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# Surface Treatment and Layer Structure in 2H-GaN Grown on the $(0001)_{Si}$ surface of 6H-SiC by MBE

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## Abstract

Heteroepitaxy of hexagonal symmetry materials is more complicated than in the more usual case of cubic systems. In the growth of layers on the (0001) surfaces, the misfit dislocations always exhibit a screw component that leads to rotation of the epilayer in a 3 dimensional growth mode. The size of the islands will depend on many factors among which the substrate surface treatment, prior to growth, may be a predominant one. In this work, a comparative study is carried out for samples grown on plasma treated samples, with and without additional substrate annealing prior to epitaxy. It is found that the defect density can be brought below $10^9$ $cm^{-2}$, which is better than one order of magnitude in comparison to the layers grown on sapphire substrates. On top of the annealed substrates, the island growth is not obvious. Whereas, misorientations as large as a few degrees can be measured inside the layers on top of non annealed substrates, justifying the occurrence of high densities of threading dislocations.

## 1. Introduction

Due to their wide band gap, GaN based wurtzite semiconductors have become very promising optoelectronic materials. Efficient laser diodes have now been fabricated for emission in the blue range [1]. The best layers are grown on sapphire by Metal Organic Vapour Epitaxy (MOVPE). Using this method, the growth is out of equilibrium and better results than Molecular Beam Epitaxy (MBE) have been achieved [2].

Silicon carbide (SiC) is an important substrate because of the possibility to combine power devices on SiC and optoelectronic compounds in the GaN based layers. Moreover, a study of the structure in samples grown by MBE is important as it can allow understanding of the growth mechanisms of these layers. Apart from the fact that SiC is still too expensive, surface cleaning has been a problem until the recent use of hydrogen plasmas for surface deoxidization. Nowadays, before growth on SiC, chemical cleaning is followed by $H_2$ plasma. Then one can apply or not, an additional thermal annealing.

In this work, a series of GaN/SiC, GaN/AlN/SiC samples have been investigated by Transmission Electron Microscopy (TEM) after various treatments before MBE growth. Although the density of defects is always high (> $10^8$ $cm^{-2}$), it is found that additional heat treatment of the substrate before growing contributes to improving the grown layer quality.

## 2. Experiment

For the growth on SiC, the $(0001)_{Si}$ surface was systematically used. In the analyzed samples three cases are dealt with:

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a. chemical cleaning in a HF based solution
b. additional $H_2$ plasma treatment (for details on chemical cleaning and plasma treatment see reference [3]).
c. additional heat treatment was carried out at 1300°C under $H_2$ for 30 min.

The MBE was carried out at 800°C, using ammonia or an Electron Cyclotron Resonance source (ECR) for $N_2$. In cases a and c, the layers were simultaneously grown and a 50 AlN buffer layer was deposited prior to GaN at the same temperature.

TEM samples were prepared in the conventional way by mechanical polishing followed by ion milling down to transparency. HREM was carried out on an ABT 002 microscope operating at 200 kV with a point to point resolution of 0.18 nm and conventional TEM was carried out in JOEL EM2010 microscope.

# 3. Results

The objective of this comparative study was to investigate a series of samples whose surfaces were prepared in different ways to try to understand the role of the surface preparation on the quality of the epitaxial layers. Examples are given for chemical cleaning; chemical cleaning + $H_2$ plasma and additional annealing of the SiC substrate at 1300°C under $H_2$ for 30 min.

In MBE samples typical defects are found in all samples, their densities are probably dependent on the substrate surface preparation as well as on the growth procedure. These defects are mainly dislocations which thread across the epitaxial layer [4] and $\{11\bar{2}0\}$ prismatic stacking faults which were found to be mainly confined inside the first 200 nm near the interface with the substrate [5]. Their atomic structure has been completely determined and their fault vector is $1/2\langle 10\bar{1}1\rangle$ [6]. More than 90 % of the observed threading dislocations have an **a** character, and only a few **c** ones are observed [4].

