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Scaling of dislocation cells in GaAs crystals by global numeric simulation and their restraint by in situ control of stoichiometry

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Abstract

Undoped GaAs crystals show dislocation cell patterns with cell dimensions of some hundred micrometer orders of magnitude larger than in metals. Nevertheless, the correlation between cell diameter and dislocation density obeys the same Holt relation. Experimental results of dislocation analysis in GaAs were correlated with global computation of the resolved shear stress distribution at growth relevant temperatures and scaled with the universal function $d = K\rho^{-1/2} = \alpha KGb\tau^{-1}$, where d is the cell size, ρ the dislocation density, K, α the constants, G the shear modulus, b the magnitude of Burgers vector and τ is the resolved shear stress. Samples grown with in situ control of stoichiometry show nearly no dislocation patterning due to the minimised native point defect content needed for dislocation climb.

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1. Introduction

After silicon the III–V compound GaAs is the most important semiconductor material. Low carbon-doped semi-insulating (SI), i.e., quasi-undoped crystals are used for high-frequency microelectronics. Today, a high level of crystal quality and reproducibility of the electrical parameters exists although dislocation-free material is not yet available. Thus, the problem of dislocation cell patterning still arises in all SI GaAs crystals, as shown in Fig. 1, independently of the growth method used [1–3]. Typical cell dimensions between 100 and 1000 μm are observed at dislocation densities in the range of 10^5–10^4 cm^{-2}, respectively [4]. Such cellular structure affects the electrical and optical parameter homogeneity in as-grown crystals. For instance, the electrical resistivity is inhomogeneously distributed across the wafers, varying by more than one order of magnitude between the cell walls and interiors [2,3]. Therefore, a costly specific post-growth annealing procedure is required in order to improve the mesoscopic homogeneity before the wafers are released for device production.

Cellular arrangements are well known from crystals with stored dislocations, which undergo external or internal stress. They have been studied the best in mechanically deformed metals and semiconductors for a long time [5,6]. In growing crystals they are the result of dynamical polygonisation due to plastic relaxation of the growth-inherent thermo-mechanical stress field. Cell formation takes place at high temperatures behind the growing melt–solid interface as has been convincingly demonstrated by real-time X-ray synchrotron topography [7].

There is a large amount of data in the literature showing the universality of the correlations between cell size d, dislocation density ρ (i.e., mean dislocation spacing $\rho^{-1/2}$) and acting shear stress τ. For metals d obeys the equation $d = K\rho^{-1/2} = \alpha KGb\tau^{-1}$, where G is the Young's modulus and b is the magnitude of Burgers vector with factors of proportionality in the range of $10 < K < 30$ and $0.2 < \alpha < 1$ [5,8,9]. However, there have been few attempts to study scaling for semiconductor compounds. Among these, mean cell diameters of $d \approx 3$ and 5 μm from transmission electron

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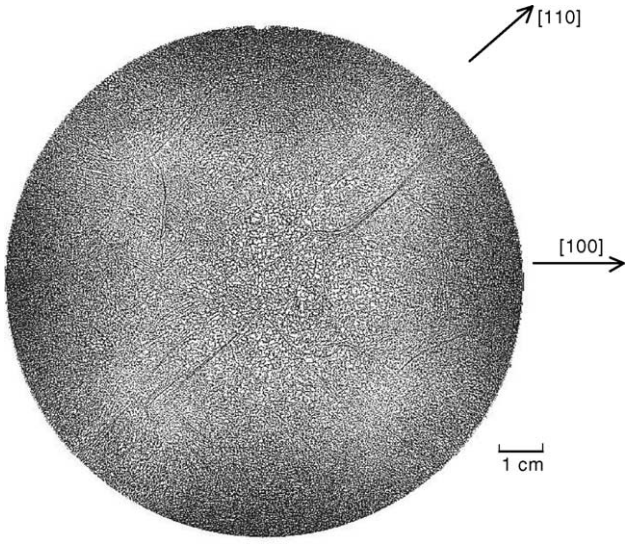

Fig. 1. Dislocation structure with typical cellular pattern revealed by KOH etching on a semi-insulating 6 in. GaAs wafer taken from the upper cylindrical part of an as-grown VCz crystal.

microscopy (TEM) examinations on post-deformed GaAs specimens [10,11] were found at $\tau = 30$ and 14.3 MPa, respectively. Geibel [12] ascertained a correlation of $d = 22Gb\tau^{-1}$ for InP single crystals bended at $0.8T_m$ (where T_m is the melting point). Recently, we correlated for the first time the cell dimensions in undoped GaAs crystals with the growth-inherent thermo-mechanical strain estimated from the calculated von Mises stresses [3]. We found comparable dependencies with post-deformed metals.

