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Determination of mechanical properties of thermally grown oxide on Fecralloy by nano-indentation

Xiaofeng Zhao, Ping Xiao *

Materials Science Centre, School of Materials, University of Manchester, Manchester M1 7HS, UK

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Abstract

Nano-indentation was used to measure the mechanical properties of the thermally grown oxide (TGO) on a Fecralloy substrate. Due to the influence both of the substrate and the indenter size effect (ISE), the measured hardness and Young's modulus of the TGO system decreased with increasing indentation depth. Models were proposed to determine the mechanical properties of the TGO with consideration of both the substrate effect and the ISE. In addition, the ratio of hardness to Young's modulus (H/E) can be related to the ratio of irreversible work to total work (W_{ir}/W_t) during the indentation process.

Keywords: TGO; Young's modulus; Hardness; Indenter size effect (ISE)

1. Introduction

The increasing use of thermal barrier coatings (TBCs) for elevated temperatures in engine turbines brings with it a need for better characterisation of the coatings [1]. In the absence of mechanical damage, the failure of TBCs typically occurs between the thermally grown oxide (TGO) and the bond coat. Therefore, knowledge of the mechanical properties of the TGO layer is of importance because they are basic parameters for simulating the stresses or predicting the failure of TBCs. Unfortunately, as the TGO is too thin (several micrometres) and buried beneath a thick ceramic coating, determination of the mechanical properties is difficult. Although the nano-indentation technique has the ability and convenience to measure the mechanical properties on a small scale, direct application of this technique to a TGO is still difficult due to the following reasons:

First, the influence of the substrate on the measured properties such as the Young's modulus and hardness [2]. The standard methods for extracting Young's modulus and hardness from the load–displacement curve were developed assuming the indented material is monolithic, but are often applied to a film/substrate system without consideration of the influence of the substrate [2]. To obtain the intrinsic hardness and Young's modulus values for thin films, a commonly used rule of thumb is to limit the indentation depth to less than 10% of the film thickness [3,4]. However, this approach is not applicable for very thin films. If the indentation depth exceeds a critical value, the substrate begins to affect the measurement.

Second, in the range of small indentation depths less than several micrometres, materials show an increasing hardness with decreasing indentation depth, which is commonly referred to as the indenter size effect (ISE) [5,6]. To avoid this effect, a large penetration depth should be employed. However, with this condition, the influence of the substrate becomes significant. Therefore, to obtain the true mechanical properties of the TGO, both the influence of the substrate and the indentation size effect must be taken into account simultaneously.

The objective of this investigation was to study the influence of a soft substrate on the mechanical properties of a hard film and to develop methods to extract the intrinsic mechanical properties using nano-indentation measurements. The TGO formed on Fecralloy substrates was chosen because they have similar mechanical properties with the real bond coat in TBCs system. Based on previous studies [7–10], models were developed to obtain the true hardness of the TGO. In addition, the relationship between the hardness and the Young's modulus was also investigated.

* Corresponding author. Tel.: +44 161 200 5941; fax: +44 161 200 3586.
E-mail address: ping.xiao@manchester.ac.uk (P. Xiao).

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2. Experiments

2.1. Sample preparation

A commercially available Fecralloy substrate (Fe72.8, Cr22, Al 5, Y 0.1 and Zr 0.1, wt.%), in the form of cold-rolled plates of 1 mm in thickness, was annealed in a vacuum at 1200 °C for 9 h to release the residual stresses. Rectangular plates with dimensions of 20 × 10 mm were cut and continually polished to 1 μm finish. Unless specifically mentioned, the thickness of all specimens after the final polishing was 0.90 ± 0.01 mm. Prior to oxidation, the substrates were cleaned in acetone. In order to obtain different thickness of TGO, the samples were oxidised in a chamber furnace at a temperature from 1150 °C to 1250 °C, with a heating/cooling rate of 3 °C min^{-1} and a dwell time of 9 h. After oxidation, a dense, continuous oxide layer was formed on the surface of substrates.