## 3.1. Chemical cleaning

When the epitaxy was carried out directly after chemical cleaning the layers always exhibit 3 dimensional growth, the defect densities are very high ($3x10^{11}$ $cm^{-2}$). In the worst cases (Figure 1), the prismatic $\{11\bar{2}0\}$ stacking faults cross the whole layers which exhibits a pronounced mosaic structure. The stacking faults, which are known to originate from the surface steps [6],accommodate displacements along the c axis between neighbouring domains which are rotated following their relaxation by the formation misfit dislocations

## 3.2. Chemical cleaning + $H_2$ plasma

When an additional $H_2$ plasma step is added to the chemical cleaning, the dislocation density can be lowered by another order of magnitude in the $10^{10}$ $cm^{-2}$ range (Figure 2) and it can be noticed that the domains are slightly larger than in the above case. The observed defects are the threading dislocations that are still mostly **a** type. The prismatic stacking faults are now only in the interface area, they may form closed domains or be terminated by partial dislocations [6] [7]. In some cases, $\{10\bar{1}0\}$ domains are observed in the interface area ( Figure 3). When examined in HREM, these domains contain amorphous material (Figure 4). In planar view, they are clearly limited by $\{10\bar{1}0\}$ facets and some of them exhibit an **a** dislocation character [8]. They constitute another factor that seems to be a consequence of the three-dimensional growth mode of the GaN layers on SiC.

## 3.3. High temperature annealing

After a 1300°C substrate annealing subsequent to the above treatments, one gains another order of magnitude in the density of dislocations, it drops now to the $10^8$ $cm^{-2}$ range (Figure 5). As this sample was grown simultaneously with the one shown in figure 2 above, the dislocation density decrease is only due to higher temperature annealing of the substrate. This is a clear indication that the structure of the GaN epitaxial layers on SiC is critically dependent on the surface of the substrate just before epitaxy.

Still, the largest fraction of the threading dislocations has an **a** character, however at the surface of the

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substrate, some impurities have segregated during the high temperature step. They are W crystallites probably due to contamination from the W of the filament used for the 1300°C annealing, and on top of them, **c** or **a**+ **c** threading dislocations originate (figure 6).

## 4. Conclusion

During the growth on top of the (0001) surface, the 3.54% lattice mismatch between GaN and SiC is released by the formation of misfit dislocations that always have a screw character. In the worst cases, they can even be pure screw **a** dislocations.

1. The steps lead to the nucleation of the layer in well separated areas of the surface followed by the growth of islands as a consequence of the large misfit involved. The stress in each island is then relieved by the formation of misfit dislocations.

2. The screw dislocations introduce systematically a rotation of each island about the (0001) growth directions [9], [10].

3. The coalescence of the islands leads to the formation of $\{11\bar{2}0\}$ prismatic defects from adjacent islands separated by adequate steps [5], [6].

