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F. Voillot, Michel Goiran, C. Guasch, J. Peyrade, L. Dinh, et al.. Dislocation slipping, a new technique to produce step-like surfaces, compatible with quantum confinement sizes. *Journal de Physique III*, 1993, 3 (9), pp.1803-1808. 10.1051/jp3:1993239 . jpa-00249042

HAL Id: jpa-00249042

<https://hal.science/jpa-00249042>

Submitted on 4 Feb 2008

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Classification
Physics Abstracts
78.65 — 62.20F

Dislocation slipping, a new technique to produce step-like surfaces, compatible with quantum confinement sizes

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(Received 10 November 1992, accepted 9 February 1993)

Résumé. — Nous proposons une méthode de génération de marches sur une surface de GaAs monocristallin. Cette méthode est basée sur la plasticité de monocristaux de GaAs. Dans les conditions de glissement simple, les surfaces $\{541\}$ présentent un ensemble de lignes parallèles de grande longueur (3 mm) suivant $\langle 561 \rangle$. L'observation au M.E.T. de cette surface par la technique des répliques permet de voir des zones très homogènes sur plus de $20 \mu\text{m}^2$, avec des marches de hauteur $h = 200 \text{ \AA}$ et de largeur $\ell = 400 \text{ \AA}$. Ces dimensions sont compatibles avec le confinement quantique et les surfaces ainsi obtenues sont une alternative aux surfaces vicinales ou à hauts indices utilisées pour la réalisation de fils quantiques par croissance directe.

Abstract. — We propose a method to generate steps at the surface of GaAs single crystals. This method is based on plasticity properties of GaAs. In the simple slip condition, the $\{541\}$ surfaces exhibit a set of very long parallel lines (3 mm) along $\langle 561 \rangle$. T.E.M. observations of these surfaces by replica technique allow to observe very homogeneous regions, over $20 \mu\text{m}^2$ with step height $h = 200 \text{ \AA}$ and step width $\ell = 400 \text{ \AA}$. Those geometrical dimensions are compatible with the quantum confinement and make the obtained surfaces alternate solutions to vicinal surfaces or high-index surfaces used to obtain quantum lines by direct growth.

Quantum well wires (QWW) have received intense interest over the past few years because of their physical properties and possible future applications in electronic or optoelectronic devices [1]. For experimental studies or applications in devices the most important step is the fabrication and control of nanostructured semiconductors.

A most widely used method has been the patterning of two-dimensional structures achieved by molecular beam epitaxy (MBE) [2, 3]. In the patterning processes however most of the active part is eliminated and the minimum lateral dimensions achieved are also much larger than the vertical one. Fluctuations in the wire sizes and mostly patterning defects are still major problems. We have proposed a new method [4] to pattern two-dimensional GaAs/GaAlAs structures by dislocation slipping. The size of the patterning tool being of the order of the

lattice parameters, the total active part of the sample is useable, and no lateral roughness appears.

Alternate methods based on direct growth of nanostructures on substrates having controllable terraces have been attempted too : in these methods the control of the growth processes and rates seem now well understood but the control of the starting terrace lengths or heights is still a major problem. Three approaches are proposed : use of misorientated substrates [5], grooved surfaces [6], or high-index surfaces [7].

We propose in this paper a new way to obtain such step-like surfaces extending over large areas using as in reference [4] deformation processes : according to single crystals plasticity, when a dislocation slips across the whole crystal, it does not change its perfect atomic arrangement and produces at the surfaces a step equal to its Burgers' vector. Repetition of such process several times along the same plane will produce a step at the surface controllable in height. Control of the repartition in the surface or the volume of the dislocation sources will allow control of the separation between different steps.

Although dislocations have been intensely studied in semiconductors, such steps at the surface have not been observed. In metals surface steps have been observed and steps separation of the order of 40 Å have been reported for copper crystals [8]. This value is close to those necessary to observe quantum confinement effect in semiconductors, so realisation of step-like surface using plasticity is possible. Besides, due to generation processes involved, these steps should not involve kinks or other energy-related defects as observed on vicinal surfaces.

This method is here tested on GaAs single crystals.

1. Sample and test.

GaAs plasticity in simple slip has been widely studied [9]. Figure 1 displays the variations of the elastic limit of GaAs single crystal *versus* temperature. These curves are obtained in a simple slip configuration with an imposed deformation rate ($\dot{\gamma}_p = 2 \times 10^{-7} \text{ s}^{-1}$).

