# ON-PROCESS MONITORING OF CERAMICS AND CERAMIC COATINGS BY LASER AE

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ABSTRACT

Microcracks are easily generated during the processing in ceramic materials due to thermal stress because ceramics have intrinsic brittleness. It is very important to control microcracks or damages in ceramics during fabrication to ensure mechanical reliability of materials and structures. Especially it becomes necessary to avoid microcracks during manufacturing in large ceramic components which are fabricated for structural applications in recent years. Also many microcracks are easily introduced in thermal spraying of ceramic coatings which are widely used for heat resistant components. These cracks control mechanical and thermal properties of coating layer. Acoustic emission (AE) method is a powerful tool to monitor in-situ damage in materials because elastic waves are generated due to brittle cracking or frictions. Conventional AE techniques use a piezoelectric ceramic element and attach it to samples in order to detect AE signals at the surface of samples. Microfractures in materials are quantitatively analyzed by the detected waveforms such as location, size and fracture mode. However, conventional AE transducer cannot be used at elevated temperature or severe environment. Laser based ultrasonic (LBU) technique has been developed to characterize materials properties and detect flaws in materials. We have developed a non-contact AE technique to detect AE signals in various environments where conventional AE technique cannot be applied. AE during sintering of alumina ceramics and thermal spying of alumina powder on steel substrates were successfully measured by laser interferometers. The effect of processing parameters on AE behavior was clearly observed by analyzing AE waveforms. One of the most advantages of this laser AE technique is to estimate the temperature where microcracks are generated. These results could give a feed back to control processing conditions in order to avoid damage in materials. It was concluded that the laser AE method was very useful to detect microcracks in ceramics during fabrication.

## 1 INTRODUCTION

Recently, ceramics is extensively applied to structural materials because of its strength, abrasion resistance, chemical stability and so on, and ceramics components become larger and more complex. Ceramic components are produced by heating and densifing green compact at elevated temperature. These components are sometimes easily fractured during sintering due to large size and complex shape of ceramic components, so it becomes a problem that this fracture interrupts manufacturing process. It is important to control the crack initiation and propagation in green compact during sintering to resolve this problem. Furthermore, it is necessary to understand the evolution of mechanical properties during sintering to control the crack behavior [1, 2].

As the operation temperature of gas turbines has been rapidly increased to achieve high efficiency in recent years, materials are required improve their stability and mechanical properties at elevated temperature above 1773 K. Thermal barrier coating (TBC) with ceramic coating layer has been developed to shield heat from the outside and increase thermal stability of the surface [3]. As these coatings are subjected to both external stress and internal thermal stress constantly or

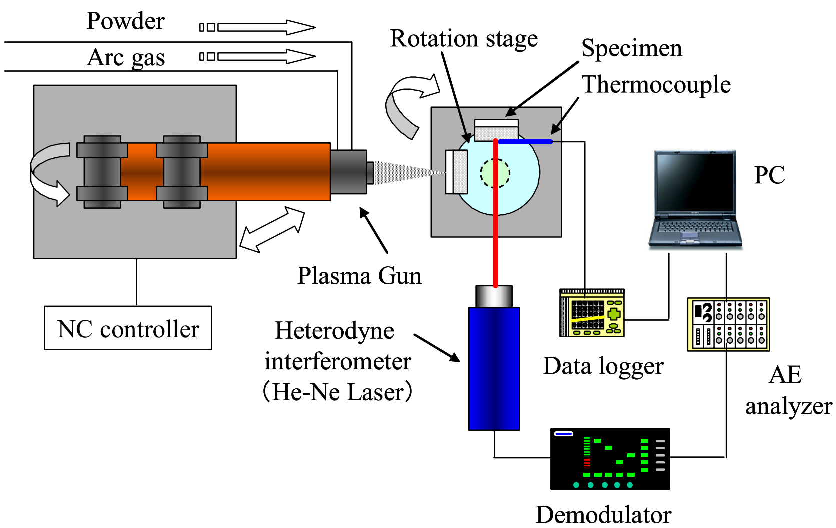


Fig. 1 Schematic figure of in-process monitoring system using laser interferometer for plasma spraying.

periodically, the evaluation of mechanical properties and failure process of TBC at elevated temperature is desired to ensure the integrity. A health monitoring of structural components in service is important to assess the integrity of TBC, and a process control is also very important to provide uniform coating thickness and maximize the coating properties, as well as an estimation of the mechanical properties of coatings such as thermal shock resistance, thermal fatigue resistance and creep resistance .

