Synthesis of carbon coated iron nitride nanoparticles by using microwave plasma technique

Bo Wei¹, Xin Jin¹, Qun Wang¹, Yongqing Li¹ and Waheed Q Khan¹
¹ School of Materials Science and Engineering, Beijing University of Technology, Beijing 100124, People’s Republic of China
² Institute of Advanced Materials, Bahauddin Zakariya University, Multan, Pakistan
* Author to whom any correspondence should be addressed.
E-mail: jinsanity@emails.bjut.edu.cn and wangq@bjut.edu.cn
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Abstract
Carbon-coated iron nitride nanoparticles were synthesized using ferrocene as the precursor material and an in-house manufactured microwave-plasma reaction system. XRD revealed that the nanoparticles contained mixed phases of iron nitride (FeN₀.₀₅₈₉ and ε-Fe₃N) and α-Fe. In the case of TEM, the powder displayed a core–shell structure with a core diameter of ∼15 nm and had a multilayer carbon structure. The particle sizes in all samples were 10–80 nm. The Raman spectra of the nanoparticles justified that the multilayer carbon coatings had an amorphous graphitic structure. The nanoparticles developed at an N₂ flow rate of 10 slpm yielded a superior Ms value of 37.2 emu g⁻¹. An increase in the N₂ flow rate exhibited a significant influence on the plasma temperature and reaction time, thereby affecting the composition of the mixed phase, the shape and size of the particles, and the uniformity and thickness of the carbon coating. This study provided a simple, efficient and economical method to prepare iron nitride magnetic nanoparticles, which is expected to be widely applied in industrial mass production.

1. Introduction

Magnetic nanoparticles are of considerable importance in research due to their unique physicochemical properties [1–3]. Nanosized particles easily to coalescence of individual particles and lose characteristic properties due to its small particle size with large specific surface area and highly surface energy. To maintain its inherent characteristics, it is necessary to modify the surface of nanoparticles [4, 5]. The common method is to coat one or more layers of other materials (organic, inorganic, protein or antibody) on the surface of magnetic particles to form core–shell structure nanoparticles [6]. Hence, the composite particles show the surface properties of the shell and maintain unique properties of the core, such as electricity, light and magnetism to realize the multifunction of the single system [7]. This ability of coated magnetic nanoparticles (core/shell) is exploited for various applications in drug delivery [8, 9], magnetic resonance imaging (MRI) [10], biosensors [11], catalysis [12, 13] and electromagnetic wave absorber [14, 15].

Iron nitride nanoparticles exhibit more attractive magnetic properties, mechanical properties and stability than iron oxide nanoparticles and comprise various crystal structures (FeN, ε-Fe₂N, δ-Fe₂N, γ-Fe₄N and δ'-Fe₄N) [16, 17]. N atoms can enter the lattice of magnetic metals in the form of covalent bond or metallic bond, and the intercalation of interstitial atoms can effectively reduce the density of the material. Combined with the magnetic volume effect, low nitrogen compounds (Fe₂N and Fe₄N) have higher saturation magnetization than their parent metal. Iron nitride magnetic nanoparticles have gained significant interest for applications such as permanent magnet [18], magnetic fluid [19] and absorbing material [20].

Carbon (graphitic or amorphous) is a preferred coating material due to its lightweight, chemical inertness, biocompatibility and ease of functionalization in comparison to other commonly applied materials (silica, boron nitride, noble metals, etc) [21, 22]. In addition, carbon material is a type of absorbing material with dielectric polarization and resistance loss as the absorption mechanism, which has been widely studied [23].
Therefore, iron nitride magnetic nanoparticles with carbon coating has dielectric polarization absorbing mechanism of the carbon shell and magnetic loss absorbing mechanism of the magnetic core, and they increase the multiple scattering and interface polarization at the interface because of the formation of many carbon-metal interfaces. Furthermore, the carbon shell with proper thickness can enhance the dielectric loss and benefit the impedance match between composite particles and air to improve the microwave-absorbing efficiency, although the saturation magnetization of the composite magnetic particles is reduced [24].

