



## Regular article

# Microstructural degradation of Electron Beam-Physical Vapour Deposition Thermal Barrier Coating during thermal cycling tracked by X-ray micro-computed tomography

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## ABSTRACT

The degradation of an Electron Beam-Physical Vapour Deposition Thermal Barrier Coating caused by thermal cycling at 1150 °C has been followed in 3D non-destructively by time-lapse X-ray micro-computed tomography ( $\mu$ -CT). Quantitative analysis of X-ray  $\mu$ -CT virtual cross-sections on small samples is validated by destructive cross-sectional scanning electron microscopy (SEM) micrographs of larger ones. The evolution of thermally-grown oxide (TGO) is quantified. The TGO/bond coat interface roughness is measured in 3D. No significant rumpling is observed. Undulations are found locally at the interface of the as-deposited sample. Such undulations can increase in amplitude during cycling providing locations for interfacial cracks to initiate.

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The application of thermal barrier coatings (TBCs) to single crystal nickel superalloys has enabled significant increases in the operating temperatures of turbine blades [1], thereby improving the efficiency of turbine engines. A typical TBC system generally consists of four layers: the superalloy substrate; the metallic bond coat; the thermally grown oxide (TGO) and the ceramic topcoat [2]. The lifetime of this multi-layer coating system is limited by coating spallation [3], to which various mechanisms contribute depending on both the coating materials and the loading conditions. Oxidation of the bond coat occurs at peak operating conditions (bond coat temperature >700 °C [2]), leading to the development of a thermally grown oxide (TGO). In addition, stress is developed due to mismatch in thermal expansion coefficients of the constituent layers [4]. For an Electron Beam-Physical Vapour Deposited (EB-PVD) TBC with a  $\beta$ -(Ni, Pt)Al bond coat, TGO rumpling under thermal cycling has been recognised as one of the primary mechanisms for interfacial delamination [5–10]. An investigation of TGO growth and the evolution of the interface under conditions representative of those in service will shed more light on the failure mechanisms of TBCs.

Most of the microstructural information collected to date regarding TBCs has been acquired using destructive cross-sectioning. This has two consequences. One is that specific features cannot be tracked as a

function of thermal cycling since different samples are examined after different numbers of cycles. Another is that crucial features, such as interface cracks, can be introduced accidentally during sample preparation. As a non-destructive characterization method, X-ray  $\mu$ -CT has been used for microstructure investigation over multiple length scales [11,12]. Zhao et al. were the first to use time-lapse  $\mu$ -CT to follow the evolution of TGO formation and damage as a function of thermal exposure [13]. Ahmadian et al. revealed the 3D cracking characteristics in cycled air-plasma sprayed (APS) TBCs [14]. Subsequently, the displacement in a Pt-diffused  $\gamma/\gamma'$  bond coat has been mapped under cyclic exposure at 1200 °C using synchrotron X-ray  $\mu$ -CT and digital volume correlation (DVC) [15]. Recently, the residual stress distribution in an APS TBC was determined using finite element (FE) models developed from the 3D micrographs obtained by X-ray  $\mu$ -CT [16].

Here we employ time-lapse X-ray  $\mu$ -CT to track the evolution of the TGO and the TGO/bond coat interface in an EB-PVD TBC deposited on a  $\beta$ -Ni(Pt)Al bond coat during thermal cycling. After validation of the small sample geometry by comparison with destructive SEM images, our aim has been to use time-lapse X-ray  $\mu$ -CT to follow and quantify TGO growth and the development of interfacial roughness as a function of thermal exposure and to relate these to the features in the as-deposited sample.

The test coupons were cut from a pristine turbine blade comprising an EB-PVD yttria stabilised zirconia (YSZ) topcoat, a  $\beta$ -Ni(Pt)Al bond coat and a CMSX-4 single-crystal superalloy substrate. The coupon was then ground to a rectangular matchstick with a cross-section

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diagonal length of  $\sim 500 \mu\text{m}$  and a length of  $\sim 10 \text{ mm}$ . This ensured that the sample was fully accommodated within the field of view (FOV) of the X-ray  $\mu\text{-CT}$  system.

The as-deposited sample was exposed to thermal cycling using a cycling furnace (Model 1608 BL, CM Furnace Inc., UK.). Each thermal cycle comprised a 5 min heating ramp to  $1150^\circ\text{C}$ , a 1 h dwell followed by forced air-cooling to ambient in 5 min. The same region of interest (RoI) in the coupon was examined by X-ray  $\mu\text{-CT}$  after 0,

10, 50, 100 and 300 thermal cycles. In the meantime, in order to validate the X-ray  $\mu\text{-CT}$  results on this small matchstick sample, a larger reference coupon ( $\sim 3 \text{ mm}$  in cross-section diagonal and  $\sim 10 \text{ mm}$  in length) was cut from the same blade and subjected to the same thermal cycling regime. This coupon was used for cross-sectional SEM investigation.

