REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

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1. REPORT DATE ***(DD-MM-YYYY)***
13-04-2006

2. REPORT TYPE
Final Report

3. DATES COVERED *(From – To)*
17 January 2005 - 17-May-06

4. TITLE AND SUBTITLE

MOCVD Growth and Etching of N-Type GaN Layers on HPVE-Grown Templates and Free-Standing GaN Substrates

5a. CONTRACT NUMBER
FA8655-05-1-3020

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER

5d. TASK NUMBER

5e. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
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8. PERFORMING ORGANIZATION REPORT NUMBER

N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

EOARD
PSC 821 BOX 14
FPO 09421-0014

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
Grant 05-3020

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report results from a contract tasking Radboud University Nijmegen as follows: The Grantee will investigate the removal of dislocations from the GaN epitaxial layer by defect-selective etching technique and comparing properties of the as-grown (i.e. containing dislocations) and dislocation-free material. This approach will allow electrical or optical measurements on the epitaxial layers with and without dislocations. The influence of dislocations on the measured material properties will also be assessed.

15. SUBJECT TERMS
EOARD, Semiconductor materials

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18, NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			MATTHEW MORGAN, Lt Col, USAF
UNCLAS	UNCLAS	UNCLAS	UL	8	19b. TELEPHONE NUMBER *(Include area code)* +44 (0)20 7514 4505

Report
MOCVD growth and etching of n-type GaN layers on HVPE-grown templates and free-standing GaN substrates

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Nijmegen, 10 June 2005

Part II: growth and characterization of Ga-polar layers

1. Experimental details

The aim of Part II:

- Growth of 0.5 micron n-type GaN layer on SI HVPE-grown, Zn-doped GaN templates and on SI MOCVD-grown, Fe-doped GaN (from Lumilog);
- Defect-selective "orthodox" etching in molten bases (eutectic KOH+NaOH=E, see ref. [7] in the first part of this report) in order to remove dislocations;
- Electrical and optical measurements of as-grown and etched n-type layers in order to find a possible influence of defects (different types of dislocations) on properties.

GaN Si-doped n-type layers were grown using standard MOCVD procedure. The time of growth was adjusted to obtain 0.5 μm thick layers. The following growth processes were performed, as specified in Table I:

Table I

HVPE template	MOCVD process	Estimated N [cm^{-3}]
#AZUR 04-140C (GaN:Fe)	MGaN 1170	~ $4x10^{18}$
#2191 (GaN:Zn)	TG 378	~ $4x10^{17}$
#2191 (GaN:Zn)	MGaN 1170	~ $4x10^{18}$
#2190 (GaN:Zn)	MGaN 1172	~ $4x10^{18}$

#AZUR 04-140C (GaN:Fe)

The surface of a quarter of 2 inch wafer AZUR 04-140C had many "crescent" growth features (see Fig. 1). This type of surface irregularities constitute effective source of steps during orthodox etching, therefore it was necessary to polish this material before MOCVD deposition of the n-type GaN layer. The quarter of this wafer was cleaved into three parts and polished mechano-chemically using in-house developed technology. The thickness of the GaN:Fe layer was only 3 μm, which allowed only very shallow polishing. The images of the surface after polishing are shown in Fig. 2: numerous structural features are revealed by DIC optical microscopy. It can be tentatively concluded that these are inclusions (precipitates?) formed most probably due to Fe doping. The presence and inhomogeneous distribution of these structural features could explain some lateral inhomogeneity of Fe distribution as was reported after SIMS analysis of the GaN sample from the same 2 inch wafer (see Report Howard Smith, 1-Apr-2005, CAMECA SIMS data\Claflin Fe in GaN Mar 05\Claflin Fe in GaN Apr 05.ppt).

After MOCVD growth the layer had extended morphological defects, which might have been formed due to the presence of micro-defects shown in Fig. 2. Two best samples have been selected, one in as-grown state (#AZUR-3) and the second was selectively etched in molten eutectic of KOH+NaOH=E (#AZUR-1). Both were sent to Wright State Center, Dayton for electrical and optical examination. The image of the etched sample is shown in Fig. 3.

