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AE measuring system at high temperature for ceramics with low thermal expansion due to microcracking

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Materials having an excellent heat shock resistance have been required for industrial products such as furnaces and heat engines receiving repeated heat shock. Aluminum titanate is promising as such a material. However, the low thermal expansion of this material results in microcracking due to thermal expansion anisotropy, so its strength is low. Therefore, it was planned to develop the material having both low thermal expansion and high strength by compositing aluminum titanate and mullite with high strength, and intermediate thermal expansion. In order to make the composite having this property, it is necessary to find out the process conditions at the optimal occurrence of microcracking by changing variously the process factors of composition ratio, sintering temperature, and raw-material particle size. AE (acoustic emission) technique can observe microcracking occurring in the interior of a material under real time, and so it is convenient for catching microcracking during cooling. Moreover, if an AE signal is analyzed, more detailed information on microcracking may be obtained and it may be expected to find out the new measures for a material evaluation. For aluminum titanate ceramics, Ohya *et al*. investigated experimentally the energy criterion of microcracking by measuring AE count rates and expansion rates during cooling [1], and it has been shown that the frequency analysis of AE waveforms is effective in the material evaluation of fiber-reinforced composites [2]. However, there have hardly been any studies on AE source waveform analysis in the ceramics for high temperature because of the difficulty of AE measurements at high temperatures. Then, firstly, the authors developed the apparatus that can automatically measure AE event counts, AE waveforms, and thermal expansion in the low thermal expansion material of microcracking-type. This paper reports the developed apparatus and the preliminary experimental result of an aluminum titanate–mullite composite.

The specimen having 85/15 wt% in the composition ratio of aluminum titanate (AT) to mullite (Mu) was prepared for a preliminary experiment: Powders of aluminum titanate with particle size 3 μm and mullite with particle size 0.8 μm were mixed, and after adding 0.5 wt% dispersant, the mixed powder was ball milled for 24 hr, and then the suspension was formed into a cylindrical bar by vibration slip casting. The cast sample was sintered at 1550 °C for 2 hr. The sintered body was 15 mm in diameter and 30 mm in length.

The measuring apparatus is shown in Fig. 1. The specimen (A) was set up in a furnace (D) and heated up to 1200 °C at a rate of 6 °C/s. No temperature control of the furnace during cooling was made in order to avoid a furnace noise. The reasons why the maximum temperature was set up in 1200 °C were as follows: Ohya *et al*. reported that the crack healing temperature of aluminum titanate ceramics was 1100 °C [3], and our experiment at temperatures above 1200 °C for an aluminum titanate–mullite composite showed no difference with Ohya's result. The expansion of a heated system composed of an alumina rod (B), composite specimen and alumina tube (C), whose diameters were 15 mm, was measured by a laser displacement meter (E) attached to the upside of a measuring apparatus. The expansion of the two parts of the rod and tube was measured in another experiment and the expansion of composite specimen only was calculated automatically from the two expansions of the heated system and the two parts by a personal computer (M). The standard temperature of thermal expansion was 1200 °C. AE waves were detected by an AE sensor (F) through the alumina rod of a wave-guide. It is one of the objects in this study to make a frequency analysis, and so a frequency wide band type PZT was used for the AE sensor. The detected AE waves were amplified by 60 dB by a pre-amplifier (G) and a discriminator (H). Ohya *et al*. counted AE events every 10 s [1]. In this study on an aluminum titanate–mullite composite, AE events were counted every 30 s because the temperature occurring in AE was unclear and the measurement interval of 10 s was too short. The judgment of event unit was very difficult because of noises and AE reflections included in the detected signal. Then, for the judgment, the system of a pair of discriminating levels, low level (VL) and high level (VH), was adopted, and the threshold voltages were set up in VL = 50 mV and VH = 80 mV considering noise levels and AE waveforms. As described later, the component of AE waves hardly existed at low frequencies below 100 kHz and at high frequencies above 1 MHz. Therefore, the detected signals were passed through a high path filter having the cutoff frequency of 100 kHz and were sampled at the rate of 2.5 Ms/s by a digital oscilloscope (I).

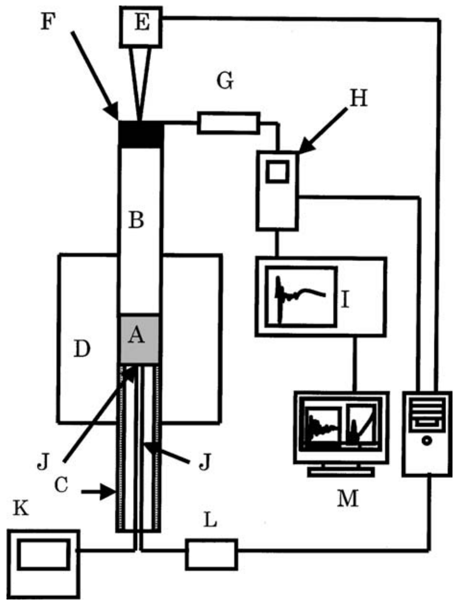

Figure 1 Measuring apparatus: (A) specimen, (B) alumina rod, (C) alumina tube, (D) furnace, (E) laser displacement meter, (F) AE sensor, (G) pre-amplifier, (H) discriminator, (I) digital oscilloscope, (J) thermocouple, (K) temperature controller, (L) digital thermometer, (M) personal computer.

