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Evaluation of in situ fracture toughness of ceramic coatings at elevated temperature by AE inverse analysis

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Abstract

This paper describes acoustic emission (AE) measurement at elevated temperature by laser techniques. AE signals originated from microfracture of Al\textsubscript{2}O\textsubscript{3}/SUS304 plasma sprayed coatings were detected using a laser interferometer during thermal cycling. The radii of microcracks were evaluated by the inverse analysis of AE signals. Microcrack radii were also studied by the numerical analysis based on the delamination model for coating materials with various fracture toughness and an initial crack size at elevated temperature. In situ fracture toughness for microcracking was evaluated by combining the results of AE inverse analysis with those of numerical analysis. This approach revealed that the in situ toughness of the coatings is 40–50 J/m\textsuperscript{2}, consistent with the results of a double cantilever beam test.

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1. Introduction

Thermal barrier coatings (TBCs) have been developed and utilized to improve the efficiency of internal combustion engines and gas turbine. In these coatings, debonding of ceramic coatings becomes severe problem and there are compelling needs to understand and evaluate the interfacial reliability and properties in order to establish the life prediction model. The failure mechanism of TBCs under thermal cycling has been well discussed in various papers and it was understood to be caused by the nucleation of microcracking at/near the interface of ceramic/bond layer or bond layer/substrate, and following growth and coalescence of these cracks [1–8]. Therefore, the present interest is especially to quantitatively evaluate those mechanisms of nucleation and growth of microcracking. However, since the cracking and growth in TBCs occur under severe environments such as elevated temperature, applicable techniques for evaluation of failure are very limited and it is difficult to quantitatively evaluate the fracture behavior and the interfacial properties with thermal treatment. Therefore, the in situ evaluation method for fracture under thermal cycle environment is required.

Measurements of acoustic emission (AE) signals using a wave-guide were tried to evaluate the temperature of fracture in coatings [9,10]. In this method relatively long wave-guide was used to avoid the damage of a piezoelectric transducer (PZT) due to heat and the detected AE waveforms were strongly depended by the wave propagation properties of wave-guide. That makes difficult to quantitatively evaluate the fracture behavior such as a location of AE source, size of microfracture, fracture mode, and so on. On the other hand, a method to detect an elastic wave at the surface using laser techniques instead of a PZT became popular [11–14]. The use of a laser interferometer has several advantages. Since a laser interferometer makes no physical contact with the observed surface, it can detect a seismic AE event at elevated temperature. Also, a laser interferometer can directly measure a surface velocity and displacement of object surface and this absolute measurement is suitable for quantitative AE source characterization. Previous works have demonstrated that this laser based AE technique was very effective to detect AE events of coatings...
at elevated temperature [15–17]. Inverse analysis of AE source was quite useful to understand the microcrack behavior in materials [18–21]. With these insights, this article tries to evaluate the in situ local fracture toughness based on the laser based AE technique and AE source characterization method.

2. Thermal cycle test of coatings

2.1. Experimental procedure

The specimen was deposited by plasma splaying of an Al₂O₃ top coat (500 μm thick) and a Ni–Cr–Al–Y bond coat (100 μm thick) onto a SUS304 steel substrate (5 mm thick). The geometry of a substrate was rectangle (15 × 15 mm²). The thermal expansion misfit (Δα) between an Al₂O₃ layer and a SUS 304 substrate is about four times as large as the misfit on a conventional Y₂O₃–ZrO₂/NiCrAlY/Ni-superalloy coating system. Since the larger thermal expansion misfit will lead to higher misfit stress, this system is expected to have much shorter cyclic life than a commercial one that is suitable for focusing the study of AE measurements by a laser interferometer and the basic understandings of the fracture behavior of ceramic coatings at elevated temperature. The cyclic heat treatment was performed at 900, 1000, 1100, and 1200 °C with an infrared image furnace. Temperature was controlled by a thermocouple placed on the surface of the specimen in the heated zone. The thermal treatment comprises a heating up to the maximum temperature with constant rate 10 °C/s, 10 s holding at the maximum temperature, and cooling to room temperature in air. The samples were thermally cycled until the large-scale delamination had been observed. During the whole period of experiment, a laser interferometer was simultaneously applied to detect AE signals generated from the microfracture of coating layer, as shown in Fig. 1. The laser beam was lead to the specimen with the reflection mirror and focused on the opposite side of the specimen, where surface was polished to improve the sensitivity.