Various methods have been developed for the synthesis of carbon-coated nanoparticles, such as laser [25], ionic beam [26] chemical vapor deposition [27], pyrolysis [28], and explosion [29]. However, the plasma synthesis technology has attracted increasing attention since the plasma chemical reaction has unique advantages [30, 31]: (i) the higher activity atmosphere makes the chemical reaction proceed very quickly, which can produce high-temperature phase compounds (such as nitrides, carbides and borides) that are difficult to synthesize by the chemical liquid phase method [32]; (ii) when the reactants leave the plasma, the cooling rate is high (up to \(10^5 \text{ K s}^{-1}\)), which is beneficial to obtain nano-scale powders; (iii) high-purity powder can be obtained in fewer processing steps, and the byproduct is a gas phase, which is easy to handle; (iv) the method easily achieves scale and automation. The most commonly used plasma synthesis methods are arc plasma, radio frequency (RF) plasma and microwave plasma. For example, Rakesh et al [33] developed carbon-coated Fe nanoparticles by plasma and ultrasonication in toluene. Borysiuk et al [34] developed carbon-coated Fe nanoparticles using the plasma arc and combustion method. Shinde et al [35] synthesized Fe/C core–shell nanostructure using the DC plasma arc discharge technique. In our previous study, we successfully synthesized iron and iron nitride carbon-coated magnetic nanoparticles using RF plasma [36]. Dong et al [37] synthesized carbon-coated LiNi0.5S0.5 powders for lithium-ion batteries using microwave plasma.

Compared with arc plasma, RF and microwave plasma have higher electron temperature, can work at lower pressure and provide higher degrees of ionization and dissociation; there is no electrode pollution, corrosion and energy waste, which is favorable to produce superior-purity materials. In addition, the microwave plasma method has unique advantages: (i) wider emission spectrum and longer lifetime of excited-state particles than other methods; (ii) possible transportation and control of microwave plasma in a specific space; and (iii) low-cost equipment, no expensive RF power supply, etc [38], as well as superior product quality [39]. In this work, we synthesized carbon-coated iron nitride nanoparticles in a custom-made microwave plasma unit using ferrocene precursor and nitrogen (N₂) as the plasma gas. Then, we studied the influence of the plasma-gas flow rate on the powder morphology and magnetic properties.

2. Experimental

2.1. Microwave plasma synthesis system

The in-house-built microwave-plasma synthesis unit in the present study is shown in figure 1. The microwave plasma apparatus consists of four major systems: (i) microwave source; (ii) microwave transmission; (iii) material reaction system; (iv) powder collection system and vacuum system. The microwave source is magnetron with a power of 1.5 kW and a frequency of 2.45 GHz. The transmission system was straight through a rectangular waveguide for microwave transfer and a large rectangular cavity to avoid damaging the magnetron, which limits the reflection of electromagnetic waves. The plasma was generated in the quartz tube (material reaction zone), which was vertically inserted in the rectangular waveguide from a wider surface. The insertion position and dimension of the quartz tube were determined based on the transmission characteristics of the wave in the waveguide. The geometry of the rectangular waveguide is shown in figure 2(a), where it is assumed that the guide is filled with air of permittivity \(\varepsilon_0\) and permeability \(\mu_0\). It is standard convention to have the longest side of the waveguide along the x-axis, so that \(a > b\). The dominant mode \(\text{TE}_{mn}\) (\(m = 1, n = 0\)) can obtain the maximum electric field and be more conducive to the plasma reaction. According to the formula of the transverse field components of the \(\text{TE}_{10}\) mode [40], (i) there is only the y-axis component of electric field \(E_y\), and the x-axis \((E_x)\) and z-axis \((E_z)\) components are zero; (ii) when \(x = a/2\) and \(z = \lambda_y^* (2k + 1)/4 (k = 0, 1, 2\ldots)\), \(E_{y,\text{max}}\) can be obtained. Here, \(\lambda_y\) is the waveguide wavelength. The selected type of rectangular waveguide is WR-340 (\(a = 86.36 \text{ mm and } b = 43.18 \text{ mm}\)). From the following equations:

