

# Synthesis and electromagnetic wave reflectivity of $\text{Si}_3\text{N}_4$ ceramic with gradient $\text{Fe}_3\text{O}_4$ distribution



Xiangming Li<sup>a,\*</sup>, Mingjun Gao<sup>a</sup>, Yun Jiang<sup>b</sup>

<sup>a</sup> School of Environment and Materials Engineering, Yantai University, Yantai, Shandong 264005, PR China

<sup>b</sup> Department of Foreign Languages, Northwest A&F University, Yangling, Shaanxi 712100, PR China

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## ABSTRACT

A  $\text{Si}_3\text{N}_4$  ceramic with gradient distribution of tri-iron tetroxide (Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$ ) was fabricated with a combined technique of chemical precipitation and directional infiltration. Electromagnetic wave could enter Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$  with little reflection because of weak impedance mismatch at its surface. Also the electromagnetic wave entering the Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$  propagated with small reflection due to continuous and gradual change of impedance resulting from the gradient  $\text{Fe}_3\text{O}_4$  distribution, and was absorbed completely by  $\text{Fe}_3\text{O}_4$  of the gradient structure.

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## 1. Introduction

Many studies have demonstrated that absorption is more useful than reflection in shielding electromagnetic wave though absorption is more difficult to achieve than reflection [1–11]. In these studies, carbon and ferrite are two typical electromagnetic wave absorbers, which are usually added in a material to enhance its electromagnetic wave absorption. However, the surface impedance mismatch of the material worsens with carbon or ferrite inclusion, which presents a difficulty in absorbing electromagnetic wave. Therefore, the electromagnetic wave absorption of a material could hardly be enhanced simply by adding carbon or ferrite [1–3].

For example,  $\text{Si}_3\text{N}_4$  ceramic with uniform distribution of pyrolytic carbon (PyC- $\text{Si}_3\text{N}_4$ ) demonstrates strong surface impedance mismatch, so there is a large part of incident electromagnetic wave reflected on the surface of PyC- $\text{Si}_3\text{N}_4$  [2]. However, due to absence of PyC at its surface,  $\text{Si}_3\text{N}_4$  ceramic with gradient distribution of PyC (Gradient-PyC- $\text{Si}_3\text{N}_4$ ) not only shows strong attenuation of electromagnetic wave but also demonstrates weak surface impedance mismatch. Thus, most of incident electromagnetic wave could enter Gradient-PyC- $\text{Si}_3\text{N}_4$  and be absorbed [3]. As demonstrated by our previous work [3], the electromagnetic wave absorption of the material could be enhanced effectively by the introduction of the absorber with a gradient distribution.

Tri-iron tetroxide ( $\text{Fe}_3\text{O}_4$ ) is one of the ferrite series

\* Corresponding author.

E-mail address: [li\\_xiangming@yahoo.com](mailto:li_xiangming@yahoo.com) (X. Li).

electromagnetic wave absorbers, which is gaining interest for its high wave-absorbing strength. However, the narrow wave absorbing band of  $\text{Fe}_3\text{O}_4$  limits its application as a good electromagnetic wave absorber [1,5–11]. Some studies have been carried out to fabricate  $\text{Fe}_3\text{O}_4$  nanoparticles with different morphology [12–14], especially urchin-like [12], dendrite-like [13], hollow spherical [14], etc., to widen the wave absorbing band of  $\text{Fe}_3\text{O}_4$ . The electromagnetic wave absorption of  $\text{Fe}_3\text{O}_4$  nanoparticles with special morphology is enhanced significantly because of large specific surface area of  $\text{Fe}_3\text{O}_4$  nanoparticles. However, the material with uniform distribution of  $\text{Fe}_3\text{O}_4$  nanoparticles also shows poor electromagnetic wave absorption due to its strong surface impedance mismatch.

In this work, a  $\text{Si}_3\text{N}_4$  ceramic with gradient distribution of  $\text{Fe}_3\text{O}_4$  (Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$ ) is fabricated with a combined technique of chemical precipitation and directional infiltration. Microstructure observation and phase identification of  $\text{Fe}_3\text{O}_4$  are carried out. The effect of infiltration pressure on  $\text{Fe}_3\text{O}_4$  distribution in Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$  is investigated. The electromagnetic wave reflectivity of Gradient- $\text{Si}_3\text{N}_4$ - $\text{Fe}_3\text{O}_4$  with different patterns of  $\text{Fe}_3\text{O}_4$  distribution is measured and discussed.