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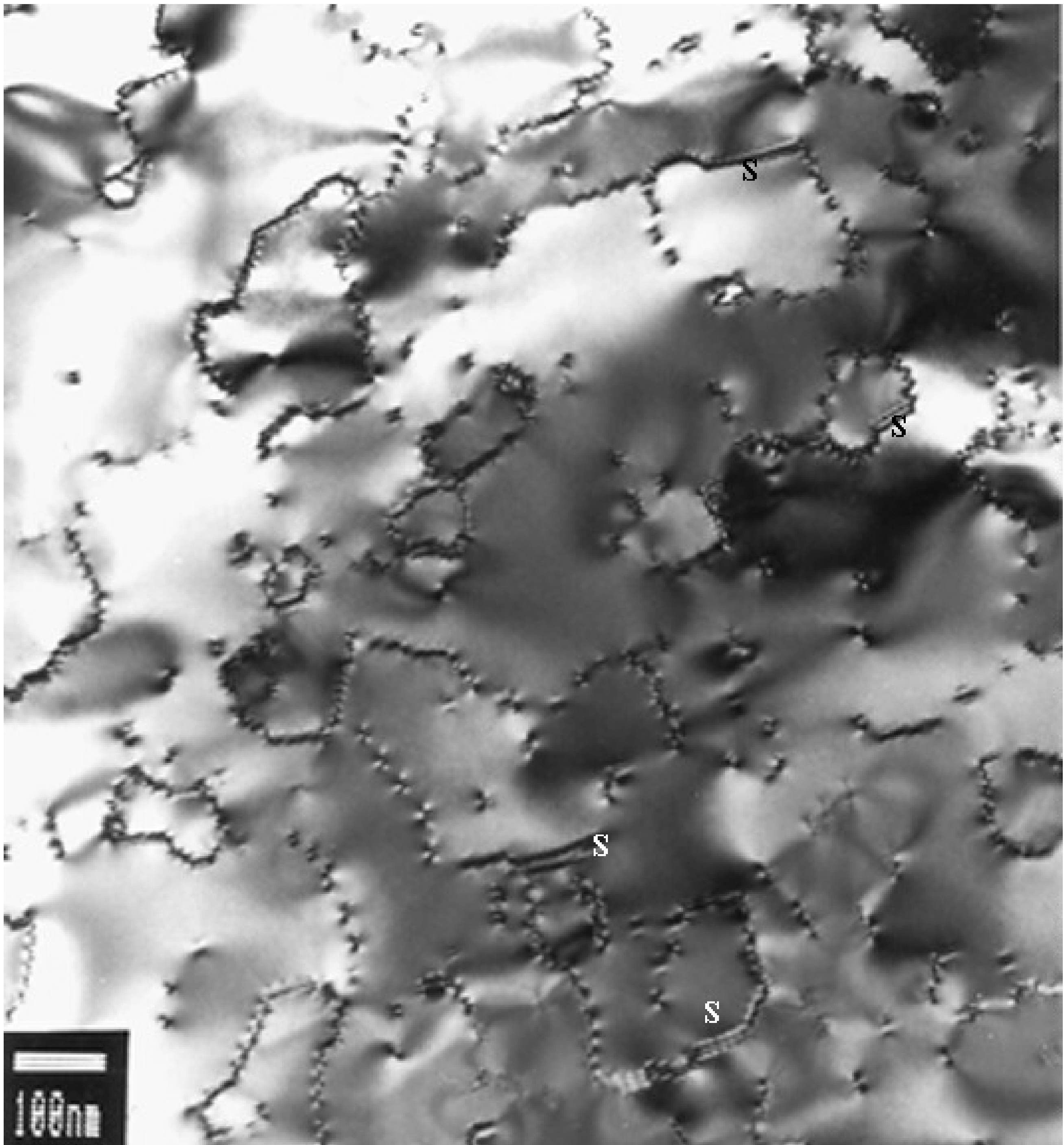


**Figure 1**. Microstructure of a GaN layer grown on 6H-SiC $(0001)_{Si}$ surface after chemical cleaning, micrograph taken close to [0001] zone axis, S: the $\{11\bar{2}0\}$ stacking faults.

