Temperature dependence of scanning electron acoustic microscopy signal in MgO and SiC

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The variation of the electron acoustic signal as a function of temperature in ceramic materials has been studied. Scanning electron acoustic microscopy (SEAM) contrast in MgO single crystals and in reaction bonded SiC, between 100 and 273 K, is discussed and compared. SEAM signal and contrast have been found to be temperature dependent in both materials as a consequence of the temperature dependence of several material parameters.

I. INTRODUCTION

Scanning electron acoustic microscopy (SEAM) has been used as a simple technique to study many different kinds of materials.[1,2] SEAM is based on the production of acoustic waves in the specimen surface due to the interaction between a solid and the primary electron beam. The acoustic waves are detected by a transducer and used to form a scanned image. The electron-acoustic signal depends on the experimental conditions of the scanning electron microscope and on the properties of the material. This technique has been widely used by varying the chopping frequency[3] in order to get information about near-surface and subsurface features in the material under study. The aim of this work is to show how the temperature can be used as a new parameter to get subsurface information.

SEAM has been previously applied to the characterization of MgO single crystals.[4,5] In the present work the temperature dependence of the SEAM signal in MgO single crystals and SiC polycrystals has been studied and the different behavior of both materials have been compared.

II. EXPERIMENTAL METHOD

The MgO single crystals used were grown by W. & C. Spicer and Co. with purity of 99.9% or 99.99%. The crystals were cleaved along (100) faces. Some samples were indented with loads of 200 g with a diamond pyramid by using the MHP microhardness attachment of a Zeiss optical microscope. The samples were coated with a transparent carbon film. The SiC samples, with a thickness of about 1 mm, were cut from a 5-mm-diam reaction bonded SiC rod (Goodfellow Metals Ltd.) and mechanically polished. Scanning electron-acoustic observations were performed on a Cambridge S4-10 scanning electron microscope.

The experimental SEAM arrangement used in this work has been previously described.[4] A square wave generator provided chopping frequencies from 40 to 240 kHz and the electron-acoustic signal was detected by a piezoelectric ceramic transducer (PZT). The acoustic signal was measured by earthing and nonearthing the specimen-transducer interface but the SEAM temperature dependence was measured with the upper side of the sample earthed and the specimen-transducer interface unearthed in order to prevent the detection of the transducer resonance spectrum. The specimen-transducer assembly used is similar to that described by Balk and Kultscher[6] but was modified to allow the SEAM signal detection in the S4-10 sample cooling module. Both PZT transducer and bottom electrode were ring-shaped and mounted directly in a copper SEAM sample holder which had to be redesigned to assure a good cooling of the sample. The sample holder is in contact with a copper cold finger, one end of which is immersed in liquid nitrogen. The sample is clamped on the top of the annular tranducer. The temperature was varied between 100 and 273 K.

III. RESULTS AND DISCUSSION

Figure 1 shows the temperature dependence of the SEAM signal in MgO single crystals and in SiC polycrystals. Measurements of different samples revealed that the shape of the curves of Fig. 1 as well as the general features of SEAM images described below, are reproducible. While the signal in MgO increases continuously by decreasing the temperature in SiC the signal reaches a maximum at about 190 K. Observation of the SEAM images in the mentioned temperature range shows that not only the signal intensity but also the SEAM images of a given sample region vary with temperature. Figure 2 shows a sequence of SEAM images of an indent in MgO, recorded at 300, 190, and 130 K, respectively. When the interface electrode is earthed, as was the case of the crystal shown in Fig. 2, a substantial part of the SEAM information is lost and only the contrast from highly deformed regions remains in MgO.[4] The temperature dependence of the contrast indicates that different regions of the crystal contribute to the SEAM image when the sample temperature changes. If one assumes that the thermoelastic coupling is the main signal generation mechanism in MgO under the present observation conditions[4] a rough estimate of the crystal slab thickness where the signal is generated, can be made by calculating the value of the thermal decay length d_t at different temperatures. By introducing the values of specific heat C and thermal conductivity K in the equation $d_t = (2K/\omega\rho C)^{1/2}$ (ρ is the density and ω the angular frequency) the values of 2 and 27 μm for d_t at 273 and 100 K, respectively, at 200 kHz are obtained. The marked temperature dependence of d_t would explain the contrast changes observed in the micrographs of Fig. 2. Although the SEAM contrast variations is the most significant effect of decreasing the temperature in MgO, the increase of signal at low temperatures is also of interest, in particular in cases of

