

# Study on structural evolution of nanostructured 3 mol% yttria stabilized zirconia coatings during low temperature ageing

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## Abstract

In the present work, the nanostructured 3 mol% yttria stabilized zirconia coatings were deposited by plasma spraying, and its structural evolution during the low temperature ageing in wet atmosphere was investigated by Raman spectroscopy. The results showed that the nanostructured 3 mol% yttria stabilized zirconia coatings had lower resistance to low temperature ageing, although the nanostructured coatings have a metastable tetragonal-prime (*t'*) crystal structure. The degradation mechanism was explained in terms of the diffusion of oxygen vacancies and OH<sup>-</sup> ion and the reactions between OH<sup>-</sup> and Y'<sub>Zr</sub> ion. The microstructure of as-sprayed coating, especially the microcracks, plays a very important role in the low temperature degradation. It can enhance and accelerate the low temperature degradation. It was also verified by wavelength dispersive spectrometer (WDS) analysis that an yttrium-rich surface was formed due to the reaction between OH<sup>-</sup> and Y'<sub>Zr</sub> ion, which resulted in the transformation of *t'* to monoclinic zirconia phase.

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## 1. Introduction

Plasma sprayed yttria-doped zirconia coatings have been widely used for the thermal protection of gas turbine engines and marine diesels, and considerably improved the combustion efficiency of engines.<sup>1–3</sup> To improve the lifetime of plasma sprayed yttria-doped zirconia coatings, many studies have been conducted on high temperature ageing and it is believed that the lifetime of plasma sprayed zirconia coatings was closely related to the transformation of tetragonal to monoclinic phase during ageing.<sup>4–8</sup> In fact, when plasma sprayed zirconia coatings were used in engine interiors as thermal barrier coatings, a low temperature degradation may take place due to the presence of moisture which is unavoidable during the fuel combustion process.

Since Kobayashi et al.<sup>9</sup> first reported the low temperature degradation of 3Y-TZP bulk ceramic in wet environment, many researches have been conducted on this phenomenon. Among

the common features about low temperature degradation,<sup>9–13</sup> the grain size was regarded as one of the key factors to improve the resistance to the low temperature degradation in wet atmosphere. It had been found that zirconia bulk ceramic with a smaller grain size exhibited a better resistance to low temperature degradation.

In addition, plasma sprayed zirconia coatings have a metastable tetragonal-prime (*t'*) crystal structure, and the *t'* phase shows high resistance to stress-induced transformation to the monoclinic phase.<sup>14–19</sup> If plasma sprayed zirconia coatings had the *t'* phase crystal structure and its grain size was less than the critical grain size of tetragonal phase, e.g., 0.3 μm for 3Y-TZP reported by Schmauder and Schubert,<sup>20</sup> the plasma sprayed zirconia coatings should have the best resistance to low temperature degradation. Our previous works demonstrated that nanostructured 3 mol% yttria stabilized zirconia coatings can be deposited by plasma spraying technology,<sup>21,22</sup> and the nanostructured coatings had the metastable tetragonal-prime (*t'*) crystal structure. Its average grain size was less than 100 nm. However, few reports on low temperature degradation of plasma sprayed nanostructured zirconia coatings can be found. So, the objective of this work is to investigate the low temperature degradation of plasma sprayed nanostructured zirconia coatings.

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Table 1  
Plasma spray parameters.

Parameters	
Ar <sub>2</sub> (slpm)	35
H <sub>2</sub> (slpm)	12
Spraying distance (mm)	120
Gun current (A)	620
Power (kW)	42
Carrier air (slpm)	3.5

In the present work, nanostructured 3 mol% yttria stabilized zirconia coatings were deposited by atmospheric plasma spraying. The structural evolutions of as-sprayed coatings during low temperature ageing in wet atmosphere were examined by Raman spectroscopy. In addition, the corresponding wavelength dispersive spectrometer (WDS) analysis was also carried out.

## 2. Samples and experimental procedures

Commercial nano-sized zirconia powders (3 mol% Y<sub>2</sub>O<sub>3</sub>) with grain sizes ranging from 30 to 80 nm (Fanmeiya Corp., Jiujiang, China) were used as the starting particles and reconstituted into micrometer-sized granules by spray drying process.

Three nanostructured coatings with a thickness of about  $2 \times 10^{-3}$  m were deposited on an aluminium alloy substrate using a Metco A-2000 atmospheric plasma spraying equipment (Sulzer Metco AG F4 gun, Switzerland). A mixture of argon and hydrogen was used as the plasma forming gas. Compressed air was used as cooling gas during plasma spraying. The optimum plasma spray parameters were listed in Table 1. After spraying, the as-sprayed coating was removed from the aluminium alloy substrate, then cut into 10 mm × 10 mm × 2 mm specimen using a SYJ-150A low speed diamond wheel saw cutoff machine (MTI Corporation, USA).