X-ray  $\mu\text{-CT}$  was carried out using a Zeiss® Versa 500 3D X-ray microscope. The X-ray tube was operated at  $90 \text{ kV}$  with a beam current

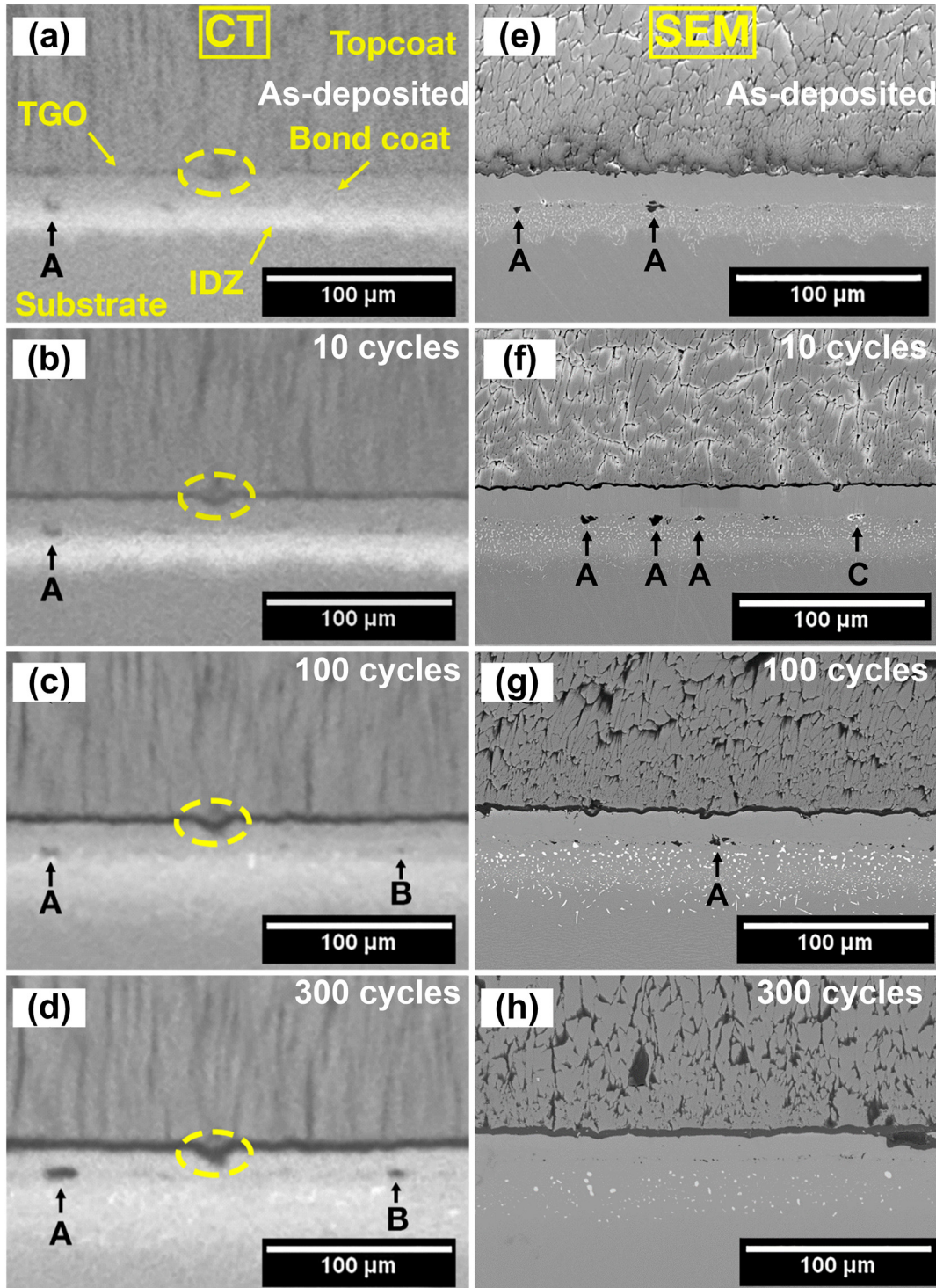


Fig. 1. X-ray  $\mu\text{-CT}$  virtual cross-sections (LHS) showing a single RoI in the centre of the TBC at different stages of thermal cycling. SEM micrographs (RHS) have been recorded for sections taken from the larger reference TBC sample after exposure to the same numbers of thermal cycles. The ellipse and labels are referred to in the text.

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