In this paper we present a more precise scaling analysis. The cell sizes experimentally determined along specific crystallographic directions of as-grown wafers will be compared with the global numeric calculation of the maximal resolved shear stress (MRSS) τ_m distribution along the crystal radius r and under consideration of given growth conditions. Finally, we will demonstrate the importance of in situ stoichiometry control during the crystal growth process for the restraint of the cell formation process.

2. Experimental

Undoped GaAs single crystals with diameters between 60 and 150 mm were grown along the [0 0 1] direction by the vapour pressure controlled Czochralski (VCz) technique [13]. This technique, a modification of the conventional liquid encapsulation Czochralski (LEC) method, is characterized by low thermal gradients and less thermo-mechanical stress down to values of 20–30 K cm^{-1} and 0.2–2 MPa, respectively. In the standard VCz version a liquid boric oxide (B_2O_3) layer is applied as melt encapsulant. Crystals obtained in this manner were grown slightly off-stoichiometric from As-rich melts. They exhibit relatively high concentrations of native point defects like As interstitials, and vacancies of both constituents. Pulling rates between 3 and 6 mm h^{-1} were used. The as-grown boules were cooled down within the growth chamber with a rate of 50 K h^{-1}. To study the correlation between crystal composition, i.e., intrinsic point defect content, and cell formation probability some VCz crystals were grown without B_2O_3 encapsulant. Growing in this mode, the melt is in direct contact with the vapour and the melt composition was adjusted in situ by controlling the arsenic atmosphere in the growth chamber. We found that a Ga-rich melt with mole fractions of $x \leq 0.45$ results in a near-stoichiometric crystal composition and, hence, in the condition for minimum intrinsic point defect concentration. More growth details were described elsewhere [14].

For examination of the dislocation patterns the as-grown crystals were cut into slices perpendicular to the growth axis and polished. The standard procedure of KOH etching at 390 °C was used to ascertain the dislocation arrangement via etch pit density (EPD) measurements. For the present analysis only wafers from the top of the cylindrical crystal section were considered. The EPD was determined by an automatic optical mapping technique [15] whereas the local mean dislocation densities (data points in Fig. 3) were each ascertained from areas of 3.92 mm × 2.94 mm. The cell dimensions were measured by the line chord method, which is a standard technique of texture analysis [16]. Finally, the mean values of the cell diameters along the ⟨1 0 0⟩, ⟨1 1 0⟩ and ⟨2 1 0⟩ directions were obtained from the Gaussian distribution.

The mean tilt angle between cells was determined by topographic measurement of the width at half maximum (FWHM) of the X-ray double crystal rocking curve (DCRC) across the polished wafers.

3. Numeric stress field calculation

Generally, the numerical modelling of the thermo-mechanical stress field in growing crystals is a well-known procedure (see e.g., [17–19]). First, the temperature distribution in the growing sample was calculated using the code CrysVUN^{++}, which is a modern powerful global finite volume program [20]. Very good agreement between computation and experiment [13,21] is obtained when comparing the modelled isotherm morphology with post-growth striation analysis. Then the temperature gradient field of the considered growth position was taken to calculate the elastic stress distribution via the tensors σ_{rr}, $\sigma_{\theta\theta}$, σ_{zz} and σ_{rz} in the axis-symmetric cylindrical coordinate system [18].

GaAs crystallises in the zinc-blende structure with the main dislocation glide system ⟨1 1 0⟩{1 1 1} consisting of 12 equivalent slip systems with Burgers vector $b = 1/2$ ⟨1 1 0⟩ [17]. Consequently, the axis-symmetric radial stress distribution was modelled by solving the stress–strain relation for an anisotropic body. As a result the crystallographically determined radial resolved shear stress distribution across the given transversal crystal cut was obtained. The calculations are based on the time-dependent crystal geometry in strong