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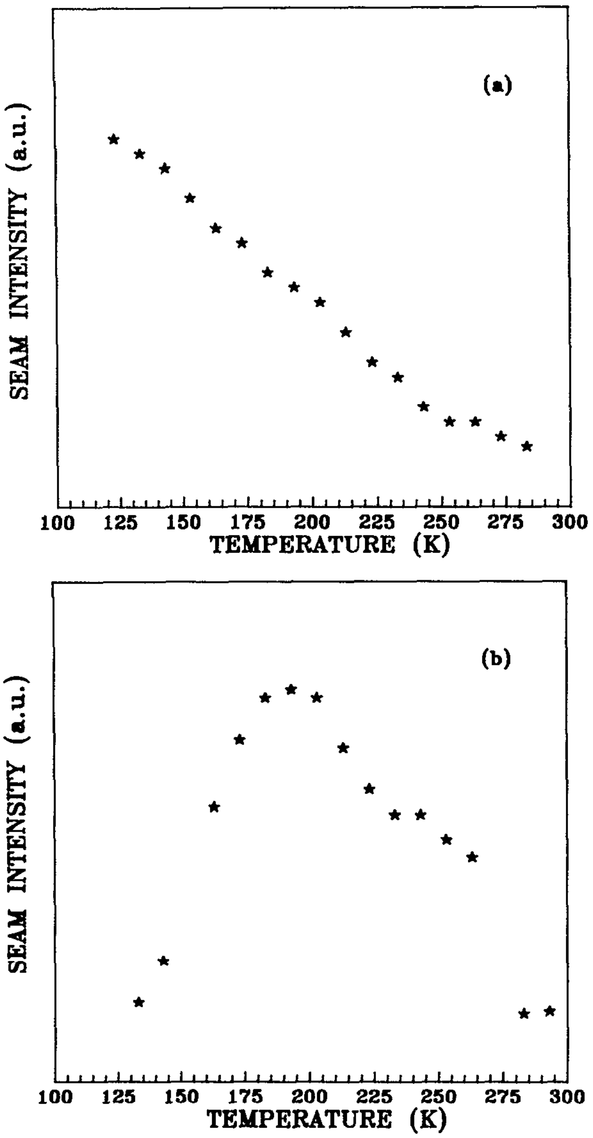

FIG. 1. Temperature dependence of SEAM signal intensity for (a) MgO single crystal, (b) SiC polycrystal.

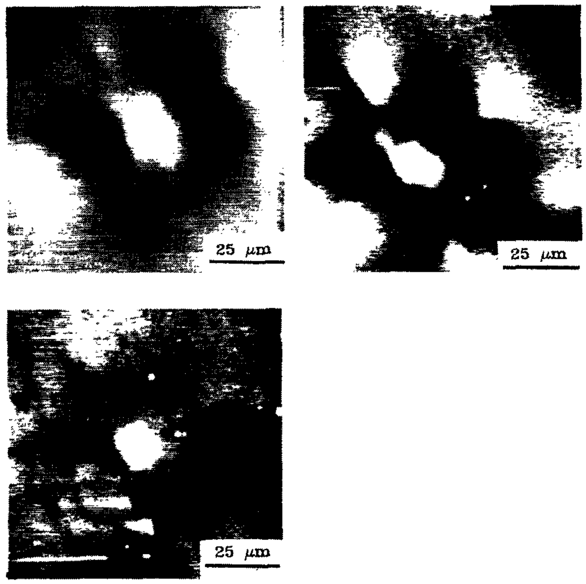

FIG. 2. MgO indented sample. SEAM amplitude images at 26 keV and (a) 121.06 kHz and room temperature, (b) 188.89 kHz and 193 K, (c) 188.99 kHz and 133 K.

low signal levels. The variation of electron acoustic signal as a function of temperature is, however, material dependent as Fig. 1 shows. In the case of thermoelastic coupling, the amplitude of the force for the generation of acoustic waves depends on material properties such as elastic constants, coefficient of thermal expansion, heat capacity and thermal conductivity.[3,7] Since all these properties depend on temperature the electron acoustic signal will depend on temperature in a different way for different materials. If in addition to the thermoelastic coupling other signal generation mechanisms such as excess carrier coupling in semiconductors are present, the temperature dependence of electron acoustic signal can be more complex. These factors would explain the fact that not a common shape has been found for the temperature dependence curves of electron acoustic signal in MgO and SiC (present work) and in Si.[8]

Figure 3 shows the emissive mode and SEAM images obtained with a beam voltage of 30 keV of the same area of a SiC sample at room temperature. In the observed area an indent had been produced. While in the emissive mode image the grain structure and the indent are clearly visible, the general structure cannot be recognized in the SEAM image although a dark region can be associated to the indent. Contrary to the present observations the SEAM technique has been previously found to be suitable to image crystal grains.[9,10] In the present case, however, the grain size of about 10 μm is small compared with the calculated value of d_t (22 μm) at room temperature and 200 kHz. We suggest that the SEAM image of Fig. 3 is formed by an average signal generated in a layer whose thickness includes several grains and pores. Since the grain structure in reaction bonded SiC is rather complex[11,12] and includes intergranular phases and pores the final SEAM image does not provide direct information about the grain structure. In order to reduce the acoustic signal generation thickness, SEAM images were recorded after reducing the accelerating voltage to 10 keV.

