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A CONTRIBUTION TO THE SCANNING ELECTRON MICROSCOPE BASED MICROCHARACTERIZATION OF SEMI-INSULATING GALLIUM ARSENIDE SUBSTRATES

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Abstract

The macroscopic behaviour of semiconducting materials is determined by the distribution of microscopic defects like dislocations, impurities and intrinsic defects. Therefore, microanalytical methods are necessary to control the influence of technological process parameters on the materials properties. In the case of GaAs substrates, measurements of the cathodoluminescence (CL) and the electron beam induced voltage (EBIV) as well as the new charging technique seem to be promising methods to perform this task. CL-micrographs of as-grown GaAs substrates show bright cellular structures, which correspond to dislocation networks. Comparative investigations by use of the new charging contrast technique indicate an increased conductivity in the bright areas. CL-measurements of annealed substrates reveal additional characteristic island-like structures in the cell interior. Both, cellular and island-like structures can also be visualized by the EBIV technique. These results can be explained by a homogeneous conductivity and an inhomogeneous distribution of the excess carrier lifetime.

Key Words: Electrical microcharacterization, scanning electron microscopy, cathodoluminescence, electron beam induced voltage, charging contrast, GaAs, dislocation networks, annealing

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Introduction

GaAs is a promising material for high speed and high frequency electronic devices. Much attention has been paid to the influence of the quality of the semi-insulating (s.i.) substrate material on the device parameters [5,13,15,16,17]. One method to investigate the quality on a micrometer scale is cathodoluminescence investigation in a scanning electron microscope. Such CL-investigations revealed cellular structures with a high luminescence yield, which correspond to dislocation networks [1,6,9]. This result is explained by a different defect concentration in the vicinity of the dislocations compared to other locations on the specimen[9]. This defect distribution is held responsible for the inhomogeneous substrate quality.

A post growth annealing process of the GaAs substrates results in an improved homogeneity of the device parameters[14] . CL-micrographs of annealed material show that the cellular structures remain unchanged and that in the cell interior additional bright island-like structures occur [9,8,11] Corresponding measurements of the CL-spectra show in both cases that merely the intensity but not the spectral composition changes locally thus creating the observed CL contrasts. However, CL investigations alone are not sufficient to clarify the influence of the annealing process on the local electronic properties which could be regarded to be the microscopic origin of the observed improvements and have therefore to be compared with other spatially resolved measurement techniques.

In the present work for the first time spatial resolved CL-measurements, measurements of the electron beam induced voltage (EBIV) and measurements of the charging contrast on s.i.-GaAs substrate material are compared.

Theory

The main problem in performing EBIV experiments on s.i. material stems from their high resistance. Fig. 1 shows the experimental set-up and the equivalent circuit. The Schottky- contact is represented by a voltage source with the induced voltage U_{ind} and the substrate is represented by the resistance R_B between the surface Schottky-contact and the backside contact. The input resistance R_M of the amplifier and R_B can be considered as a voltage divider network and the measured voltage U_M is then given by:

$$U_M = U_{ind} * \frac{R_M}{R_M + R_B} \qquad (1)$$

Since the bulk resistance R_B in s.i. material is very high a sufficient signal U_{ind} can only be gained by using a high impedance amplifier. A chopped electron beam generates acoustic waves in the specimen. In a piezoelectric material like GaAs these acoustic waves induce a voltage at the specimen surfaces. In order to avoid a superposition of the EBIV with the electron acoustic signal, we did not use a chopped electron beam. Therefore, an improvement of the signal to noise ratio by use of a lock-in amplifier technique was not possible. Equation (1) implies that the EBIV-signal depends on the carrier lifetime, which influences the induced voltage U_{ind}, as well as on the bulk resistance R_B.