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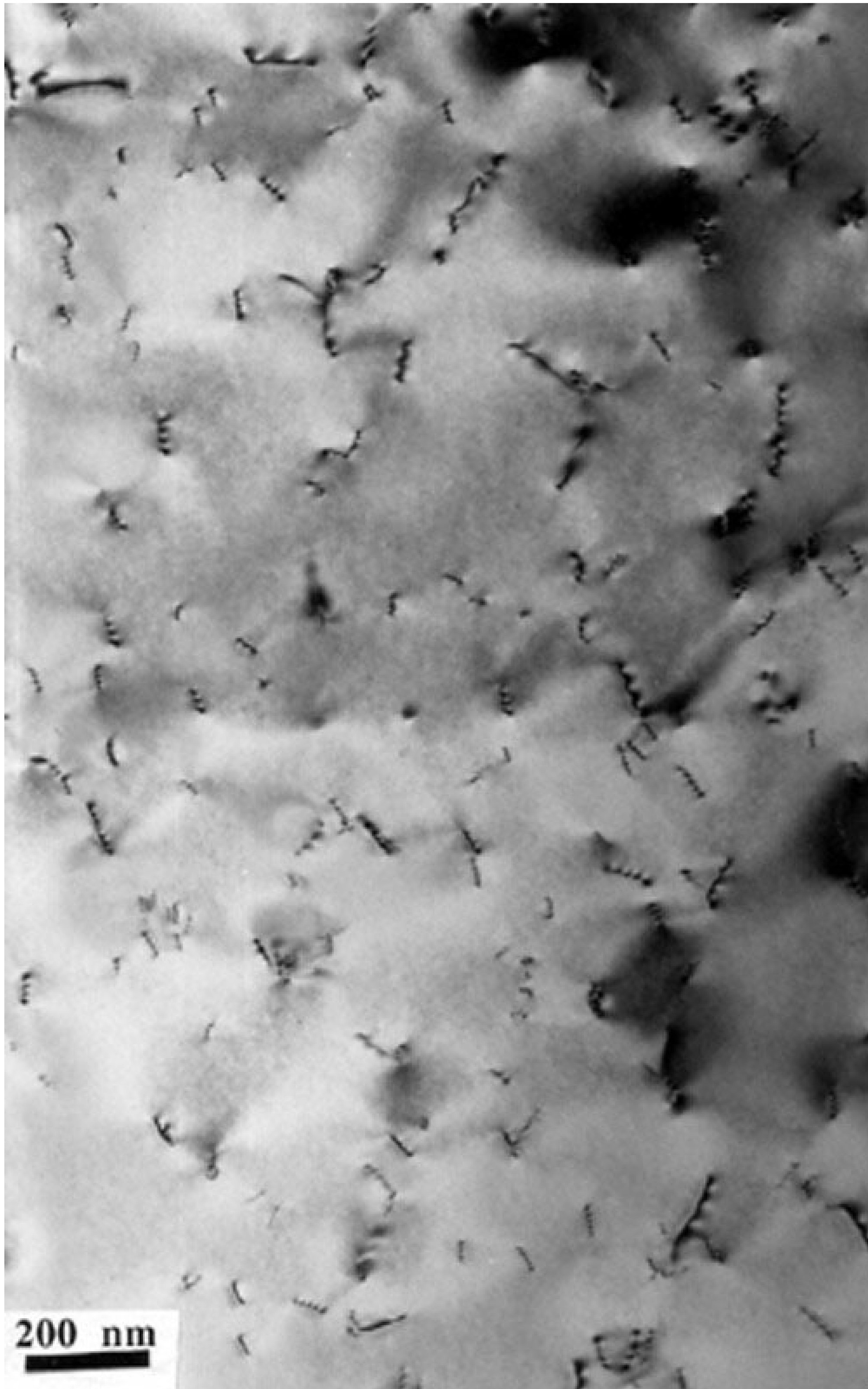


**Figure 2**. Morphology of a GaN layer grown on a hydrogen plasma cleaned SiC substrate, BF image taken at $g = 00\bar{1}1$.