\[
\lambda_y = \frac{\lambda}{\sqrt{1 - (\lambda/\lambda_c)^2}}
\]

\[
\lambda_c = 2\sqrt{(m/a)^2 + (n/b)^2}
\]

it can be calculated that \(\lambda_y\) is 173 mm at 2.45 GHz. Here, \(\lambda\) and \(\lambda\) are the cutoff wavelength and working wavelength. Overall, the inserted position of the quartz tube is \(x = 43.18 \text{ mm } (a/2)\) and \(z = 130 \text{ mm } (\lambda_y^* 3/4)\). The results of contrast in figure 2(b) indicate that there was good consistency between calculated data and simulation data (by HFSS software, ANSYS). The smaller diameter of the quartz tube limits the reaction zone.
and affects the preparation efficiency. However, a larger diameter of the quartz tube changes the wall-current distribution of the rectangular waveguide [40]. In general, the diameter of the quartz tube should be less than half of the working wavelength [41]. Here, we selected 30 mm for the present unit.

Synthesized carbon-coated nanoparticles were dispersed onto the wall of the powder collection chamber, which was connected to the vacuum pump. Ultrafine filters were used as the air separators between the powder-collecting chamber and the vacuum system. The powder was collected in the filter, and the rotary pump pumped out the gas. Powder collected in the filters was only used for characterization.

2.2. Synthesis of carbon-coated iron nitride particles by microwave plasma
The microwave plasma was generated by using pure N₂ (99.99% purity) at a flow rate of 3 slpm and 0.04 MPa in the presence of microwaves. Then, the N₂ plasma gas flow rate was varied to be 5 slpm (S₁), 10 slpm (S₀), 15 slpm (S₂) and 20 slpm (S₃) to study its effect on the powder morphology and magnetic properties. The flow of plasma gas was controlled by using the attached mass flow meters. During the reaction in the microwave-plasma reaction site, a certain amount of cooling water (20 °C) was first introduced to reduce the temperature of the magnetron and outer wall of the powder-collecting chamber.

Ferrocene (Sigma-Aldrich, F408-100G) was used as a precursor for the production of iron nitride carbon-coated nanoparticles. Ferrocene is considered a suitable regent due to its nontoxicity and lower flammability [42]. Ferrocene was heated at 140 °C in N₂ atmosphere using a heat bed, and vapors were uniformly injected into the reaction zone (quartz tube) with the carrier N₂ gas. The ferrocene vapor pressure can be calculated using

![Figure 1. Schematic diagram of the microwave-plasma synthesis apparatus to synthesize carbon-coated iron nitride nanoparticles.](image1)

![Figure 2. Diagram of the (a) geometry of a rectangular waveguide and (b) electric field intensity distribution in the x-z plan.](image2)
Clark and Glew equation [43]. The flow rate of the carrier gas was adjusted at 1.5 slpm and a pressure of 0.04 MPa. The ferrocene flow rate was calculated using the attached mass flow meters.

The achieved carbon-coated nanoparticles produced by a higher N2 plasma gas flow rate (10 slpm) were blackened, and those by the lower N2 plasma gas flow rate (5 slpm) partly maintain the same color as ferrocene. Hence, the gas flow rate of 10 slpm could generate adequate dissociation of plasma gas to completely react with Fe vapors to produce iron nitride, while the rate of 5 slpm cannot.

2.3. Characterization of carbon-coated iron nitride nanoparticles

To find the structure and phases of the carbon-coated iron nitride nanoparticles, XRD (Shimadzu - 7000) was performed. The morphology of the carbon-coated iron nitride powder was characterized using TEM (TEM, JEM - 2010, Japan). The samples for TEM were prepared by sonication of powder in ethanol solution at 30 °C. The homogeneous suspension was spin-casted on copper TEM grids to be coated with conductive carbon. The particle size distribution of powders was measured using the software ImageJ. A Raman spectroscope equipped with a 532 nm laser (Thermo Fisher Scientific) was used to study the structural properties of the graphite shell. Magnetic parameters of the samples were measured using VSM (Lake Shore, 7307, VSM) at room temperature.