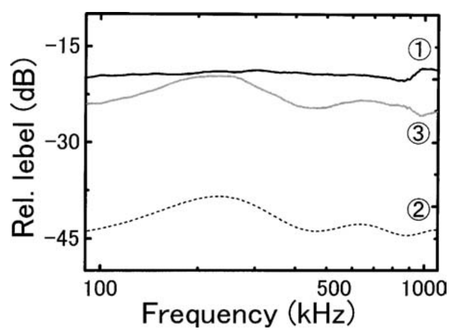

Figure 2 The transfer function of alumina rod at 600 °C: ① frequency characteristic of AE sensor, ② frequency characteristic of alumina rod at 600 °C including AE sensor, ③ transfer function.

The AE signal which passes through the alumina rod is influenced by not only damping but also dispersion due to it. Therefore, it is necessary to correct the frequency analysis result of observed AE waveforms by the transfer function of alumina rod. Since the alumina rod is under complicated temperature conditions, the following two experiments were made in order to obtain the transfer function: (1) Two AE sensors were contacted directly, and then the frequency characteristic of AE sensor shown by the ① in Fig. 2 was measured by inputting a pulse to one side and detecting at the other side. (2) The alumina tube was changed to an alumina rod, and then two AE sensors were contacted to each end of the two rods, and after heating the rods at 300 or 600 °C, the frequency characteristics of alumina rod at each temperature including the AE sensor were measured using the same method as above (1). The results showed that there was little difference between the frequency characteristics at each temperature. The frequency characteristic of alumina rod at 600 °C including the AE sensor is shown by the ② in Fig. 2. The transfer function of alumina rod shown by the ③ in Fig. 2 was found by calculating from two frequency characteristics of ① and ②. In addition, the ② in Fig. 2 was processed using a curve approximation by computer because of the obscurity of measured data.

A preliminary experiment was made for the specimen with the composition ratio of 85/15 in the weight ratio of aluminum titanate to mullite. The measured results of AE count rates and expansion rates during cooling are shown in Fig. 3. The vertical axis of the left-hand side expresses the AE event counts every 30 s and that of the right-hand side expresses the change of expansion rate. It is observed that many AE waves occur near 600 °C where contraction changes into expansion. This shows well the characteristic of aluminum titanate that the thermal expansion becomes small apparently because of the occurrence of microcracking. An example of detected AE waves is shown in Fig. 4. Detected AE waves were analyzed by performing fast Fourier transformation (FFT) in the frequency range of 0.1–1 MHz

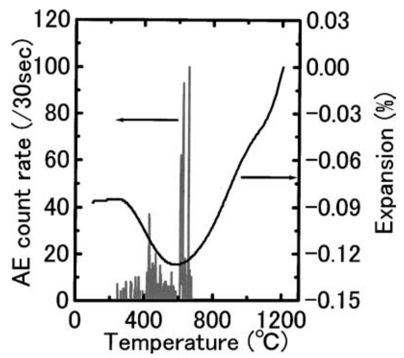

Figure 3 The thermal contraction and expansion curve and the acoustic emission count rate of the aluminum titanate–mullite composite with 85/15 composition ratio on cooling after heating at 1200 °C.

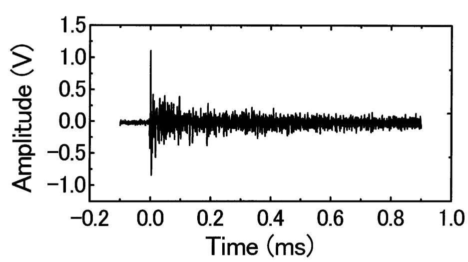

Figure 4 An example of AE waves.

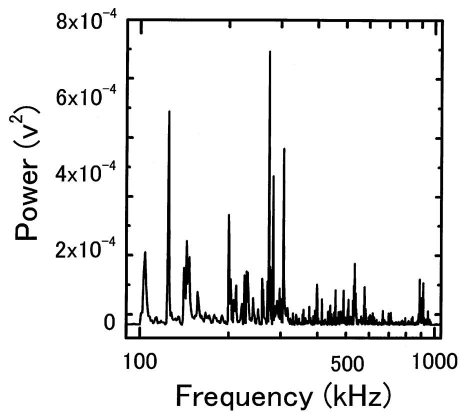

Figure 5 An example of power spectra in AE waves, which was corrected by transfer function.

and corrected using above transfer function. An example of the results is shown in Fig. 5. It is observed that the components of AE waves concentrate near 300 kHz.

It became possible by this apparatus to automatically measure the AE event counts, the AE waveforms, and the thermal expansion during cooling in the ceramics with low thermal expansion due to microcracking. Hereafter, it is scheduled to measure a few of AE parameters and bending strength in aluminum titanate–mullite composites made by changing variously the composition ratio, sintering temperature, and raw-material particle size, and to find the strength evaluation method and the optimum process conditions by considering the relationships between AE parameter and bending strength.

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