Signal generation mechanisms in scanning-electron acoustic microscopy of ionic crystals

M. Urchulutegui and J. Piqueras
Departamento de Fisica de Materiales, Facultad de Fisicas, Universidad Complutense, 28040 Madrid, Spain

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MgO crystals have been studied by scanning-electron acoustic microscopy under different experimental conditions. Contrast mechanisms in imaging are discussed and compared. The experimental results obtained by earthing or nonearthing the specimen-transducer interface suggest the existence of a signal generation mechanism that is related to the ionic nature of these kind of crystals. Electron-acoustic microscopy appears then to be a useful tool for the characterization of ionic materials.

INTRODUCTION

Scanning-electron acoustic microscopy (SEAM) was developed in 1980 (Refs. 1 and 2) and has been mainly used in the last few years in the characterization of metals and semiconductors. Recently,[3] SEAM has been also applied to the observation of different surface and subsurface features in MgO crystals. It was concluded in Ref. 3 that the results obtained cannot be explained only by a thermal mechanism of acoustic signal generation but also nonthermal mechanisms have to be considered. In this work the SEAM contrast of subgrain boundaries and deformed regions of MgO single crystals is further investigated under different experimental conditions in order to get information about the nature of SEAM signal generation mechanisms in ionic crystals.

EXPERIMENTAL METHOD

The MgO single crystals used were grown by W. & C. Spicer with a purity of 99.9% or 99.99%. The crystals were cleaved along (100) faces. Some samples were indented with loads between 20 and 200 g with a diamond pyramid by using the MHP microhardness attachment of a Zeiss optical microscope. SEAM observations of the samples were performed on a Cambridge S4-10 scanning-electron microscope.

For SEAM measurements a chopping system consisting of a pair of condensor plates and beam blanking electronics to create a periodically modulated beam is used. A square wave voltage with frequencies up to 240 kHz is produced by a function generator. The sound signal is detected by a piezoelectric ceramic transducer (PZT) on which the samples are clamped. The specimen-transducer assembly is similar to that described by Balk and Kultscher.[4] The amplification is carried out by a low-noise preamplifier, a lock-in amplifier receiving the reference signal from the function generator, and a video amplifier. The signal was detected at the reference frequency f or at $2f$. The acoustic signal was measured either with the upper side of the sample earthed and the specimen transducer interface unearthed or with both sample surfaces earthed. Further details on the SEAM system are given in Ref. 3.

RESULTS

The SEAM images of the subgrain boundaries, obtained when the interface electrode was unearthed, show a dark boundary with bright bands at the sides as previously described.[3] No significant changes in the width of the subboundary image are observed when the chopping frequency is increased from 40 to 240 kHz. When both sample surfaces are earthed the electron acoustic signal decreases between two and three orders of magnitude. In this case the subboundary shows a blurred contrast and the bright lateral bands are not observed. By increasing the frequency, the linear (detection at the chopping frequency f) image of the subboundary appears better resolved (Fig. 1). In the nonlinear (detection at the frequency $2f$) mode the image of the subboundary (Fig. 2) seems to be rather independent of the chopping frequency.

By imaging deformed regions, the difference in the SEAM signals obtained in the two conditions (interface electrode earthed and unearthed) is much more striking than in the case of subboundaries. Figure 3 shows the corresponding images of an indent. Although the topography contributes to the image when the electrode is unearthed, many of the features observed in Fig. 3(a) are of acoustic origin as described in Ref. 3. By earthing, most of the electroacoustic information is lost but part of it remains, causing the dark acoustic rossette of Fig. 3(b). A correspondence between dark SEAM contrast and deformed regions is observed also in crystals damaged as a result of cleaving as Fig. 4 shows. The deformed region is identified through its association with high-cathodoluminescence emission.[5]