## 2. Experimental procedure

The porous  $\text{Si}_3\text{N}_4$  ceramic fabricated in our previous work [15] was machined into preform with a dimension of 180 mm × 180 mm × 5 mm, and then was assembled in a device (Fig. 1) to infiltrate  $\text{Fe}_3\text{O}_4$  nanoparticles directionally. Before infiltration process, a solution of  $\text{FeCl}_3$  (0.024 mol/L) and  $\text{FeCl}_2$  (0.012 mol/L)

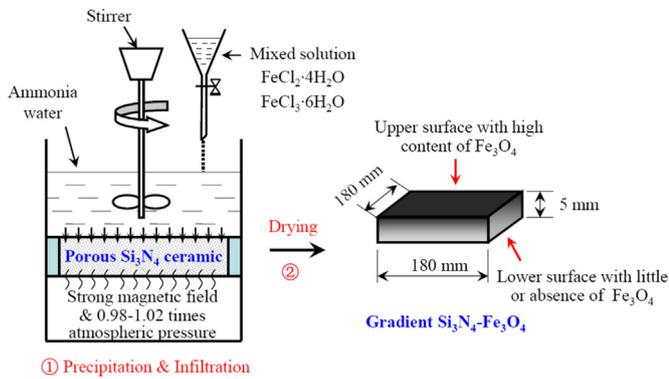


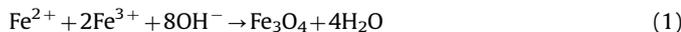
Fig. 1. Schematic of the process of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$  fabrication.

was prepared by mixing  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  with distilled water. As shown in Fig. 1, an ammonia water with concentration of 5–6 mol/L was poured into the chamber above the preform, and then the mixed solution dripped slowly into the ammonia water. During the addition process, the ammonia water was stirred rapidly and a big powerful magnet was placed beneath the preform to speed the infiltration of reaction-derived  $\text{Fe}_3\text{O}_4$  nanoparticles into the preform. When there was no liquid seeping out from the lower surface, the preform was taken out and dried at  $90^\circ\text{C}$  for 5 h in air. The distribution of  $\text{Fe}_3\text{O}_4$  in the preform could be controlled and adjusted by changing the pressure in the chamber at the bottom of the device (Fig. 1). The suitable pressure range was 0.9–1.05 times atmospheric pressure. For the convenience of the following discussion, the Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$  prepared with  $m$  times atmospheric pressure was denoted as Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4\text{-}m$ .

The microstructure was observed with a scanning electron microscopy (SEM, S-4800, Hitachi, Japan). Phase analyses were conducted by X-ray diffraction (XRD, X'Pert Pro, Philips, Netherlands). The  $\text{Fe}_3\text{O}_4$  distribution was analyzed with an energy dispersive X-ray spectrometer (EDS, Genesis XM2, EDAX, USA) during SEM analysis. The electromagnetic wave reflectivity was measured with a Naval Research Laboratory (NRL) testing system [16,17].

### 3. Results and discussion

During infiltration, as the mixed solution dripped slowly into ammonia water, more and more  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  ions in ammonia water react with  $\text{OH}^-$  to produce  $\text{Fe}_3\text{O}_4$  according to the following reaction equation.



As known from our previous work [15], there are lots of well-connected pores formed by bonding the rod-like  $\text{Si}_3\text{N}_4$  particles with each other in porous  $\text{Si}_3\text{N}_4$  ceramic. At the beginning of infiltration process, with the help of powerful magnet beneath the preform, the reaction-derived  $\text{Fe}_3\text{O}_4$  nanoparticles in ammonia water enter the preform along the well-connected pores and deposit in the pores gradually. As infiltration process goes on, the pores in the preform get smaller and smaller due to continuous deposition of  $\text{Fe}_3\text{O}_4$  nanoparticles. The  $\text{Fe}_3\text{O}_4$  nanoparticles could hardly arrive at deeper place in the preform but deposit in the pores near the upper side of the preform. Finally,  $\text{Fe}_3\text{O}_4$  nanoparticles could only deposit on the upper surface of the preform when the pores near the upper side of the preform are stuffed.