Acoustic emission (AE) technique is a promising tool for reliability assessment of materials because it can monitor the generation and growth of microcrack in real time. However, conventional contact AE technique has a limit in application at elevated temperature, because a conventional piezoelectric transducer cannot be used above about 800 K. We have investigated the non-contact AE measurement technique using laser interferometer as a AE sensor [4-7]. This laser AE technique has several advantages such as non-contact measurement, absolute velocity measurement of AE signals, and applicability for severe environment. The purpose of this study is to investigate the influence of processing parameters on the generation and growth process of defects during fabrication process of ceramics by means of an in-situ monitoring system based on laser AE technique.

## 2 EXPERIMENTAL

### 2.1 In-situ monitoring during plasma spray coatings

Experimental setup of in-process monitoring system for plasma spraying is shown in Fig. 1. A sample was fixed to the stage equipped on a turntable in the direction perpendicular to the spraying direction. This coating process consists of three stages: First is a preheating of the substrate (stage-1), second is a spraying of topcoat (stage-2), and the last stage is an air cooling of the coated specimen (stage-3). After preheating of the surface by gun stroking without powder feeding, a feeding-rate control valve was opened to start the spraying of the topcoat. When the thickness of

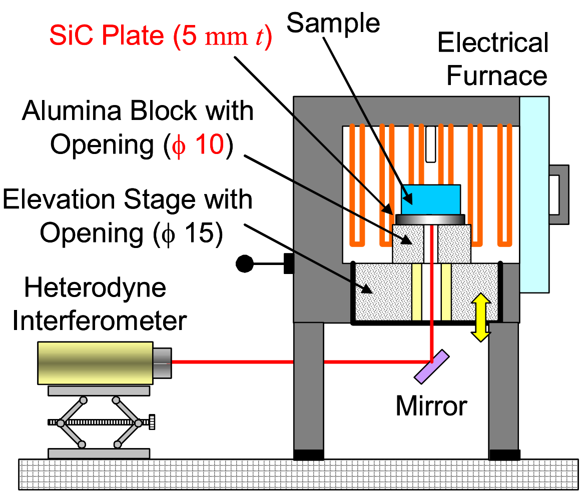


Fig. 2 Schematic figure of in-process monitoring system using laser interferometer for sintering of ceramics.

topcoat reached a desired value, the spraying apparatus was switched off and then the specimens were air-cooled until room temperature. After spraying, the sample stage was turned and the rear surface was faced to the laser beam of interferometer. AE signals during cooling period were detected using a heterodyne type interferometer (AT-0022, Graphtec Corp.). Acquisition of AE signals was started after the power source of the spraying robot, exhaust duct and compressors were switched-off to avoid the influence of mechanical or electromagnetic noise on AE signals. Some dead time for AE measurement was required to adjust the focus of laser beam on the measuring surface. Total dead time before the start of an acquisition was about 1 min. Low noise type demodulator (AT-3600S, Graphtec Corp.) was used to measure an out-of-plane surface velocity on a sample with range of 1 mm/s/V. In order to reduce noise level, output signals were filtered with high pass filter (HPF) of 50 Hz and low pass filter (LPF) of 200 kHz. Detected AE waveforms were recorded by AE analyzer (DCM-140, JT-Toshi Corp.).

2.2 In-situ monitoring during sintering of alumina

Experimental setup for sintering of ceramics is shown in Fig. 2. Pre-treated alumina green compact, 35 by 35 by 20 m, was heated by electrical furnace, and AE signals during sintering were measured by using laser interferometers. Deep notch was introduced into some samples to investigate the effect of stress distribution. Two types of sintering pattern with high and low shrinkage rate were also used to change the stress during sintering. Refractory was holed to introduce a laser beam and AE signals through SiC plate was detected by the above measuring system. Generally long waveguide is used to measure AE signals at elevated temperature in order to avoid the influence of heat on AE transducers. However, it is very difficult to characterize AE sources quantitatively because AE signals through waveguide are strongly affected by the shape of waveguide. AE signals detected by this setup have an advantage over the ones using waveguide.

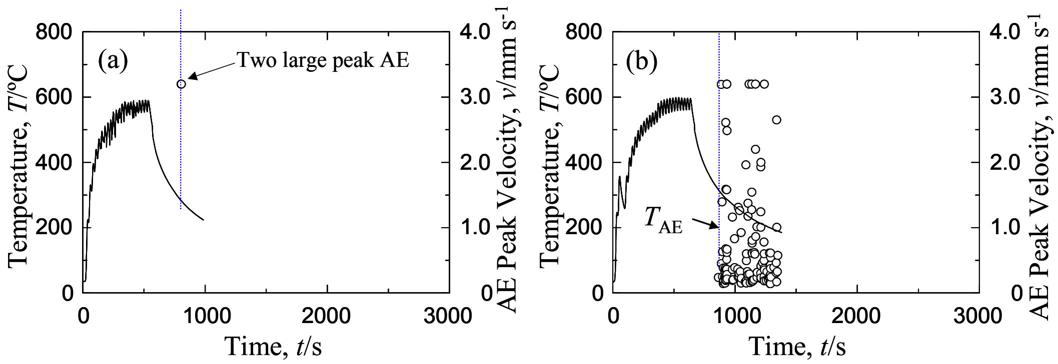


Fig. 3 Relationship between temperature history and AE behavior for different thickness of bond coat, (a) $d_{BC}$= 0 mm and (b) $d_{BC}$= 70 µm

