Study on Preparation and Properties of Fe_2O_3 Nanopowders

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Abstract. The Fe_2O_3 nanopowders were prepared by Solid Phase Reaction. The phase composition, microstructure, magnetic properties and gas sensor properties were investigated by XRD, TEM, VSM and gas sensor measuring system. It was shown that the precursor could be transformed into γ-Fe_2O_3 of the spinel structure when it was sintered at 300˚C and transformed into α-Fe_2O_3 of the corundum structure when it was sintered at 400˚C. The gas sensor of γ-Fe_2O_3 was more sensitive to the alcohol. In addition, it was found that heat insulation effect was improved evidently by adding two types of nanopowders to the paint.

Introduction

The α-Fe_2O_3 was called hematite in natural stability condition. And the γ-Fe_2O_3 was called maghemite in natural metastability condition.

In recent years, γ-Fe_2O_3 nanopowders was widely used in the field of magnetic refrigeration、magnetic recording、gas sensitive material、inorganic pigment、catalyst and biomedicine, etc[1-4]. Compared with γ-Fe_2O_3, the structural stability and gas sensitivity of α-Fe_2O_3 nanopowders were weak[5]. The γ-Fe_2O_3 and the α-Fe_2O_3 nanopowders could play a good role of close in the film due to the small partical size and good dispersion[6]. They had good ability of heat resistance and light resistance, and their effects on the atmosphere、alkaline and dilute acid was very stable. Therefore, the corrosion in hibition type of pigment such as aluminum flake pigments and zinc phosphate were often used to enhance the antirust function. In addition, it could enhance the ability of thermal radiation and the effect of thermal barrier by putting them into the paint.

In this paper, we put the xerogel calcined under a certain temperature by Solid-state Grinding Method at Room temperature. As a result, the γ-Fe_2O_3 nanopowders were sintered at 300˚C and the α-Fe_2O_3 nanopowders were sintered at 400˚C.

Experiment

The Synthesis of the Samples

Reagent: $C_4H_6O_6$ and $Fe(NO_3)_3$ $9H_2O$, all were analytical pure.

Put the equimolar $C_4H_6O_6$ and $Fe(NO_3)_3$ $9H_2O$ into the agate mortar and grind them sufficiently. Then we got the yellowish-green paste intermediates. Put the intermediates in the drying oven for drying for 3h at 80˚C and then adjust the drying oven to 100˚C , let it dry for 1h. We divided the xerogel into two, called sample 1 and sample 2, calcined for 1h at 300˚C and 400˚C , obtained γ-Fe_2O_3 and α-Fe_2O_3 nanopowders.

Solid-state Grinding Method at Room temperature belongs to mechanics. The reactants were broken under the action of external force and made their particals became smaller and free energy incresed, generating heat which promoted the reaction due to the existence of friction at the same

time[7]. In the grinding process, the nine crystal water of $Fe(NO_3)_3 \cdot 9H_2O$ was released, dissolving parts of the reactants and accelerating the process of the reaction furtherly.

Characterization of the Sample

Determine the phase of the sample by the XRD (Cu target Ka, 40 kV tube voltage, tube current 120 mA). Analyze the infra-red spectrum of sample by Perkin-Elmar 883 spectrograph at 400~4000cm^{-1} frequency range. Measure the magnetic properties of the sample by vibrating sample magnetometer (M7310). Measure the relationship between sensitivity and operating temperature of the gas sensor which made by the powder samples by using Gas Sensor Test System.

Results and Discussion

Figure 1 a, b respectively, of XRD spectrum of sample 1 and sample 2. As could be seen, sample 1 was a spinel structure of γ-Fe_2O_3 nanopowders and sample 2 was a corundum structure of α-Fe_2O_3. The studies of JING Zhi-Hong [8] had shown that nanoscale γ-Fe_2O_3 could be fully translated into the most stable α-Fe_2O_3 crystalline phases when the calcination temperature reached 600˚C . Theoretically, it should be as our results that γ-Fe_2O_3 was transformed to α-Fe_2O_3 when heated over 400˚C in the air.

Calculated the average grain size of sample 1 and sample 2 was 11nm and 18nm respectively by Scherrer formular $D=K\lambda/B\cos\alpha$.