DISCUSSION

The SEAM generation mechanism is typically explained by the conversion of an electron-beam-induced heat distribution into sound by means of the thermal expansion coefficient (thermal coupling) but evidence has been found[6] of electrostrictive and piezoelectric couplings which are nonthermal mechanisms. Since MgO has no piezoelectric properties the latter mechanism will not be discussed further. The electrostrictive coupling is basically nonlinear [6] and we therefore assume that in our linear images the elec-

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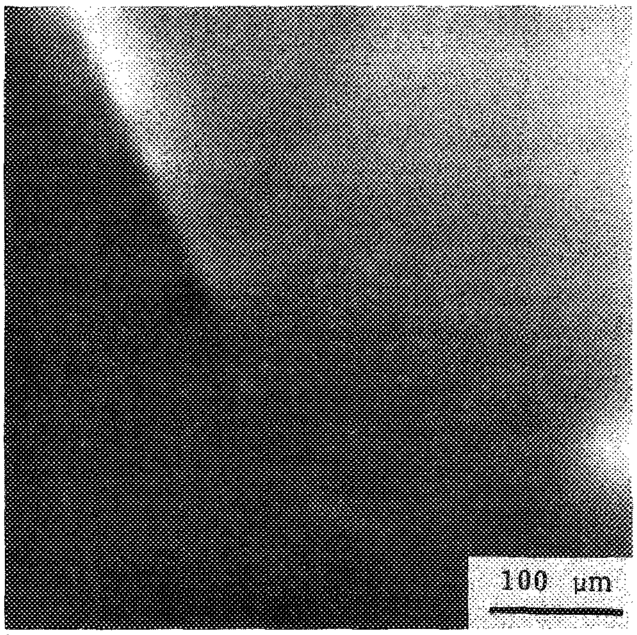
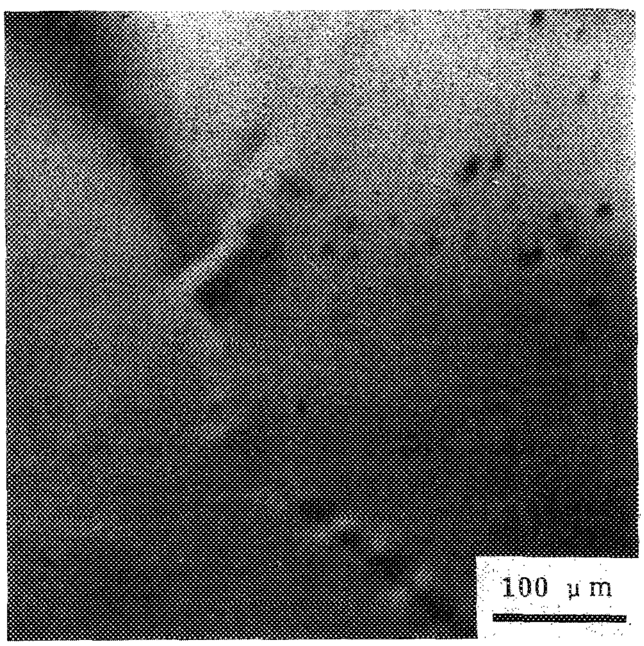
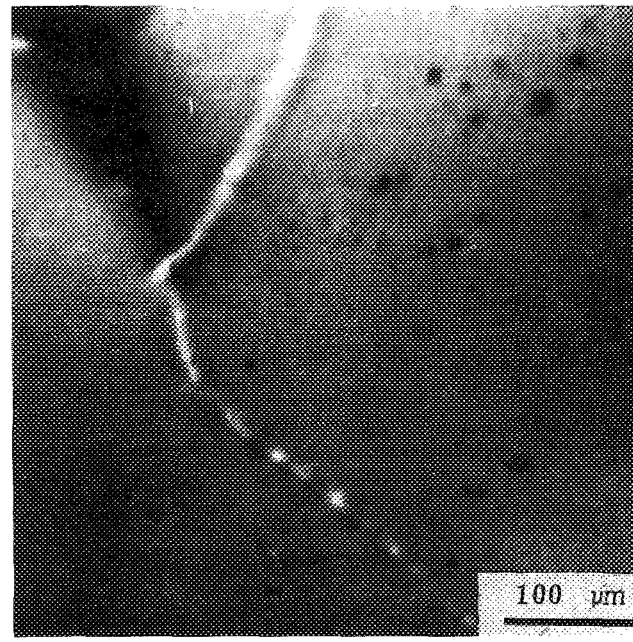