3. Results and discussion

The XRD patterns of the samples (S0, S1, and S2) produced at various N2 plasma gas flow rates are shown in figure 3. All samples contain four clearly visible peaks at 38.29°, 41.22°, 43.71° and 49.37°, which were designated as the \{110\}, \{002\}, \{111\}, \{201\} crystal planes of the \(\varepsilon\)-Fe3N phase (PDF # 086-0232). Two diffraction peaks at 50.02° and 74.24° for planes \{200\} and \{220\} of FeN 0.0589 (PDF #75-2130) were also identified in the samples. The x-ray reflection of as-synthesized powder shows that it contained mixed phases of \(\varepsilon\)-Fe3N (hexagonal closed packed structure) and FeN 0.0589 (tetragonal structure) regardless of the N2 flow rate. Moreover, peaks of \(\alpha\)-Fe (PDF #99-0064) were observed at 2\(\theta\) values of 44.67° and 65.02° for the \{110\} and \{200\} crystal planes. Phase FeN 0.0589 is energetically unfavorable, so it could have been transformed into \(\alpha\)-Fe [43, 44]. Furthermore, the chamber cooling of the \(\varepsilon\)-Fe3N phase at high temperature could result in its transformation to \(\alpha\)-Fe. There is no apparent peak of Fe3C, possibly due to the longer residence time of nanoparticles in the high-temperature region, and lower-temperature gradients limited the formation of Fe3C [45]. In other words, the achieved product was free of iron carbides, and similar results were reported by Feng et al [36].

With the increase in the N2 flow rate from 10 slpm (S0) to 20 slpm (S2), the peak of the Fe3N phase had a slight decrease in intensity, while the intensity of the \(\alpha\)-Fe peak increased. This behavior can depend on the kinetics of
the reaction: an increase in \( \text{N}_2 \) plasma velocity limits the residence time for Fe vapors formed from the ferrocene decomposition to completely react with \( \text{N}^+ \) to produce iron nitride.

The broad diffraction peak at 25.80° is assigned to the \([110]\) planes of the hexagonal graphite structure. The peak is asymmetric, broader and lower than that of the well-crystallized graphite, which indicates a small crystalline dimension. Due to the high defect density, amorphous carbon leads to higher \( I_D \), which leads to a low \( I_G \).

Based on the diffraction pattern of XRD, a semi-quantitative analysis was performed using the reference intensity ratio method (RIR) to validate the trend of phase content at various \( \text{N}_2 \) flow rate. As shown in table 1, with the increase in \( \text{N}_2 \) flow rate, the content of \( \alpha\)-Fe and \( \text{FeN}_{0.0589} \) increased, while the content of \( \text{Fe}_3\text{N} \) decreased. The reason could be the transformation of phase \( \varepsilon\)-Fe,N to phase \( \alpha\)-Fe with the increase in plasma temperature due to the higher nitrogen flow rate. Furthermore, Fe vapors formed from the ferrocene decomposition incompletely reacted with \( \text{N}^+ \) to form iron nitride with a low nitrogen content in a short residence time.

To analyze the carbon structure formed by a microwave-plasma reaction system, Raman spectra of all samples were obtained. There were peaks at 2698 cm\(^{-1}\) and 2708 cm\(^{-1}\), which are consistent with the characteristic peaks of the graphite structure. The \( I_D \) peak in figure 4(f) is small in all samples. Some irregular-shaped particles were developed and contained laced carbon layers. An increase in \( \text{N}_2 \) flow rate to 20 slpm favored the formation of active \( \text{N}^+ \) species but limited the amorphous carbon (peak in figure 4) to completely shield magnetic particles (figure 4(f)). In addition, the irregular shape of the core could be due to the mixed phases of iron and iron nitrides of different crystal structures. It was seen that few particles agglomerated because the rupture or absence of layers of carbon provided freedom to adjacent particles to form anisotropic magnetic poles, and the mutual attraction between magnetic poles reduced the distance among particles, so they might even come in contact.

