonded with the SiC particles and Fe matrix. Due to the action of the Ti-coating and Mo-coating, both Ti-coated and Mo-coated SiC particles reinforced the composites, allowing these to have better mechanical properties. Furthermore, Mo-coated composites have the best mechanical properties. The bending strength, impact strength and hardness is 15.9%, 27.8% and 4.7% higher, when compared to uncoated composites, respectively, and the wear mass loss is half of that for uncoated composites. Wang et al. [23] used powder embedding reaction coating technology to form a Cr coating layer on the SiC surface, and deposited Cr-coated SiC particles on the carbon steel substrate using plasma surfacing technology. The results indicated that the interface reaction led to chemical composition changes between the reinforcements and matrix, and that SiC chemically bonded with the carbon steel substrate. Compared to non-coated Fe-based alloys, the hardness and wear resistance of Cr-rich SiC coatings increased by 10% and 50%, respectively. Zhang et al. [24] manufactured Ni-coated SiC particles using chemical deposition, and sintered SiCp/Fe composites. The results revealed that the tensile strength and elongation of composites reinforced by Ni-coated SiC particles significantly improved. That is, the Ni coating effectively reduced the direct contact between SiC particles, and reduced the possibility of forming microcracks. Furthermore, the bonding strength also significantly improved.

Although the application of metallic coatings on the surface of SiC particles can improve the mechanical properties of composites, the expected property increase was not observed, and information related to metalized SiC reinforced Fe-based matrix composites remain limited. Thus, it is important to investigate the interface modification of SiC, and the manufacturing of high quality metal matrix composites. In the present study, the effects of SiC interface modification on the properties of SiC-reinforced Fe matrix composites were investigated. Copper (Cu) was chosen as the material to adhere to the SiC surface. The Cu/SiC particles were obtained by high energy ball milling, and the subsequent mixing with Fe powders to fabricate SiC-reinforced composites by
conventional sintering. The microhardness test and Charpy pendulum impact test were used to perform the comparison study of raw SiC and Cu/SiC particles on Fe matrix composites.

2. Materials and methods

2.1. Raw materials
Elemental Cu powders (>99.5% in purity, produced by Sinopharm Chemical Reagent Co., Ltd) and SiC particles (>99.99%, produced by Dongtai Aidong Abrasive Mould Factory) were used as raw materials to prepare the Cu/SiC particles. The particle size of the Cu powder was within the range of 250–300 mesh. SiC is an irregularly shaped particle, with a particle size of 2.118 μm and a D$_{50}$ of 24.875 μm. The matrix material was reduced iron powders (produced by Zhengzhou Hengchang Metal Material Co., Ltd), which had a particle size of 100 μm.

2.2. Preparation of Cu/SiC particles and the orthogonal experiment
The Cu/SiC particles were prepared by high energy ball milling. The mixed powders comprised of 40 wt% of SiC particles and 60 wt% of Cu powders. 304 stainless steel balls were used as the grinding and mixing media. Each container had 100 small balls with a diameter of 10 mm, and 20 large balls with a diameter of 20 mm. Furthermore, each container contained 100 ml of anhydrous ethanol, which was used as process control agent.

In order to make the raw SiC and Cu/SiC particles comparable, there was a need to control the size of the Cu/SiC particles. Thus, the L9($3^4$) table was used for the orthogonal experiment, in order to investigate the relationship between the ball milling parameters and particle size. Powder to ball ratio, milling speed and milling time were selected as the orthogonal design factors, and each factor had three levels. The factors and levels are listed in table 1. The median diameter (D$_{50}$) was set as the dependent variable. According to the results, raw SiC particles with the corresponding size were screened out using a sieve for the comparison experiment.

