

# Measurement of residual stress in air plasma-sprayed $Y_2O_3$ – $ZrO_2$ thermal barrier coating system using micro-Raman spectroscopy

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## Abstract

Residual stress in air plasma-sprayed  $Y_2O_3$  stabilized  $ZrO_2$  thermal barrier coating (TBC) has been measured using micro-Raman spectroscopy. The frequency shift of Raman peak from tetragonal phase ( $\approx 640\text{ cm}^{-1}$ ) is used for determination of local stress in TBC. The relation between Raman peak shift and applied uniaxial stress is measured using a freestanding specimen. The relation is used to determine actual residual stress of the TBC layer coated on a stainless steel substrate. The uniaxial stress,  $\bar{\sigma}_{tbc}^U$ , and Raman peak shift,  $\Delta\nu$ , on the freestanding TBC layer follows:  $\Delta\nu = \bar{I}\bar{\sigma}_{tbc}^U$  ( $\bar{I} = 25\text{ cm}^{-1}\text{ GPa}^{-1}$ ). The average in-plane residual stress in this layer is smaller than that obtained from thermal stress, although the trend of measured values versus thickness follows the estimated thermal stress-substrate thickness relation. This experimental evidence suggests that measurement by micro-Raman spectroscopy is an effective tool to determine local and average residual stress in the  $Y_2O_3$  stabilized  $ZrO_2$  TBC layer.

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## 1. Introduction

Air plasma-sprayed thermal barrier coatings (APS–TBCs) have been used in various industrial components where a heat shield is required [1–3]. The performance and lifetime of APS–TBCs are strongly affected by the condition of residual stress stored in TBC layers because these stresses lead to cracking of the coating layer, shape change and spallation during service. Therefore, the measurement of residual stress in a TBC layer is an important research subject. Such measurement as, X-ray diffraction [4,5], neutron diffraction [6,7], curvature measurement [8,9] and substrate removal and strain measurement technique [9,10] are useful engineering methods and are already being used for various kinds of TBCs [4–10]. The disadvantage of these techniques is the difficulty in measuring the local residual stress.

Micro-Raman spectroscopy has been applied to measure residual stress in  $ZrO_2$  [9] and also residual stress of  $Y_2O_3$  sta-

bilized  $ZrO_2$  TBC [11,12]. The spectroscopic technique usually uses a few micron-meter order laser beam as an excitation source and the method provides local stress information. Therefore, it is possible to measure stress distribution, although the measured area is limited to near the surface.

However, measurement of the residual stress in a TBC layer by Raman spectroscopy has not been examined to detail and further research is needed. The present study focused on the measurement of residual stress in an air plasma-sprayed  $Y_2O_3$  stabilized  $ZrO_2$  TBC layer: (i) determination of stress-Raman shift relation and (ii) change of residual stress in the TBC layer with a thickness using obtained stress-Raman shift relation. Based on these results, discussion is made on the residual stress of APS–TBCs is discussed.

## 2. Experimental procedure

### 2.1. Coating material and microstructure

Air plasma-sprayed 8 wt%  $Y_2O_3$ – $ZrO_2$  TBC (hereafter termed 8Y– $ZrO_2$ ) coating on SUS-304 stainless steel was used,

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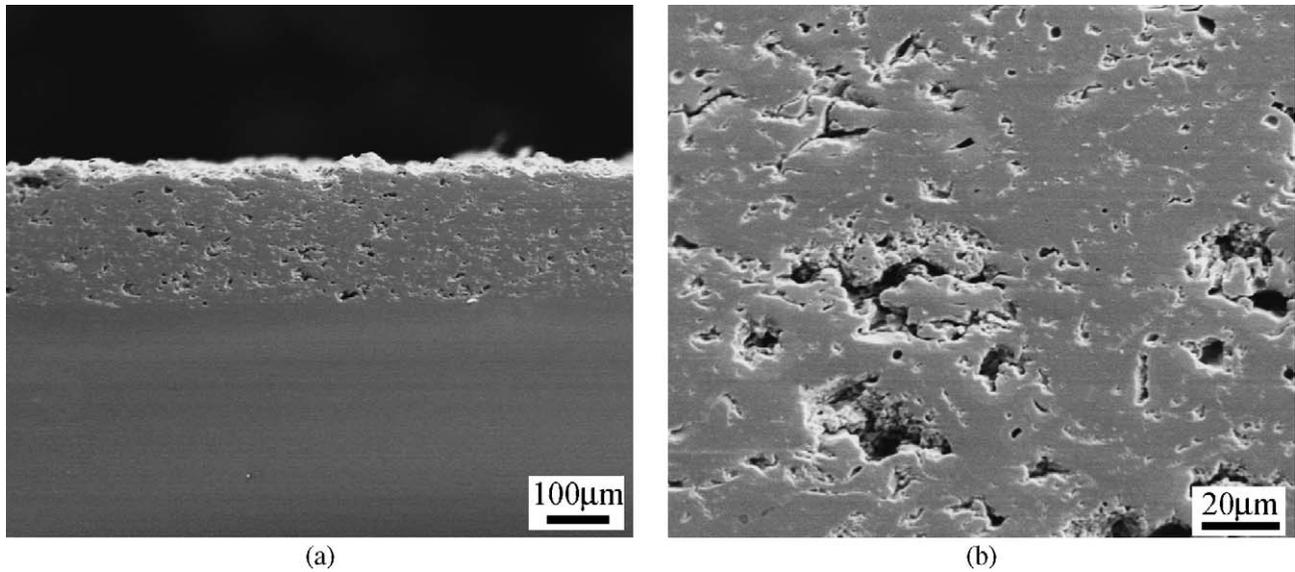


