Preparation of Fe-Diopside Matrix Composites from Iron Tailings and Fly Ash

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Abstract. Iron-diopside matrix composites were prepared by using iron tailings, fly ash and iron powder as main raw materials. The samples with iron contents of 20%, 30%, 40% and 50% were investigated and compared with the basic ceramic which without iron addition. By using the methods of X-ray diffraction (XRD), scanning electron microscope (SEM), flexural strength test, the phase identification, microstructure and mechanical properties of samples were determined. The results show that the optimum addition amount of iron is 40%, under the sintering temperature 1200°C and holding time 90min, the main phase of the samples is diopside, iron and a small amount of quartz. The morphology of diopside is found to be columnar crystals. The sample prepared under these conditions has the maximum flexural strength of 140MPa, which is 105.9% higher than that of the basic ceramic, with a density of 3.6g/cm³. A new method of preparing composites from solid wastes has been developed.

1. Introduction

With the rapid development of the global economy, industrial solid wastes have increased dramatically, such as blast furnace slag [1], steel slag [2], iron tailings [3], fly ash[4], red mud [5], etc, which not only occupied a lot of land, but also caused pollution to the environment. Therefore, the effective utilization of solid wastes has attracted the attention of many countries. In China, the amount of metal tailings deposited is nearly 5 billion tons at present, and the annual discharge is up to 500 milling tons, among which, the iron tailings account for 30% of the total. The comprehensive utilization ratio of the iron tailings is only about 25%, which will inevitably result in a large accumulation of iron tailing, occupying and destroying more natural resources [6-8]. Many researchers have made in-depth study on the comprehensive utilization of iron tailings. These are not only to solve the practical problems of utilization of iron tailings, but also to provide a sustainable path for development of human beings.

The researchers studied the preparation of ceramic materials by using iron tailings because they are mainly rich in SiO₂, Al₂O₃, CaO, Fe₂O₃. Li et al prepared glass-ceramic with iron tailing and fly ash. The main crystalline phase is pyroxene [9].Iron tailings can also be used as raw materials to prepare ceramic tiles, and the mechanical properties of the sample are generally greater than the standard [10].

Fly ash is the solid waste discharged from coal-fired power plant. Nowadays, the total amount of fly ash has exceeded 1billion tons. What’s more, it is increasing at a rate of 100 million tons per year in China. The large amount of fly ash occupies cultivated land, which pollutes water resources,
atmospheric environment and even harms human health. Therefore, it is of great significance to realize the comprehensive utilization of fly ash [11-14].

The composition of fly ash is generally, SiO$_2$ 33-59%, Al$_2$O$_3$ 16-35%, Fe$_2$O$_3$ 1.5-19%, CaO 0.8-10%, MgO 0.7-1.9% [15]. On account of its composition characteristics, the utilization of fly ash can be divided into three types. The first is the preparation of building materials such as cement and concrete. M.J. McCarthy et.al describes a study to investigate the use of high volume fly ash as a cement component in concrete, the results show that high fly ash levels can be used in combination with Portland cement to produce concrete covering the range of design strengths typically required in practice [16]. The second is the recovery of valuable elements from fly ash, Gao et al. extracted silica from fly ash via organic acid/inorganic alkali/ultrasound-assisted joint process. The results show that the final yield and purity of SiO$_2$ were 51% and 98.65%, respectively [17]. And the third application of the fly ash is the preparation of ceramic materials. Peng et al. investigated the glass-ceramics by using melt high alumina fly ash with fluxing additives. Nano-crystal glass-ceramics with anorthite (CaAl$_2$Si$_2$O$_8$) and wollastonite (CaSiO$_3$) as the main crystal phase are obtained through specific thermal treatment. The physical and mechanical properties of the glass-ceramics, such as density, thermal expansion coefficient, hardness and flexural strength were studied, the results show that the chemical composition of fly ash was quite suitable as the raw materials for structural glass-ceramics, and the maximum value of flexural strength was 103MPa [18].

The ceramic-metal composites are mainly made up of both ceramic phase and metal phase, in which the higher hardness and excellent wear resistance are given by harder ceramic phase, besides the plasticity and tenacity are provided by the metal phase. Compared to single phase ceramic materials, metal-reinforced ceramics exhibit attractive mechanical properties, especially improved fracture toughness [19]. Therefore, the ceramic-metal composites is usually a very crucial material.

