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# Thermal deformation of Y<sub>2</sub>O<sub>3</sub> partially stabilized ZrO<sub>2</sub> coatings by digital image correlation method

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#### ABSTRACT

A contact-free method based on the digital image correlation (DIC) technology has been established to measure the in-plane thermal deformation of yttria stabilized zirconia (YSZ) bulk and YSZ free-standing coatings prepared by electron beam physical vapor deposition (EB-PVD). The results show a superb test agreement between the DIC method and the mechanical dilatometer. Moreover, thermal strain of the sintered YSZ bulk as well as the coatings caused by phase transformation of monoclinic-tetragonal phase after 15 h thermal exposure at 1450 °C are determined by the DIC method. The volumetric changes induced by the phase transformation of YSZ coatings are measured during heating and cooling, which are helpful for understanding the stress state and lifetime prediction of thermal barrier coating (TBC) system.

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#### 1. Introduction

8 wt.% yttria stabilized zirconia (YSZ) is the most commonly used thermal insulation material in Thermal Barrier Coatings (TBCs) due to its higher melting point, lower thermal conductivity and excellent mechanical properties [1–4]. The dominating phases in zirconia are tetragonal phase, monoclinic phase and cubic phase. The dopant yttria stabilizes zirconia in its cubic or tetragonal form and suppresses the tetragonal to monoclinic destructive phase transformation [5]. However, full stabilization to the cubic phase can compromise the cyclic thermal fatigue life [6], which is not favorable. Lower dopant concentration stabilizes the tetragonal phase which coexists with tetragonal metastable phases at high temperatures [7]. While cooling, these tetragonal metastable phases transform into monoclinic phase, which leaves behind a volumetric change [8] resulting in mechanical instability of the material. Because of the characteristics of martensitic transformation [9], this phase transformation could be induced by stress field, which is the so called transformation toughening [8]. Similarly, a reverse stress field could restrict the phase transformation and result in the incomplete volumetric change. Therefore, a good knowledge about the practical phase transformation induced by volumetric changes in service condition is essential for the assessment of the stress evolution and lifetime prediction of the TBCs.

The commonly used method for the thermal deformation measuring is the mechanical dilatometer [10,11] with a long push rod to detect the sample displacement during heating or cooling. However, it is not suitable for materials which are extremely fragile and therefore incapable of actuating the comparatively heavy rod. The YSZ layers of TBCs are normally formed by electron beam physical vapor deposition (EB-PVD) or plasma spray to the thickness about 100  $\mu$ m [1]. Their in-plane deformation therefore clearly cannot be detected by mechanical dilatometer.

Recently, a novel non-contacted, image based, method for micro-deformation measurement called digital image correlation (DIC) technology has been well developed [12–14], which is considered as an appropriate method to study the deformation of 2D materials [15–17]. The application of DIC on the YSZ and TBC measurement, however, has not been well developed. To the best of our knowledge, it is always used for the YSZ coating crack propagation detection [18,19], the bond coat thermal expansion coefficient measurement [11] as well as the deflection test of TBCs during bending and consequently deriving the apparent elastic modulus [19–21]. Seldom report has been found about the study on the thermal deformation of YSZ coating by using DIC technology, especially the deformation induced by phase change.

In this study, a setup and corresponding method were presented based on the DIC technology for the thermal deformation measurement. And the thermal strain of cold-pressing sintered YSZ bulk and free standing EB-PVD YSZ coating during heating and cooling from room temperature (RT) to 1100 °C are obtained by the proposed method. Finally, the strain variation caused by phase change was derived, which represented the practical volumetric variation amount during the incomplete transformation between the tetragonal and monoclinic phases.

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#### 2. Experimental

#### 2.1. Experimental setup

For the image based thermal deformation test by DIC, the thermal loading as well as the real-time observation and photographing are required. The high temperature metallographic microscope (Leica DM4000M microscope, Germany, and Linkam TS1500 heating stage, England) is therefore used as shown in Fig. 1. In order to make compatible for the high temperature observation through the window of the heating stage, a long working distance lens is installed. And a 6.6 megapixel digital camera is equipped on the microscope to capture the high resolution image for correlation calculations.