From post mortem and live *in situ* microscopic observations, two regions can be identified :

- the region (1), corresponding to low temperatures in which the deformation is achieved by thermally activated slip, and where the strain, for a fixed deformation rate, increases rapidly when the temperature is lowered. At very low temperature, twinning stress is quickly reached and brittle fracture happens ;
- in the region (2), the GaAs single crystal is more ductile. High temperature mechanisms as cross slip are preponderant.

Between these two extreme situations, both deformation mechanisms are active in the transition region (3).

We have chosen our deformation conditions in the low stress part of region (1). In this case the material is ductile enough and simple slip is only involved. Moreover, this temperature is low enough to prevent arsenide evaporation.

In our experiment, we have used n type GaAs single crystals elaborated by horizontal Bridgman ($n = 10^{15} \text{ cm}^{-3}$). The mean residual dislocations density is 10^8 m^{-2} .

The edges of the parallelepipedic test-bar are cut along $\langle 123 \rangle$, $\langle 111 \rangle$ and $\langle 541 \rangle$ directions. The sample size is 12 mm \times 2.7 mm \times 3.5 mm (Fig. 2). All faces are mechanically polished. In order to perform T.E.M. on surface replica, the (541) face is mechanochemically polished.

A compressive stress is applied along the $[123]$ direction of the test-bar, in order to achieve simple slip condition for deformation. In this configuration, the Schmidt factor of the $[101](111)$ system is the highest, thus it will be the more efficient (see Tab. I). The chosen temperature is 673 K and the rate is $2 \times 10^{-7} \text{ s}^{-1}$.

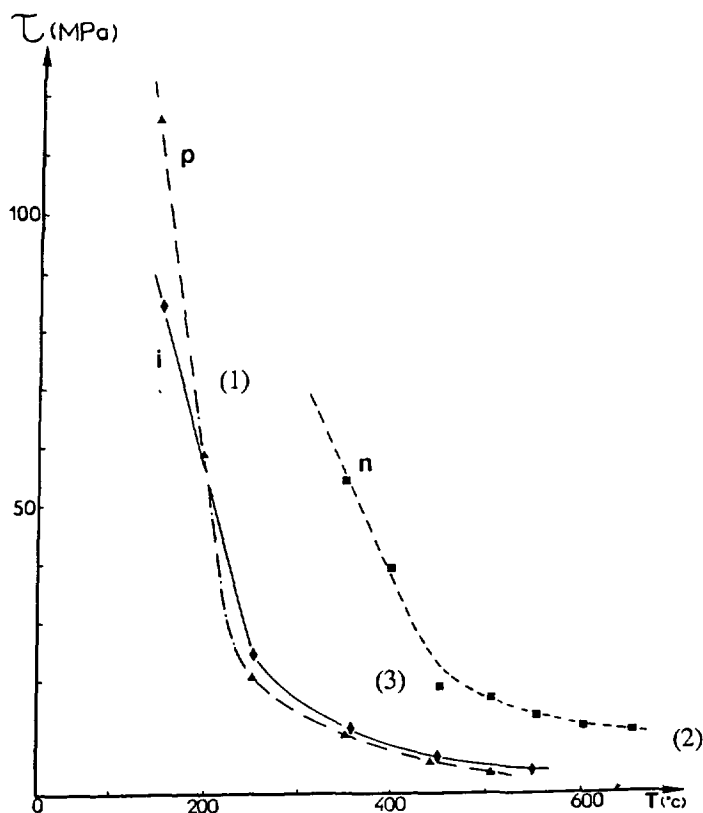


Fig. 1. — Elastic limit of intrinsic (i), p type (p) and n type (n) GaAs, versus temperature ($\gamma_p = 2 \times 10^{-7} \text{ s}^{-1}$).

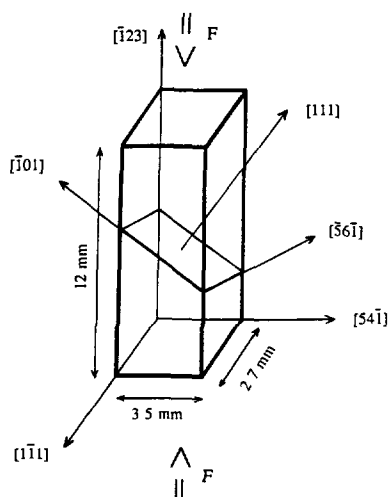


Fig. 2. — Orientation and size of the test-bar. Stress direction is indicated by the two arrows labelled F .

