#### exposed to high temperatures, such as air-craft engines, gas turbines, reciprocating engines, and power switchgears, are itoring of the damage test. In this manner, t

ularly enables to the non-destructive evaluation of failure cycle.

burner heating test, heat cyclic resistance, Japa-

nese industrial standard, temperature gradient,

thermal barrier coatings

**Testing Method for Heat Resistance Under** 

**Temperature Gradient** 

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"Testing Method for Heat Resistance under Temperature Gradient" is a Japanese Industrial Standard (JIS) newly established by the Minister of Economy, Trade and Industry, after deliberations by the Japanese Industrial Standards Committee, in accordance with the Industrial Standardization Law. This standard specified the testing method for heat resistance under temperature gradient of materials and coated members of equipment exposed to high temperature, such as aircraft engines, gas turbines, and so on. This paper introduces the principle and overview of the established standard. In addition, taking the heat cycle test using the burner rig for instance, we specifically illustrate the acquirable data and their analysis in the standard. Monitoring of the effective thermal conductivity and acoustic emission partic-

subject to temperature gradient at all times. The temperature gradient determines the macro/micro stress distributions in the materials and coatings, and it influences thermal cycle damage during the repetition of start-up and shutdown operations of the equipment. Therefore, development for such materials and coatings require a standard testing method for heat resistance under temperature gradient. So far, the uniform heating test and the thermal shock test with water-quenching have been applied to evaluate heat resistance (Ref 1, 2). However, these methods cannot realize a heat resistance test with simulation of an actual temperature gradient, because the inner temperature dis-

The materials and coated members of equipments

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tribution of the test pieces is automatically determined by their shape, material, and test environment only.

On the other hand, the heat-resistance tests involving heat treatment on the surface of the test piece, such as the burner rig test, has so far been applied as a simulative test using a small test piece (Ref 3, 4). These methods are capable of realizing necessary temperature gradient by controlling both the surface and rear face temperatures of the test piece. In addition, combining these heating tests with an acoustic emission method enables an in situ monitoring of the damage progress during the cyclic heating test. In this manner, the surface heating tests have been demonstrated to be effective for evaluation of heat resistance under temperature gradient. However, no industrial standard regarding such a surface-heating test has been established worldwide. Therefore, an exploratory committee for the standardization was organized in 1998, and consequently new Japanese Industrial Standard named "Testing Method for Heat Resistance under Temperature Gradient" was established by the Minister of Economy, Trade, and Industry in 2005, after deliberations by the Japanese Industrial Standards Committee, in accordance with the Industrial Standardization Law (Ref 5).

This article firstly describes the outline of this newly established Japanese Industrial Standard. Subsequently, we introduce a practical example of the cyclic heating test for thermal barrier coatings (TBCs) using a burner heating apparatus, according to this standard.

# 2. Testing Method for Heat Resistance Under Temperature Gradient (JIS H7851)

This standard specifies the testing apparatuses and test pieces in order to achieve commonality of the heat cyclic evaluation as outlined below.

Keywords

1. Introduction

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### 2.1 Principle

The principle of this new standard is indicated in Fig. 1. The test piece mounted on the jig is subjected to temperature gradient by heating its surface and cooling the rear surface of the jig. The heating source shall be chosen from among burner heating, arc heating, plasma heating and beam heating by lamp, laser, or electron beam. The cooling source, such as water and gas, should have the performance enough to obtain the required temperature gradient. The surface temperature of the test piece  $T_h$  and the inner temperatures of the jig shall be measured in real time by a radiation thermometer and thermocouples, respectively. These measurements can provide the heat flux Q and the temperature difference  $\Delta T$  of the test piece as follows:

$$Q = -\lambda_s \cdot dT/dx \tag{Eq 1}$$

$$\Delta T = T_h - T_c \tag{Eq 2}$$

where  $\lambda_s$  and dT/dx are, respectively, the thermal conductivity of the jig and the temperature gradient which is obtained from the extrapolation of three temperatures in the jig. The surface temperature,  $T_h$ , of test piece can be measured by a noncontact type thermometer such as a radiation thermometer. The rear surface temperature,  $T_c$ , can be obtained from the extrapolation. Additionally, the effective thermal conductivity of the test piece can be calculated as:

$$\lambda_{\rm eff} = Q \cdot t / \Delta T \tag{Eq 3}$$



Fig. 1 Principle of the "Testing Method for Heat-Resistance under Temperature Gradient" (JIS H7851)

where t is the thickness of the test piece. Under the above temperature gradient, heating/cooling cycle test is conducted. Evaluations of the damage state and fracture morphology after the cycle test, such as surface and cross-sectional observations, can determine the cyclic thermal resistance of test pieces. Particularly, combining an acoustic emission method enables *in-site* monitoring of crack propagation during the heat cycle test.

#### 2.2 Test Pieces

The test piece should be mounted on the jig by either coating or mechanical bonding as shown in Fig. 2a and b. Otherwise, the test piece may have its portion functioning as a jig as shown in Fig. 2c. Additionally, the test piece and jig should be shaped as in Fig. 2d, where thermocouple holes allow for monitoring the thermal gradient in the jig.