Recently, the research of ceramic-metal composites materials is mainly focused on the carbide such as TiC, WC [20, 21] and the oxide matrix, for instance, Al$_2$O$_3$ [22], etc. Although the performance is excellent, the preparation of composites is usually made of chemical pure raw material, the preparation process is hot pressing or spark plasma sintering, which will inevitably lead to the complexity of the preparation process and the high cost of manufacturing, which limits the wide application. The traditional method for preparing ceramic composite are mostly made of fine chemical powder, which has the disadvantages of high energy consumption and high cost. In recent years, many researches have been conducted on the preparation of ceramic materials from natural minerals, exploring a new way to prepare high performance composites at low cost [23]. Many researchers synthesis composites by using the natural ilmenite which was reduced by the carbon, aluminum, magnesium, calcium and other reduction agents to form hard phase TiC, TiN and metal phase Fe [24, 25]. It will not only reduce the cost but also obtain better properties that synthesizing ceramic composites by reaction sintering from minerals.

It is generally known that solid wastes such as iron tailings and fly ash can be used as raw materials for preparing ceramics or glass-ceramics, while the preparation of composites from solid waste has not been reported. In an effect to expand the way of high-value utilization of these solid wastes, iron tailings and fly ash were used as main raw materials in this paper, and iron powder was added as metal phase to prepare cheap composites. The aim is to develop a king of cheap composites that can be widely used in mining or metallurgical industries. The materials were characterized by XRD, SEM and flexural strength test, and the relationship between the microstructure and the macroscopic properties of the samples was discussed.

2. Materials and methods

2.1. Materials
The tailings of Bayan Obo separation in Inner Mongolia (provided by the team studying mineral processing, Inner Mongolia University of Science and Technology, Baotou, China) and the fly ash(Provided by a power plant in baotou, Inner Mongolia, Baotou, China) of a power plant are used as
the main raw materials. The chemical compositions of the raw materials are shown in Table 1. It has been reported that diopside crystal phase can significantly improve the flexural strength of the sample. In order to improve the mechanical properties of the sample, pure MgO was added in the experiment to produce more diopside phases [26]. According to the chemical composition and characteristics of the raw materials, combined with the phase diagram of the CaO-Al₂O₃-SiO₂ ternary system and the preliminary experiment, the specific formula and compositions of the experiment are shown in Tables 2 and 3.

Table 1. The main chemical compositions of raw materials.

| Name          | SiO₂ | CaO  | Al₂O₃ | MgO  | Na₂O | K₂O | TFe | REO | CaF₂ |
|---------------|------|------|-------|------|------|-----|-----|-----|------|
| Iron tailing  | 18.17| 13.26| 1.69  | 2.46 | 2    | 0.5 | 20.11| 5.13| 4.2  |
| Fly ash       | 56.34| 9.5  | 23.22 | 3.15 | 1.1  | 3.15| 7.02 | —   | 0.14 |
| Quartz        | 97.88| 0.04 | 1.48  | 0.06 | —    | —   | 0.38 | 0.15| —    |

Table 2. The compositions of composites formulation.

| No. | Tailing | Fly ash | Quartz | CaO  | MgO  | Iron |
|-----|---------|---------|--------|------|------|------|
| C0  | 40      | 30      | 20     | 5    | 5    | 0    |
| C1  | 32      | 24      | 16     | 4    | 4    | 20   |
| C2  | 28      | 21      | 14     | 3.5  | 3.5  | 30   |
| C3  | 24      | 18      | 12     | 3    | 3    | 40   |
| C4  | 20      | 15      | 10     | 2.5  | 2.5  | 50   |

Table 3. The chemical compositions of composites formulation.

| No. | SiO₂  | CaO  | Al₂O₃ | MgO  | Na₂O | K₂O | TFe | REO | Fe  |
|-----|-------|------|-------|------|------|-----|-----|-----|-----|
| C0  | 41.00 | 16.07| 7.64  | 6.88 | 0.85 | 1.15| 10.33| 2.05| 0   |
| C1  | 35.18 | 10.48| 6.11  | 5.50 | 0.68 | 0.92| 8.26 | 1.64| 20  |
| C2  | 30.78 | 9.17 | 5.35  | 4.82 | 0.59 | 0.80| 7.23 | 1.44| 30  |
| C3  | 26.38 | 7.86 | 4.59  | 4.13 | 0.51 | 0.69| 6.20 | 1.23| 40  |
| C4  | 21.99 | 6.55 | 3.82  | 3.44 | 0.43 | 0.58| 5.17 | 1.03| 50  |

