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Microstructural comparison of material damage in GaAs caused by Berkovich and wedge nanoindentation and nanoscratching

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Nanoindentations and nanoscratches in GaAs under Berkovich and 60° wedge indenters were investigated via transmission electron microscopy. Dislocations, twins and slip bands are convergent under the Berkovich indenter, whereas they are only divergent under the 60° wedge. This difference is explained in terms of crystallography and geometry. Median cracks were created under the wedge indenter although only diverging bands are observed. Finally, twinning was found to be the main deformation process occurring during indentation, whereas only slip bands and perfect dislocations are observed during scratching.

Keywords: Nanoindentation; Scratch; GaAs; Deformation process

During recent years, many studies have been dedicated to semiconductor indentation and scratching, in particular of gallium arsenide (GaAs) due to its range of applications in the microelectronics industry [1–5]. Nanoindentation is frequently used to investigate mechanical properties such as hardness and Young's modulus at the nanoscale in semiconductors [4–6]. Initially, scratching was used by many scientists as the first step to cleave semiconductors [7–12], to investigate delamination of layered materials [13], thin films [14] or tribological contacts [15]. The objective of this paper is to analyze the deformation mechanisms during nanoindentations and nanoscratching between various tip geometries via high-resolution transmission electron microscopy (HRTEM).

GaAs is used in many applications, but its mechanical deformation mechanisms are only partially known. Numerous studies agree that the initial nucleation of dislocations, also called the elastic–plastic transition, is associated with a pop-in in the load–displacement curve at nanoindentation [3,16–22]. Glide planes are $\{1\,1\,1\}$ and the glide directions are $\langle 1\,1\,0\rangle$, leading to a rosette arm pattern. Depending on the doping and test conditions, the rosette arms exhibit various shapes and microstructures: Choi et al. [23] demonstrated that α- and β-dislocations are created along the [110] and $[\bar{1}\,1\,0]$ directions, respectively, and they have dissimilar velocities and mobilities [1,2,23], resulting in asymmetric rosette patterns [1,4,24]. This finding has been questioned by numerous authors [3,25,26] who did not observe such a pattern. Diverse results are also reported in the literature for the rosette microstructure under indentations on (0 0 1) GaAs wafers. Most authors classified their observations in respect to the applied load, making comparisons between the results difficult since indenter tip geometry and tip radius vary from experiment to experiment. For indentation loads of less than 0.55 mN, only perfect dislocations are visible [3]. Between 10 and 100 mN, perfect dislocations were seen only along $[\bar{1}\,1\,0]$, whereas partial dislocations associated with stacking faults and/or twins were only visible in the [1 1 0] direction [24–26]. However, for indentations at 50 mN, Lefebvre et al. [27] observed twins only in the $[\bar{1}\,1\,0]$ direction and only perfect dislocations in the [1 1 0] direction. At loads ranging from 2 to 100 mN, the main deformation mechanism during indentation was found to be microtwinning [28–31]. Furthermore, TEM investigations showed only converging glide systems under indentations with Vickers [1,2,26], spherical [4,10,20–22] and Berkovich [19,29–31] tips. Finally, dislocations, slip bands and twins were visible under indentation while only slip bands and dislocations took place under scratching [32]. From this review, it is clear that the deformation of GaAs is still not fully understood. More complete reviews are given in Refs. [4,28,33,34].

In this study, pure GaAs (1 0 0) samples $5\times5\,mm^2$ were cut from a 150 μm thick wafer doped with 1–

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2×10^{18} silicon atoms/cm^3 and with a miscut of 2° to the [1 1 0] direction. Indentations were made with a MTS Nanoindenter XP (MTS/Nanoinstruments, Oak Ridge, TN) using two types of indenter tip: a Berkovich and a wedge. The wedge indenter is a pyramid which has an elongated contact edge with a length $L = 55$ µm and an opening angle of 60°, and is hereafter called the 60° wedge. Indentions and scratches were carried out along either the [1 1 0] and/or $[\bar{1}\,1\,0]$ direction based on a planar representation given by Sumitomo [35] with an applied load of 80 mN. The velocity and length of the scratch was 200 µm s^{-1} and 500 µm, respectively. Two electron microscopes were used: a conventional Philips EM430 (LaB$_6$ cathode) and a high-resolution Philips CM300 (equipped with a field emission gun). Both TEMs operated at 300 keV and had spatial resolutions of 2.5 and 1.8 Å, respectively.

Figure 1 shows TEM cross-sections through an indent and a scratch performed with a Berkovich tip, showing very different deformation defects. The lamella in Figure 1a was cut through an indent according to Figure 3a. Two systems of converging bands crossing each other, as well as a dense network of dislocations, are visible, which is consistent with the literature [19,29–31]. These defects make an angle of 54.6° with the surface and thus lie in equivalent {1 1 1} planes. The selected-area electron diffraction (SAED) pattern inset taken in the [1 1 0] zone axis was obtained from the deformed area. It exhibits double spots resulting from twinning [36] which is confirmed by the dark-field images of the twinned area taken with the diffraction spot corresponding to the matrix (Fig. 1b), and the one generated by the twin (Fig. 1c), respectively. The streaks visible in the inset in Fig. 1a (indicated by white arrows) can arise from elastic strain effects and defects such as microtwins, stacking faults and slip bands [36]. Only one crack in the material was generated as shown by the white arrow in Figure 1a.