A commercial heterodyne laser interferometer with He–Ne laser (Graphitec Co. Ltd, AT0022) was used, which is so called a Laser Doppler Vibrometer (LDV) and can directly measure the surface velocity of an object. Low pass filter of 300 kHz and high pass filter of 200 Hz were applied to reduce the noise level. Detected AE waveforms were recorded by the wave memory (JT toshi Co. Ltd, DCM-120) with sampling rate of 200 ns and the length of 2 kwords.

2.2. AE behavior

The temperature at the measurement point focused by laser beam was over 1100 °C for the samples of holding temperature 1200 °C, shown in Fig. 2. It was impossible to measure AE events using a PZT because this temperature was extremely higher than Quie point. Fig. 3 shows the typical temperature history and detected AE events for each holding temperature, where different symbols stand for the order of thermal cycling. It clearly indicates that a laser interferometer has an advantage to measure AE under such high temperature environment. Fig. 4 typical AE waveforms detected during cooling for the sample kept at temperature of 1100 °C. The difference between AE waveforms seems to directly correspond to the variation of fracture behavior. Most of AE signals were generated during only cooling period and detected at the temperature less than about 400 °C. As the holding temperature decreased, it took more cycles to cause the large scale delamination, but the effect of holding temperature was not so remarkable in this experiment.

2.3. Microfracture observation

Fig. 5 shows the cross-section of the specimen after (a) the first cycle and (b) after a large scale delamination. Delamination parallel to the interface, at about 50 μm far...
from the interface in the ceramic layer, was observed in the ceramic layer. Cracks perpendicular to the interface were not observed in Al₂O₃ layer. This indicates that AE signals were mainly generated due to delamination parallel to the interface in the ceramic layer. The scanning acoustic microscope (SAM) with acoustic probe (25 MHz) was applied to observe the fracture of interfaces and C-scan images of the specimen with holding temperature of 1100 °C after each thermal cycle are shown in Fig. 6. Two cracks with about 1 mm diameter appeared in the center region after the first cycle, which are indicated by white arrow. The generation of another crack and coalescence between cracks were recognized in the second and the third cycle. Those cracks merged each other and propagated all over the specimen area at the fourth cycle. These cracks correspond to the delamination in the ceramic layer shown in Fig. 5. Therefore, it is demonstrated that the fracture behavior of coatings proceeds with step-by-step manner, that is, after the generated cracks were arrested once, the criteria of crack propagation or crack generation were satisfied again, and then the cracks began to propagate. Repeat of this process while thermal cycling results in the final failure.

3. Acoustic emission source characterization

3.1. Inverse analysis of AE

Microcracking of materials can be represented by the seismic moment tensor in dislocation theory and seismology [18] shown in Appendix A. We introduce two assumptions to apply this theory for present analysis. At first, a delamination parallel to an interface occurs under tensile fracture mode based on various theoretical analysis [22–24]. Secondly, microcracks are assumed to occur only in the ceramic layer based on SEM observation results. Therefore, the theoretical treatment for microfracture in homogeneous medium can be applied to describe the delamination in the ceramic layer. Due to these assumptions, the out-of-plane displacement at detection point \( U_3(x',t) \) is written as

\[
U_3(x',t) = \frac{\lambda}{\lambda + 2\mu} \left[ G_{31.1}(x',x,t) + G_{32.2}(x',x,t) \right] + \frac{\lambda + 2\mu}{\lambda} G_{33.3}(x',x,t) \ast \Delta D. \tag{1}
\]

Fig. 3. Typical temperature history of heated surface and detected AE events, (a) holding temperature of 1100 °C, (b) holding temperature of 900 °C. Symbols stand for the cycle number when AE events were detected.