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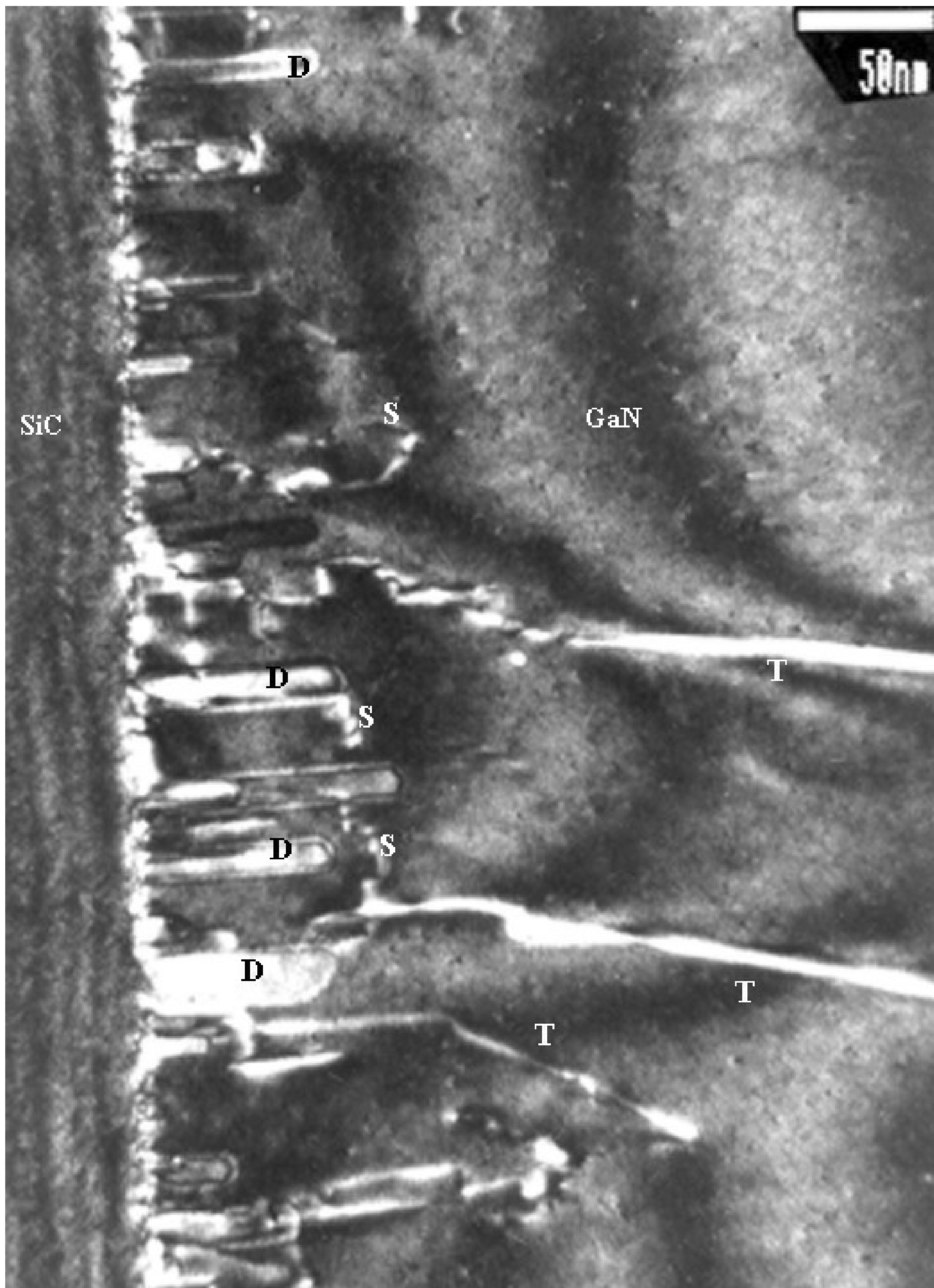


**Figure 3**. Cross section TEM view showing S: {11$\bar{2}$ 0} stacking faults and D: {10$\bar{1}$ 0} domains, the image is underfocussed in order to enhance the contrast of the domains walls, g: 0002,T: the threading dislocations which have a **c** component.

