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Effects of sintering process on preparing iron-based friction material directly from vanadium-bearing titanomagnetite concentrates

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ABSTRACT

In order to optimize an innovative two-stage process for preparing an iron-based friction material directly from vanadium-bearing titanomagnetite concentrates, this paper focuses on the effects of sintering process on the microstructures and properties of an iron-based friction material. On one hand, the samples were sintered at 900 °C, 950 °C, and 1000 °C for 3 h respectively; On the other hand, the samples were sintered at 1000 °C for 1 h, 2 h, 3 h, and 4 h respectively. As a result, after the samples were sintered at above 950 °C for more than 3 h, a lot of laminated microstructures appear in these samples owing to the formation of a large number of pearlites. Besides, the density, the hardness, and the friction coefficient of this material are positively correlated to the sintering temperature or the sintering time, and the wear rate of this material is negatively related to the sintering temperature or the sintering time. This study can contribute to the attainment of much clearer insight into the effects of sintering process and lay the foundation of practical application of this innovative two-stage process.

1. Introduction

As an important metal matrix composite, iron-based friction material has been applied widely in clutch or brake mechanisms of aeroplanes, tanks, trucks, ships, tractors, engineering machinery and metal-cutting machines. Conventionally, based on pure substance powders, iron-based friction material is produced by means of powder metallurgy. Generally, it is costly and time-consuming to prepare various pure substance powders.

At present, some ferro-matrix composites can be prepared directly from natural minerals by means of in-situ synthesis technology, which have caught great interests of researchers because of great process simplification and cost decrease. Welham et al. discussed the carbothermic reduction of ilmenite and rutile in detail [1], and made successful attempts to produce TiN/TiC–Fe composites cheaply from ilmenite concentrate [2]. Jayasankar et al. succeeded in synthesizing Fe–TiC composites from cheap raw materials such as mild steel scrap, ilmenite, and petroleum coke [3]. Razieh Khoshhal et al. used cheap ilmenite to synthesize Fe–TiC/Al_2O_3 composite [4]. Based on ilmenite, carbon black, and aluminum powder, Mansour Razavi et al. prepared Fe–TiC–Al_2O_3 hybrid nano-composite via carbothermic reduction caused by mechanical activation [5].

However, up until now, seldom documents are concerned with the material preparation from the abundant vanadium-bearing titanomagnetite by means of in-situ synthesis technology. As a special and complex iron ore, the vanadium-bearing titanomagnetite mainly consists of ferrous oxides, titanium oxides, vanadium oxides and other oxides such as Al_2O_3, SiO_2, CaO, and MgO [6]. After the beneficiation process, vanadium-bearing titanomagnetite concentrates are produced with a high content of total iron. At present, these concentrates are mainly applied in the ironmaking process. During this process, most of the iron and part of the vanadium can enter the hot metal, but almost all of the titanium remains in the slag to form the high titanium slag. Hitherto, no an appropriate and economical method has been found to utilize this kind of slag [7]. At the same time, these oxides such as Al_2O_3, SiO_2, CaO, and MgO in the vanadium-bearing titanomagnetite concentrates are regarded as impurities and must be removed during this ironmaking process. Consequently, this application in the ironmaking process gives rise to an enormous waste of precious elements in the vanadium-bearing titanomagnetite concentrates.

On the other hand, theoretically, based on selective in-situ carbothermic reactions of the vanadium-bearing titanomagnetite concentrates in a specific condition, the ferrous oxides can be reduced to metal iron, the titanium oxides can be converted into TiC and the vanadium oxides can be converted into VC, yet Al_2O_3, SiO_2, CaO, and MgO cannot be reduced by carbon because of their chemical stability. As a matter of fact, TiC, VC, Al_2O_3, SiO_2, CaO, and MgO are useful hard particles in iron-based friction material. Consequently, it is possible to find an alternative method to prepare an iron-based friction material by means of selective in-situ carbothermic reactions of the vanadium-bearing titanomagnetite concentrates.

Based on the above backgrounds, Guangming Zhang and Keqin Feng have recently invented a two-stage process, consisting of a selective

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pre-reduction stage and a final sintering stage, to prepare iron-based friction material directly from the vanadium-bearing titanomagnetite concentrates [8]. In general, as a key step of powder metallurgy, sintering process plays an important role in the quality of final product. Both the sintering temperature and the sintering time have great effects on the microstructures and the properties of the material. If the sintering temperature and the sintering time are not proper, the properties of the material cannot be guaranteed. Besides, an appropriate sintering temperature and sintering time can contribute to the attainment of economical sintering process since they can help to reduce the energy consumption of sintering process. Thus, this paper focuses on the effects of the sintering temperature and the sintering time on the microstructures and properties of an iron-based friction material with the main chemical compositions of 74 wt.% Fe, 8 wt.% Cu, and 5 wt.% C. The corresponding results are helpful to optimize the sintering process and lay the foundation of the practical application of this two-stage process.

2. Materials and methods

The powders of the vanadium-bearing titanomagnetite concentrates were supplied by Panzhihua Steel Group Corporation in China, and the main chemical compositions are listed in Table 1. High purity graphite powders were used as a reductant and a lubricant.

