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Original Research Paper

Formation phenomena of iron oxide-silica composite in microwave plasma and DC thermal plasma

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ABSTRACT

Iron oxide-silica composite was synthesized using atmospheric microwave plasma and DC thermal plasma. There has recently been increasing interest in predicting the final product during vapor phase synthesis using plasma because of difficulty obtaining desirable product. In this study, vapor phase synthesis of iron oxide-silica composite from iron pentacarbonyl ($\text{Fe}(\text{CO})_5$) and tetraethyl orthosilicate ($\text{SiC}_8\text{H}_{20}\text{O}_4$, TEOS) was conducted using various Fe/Si ratios and different types of plasma to identify the formation mechanism in the Fe-Si-O multi-component system. The morphologies and phase compositions of the synthesized particles were analyzed and compared. The results showed that the Fe/Si ratio and the type of plasma influenced the morphologies and the phase composition. A thermodynamic consideration was introduced to investigate the particle formation phenomena, which could explain the differences induced by varying the Fe/Si ratio and type of plasma. The particle formation mechanism was divided into a condensation step and a diffusion step. At the condensation step, the Fe/Si ratio determined the condensation temperature, which is related to the morphology. At the diffusion step, the quenching rate of the plasma determined the degree of diffusion, which was related to the phase composition and formation of the external layer.

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1. Introduction

Magnetic materials have been widely applied to magnetic fluids [1,2], data storage [3], biochemical applications (e.g., magnetic resonance imaging [4,5] and drug delivery [6]) and environmental remediation [7]. Among numerous magnetic materials, iron oxides are attracting considerable interest because Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$ exhibit superparamagnetic properties when they exist as particles of <100 nm; accordingly, the synthesis of those nanoparticles has been continuously attempted [8–16]. Moreover, biochemical applications of iron oxide have been actively investigated in recent years [17]. However, pure iron oxides cannot be applied because of large agglomeration, changes in magnetic properties and degradation in biological systems. Therefore, many researchers have investigated iron oxides coated with inert materials or iron oxide/inert material composites as potential candidates to solve those problems [18–20]. Coating iron oxide with inert material, especially silica, provides enhanced colloidal stability and

increased chemical resistance while maintaining the original magnetic property.

Various methods of preparing coated iron oxide particles have been investigated, including use of carbon/iron oxide [21–24], ceramic/iron oxide [25,26] and metal/iron oxide [27,28]. The preparation methods include liquid phase synthesis such as sol-gel, co-precipitation and controlled hydrolysis, in which additional processes must be conducted to remove organic compounds used in the preparation process that limit their applications. Nevertheless, it is difficult to completely remove the organic compounds adsorbed on the products. Accordingly, interest in employing vapor phase synthesis as a potential solution to this problem has grown [29–31]. As an alternative to liquid phase synthesis, vapor phase synthesis leads to high purity products without post-treatment.

Plasma processing is a well-known method of vapor phase synthesis used to prepare nanoparticles. Thermal plasma and microwave plasma are typical sources used for vapor phase synthesis. The high temperature of these plasmas (about 10,000 K and 3000 K in the highest temperature region, respectively) leads to vaporization or decomposition of raw material to the gaseous phase, after which ultrafine particles are obtained during

