ARTICLE IN PRESS

Ceramics International xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

High temperature fracture toughness and residual stress in thermal barrier coatings evaluated by an in-situ indentation method

Zhaoliang Qu[a,b], Kai Wei[c], Qing He[d], Rujie He[a,*], Yongmao Pei[e,*], Shixing Wang[d], Daining Fang[a,e]

[a] *Institute of Advanced Structure Technology, Beijing Institute of Technology, Beijing 100081, People's Republic of China*
[b] *School of Mechanical Engineering, Beijing Institute of Technology, Beijing 100081, People's Republic of China*
[c] *State Key Laboratory of Advanced Design and Manufacturing for Vehicle Body, Hunan University, Changsha, Hunan 410082, People's Republic of China*
[d] *Surface Engineering Research Institute, Chinese Academy of Agricultural Mechanization Sciences, Beijing 100083, People's Republic of China*
[e] *College of Engineering, Peking University, Beijing 100871, People's Republic of China*

ARTICLE INFO

Keywords:
TBCs
In-situ indentation method
High temperature fracture toughness
High temperature residual stress

ABSTRACT

High temperature fracture toughness and residual stress are important for the evaluation of TBCs. In this paper, an in-situ high temperature indentation method was originally developed to investigate the high temperature fracture toughness and residual stress in a typical TBC, nanostructured 8 wt% yttria partially stabilized zirconia (YSZ) coating. The cracks caused by in-situ high temperature indentation tests were observed, and high temperature fracture toughness and residual stress were experimentally measured. The fracture toughness was measured to be 1.25, 0.91 and 0.75 MPa*m$^{1/2}$ at 25, 800 and 1000 °C, respectively. The residual stress was measured to be − 131.3, − 55.5 and − 45.5 MPa, correspondingly. Moreover, the residual stress and fracture toughness both decrease with increasing environmental temperature. It is also found that the fracture toughness without consideration of residual stress is significantly larger than the intrinsic fracture toughness, which may result from the compressive stress state.

1. Introduction

Thermal barrier coatings (TBCs) are widely used to improve fuel efficiency, increase gas temperature and prolong the life of the components in turbines for propulsion and power generation [1–3]. TBCs usually suffer severe working conditions in service, including high temperatures, high pressures, thermal shock, oxidative and corrosive environments, and complicated mechanical loadings. Such severe working conditions usually lead to catastrophic premature failures and considerably restrict the widely applications of the TBC. Recently, numerous studies have focused on the failure mechanism in order to improve TBCs' reliability, and provide guidance for their design and applications [3–5]. Generally, it is widely known that the changes of stress state and mechanical properties, especially such as hardness, elastic modulus and fracture toughness, are the key factors on the failure mechanism of TBCs [5]. Thus, typical characterization methods, including macroscopic mechanical test, ultrasonic measurement, indentation, Raman spectroscopy, are reported to evaluate the stress state and mechanical properties (i.e. hardness, elastic modulus and fracture toughness) [6–10]. Specially, it should be noted that the stress state and mechanical properties should be well evaluated under high temperature since TBCs usually work under high temperature service environment. Unfortunately, currently reported methods are always conducted in "as-coated" or "ex-situ (after heat treatment)" conditions, and the high temperature stress state and mechanical properties of TBCs cannot be obtained. Thus, it is urgent to develop a method to evaluate the high temperature stress state and mechanical properties of TBCs at high temperatures.

High temperature indentation, as an in-situ mechanical testing method, has distinguished advantages in evaluating the mechanical behaviors of multilayered TBCs [11–14]. In our previous work [14], the hardness and Young's modulus of nanostructured 8 wt% yttria partially stabilized zirconia coatings were measured by high temperature indentation in air up to 1200 °C. Stress state plays an important role in the crack evolution of TBCs, and the fracture toughness represents resistance ability TBCs containing a crack to fracture failure. Unfortunately, few studies reported fracture toughness and stress state of TBCs at high temperatures. In this study, an in-situ high temperature indentation method was therefore developed to investigate the high temperature fracture toughness and residual stress of nanostructured 8 wt% yttria partially stabilized zirconia (YSZ) coatings at elevated temperatures. The aim of this work is to provide useful guideline for the study of the failure mechanism for TBCs.