Table 1. Composition of the synthesized powders by XRD.

| Phases  | \( \alpha\)-Fe (wt%) | \( \text{Fe}_3\text{N} \) (wt%) | \( \text{FeN}_{0.0589} \) (wt%) |
|---------|----------------------|-------------------------------|-------------------------------|
| \( S_0 \) | 14                   | 79                            | 7                             |
| \( S_1 \) | 21                   | 60                            | 19                            |
| \( S_2 \) | 33                   | 43                            | 24                            |

Based on the XRD and TEM results, carbon-coated mixed-phase magnetic particles of iron nitride were successfully synthesized using the adopted procedure, and the \( \text{N}_2 \) flow rate slightly affects the morphology of nanoparticles.

To analyze the carbon structure formed by a microwave-plasma reaction system, Raman spectra of all samples were obtained. There were peaks at \( \sim 1330 \text{ cm}^{-1} \), \( 1585 \text{ cm}^{-1} \), and \( 2660 \text{ cm}^{-1} \), which are consistent with the characteristic peaks of the graphite structure. The \( D \) peak near \( 1330 \text{ cm}^{-1} \) originates from defects, disorder, and finite particle size effects. The \( G \) peak, which is the main characteristic peak of the graphite structure and initiated by the in-plane vibration of the sp\(^2\) atom in the graphite structure. The intensity ratio \( I_D \) between D band and G band is an important factor of the graphitization degree. Due to the high defect density, amorphous carbon leads to higher \( I_D \), which leads to higher \( I_D/I_G \). With the increase in \( \text{N}_2 \) flow rate from 10 slpm to 20 slpm, \( I_D/I_G \) slightly decreased, which indicates that the amorphous carbon in the sample was reduced when the graphitization degree increased. The \( G' \) peak, which is also known as the 2D peak, appears near \( 2660 \text{ cm}^{-1} \). It is a second-order Raman peak of double phonon resonance, which is used to characterize the stacking mode of carbon atoms. In addition, the \( G' \) peak can be used to distinguish the number of carbon or graphene layers depending on the peak width. Therefore,
carbon coatings formed on iron nitride nanoparticles have distorted the graphite crystal structure with a multilayer of carbon with turbostructural stacking and a $G'$ peak width of $\sim 130$ cm$^{-1}$. Thus, carbon coating formed by microwave plasma contains more amorphous carbon than reported in our previous work using arc plasma [36]. With the increase in N$_2$ flow rate, the $G'$ peak width slightly decreased, which indicates that the number of carbon coating layers in the sample is reduced with short residence time of species of ferrocene. To understand the structural properties of carbon in-depth, further investigation is required, which will be presented in future study.

The magnetic properties of the as-synthesized nanoparticle samples at various N$_2$ flow rates were measured at room temperature using VSM. The hysteresis loops of all samples are shown in figure 5(a). The curves show that these particles have nearly zero hysteresis and can rapidly respond to the applied external field, which indicates that the particles are superparamagnetic. The FC-ZFC measurement of the as-synthesized sample ($S_0$) was completed. As shown in figure 5(b), the curve shows the characteristic of typical superparamagnetic materials. The coercivity field ($H_c$), saturation magnetization ($M_s$) and remnant magnetization ($M_r$) are shown in