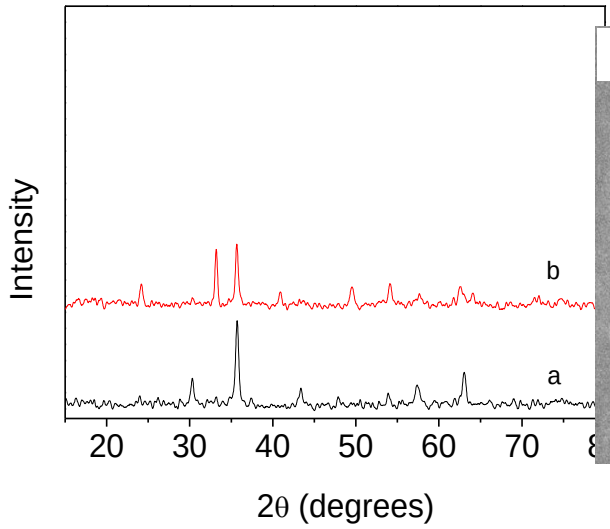

Fig.1 The X diffraction pattern **of** samples

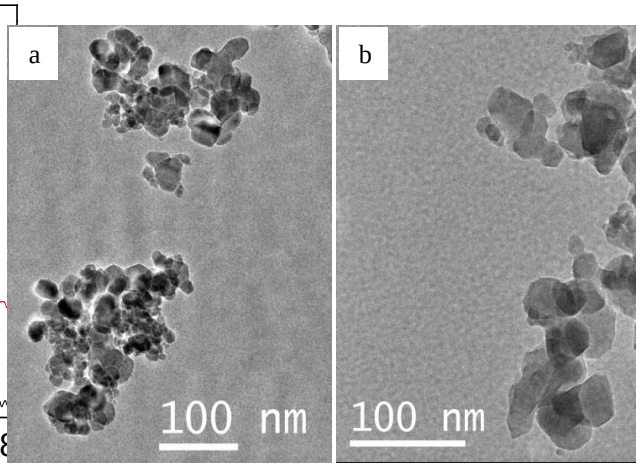

Fig 2 The TEM images of samples

Figure 2 a, b were the TEM photo of powder which called sample 1 and sample 2 respectively. It could be seen, the boundary of sample 2 was obvious and the partical dispersion was better due to the higher calcination temperature that made particles larger. Sample 1 had reunion phenomenon. The particle size of sample 1 and 2 were all below 50nm. Particle size was larger than the average grain size, because the cause of the reunion, a particle was generally a combination of several grains.

Figure 3 a, b were the infrared spectrogram of the intermediates and sample 1 respectively. In figure 3 a, the position of 1630cm^{-1} absorption peaks were significantly enhanced, which was mainly the anti-called absorption peak of carboxylic acids, and the absorption peak at 530cm^{-1} was characterized by the spectral band for the tartrate crystals, it indicated that the ion of Fe^{3+} had coordinated with the carboxyl of tartaric acid in grinding process. While the figure 3 showed the infrared spectrum characteristics of metal oxide.

Figure 4 a, b were the room temperature magnetic hysteresis loops of sample 1 and sample 2 respectively, which could be observed that sample 1 was larger than sample 2 for the specific saturation magnetization due to the γ-Fe_2O_3 was magnetic , but the pure α-Fe_2O_3 was non-magnetic,

while there was a small amount of γ-Fe_2O_3 nano-particles exited in sample 2 just because of the calcinations temperature was only 400˚C .

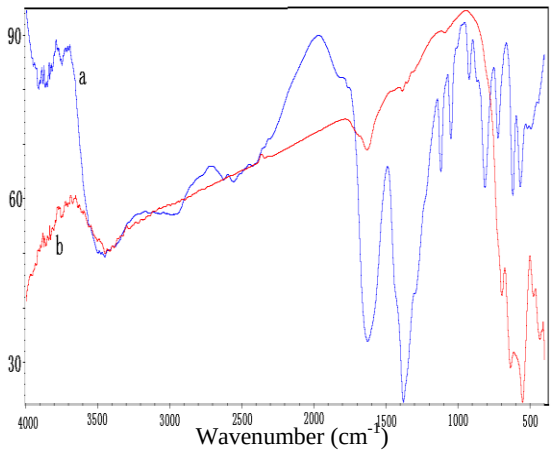

Fig.3 The FT-IR spectra for (a) Intermediate and (b) sample 1

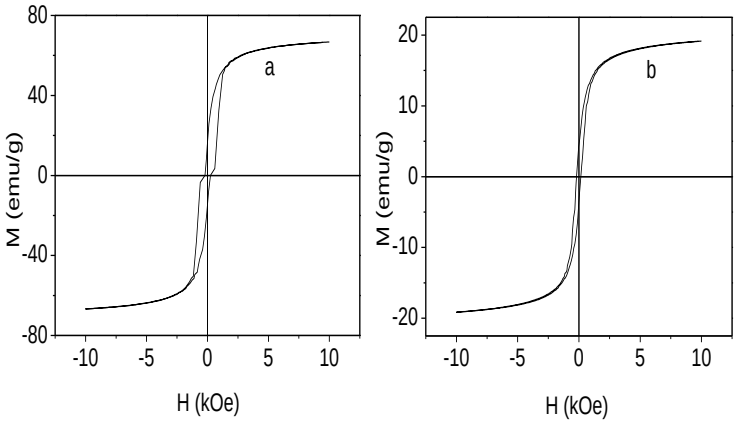

Fig.4 The magnetic hysteresis loops of samples

Sample 1 and sample 2 were porphyrized in an agate mortar and made them into paste, regarding terpineol which contain 3% of ethyl cellulose as adhesives. Then painted them on the Al_2O_3 insulating porcelain tube and calcined for 1 hour at 300˚C after stoving. Finally, put the nichrome resistance-silk into the tube core that we got, picked the tube core on the header, made a gas sensitive element after sealing cap.