---

* Corresponding authors.
E-mail addresses: herujie@bit.edu.cn (R. He), peiym@pku.edu.cn (Y. Pei).

https://doi.org/10.1016/j.ceramint.2018.01.230
Received 10 January 2018; Received in revised form 24 January 2018; Accepted 27 January 2018

Please cite this article as: Qu, Z., Ceramics International (2018), https://doi.org/10.1016/j.ceramint.2018.01.230

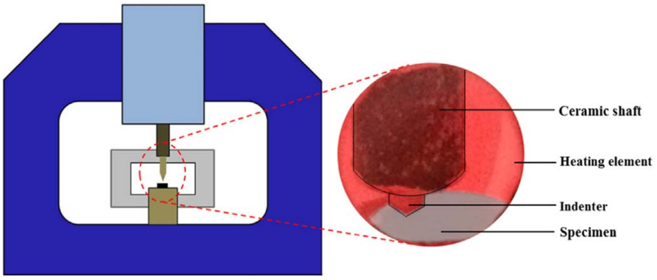

Fig. 1. Diagrammatic sketch of the in-situ high temperature indentation testing system assembled in our laboratory.

2. Material and methods

Nanostructured YSZ coatings, kindly provided by Beijing Golden Wheel Special Machine Co., Ltd (Beijing, China), were chosen and evaluated in this study. The coatings were prepared by air plasma spraying process. The coatings were deposited on flat polycrystalline alumina rather than superalloy since superalloy may oxidize and creep at high temperature, and hence preclude the indentation investigations. The thickness range of ceramic coatings is 300–400 μm. Specimens with 15 mm × 15 mm × 3 mm (length × width × thickness) were prepared for high temperature indentation tests. Prior to indentation tests and microstructure observations, specimens were ground and polished with diamond slurries down to a 1 μm finish. Afterwards, the specimens were carefully cleaned by ultrasonic oscillator, and completely dried by a drying machine.

Subsequently, as shown in Fig. 1, the prepared nanostructured YSZ coatings were characterized using the in-house designed and constructed in-situ high temperature indentation testing system [15]. A muffle furnace is used to provide a high temperature environment. The temperatures are measured by a thermocouple near the specimen table, which locates in the uniform temperature zone. The measurement temperature is almost the same with the actual testing temperature. Before indentation tests, specimens were placed inside the high temperature furnace in ambient laboratory atmosphere. Then, the specimens were heated to the target testing temperatures, and held at the target testing temperature with a dwelling time to reach a thermal equilibrium state. The target testing temperatures were chosen as 25, 800 and 1000 °C. Since the cube corner indenter is easier to produce cracks than the Vickers or Berkovich indenters at a given load, a cube corner indenter was used to perform in-situ high temperature indentation tests on the top surface of the coatings. Load-controlled mode was adopted to exactly control the maximum indentation load during indentation tests. The maximum indentation loads were kept as 80, 100 and 120 N, with a dwelling time of 20 s. After in-situ high temperature indentation tests, the images of residual impressions were observed by a scanning electron microscope (SEM, FEI Quanta 250, USA) equipped with an X-ray EDS analyzer attachment (EDAX Inc., USA), and the lengths of indentation cracks were measured and collected.

It should be noted that while the cube corner indenter was used to evaluate residual stress and fracture toughness, the Young's modulus and hardness should be measured by standard indentation techniques using a Berkovich indenter. In this study, the measurements of Young's modulus and hardness for TBCs at different temperatures were performed using in-house constructed high temperature indentation testing system [15] equipped with a Berkovich sapphire indenter. Corresponding to the previous fracture indentation tests, the testing temperatures were also set as 25, 800 and 1000 °C. The indentation tests were conducted under a maximum load of 3 N, with a nominally constant loading rate of 0.1 N/s during the loading and unloading cycle. At least 5 tests were performed to determine the average value at each selected testing temperature.