Figure 1d is a TEM bright-field image of the transverse cross-section through a scratch. The scratch was made in the [1 1 0] direction with the edge first. The SAED pattern inset in the [1 1 0] zone axis, taken from the area below the scratch, does not exhibit double spots or streaks, clearly demonstrating the absence of twins. The diffracted spots have an elongated shape, indicating severe material deformation caused by the scratching process. No dislocations are visible directly under the scratch imprint; however, a dense network of dislocation bands is observed deeper in the material above the lateral crack. In the pile-up regions, on the sides of the scratch, numerous dislocations and slip-bands were generated, indicated by white arrows in the magnified bright-field image inset in Figure 1d. Two types of cracks are visible: (a) the wide open one corresponds to a lateral crack and (b) the one perpendicular to the surface is defined as a median crack. Around the median crack, a large density of dislocations is generated due to high stress concentrations (white arrows in Fig. 1e), which remain even after the stress has been released via crack formation and opening.

To compare the defects induced by indentation and scratching with the Berkovich tip, a special lamella was prepared based on Figure 2a. The residual imprint depth after scratching is constant at about 500 nm. At the scratch surface, wavy lateral cracks were induced through stochastic movement of the tip. Beneath the indenter, starting at 2 µm, repeated tensile microcracks were formed. Under the scratched area, dislocations and slip bands are observed on one of the {1 1 1} planes forming an angle of 54.6° to the surface. No twinning is observed as confirmed by the SAED pattern taken in the [1 1 2] zone axis (Fig. 2c). Below the indent (Fig. 2b and e), deformation occurs by a mixture of converging twinning and dislocation bands. This is supported by the double spots in the SAED pattern (Fig. 2d), with two twinning systems (denoted 1 and 2) indicated by white arrows. The twinning visible in Figure 2e seems to cross the wide open lateral crack. This is an artifact due to the

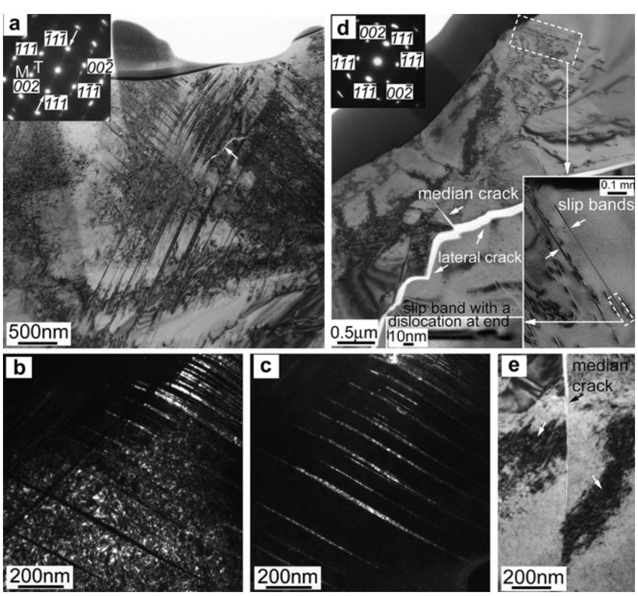

Figure 1. (a) TEM cross-section through the area damaged after indentation of GaAs with a Berkovich tip showing convergent twinning (twinning plane [1 1 1]) crossing under the indented area. The SAED pattern inset is taken in the [1 1 0] zone axis and contains double spots originating from this twinning. (b, c) Dark-field images of the twinning taken with the diffracted spots corresponding to (b) matrix and (c) twins, respectively.

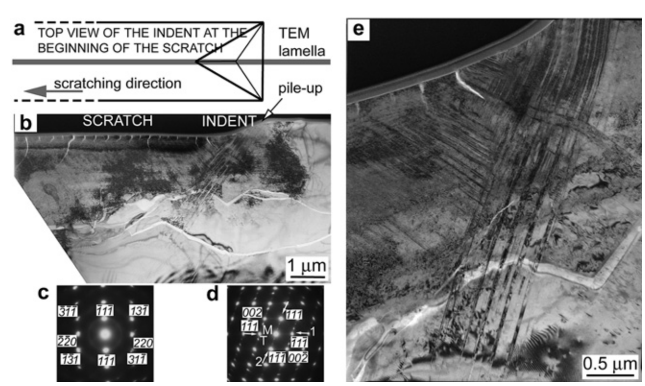

Figure 2. (a) Sketch showing the positioning of a particular lamella cut by a focussed ion beam through a Berkovich indent and the beginning of the subsequent scratch. (b) TEM bright-field general view of the indent and scratch, which is decorated by short, wavy lateral cracks. (c, d) SAED patterns taken in the [1 1 2] zone axis from the areas under the scratch and the indent, respectively. (e) Detailed bright-field view of the area under the indent with convergent twinning and the beginning of the scratch.