



# Influence of the different organic chelating agents on the topography, physical properties and phase of SPPS-deposited spinel ferrite splats



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## ARTICLE INFO

### Article history:

Received 26 April 2013

Received in revised form 17 June 2013

Accepted 15 July 2013

Available online 24 July 2013

### Keywords:

Splat

Solution precursor plasma spray

Chelating agents

Cobalt ferrite

Topography

Phase composition

## ABSTRACT

Different types of organic chelating agents (polyvinyl alcohol, citric acid, and oxalic acid) were added during the preparation of cobalt ferrite precursor. The resulting cobalt ferrite solutions were then used in the solution precursor plasma spray (SPPS) process to deposit single splats onto mild steel substrates. It was revealed that the thermal oxidation of cobalt and iron salts into cobalt monoxide (CoO), maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), cobalt oxide (Co<sub>3</sub>O<sub>4</sub>), cobalt oxyhydroxide (CoOOH), goethite ( $\alpha$ -FeOOH), hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), magnetite (Fe<sub>3</sub>O<sub>4</sub>), and cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>) occurred largely within the deposited splats. The surface topographies of the splats exhibited unmolten, partially molten, and completely molten splats. The type of organic chelating agents affected significantly the splat thickness, percentage of cobalt ferrite phase, degree of splashing and equivalent diameter of the splats.

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## 1. Introduction

Cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>) is considered as an interesting metal-oxide material because of its unique magnetic properties [1]. In our previous study [2], cobalt ferrite nanoparticles were prepared using polyvinyl alcohol (PVA) and citric acid (CA) as two chelating agents, and these two types of resulting cobalt ferrite nanoparticles exhibited effective antibacterial activity against *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*). In the test of lysogeny broth (LB)-agar plates, the cobalt ferrite nanoparticles demonstrated surface-dependent antibacterial activities and, therefore, could have potential applications in drug delivery and other biomedical systems. However, studies of their biocompatibility and antibacterial properties are limited and more research is needed for an enhanced understanding.

### 1.1. Chelating agents

Chelating agents are used in inorganic chemistry to prevent particle agglomeration by reducing condensation reactions during liquid phase synthesis. They have been proven to control the cobalt ferrite phase in sol–gel chemical synthesis [3,4]. Chelating agents

can function as good selective flocculants because of their metal specificity and selectivity. From the synthesis point of view, chelating agents present certain advantages over conventional mineral processing in term of metal selectivity. There are many reagents that may be used with chelating agents in specific applications because of their varieties of major donor atoms which are sulphur, nitrogen, oxygen and phosphorus. Understanding the chemical behavior of these donors is essential to predict the properties of chelating groups. Normally, the choice of a chelating agent or a group is based on its function in analytical metal separations.

Most of the chelating groups form the necessary chemical complexes with almost all of the transition and many non-transition metals and, therefore, specificity is not as qualified as for selective adsorption on minerals. The selectivity is determined by consideration of differences in stability constants and chelate formation under various solution conditions. Moreover, the contribution from the donor atoms as they are located in the mineral lattice has to be considered in obtaining the selectivity. Also, the solubility of the mineral, in addition to that of the metal chelate, has a pronounced influence on the selectivity and the collection power of the chelating agent.

### 1.2. Thermal spray processing of ferrite coatings

Plasma spraying has been used for producing ferrite coatings since the 1970s [5] and has shown to be useful for fabricating planar

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inductors, magnetoresistance (MR) sensors, and thermistors. Liang et al. [6] used plasma spraying to produce cobalt ferrite coatings for magnetostrictive sensors, and found that the mixture of cobalt and iron oxides caused unstable saturation magnetostriction of the coatings and limited their applications. Moreover, it is challenging to feed powders with particle size less than 5–10  $\mu\text{m}$  into a conventional plasma spray system to obtain the nanostructured coatings due to the effect of surface forces on powder flowability [7].

However, solution precursor plasma spraying (SPPS), a versatile technique, allows coatings to be produced at a nanoscale with a wide spectrum of coating structures and architectures [8]. The SPPS technique uses molecularly mixed metal-ion liquid precursors of the associated metal oxides, rather than the spray grade powdered feedstock of specific particle size range that is usually used for the plasma spray process. It also offers advantages such as precise control over final thickness and morphology [9], and ability to deposit nanostructured coatings [10]. In addition, SPPS reduces health and safety concerns associated with the handling of nanoparticles and, therefore, has a positive impact on the environment during feedstock production [11].