Fig. 4. Detected AE waveforms in the case of holding temperature of 1100 °C, and (a) and (b) correspond to the symbols a and b in Fig. 3, respectively.
where \( \lambda \) and \( \mu \) are the Lamé constants, \( G_{ij}(x', x, t) \) is a differential of Green’s function \( G_{ij}(x', x, t) \) which means the displacement field in the direction \( x_i \) at position \( x' \) at the time \( t \) due to an impulsive force in the direction \( x_j \) at \( x \) at time \( 0 \). Source function \( \Delta D \) is the \( j = k = 3 \) component of the deformation moment tensor \( D_{jk}(x, t) \) and \( * \) represents a convolution integral with respect to time. Therefore, it can be understood that the source function due to microcracking can be obtained from deconvolution of Eq. (1), if the Green’s function \( G_{ij}(x', x, t) \) is evaluated and the out-of-plane displacement of detection point \( U_j(x', t) \) is experimentally measured.

3.2. Calculation of Green’s function and deconvolution

Green’s function has to be calculated in order to evaluate a source function due to microcracking. Analytical and experimental methods have some limitations for applying an ambient finite medium and numerical simulation is indispensable for obtaining more accurate Green’s functions of finite media [25]. Therefore, they were evaluated by using dynamic finite element method (FEM) code (LS-DYNA3D, Livermore software Co. Ltd) in the present work. The material properties in Table 1 were used for the calculation and Green’s functions were obtained by inputting the corresponding dipole moment at the 50 \( \mu \text{m} \) height in the ceramic layer from the interface at the center point of specimen. Also the dipole moment was assumed to have the same time function with step like wave of rising time of 2 \( \mu \text{s} \). By substituting calculated Green’s functions and the detected AE signals into Eq. (1), we could obtain AE source functions for each AE events after deconvolution calculation.

3.3. Evaluation of crack radius from AE

Assuming that a microcrack is a penny shape crack subject to a normal tensile stress \( \sigma \), we can estimate the crack radius \( a \) as

\[
a = \left[ \frac{3(1 - 2\nu)D_0/16(1 - \nu^2)\sigma}{\mu} \right]^{1/3}
\]

where \( \nu \) is the Poisson’s ratio and \( D_0 \) is the source intensity, which is the first peak value of calculated \( \Delta D \) [18]. The tensile strength of plasma sprayed Al\(_2\)O\(_3\) of 20 MPa was used for calculation. The distribution of microcrack radii is shown in Fig. 7 for detected AE events of all experimental conditions. Microcrack radii are estimated in the range of 100–500 \( \mu \text{m} \). The result shows good correlation with SAM

### Table 1

| Material properties used in the calculation of stress intensity factor by FEM |
|---------------------------------|-----|-----|
| Thickness (mm)                  | SUS304 | Al\(_2\)O\(_3\) |
| Elastic constant (GPa)          | 5.0  | 0.5  |
| Poisson’s ratio                 | 0.30 | 0.25 |
| Density (\( \times 10^3 \) kg/m\(^3\)) | 7.8  | 1.6  |
| Thermal expansion coefficient (\( \times 10^{-6} \text{ K}^{-1} \)) | 16   | 8    |
images and SEM observation. This result was used to estimate the in situ fracture toughness in Section 4.

4. Evaluation of in situ fracture toughness

In this section, we propose the method to evaluate the in situ fracture toughness by the result of AE source characterization and the pop-in fracture model. The methodology is schematically shown in Fig. 8. The radius of microcrack generated during thermal cycle test can be evaluated by the AE inverse analysis as mentioned in Section 3.3. Therefore, if the crack radius is given as the function of fracture toughness by a fracture model, we can estimate the in situ fracture toughness for each AE event.