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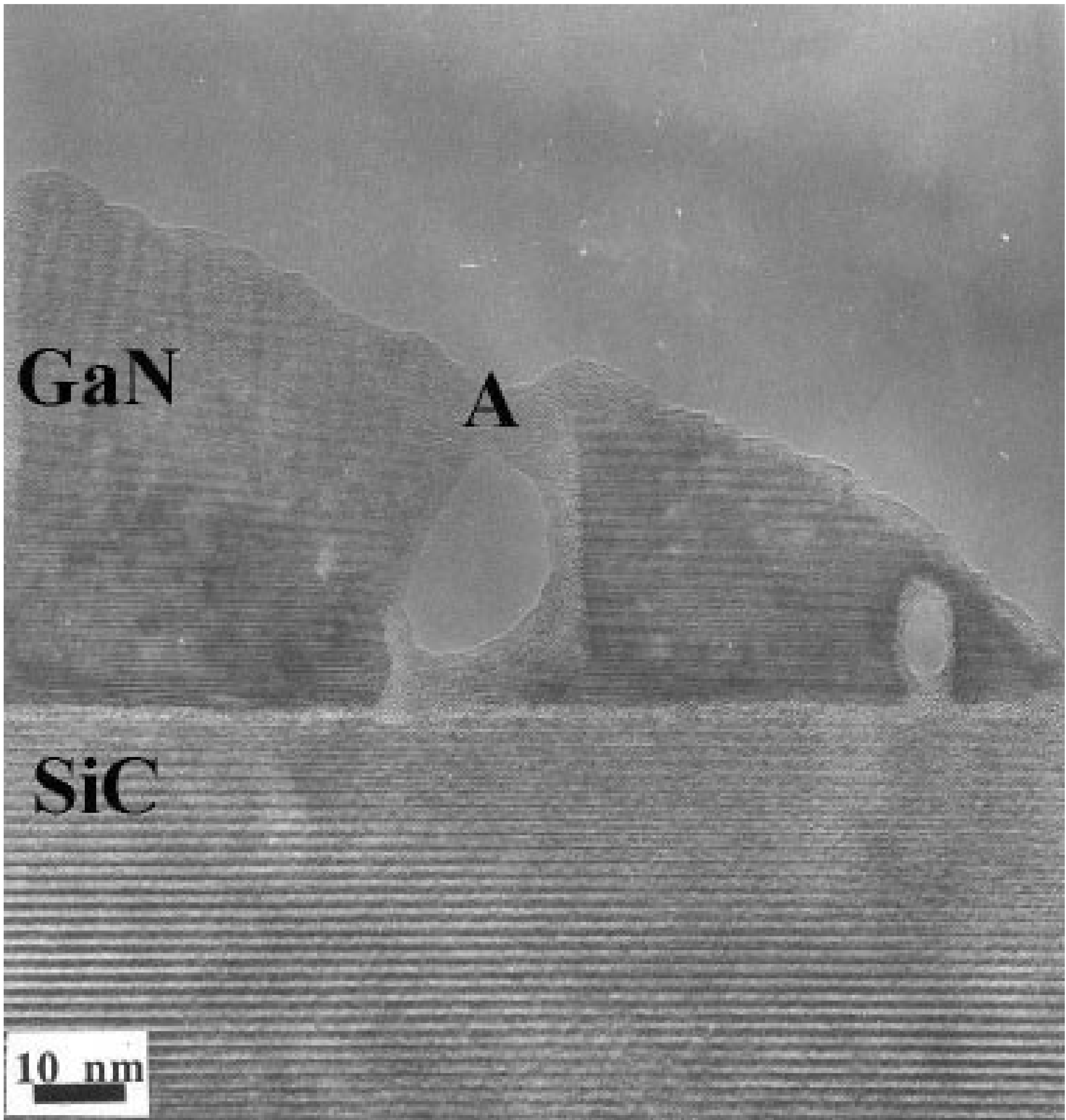


**Figure 4**. The $\{10\bar{1}0\}$ domains: HRTEM image along the $[11\bar{2}0]$ zone axis, A: remaining amorphous of the domain.

