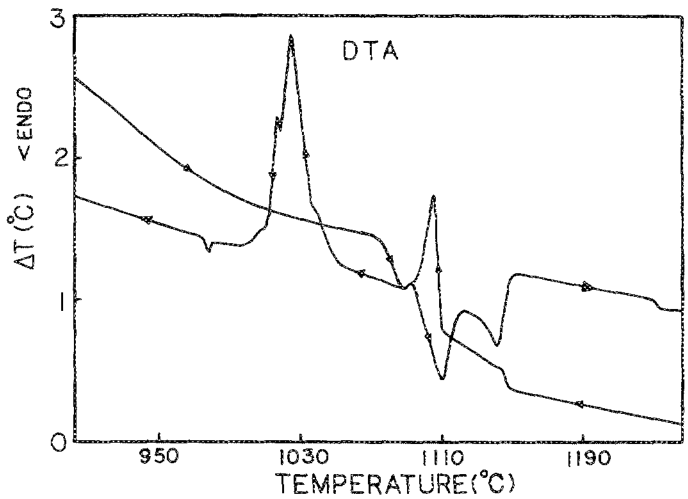

FIG. 5. Differential thermal analysis of the as-cast Pr-Co-B alloy used for magnet fabrication.

pound is approximately 100 kOe. The shoulder that exists in the low negative values of the magnetic field of the hysteresis loop (Fig. 1) is most likely due to the presence of the nonuniaxial $PrCo_4B$ phase.

Figure 4 shows two optical micrographs. The first [Fig. 4(a)] corresponds to a low coercivity (2.5 kOe) magnet, sintered at a low temperature (~1060 °C). The second corresponds to a high coercivity (5.2 kOe) magnet, sintered at a high temperature (~1100 °C). The low coercivity magnet shows high concentration of voids and small, irregular, non-well-defined grains. On the other hand, the high coercivity magnet shows a low concentration of voids and large, very well-defined grains. These microstructural features can be interpreted by examining the differential thermal analysis (DTA) of the starting as-cast alloy of the same composition shown in Fig. 5. This figure shows at least three endothermic processes occurring upon heating the material above approximately 1070 °C. Although a detailed investigation to precisely identify these processes has not yet been carried out, we believe that they involve some partial melting. Accordingly, at high sintering temperatures the presence of a liquid phase appears to have facilitated the observed densification and extensive grain growth under near equilibrium conditions. Physical observation of the magnets after sintering at high temperatures, strongly suggests the idea of the presence of a liquid phase during sintering.

The results reported above are preliminary and optimization studies are under way. These will be focused on alloy composition and a suitable optimization of the processing conditions. The effect of the presence of other phases will be examined. Emphasis will also be directed at acheiving highly dense magnets with small grains. We believe that this may lead to further enhanced values of intrinsic coercivity comparable with those of the Fe-containing magnets.

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Scanning electron acoustic microscopy observations of twins and grain boundaries in III-V materials

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Polycrystalline GaP and InP have been observed by scanning electron acoustic microscopy. While grain boundaries show a weak contrast, twinned regions are clearly revealed. Results suggest that the contrast observed is related to signal generation by a piezoelectric-coupling mechanism.

Grain and twin boundaries in semiconducting materials influence some structure-sensitive parameters like carrier mobility or lifetime. Therefore, a detailed study of such boundaries is required for a complete understanding of transport phenomena in polycrystalline semiconductors. Several techniques based on the scanning electron microscope (SEM), such as cathodoluminescence (CL), or electron beam-induced current (EBIC), can provide information about grain and twin boundary structures in a nondestructive way. In particular, boundaries are readily observed with these techniques in several III-V compounds. In recent years, the scanning electron acoustic microscope (SEAM) has shown its capability to image grain boundaries in metals[1] as well as in silicon.[2] On the other hand, SEAM images of the grain structure of elastically anisotropic polycrystals are possible because of the orientation contrast between different oriented grains.[3] In the present work, SEAM and CL measurements have been carried out for the observation of twins and grain boundaries in GaP and InP.

The samples used in this investigation are polycrystalline GaP and InP from Metals Research. SEAM and CL observations of the samples were performed on a Cambridge S4-10 scanning electron microscope. The experimental SEAM arrangement used in this work has been previously described.[4] The amplitude A and the phase delay ψ of the detected signal with respect to the reference square wave signal can be separately imaged. The acoustic signal can be detected at the reference frequency f (linear mode) or at $2f$ (nonlinear mode[2]). To prevent spurious EBIC signals, which could be introduced into the SEAM contrast, both sample surfaces were earthed.