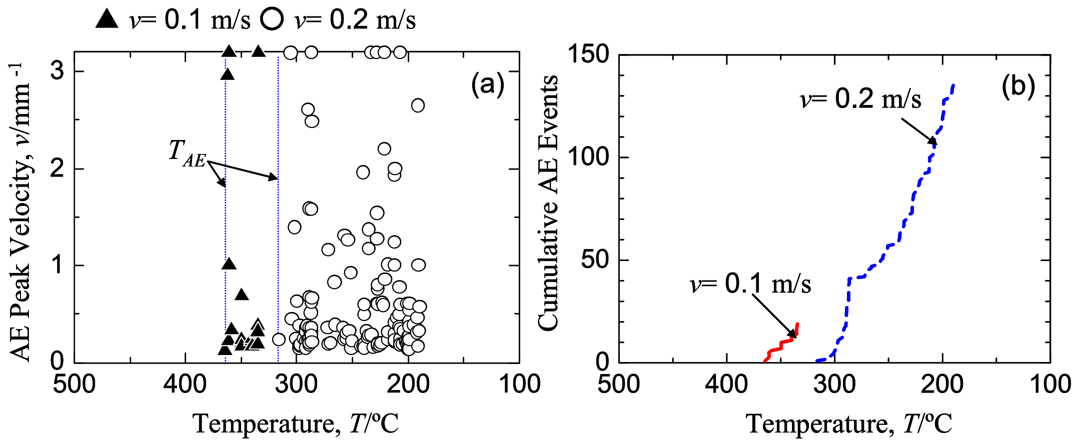


Fig. 4 Relationship between the (a) amplitude or (b) cumulative AE event and generation temperature of AE for different traverse speed of spraying gun.

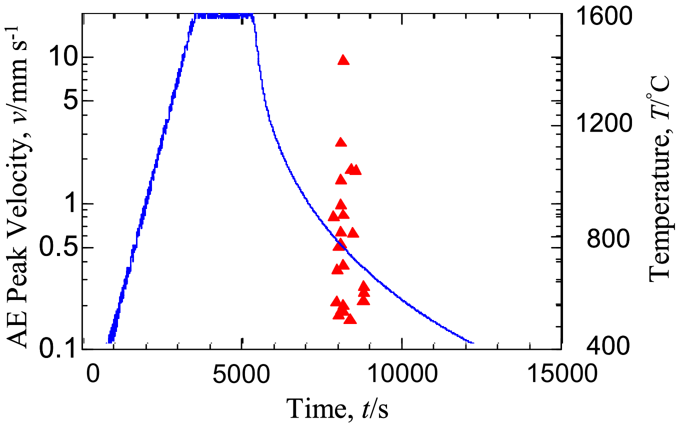


Fig. 5 Typical AE behavior during sintering of alumina detected by laser AE technique.

## 3 RESULTS AND DISCUSSION

Figure 3 shows the temperature history and AE behavior for the different thickness of bond coat, where the other processing parameters were common, that is, pre-heating temperature $T_p$ was 773 K, thickness of top coat $d_{TC}$ was 1 mm, and gun velocity v was 0.1 m/s. In the sample without bond coat, only two AE with large amplitude were detected and immediately ceramic layer was delaminated. On the other hand, a large number of AE events with various amplitudes in the sample of thickness of bond coat $d_{BC}$ of 70 μm were detected in the temperature range of 200 K before the delamination of top coat.

Figure 4 shows the relationship between the amplitude and generation temperature of AE for different traverse speed of gun, where $d_{BC}$ was 70 μm, $T_p$ was 773 K and $d_{TC}$ was 1 mm. AE generation temperature $T_{AE}$ in the sample of 0.1 m/s was higher compared with that in the sample of 0.2 m/s. The number of AE events in the case of 0.2 m/s was lager than that of 0.1 m/s, and the temperature range during AE generation of 0.2 m/s sample was wider than that of 0.1 m/s. Many interlamellar microcracks were observed in top coat of coating samples, and delamination was found near the interface between top coat and bond coat. It is difficult to quantitatively characterize the damage induced sample during coating. However, non-contact measurement of AE behavior enables to estimate the damage during coating process.