Table I. — Calculated Schmidt factor for stress applied along $\langle 123 \rangle$; φ is the angle between stress and plane directions ; λ is the angle between stress and \mathbf{b} directions.

Slip plane	(111)			$(\bar{1}\bar{1}\bar{1})$			$(1\bar{1}\bar{1})$			$(11\bar{1})$		
$2 \times \mathbf{b}$	$[\bar{1}10]$	$[01\bar{1}]$	$[10\bar{1}]$	$[\bar{1}10]$	$[01\bar{1}]$	$[10\bar{1}]$	$[\bar{1}10]$	$[01\bar{1}]$	$[10\bar{1}]$	$[\bar{1}10]$	$[01\bar{1}]$	$[10\bar{1}]$
φ (°)	51.88	51.88	51.88	22.20	22.20	22.20	90	90	90	72.02	72.02	72.02
λ (°)	55.46	79.10	40.89	79.10	79.10	67.79	79.10	40.89	19.10	55.46	67.79	19.10
f	0.35	0.12	0.46	0.17	0.17	0.35	0	0	0	0.17	0.12	0.29

When the single crystal is stressed, dislocation sources emit loops formed by six dislocation segments : two screw dislocations, two $60^\circ \alpha$ and two $60^\circ \beta$. These loops increase with the stress and propagate throughout the crystal. When the dislocation loop reaches the edges of the sample, a (111) plane has slipped along the $[10\bar{1}]$ direction, leaving on the $\{54\bar{1}\}$ surfaces a step characterized by the Burgers vector of the dislocation. The direction of this step is $\langle \bar{5}6\bar{1} \rangle$.

2. Surface observations.

We used double print replica method (parameters : silver thickness = $4 \mu\text{m}$, carbon thickness = 30 nm) followed by a chrome shade (thickness = 5 nm , tilting angle = 30°) (Fig. 3). These replica are then observed by T.E.M. ; due to the poorly contrasted roughness, the observations are performed in unfocused condition (Figs. 4a, b). This method allows the determination of step height $h = \ell_B \cdot \tan 30^\circ$ and width $\ell = \ell_B + \ell_{Cr}$. The most important drawback is the loss of information related to « small » steps having $h/\ell_B < \tan 30^\circ$.

Both pictures show typical observation of the topology for two different zones of the $\{54\bar{1}\}$ surface after deformation. In both cases, straight lines with length greater than $10 \mu\text{m}$ are visible. They are parallel to the $\langle \bar{5}6\bar{1} \rangle$ direction, as predicted by the chosen deformation conditions.

Figure 4a shows an inhomogeneous region which can be divided in three parts : region (a) with low ($h = 100 \text{ \AA}$) and narrow ($\ell = 300 \text{ \AA}$) steps, region (b) with higher steps

