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Preparation of zirconia-magnesia nanocomposite powders and coating by a sucrose mediated sol-gel method and investigation of its corrosion behavior

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ABSTRACT

The zirconia-magnesia nanocomposite powders and coating were prepared using a new sucrose- mediated sol-gel method. The effect of sucrose content to metal ions and calcination temperature on the morphologies and phase of the products were investigated. The samples were characterized by XRD, FTIR and FE-SEM analysis. The results showed that the cubic phase of ZrO_2-MgO with an average size of 30 nm was formed in the presence of sucrose as a gel agents. Finally, the corrosion behavior of optimized sample has been evaluated.

1. Introduction

Nanostructured composite materials have established better performance and are capable of meeting requirements of structural materials in wide applications. The nano-sized ZrO_2–MgO is one the most important categories of nanocomposite which have attracted a great deal of interest recently due to their properties such as excellent chemical and thermal stability, high porosity and large surface area and good corrosion barrier behavior [1–4]. These oxides have been used for widely industrial applications like sensors, microelectronic devices, refractory materials, biomaterials and fuel cells as well as catalyst properties [5,6]. Therefore, many challenges have been done for optimization of synthesis methods of these oxides. The most commonly used process for fabricating of MgO–ZrO_2 ceramics are sol-gel, mechanical alloying, co-participated and plasma-sprayed [4–15]. The reliability of sol–gel method for many practical applications are largely influenced by the intrinsic properties of this method, which it is controlled by the sol–gel processing parameters [16,17].

The eutectoid decomposition in ZrO_2-8 mol% and 11 mol% MgO were studied by Czeppe et al. [18]. They observed that the reaction products at the grain boundaries were monoclinic or tetragonal ZrO_2 phases and MgO precipitates of two different cellular morphologies and eutectoid decomposition in ZrO_2–MgO is controlled by the interface. Tian and coworkers [19] found that the Mg^{2+} doped into zirconia lattice could stabilize the tetragonal phase and prevent zirconia crystals from excessive growth. Das and coworkers [13], have shown that MgO particles occupy the inter-granular positions between ZrO_2 polycrystalline.

This work presented the synthesis of ZrO_2.MgO nanocomposites by a new sol–gel method using sucrose as both chelating and gel agent in different mole ratios. Then, the optimized sample has been selected for the investigation of corrosion behavior.

2. Materials and methods

2.1. Preparation of ZrO_2–MgO nanocomposites

The raw materials were prepared from Merck company and were used without further purification. The ZrO_2-MgO nanocomposites were prepared by various molar ratios of sucrose as both chelating and gel agents. The summary of selected parameter is reported in Table 1. The appropriate mole ratios of $Mg(NO_3)_2{\cdot}6H_2O$ (2 mmol) and $ZrOCl_2{\cdot}8H_2O$ (1 mmol) were dissolved in 100 ml of deionized water. Then, according to Table 1, various molar ratios of sucrose were separately added into the solution, and the mixture was vigorously stirred at 80–150 °C until highly viscous gels were formed. Finally, the resulting gel was calcined at 500–700 °C for 2 h. The optimized sample was selected and used for corrosion behavior. The procedure for the synthesis of ZrO_2-MgO nanocomposites has been illustrated in Fig. 1.

2.2. Preparation of ZrO_2–MgO nanocoating

At this stage, the sol of sucrose-Mg, Zr ions was created at 80 °C and then the steel electrode (316 L) was covered by dip coating method with three times. The electrodes are placed in the furnace and calcined at different temperatures to form a ZrO_2-MgO nanocoating.

The Polarization tests (TAFEL diagrams) were performed with a 3% solution of NaCl by potentiostat-galvanostats (AUTOLAB) instrument at −200 mV potential to +200 mV (open circuit potential (OCP)) with a scan rate of 1 mV/s.

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Table 1
summary of parameters design.

Sucrose to metal ions molar ratioes	$ZrOCl_2 \cdot 8H_2O$	$Mg(NO_3)_2 \cdot 6H_2O$	Sample
1:1	1	2	**A1**
2:1	1	2	**A2**
3:1	1	2	**A3**
4:1	1	2	**A4**

2.3. Materials characterization

The X-ray diffraction measurements were performed by a Phillip X′pert diffractometer (Model MPD-XPERT) w. A CuK_α beam with a wavelength of (λ) =1.54 Å was used as the radiation source. The diffractometer was operated at 40 kV and 30 mA using a scanning step of 0.030 in 2θ, and dwell time of 1 s was used. The FTIR spectra for all samples were carried out by using a Bruker TENSOR 27 infrared spectrophotometer. The field emission scanning electron microscope (FE-SEM; Model Mira3-XMU, TESCAN) were used for morphology evaluation of the samples. Before taking FESEM images, the samples were Au-coated with desk sputter coater (DST3 model, nanostructured coating Co., made in Iran).