2.3. Preparation of Cu/SiC reinforced Fe-based matrix composites
Before pressing, the Cu/SiC particles and Fe powders were mixed by ball milling. Each ball mill jar contained four ø20-mm big balls and 20 ø10-mm small balls. These were used as stirrers, in order to uniformly disperse the Cu/SiC particles in the Fe powder. The ball milling parameters were as follows: powder to ball rate, 1:5; mill speed, 200 r min$^{-1}$; mix time, 15 min. The mixture of Cu/SiC particles and Fe powders was made into a rectangular specimen (10 × 10 × 55 mm$^3$) for the Charpy pendulum impact test and microhardness test. All the composites (no matter raw SiC reinforced composites or Cu/SiC reinforced composites) used for property tests all have the same SiC content–10% filling mass fraction, pressed at 500 Mpa. Using conventional sintering in a resistance furnace (XD-1200N-3, Zhengzhou Brother Furnace Co., Ltd), the green compacts were buried in graphite powders to prevent sample oxidation. Then, the specimens were heated from room temperature to 600 °C at a rate of 10 °C min$^{-1}$, allowed to rest for 30 min, and subsequently heated to 1,000 °C at a heating rate of 10 °C min$^{-1}$. Finally, the specimens were sintered at 1,000 °C for two hours, and cooled to room temperature at a rate of 10 °C min$^{-1}$.

2.4. Characterization of Cu/SiC particles and composites
The microstructure of Cu/SiC particles and interface reaction were observed by scanning electron microscopy (SEM; JSM-IT300, JEOL). The phase composition of the composites was determined by x-ray diffraction (XRD) with Cu-Kα radiation (SmartLab, Rigaku Corporation, 3KW), at a scanning speed of 10° min$^{-1}$. The element distribution was determined by energy dispersive spectroscopy (Oxford Instruments), which was combined with the scanning electron microscope to evaluate the metal coating on the SiC surface. The particle size of all powders was measured using a laser particle size analyzer (Mastersize 2000).

The microhardness test was performed using the Vickers Hardness Tester (MicroVicker TouchHV-1000A, MEGA Instruments [Suzhou] Co., Ltd) at a load of 500 gf for 15 s. The HV measurements were averaged by 10 repeated tests for each sample.

The Charpy pendulum impact test was performed using the Impact Testing Machine (JBSA-450, MEGA Instruments [Suzhou] Co., Ltd). The impact strength (P) is expressed in equation 1.

| Level | Powder to ball ratio | Speed of milling | Milling time |
|-------|----------------------|-----------------|-------------|
| 1     | 1:10                 | 200             | 5           |
| 2     | 1:15                 | 250             | 10          |
| 3     | 1:20                 | 300             | 15          |
The J in equation 1 refers to the expended work of a pendulum while the specimen broke off, and the S in equation 1 refers to the area of the fracture. The impact strength (P) refers to the average of three repeated tests. In the present study, the impact energy was 250 J, and the experimental environment was air.
3. Results and discussion

3.1. Characteristics of Cu/SiC particles

Figure 1(a) presents the digital image of raw SiC particles, showing the original state of SiC particles. The raw SiC particles were presented in green color. Figure 1(b) presents the SEM image of raw SiC particles. As shown in the figure, the SiC particles were irregularly shaped, and most of these presented as strips and blocks, with obvious edges and corners. At high magnification, as encircled in figure 1, it can be clearly observed that the surface of SiC particles was smooth, and that no foreign matter was adsorbed. After the SiC particles and Cu powders were fully mixed and grounded by high-energy ball milling, the Cu/SiC particles were placed in a vacuum drying oven for drying. Figures 2(1)–(9) presents the SEM images of Cu/SiC particles with nine different ball milling parameters. As shown in the image, for sample 1, 2, 3, 4, 5, 7 and 8, a large number of SiC particles still had lumps, with obvious edges and corners, but the surfaces were smooth, and had no attachments. Thus, in these seven samples, the SiC particles and Cu powders only mixed with each other, and the adhesive effect of the Cu powders was not good. However, the results for sample 6 and 9 were opposite to that for the previous samples. As shown in sample 6 and 9, most of the SiC particles were spherical, and the surface was rough. This means that some foreign substances attached to the surface of the SiC particle. In comparing the digital images of Cu/SiC (figures 3(a) and (b)) and raw SiC particles (figure 1(a)), it can be clearly observed that there was a clear change in color. The color of each part of the Cu/SiC particles is uniform, indicating that the adhesion effect of Cu is uniformly dispersed throughout the mixed powder.