2.2. Methods

100g of a batch was prepared according to Table 2. The powder was mixed for 4h in a ball milling at 300 rpm, the ball milling medium was anhydrous ethanol, the ball to powder ratio (BPR) was 4:1, slurry was obtained and then dried for 24 under 95℃. Samples of φ40mm×5mm were hydraulically compacted by using uniaxial pressing at 30MPa. The shaped samples were fired at different temperatures in a sintering furnace under argon atmosphere, the gas flow was 1L/min. The heating rate was 5℃ /min under different holding time. After sintering, the samples were removed with the furnace cooling to room temperature.

The crystalline phases were determined by X-ray diffraction(XRD, X’pert Pro Powder, PANalytical), the Cu target as X-ray tube was operated at 40KV and 40mA, and the 2θ scan range was from 10°to 80°, with a step size of 0.02° and a scan speed of 0.3s/step. The microscopic structures were investigated by scanning electron microscope(SEM, SUPRA 55 FESEM, Carl Zeiss Jena). Before SEM test, the samples were etched with HF 5% for 90s at room temperature, and then coated with gold to be analyzed.

The densities were measured by Archimedes method. Each value is the mean value of measurements made with five samples. Linear shrinkage(LS) was determined the length difference between the green(L₁) and the fired sample(L₂), and then calculated by using Eq.(1):

\[ LS(\%) = 100 \times \frac{(L_1 - L_2)}{L_1} \]  

(1)
The three-point flexural strength (FS) of rectified parallelepiped bars (3mm × 4mm × 40mm) of samples were tested by the CSS-88000 electronic universal testing machine, and then calculated using Eq. (2):

\[ FS (\text{MPa}) = 3 \times \frac{F}{I (2 \times b \times h^2)} \]  

Where F is the breaking load (N); \( l \) is the span between the support rods (mm); b is the width of the test sample (mm); h is the minimum thickness of the test sample measured after the test along the broken edge (mm).

3. Results and discussion

3.1. Physical and mechanical properties

The variation curves of the density and flexural strength of the samples with different iron contents are shown in Figure 1. The density of samples with iron content of 20%, 30%, 40%, and 50% are 3.19 g/cm³, 3.36 g/cm³, 3.52 g/cm³, and 3.68 g/cm³, respectively. The density increases along with the increasing of iron content. The reason for this phenomenon is obvious, since the density of iron is 7.86 g/cm³, which is greater than the density of ceramic phase. Therefore, the addition of iron content will result in the increasing of the density of samples.

![Figure 1. Density and flexural strength of composites with different iron content](image)

The variation of the flexural strength is different from that of the density, which is first decreased and then increased. It can be seen in the figure, when the content of iron is 20%, the flexural strength of the sample is only 35 MPa, which is lower than the 68 MPa of the basic ceramic. When the content of iron reaches 30%, the flexural strength increases to 106 MPa. When the content of iron increases to 40% and 50%, the reinforcing effect of metal phase is more obvious, the flexural strength reaches 140 MPa and 152 MPa, respectively. Compared with the ceramic sample without iron addition, the flexural strength increases by 105.9% and 123.5%, respectively. The reason why the flexural strength of samples decreased first is that the metal phase with lower content is added and dispersed in the ceramic phase, which does not enhance the strength of the samples. And then is gradually increasing...
with the increase of the content of iron, mainly due to two factors, firstly, the flexural strength of the sintered iron is about 300MPa generally. Secondly, the increase of the metal phase causes part of metal connected in the ceramic, which has the effect for enhancing the strength of the materials. The flexural strength of samples is increased with the increasing of the iron content. In order to ensure the performance of materials and the utilization ratio of the wastes, thus the optimum addition of iron is 40%.