3. Results and discussion

To determine the optimized calcination temperature for MgO-ZrO_2 nanocomposites, X-ray diffraction (XRD) analysis was performed on A4 samples calcined at 500–700 °C for 2 h. The typical XRD patterns shown in Fig. 2. According to Fig. 2a, at low calcination temperature (500 °C), the diffraction background of the sample was relatively high. With increasing calcination temperature from 500 °C to 700 °C, the crystallization of both MgO and ZrO_2 phases was completed, and no impurity peaks appeared in this temperature. It could be seen that the reflections of these nanocomposites were matched well with the JCPDS No. 75-0447 (MgO) and JCPDS No. 27-0997 (ZrO_2). The phase of MgO and zirconia was the Periclase and cubic form, respectively. The lattice

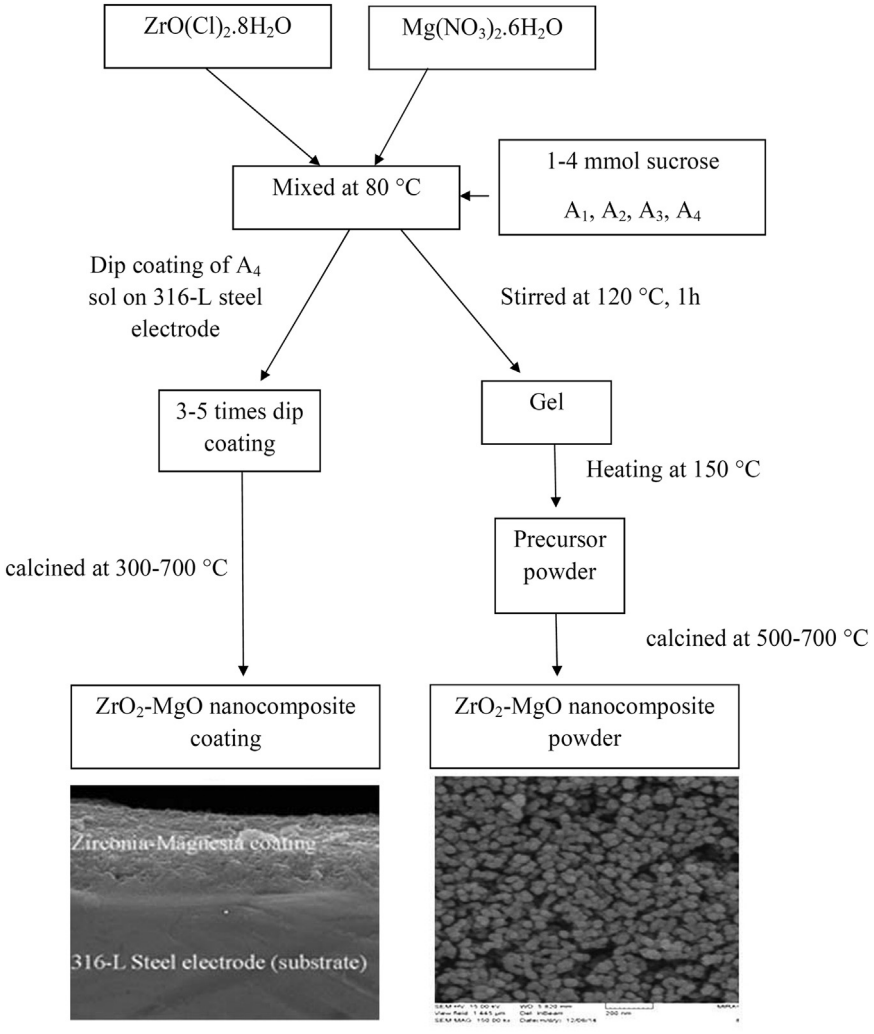

Fig. 1. The schematic for the synthesis of ZrO_2-MgO nanocomposite powders and coating.