



# Evolution of phase composition and fluorescence properties in zirconia degraded under hydrothermal conditions



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## ABSTRACT

In the present study, 8YSZ:Eu<sup>3+</sup> powder was synthesized using co-precipitation route. The evolution of phase composition and fluorescence properties of 8YSZ:Eu<sup>3+</sup> powder have been assessed after being subjected to hydrothermal degradation for different times. Morphology and microstructure of the sintered powder were observed by transmission electron microscope (TEM) and scanning electron microscope (SEM). XRD, Raman spectra, photoluminescence (PL) spectra and the decay curve were used to evaluate the phase composition variation and fluorescence properties variation of the powder. The results showed that 8YSZ:Eu<sup>3+</sup> powder had a pure tetragonal phase structure. Under hydrothermal degradation conditions, due to the reaction of OH<sup>-</sup> and oxygen vacancy, the transformation of tetragonal to monoclinic occurred, furthermore the fluorescence properties of the powder were altered.

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## 1. Introduction

Thermal barrier coatings (TBCs), known as thermally insulating ceramic coatings, are widely used to protect critical metallic parts (blades, vanes, seals, shrouds and combustor chambers) of aircraft engines as well as land-based gas turbines at high temperatures. They provide thermal insulation of the metallic substrates in addition to increasing operational temperatures and the efficiency of power generation plants and engines [1,2]. Typical TBCs system usually consists of three layers which are applied onto a substrate or base metal (BM): a MCrAlY (M represents Ni, Co or Ni and Co) bond coat as oxidation resistant layer, a porous and thermally insulating ceramic top coat, and the thermally grown oxide (TGO) [3,4]. Up to now, two main coating processes are used to deposit TBCs for industrial applications, namely the electron beam physical vapor deposition (EB-PVD) and the air plasma spray (APS), each generating specific layer morphology, deposit microstructure and thermo-physical properties.

Nowadays, the current state-of-the-art ceramic top coat material is 3.5–4 mol% Y<sub>2</sub>O<sub>3</sub> partially stabilized zirconia (YSZ). Compared with other ceramics, YSZ has excellent mechanical

properties, such as high strength and fracture toughness combined with good wear resistance and, above all, a thermal expansion coefficient close to that of metallic substrates [5,6].

Observations of coatings after service indicate that they can fail by cracking, delamination, and spallation, typically on cooling to room temperature [7,8]. A primary failure mechanism involves oxidation at the interface of ceramic layer and bond coat which creates TGO layer at the interface. At higher temperature, additional mechanisms, particularly porous coating sintering and chemical reaction between the ceramic top coat and bond coat result in the formation of cracks in the coating and higher thermal conductivity, and accelerate the spallation failure of TBCs [9,10]. On the other hand, stresses resulting from volumetric changes accompanying phase transformation of TBCs during service is another key factor in controlling the TBCs life [7].

Under the influence of humidity the material is susceptible to a spontaneous t–m phase transformation generally beginning on the surface due to the oxygen vacancy annihilation [11]. This phase transformation is of martensitic type and induces a volume expansion (approximately 3–5 vol.%), which can induce significant stresses and, possibly, the nucleation and the propagation of cracks within the TBCs, thus reducing its lifetime, eventually contribute to the failure of the TBCs system [12]. This transformation, though rarely seen in service parts, is considered undesirable since it can

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be accompanied by intergranular microcracking and result in mechanical degradation of the coating. To date, there has been no systematic research to study the evolution of the phase composition in the presence of a humid environment. Thus, it is necessary to know the phase composition evolution in YSZ coating or powder under humid environment, which is important for understanding the degradation and failure of TBCs.

Various methods have been employed to assess the failure of the TBCs. However, some methods, such as fracture mechanics approach, modified four-point bending test and indentation method would destroy the structure of the coatings. Some methods such as neutron diffraction, XRD, luminescence spectroscopy [13], curvature measurement [14] and Raman spectroscopy [15] as non-destructive diagnostic tools could reliably assess the damage state of TBCs and would alleviate the risk of TBCs premature failure. Among those, luminescence spectroscopy is a valid tool to detect the degradation and the thickness of the degraded layer.