The variation curves of the linear shrinkage and flexural strength of C3 sample at different sintering temperatures are shown in Figure 2. With the increasing of sintering temperature, the linear shrinkage and flexural strength are increased. When the sintering temperature is 1180°C, the linear shrinkage of the sample is only about -4.2%, which means that the sample does not start densification, and the flexural strength is only 67MPa. When the sintering temperature is increased to 1190°C, the sample is substantially densified, its linear shrinkage reaches -8.5%, and the flexural strength increases to 118MPa. When the sintering temperature is up to 1200°C, the sample has been completed densification, and the linear shrinkage and flexural strength are -10.2% and 140MPa, respectively. As the sintering temperature increases to 1210°C, the sample expands and the strength decreases, indicating that the sample has been overheated. Therefore, the optimum sintering temperature is 1200°C.

![Figure 2. Linear shrinkage and flexural strength of sample C3 at different sintering temperatures](image)

Figure 3 shows the curve of flexural strength of C3 sample at different holding times. With the increasing of the holding time, the flexural strength of the sample first increased and the then decreased. The highest flexural strength of the basic ceramic C0 is 68MPa, which is at the sintering temperature of 1200°C and the holding time of 30min. However, the C3 sample with 40% iron added has a flexural strength of only 64MPa at holding time of 30min. When the holding time is increased to 60min, the flexural strength is abrupt increased to 131MPa. The flexural strength reaches 140MPa at the holding time of 90min, while decreasing significantly at 120min. It indicates that the addition of the metal phase hinders the densification of the ceramic phase, which means that it is necessary to extend the holding time to obtain more excellent sample [27, 28]. However, it will cause the abnormal growth of the ceramic grain and reduces the mechanical properties of the composites hat the holding time is too long. In conclusion, the best holding time is 90min.
3.2. Phase identification

In order to determine the phase composition of the samples, the XRD analysis of each sample was carried out. The results of the analysis are shown in Figure 4. The main crystalline phase of the C0 sample is diopside and quartz. In the C1 to C4 samples, the main crystalline phases are diopside and iron. The reason for this is that the SiO$_2$, CaO, MgO and Al$_2$O$_3$ in the sample will form pyroxene crystals at high temperature, since the silicon-oxy tetrahedron of the pyroxene crystals has a chain structure, which makes it possible to accommodate different cations [29]. The iron ions contained in the raw materials are similar in radius to calcium and magnesium ions. Therefore, after the iron ions enter the pyroxene crystal, the solid solution of calcium, magnesium, aluminum and iron ions is formed, and finally the diopside phase is formed [30]. The diffraction peak of iron appears in all samples, and the intensity gradually increased with the increasing of iron content.

It can be seen clearly from the enlargement that the intensity of the diffraction peaks in the quartz phase is significantly reduced due to the addition of iron. However, the intensity of the diopside diffraction peak on the right is not reduced by the decrease of the ceramic composition but remains essentially unchanged. It shows that the addition of iron promotes the unreacted quartz to combine with calcium ions, magnesium ions and iron ions to form diopside phase. The reason for this phenomenon may be due to the addition of iron powder containing a certain amount of iron oxide, and these iron oxide mainly have two functions, the first is that the increasing of iron oxide content in the sample results in the decrease of the melting temperature of Fe$_2$O$_3$+ CaO+MgO+SiO$_2$. It leads to more liquid phase is formed by combining unreacted SiO2 during the sintering process. And the liquid phase will form glass after condensation process. The second function of the iron oxide is that it can be used as nucleating agent, so that glass is crystallized during the cooling process, and result in more diopside phase is formed, thus increasing the crystallization ratio of the sample.

