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# Adhesion strength of ceramic top coat in thermal barrier coatings subjected to thermal cycles: Effects of thermal cycle testing method and environment

Masakazu Okazaki [a,*], Satoshi Yamagishi [a], Yasuhiro Yamazaki [b], Kazuhiro Ogawa [c], Hiroyuki Waki [d], Masayuki Arai [e]

[a] Department of Mechanical Engineering, Nagaoka University of Technology, 1603-1 Kamitomiokamachi, Nagaoka-shi, Niigata 940-2188, Japan
[b] Department of Mechanical Engineering, Niigata Institute of Technology, Japan
[c] Fracture and Reliability Research Institute, Tohoku University, Japan
[d] Department of Mechanical Engineering, Iwate University, Japan
[e] Materials Science Research Laboratory, Central Research Institute of Electric Power Industry, Japan

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ABSTRACT

This paper deals with the adhesion strength of ceramic top coat in thermal barrier coatings (TBCs) subjected to thermal cycles under several different test conditions. Here the TBC specimens consisting of 8% yttria stabilized zirconia, CoNiCrAlY alloy bond coat and Ni-base superalloy were prepared by plasma spraying. The isothermal exposure and the thermal cycles were applied to the TBC specimens by several conditions at high temperatures. A series of the test results clearly demonstrated that the adhesion strength of the top coat was significantly changed by the application of thermal cycles and by the isothermal exposure. It was also found that the thermal fatigue damage might be evolved depending on of the testing method by which the thermal cycles are applied. Some background of these findings were discussed, based on the measurements of elastic modulus, tensile strength, and thermal conductivity of the ceramic top coat, as well as both the thermally grown oxide at the top coat/bond coat interface and the residual stress in the TBC specimens.



## 1. Introduction

Thermally insulating ceramic coatings, known as thermal barrier coatings (TBCs) are essential to improve the performance and efficiency of advanced gas turbines in service at extremely high temperatures [1–6]. The key role of TBCs is, of course, to protect the metal substrate from high temperature oxidation and environmental attack [1,2]. In general the TBC system consists of at least three layers; ceramic top coat, bond coat and metal substrate.

The most critical issue limiting durability of TBCs is spallation of the ceramic top coat. Once this type of damage is realized, hot section components made of superalloy substrate may be overheated, resulting in complete failure. Adhesion strength is a major parameter characterizing the resistance of the ceramic top coat against spallation [7,8], where the strength has been often evaluated according to the ASTM standard [7]. In general the adhesion strength of top coat is significantly changed during service. (i) Thermal stress, which is promoted by the mismatches in thermal expansion coefficient and thermal conductivities between the metal substrate, bond coat and ceramic top coat, and (ii) the influence of environment, i.e. formation of thermally grown oxides (TGOs) at the bond coat/top coat interface, are essential factors [9,10,12–14]. The interaction between both factors is also important in some cases [12–14]. In the actual TBCs in service the thermal cycle failure is often a critical issue to be concerned, to which all of the above factors commit. Nevertheless, the basic understanding of failure mechanisms and their interaction still is still on the way of research.

It is an objective of this work to get basic understanding on effects of thermal cycle testing method on the thermal fatigue damage of TBCs, through the measurements of both the mechanical and physical properties of top coat and the remaining adhesion strength.

## 2. Experimental procedures

### 2.1. Preparation of specimens

The TBC specimens consisting of three layers; Ni-base superalloy, bond coat and top coat, were prepared in the present work. Here, an 8 wt.% yttria partially stabilized zirconia (YSZ), METCO 204NS, and a CoNiCrAlY alloy, AMDRY9951, were selected as the top and bond coat materials, respectively. The chemical compositions of the powders used are listed up in Table 1. The substrate

* Corresponding author.
*E-mail address:* okazaki@mech.nagaokaut.ac.jp (M. Okazaki).

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**Table 1**
Chemical compositions of the powders used (wt.%).

| (a) Top coating powder | | | | | | |
|---|---|---|---|---|---|---|
| $ZrO_2$ | $Y_2O_3$ | $HfO_2$ | MgO | $SiO_2$ | $TiO_2$ | CaO |
| 90.5 | 7.5 | 1.6 | <0.01 | 0.04 | 0.11 | 0.02 |
| *(b) Bond coating powder* | | | | | | |
| Co | Ni | Cr | Al | Y | | |
| 38 | 32 | 21 | 8 | 0.5 | | |