To observe the adhesion of the foreign matter adsorbed by the SiC surface, EDS was performed on the surface of the SiC particles in sample 6 and 9. Figures 4(a) and (b) present the EDS mapping of sample 6 and 9.
respectively. It was apparent that a substantial amount of element Cu could be detected on the surface of SiC particles, and the content of Si was low. That is, these foreign substances were Cu powders, a relatively dense Cu powders had adhered to the SiC surface. In addition to the obvious adhesive effect observed on the surface of large particles, some elements of Cu could also be detected on the smooth surface of small SiC particles, as encircled in figure 4(b), but the content was low. During the process of milling, the Cu powder became deformed after this was initially impacted and subsequently attached to the surface of SiC particles. With the extension of ball milling time, the Cu powders continued to be refined. These refined Cu powders had high surface energy, and were in an unstable state. Eventually, these reduced the surface energy by agglomerating or adsorbing on the surface of SiC particles, allowing small sized SiC particles to have a Cu powders. Compared to sample 6 (the powder to ball ratio was 1:15, 300 r min\(^{-1}\), five hours), sample 9 (the powder to ball ratio was 1:20, 300 r min\(^{-1}\), 10 h) had a longer milling time and harder impact energy. Thus, the adhesion and crushing of Cu powders was more obvious. Furthermore, sample 9 had a more significant adhesive effect.

Figure 5(a) presents the XRD patterns for sample 6 and 9. As shown in the figure, merely the peaks of SiC and Cu could be observed in the XRD pattern, while the peak for copper silicide was not detected. This means that there were no obvious chemical reactions between the SiC particles and Cu powder during the process of ball milling, and that the Cu powder did not chemically bind with the SiC particles. Figure 5(b) presents the particle size distribution for sample 6 and 9. As shown in the figure, sample 6 and 9 had an approximate particle size distribution, the particle size of the two powders was distributed within the range of 2.7–40.0 \(\mu m\), and the \(D_{50}\) for sample 6 and 9 were 8.429 \(\mu m\) and 7.402 \(\mu m\), respectively. Furthermore, as shown in figures 2(6) and (9), it can be observed that particles smaller than 1 \(\mu m\) were mainly broken SiC particles and Cu powders. This made up the small percentage of the powder, indicating that the refining effect of ball milling was not obvious at this time, and that the deformation of Cu powders and the adhesion on the SiC surface mainly occurred at this stage. Therefore, Cu/SiC particles with a particle size of 5–20 \(\mu m\) occupied the main part of powders. The similar particle size distribution for sample 6 and 9 means that these two powders can be used as a comparative experiment, without considering the effect of particle size on the experimental results. All these phenomena indicate that the use of high energy ball milling can make the Cu powders adhere to the surface of SiC particles. This allows SiC particles and Cu powders to be physically bonded.
3.2. Results analysis for the orthogonal optimization experiment

The median diameter of particles with each ball mill parameter after ball milling and the range analysis results are listed in table 2 and table 3. Based on the data in table 3, the powder to ball ratio had the largest range, which was 2.413. This means that among the three factors, the powder to ball ratio had the greatest influence on the decline in particle size, followed by the milling time, and the influence of milling speed was minimal. Therefore,

Table 3. Results for the orthogonal optimization experiment.

| Number | Powder to ball ratio | Speed of milling, r min⁻¹ | Milling time, hours | D50, μm |
|--------|----------------------|---------------------------|---------------------|---------|
| 1      | 1:10                 | 200                       | 5                   | 12.302  |
| 2      | 1:10                 | 250                       | 10                  | 9.763   |
| 3      | 1:10                 | 300                       | 15                  | 8.376   |
| 4      | 1:15                 | 200                       | 10                  | 9.208   |
| 5      | 1:15                 | 250                       | 15                  | 8.147   |
| 6      | 1:15                 | 300                       | 5                   | 8.429   |
| 7      | 1:20                 | 200                       | 15                  | 6.649   |
| 8      | 1:20                 | 250                       | 5                   | 9.150   |
| 9      | 1:20                 | 300                       | 10                  | 7.402   |
| K₁     | 30.441               | 28.159                    | 29.881              |         |
| K₂     | 25.784               | 27.060                    | 26.673              |         |
| K₃     | 23.201               | 24.207                    | 23.172              |         |
| k₁     | 10.147               | 9.386                     | 9.960               |         |
| k₂     | 8.595                | 9.020                     | 8.891               |         |
| k₃     | 7.734                | 8.069                     | 7.724               |         |
| Range  | 2.413                | 1.317                     | 2.236               |         |