#2191 and 2190 (GaN:Zn)

The HVPE-grown templates (#2191 and 2190, thickness of the GaN layers ~60 μm) had flat surface on the majority of the 2 inch wafers and were used as-delivered for MOCVD growth. The following samples were selected, etched and submitted to Dayton for measurements:

Table II

Code	Etching conditions E/t[°C]/time	Figure
#2190-7	As-grown	
#2190-4	E/380/1.5'	
#2190-5	E/380/2'	
#2190-1.6	E/240/60'	
#2191-13.3	As-grown	
#2191-13.2	E/380/2'	Fig.4a
#2191-13.4	E/380/1.5'	
#2191-13.5	E/240/60'	Fig.4b, Fig.5

The selection of remarkably different etching temperatures and time is based on the following reasoning. In the first report (J.L. Weyher, "Study of the influence of dislocations on electrical properties of GaN hetero-epitaxial layers", Warsaw, 12 December 2003) it was shown that in order to obtain deep pits (up to 0.5 μm) with large angle of inclination of the side walls of the etch pits (α ≥ 45°) it is necessary to perform etching in relatively low temperature (200-240°C) and for a long time (up to 60'). In that report, however, one parameter could not have been judged correctly, namely the total density of dislocations revealed at low temperature in the 0.5 μm thick MOCVD-grown GaN layer. The HVPE-grown templates had large growth hills, which resulted in the presence of numerous steps on the surface and selective etching of dislocations only in the valleys (see Fig.8b-c and the comments in the above mentioned Report). Detailed

study performed later (J.L. Weyher, L. Macht, S. Krukowski, unpublished) showed that revealing of all types of dislocations (edge, mixed and screw) in HVPE- and MOCVD-grown GaN requires etching in molten E at temperatures above 350ºC. In addition it was found, that the difference in the activation energy of formation of pits on dislocations with screw component is not large, which suggests that it is caused rather by differences in dislocation decoration and not by differences of the Burgers vector. It was concluded, therefore, that activation energy of these dislocations might be dependent on the growth method, doping type and level. Secondly, the dislocations with large Burgers vector, i.e. screw-type, are etched as first at much lower temperature than that, critical for revealing edge dislocations (i.e. 350°C). All these conclusions are supported by the results obtained by etching samples #2190-5, #2190-4, #2191-13.2, #2191-13.4 at 380°C for 1.5-2 minutes and #2190-1.6, #2191-13.5 at 240°C for 60 minutes. As a matter of example Fig. 4 shows the result of etching at high and low temperature and Fig.5 confirms that the depth of pits after etching at low temperature is comparable with the thickness of the n-type GaN layer.

These experimental facts indicate that there is a basic contradiction in the necessity of etching at low temperature in order to obtain large α and etching at high temperature for removal of all dislocations. The following experimental procedure could be suggested in order to overcome this problem: first the samples should be etched at low temperature/long time in order to remove dislocations with screw-type component of Burgers vector. After performing electrical and optical measurements the samples should be etched second time at high temperature in order to form pits on the low energy edge-type dislocations and the electrical/optical measurements should be repeated. This refers specifically to samples labelled #2190-1.6 and #2191-13.5 which should be etched second time at high temperature in order to remove the edge-type dislocations.

The main uncertainty: for removing the edge dislocations it is necessary to perform etching at 360ºC (or higher) for relatively long time. At the same time the pits formed already on the screw-type dislocations will grow remarkably and the inclination angle of the side walls might become smaller. These growing pits may "sweep" the pits forming on the edge-type dislocations. But this uncertainty will be verified experimentally after second etching.