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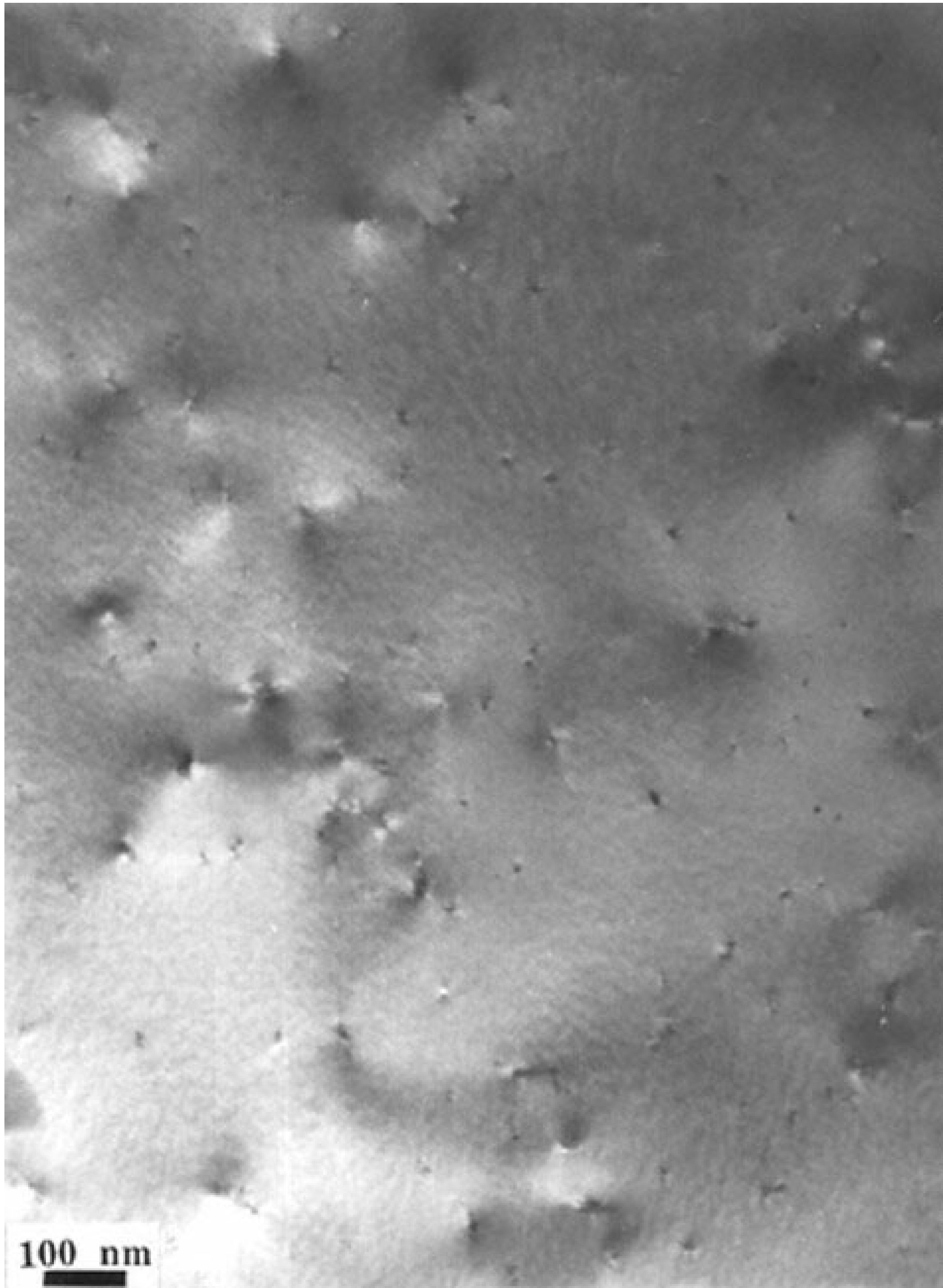