Since the nano-indentation measurement is sensitive to the surface roughness of the samples, all the oxide layer were carefully polished to 1 μm finish and followed by a final polishing using a suspension of 40 nm colloidal silica.

2.2. Nano-indentation

Elastic modulus and hardness were obtained using Nano-Indenter™ XP (MTS, USA) with a Berkovich indenter. Before the experiment, the system was calibrated with a standard fused-silica specimen, and the indentation was performed using a constant nominal strain rate of 0.05 s^{-1}. After calibration, an array of 6 × 6 indentations was made both on the surface of the TGO and the substrate, with an interval of 100 μm between each indentation. The Poisson's ratio of the TGO and the substrate were taken as 0.25 and 0.30, respectively.

2.3. Data analysis and correction

Like most metallic materials, the deformation in the Fecralloy substrate by nano-indentation usually pushes out into the side of the indenter, which is referred to as a 'pile-up' (Fig. 1A). When this happens, the actual contact depth is larger than the measured contact depth. As a consequence, the contact area will be underestimated by the system and thus the measured properties will be overestimated. In order to correct this effect, the contact area measured by atomic force microscopy (AFM) was used for the calculation of each indentation point. The detailed procedure for this correction are described elsewhere [4,11,12]. However, for the hard TGO, no 'pile-up' effect was observed in almost all of the indents (Fig. 1B). Therefore, the hardness and Young's modulus were evaluated by the Oliver–Pharr method [3].

2.4. Microstructural characterisation

The thickness of the TGO layer was measured using a scanning electron microscopy (SEM, Philips XL30). To study the micro-structural change after indentation, a focused ion beam (FIB, FEI Nova 600 DualBeam system) was employed to fabricate the cross-section of the indenter impressions. These cross-sectional samples can be further thinned to electron transparency for transmission electron microscopy (TEM) observations [13]. Both the micro-structure and phases of the TGO were identified by an analytical transmission microscopy (Tecnai™ G^2 F30).

3. Analysis: separation of the film hardness from the substrate

The effect of the substrate on the hardness of a thin film has been studied by many researchers [7–9,14–18]. Jonsson and

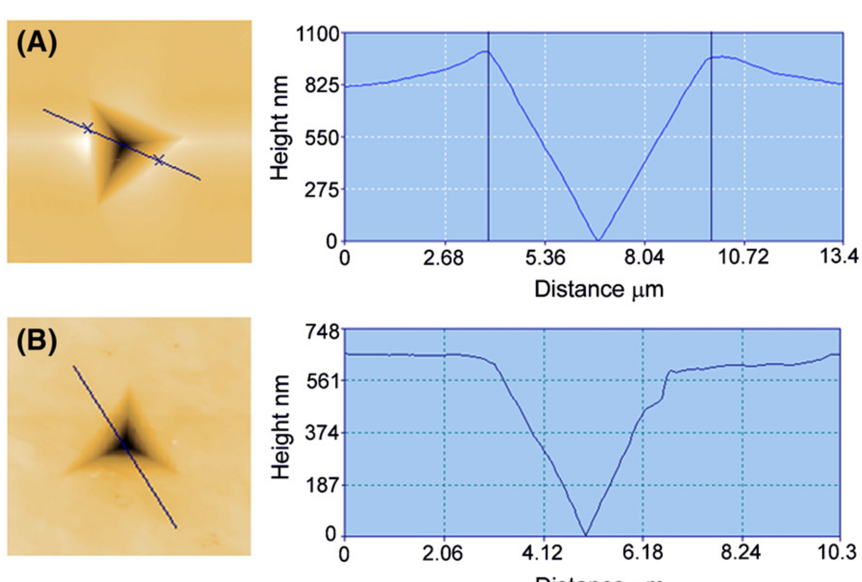

Fig. 1. Typical AFM images of impressions developed by a Berkovich indenter on (A) a Fecralloy substrate annealed at 1200 °C for 9 h and (B) a TGO formed at 1200 °C with a thickness of about 3 μm. The indentation depths for both samples are 1100 nm.