Two different ageing experiments were designed to investigate the low temperature ageing. One was carried out in the water bath (CU600, Zhisun, Shanghai) at 40, 60, 80 °C for 120 h and 100 °C for 24 h. The other was in water vapor at the 0.133 Pa pressure of water vapor. In the latter, the specimens were firstly put into an evacuated sealed silica tube, which was positioned in the tubular furnace, then aged in a steam of humid air with a flow rate of 10 mL/min at 250, 450, 650 °C for 30 h. The schematic illustration of the water vapor experiment apparatus was shown in Fig. 1.

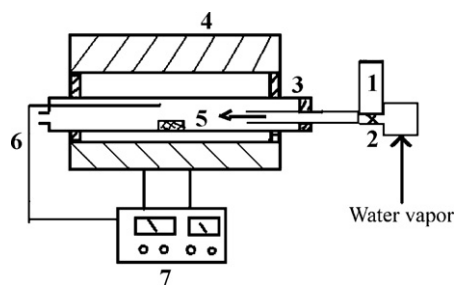


Fig. 1. Schematic diagram of experimental apparatus. 1: water vapor flowmeter; 2: needle valve; 3: quartz tube; 4: furnace; 5: specimen; 6: thermocouple; 7: temperature controller.

The phase evolution of aged coatings was identified by a LabRam-1B micro-Raman spectrometer (Dilor, France). For Raman spectrum analysis, the radiation of the 632.8 cm<sup>-1</sup> line from a He–Ne laser was used as the excitation source and with 6.4 mW incident power and 100 s for each specimen. The main reason for using Raman spectroscopy in this study is that the tetragonal and monoclinic polymorphs of zirconia are reported to have distinct and characteristic Raman spectra.<sup>23–25</sup> A small amount of tetragonal or monoclinic phase could be detected in a polycrystalline zirconia material by its distinguishing lines. The monoclinic doublet (at 181 and 192 cm<sup>-1</sup>) and the tetragonal bands (around 148 and 264 cm<sup>-1</sup>) are well separated over the range 100–1000 cm<sup>-1</sup>.

The relative fraction of the monoclinic phase,  $f_m$ , was calculated based on the Raman intensities of monoclinic doublet (at 181 and 192 cm<sup>-1</sup>) and the tetragonal bands (at 148 and 264 cm<sup>-1</sup>) using the relation

$$f_m = \frac{I_m^{181} + I_m^{192}}{k(I_t^{148} + I_t^{264}) + I_m^{181} + I_m^{192}}$$

where the value of  $k$  was taken to be 0.97.<sup>26,27</sup> The superscripts refer to the Raman shift of the characteristic peaks; the subscripts,  $t$  and  $m$ , refer to the tetragonal and monoclinic phase, respectively.

The surface morphology and cross-section area of the nanostructured zirconia coatings were examined by field emission scanning electron microscope (FESEM, JSM-6700F, JEOL, Japan) and scanning electronic microscopy (SEM, EPMA-8705QH, Shimadzu, Japan). The concentration of yttrium in specimens after ageing in water at 100 °C for 24 h was determined by wavelength dispersive spectrometer (WDS, JXA-8100, JEOL, Japan).

## 3. Results and discussion

### 3.1. Characterization of nanostructured zirconia coatings

Fig. 2 presents the Raman spectra of the as-sprayed nanostructured zirconia coating. It can be seen that four Raman lines occurred at 153.7, 261.1, 472.8 and 642.8 cm<sup>-1</sup> could be

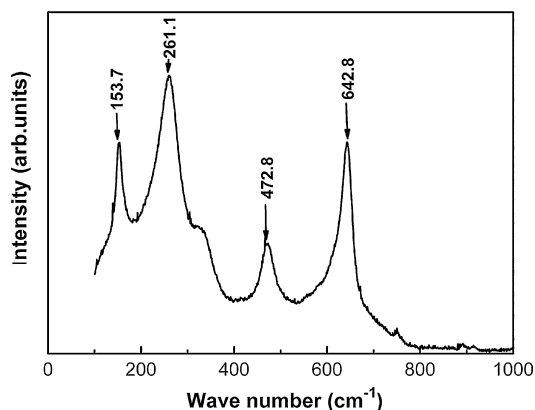


Fig. 2. The Raman spectrum of nanostructured 3 mol% yttria stabilized zirconia coatings.

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