Zhao et al. [16] prepared LZ<sub>7</sub>C<sub>3</sub>/8YSZ:Eu double-ceramic-layer TBCs. After thermal cycling test, 8YSZ:Eu sublayer exposed and produced visible luminescence under ultraviolet illumination. The result showed that application of a Eu<sup>3+</sup>-doped luminescence sublayer can be a simple and useful non-destructive technique to indicate the spallation and damage degree of DCL coatings. Zhao et al. [17] developed a non-destructive inspection technique to measure the residual stresses in TBCs by using Eu<sup>3+</sup> photoluminescence piezo-spectroscopy. The result showed that this method can successfully apply in detecting residual stress in plasma sprayed TBCs.

In recent years, nanostructured zirconia based TBCs deposited by atmospheric plasma spraying have been the focus of attention. It was reported that nanostructured thermal barrier coatings had high bonding strength, low thermal conductivity and prolonged thermal cycling lifetime [18]. Accordingly, nanostructured TBCs are expected to provide better performance than the conventional TBCs. Furthermore, nanostructured powders are expected to provide better performance than the bulk materials before deposited on the superalloy substrates. There are several chemical methods to synthesize nanostructured zirconia powders. The solution based synthesis methods are chemical co-precipitation, hydrothermal, sol-gel, microemulsion, and microwave. Among these, chemical co-precipitation is a low cost method for controlled synthesis of nanostructured zirconia powders.

In the current study, nanostructured 8YSZ doped with 1 mol% Eu<sup>3+</sup> was synthesized via chemical co-precipitation. Here we focused the attention on the evolution of phase composition and fluorescence properties after the powder had been exposed to accelerated aging by using hot water vapor in autoclave. XRD and Raman spectra were used to evaluate the phase composition of the powder. Photoluminescence (PL) spectra and the decay curve were used to investigate the fluorescence properties of the powder.

## 2. Experimental

### 2.1. Materials

Zirconium(IV) oxychloride octahydrate (ZrOCl<sub>2</sub>·8H<sub>2</sub>O ≥ 99.0%, Sinopharm Chemical Reagent Co., Ltd), yttrium nitrate hexahydrate (Y(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O ≥ 99.0%, Sinopharm Chemical Reagent Co., Ltd), europium (Eu<sub>2</sub>O<sub>3</sub>, 99.99%, Shanghai Yuelong New Materials Co., Ltd), nitric acid, polyethyleneglycol (PEG), ammonia were used as starting materials. All the chemicals were analytical reagent.

### 2.2. Synthesis and thermal treatment of the powder

8YSZ:Eu<sup>3+</sup> powder was synthesized using chemical co-precipitation method. The content of Y<sup>3+</sup> and Eu<sup>3+</sup> ions were 8 mol% and 1 mol% in the powder, respectively. Oxide powder (Eu<sub>2</sub>O<sub>3</sub>) was heat-treated at 1000 °C for 2 h in air because rare earth oxide is hygroscopic. Appropriate amount of Eu<sub>2</sub>O<sub>3</sub> was firstly dissolved in

dilute nitric acid and then mixed with a stoichiometric ZrOCl<sub>2</sub>·8H<sub>2</sub>O and Y(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O aqueous solution to give a final molar content of 0.08 mol with vigorous stirring. After 30 min of mixing, the solution was then mixed with 3.0 wt% of polyethylene glycol (PEG) as a dispersant because an appropriate amount of PEG can greatly decrease agglomeration. The obtained solution was stirred for 30 min to obtain a homogeneous solution. After homogenization, ammoniacal solution was added drop wise till the pH of the solution reached to 9. The agitation continued for 30 min after the reaction was completed. The precipitate was aged in the original liquid for 24 h and the resultant gelatinous precipitate was then washed and filtered thoroughly three times with a de-ionized water and additional two times with absolute ethanol to remove impurities. The obtained wet zirconium hydroxide was dried at 80 °C for 24 h and ground into powder.