(a)



(b)



(c)

FIG. 1. SEAM image of subgrain boundaries (specimen-transducer interface earthed). Linear SEAM amplitude image at 25 keV and (a) 41.32, (b) 123.36, and (c) 183.61 kHz.

trostrictive contribution can be neglected. On the other hand, the electron beam can generate space charges in the sample causing the generation of an internal EBIC signal that contributes to the transducer output. This nonacoustic contribution is eliminated when both sample surfaces are earthed. Thus, in the linear image obtained with unearthed interface electrode only signals generated by thermal coupling and an EBIC-type component would be, in principle, present.

Up to now the possibility of a contribution to the specific SEAM signal for ionic crystals has not been discussed. However, the ionic motion associated to the sound wave could cause an electric field contributing to the transducer output. In that case the electric field should be associated with acoustic modes of vibration because these are the only modes related to dilation.[7] A similar effect occurs, in fact, in piezoelectric materials where the strain accompanying the wave produces an additional electric field component.[8,9] Equations of lattice wave in ionic crystals show that, in principle, the polarization is nonzero for optical modes and is zero for acoustic modes,[10] but it has been pointed out[11] that in some centrosymmetric ionic crystals, especially those with different ion masses, a polarization wave is associated with long-wavelength acoustics rather than the optical vibrations of the lattice. It seems then that, regardless of the particular mechanism, the possibility of the "ionic" contribution to the acoustic signal detected in the transducer cannot be ruled out. This is discussed in the following in terms of the present results.

In the thermal coupling model the resolution of the

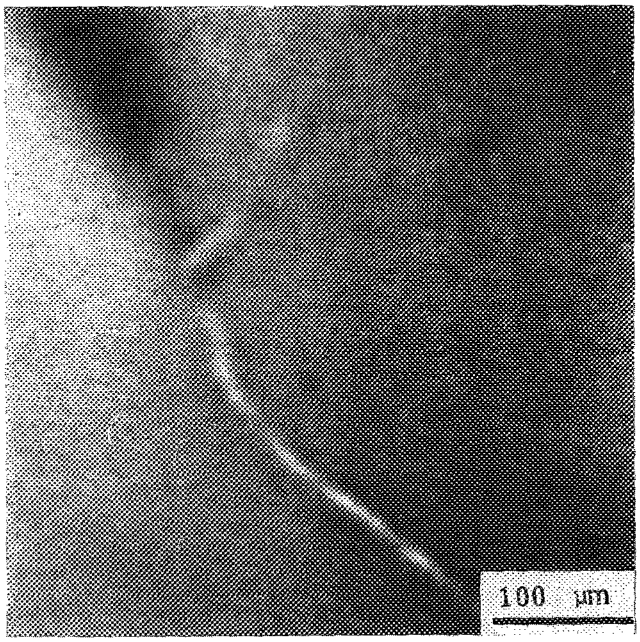


FIG. 2. Subgrain boundaries (specimen-transducer interface earthed). Nonlinear (2*f*) SEAM amplitude image at 25 keV and 121.72 kHz.

SEAM image is proportional to $\omega^{-1/2}$ ($\omega = 2\pi f$). However, as mentioned above, the width of the subboundary image does not vary with the frequency indicating that the thermal mechanism does not determine the contrast. This could be explained by the existence of nonthermal mechanisms or by a high stray (EBIC) signal. The latter possibility is supported by the fact that when the electrode is earthed the signal drastically decreases and the subboundary width shows a certain frequency dependence. It then appears possible that thermal coupling is one contrast mechanism of subboundaries and the fact that nonlinear images are obtained suggests the existence of electrostrictive coupling as well.