4.1. Pop-in fracture model

The roughness of the interface is considered to be a dominant factor to cause failure of the coatings. Many researches considered the stress field and crack initiation due to interface morphology in order to investigate complicated fracture phenomena of coatings [8,22–24]. That is, some interfaces with non-planarity exist in initial states and a crack grows at these interfaces during thermal cycles. As this time stress concentrations are induced around the non-planar region that cause cracks to form and propagate. If there exists a sinusoidal interface in an elastic system and thermal expansion coefficient of coatings is less than substrate, tensile stress is produced at the crest of the interface and compressive stress at the valley for perpendicular direction of the interface during cooling. A fracture model for the delamination of the thin film system was proposed by Clarke et al. [26], where they assumed the propagation of the crack at the sinusoidal interface between thin film and substrate and formulated the fracture energy of the interface as the function of the initial crack size, propagated crack size and temperature. If the crack size can be known, the fracture energy can be estimated by applying their methodology. In the present experiment results, cracks were generated and propagated in the coating layer shown in Fig. 5. Therefore, we developed the method based on numerical analysis in order to apply their approach to present results.

Let us consider the elastic bi-material system which consists of coating and substrate layers with sinusoidal interface of the curvature radius $R$ and angle $\theta$. That bond layer is assumed to have the same property as substrate. An initial penny-shaped crack with radius $a_0$ exists in the coating layer at the position $h$ from the interface, shown in Fig. 9. When the specimen is subjected to uniform temperature difference $\Delta T$ over the specimen with initially stress-free state, the stress concentration occurs at the crack tip. Based on these assumptions, variation of fracture energy $G(= (1 - \nu^2)K_f^2/E)$ of the crack tip as the function of crack radius due to thermal stress was analyzed by the commercial FEM code MARC (Marc Co. Ltd). Eight node axisymmetric elements were used and the axis of symmetry was set at the center of the crest part of interface with periodic boundary conditions. Mode II stress intensity factor was not taken account of in this analysis. Material properties shown in Table 1 are also assumed for the calculation and the interface curvature radius $R = 1$ mm and angle $\theta = 60^\circ$ finite element method were used as effective values.

Fig. 10 stands for the variation of normalized stress intensity factor by $\Delta T$ as the function of crack radius $a$, where dots mean the calculated values and the solid line is the polynomial fitting curve, $f(a/R)$. The stress intensity factor increases with the increase of crack radius and decreases after reaching at the maximum value. By assuming the criterion for crack propagation as $I_C \leq I$, where $I_C$ is the fracture toughness of the ceramic layer, the condition of crack propagation using the approximated function $f(a/R)$ is rewritten as

$$\frac{I_C}{\Delta T^2} \leq \frac{1 - \nu^2}{E} f(a/R)$$

Therefore, according to the inequality of Eq. (3), crack will grow when the fracture energy is higher than the function of

![Fig. 8. Procedure for evaluation of in situ fracture toughness.](image)
the right hand of the inequality, where schematic is shown in Fig. 11. If a pre-existing crack with the size $a_0/R$ in the initial state ($\Delta T = 0$) exists and temperature starts to decrease, pop-in will occur and stop when the crack size reaches to $a_c/R$ at $\Delta T = \Delta T_C$.

We can estimate the crack size generated from the initial flaw size at the ambient temperature and fracture toughness using the above pop-in model. Fig. 12 shows the result of calculation with variation of cracking temperature and fracture toughness $\Gamma_C$. The temperature difference $\Delta T$ required for cracking is increased as $\Gamma_C$ increases. For example, $\Delta T \sim 300 \, ^\circ C$ for $\Gamma_C = 5 \, J/m^2$ is required to create a crack of radius $a_c = 0.2R$, and $\Delta T \sim 1000 \, ^\circ C$ for $\Gamma_C = 40 \, J/m^2$. Smaller $\Gamma_C$ induces larger crack for the fixed $\Delta T$. Also, the variation of generated crack size can be obtained from the variation of initial crack size, shown Fig. 12. This calculation demonstrates that the initial flaw size and the pop-in crack size have unique relationship and

\[
\frac{(1 - V^2)}{E} f(a/R)^2
\]

Fig. 9. Schematic of pop-in fracture model illustrating crack propagation and arrest in coating layer at height of $h$ from sinusoidal interface.