In the pre-reduction stage, 83 wt.% vanadium-bearing titanomagnetite concentrates and 17 wt.% graphite powders were mixed in planetary ball mill at 400 rpm for 3 h. Then these mixed powders were placed in a vacuum sintering furnace to be heated to 1300 °C. For the need of carbothermic reactions, the time of heat preservation was 3 h. These pre-reduced powders contain 74.42 wt.% MFe, 10.32 wt.% TiC, 5.90 wt.% Al_2O_3, 4.71 wt.% SiO_2, 1.23 wt.% CaO, 1.25 wt.% MgO, and 0.53 wt.% VC. According to the experimental mass loss, it can be calculated that the percentage of ferrous oxides reduced to metal iron is about 96%, the percentage of $FeTiO_3$ converted into TiC is about 75% and the percentage of V_2O_5 converted into VC is about 94% during the pre-reduction stage. The corresponding selective in-situ carbothermic reactions of the vanadium-bearing titanomagnetite concentrates have been detailed in another paper [8].

In the sintering stage, according to the main chemical compositions of the pre-reduced powders and the main chemical compositions of 74 wt.% Fe, 8 wt.% Cu, and 5 wt.% C in typical iron-based friction material, supplementary iron powders, copper powders, and graphite powders were added into the pre-reduced powders to be ground and mixed. After that, the mixed powders were placed in steel mold and pressed under 400 MPa by oil hydraulic press machine to obtain the samples of Φ13.26 mm × 9 mm. These samples were divided into two groups. One group was used to study the effects of the sintering temperature, in which the samples were sintered at 900 °C, 950 °C, and 1000 °C for 3 h respectively. The other group was used to study the effects of the sintering time, in which the samples were sintered at 1000 °C for 1 h, 2 h, 3 h, and 4 h respectively.

The microstructure of the iron-based friction material was investigated by SEM. The density was tested via Archimedes drainage method and the Brinell hardness was tested by means of HBE-3000A. In addition, the friction-wear test between the iron-based friction material and GCr15 was carried out by using M-2000A friction and wear test machine according to the specification of GB/T 12444–2006.

Table 1
Main chemical compositions of the vanadium-bearing titanomagnetite concentrates (wt.%).

TFe	FeO	TiO_2	V_2O_5	Al_2O_3	SiO_2	CaO	MgO
53.17	30.02	12.65	0.56	4.87	3.89	1.02	1.03

3. Results and discussion

3.1. Effects of the sintering temperature on the microstructures and properties of the iron-based friction material

Based on SEM test, the characterization of the laminated pearlites in the etched final samples sintered at 900 °C, 950 °C, and 1000 °C respectively is demonstrated in Fig. 1 after the final samples were etched by using 2% nitric acid solution in alcohol.

The characterization of the laminated pearlites in the etched iron-based friction material, shown in Fig. 1, demonstrates that the higher sintering temperature can facilitate the formation of the laminated pearlites. After the samples were sintered at 900 °C, a small amount of laminated pearlites appear in the materials. Meanwhile, after the samples were sintered at 950 °C and 1000 °C respectively, there are more and more laminated pearlites in the materials. Theoretically, according to the Fe–C phase diagram, with the increase of the sintering temperature, more and more phase changes from α-Fe to γ-Fe take place and the homogenization of carbon atoms in γ-Fe can be enhanced during the sintering process. Consequently, by means of the decomposition of austenite during the subsequent cooling process, the formation of laminated pearlites can be reinforced.

The effects of the sintering temperature on the density and the Brinell hardness of the iron-based friction material are shown in Fig. 2. In addition, the effects of the sintering temperature on the friction coefficient and the wear rate of the iron-based friction material are illustrated in Fig. 3.

It can be seen from Figs. 2 and 3 that the higher the sintering temperature is, the larger the density, the Brinell hardness and the friction coefficient are, correspondingly, the lower the wear rate is. Comparatively, the hardness rises very quickly when the sintering temperature varies from 950 °C to 1000 °C, the wear rate declines very slowly when the sintering temperature varies from 950 °C to 1000 °C and the friction coefficient increases very rapidly when the sintering temperature changes from 900 °C to 950 °C.

Generally, there is a positive correlation between the sintering temperature and the density of the sintered material [9]. The higher sintering temperature can contribute to the densification of the sintered material by means of facilitating the formation of sintering necks, the growth of sintering necks and the movement of grain boundaries. The hardness of the iron-based friction material is related to its density and the amounts of various phases in the material. Since the higher sintering temperature can make the material density larger and can also facilitate the formation of the pearlites in the iron-based friction material, it is helpful to raise the hardness of the iron-based friction material. In general, there is a negative correlation between the wear rate of material and the hardness of material. Thus, the higher the sintering temperature is, the larger the hardness of the iron-based friction material is and the lower the wear rate is.

Based on the previous discussions about the effects of the sintering temperature on the microstructures of the materials, at lower sintering temperature, more free graphite particles can be left to act as lubricating phases in the iron-based friction material. Thus, the lower the sintering temperature is, the lower the friction coefficient is.

3.2. Effects of the sintering time on the microstructures and properties of the iron-based friction material

By means of SEM test, the characterization of the laminated pearlites in the etched final samples sintered for 1 h, 2 h, 3 h, and 4 h respectively is illustrated in Fig. 4 after the final samples were etched by using 2% nitric acid solution in alcohol.

The characterization of the laminated pearlites in the etched materials sintered for different hours, shown in Fig. 4, demonstrates that the longer sintering time can also facilitate the formation of the laminated pearlites. Generally, the longer sintering time can facilitate the phase