# 3. Cyclic Heating Test with Burner Heating Apparatus

Among the heating sources that are listed in the standard, the burner heating system is one of the most effective methods because it is capable of properly simulating actual environmental conditions in high-temperature equipments. The following paragraphs will describe one example of the cyclic heating test with a burner heating apparatus according to the new standard. Plasma-sprayed TBCs were chosen as the test pieces.

# 4. Experimental Procedure

### 4.1 Burner Heating Test Apparatus

Figure 3 shows the burner heating test apparatus. The test piece was mounted to the supporting holder with the water-cooling functioning to cool the back face of the jig. The temperature of the water was kept constant by a chiller unit. Combustion of oxygen/hydrogen  $(O_2/H_2)$  was



**Fig. 2** Schematic of the test piece. The test piece shall be (a) soldered or mechanically bonded to the jig with the base material, (b) coating provided to the jig, or (c) a part of test piece functioning as jig. (d) The typical shape and dimension of the test piece



Fig. 3 Schematic of the burner apparatus for heating test

applied for the burner flame, and the flow rate of the gases controls the burner output. As described above, the temperatures on the surface and in the jig were monitored at all times by the radiation thermometer and thermocouples, respectively. The acquired temperature data were converted into heat flux, temperature difference, and effective thermal conductivity in real time by a computer. The shutter has a function to interrupt the flame, and the repeating ON/OFF of the shutter realizes the heat cycle. Additionally, the acoustic emission (AE) sensor mounted on the jig. This sensor is also capable of detecting the initiation and propagation of cracks in real time.

### 4.2 Thermal Barrier Coating Test Piece

The duplex coating test pieces consisting of partially stabilized zirconia (PSZ) and NiCrAlY alloy layers were prepared by air pressure plasma spraying, as indicated in Fig. 4. The thickness of the PSZ layer and NiCrAlY layers were approximately 340 and 80  $\mu$ m, respectively. Type 304 stainless steel was chosen as the material for jig.

# 5. Results and Discussion

The heat cycle tests were conducted by gradually increasing the burner output at each cycle. As seen in Fig. 5, the temperature of the heated surface linearly increases with increasing burner output. This result indicates that the temperature condition of the outer surface can be controlled by the adjustment of the burner output. The temperature difference  $\Delta T$  is also one of the important thermal parameters. In the present result, the effective thermal conductivity is slightly increased by increasing the burner output. If the thermal conductivities for the TBCs in Eq 3 were invariable, the temperature difference should be varied by the burner output, i.e., the heat flux Q. In other words, this slight variability of the  $\Delta T$ would be attributed to the increase in the thermal con-



Fig. 4 Optical micrograph of cross-section for thermal barrier coating test piece



**Fig. 5** Relationships between the burner output, the temperatures of front and rear faces, and temperature gap

ductivity of TBCs during the cyclic test. In actual, the calculated effective thermal conductivity monotonously increased until the sixth cycle with Q = 0.6 M W/m<sup>2</sup> as seen in Fig. 6. Sintering behavior in the porous bond coat prepared by the APS could explain this increase in the effective thermal conductivity. But we must explore a more satisfactory reason down the line because this result conflicts with our previous study in which the thermal conductivity showed no change as the cyclic heating with the constant burner output (Ref 4). In any case, the heating test according to this standard can provide the





Fig. 6 Relationships between the heat flux, effective thermal conductivity, and AE hit counts

reasonable values of effective thermal conductivity which are in good agreement with the experimental and analytical values in the literatures (Ref 6, 7).

On the other hand, the effective thermal conductivity did not continue to increase but began to decrease as seen at the eighth cycle in Fig. 6. This decrease implies the occurrence of cracks preventing the heat transfer. Such cracks are obviously parallel to the coating and jig interface so that they directly cause the fracture of the TBCs. Therefore, progression of fracture in the test piece can be detected by decreases in effective thermal conductivity by this particular method. In the case of TBCs, the cracks parallel to the interface between the top coat and the bond coat have been theoretically and experimentally proven to be generated and propagated during heating (Ref 8-10). Thus, the increase in the AE hit counts during heating in Fig. 6 definitely corresponds to significant cracking. Consequently, analysis of the AE data may help to determine the state of internal damage of the test piece as well as the effective thermal

conductivity. Finally, Fig. 6 indicates that the test piece failed at the cycle of  $Q = 0.6 \text{ M W/m}^2$ .

## 6. Summary

"Testing Method for Heat Resistance under Temperature Gradient" is a Japanese Industrial Standard (JIS) newly established by the Minister of Economy, Trade, and Industry. This standard specifies the testing method for heat resistance of materials and coated members of equipment exposed to high temperature under temperature gradient. An example test using a burner heating apparatus reveals the cyclic failure of thermal barrier coating test piece by analyses of the effective thermal conductivity and AE hit counts.

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