Figure 6. SEM image of Cu-coated SiC particles with the following ball milling parameters: (a) the powder to ball ratio was 1:20, milling time was 15 h, and milling speed was 300 r min⁻¹; (b) the powder to ball ratio was 15:1, milling time was 15 h, and milling speed was 300 r min⁻¹; (c) the powder to ball ratio was 20:1, milling time was 15 h, and milling speed was 250 r min⁻¹; (d) the powder to ball ratio was 20:1, milling time was five hours, and milling speed was 300 r min⁻¹.
from the point of preparing finer powders, the best parameters are, as follows: powder to ball ratio, 1:20; milling time, 15 h; milling speed, 300 r min\(^{-1}\). Figure 6 presents the SiC particles prepared using these ball milling parameters. It was apparent that although Cu/SiC particles with the smallest particle size could be obtained under these parameters, the adhesive effect was not as ideal as expected. Increasing the powder to ball ratio, milling time and milling speed would undoubtedly be beneficial for the preparation of fine SiC particles. However, the higher the parameters are set, the more violent the impact would be on the SiC particles and Cu powders. This would accelerate the work-hardening of Cu powders, and reduce the plasticity and toughness of the metal. As a result, the Cu powders would fall off from the SiC surface, and this would become more obvious as the milling time increases. Form the point of the adhesive effect, proper ball milling parameters should be selected, in order to prepare an ideal Cu/SiC particle. Sample 6 and 9 had a better Cu adhesive effect, when compared to the finest SiC particles.

Figure 7 shows the relationship among the three different powder to ball ratios, with a D\(_{50}\) parameter of 300 r min\(^{-1}\) and milling parameters of 15 h and powder to ball ratio of 1:20. (c) The D\(_{50}\) milling time curve, with the parameters of 300 r min\(^{-1}\) and powder to ball ratio of 1:20.

Figure 7. (a) The D\(_{50}\)-powder to ball ratio curve, with the parameters of 300 r min\(^{-1}\) and milling time for 15 h; (b) The D\(_{50}\)-milling speed curve, with the parameters of milling time for 15 h and powder to ball ratio of 1:20; (c) The D\(_{50}\) milling time curve, with the parameters of 300 r min\(^{-1}\) and powder to ball ratio of 1:20.
ball ratio had a significant effect on the refinement of the powders. Under the conditions of 15:1 and 20:1, the degree of refinement of these two was similar. However, the condition of 15:1 had a better adhesive effect, which fit the D_{50} powder to ball ratio curve well, indicating that the powder to ball ratio was not as big as possible.

Figure 7(b) presents the curve diagram for the relationship between D_{50} and milling speed, with the following parameters: milling time, 15 h; powder to ball ratio, 1:20. Figures 2(g), 6(c) and (a) presents the SEM images for the three milling speeds with the same powder to ball ratio and milling time. It was obvious that milling speed had a significant effect on the decline in particle size. That is, as the milling speed increased, the particle size sharply decreased, conforming to the law described in figure 8. When the milling speed was 200 r min^{-1}, the adhesive effect of Cu was remarkable, but most of the SiC surfaces had no Cu. As the milling speed reached 250 r min^{-1} and 300 r min^{-1}, the adhesive effect of Cu became worse, and the Cu powders were refined into smaller particles and dispersed in the mixed powder (figures 6(c) and (a)).

When the milling speed was increased, the milling balls received a greater resultant force in the radial direction of the milling container [19], which increased the impact power of the milling ball, and made the collision between milling balls more violent and frequent. As a consequence, the powders were quickly grounded into finer particles, and the particle size rapidly decreased. However, the increase in impact to the powder accelerated the work-hardening of metal powders, causing the plasticity and toughness of the Cu powder to decline. Due to this, the Cu powders that attached to the SiC surface fell off with the action of the impact, and refined into smaller particles. Hence, milling speed has a significant influence on the coating effect of the metal.

Figure 7(c) presents the relationship between particle size and ball milling time, with the following parameters: milling time, 300 r min^{-1}; powder to ball ratio, 1:20. It can be clearly observed in figure 7(c) that the particle size continued to decrease, and in the early stage of ball milling, the particle size rapidly decreased. However, in the later stage, the particle size slowly decreased. In the process of ball milling, the powders were affected by both fracture and cold welding. At the initial stage of ball milling, fracture was the main factor for the decline in size of the particles, and the change in particle size was obvious. As the milling time increased, the SiC particles and Cu powders were refined into finer particles. At this stage, the cold-welding effect became gradually obvious, and finally, the cold-welding effect and fracture effect tended to balance. Thus, it is hard to grind finer particles.