**Figure 5**. Micrograph of GaN layer grown on high temperature SiC substrate: weak beam, dark field image, g: 0002.

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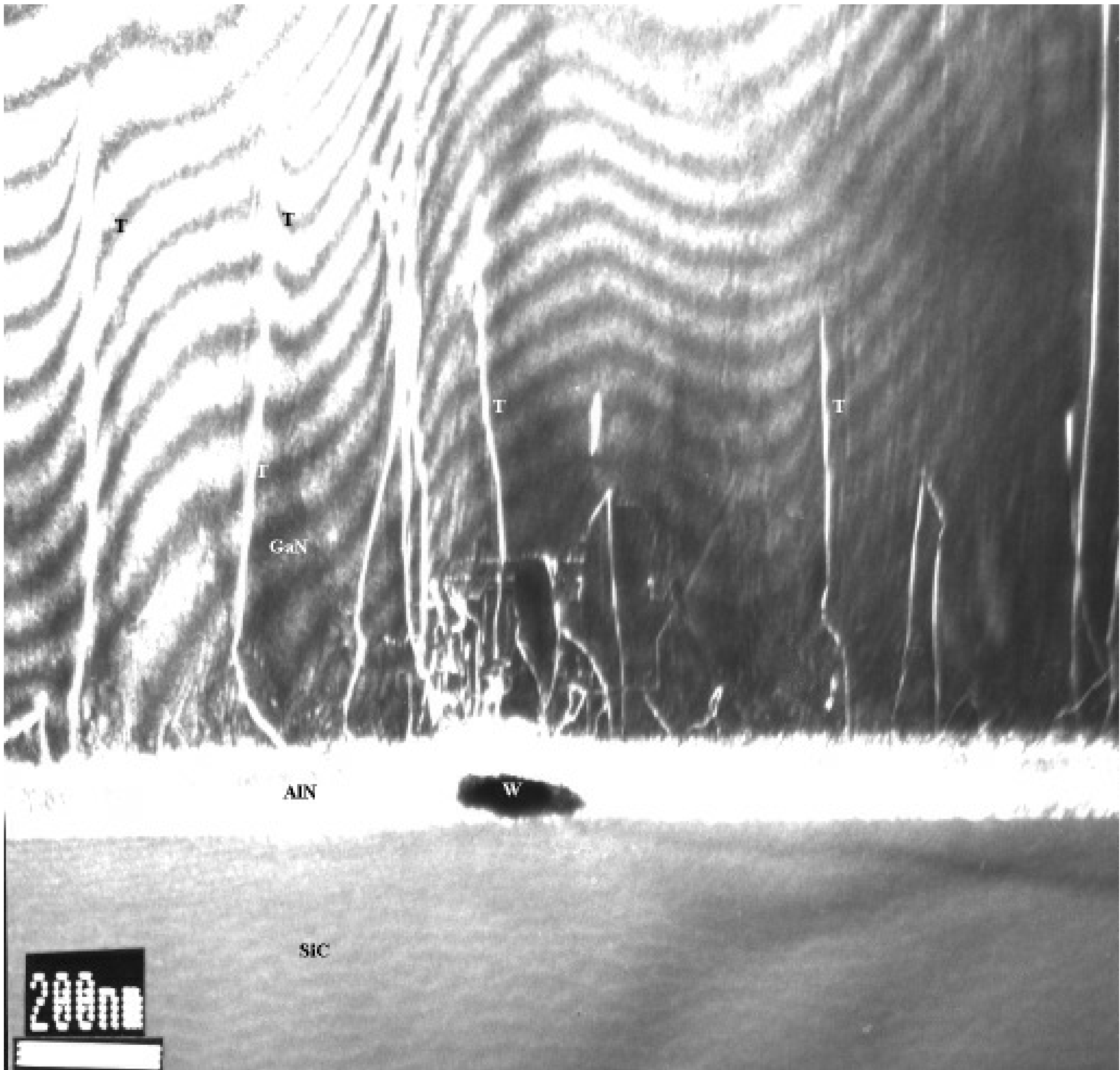


**Figure 6**. Precipitates at the SiC surface which originate in **c** and **a+c** dislocations.

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