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## NONLINEAR SCANNING ELECTRON ACOUSTIC MICROSCOPY

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**Résumé** - La microscopie électronique acoustique par balayage non-linéaire est une technique spéciale de la microscopie acoustique, qui utilise les amplitudes et les phases des harmoniques, particulièrement le second harmonique de l'onde acoustique provenant d'un faisceau électronique modulé sur une certaine fréquence de base. Comme ces harmoniques sont déterminés par le couplage non-linéaire entre le son et le solide, ils détectent d'une manière très sensible les inhomogénéités du matériau avec une grande résolution spatiale.

**Abstract** - Nonlinear scanning electron acoustic microscopy is a special technique of acoustic microscopy which uses amplitudes and phases of higher harmonics, especially the second harmonic, of the sound wave originated by an electron beam modulated at a certain ground frequency. As these harmonics are determined by the nonlinear coupling between sound and the solid, they reveal very sensitively material inhomogeneities with high spatial resolution.

## INTRODUCTION

Scanning electron acoustic microscopy (SEAM) is a recently developed technique /1-4/, which allows determination of microscopic variations of material parameters associated with sound generation and propagation within the examined material. Until now SEAM has been used only in the linear mode. This means: the electron beam is modulated by a sine or square wave generator via a chopping device at a certain frequency, the sound wave is detected by a transducer mounted to the bottom of the specimen, the signal is amplified by means of phase-sensitive lock-in amplification at the same frequency. Thus only linear coupling mechanisms between sound and specimen are used for the electron acoustic (EA) image formation.

## NONLINEAR ELECTRON ACOUSTICS

As can be shown by consideration of primary electron beam parameters and material properties, nonlinear interaction may occur due to several reasons: non-validity of Hooke's law because of the large amplitudes within the generation volume and thus an anelastic behaviour, nonlinear coupling of sound because of piezoelectricity or space charges, finally nonlinear interaction between free carriers and sound in a semiconductor material /5/. These nonlinearities lead to deformation of the original wave and to generation of harmonic waves, especially the second harmonic. Amplitudes and phase shifts of these harmonics are strongly related to material parameters. In this paper especially nonlinear interactions are used for the production of micrographs. By simultaneous measurement of amplitudes, phases and phase difference of ground wave and the chosen harmonic, a large amount of data on the solid can be gained. The realization of nonlinear SEAM has been carried out for a frequency range from several kHz up to 50MHz and to the use of second and fourth harmonic /6/, though applications of this paper are restricted to a chopping frequency of 100kHz and to the second harmonic. Fig.1 shows the principal experimental arrangement.

## APPLICATIONS

Fig.2 and fig.3 are examples of EA investigations of an InP single crystal substrate with Zinc doped regions. Zinc has been diffused in the bright regions of the secondary

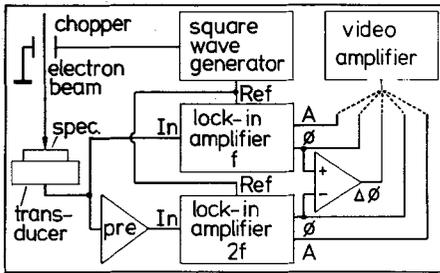


Fig. 1- Experimental set-up

Fig.2d is a phase difference image between ground wave and second harmonic showing up many inhomogeneities even in the undoped areas. As the second harmonic image depends on a high signal situation, it is very suitable for high spatial resolution, further it reveals more details on the material. This can be seen in the EA images of fig.3 taken within sections of the doped regions. Fig.3a is a 30keV micrograph showing white lines which can be associated with crystal dislocations. When lowering the primary energy to 5keV, the signal is mainly generated in the surface near region. Then the EA image (fig.3b) shows pitch like structures, from which a spatial resolution of less than  $0.2\mu\text{m}$  can be deduced. In fig.4 a second harmonic EA micrograph is shown for a uniformly diffused InP surface. Besides of many other effects, it shows variations of the EA amplitude due to flow channels caused by the liquid phase process. The black circular area is etched down to the undoped material as a reference.

A striking application of nonlinear SEAM has been the examination of solar graded polycrystalline silicon, which has not obtained any kind of specimen preparation. In spite of a rough surface, imaging of grain boundaries has been possible with high sensitivity and spatial resolution. Fig.5 shows some remarkable results (for more details see /4/). Fig.5a-c give a low magnification overlook. Whereas in the SE+RE image only surface topography can be seen, both amplitude and phase EA images of the second harmonic yield the polycrystalline structure. Fig.5d-e demonstrate the high spatial resolution possible with the  $A(2f)$ -mode. The imaged area is a grain boundary, showing a black contrast, surrounded by an about  $20\mu\text{m}$  wide bright region on both sides. These regions correlate to so-called denuded/zones of decreased oxygen and carbon concentration. The corresponding phase image of fig.5f shows a rapid signal variation at the boundary itself allowing a precise determination of the boundary location. When comparing linear and nonlinear modes for this application, especially for twin boundaries, a significant contrast difference occurs. Whereas in the second harmonic image boundary and denuded zones show up clearly (fig.5h), the linear EA image of the same section gives only a change in the amplitude from one grain to the other (fig.5i). In choosing the primary electron beam energy one has to be careful in those cases, in which the boundary is inclined with a small angle to the specimen surface. Then the electron beam can reach the surrounding bright areas at various depths corresponding to the energy dissipation of the electrons. As a result an integration of the bright signal occurs, which seems to broaden the denuded zone, as shown in fig.5k-m for an example, which yielded an overall width of the wide region of  $20\mu\text{m}$  at a primary energy of 5keV.