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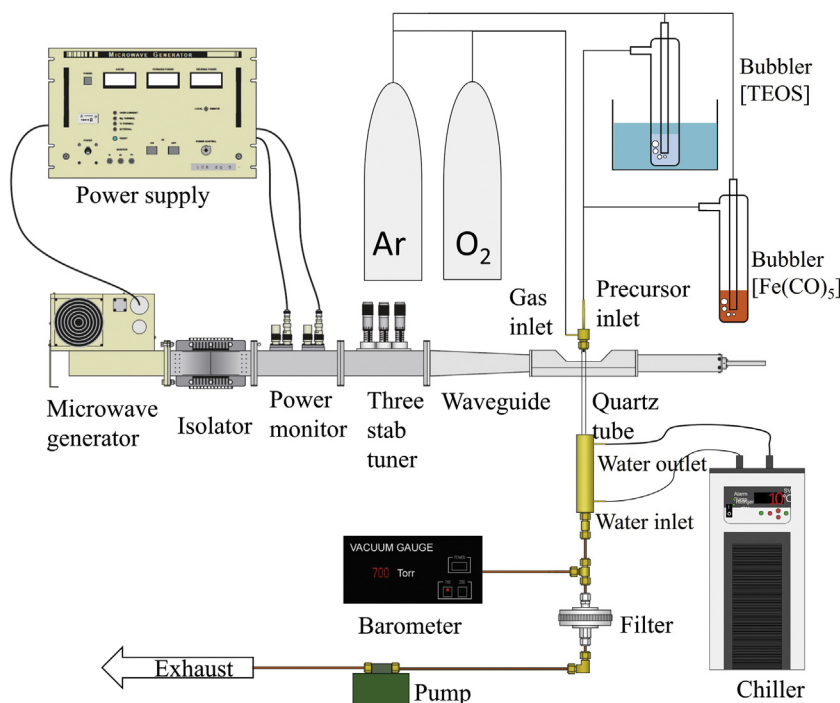


Fig. 1a. Experimental apparatus used for atmospheric microwave plasma.

Table 1
Antoine equation parameters of $\text{Fe}(\text{CO})_5$ and TEOS.

	Temperature (K)	A	B	C
$\text{Fe}(\text{CO})_5$	266.7–378	5.18943	1960.896	−0.228
TEOS	289–441.7	4.17312	1561.277	−67.572

Table 2
List of the experiments and the samples using the plasmas.

Experiment & Sample		Precursor	Temperature (K)	Carrier gas (Ar, ml/min)	Injection rate (ml/min)	Fe/Si
Microwave Plasma	MWP3	Fe(CO) ₅	298	300	9	3
		TEOS	323	300	3	
	MWP1	Fe(CO) ₅	298	200	6	1
		TEOS	323	600	6	
	MWP033	Fe(CO) ₅	298	100	3	0.33
		TEOS	333	500	9	
DC thermal Plasma	DCP3	Fe(CO) ₅	298	300	9	3
		TEOS	323	300	3	
	DCP1	Fe(CO) ₅	298	200	6	1
		TEOS	323	600	6	
	DCP033	Fe(CO) ₅	298	100	3	0.33
		TEOS	333	500	9	

simultaneous nucleation and quenching. Numerous studies have reported the synthesis of nanoparticles by those plasmas. Nevertheless, one of the main issues associated with vapor phase synthesis is difficulty in control of the phase composition in the particle. Recent studies have frequently dealt with multi-component particles such as doped ceramic, coated material and spinel (AB_2O_4) since their unique properties have attracted a great deal of interest in the application field [32–34]. When compared to single-component systems, multi-component systems lead to the formation of a greater variety of products by altering the experimental conditions (e.g., ratio of components, injection method). However, the particle generation phenomena have not yet been explained because of difficulties in predicting behaviors of various chemical

species in the high temperature region. To prepare desirable multi-composite particles from plasma processing, a better understanding of the multi-component systems in plasma is required.

This study had two primary purposes. The first was to investigate the particle formation mechanism in an Fe-Si-O multi-component system obtained from the decomposition of iron pentacarbonyl [$\text{Fe}(\text{CO})_5$] and tetraethyl orthosilicate [$(\text{C}_2\text{H}_5\text{O})_4\text{Si}$], known as TEOS]. The second was to identify the operation conditions such as the type of plasma and the Fe/Si ratio needed to synthesize desirable product. DC thermal plasma and atmospheric microwave plasma were used to synthesize iron oxide-silica composite and the results were compared. Finally, a particle formation mechanism was proposed based on comparison of the particle

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