Fig. 10. Relationship between normalized stress intensity factor by temperature difference and normalized crack radius, which was calculated by FEM.

Fig. 11. Schematic of generation of pop-in crack due to thermal stress during cooling [26].
generated crack size is independent of cracking temperature. That is, initial crack size and fracture toughness determine the generated crack size. By combining both plots, the fracture map for pop-in fracture model can be described.

4.2. In situ fracture toughness

We can estimate the initial crack size and in situ fracture toughness of each AE event from this map because the generated crack size and the temperature difference $\Delta T$ are experimentally obtained by AE measurement and inverse analysis. The dot symbols in Fig. 12 stand for the experimental results from the AE source characterization in the case of the holding temperature of 1200 °C. It is concluded that the in situ fracture toughness of the coatings is estimated as about 40–50 J/m² and the initial crack radius is about 200 μm. This value is in good agreement with the fracture toughness obtained from DCB test or DT test for Al₂O₃ coatings [27]. Since AE events occurred during the 1st–4th thermal cycle at less than 400 °C nearly closed to room temperature, this coincidence between fracture toughness values estimated from different methods seems to be reasonable. If materials degrade after more thermal cycles or AE events occur at higher temperature, the estimated fracture toughness from AE measurement may demonstrate a significant difference from the obtained fracture map. This toughness is in good agreement with the value obtained from the other mechanical test. It was demonstrated that AE measurement by a laser technique and AE source analysis were effective for the understanding of fracture behavior of TBCs and the evaluation of in situ fracture properties such as crack radius and fracture toughness at elevated temperature.

5. Conclusions

AE measurement using a laser interferometer could evaluate the fracture behavior of coating layer during thermal cycle test, and the in situ fracture properties were evaluated by combining the AE source characterization technique and the proposed pop-in fracture model. The radius of cracks generated during the experiment was evaluated as 100–500 μm and the fracture toughness of the coatings as 40–50 J/m² from the obtained fracture map. This toughness is in good agreement with the value obtained from the other mechanical test. It was emphasized that the present methodology with proper fracture model can be used to evaluate in situ properties such as initial crack and fracture toughness of coatings during the thermal cycles.

Appendix A. General theory of acoustic emission [18]

Consider an elastic domain occupied by a given three-dimensional body. If the point source approximation can be applied, we can describe the displacement field $U_j(x', t)$ due to moment tensor $D_{jk}(x, t)$ as

$$U_j(x', t) = G_{ij}(x', x, t) \ast D_{jk}(x, t) \quad (A1)$$

where $G_{ij}(x', x, t)$ is the Green’s function that is the displacement field in the direction $x_j$ at position $x'$ at time $t$ due to an impulsive force in the direction $x_i$ at $x$ at time 0. The comma indicates a differentiation and $\ast$ means convolution.
a convolution integral with respect to time. Moment tensor $D_{jk}(x, t)$ is represented by mode and intensity of the fracture as following

$$D_{jk}(x, t) = C_{jkmn}[u_m(x, t)]v_n(x)\Delta A$$  \hspace{1cm} (A2)

where $v_n(x)$ is the normal vector to surface, $\Delta A$ is the area of surface, and $C_{jkmn}$ is the elastic stiffness tensor. For isotropic materials, $C_{jkmn}$ can be written as

$$C_{jkmn} = \lambda \delta_{jk} \delta_{mn} + \mu (\delta_{jm} \delta_{kn} + \delta_{jn} \delta_{km})$$  \hspace{1cm} (A3)

where $\lambda$ and $\mu$ are the Lamé constants, and $\delta_{jk}$ is the Kronecker’s delta. Therefore, in the case of tensile fracture, that is, $j = k = 3$, moment tensor components can be represented as

$$[D^3] = \begin{bmatrix} \frac{\lambda}{\lambda + 2\mu} & 0 & 0 \\ 0 & \frac{\lambda}{\lambda + 2\mu} & 0 \\ 0 & 0 & 1 \end{bmatrix} \Delta D$$  \hspace{1cm} (A4)

where $\Delta D$ stands for $D_{33}$.

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