3. Results and discussion

Indentation method has been widely used to investigate the fracture behaviour of a coating system [16,17]. Considering the residual stresses in the coating [16,17], based on the principle of superposition, the total stress intensity factor K_I can be given by:

$$K_I = \chi P/c^{3/2} + 4\sigma_r\sqrt{t}/\sqrt{\pi} - (2\sigma_r t)/(\sqrt{\pi c}) \tag{1}$$

where $\chi = \delta(E/H)^{1/2}$, and $\delta = 0.036$ is a geometric factor, for a cube corner indenter. E and H are Young's modulus and hardness, respectively. P is the peak indentation load, and c is the average crack length. σ_r is the average stress in the coating, and t is the thickness of the coating.

For equilibrium fracture conditions, the cracks extend when K_I reaches a critical value, K_{IC} (fracture toughness). Eq. (1) can then be rewritten as:

$$P/c^{3/2} = (K_{IC} - 4\sigma_r\sqrt{t}/\sqrt{\pi})/\chi + ((2\sigma_r t)/(\chi\sqrt{\pi}))c^{-1/2}. \tag{2}$$

A linear relationship is found between the ratio $P/c^{3/2}$ and $c^{-1/2}$. Here, $2\sigma_r t/(\chi\sqrt{\pi})$ and $(K_{IC} - 4\sigma_r\sqrt{t}/\sqrt{\pi})/\chi$ denote the slope and intercept of Eq. (2), respectively. Then, residual stress and fracture toughness can be determined by the values of slope and intercept, respectively. To obtain a better linear equation, a series of indentation tests should be performed under different maximum indentation loads, and the corresponding crack lengths should be measured. In this study, indentation tests were conducted under different loads (80, 100 and 120 N) upon the top surface of TBCs at various temperatures. Then, the residual indentation impressions were observed by SEM, and the lengths of indentation cracks (c_1, c_2 and c_3) were measured. Typical SEM observations of residual indentation impressions at different temperatures are presented in Fig. 2. It can be found that the shapes of residual indentation impressions were typically triangular, and three

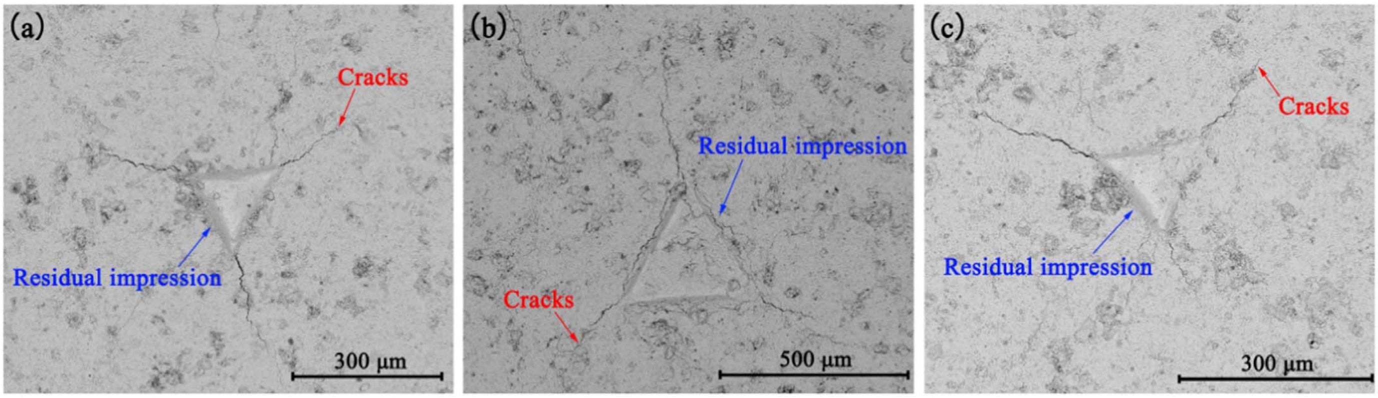

Fig. 2. The typical SEM observations of residual indentation impressions at (a) 25, (b) 800 and (c) 1000 °C, respectively.