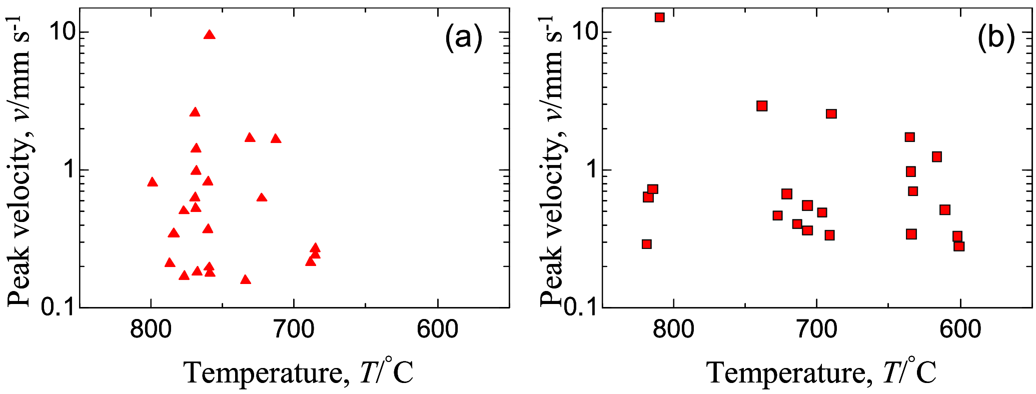


Fig. 6 AE behavior during sintering, (a) smooth specimen and (b) notched specimen.

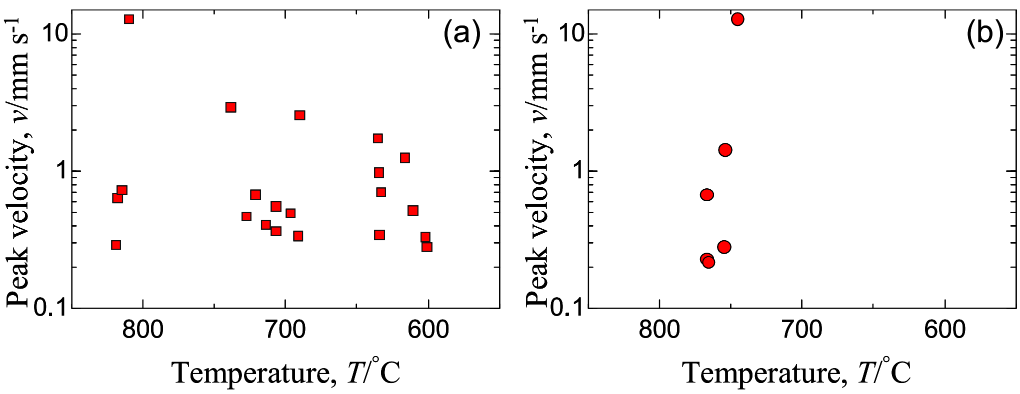


Fig. 7 AE behavior during sintering, (a) high shrinkage rate and (b) low shrinkage rate.

AE signals detected during cooling process could be classified into two types by the peak frequency, that is, Type-A and Type-B, respectively. Type-A signals have a peak around 75 kHz, on the other hand Type-B signals have several characteristic peaks in 100-200 kHz. Type-A signals were especially detected before the final delamination of specimens, and Type-B signals were broadly observed during cooling process.

Figure 5 shows a typical AE behavior in sintering of alumina, where AE generated at cooling period of processing process. Figure 6 shows the AE behaviors without and with notch. AE generation temperature of notch sample was higher than that of smooth sample, and AE continuously occurred until lower temperature. Figure 7 shows the effect of shrinkage rate of sintering on AE behaviors. AE generation temperature of high shrinkage rate, -0.171, sample was higher than that of low shrinkage, -0.143, rate one and many AE signals were detected in high shrinkage rate sample. These different AE behaviors clearly imply the stress distribution during sintering.

## 4 SUMMARY

We developed a non-contact in-process monitoring system for ceramics and coatings with laser AE technique, and applied this system to the detection of microfracture during fabrication process. Conclusions of this study are as follows:

(1) Using the developed in-process monitoring system with laser AE technique, AE generated in cooling process of plasma spraying and alumina sintering was successfully detected.

(2) Processing conditions in plasma spraying affected AE behaviors such as generation temperature, number of AE and frequency characteristics.

(3) Difference in stress distribution due to notch and shrinkage rate during sintering of alumina also induced different AE generation temperature.

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