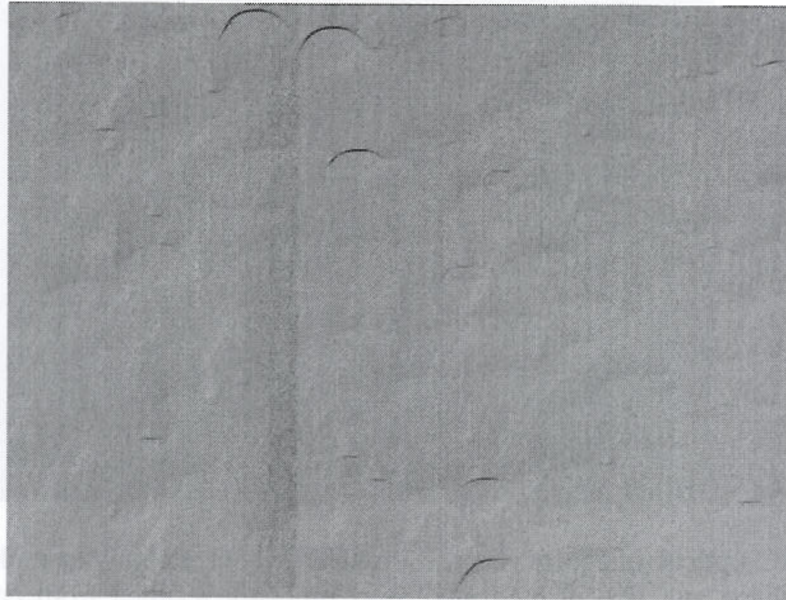


Fig.1. DIC optical image of as-grown sample #AZUR 04-140C (GaN:Fe).

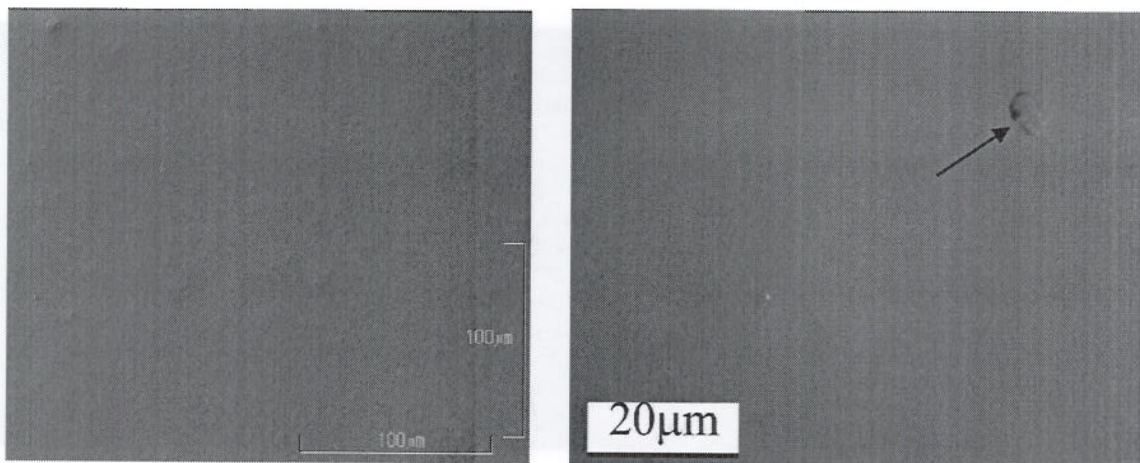


Fig.2. DIC optical images of sample #AZUR 04-140C (GaN:Fe) after mechano-chemical polishing. The arrow indicates large size inclusion.