3.3. Properties comparison of raw SiC and Cu/SiC particles reinforced Fe-based matrix composites
Three kinds of SiC particles (raw SiC, sample 6 and sample 9) were used to prepare the corresponding Fe matrix composites, each composite had the same reinforcement content (10 wt%) and sintering parameters (sintering at 1000 °C for 2 h).

The hardness, impact strength data of the three kinds of SiC reinforced Fe matrix composites are listed in table 4. It can be observed that composites reinforced with Cu/SiC particles had better properties, and the properties of composites reinforced with sample 9 were the highest, with the hardness and toughness reaching

![Figure 8. SEM images and EDS line on the polished surfaces: (a) raw SiC reinforced, (b) sample 6 reinforced, (c) sample 9 reinforced.](image-url)
239.97 HV0.5 and 12.1 KJ·m⁻², respectively. Compared to raw SiC particles, the hardness and impact strength for sample 9 increased by 15% and 12.24%, respectively. These results suggest that due to the Cu attached to the SiC surface, the properties of the composite improved.

Figure 8 presents the SEM images of the polished surfaces of the three composites. Figure 8 presents the raw SiC-reinforced Fe matrix composites. It can be clearly observed from the figure that the bonding strength between raw SiC particles and the Fe matrix was weak, because multiple cracks could be observed at the interface. Due to the difference in crystal structure, the thermal expansion coefficient of SiC particles and the Fe matrix was very different. The SiC particles had a lower thermal expansion coefficient, which resulted in inconsistencies in the deformation of SiC particles and the Fe matrix as the temperature changed. Therefore, the mismatch of deformation degree resulted in a large amount of residual stress generated at the interface. When the effect of residual stress exceeded the plastic limit that the material could withstand, cracks occurred, especially at the sharp angles of the SiC particles, and the stress concentration accelerated the crack growth, as shown in figure 8(a). The Cu powders adhering to the surface of SiC balanced the difference between the SiC particle and Fe-based matrix in the thermal expansion coefficient, causing the residual stress at the interface to decrease. As a result, the generation of cracks decreased, as shown in figures 8(b) and (c).

Figure 9 presents the XRD patterns for sample 6 and 9, with reinforced Fe-based composites. This indicates that in sample 6, the peaks for SiC, the Fe matrix and Cu could be clearly observed in the patterns, while no obvious iron silicon and copper silicide peaks were observed. For sample 9, slight diffraction peaks of Cu₃Si could be detected. It is apparent that during the powder metallurgy processing, SiC was decomposed into Si and C, and element Si reacted with Cu to form Cu₃Si. Obviously, in sample 6 and sample 9, the SiC particles
chemically bonded with the Cu. Furthermore, regardless of whether it was sample 6 or 9, no iron silicide peaks were detected, showing that the Cu adhering to the surface acted as a barrier that suppressed the chemical reaction between SiC particles and the Fe matrix.

An EDS line scan was performed from the matrix to the SiC particles, in order to characterize the structure and element distribution, as shown in figure 8. Figure 10 presents the results for the elements distribution of Cu, Si, C, O and Fe. As shown in figure 10(a), a small amount of Si element could be detected in the reaction region and the matrix, suggesting that merely a very small part of SiC was dissolved, and that it was difficult to form silicide. This was also consistent with the XRD diagram, in which merely a small amount of Cu3Si could be detected, and that the metallic Cu on the surface of SiC inhibited the chemical reaction between the matrix and reinforcing particles. The element Cu was mainly distributed in the matrix and reaction region, and the content was almost the same with that in the two regions. The Cu in the matrix was mainly derived from Cu powders that did not attached to the surface of SiC particles, and this uniformly dispersed in the matrix metal under the action of the mixture. It can be observed that the strengthening effect of Cu on the composites was not only reflected in the improvement of interface bonding, but also in the strengthening of the matrix.

In addition, a small amount of O could be detected on the SiC side, indicating that the sample was slightly oxidized. Since the pressed sample was not completely dense, a small part of the air was substituted into the sample during the process of pressing the powder. The Gibbs free energy of the oxidation reaction was much lower than that of the reduction reaction, which resulted in the formation of a small amount of oxide on the SiC side.
lower than that of the forming silicide. Furthermore, it was very easy to oxidize the sample under high temperature conditions. Some unmatched peaks in figure 9 may have been caused by the sample oxidation.

