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Ultra Fine Pure Iron Powder†

1. Introduction

JFE Steel and JFE Chemical developed a new soft magnetic material, ultra fine pure iron (UFPI) powder with an average particle size of approximately 1 μm, which is smaller than that of the carbonyl iron power used in inductors for high frequency power supplies and similar devices[1]. UPIF powder can be handled in the air in spite of its ultrafine particle size, because each of the particles is covered with a stable oxidized layer on its surface.

2. Manufacturing Method

UFPI powder is manufactured from iron oxide (hematite: Fe_2O_3) obtained by spray roasting of the waste hydrochloric acid used in washing cold rolled steel. In the manufacturing process, this iron oxide powder is reduced in hydrogen at 500–700°C, cooled to room temperature, and then held in a mixed gas of 5–10vol%O_2-N_2, to oxidize of the particle surface. An ultrafine powder can be obtained because the reduced metallic iron particles do not undergo agglomeration due to sintering in this reduction temperature range. Furthermore, because the particle surface is stabilized by oxidation treatment after reduction, a rapid oxidation reaction in air is prevented.

3. Powder Characteristics

3.1 Chemical Composition and Particle Size Distribution

Table 1 shows the chemical composition of this product. **Table 2** shows the diffraction intensities of the ferrous crystal phases detected by X-ray diffraction. The purity of iron is high in comparison with that of general-purpose iron powders for powder metallurgy. The oxygen content is high in comparison with other impurities due to the oxidized layer which is formed on the surface. The phases which form particles are ferrite (α-Fe) and magnetite (Fe_3O_4). The latter is considered to be contained in the oxidized layer on the particle surface.

Table 1 Chemical contents of UFPI powder

Element	Content (mass%)
Total Fe	>98
C	<0.02
N	<0.001
O	0.80

Table 2 Diffraction intensities of ferrous crystal phases detected by X-ray diffraction

Phase	X-ray intensity*
α-Fe	100
Fe_3O_4	0.1

*Relative intensity

The average particle size of UFPI powder measured by Kozeny Carman method is 0.85 μm, which is roughly equal to the average size of the raw material iron oxide powder.

3.2 Particle Morphology

The particle shape and size distribution were observed by scanning electron microscope (SEM), transmission electron microscope (TEM), and scanning ion microscope (SIM). The thickness of the oxidized layer on the particle surface was estimated a depth profile of oxygen across the particle surface by Auger electron spectroscopy (AES).

Photos 1, **2**, and **3** show SEM, TEM, and SIM images of this product, respectively. In Photo 1, the particle shape is irregular, resembling that of the raw material iron oxide powder, and virtually any conjunction between particles formed by sintering during reduction process could not be observed. Considering the facts that grain boundaries cannot be observed within the particles, as shown in Photos 2 and 3 (particle cross section at part indicated by an arrow), as well as the X-ray diffraction results, the particles appear to be single crystals of α-Fe.

On the basis of the depth profile obtained by AES, the oxygen is enriched on the particle surface approximately 20 nm in thickness. This is considered to be the oxidized surface layer which formed on the particle.

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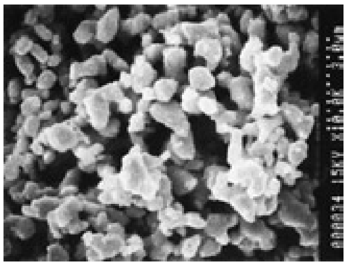

Photo 1 SEM image of UFPI powder

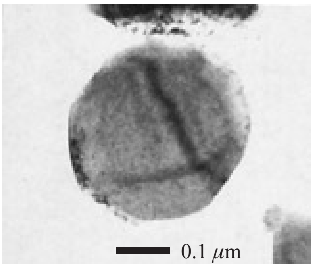

Photo 2 TEM image of UFPI powder

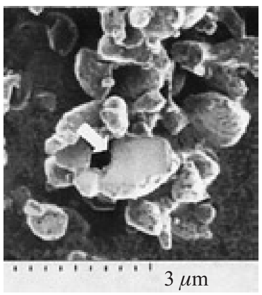

Photo 3 SIM image of UFPI powder (A cross-section view is indicated by an arrow)

Table 3 Magnetization and coercive force of UFPI powder at 800 kA/m

Magnetization, M_{800k} (Wb · m)/kg	$2\,512 \times 10^{-7}$
Coercive force, H_c (A/m)	1 968

4. Magnetic Properties

Table 3 shows the magnetization, M_{800k} and coercive force, H_c of UFPI powder measured by a vibrating sample magnetometer (VSM) under a maximum applied magnetic field of 800 kA/m. The value of M_{800k} is approximately 9% lower than the saturation magnetization (20°C, $2\,738 \times 10^{-7}$ Wb · m/kg)[2)] of bulk iron material. This corresponds to the reduction in the metallic iron content due to formation of the oxidized surface layer.