The charging contrast of s.i.-GaAs has for the first time be reported in ref [18]. It can be observed at low temperatures using high

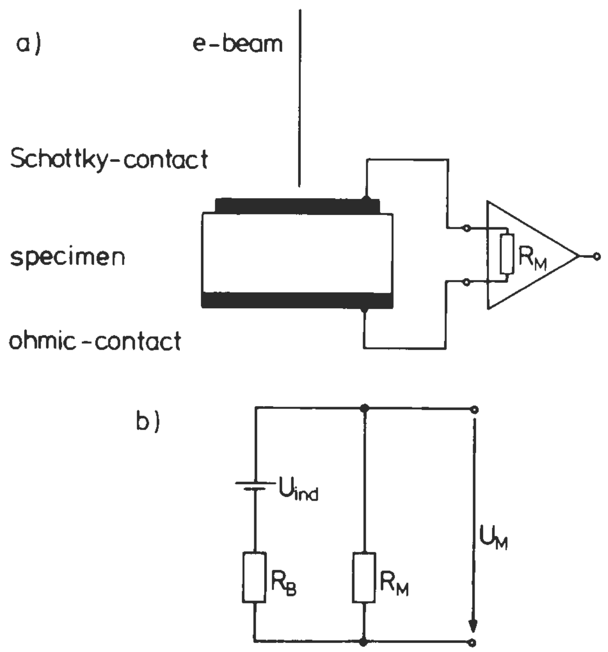

Figure 1: (a) experimental set-up for EBIV measurements (b) equivalent circuit

beam currents in the secondary electron image. The contrast formation can be explained by the following equilibrium equation of the primary beam current I_O, the absorbed current I_{abs} and the secondary and backscattered electron current I_{SE}, I_{BE} :

$$I_O = I_{abs} + I_{SE} + I_{BE} \qquad (2)$$

We can transform the right side of this equation, if we use the following relations:

$$I_{abs} = U_S / R_S \qquad (3)$$

$$I_{SE} + I_{BE} = I_O * \sigma(eU_K - eU_S) \qquad (4)$$

U_S is the specimen surface potential, R_S is the resistance between the irradiated area and ground, and σ is the secondary and backscattered electron yield as a function of the cathode potential U_K and the specimen surface potential U_S. We get:

$$1 - \frac{U_S}{I_O * R_S} = \sigma(eU_K - eU_S) \qquad (5)$$

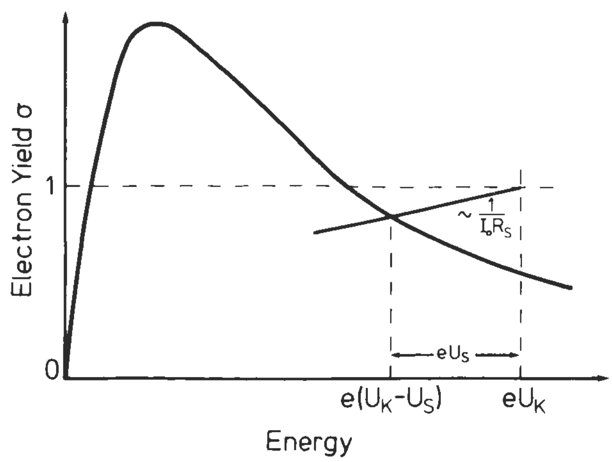

Figure 2: Influence of the resistance of s.i. material on the electron yield (see eq. 5)

In fig. 2 both sides of eq. 5 are shown in the same diagram as functions of (eU_K - eU_S). The intersection point determines the surface potential U_S and the corresponding secondary and backscattered electron yield σ. Since the slope of the linear function is inversely proportional to I_O*R_S the secondary and backscattered electron yield σ increases with a decreasing resistance R_S. Charging contrast micrographs thus give information about the local resistance between the specimen surface and ground. This resistance depends not only on the local resistivity of the irradiated specimen area but is also influenced by the resistivity of distant specimen regions. Therefore, the spatial resolution of the charging contrast technique depends on the specimen properties and cannot be predicted for a particular case.