In the image of an indent [as that shown in Fig. 3(a)] obtained with unearthed interface electrode, the main features have been shown[3] to be real electroacoustic signals. The appearance and resolution of the image changes somewhat with frequency, indicating the existence of a thermal coupling mechanism. The fact that most of the acoustic signal is lost by earthing, as a comparison of Figs. 3(a) and 3(b) shows, indicates that an electric field signal of acoustic origin is present. Since we are not dealing with piezoelectric material we suggest that an ionic contribution to the acoustic signal possibly in the form of a polarization wave as mentioned above, is present in these kind of crystals. With the existence of an ionic coupling it follows that when observations are performed with the interface electrode earthed, a substantial part of the electroacoustic information is lost. Figures 3 and 4 suggest that, in deformed crystals, only a signal from highly deformed regions remains when the electrode is earthed.

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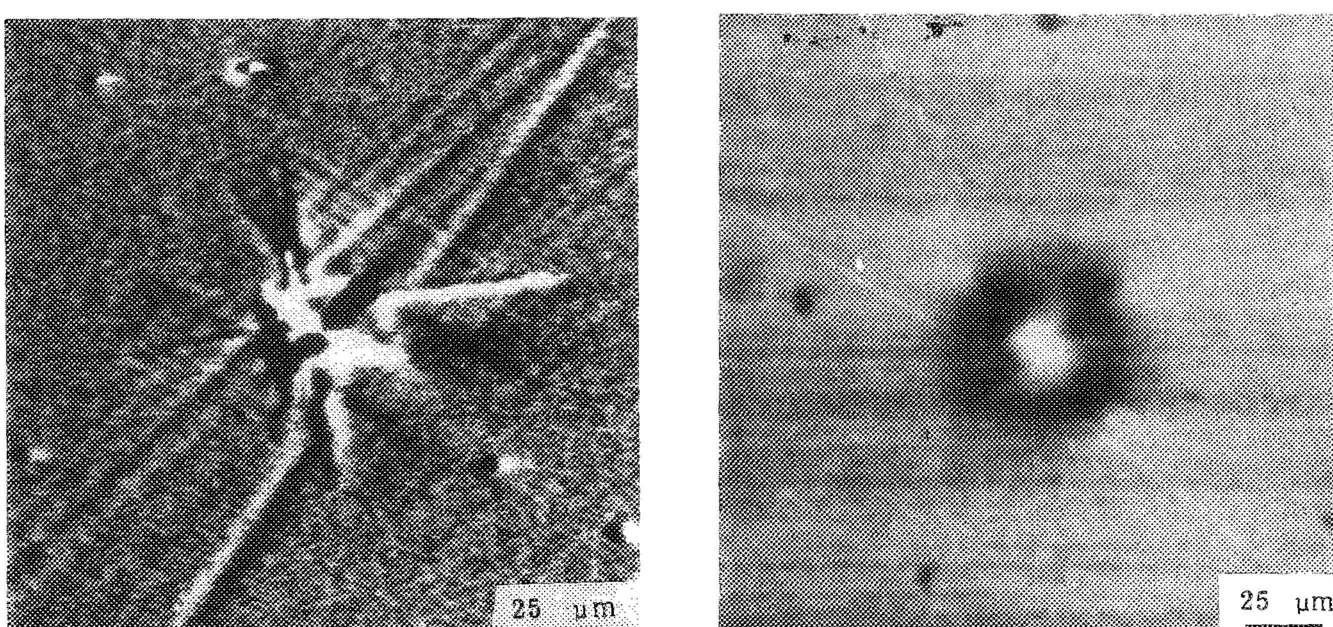

(a) (b)

FIG. 3. Indented sample. Linear amplitude image at 20 keV and (a) 70 (unearthed) and (b) 197 kHz (earthed).