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and backscattered (SE+RE) electron image of fig.2a. Whereas this contrast has been only in the 1<sup>st</sup>-order, the EA micrographs show very good contrast. The linear EA amplitude image ( $A(f)$ ) of fig.2b only gives a uniform distribution determining the doped regions. The second harmonic EA amplitude image ( $A(2f)$ ) of fig.2c is more detailed and shows inhomogeneities, preferably at the edges of the structures and at the narrow gates, which might be due to variation of the diffusion depth achieved locally, as the primary electron penetration depth correlates quite accurately with the average diffusion depth of this sample.

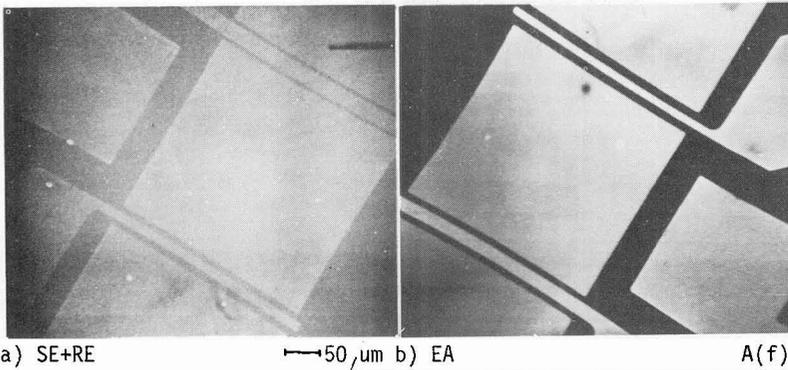
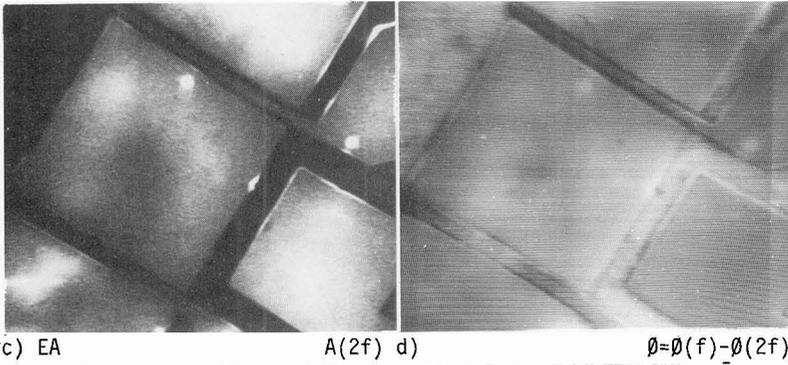


Fig. 2 - EA images of Zn-diffused regions in InP for a primary electron energy of 30keV



a) SE+RE  $\longleftarrow$ 50,um b) EA A(f)  
c) EA A(2f) d)  $\emptyset=\emptyset(f)-\emptyset(2f)$

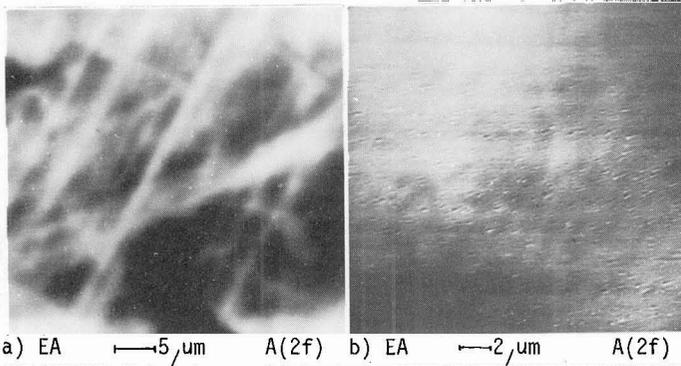


Fig. 3 - Comparison of non-linear EA images of crystal imperfections for various primary electron energies: a) 30keV; b) 5keV