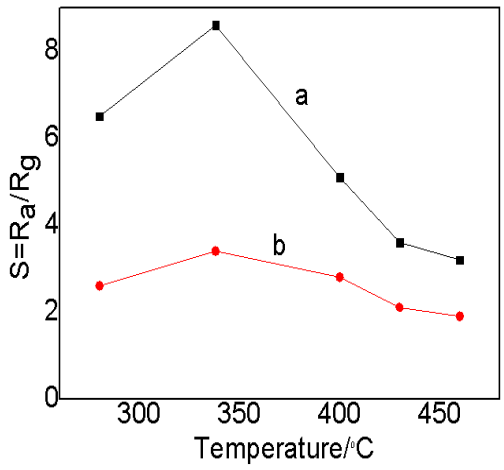

Fig.5 Gas sensitivity vs operating temperature of the sensors calcined

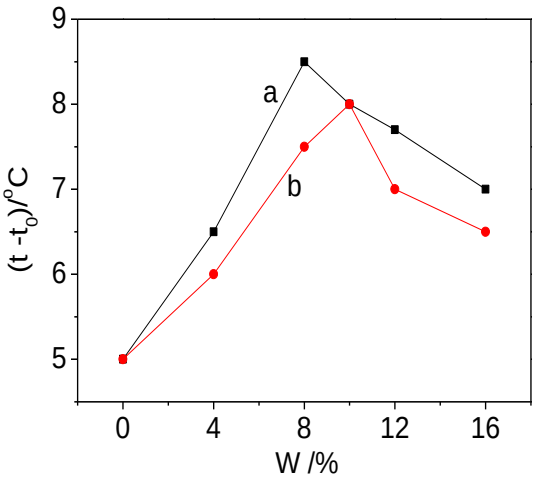

Fig.6 Fe_2O_3 content on coating of heat insulation effect

Text the sensitivity of two gas sensitive elements for the 1‰ ethanol in different temperature with 10V loop voltage. Figure 5 a, b were the relation of gas sensitivity and operating temperature of the gas sensors which made from sample 1 and sample 2 respectively. From figure 5, we knew that the two gas sensitive elements both were changed with the development of operation temperature, and they each corresponding to a maximum of the temperature which was the best working temperature, and the corresponding temperature both were 338˚C . The sensitivity of gas sensitive element which made from sample 1 was much higher than that made from sample 2, it mainly because of the gas-sensitivity of γ-Fe_2O_3 was better than α-Fe_2O_3. In addition, the gas contact area was bigger after it

making into gas sensitive element also due to the small partical size and larger surface area of the sample 1.

Put nanometer powder of sample 1 and sample 2 in coating, it could be served as pigment, also could played a role of rust protect and it could enhance the thermal barrier effect of the coating, because that the inverse spinel Fe_2O_3 could enhanced thermal radiation ability of the coating and emited the heat absorbed from sunlight to the space by thermal radiation. Figure 6 a, b showed the effect for sample 1 and sample 2 content in titanium dioxide on coating of heat insulation. When the content of γ-Fe_2O_3 was 10%, thermal barrier effect could enhance 8.5˚C . Continue to increase γ-Fe_2O_3, thermal barrier effect will reduce. When the content of α-Fe_2O_3 was 11%, thermal barrier effect could enhance 8˚C . Continue to increase α-Fe_2O_3, thermal barrier effect will reduce.The thermal barrier effect of γ-Fe_2O_3 was better than α-Fe_2O_3, because γ-Fe_2O_3 was inverse spinel, material with inverse spinel had the characteristics of high thermal efficiency, so it extensively serves as the packing of energy saving coating. The research of the infrared meteorology showed that around the areas of wavelength 8~13.5μm, the infrared radiation on ground could directly radiate to the outer space. Therefore if radiance of coating in this wave band was high as possible, then in the surface of radiator, quantity of heat could efficiently launch to the outer atmosphere by infrared radiation to achieve adiabatic goal.

Conclusion

γ-Fe_2O_3 and α-Fe_2O_3 nanopowders were prepared by solid-state grinding method. The product was spherical and the particle size was below 50nm.

The magnetic properties and gas sensing properties of γ-Fe_2O_3 and α-Fe_2O_3 nanopowders were studied.

γ-Fe_2O_3 and α-Fe_2O_3 were added to the heat insulating coating. The thermal radiation ability of the coating was enhanced, and the heat insulation effect was obvious.

Acknowledgments

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