Figure 1 shows the SEAM and visible (300–800 nm) CL images of the same area of a GaP sample. The corresponding image of the emissive mode was featureless. Figure 2 shows the emissive mode and SEAM images of an InP sample. In order to get a reference for the SEAM image, the sample was etched as described in Ref. 5 revealing grain and twin boundaries, as seen in the emissive mode image. For InP samples, CL images were obtained using a Ge detector (North-Coast EO-817) with optimum spectral response in the range of 0.8–1.7 μm. In these images, dark grain boundaries are observed but appear crossed by a dense array of dark lines, probably related to polishing damage.

In both materials investigated in this work, no variation of the spatial resolution has been observed when the chopping frequency was varied from 10 to 240 kHz. On the other hand, the nonlinear signal (at the frequency $2f$) was very weak and only noisy images are obtained in this mode. Comparison of CL and SEAM images shows that both techniques provide different information. The CL contrast appears mainly localized at grain boundaries. The SEAM contrast of grain boundaries is, on the contrary, rather low while twins which are not observed in CL images are clearly revealed. The SEAM contrast at twins is higher in the amplitude than in the phase images but the latter sometimes show additional features (marked A in Fig. 2).

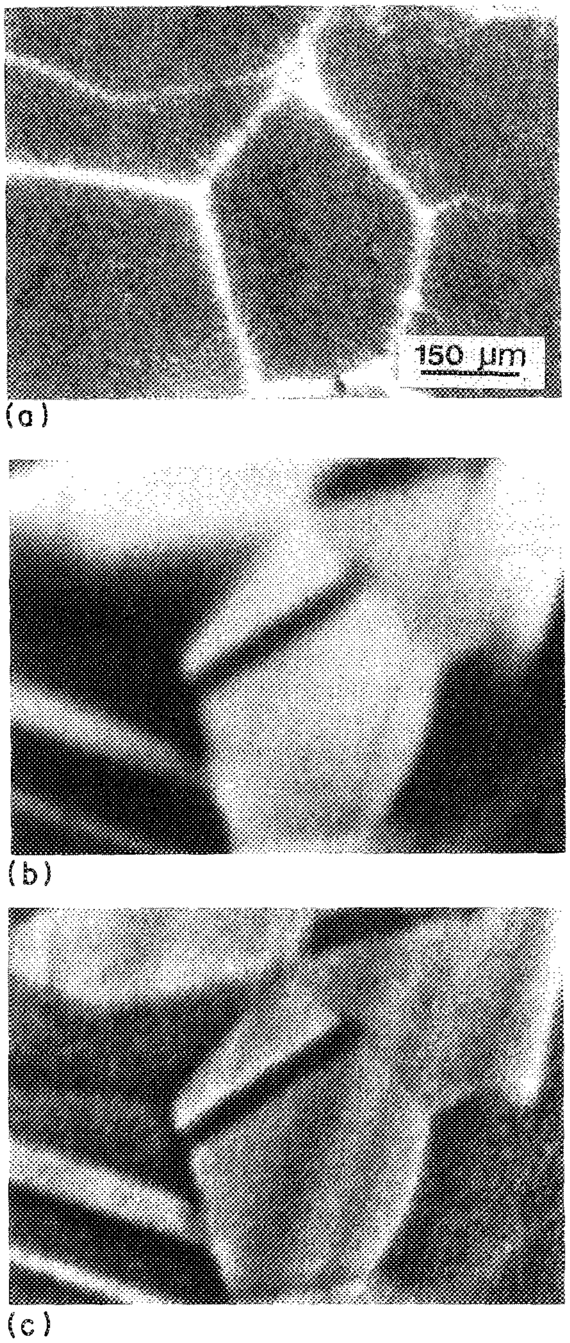

FIG. 1. Polycrystalline GaP sample. (a) CL image, (b) linear SEAM amplitude image, and (c) phase image of the same region at 203.7 kHz.

The SEAM signal generation mechanism is often explained by the conversion of an electron beam-induced heat distribution into sound (thermal wave coupling[6]), but evidence has been found of the existence of piezoelectric and electrostrictive couplings in III-V semiconductors.[7] Theory for the thermal wave coupling predicts that the spatial resolution of the SEAM image is proportional to $f^{-1/2}$. Such a dependence has been found in metals[8] but the present results indicate that in GaP and InP the thermal coupling does not determine the contrast. The electrostrictive or excess carrier coupling in semiconductors is basically nonlinear so that a high $2f$ signal is to be expected if this kind of coupling has a high contribution to the sound generation. As mentioned above only a weak $2f$ signal has been observed which seems to rule out the possibility of excess carrier coupling.