Figure 11 presents the fracture morphologies of composites reinforced with the three kinds of SiC particles. The surface of the fracture surface is uneven, showing the characteristics of brittle fracture on the macroscopic level. As shown in figure 11(b), under high magnification, broken SiC particles and the absence of reinforcements could be observed in the dimples, and obvious cracks could be observed at the interface between the SiC particles and the Fe matrix. This suggests that the bonding strength between the SiC particles and Fe matrix was weak, the effect of the interface reaction and the difference in thermal expansion coefficient resulted in the formation of a bad interface region. The weakening of the interfacial bonding made the properties of the composite material decline. As shown in figures 11(c) and (e), there was no obvious change in the morphology of the fracture, The fracture surface is relatively flat and continued to show the characteristics of a brittle fracture. Under high magnification, as shown in figures 11(d) and (f), complete SiC particles could be found in the metal matrix. Furthermore, the bond between the matrix and reinforcement was fine, and no obvious particle shedding and cracks were observed. As described above, on the sides close to the SiC, the Cu powders and SiC particles formed a stable chemical bond through chemical reactions, while on the side close to the Fe matrix, the Cu and iron could be stably connected due to good affinity. Due to the action of the Cu, a sturdy interfacial bonding was formed at the interface region between the SiC particles and Fe matrix, and less particle shedding was found at the interface. In addition, the Cu powders adhered to the surface of SiC not only acted as a barrier to inhibit the direct contact between SiC particles and the Fe matrix to increase the bonding strength, but also alleviated the difference in thermal expansion coefficient between the matrix and reinforcement. Thus, this effectively reduced the stress concentration at the interface, slowed down the possibility of rapid crack growth, and improved the interface structure. However, from the property tests result, the properties of sample 6 and sample 9 were not improved much. The hardness and impact toughness of sample 6 were improved by 12.83% and 5.84% respectively, and the hardness and impact toughness of sample 9 were improved by 15% and 12.24% respectively. Although it can be observed that, in the figures 11(d) and (f), SiC is closely combined with the surrounding matrix, but the aggregation of SiC can also be observed, which may be the reason for the small improvement in material properties.

All these phenomena indicates that the matrix and the Cu/SiC particles form a chemical bond, and effectively inhibits the reaction between SiC particles and the Fe matrix, improving the interface bonding. Furthermore, Cu/SiC particles reinforced Fe-based metal matrix composites had better properties, comparing to raw SiC reinforced composite, the hardness and impact strength have been improved.

4. Conclusion

(1) The use of high energy ball milling induce Cu adhesion on the interface of SiC particles. Cu powders were constantly deformed, broken and refined under the impact of the grinding ball, and these adhered to the surface of SiC particles. The ball milling did not cause a chemical reaction between SiC and Cu, and these were physically bonded.

(2) After analyzing the results of the orthogonal optimization, the best ball milling parameters for preparing the finest Cu/SiC particles were obtained: powder to ball ratio, 1:20; mill speed, 300 r min⁻¹; milling time, 15 h. The SiC particles prepared under these conditions had a median diameter of 4.406 μm, but the adhesive effect was not good.

(3) Compared to raw SiC-reinforced Fe-based matrix composites, both the hardness and impact toughness of Cu/SiC-reinforced composites improved. Sample 9 had a better adhesive effect, and had better properties. The hardness increased to 239.97 HV0.5, which was 1.15 times of that for raw SiC particles. The impact strength increased to 12.1 KJ·m⁻².

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.
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References