Figure 5 shows the EDS analysis of the C3 sample, and Figure 5(a) is divided into three areas, white, gray and black. Iron is enriched in the white area and there is no other element, indicating that the region is iron phase, which is consistent with the previous analysis. The gray region is rich in O, Si, Ca, Mg and Al, indicating that these regions are rich in diopside crystals. The black region contains higher O and Si elements, showing that SiO$_2$ is enriched in this region, which is consistent with the precious XRD analysis results. The sample mainly contains iron, diopside and quartz.
3.3. SEM analysis of composites
In order to reveal the dispersion states of the iron phase in the samples, the back-scattering image of samples with different content of iron is shown in Figure 6, which is divided into two colors, white and black. The white is the iron phase and the black is the diopside phase and the glass phase. The sample with 20% Fe content is shown in Figure 6(a). It can be seen that the iron is disperse in the ceramic phase. The metal phase does not play a role in reinforcing the ceramic phase from the above analysis of mechanical properties, but rather can be seen as an impurity phase existing in the ceramic phase, which decreases the mechanical properties of the material. The sample with 30% Fe content is shown in Figure 6(b), although the iron is not connected in large areas, it has aggregated on a small scale, which can play a role in hindering the expansion of cracks and improve the mechanical properties of materials. Figure 6(c) shows a sample with 40% Fe content, the iron is uniformly distributed in the ceramic matrix as well as basically connected. In the process of crack development, metal phase will inevitably encounter the deflection of the crack, meanwhile, the energy is required to increase, which results in the mechanical properties of the sample is improved. Figure 6(d) shows the sample with 50% Fe content, iron exists in the ceramic matrix in a substantially connected, which can improve the strength of the sample.
Figure 6. Back scattering images of composites with different content of iron (a):20% of Fe, (b):30% of Fe, (c):40% of Fe, (d)50% of Fe

Figure 7. SEM images of composites in different content of Fe: (a) 0% of Fe, 1200°C, 30 min; (b) 20% of Fe, 1200°C, 90min; (c) 30% of Fe, 1200°C, 90min; (d) 40% of Fe, 1200°C, 90min; (e) 50% of Fe, 1200°C, 90min

The SEM images of composites in different content of Fe are shown in Figure 7, the iron contents of (a) to (e) are 0%, 20%, 30%, 40% and 50%, respectively. In Figure 7(a), the crystal consists mainly of granular crystals and columnar crystals, the crystal length of columnar crystals is approximately $14 \sim 16\mu m$. These two crystals are intertwined to ensure the macroscopic mechanical properties of the sample. However, due to the high content of granular crystals and the large aspect ratio of the columnar crystals, the basic ceramic sample without iron has a flexural strength of only 68MPa. While with the addition of iron content, the number of granular crystals and the aspect ratio of columnar crystals is significantly decreased. Meanwhile, the crystals interweave and grow together, significantly improving the mechanical properties of the sample. This is mainly due to two reasons, firstly, it is well known that long holding time is advantageous to the crystal development. Therefore, the holding time of the sample is 90min, which can promote the granular crystals in the basic ceramics to develop into columnar crystals. Secondly, the addition of the second phase of metal can inhibit the crystal
development and hinder the abnormal growth of the crystal. The second phase particles generally inhibit the grain boundary migration of the matrix. However, the matrix grain boundary can pass through the dispersed particles and be absorbed into the crystal grain for the fine dispersion particles. The larger particles remain at grain boundaries and cannot be absorbed by matrix grains [31]. So, the grain length in the sample decreases obviously, and there is no grain with larger aspect ratio.

The sample with 40% iron has a 105.9% increase in flexural strength compared to the basic ceramic sample. There are two main reasons for this, firstly, the addition of iron changes the shape of the crystal, transforms from granular to columnar crystals, and grows together. Secondly, the added metal phase exists in the ceramic matrix as a reinforcing phase, hinders the expansion of cracks, and improves the mechanical properties of the sample.

4. Conclusions
In this paper, a novel Fe-diopside based composites were successfully prepared with iron tailings and fly ash as main raw materials. It not only provides a new idea to solve the practical problem of solid waste, but also produces a cheap composites material. The optimum iron content of the sample is 40%. The preparation process is: sintering temperature 1200°C, holding time 90min. The density of sample is 3.6g/cm³ and the flexural strength is 140MPa. Compared with the basic ceramic sample without iron, the flexural strength is increased by 105.9% and the properties are improved significantly.

The main phase of the samples is diopside phase, iron and a small amount of quartz phase. It can be seen that more diopside phase is formed because of the addition of iron to promote the unreacted quartz to participate in the crystal phase reaction by XRD analysis. The results of SEM show that the iron exists in the ceramic phase in the form of connectedness, and the interface between the metal and the ceramic phase is relatively close. The ceramic samples without iron are composed of granular and columnar crystals, and the content of granular crystals is higher. However, after the addition of iron, the crystal development is promoted. The sample are mainly columnar and interlaced and grown together, which is beneficial to the improvement of the mechanical properties of the samples. Moreover, the metal phase can hinder the crack expansion. Therefore, the mechanical properties of the samples can be significantly improved.

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