The SPPS deposit is built up by successive impacts of molten droplets. The understanding of the topography, composition and phase of the initially deposited individual splats is important since these splats are fundamental elements and will ultimately determine the microstructure and properties of the coatings. It has been reported that the morphology of the splat, i.e., the 'brick' for the build-up of the deposit, depends on several factors such as particle temperature and velocity, and substrate roughness [12]. The study of the formation and phases of a particular splat is useful for qualitative analysis of the thermal spray coating, especially for gas sensor [13] and biomedical applications [14], as the different phases of deposited splats may result in different sensing ability and biocompatibility. Similarly, the magnetic performance of cobalt ferrite, cobalt oxides and their derivatives depends significantly on their phases [15]. However, no studies have been undertaken of the effect of different chelating agents in the solution precursor preparation process on the morphology and phase composition of the deposited splats.

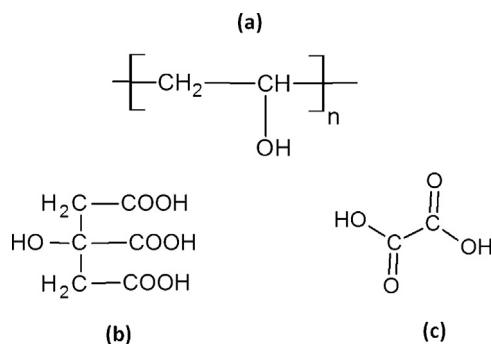


Fig. 1. The chemical structure of chelating agents: (a) poly vinyl alcohol, (b) citric acid and (c) oxalic acid.

### 1.3. Techniques to examine phases

A structure-sensitive and localized technique is required to characterize various cobalt ferrite phases that exhibit slightly different structures within a splat. Conventionally available techniques have their limitations. For example, X-ray diffraction can only detect phases within a relatively large volume, and cannot provide sufficient information when amorphous phases are the major components. Infrared (IR) spectrometry provides detailed information about molecular vibrations; however, it cannot provide information concerning wavelength shifts lower than  $400 \text{ cm}^{-1}$ . The Raman spectroscopy technique, on the other hand, can provide information on the short- and intermediate-range ordering in solids. Raman spectroscopy allows direct and nondestructive detection from the sample surface with spatial resolution on a micrometric scale that is 100 times greater than infrared resolution.

The present study deals with the SPPS deposition of cobalt ferrite solution precursor using water based solutions of cobalt nitrate and iron nitrate salts. Three types of chelating agents, citric acid (CA), poly vinyl alcohol (PVA) and oxalic acid (OA), as presented in Fig. 1, were used. The concentration of all chelating agents in the solution precursor was fixed for the entire experiment.

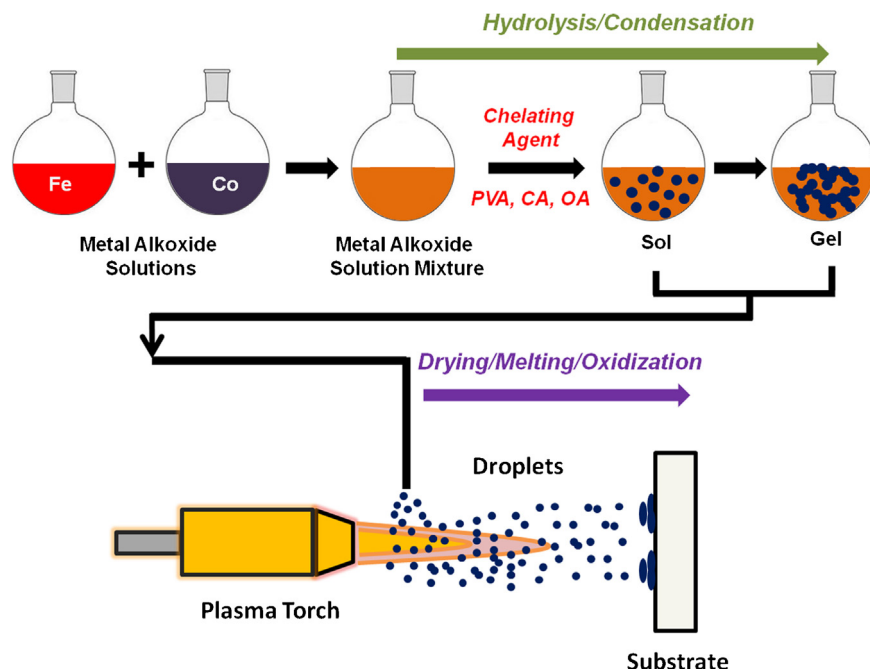


Fig. 2. Schematic of the solution precursor plasma spray delivery process.

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