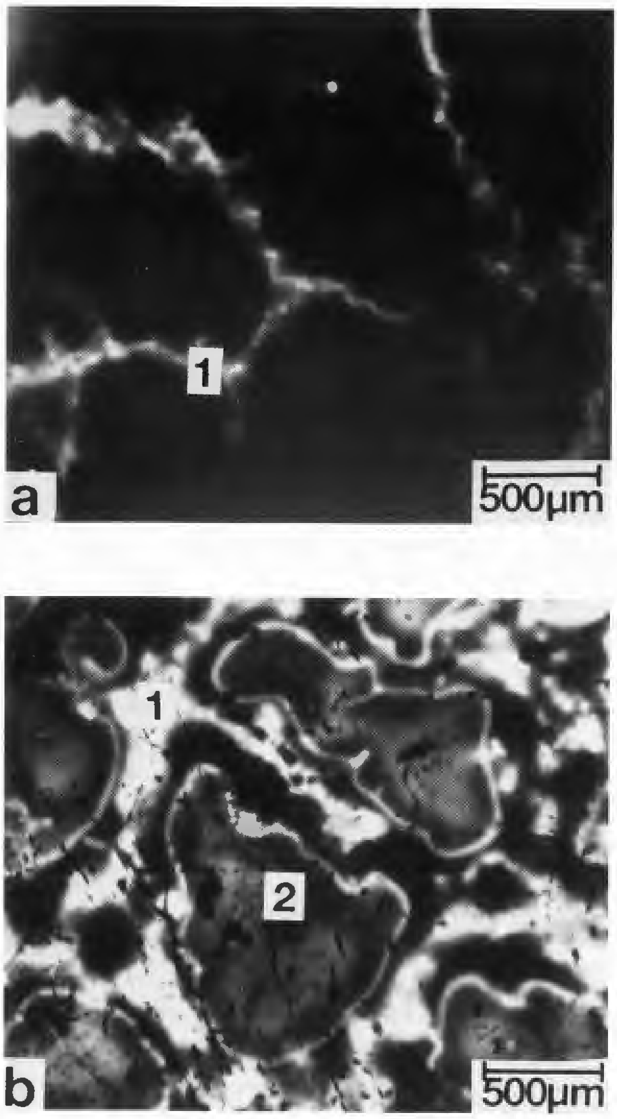

Figure 3: CL-micrographs of an as-grown (a) and an annealed substrate (b): cell walls (1), island-like structures (2)

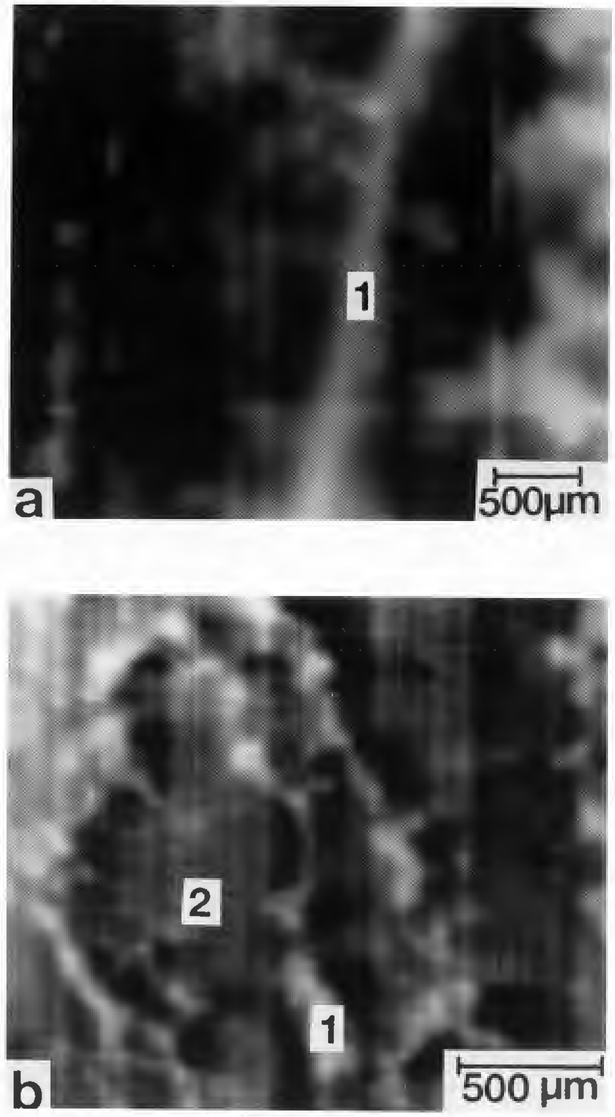

Figure 4: EBIV-micrographs of an as-grown (a) and an annealed substrate (b): cell walls (1), island-like structures (2)

