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Effect of cooling rate on structural and electromagnetic properties of high-carbon ferrochrome powders

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ABSTRACT

The structural and electromagnetic properties of high-carbon ferrochrome powders (HCFCP) obtained at different cooling rates were respectively investigated by means of optical microscope, X-ray diffractometer, electron probe as well as the vector network analyzer in the frequency range of 1–18 GHz. The results show that the cell structure of main phase, $(Cr,Fe)_7C_3$, transforms from hexagonal to orthogonal with the improvement of cooling rate. Meanwhile the mass ratio of Cr to Fe in $(Cr,Fe)_7C_3$ gradually declines, while that for CrFe goes up. Both the real part and the imaginary part of relative complex permittivity of HCFCP are in an increasing order with cooling rate rising in most frequencies. For comparison, the relative complex permeability presents an opposite changing tendency. The peaks of the imaginary part of relative complex permeability appearing in low and high frequencies are attributed to nature resonance. The reflection loss of HCFCP gradually decreases as cooling rate reduces and frequency enhances. At 2.45 GHz, the algebraic sum of dielectric loss factor and magnetic loss factor increases first and then decreases in the temperature extent from 298 K to 1273 K.

1. Introduction

Owing to the extensive use of stainless steel and high strength tool steel in various areas, high-carbon ferrochrome (HCFC) as a sort of metallurgical feedstock material has attracted a considerable attention from industries, which also has propelled the further development of metallurgical industry [1–4]. Yet the conventional liquid-phase decarburization processing routes of HCFC such as oxygen blowing and electro-silicothermic methods, have always faced a severe problem that the generation of large amounts of toxic chromium slag does harm to the ecological environment and human health [5–6]. In recent years, solid-phase decarburization process of HCFC without the production of chromium slag has been gradually introduced, and the degree of decarburization depends on its phase structure. Navara et al. [7] have pointed out HCFC involves the following phases, the primary phase $(Cr,Fe)_7C_3$, peritectic phase $(Cr,Fe)_{23}C_6$ and eutectic phase $(Cr,Fe)_7C_3$–CrFe, offering a promising guidance for solid-phase decarburization. Unfortunately, the popularity reveals a slow program on account of low heating efficiency of the traditional heating method [8]. Microwave, is a promising heating technology as seen from both the metallurgical and material points of view, showing simultaneous ordering of non-contact, pollution free and fast distribution of thermal energy within the object of interest, and has been utilized into a series of explorations and experiments, opening up immense feasible routes [9–12]. In consideration of the high reflectivity of bulk metallic materials for microwave because of so-called skin-effect, HCFCP is generally regarded as the raw material for solid-phase decarburization in microwave filed [13–14]. Hao et al. [14] applied microwave heating technology to solid-phase decarburization test of HCFCP, exhibiting an acceptable decarburization rate, the value of 82.96%, when heated to 1373 K then thermal insulation 60 min.

As most clearly pronounced by researchers, the microwave-absorption performance and temperature-rising characteristic of substances in microwave field significantly rely on their electromagnetic performances mainly involving dielectric and magnetic properties, which are generally represented by the relative complex permittivity ($\varepsilon_r = \varepsilon_r' - j \cdot \varepsilon_r''$) and relative complex permeability ($\mu_r = \mu_r' - j \cdot \mu_r''$). The real part of relative complex permittivity (permeability), ε_r' (μ_r'), shows the stored electric (magnetic) energy via polarization mechanisms (the alignment of magnetic dipoles along the direction of the magnetic field) and the imaginary part, ε_r'' (μ_r''), represents the loss of electric (magnetic) energy via dielectric relaxation mechanisms (natural resonance, magnetic hysteresis and eddy current loss) [15–16]. In addition, the believable explanations for the mechanism of microwave heating have been given in some previous literatures [13,16–18]. Li [19] focused

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on electromagnetic properties of HCFCP decarburized by microwave heating, and pointed out electromagnetic properties would vary along with changing in carbon content of HCFCP. In terms of phase composition of HCFC [7], more interest of academics for electromagnetic properties of HCFCP has been drawn in main phases, $(Cr,Fe)_7C_3$ and CrFe. Chen [20] discussed the intrinsic nature of Fe-doped o-Cr_7C_3, namely $(Cr,Fe)_7C_3$, basing on density functional theory (DFT) and indicated the importance of structural performance of $(Cr,Fe)_7C_3$ on microwave dielectric properties of HCFCP; With the same method, Konyaeva et al. [21] investigated the structural and magnetic properties of $(Cr,Fe)_7C_3$; As for CrFe, Froideva [22] studied the magnetic and structural properties of CrFe using imaging magnetic microspectroscopy and ab initio method.

Due to the distinct discrepancy on cooling rate for different sections of liquated HCFC in industrial large-scale cooling process, the type, structure and electromagnetic performance of solid-phases present relatively complex diversities. In previous studies, however, the effects of cooling rate on phase structure as well as further electromagnetic properties of HCFCP have never been comprehensively reported, remaining a real road-block to the practical applications of microwave in solid-phase decarburization of HCFCP. In current paper, the research focuses on the comparation in the structural and electromagnetic properties of HCFCP prepared at different cooling rates in order to determine the optimal microwave heating material and further guide the solid-phase decarburization process in microwave field. As the potential fact of microwave heating by multiple frequency in the future industrial development, other frequencies in the limit of 1–18 GHz are also considered besides 2.45 GHz [23–25].