Fig. 1. A typical example of polished transverse section of a TBC layer: (a) entire view and (b) TBC layer.

which contained 16 wt% Cr, 8.5 wt% Co, 3.5 wt% Al and 3.5 wt% Ti and the remainder Fe. The coated material was obtained from Toshiba Corp., Tokyo, Japan. Thickness of the freestanding  $8\text{Y-ZrO}_2$  TBC layer and it coated on a stainless substrate, respectively, were  $\approx 500$  and  $\approx 300$   $\mu\text{m}$ . Fig. 1 shows a typical polished transverse section of the layer observed by scanning electron microscope (SEM); it contains many irregularly shaped pores and inter-splat boundaries. Detailed observation of the polished in-plane section shows that the pore shape and size are completely random (Fig. 1(b)). The entire volume fraction of pore,  $f_p$ , measured by image analysis was  $f_p \approx 0.22$ .

To identify the crystalline structure of the TBC layer, X-ray diffraction analysis was done on the coated surface. The analysis was performed by an X-ray diffractometer (RINT2000, Rigaku Co. Ltd., Tokyo, Japan) using  $\text{Cu-K}\alpha$  radiation under an acceleration voltage of 50 kV and a current of 300 mA. Fig. 2 shows the X-ray diffraction profile of the layer with well identified

peaks, and indicates that this layer consists of tetragonal and cubic phase  $\text{ZrO}_2$ .

## 2.2. Raman spectroscopy for local stress measurement in TBC layer

Raman spectroscopy was done using two kinds of specimens: (i) a freestanding TBC layer and (ii) a TBC layer coated on a stainless substrate. A freestanding specimen was used to measure the relationship between compressive stress and Raman peak shift of the layer. To obtain the freestanding TBC layer, a coated material was dipped into 20 wt% ferric chloride solution (300 K) and slowly etched with stainless substrate. Before dipping, the surface of coated specimen was slightly polished to obtain a section parallel to the freestanding specimen. The extracted TBC layer was carefully cleaned with distilled water and completely blown dry with air. Then the specimen was cut and slowly polished into a rectangular shape with dimensions of 2.0 mm  $\times$  2.0 mm  $\times$  0.5 mm thick. The final polishing of specimen surfaces was done with 1  $\mu\text{m}$  diamond paste finish.

The freestanding TBC specimen was attached to a specially designed homemade loading device, which was then placed on the Raman measuring equipment and Raman spectroscopic analysis was carried out under a stepwise loading condition. Care was taken to align the loading axis and avoid buckling of the freestanding specimen. Fig. 3 shows a schematic drawing of the loading set-up and diagram of the spectroscopic system for the measurement of a Raman spectrum (NRS-1000: Special Version, JASCO Co., Tokyo, Japan) for a freestanding TBC layer spectrum under applied loading. Prior to measurement, the specimen was loaded at a rate of 30  $\mu\text{m}/\text{min}$  to a set point load; the time required for measurement of the spectrum was 15 s. The effect of this holding time on stress–strain relaxation in the specimen could be neglected for the stress–strain response of the TBC layer.

Raman spectroscopic measurements were made at a fully controlled temperature ( $297 \pm 1$  K) in ambient air. A green laser

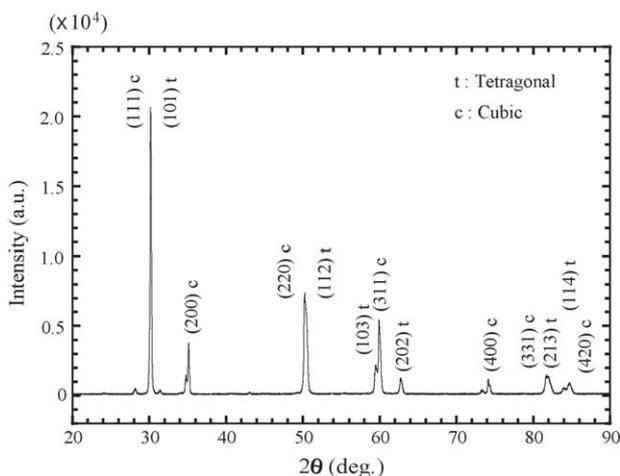


Fig. 2. X-ray diffraction profile of the TBC layer.

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