The powder was subsequently thermal treated in an Al<sub>2</sub>O<sub>3</sub> boat at a temperature of 800 °C for 6 h with a heating rate of 5 °C/min.

### 2.3. Hydrothermal degradation test

Hydrothermal degradation treatments were carried out at 150 °C in an autoclave with dwelling times of 24, 48, 96, 192 and 384 h. The ratio of the 8YSZ:Eu<sup>3+</sup> powder and the H<sub>2</sub>O was 1:10 g/ml. After degradation to the designed time, the autoclave was taken out and cooled down in air to the room temperature. The powder was then filtered and dried at 80 °C for 24 h. Immediately after hydrothermal degradation treatment, the powder was examined by X-ray diffraction, Raman spectra, luminescence decay curve and fluorescence spectra to evaluate the relative amounts of the various phases and the variation of the fluorescence properties.

### 2.4. Characterization

Microstructure and particle size of the powder were observed by Hitachi S-4800 scanning electron microscope (SEM) with an accelerating voltage of 10 kV. To obtain better image resolution, a thin gold film was deposited onto the surfaces of the powder by radio frequency sputtering. The morphology of the powder was obtained using a TECNAI G<sup>2</sup> F-20 transmission electron microscope by employing an accelerating voltage of 200 kV. For recording TEM images first powder was ultrasonically dispersed in ethanol. A drop of this solution was placed on carbon-coated copper grid and dried at 50 °C.

The crystalline phase composition of all powders were determined by X-ray diffraction using a Bruker D8 Focus powder X-ray diffractometer with Cu K $\alpha$  radiation ( $\lambda = 0.1540598$  nm). The operation voltage and current were 40 kV and 40 mA. Region from 10° to 90° 2 $\theta$  range was recorded with a scan speed of 0.5 s/step and a scan step of 0.04°. High-resolution X-ray patterns in the 72° < 2 $\theta$  < 76° and 27° < 2 $\theta$  < 32° range were also recorded so as to identify the zirconia phases present. The proportion of the monoclinic and tetragonal or cubic phases was quantified by converting the X-ray intensity to volume fraction using the relationship in the following section. The pseudo-cubic description of tetragonal zirconia is used in this work, i.e. it is considered as a distorted cubic lattice, similar to previous studies on TBCs materials [12]. Therefore, the tetragonal peaks in the range of 72–76° are assigned the indices (004)<sub>t</sub> and (400)<sub>t</sub>. Raman spectra were collected through a Renishaw 2000 model confocal microscopy Raman spectrometer with a CCD detector and a holographic notch filter (Renishaw Ltd., Gloucestershire, UK) at ambient conditions, using an operating wavelength of 514.5 nm from an air-cooled argon ion laser to excite the SERS.

The photoluminescence (PL) measurements were performed on a Hitachi F-7000 spectrophotometer equipped with a 150 W xenon lamp as the excitation source. The spectral resolution is 1.0 nm. The luminescence decay curves were obtained from a Lecroy Wave Runner 6100 digital oscilloscope (1 GHz) using a tunable laser (pulse width = 4 ns, gate = 50 ns) as the excitation (Continuum Sunlite OPO) source. The data obtained in the experiments were processed using the software Origin 8.0.

## 3. Results and discussion

### 3.1. Morphology of as-prepared 8YSZ:Eu<sup>3+</sup> powder

The morphology, particle size and microstructure of the synthesized powder were characterized by SEM, TEM and HRTEM. Fig. 1 shows the SEM images of the as-prepared 8YSZ:Eu<sup>3+</sup> powder at 800 °C for 6 h. From Fig. 1, we can see that the 8YSZ:Eu<sup>3+</sup> powder has a nearly spherical or ellipsoidal morphology with the particle size of about 15 nm. Some agglomerates can also be observed in the powder, which may be caused by the uncontrolled coagulation during the process of precipitation and sintering. Since the powder was prepared through wet-chemical route, the agglomeration phenomenon is expected to take place [19].

Typical TEM images of 8YSZ:Eu<sup>3+</sup> powder annealed at 800 °C for 6 h are shown in Fig. 2. Fig. 2a and b shows the morphology of

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