2. Experimental

2.1. Experimental raw material and equipment

The raw material for experiment, massive HCFC, is provided by Zhongxin Manganese Mining Co., LTD. in Guangxi, China and the chemical composition is listed in Table 1.

The initial block HCFC was melted in ZG-0.5 type high frequency vacuum induction heating furnace, the maximum input power of 15 kW. The device used for cooling process was SX3-12-16 type resistance furnace equipped with water-cooled system. The metallographic morphology, phase structure and microscopic component analysis of HCFC undergoing refusion and solidification were observed adopting Axio Scope. Al optical microscope (OM), D8-ADVANCE X-ray diffractometer (XRD) with a Cu Kα radiation source (the operating parameters of 40 kV, 40 mA, 2θ from 10–90°) and the JXA-8800r electron probe (EPMA) respectively. Electromagnetic parameters of HCFCP, ε_r', ε_r'' as well as μ_r', μ_r'', were measured in the frequency range of 1–18 GHz by applying the HP8722ES vector network analyzer.

2.2. Experimental method

500 g of block HCFC placed in corundum crucible was heated in high frequency vacuum induction furnace until melted completely and then keeping warm for 3 min to ensure the liquid composition achieving an uniform stability. Therewith transfer the crucible from the induction furnace to the resistance furnace quickly. By this time the temperature in the resistance furnace was kept around 1973 K close to the melting point of HCFC, and then immediately converting the operating mode from heat preservation to cooling at a speed of 283 K min^{-1}. Carrying out another two groups of melting experiments in terms of above method, but cooling rate were set up to 323 K min^{-1} and 423 K min^{-1} respectively. The choice of cooling rate is based on the industrial practice. Until three HCFC samples are down to ambient temperature, sampling for subsequent examination and analysis. Better comparison could be ensured as this examination was conducted at the same location of the three samples. Here we would give an illustration that carrying out the experiments, refusion and solidification with a certain rate, is mainly because the cooling rate of each section in raw massive HCFC is inconclusive and can not directly be used to support the purpose of the research.

Table 1
Chemical composition of HCFC.

Chemical components/$w_{(B)}$%							
Cr	Fe	C	Si	Mg	Ca	Al	Others
55.79	32.76	8.16	2.34	0.058	0.048	0.077	0.767

Three sorts of HCFC specimens prepared through above means were grinded to fine powders with particle sizes of less than 74 μm, and then compacted into samples in a sheet form with dimensions of $(22.86 \times 10.16 \times 2.00)$ mm^3 under an enough large uniaxial pressure, instead of the application of the paraffin as a class of binder shown in Li's research [19]. The relative complex permittivity ($\varepsilon_r = \varepsilon_r' - j \cdot \varepsilon_r''$) and permeability ($\mu_r = \mu_r' - j \cdot \mu_r''$) were measured on a vector network analyzer at room temperature, namely 298 K, in the frequency range from 1 to 18 GHz. The dielectric loss factor (tan δ_ε) and magnetic loss factor (tan δ_μ), equal to the ratio of imaginary part and real part, indicate the heating effect in microwave field. In particular, the tan δ_ε and tan δ_μ at 2.45 GHZ were analyzed in the range from 289 K to 1273 K, directly predicating the heating characteristic of HCFCP in industrial microwave frequency. Here it is noteworthy that the measurement of electromagnetic parameters as temperature rises was carried out by the self developed equipment displayed in Fig. 1. For the measurement of HCFCP from 1 to 18 GHz at room temperature, the heating unit in Fig. 1 is unnecessary. In order to ensure the credibility of the dielectric and magnetic measurements, the repetitive operations were performed, and the additional results were extremely similar to the results shown in paper with a largest error of nearly three percent. Therefore the measurements of electromagnetic parameters in this paper are reliable adequately.

According to the transmit-line theory [26,27], the reflection loss (R_L, dB) of HCFCP was calculated by Eq. (2), where d and Z_{in} stand for the thickness and input impedance of the absorber respectively, f is microwave frequency and c is the velocity of electromagnetic waves in free space.

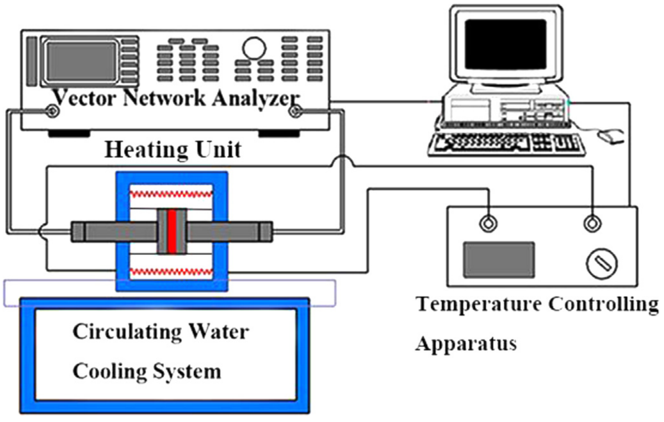

Fig. 1. Schematic diagram of the measurement of electromagnetic parameter for HCFCP at high temperature.