5. Examples of Application

5.1 Powder Cores

The UPIF powder of a soft magnetic material can be applied for iron powder cores. The powder cores were prepared by mixing UFPI powder with 5 mass% phenol resin, followed by compacting at 686 MPa to a ring shape with an outer diameter of 12 mm, inner diameter of 7 mm, and thickness of approximately 3 mm.

Table 4 Initial permeability and Q value of UFPI powder core

	UFPI
μ_i at 1 MHz	22
Q_{max}	69.0
f at Q_{max} (MHz)	7

Number of turns: 10

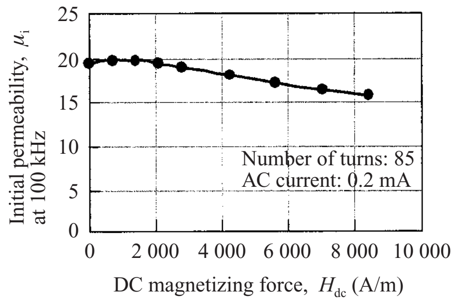

Fig. 1 Initial permeability of iron powder core vs. DC magnetizing force

Table 4 shows the initial permeability, maximum Q value, Q_{max}, and frequency, f at which Q shows Q_{max} in the UPIF powder core. In the frequency region up to 10 MHz, the UFPI powder core showed high permeability. Thus, uisng UFPI powder, it is possible to manufacture inductors with high permeability and low loss.

Figure 1 shows the DC superposition characteristic of inductance of the same specimen at 100 kHz. Because the initial permeability of the UFPI powder core is high across the range of DC field intensities up to 8 800 A/m, application to power inductors is expected.

Cores manufactured using UFPI powder perform high permeability irrespective of whether a DC field exists or not. This is considered to be attained to the single-crystal structure without grain boundaries which would prevent the propagation of domain walls within the powder particles as shown in Photo 2.

5.2 Remediation Agent for Contaminated Water and Soil

Iron powder has attracted attention as an environmental purification material for degrading volatile organic chlorine compounds (VOC), which have been subject to stricter regulation as environmental pollutants in recent years[3)]. The degradation performance of UFPI powder was investigated using trichloroethylene (TCE), which is one VOC, as the substance to be degraded.

Figure 2 shows the change in TCE concentration over time when UFPI powder and a comparison reduced

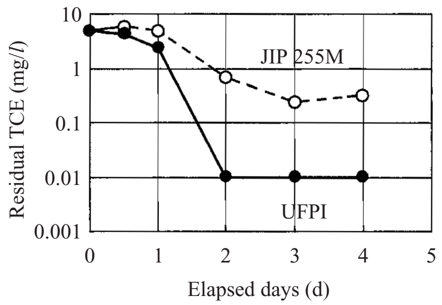

Fig. 2 TCE degradation property of iron powders

iron powder for powder metallurgy (JIP255M, manufactured by JFE Steel) were mixed in 50 m*l* of an aqueous solution containing 5 mg/*l* of TCE, respectively, and then held under the same conditions. The ordinate shows the residual TCE concentration in the water in accordance with the head space method. With the mixture containing UFPI, the TCE concentration decreased more rapidly than with the comparison material, and reached the limit of the detectable concentration with the TCE gas detection tube on the second day. In addition to the small particle size of the UFPI powder, it is assumed that a galvanic cell is formed in the water because metallic iron coexists with the iron oxide at the particle surface layer, and the VOC degradation reaction[4] is accelerated by the resulting exchange of electrons. The average particle size of less than 1 μm allows injection into soil, and thus also enables application to soil purification by the *in-situ* degradation method[5].

6. Conclusions

UFPI powder is a high-purity ultrafine iron powder with an average particle size of less than 1 μm which can be handled in the air.

(1) The UFPI powder core shows high permeability in the frequency range up to several 10s of MHz, and also performs the same tendency under superposition of a strong DC magnetic field. Based on these advantages, UPIF powder is expected to be applied for electronic parts for which low loss in the high frequency range and high magnetic flux density are required, including large current inductors for use in the CPU drive power supply of personal computers.

(2) UFPI powder rapidly degrades VOC due to its large specific area and the existence of a 2-layered structure consisting of metallic iron and iron oxide at the particle surface, which forms a galvanic cell in water. Its small particle size also enables injection into soil, making it possible to apply UFPI as a soil remediation agent.

References

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