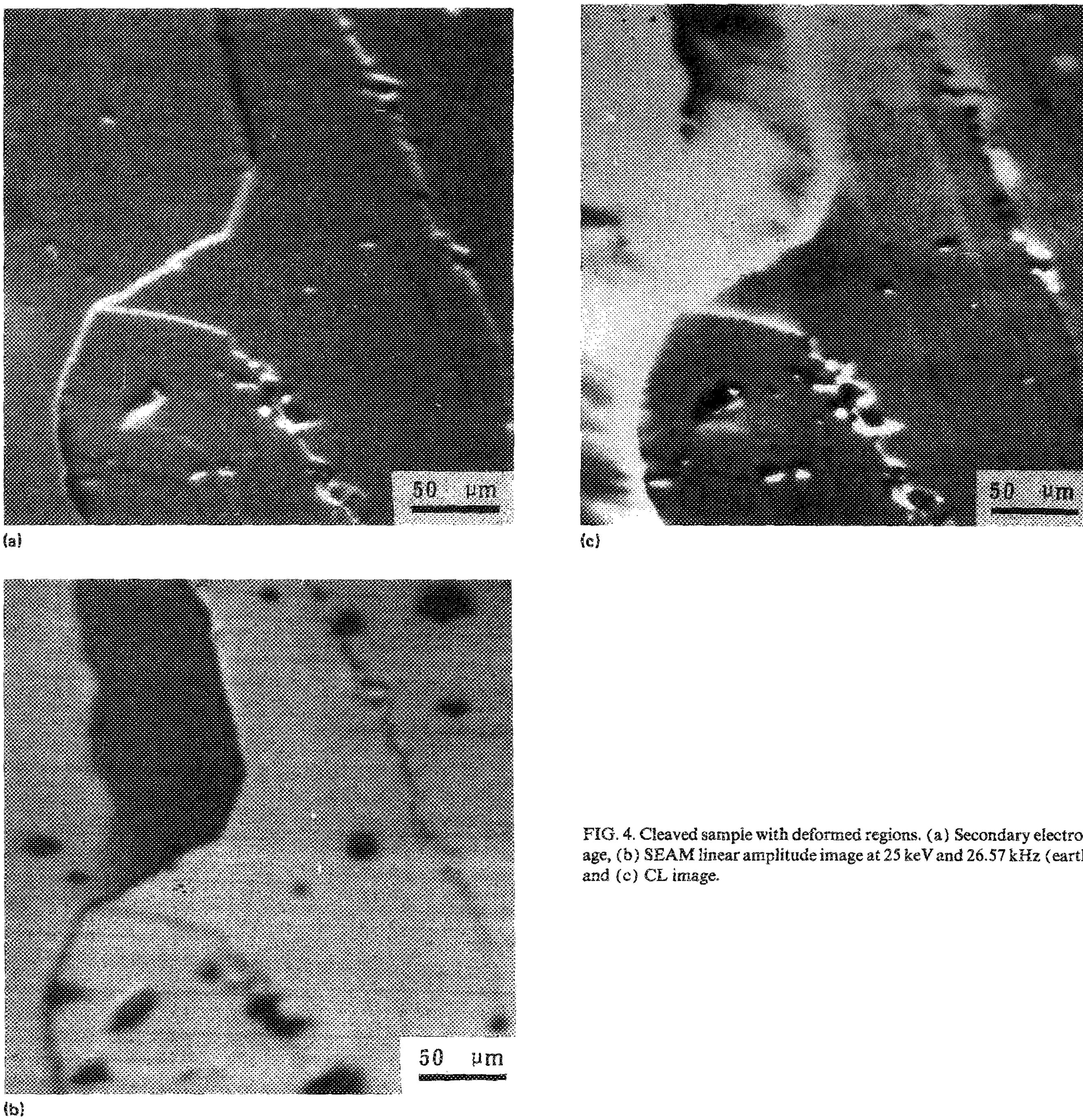

FIG. 4. Cleaved sample with deformed regions. (a) Secondary electron image, (b) SEAM linear amplitude image at 25 keV and 26.57 kHz (earthed), and (c) CL image.

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