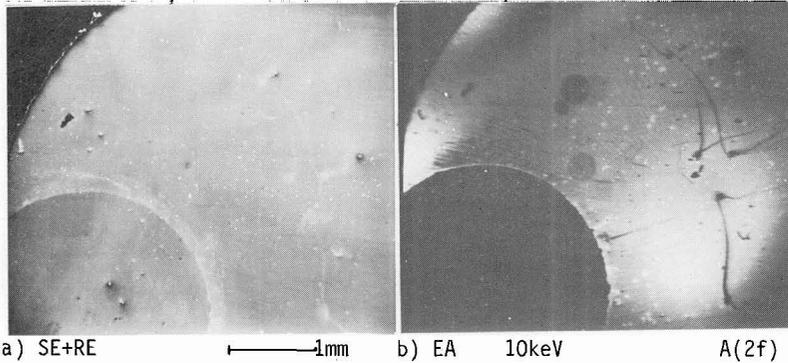


Fig. 4 - EA micrograph of an InP substrate diffused with Zn, the circular area is etched as an undoped reference

a) SE+RE  $\longleftarrow$ 1mm b) EA 10keV A(2f)

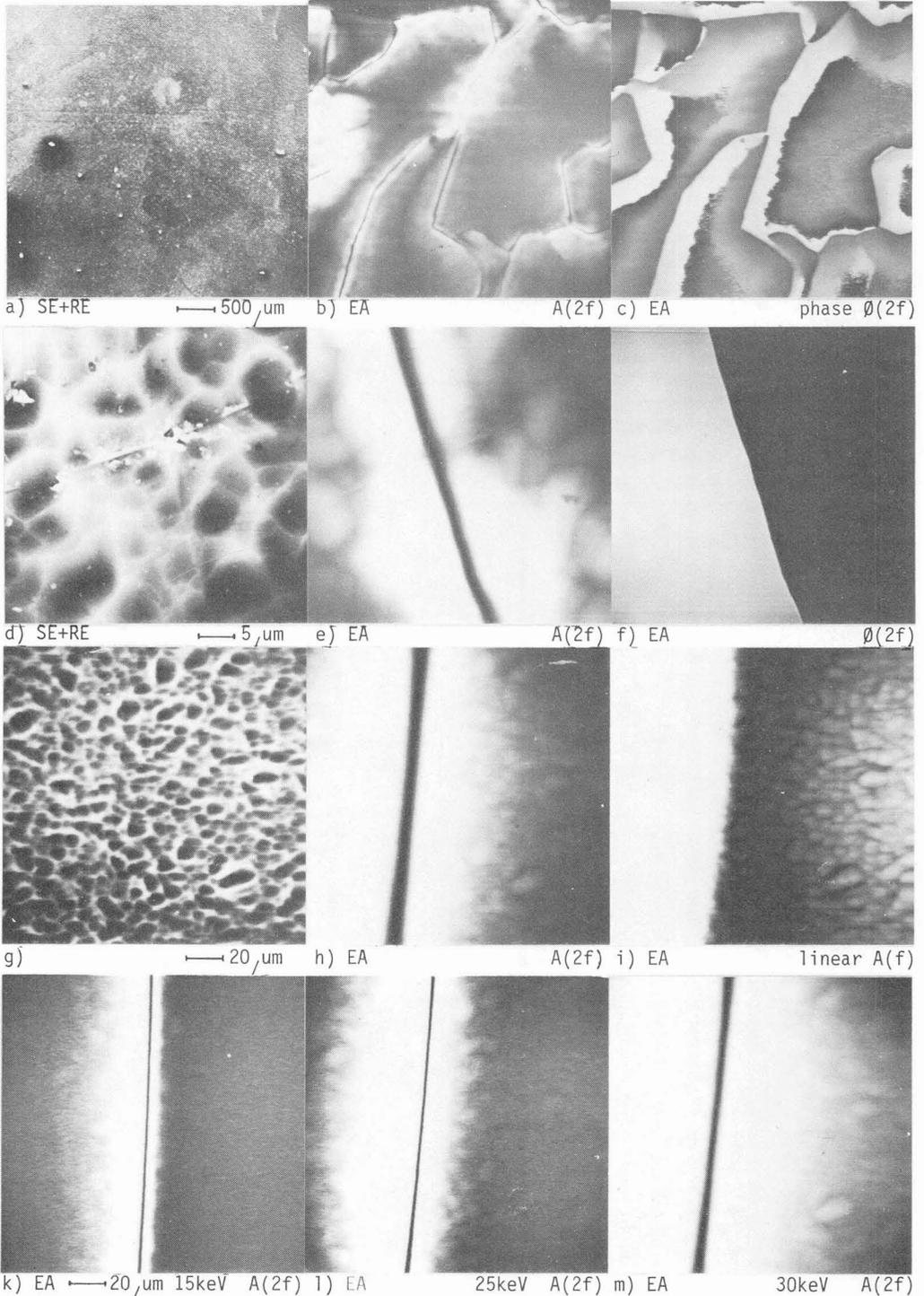


Fig. 5-Electron acoustic images of solar grade polycrystalline silicon