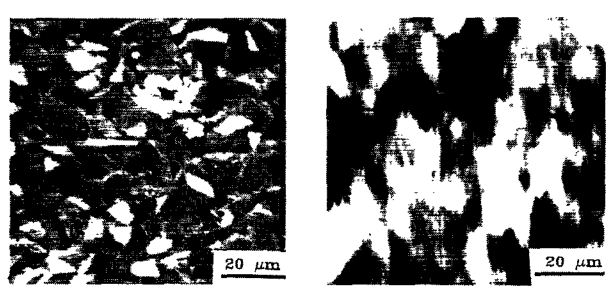

FIG. 3. SiC indented sample (a) secondary electron image, (b) SEAM amplitude image at 27 keV and 229.42 kHz.

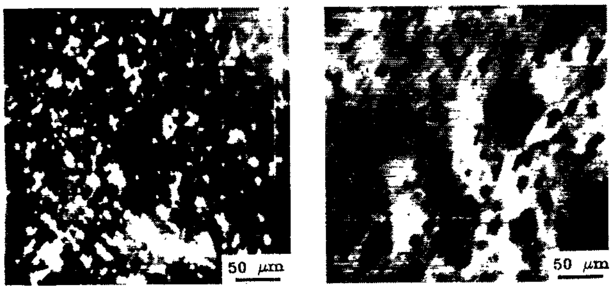

FIG. 4. SiC sample (a) secondary electron image, (b) SEAM amplitude image at 10 keV and 47.22 kHz.

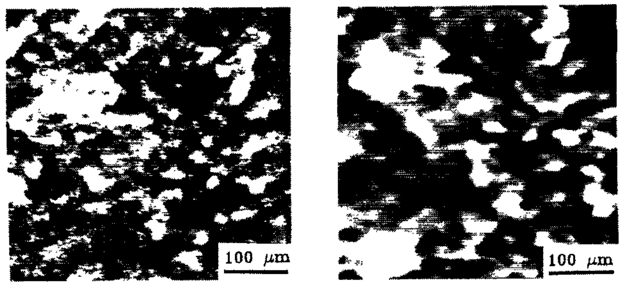

FIG. 5. SiC sample. SEAM amplitude images at 30 keV, 140.26 kHz and (a) room temperature, (b) 133 K.

Although the grain structure is still not revealed, comparison of emissive mode and SEAM images, as those of Fig. 4, indicates that the dark regions in SEAM image are related to the presence of pores. Region marked *A* in Fig. 4(b) is an indent. The dark contrast would arise from cracks, microcracks, pores and deformed regions in and around the indent.

As in MgO, decreasing the temperature increases d_t and therefore the signal generation thickness should increase. Figure 5 shows SEAM images of the same sample area at room temperature and at 130 K, respectively. Some contrast changes, as the broadening of dark zones by decreasing temperature, are observed. On the other hand changing the frequency at room temperature, in the range of 40–240 kHz did not produce contrast changes. In some materials, nonlinear processes in the SEAM signal generation mechanism lead to signals at harmonics of the chopping frequency.[6] By tuning the detector frequency to $2f$, nonlinear images are obtained that can show improved spatial resolution and new contrast features. Nonlinear images in MgO have been previously described[4] while in the present work nonlinear $2f$ signals in SiC were not detected.

The present results show that in SiC, sample temperature and beam energy are the main factors determining the SEAM signal generation depth. In reaction bonded SiC, SEAM images reveal a distribution of pores or open spaces in the sample while details of grain structure could not be resolved which is due to the high d_t value as compared with the grain size in this material. The observations in both kinds of samples used here indicate that decreasing the temperature can be useful, in some materials, to increase the intensity of SEAM signal and to increase the information depth. On the other hand resolution is decreased by lowering the temperature in MgO and SiC and this fact makes the technique unsuitable to study polycrystalline structures with small grain size.

ACKNOWLEDGMENTS

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