![Figure 4. TEM and HRTEM images of carbon-coated iron nitride nanoparticles synthesized samples by the microwave plasma reaction system at various N$_2$ flow rates of (a), (b) 10 slpm, (c), (d) 15 slpm and (e), (f) 20 slpm; (g) nanoparticle size distribution and (h) Raman spectrum of samples $S_0$, $S_1$, and $S_2$.](image)
The saturation magnetization of bulk Fe$_3$N is 123 emu g$^{-1}$ [36]. The di-magnetic layer of carbon was present on the surface of nanoparticles (figure 4(b)), which decreased the net magnetic moment by interacting with the 3$d$ electrons [49]. Graphite is diamagnetic with a magnetic susceptibility of $-21.5 \times 10^{-6}$ emu g$^{-1}$ parallel and $0.5 \times 10^{-6}$ emu g$^{-1}$ perpendicular to the magnetic field [50]. Thus, the di-magnetic phase (carbon) has a harmful effect on the saturation magnetization per unit mass. Similar findings have been reported, where the saturation magnetization is reduced by carbon-coated iron and iron nanoparticles [17, 36]. Another reason is that carbon-coated magnetic particles are not pure Fe$_3$N phase or bulk iron, which can lead to the lower saturation magnetization than that for bulk iron or iron nitride. Samples S$_1$ and S$_2$ have $M_s$ of 26.5 emu g$^{-1}$ and 17.1 emu g$^{-1}$, respectively. With an increase in N$_2$ flow rate, $M_s$ of the carbon-coated magnetic particles decreased. As shown in table 1, the $\alpha$-Fe/$(\alpha$-Fe + Fe$_3$N) ratio increases with the increase in N$_2$ flow rate. Compared with carbon-coated Fe$_3$N nanoparticles, carbon-coated $\alpha$-Fe nanoparticles have a weak ferromagnetic phase, which decreases the magnetic properties of the prepared samples. The saturation magnetization of carbon-coated $\alpha$-Fe is 20 emu g$^{-1}$ [36].

It is known that for iron nitride thin films, the coercivity is affected by the nitrogen content, anisotropy and inhomogeneity (grain size, stress and surface roughness, etc) [51]. In this work, the surface of FeN nanoparticles shows no significant change with plasma velocity. The coercivity for mixed –Fe$_3$N + –Fe was due to different phases, grain sizes and grain shapes.

### 4. Conclusions

The 2.45-GHz microwave plasma system using C$_{10}$H$_{10}$Fe as the precursor and N$_2$ as the plasma gas was employed to produce one-step carbon-coated iron nitride nanoparticles. The results showed that the carbon-coated iron nitride nanoparticles prepared by this method had considerable saturation magnetization and uniform carbon coatings. The achieved core of the product comprised mixed phases of iron nitride (FeN$_{0.0589}$ and $\varepsilon$-Fe$_3$N) and tiny amounts of $\alpha$-Fe, but it did not contain any weak magnetic phase of iron carbide. Composite nanoparticles had multilayer amorphous carbon shell with the size of 10–80 nm. This method can provide an effective, efficient, and economical direct synthesis of carbon-coated nanoparticles. Furthermore, the increase in N$_2$ flow rate from 5 s lpm to 20 s lpm produced significant effect on the plasma temperature and reaction time, which affected the composition of the mixed phase, shape and size of the particles, magnetization and uniformity and thickness of the carbon coating. The composite properties prepared at the nitrogen flow rate

### Table 2. Magnetic properties of the as-synthesized powder samples.

| Properties       | S$_0$ | S$_1$ | S$_2$ |
|------------------|------|------|------|
| $H_c$ (Oe)       | 51.4 | 57.0 | 60.8 |
| $M_s$ (emu g$^{-1}$) | 37.2 | 26.5 | 17.1 |
| $M_r$ (emu g$^{-1}$) | 0.52 | 0.53 | 0.55 |

![Figure 5. (a) Magnetic hysteresis loop of the as-synthesized powder samples; (b) FC-ZFC curve of as-synthesized sample S$_0$.](image-url)
of 10 slpm presented an ideal core–shell nanostructure morphology with a core diameter of ∼15 nm, a shell of ∼7 nm, and optimal saturation magnetization of 37.2 emu g⁻¹. From the aspect of absorbing mechanism, such core–shell magnetic nanomaterials are notably suitable for microwave-absorbing materials. Therefore, carbon-coated iron nitride nanoparticles for absorbing materials produced by microwave plasma can be a promising approach for commercialization.

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ORCID iDs

Xin Jin https://orcid.org/0000-0003-2082-2442

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