Experimental Procedure and Specimen Material

The CL experiments were performed in a scanning electron microscope based measurement system, which has been described in detail in ref[10]. The CL micrographs were taken with a photomultiplier with a sensitive wavelength range between 300 nm and 850 nm. For all CL measurements a primary electron beam energy of 30 keV and a beam current of about 10 nA were used. The specimen temperature was 77K.

The EBIV-measurement circuit was simply realized by connecting the Schottky-contact and the ohmic backside contact with a preamplifier, which had an input resistance of 100 MΩ. The EBIV-experiments were done at room temperature, because the bulk resistance R_B increases with decreasing temperature and the signal U_M becomes too small. The beam energy was 30 keV and beam current was about 100 nA.

The charging contrast occured at temperatures below 30 K. Our measurements were performed at 10 K with a beam energy of 30 keV and a beam current of about 100 nA.

The specimens were cut from LEC-grown undoped s.i.-GaAs wafers. We investigated as-grown material (specimen 1) and material, which has been ingot-annealed for 30 h at 1000 °C (specimen 2). Schottky-contacts for the were fabricated by evaporating a 10 nm thick chromium layer and subsequently a 50 nm thick gold layer. Ohmic contacts were made by evaporating a Au/Ge layer.

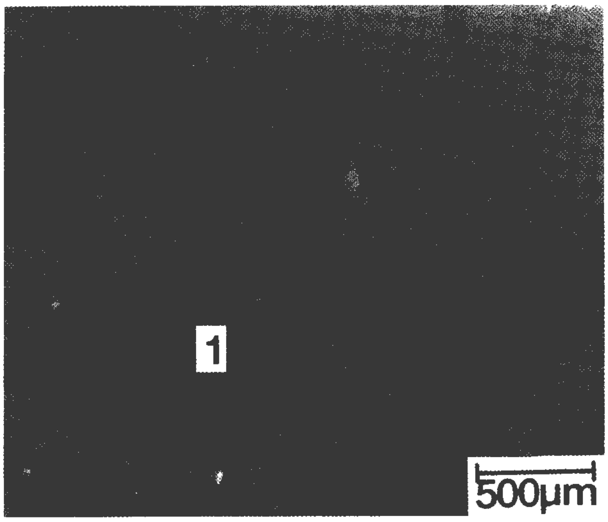

Figure 5: Charging contrast micrograph of the same as-grown substrate as in figure 3a:cell walls (1)

Results

The CL-micrograph (fig. 3a) of an area of specimen 1 shows the cellular structure with bright cell walls (1) and dark cell interior. In fig. 3b a CL-micrograph of an annealed substrate (specimen 2)is shown. The cell walls (1) and the island-like structures (2) can clearly be seen.

The micrographs in fig. 4 show, that the electron beam induced voltage increases at the cell walls in as-grown (fig. 4a) as well as in annealed material (fig. 4b). In the annealed substrate the additional island-like structures occur. The weak parallel lines are due to 50 Hz noise.

The charging contrast micrograph in fig. 5, which shows the same specimen location as fig. 3a, reveals a dark contrast at the cell walls. On annealed material, however, no contrast under similar beam conditions could be observed.