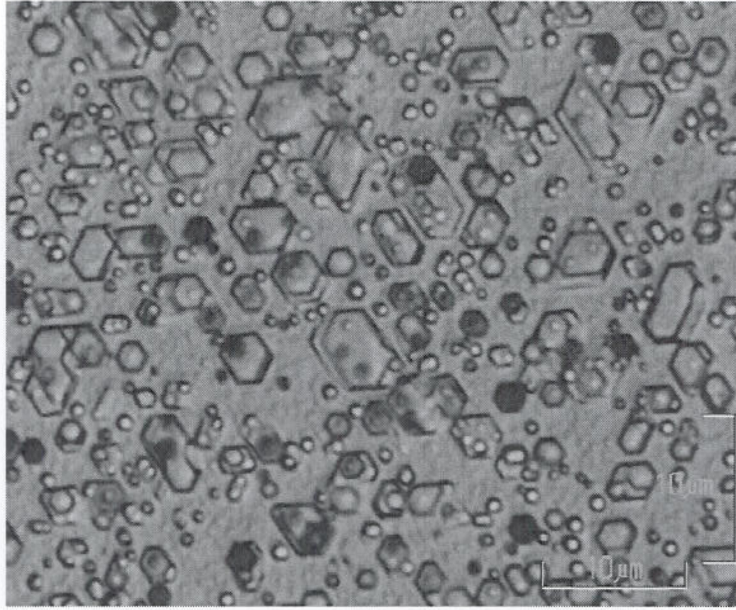
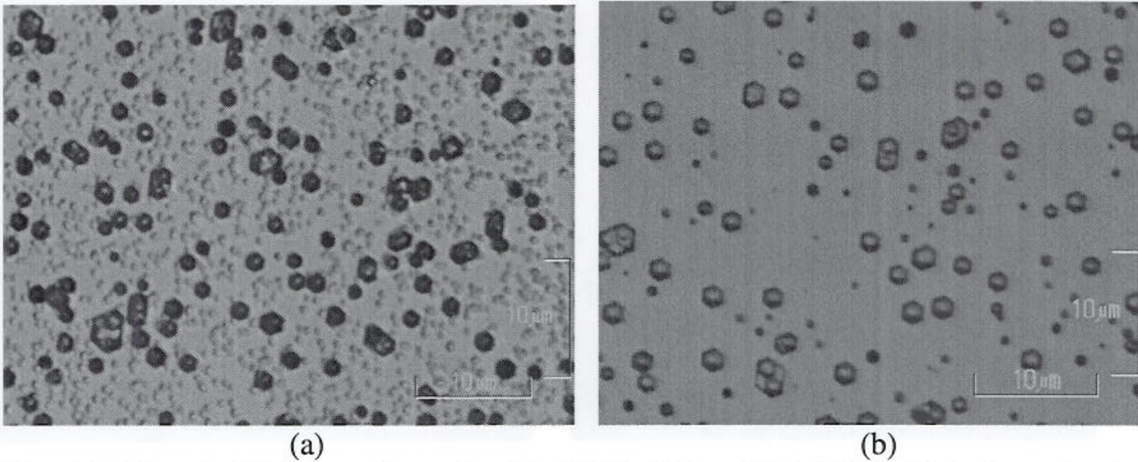


Fig. 3. DIC optical image of sample #AZUR-1 after etching in molten E. Note very irregular shape and size of etch pits, most probably resulted from complex pattern of dislocations formed on micro-defects emerging at the surface of polished GaN:Fe substrate.



(a) (b)

Fig. 4. DIC optical images of samples (a) #2191-13.2 and (b) #2191-13.5 after selective etching in conditions specifies in Table II.