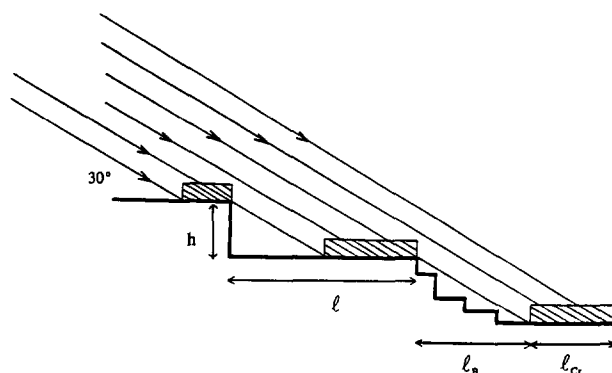
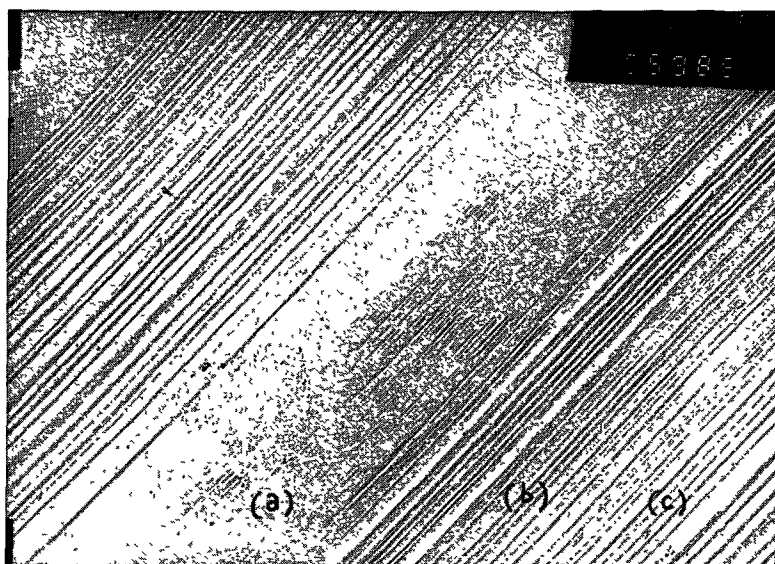
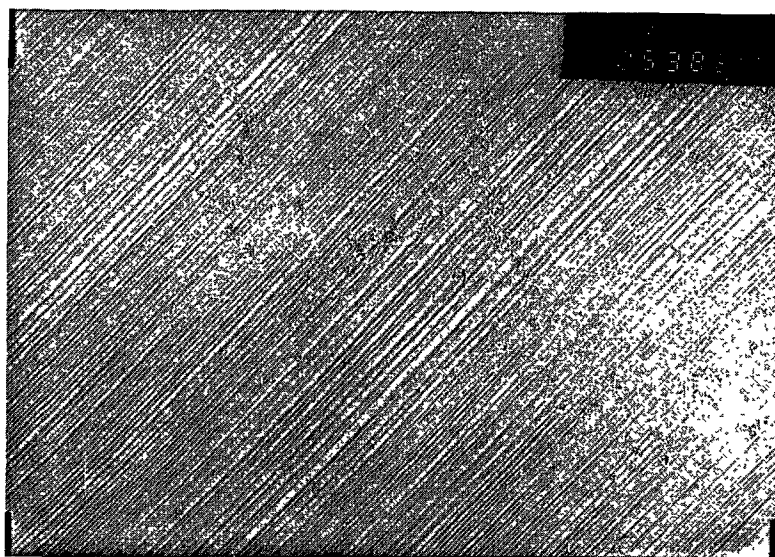


Fig. 3. — Chrome shade on double print replica ; ℓ_{Cr} is the chrome width, ℓ_B the shaded width, ℓ the whole step width and h the step height.



a)



b)

Fig. 4. — T.E.M. observation of the (541) surface replica. The dark lines are the image of the chrome deposition. Figure 4a is taken in a relatively inhomogeneous region and figure 4b in a very homogeneous one. For the two figures, 1.2 cm corresponds to $1\text{ }\mu\text{m}$.

($100 \text{ \AA} < h < 400 \text{ \AA}$) and region (c) with great height ($h = 450 \text{ \AA}$) and width ($\ell = 1\,000 \text{ \AA}$). Due to the method, this last region can be regarded as an inhomogeneous region made of steps comparable to the one of the (a) region but masked by some higher steps (see Fig. 3).

Figure 4b shows a relatively homogeneous region over $20 \mu\text{m}^2$ area, in which the height h value is between 100 \AA and 400 \AA , and the step width ℓ between 400 \AA and 600 \AA . In the high left corner of the picture exists a very homogeneous region composed by steps with $h = 200 \text{ \AA}$ and $\ell = 400 \text{ \AA}$.

Those results confirm that deformation of GaAs single crystal, in simple slip conditions, can generate step-like surfaces with geometrical characteristics compatible with quantum confinement. Steps height is about 10 times greater than the lattice width, so subsequent deposition by M.B.E. on such surfaces would lead to the generation of quantum lines in an easy way.

Theoretically the lateral roughness is no more than the atomic step, but the observation method used make it maximum to 40 \AA . The main problem is the heterogeneity of the surface on a large scale due to secondary slip, sources distribution and local hardening, so these structures will be useable only with local characterization techniques.

Conclusion.

We demonstrated that plasticity properties such as generation of slip lines by dislocations slip are efficient to generate $\{541\}$ step-like surfaces. The terrasses height and width are consistent with quantum confinement conditions. This process is an alternate to angle polished vicinal surfaces.

The obtained surfaces are not flat over a large scale but show areas of several μm^2 which exhibit a very good homogeneity in terrasses geometrical dimensions.

Nevertheless, to our knowledge, M.B.E. growth on $\{541\}$ surfaces is not developed yet. For this reason we have started a study of deformation systems on (100) GaAs substrates, commonly used as a substrate for M.B.E. growth.

Acknowledgments.

We wish to thank Mr Saur for preparation of the replica used in this work.

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