[1] Zhang H C, Gao J F and Huang L Y 2010 Analysis and prevention of fracture cause of high chromium cast steel roller Hot Working Technology 39 183–4
[2] Choi J 2018 Strong potential of commercialized high Mn steels and process for various applications Metallurgia Italiana 7 69–77
[3] Girelli L, Pola A, La V and Gelfi M 2017 Performance optimization of high resistant white cast iron for severe working applications Metallurgia Italiana 6 5–10
[4] Ma S D and Zhang J J 2016 Wear resistant high boron cast alloy - a review Reviews On Advanced Materials Science 44 54–62
[5] Zheng Y, Zhou Y and Li S 2017 Preparation and mechanical properties of TiC-Fe cermet and TiC-Fe/Fe bilayer composites J. Mater. Eng. Perform. 26 4933–9
[6] Li J, Zong B Y, Wang Y M, Wang Y M and Zhuang W B 2010 Experiment and modeling of mechanical properties on iron matrix composites reinforced by different types of ceramic particles Mater. Sci. Eng. A 527 7545–51
[7] Wang F C, Du X D, Zhan M J, Lan J W, Zhou D, Liu G F and Shen J 2015 Microstructure and mechanical properties of Cr-SiC particles-reinforced Fe-based alloy coating J. Mater. Eng. Perform. 24 4673–80
[8] Shang F J, Wang W X and Zhou J 2022 Interaction mechanism and wear resistance of Ni-encapsulated Al2O3 particles reinforced iron matrix composites Rare Met. Mater. Eng. 51 422–8
[9] Wei D Q, Shi S Y and Li X K 2015 Preparation of Cu matrix composites coated and Mo-coated SiC particles. Mater. Today Proc. 18 6933–8
[10] Hu Z H, Ning K J and Lu K 2016 Study of spark plasma sintered nanostructured ferritic steel alloy with silicon carbide addition Mater. Sci. Eng. A 670 75–80
[11] Tang W M, Zheng Z X, Wu Y C and Jin Z H 2006 Interface Stability of the SiC Particles/Fe matrix composite System Journal of Wuhan University of Technology, Materials science edition 21 49–53
[12] Wang M T, Wang Z C and Zong Y P 2021 Sintering and mechanical properties of (SiC + TiCx)(p)/Fe composites synthesized from Ti3AlC2, SiC, and Fe powders Materials 14 9
[13] Hu Z H, Ning K J and Lu K 2016 Tribology characteristics of ex situ and in situ tungsten carbide particles reinforced iron matrix composites produced by spark plasma sintering J. Alloys Compd. 704 260–8
[14] Cui S L and Jung I H 2017 Critical reassessment of the Fe-Si system J. Eur. Ceram. Soc. 37 6135–41
[15] Cui S L and Jung I H 2017 Critical reassessment of the Fe–Si system Materials Science & Engineering. A, Structural materials: properties, microstructure and processing 689 225–41
[16] Zhang Y, Zong B Y and Cao X 2015 Electroleoclone copper plating on particulate reinforcements and effects on mechanical properties of SiCp/Fe composite Surf. Eng. 31 232–9
[17] Tatariko P, Casalegno V and Reece M J 2016 Joining of CVD-SiC coated and uncoated fibre reinforced ceramic matrix composites with pre-sintered Ti6Al4V MAX phase using spark plasma sintering J. Eur. Ceram. Soc. 36 3957–67
[18] Strojny-Nedza A, Egizabal P and Chmielewski M 2020 Corrosion and thermal shock resistance of metal (Cu, Al) matrix composites reinforced by SiC particles Materials Bulletin of The Polish Academy of Sciences-Technical Sciences 68 1227–36
[19] Fang W T F, Huang X and Huang H 2021 Microstructure inheritance of matrices of SiC/Ti6242 composites from precursor wire coatings by alpha plus beta phase field consolidation Rare Met. 40 3676–84
[20] Wei D Q, Shi S Y and Li X K 2015 Preparation of Cu/SiC mixed powder by high energy ball Milling. Special Casting & Nonferrous Alloys 35 565–8
[21] Ma H B, Lu Y, Lu H B and Meng X K 2017 Fabrication of Ni/SiC composite powder by mechanical alloying and its effects on properties of copper matrix composites Int. J. Mater. Res. 108 213–21
[22] Chang B, Zang J B, Wang Y H, Yu Y Q, Lu J and Xu X P 2018 Comparison study of Fe-based matrix composites reinforced with Ti-coated and Mo-coated SiC particles. Mater. Chem. Phys. 204 154–62
[23] Wang F C, Du X D, Zhan M J, Lan J W, Zhou D, Liu G F and Shen J 2015 Microstructure and mechanical properties of Cr-SiC particles-reinforced Fe-based alloy coating J. Mater. Eng. Perform. 24 4673–80
[24] Zhang Y B, Zong Y P, Cao X J and Zhang L. 2012 Effects of chemical Ni plating of reinforcing particles on properties of SiCp/Fe Composite Journal of Materials Research 26 483–8
[25] Wei D Q, Shi S Y and Li X K 2015 Preparation of Cu/SiC mixed powder by high energy ball Milling. Special Casting & Nonferrous Alloys 35 565–8