Discussion

CL is the radiation of a specific recombination process of excess carriers. CL micrographs thus reflect the local recombination behaviour of a specimen. The origin of EBIV is the separation of free excess carriers in an electric field of a pn-junction or a Schottky-contact. Variations of the EBIV signal indicate variations of the free carrier lifetime and of the local electrical conductivity (see eq. 1). If we suppose an increase of the carrier lifetime at the bright locations of the specimen the equilibrium condition of generation and recombination:

$$g = 1/\tau * \delta n \qquad (6a)$$
$$g = 1/\tau * \delta p \qquad (6b)$$

where g is the generation density, τ the carrier lifetime, and δn, δp the excess carrier concentrations, predicts for such locations an increased concentration of excess carriers. Under this condition, more carriers can be separated by an internal electric field and more carriers can recombine radiatively. Therefore, both CL-yield and EBIV increase with increasing carrier lifetime and this can be taken for an explanation of the bright structures in the CL- and EBIV-micrographs (fig. 3a,b fig. 4a,b).

The observed CL contrast of both as grown and annealed GaAs substrates can be traced back to the inhomogeneous distribution of a competitive recombination process which is either non-radiative or radiative, but with a wavelength above 850nm, which cannot be detected by the photomultiplier used. Bright contrasts on the specimen (cell walls, islands) are explained by a concentration of the responsible competitive recombination centers which is less than at the dark specimen locations [8,11]. It was shown by infrared absorption measurements that the concentration of the main native donor, the EL2, which is ascribed to an As-atom on a Ga site, also increases at the cell walls [12]. As a consequence, it can be concluded that locations with an increased EL2 concentration show a decreased Ga-vacancy concentration. The Ga-vacancy or a defect associated with a Ga-vacancy is discussed in the literature to be a non-radiative recombination center [4,3]. It was therefore assumed that the Ga-vacancy is involved in the competitive process. At locations, where the concentration of the Ga-vacancies is low, the carrier lifetime is expected to be higher than at locations where the concentration of the Ga-vacancies is high. Although, the experimental determination of the carrier lifetime via measurements of the photocurrent profiles across s.i.-GaAs wafers indicated an increased carrier lifetime at the cell walls as expected [7], these investigations suffered essentially from a low spatial resolution.

The results of the charging contrast measurements unambiguously suggest an increased conductivity at the cell walls of as-grown substrates. If we suppose an increased carrier concentration at these locations, this leads to a locally decreased resistance R_B (eq. 1) and to an increased measured voltage U_M, thus creating a bright EBIV contrast as actually observed. There is evidence that the CL yield in GaAs increases with increasing carrier concentration [2]. Therefore the observed bright contrasts at the cell walls can be explained by a locally increased conductivity, which is probably due to an increased concentration of the main donor EL2. This is not in contradiction to the assumed existence of a competitive recombination process, but rather is an alternative interpretation of the results. Probably, both alternatives, a local change of the carrier lifetime or of the electrical conductivity - both related to the local distribution of the EL2 -

actually occur.

In contrast to as-grown substrates no charging contrast was observed at the annealed material. This behaviour suggests a homogeneous conductivity even on a micrometer scale, but from the CL- and EBIV-measurements which show additional island-like structures one should also expect a corresponding charging contrast behaviour. Whether or not the observations on annealed material are due to the electron beam parameters used or represent a real specimen effect, remains as a matter of question and has to be the subject of further investigations.

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Discussion with Reviewers

J.F.Bresse: There is a contradiction between your CL and EBIV results and the charging method for the annealed substrate. How can you explain that? Is there any effect of the injection level?

Authors: Our experimental results can be explained by an homogeneous conductivity of the annealed substrates, so that no charging contrast can occur. In this case the observed CL and EBIV contrasts would indicate an inhomogeneous carrier lifetime. On the other hand, we cannot exclude that a charging contrast occurs at different beam conditons, which would indicate an inhomogeneous conductivity. Further investigations on the charging contrast mechanism are necessary to clarify this question.

J.F.Bresse: In the charging method a black contrast is related to an increase of the ratio U_S/R_S . Is there any effect of an increase of the surface potential due to a barrier phenomenon at the cell boundary which can also explain the black contrast at the cell boundary?

Authors: The charging contrast gives information about the local resistance between the irradiated specimen area and ground. This resistance can be influenced by the local resistivity as well as by the presence of potential barriers.