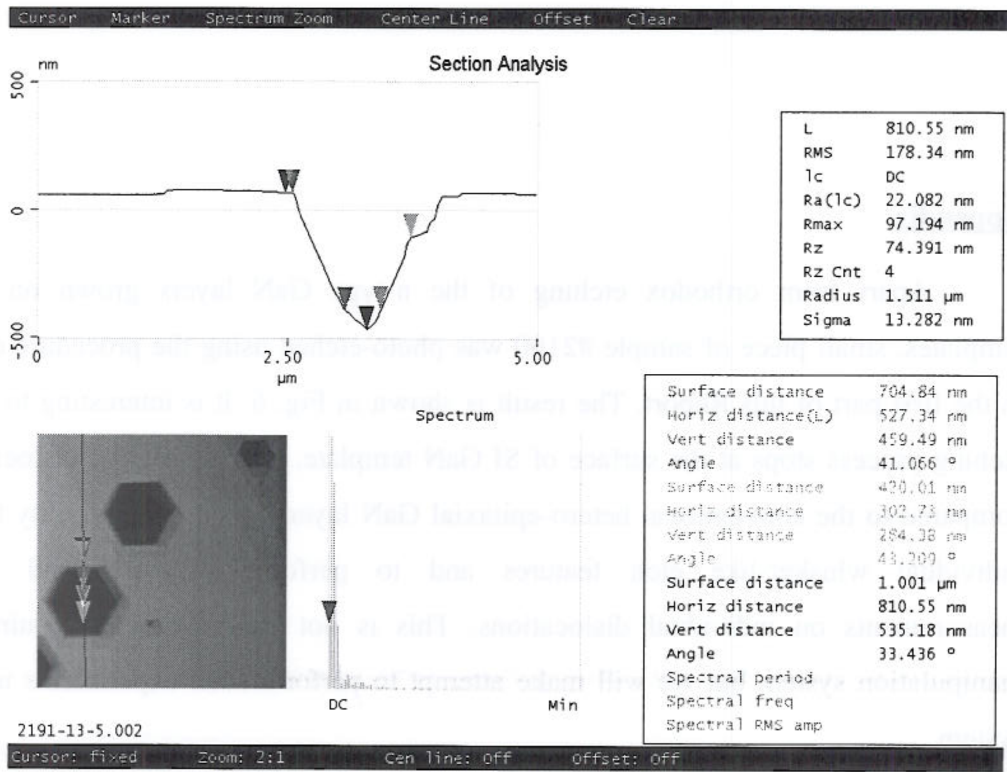


Fig. 5. AFM image and section profile across large pit in sample #2191-13.5. The depth of the pit is 535 nm and the inclination angle of the side walls of the pit α=41-43°.

Acknowledgement of Sponsorship:
Effort sponsored by the Air Force Office of Scientific Research, Air Force Material Command, USAF, under grant number FA8655-05-1-3020. The U.S. Government is authorized to reproduce and distribute reprints for Government purpose notwithstanding any copyright notation thereon.

Appendix.

Apart from orthodox etching of the n-type GaN layers grown on SI GaN templates, small piece of sample #2190 was photo-etched using the procedure described in the first part of this Report. The result is shown in Fig. 6. It is interesting to note that etching process stops at the surface of SI GaN template. Low density of dislocations, as compared to the conventional hetero-epitaxial GaN layers, opens a possibility to extract individual whisker-like etch features and to perform structural and electrical measurements on individual dislocations. This is not easy task and requires nano-manipulation system but we will make attempt to perform such experiments using FIB system.

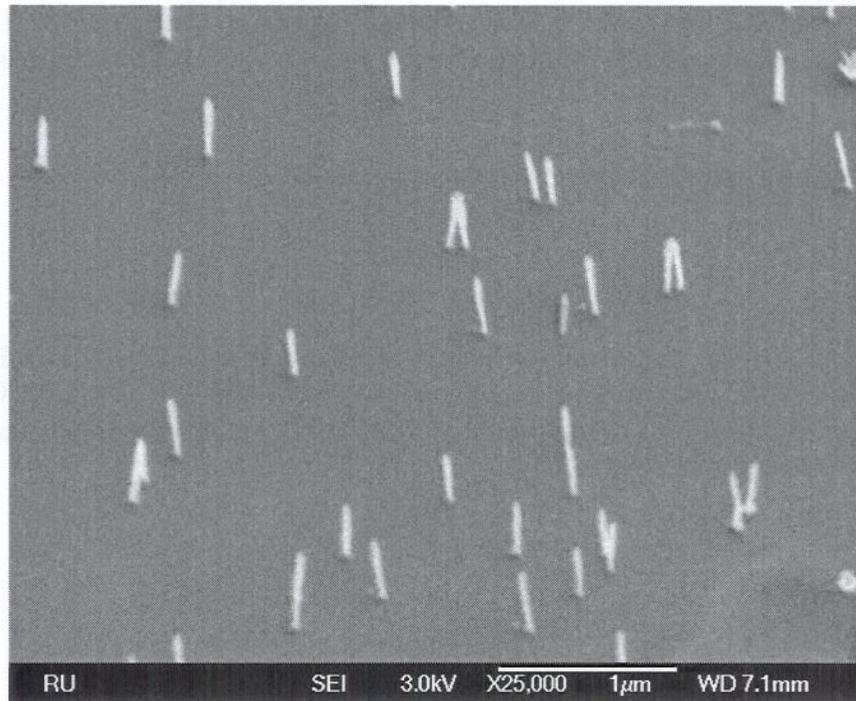


Fig. 6. SEM image (45°. tilted sample) of PEC-etched MOCVD-grown n-type GaN layer (sample labeled #2190-1.1).