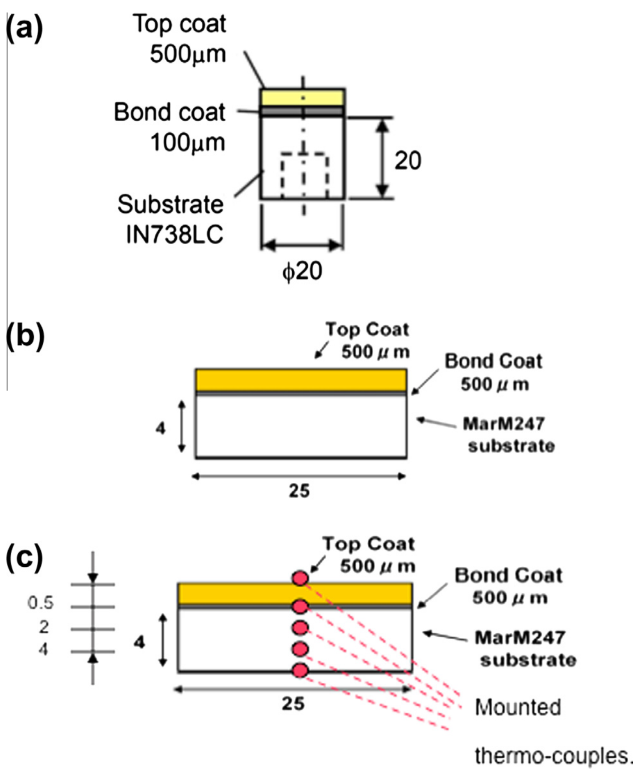


**Fig. 1.** Geometry of specimens used. (a) Adhesion strength, (b) residual stress, and (c) thermal conductivity.

material employed was a polycrystalline Ni-base superalloy, Mar M247.

The geometries of TBC specimens used are illustrated in Fig. 1. These TBC specimens were fabricated as follows: after spraying the bond coat alloy by 100 µm in thickness on the substrate, the YSZ top coat was overlay coated by 500 µm in thickness. These processes were managed and performed by atmospheric plasma spraying at Plasma Giken Co. Ltd., Japan. The details are presented in Refs. [11,18].

Moreover, the free-standing top coat specimens were also prepared to measure their some basic properties: elastic stiffness, tensile strength and thermal conductivity. Here, for the former two properties, the specimens were extracted by removing the substrate material from the TBC specimen by chemical solution technique. The outline of the measurement method will be given in each subsection in next chapter.

### 2.2. Thermal cycle and isothermal exposure tests

Either thermal cycles or isothermal exposure was applied to the TBC specimen in air, according to the test conditions summarized in Table 2. Thermal cycles were applied, following the two different type of test methods; hereinafter, denoted by *METHOD-I* and *-II* in this work, respectively. In the "*METHOD-I*" one single furnace was used to cyclically heat-up and cool-down the TBC specimen under such a cycle frequency low enough that the temperature in the TBC specimens might be changed in steady state without significant temperature gradient [17] (Fig. 2a). The heating and cooling rates employed in this work were approximately 0.133 °C/s and 0.067 °C/s, respectively. The control of test temperature was conducted via R type thermocouples which represented the temperature within the electric furnace. At the same time the TBC specimen temperature was monitored by the thermocouples welded on the substrate, to ensure insignificant temperature difference between the furnace and specimen. In the "*METHOD-II*" thermal cycle test, on the other hand, two furnaces isolated with each other; higher and lower furnaces, were used. The temperature of the two furnaces was kept constant so that they corresponded to higher and lower temperatures in the thermal cycle test. At the same time, the specimen temperature was continuously monitored by the thermocouples, as well. During the test the TBC specimen traveled periodically between the two furnaces, through a mechanical driving system. Here, the traveling time was so short (i.e., within 30 s) that the temperature gradient in the specimens was significant during the traveling period, as will be given in Section 3.2. The heating and cooling rates in the *MEHOD-II* were approximately 1.5 °C/s and 0.67 °C/s in average, respectively.

The thermal cycle test conditions are summarized in Table 2. The isothermal exposure test was also carried out in air, by means of an electric furnace.

After applying either thermal cycles or isothermal exposure, the residual adhesion strength of the ceramic top coat was evaluated, according to the ASTM standard, C633 [7], as a representative measure to assess the damages. The residual stress in the top coat was also measured by the stress relief method. Here the stress was evaluated from the measurement of relief strain between before/after the removal of metallic substrate and bond coat.

## 3. Results and discussion

### 3.1. Isothermal exposure test

Elastic stiffness of the ceramic top coat was measured by applying an external tensile load to the free-standing 0.5 × 10 × 70 mm rectangular plate specimen, where the tab plates were adhered on the both sides of the specimen parts for clamping (Fig. 3a). Stress–strain relation was monitored via a load cell and a strain gauge directly adhered at the center of specimen gauge section (see Fig. 3a), from which slope the elastic modulus was determined. Fig. 3b reveals some typical stress–strain curves. The elastic modulus evaluated is summarized in Fig. 4 as a function of isothermal

**Table 2**
Test program of thermal cycle and isothermal exposure tests.

| Type of test | Environment | Temperature/temperature range | Time/number of cycle |
|---|---|---|---|
| High temperature (isothermal) exposure | In air | 800 °C, 900 °C, 1000 °C, 1100 °C | 0, 100, 300, 1000 h |
| *METHOD-I* thermal cycle (uniform heating and cooling) | In air (in vac.) | 1000–400 °C, 1000–500 °C, 900–400 °C, 900–650 °C | 0, 10, 100, 1000 (cycles) |
| *METHOD-II* thermal cycle (non-uniform heating and cooling) | In air | 1000–400 °C | 0, 10. 100,1000 (cycles) |