GaP and InP are piezoelectric materials, so that piezoelectric coupling contributes to the sound generation. In fact, the piezoelectric-constant matrix of III-V compounds is anisotropic,[9] which could explain the observed high contrast at twins. In regions with different orientations the acoustic signal generation by piezoelectric coupling would be different. Furthermore, the transducer we used detects

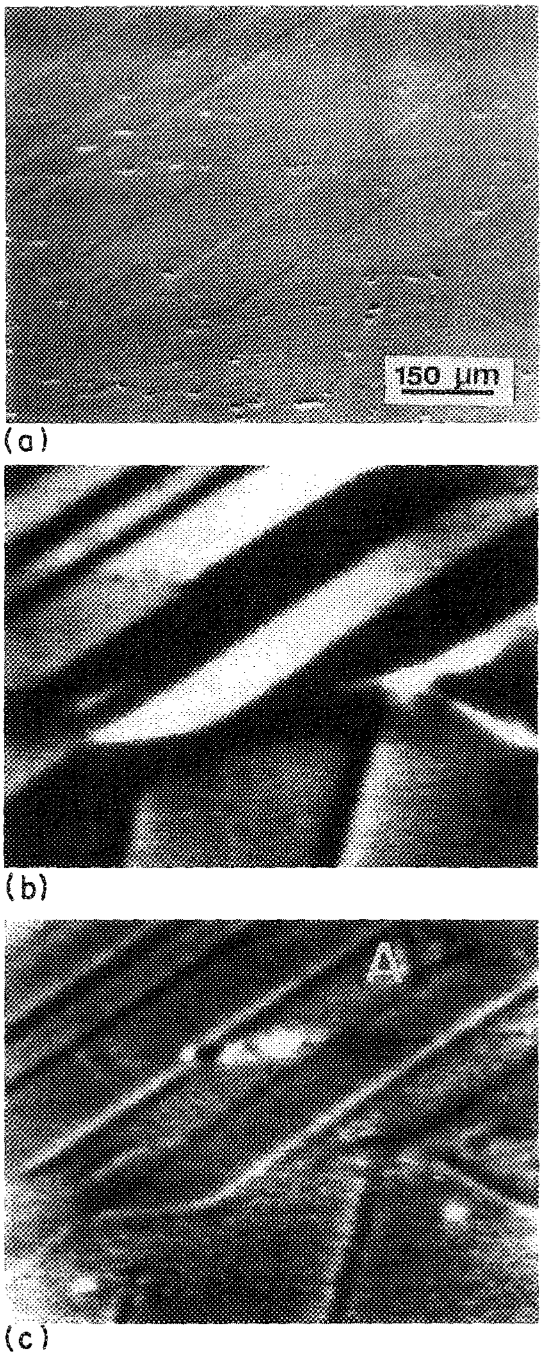

FIG. 2. Polycrystalline InP sample. (a) Emissive mode image of an etched region, (b) linear SEAM amplitude, and (c) phase image of the same zone at 223.9 kHz.

the longitudinal component of the acoustic wave, which depends on the crystallographic orientation of the observed region. Another possibility which explains the image contrast of twinned regions is the elastic anisotropy, as observed in several polycrystalline metals.[3] However, this possibility would involve the thermal wave coupling that, as stated above, does not determine the contrast in the present case. On the other hand, highly elastical anisotropic materials exhibit particularly good grain contrast in the SEAM images.[3] The observed contrast of twins in GaP and InP crystals is fairly good, although such materials do not present highly anisotropic elastic properties. In particular, the anisotropy ratio, defined as $2C_{44}/(C_{11}-C_{12})$, is smaller for GaP and InP than for Cu. In order to check the possible effect of the elastic anisotropy on twin contrast, twinned Cu samples were also studied. The twinned regions were clearly observed in the emissive mode of the SEM when etched samples were used, but the SEAM contrast of twins and grains was very weak as compared with the contrast observed in GaP and InP.

It is concluded from the present observations that SEAM can be used in the characterization of polycrystalline III-V materials and that provides complementary information to that obtained by other SEM-based characterization techniques. The results suggest that the observed contrast is associated with the piezoelectric character of these materials.

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