For the comparison and assessment of the DIC test results, the thermal deformation of the YSZ bulk is also measured by mechanical dilatometer (NETZSCH DIL402C, Germany). The YSZ phase transformations during cooling and heating procedures are determined by Differential Scanning Calorimetry (DSC, NETZSCH STA449C, Germany). And the YSZ phase contents are measured at room temperature (RT) by X-ray Diffraction (XRD, Rigaku D/max 2200PC, Japan) with CuK $\alpha$  radiation.

#### 2.2. Specimen preparation

The YSZ bulk is prepared by cold-pressing and sintering method. The yttria (99.99%) and zirconia (99.99%) oxide powders (wt.:wt.= 8:92) are mechanically mixed by ball milling method. Then the mixed powder is glued by wax-oil-polyethylene binder and pressed at RT with a pressure of 21 MPa to form the cold pressed bulk. After heat treatment at 1500 °C for 12 h in air, the cold pressed bulk eventually becomes sintered. The free standing YSZ coatings are detached from TBCs, which are deposited by EB-PVD, by dissolving the substrate and bond coat in the mixture of  $H_2O_2$  and HCl (V:V = 1:2). Then the coatings are subjected to the heating exposure at 1450 °C for 5 h, 10 h, 15 h, 20 h and 25 h respectively. Finally, due to the size limitation of the furnace heating stage, the sintered bulk and detached coatings are cut into sizes of 2 mm×2 mm×1 mm and 2 mm×2 mm respectively. The surface morphologies of the YSZ bulk and coatings are shown in Figs. 2 and 3 respectively. The native patterns or spots on the sample surfaces provide sufficient surface features for DIC measurements. Even at higher temperature, these features still keep stable, which is very benefical to an accurate measurement.

#### 2.3. Thermal deformation test by DIC

Digital images, taken before and after the deformation of an object, represent the positions of the object at these two moments of time. To retrieve the displacements at a certain region of interest on the object, a small subset surrounding this point in the reference image is selected to match the similar subset area in the target image. This process is called digital image correlation. To quantify the degree of correlation, various correlation coefficients have been introduced, such as cross correlation coefficient [22] and its various derivatives [23]. Digital image correlation procedures are always time-consuming because considerable calculations need to be carried out to obtain the displacement value through searching either the maximum or minimum value of the correlation coefficient. Considering the time consumption and calculation accuracy, a normalized covariance correlation coefficient [12] is undertaken in this paper, which is defined as:

$$C = \frac{\sum_{i=1}^{m} \sum_{j=1}^{n} \left[ f(x_{i}, y_{j}) - \bar{f} \right] \left[ g(x_{i}^{*}, y_{j}^{*}) - \bar{g} \right]}{\sqrt{\sum_{i=1}^{m} \sum_{j=1}^{n} \left[ f(x_{i}, y_{j}) - \bar{f} \right]^{2}} \sqrt{\sum_{i=1}^{m} \sum_{j=1}^{n} \left[ g(x_{i}^{*}, y_{j}^{*}) - \bar{g} \right]^{2}}}$$
(1)

where,  $(x_i,y_j)$  and  $(x_i^*y_j^*)$  are the positions of the calculation points in the reference and target images, respectively.  $f(x_i,y_j)$ ,  $g(x_i^*y_j^*)$ ,  $\bar{f}$ , and  $\bar{g}$  are the gray values of each pixel and the mean gray values of whole pixels in the reference and target images, respectively. In the present work, the digital image correlation process is implemented by Matlab<sup>TM</sup>. And correlation subset size of  $41 \times 41$  pixels is used. The magnification of the microscope used is  $\times$  100. Every one pixel in the captured images represents the length of 0.58  $\mu$ m. The noise floor of the DIC test setup is plotted in Fig. 4, which shows a mean squared error of 0.36 pixels for the displacement measurement. When the size of the interesting region for correlation calculation is set to  $1024 \times 768$  pixels, the resolution of the average strain in the whole field is 2e-4, which is adequate to the thermal strain ( $\sim$ 10 $^{-3}$ ) measuring of YSZ.

The thermal strain test procedure is shown in Fig. 5. The reference image is firstly taken at RT. Then the object images, which record



Fig. 1. High temperature metallographic microscope.

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