Fig. 2(a) and (b) show the microstructures at the upper and lower surfaces of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$  respectively. As predicted, the pores among  $\text{Si}_3\text{N}_4$  particles at the upper surface of Gradient-

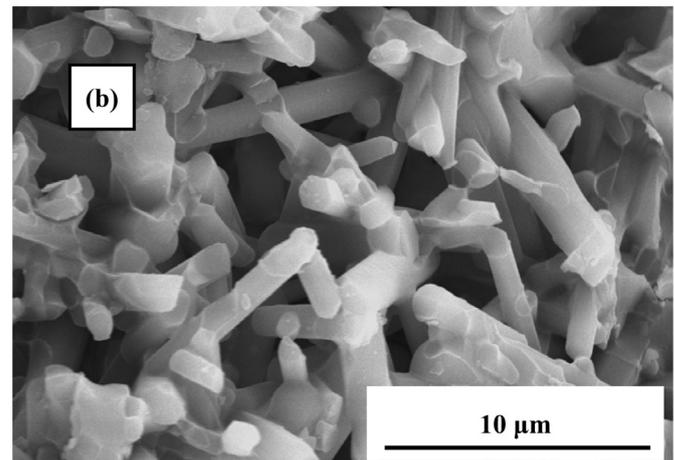
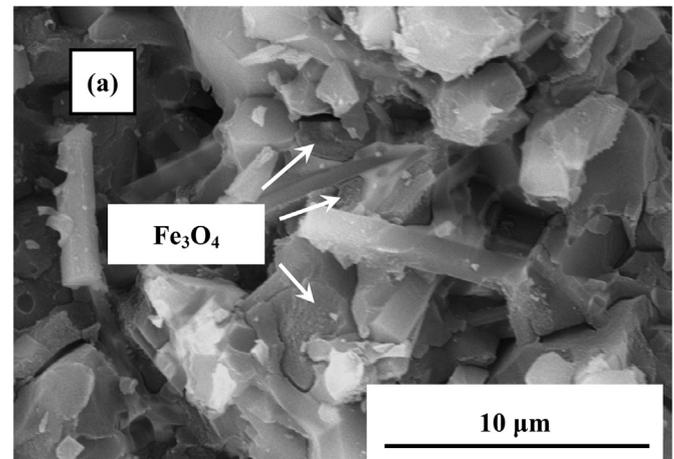


Fig. 2. Microstructures at the (a) upper and (b) lower surfaces of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$ .

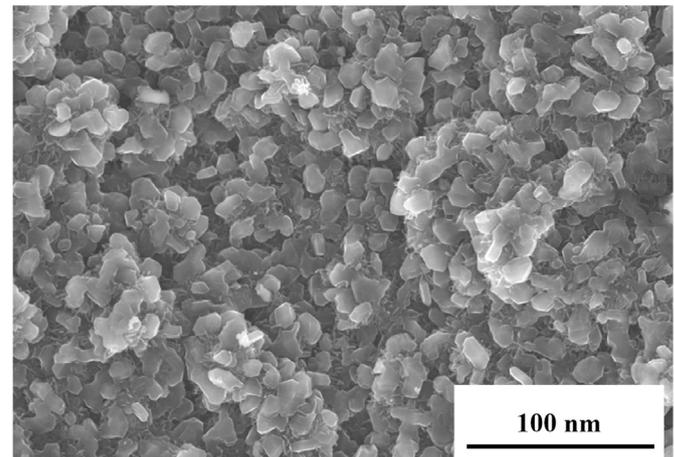


Fig. 3. High-magnification micrograph of the  $\text{Fe}_3\text{O}_4$  nanoparticles.

$\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$  are filled with  $\text{Fe}_3\text{O}_4$  nanoparticles, while the lower surface of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$  is still porous with no  $\text{Fe}_3\text{O}_4$  nanoparticles detected. Accordingly, it is inferred that there is a gradient distribution of  $\text{Fe}_3\text{O}_4$  in Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$ . In addition, Fig. 3 shows the high-magnification micrograph of the  $\text{Fe}_3\text{O}_4$  nanoparticles in the pores at the upper surface of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$ . The  $\text{Fe}_3\text{O}_4$  nanoparticles stack closely with each other and have uniform diameters of about 15–20 nm.

After infiltration, there is a number of  $\text{Fe}_3\text{O}_4$  nanoparticles deposited on the upper surface of Gradient- $\text{Si}_3\text{N}_4\text{-Fe}_3\text{O}_4$ . Fig. 4

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