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AVS JTG, Fall 2017 Talk

Evaluation of crystal damage in Mg implanted GaN by RBS, 3D-Atom Probe and TEM

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Outline

1. Background / Motivation
2. Samples
3. Experiment
4. Result
5. Conclusion

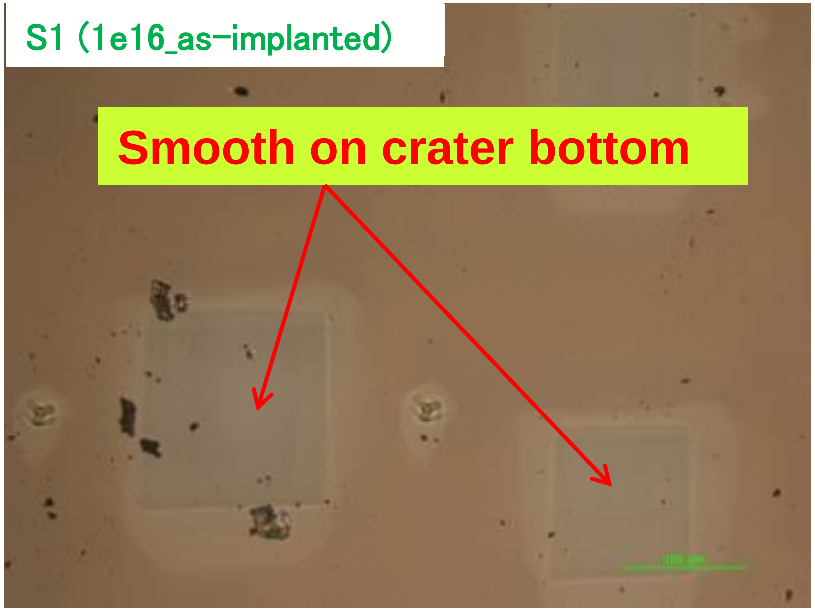
Background・Motivation

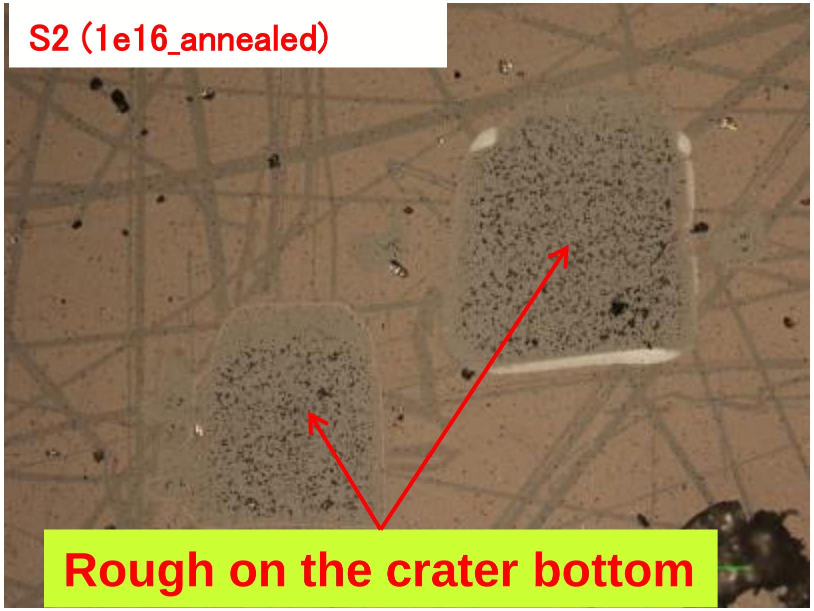
GaN is an attractive material for high-power and high-frequency devices due to its wide band gap and high saturation velocity. However it is difficult to achieve p-type conduction by Mg ion implantation.

In order to know the cause of the difficulty of p-type formation, as a basic research we measured Mg ion implanted GaN crystal samples with various implanted condition before and after annealing by Rutherford Backscattering Spectrometry (RBS), 3D-Atom Probe (3DAP) and TEM.

Background・Motivation

In addition, we performed SIMS analysis to see Mg depth profile with this round of experiments .
We found roughness at SIMS crater bottom in the annealed sample and saw a difference of roughening between as-implanted and annealed samples. It was a factor to further investigate the cause of the difference in roughness.





Optical microscope photo on Crater bottom in 1E16 as-implanted and 1E16 annealed after SIMS measurement are shown.

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Samples

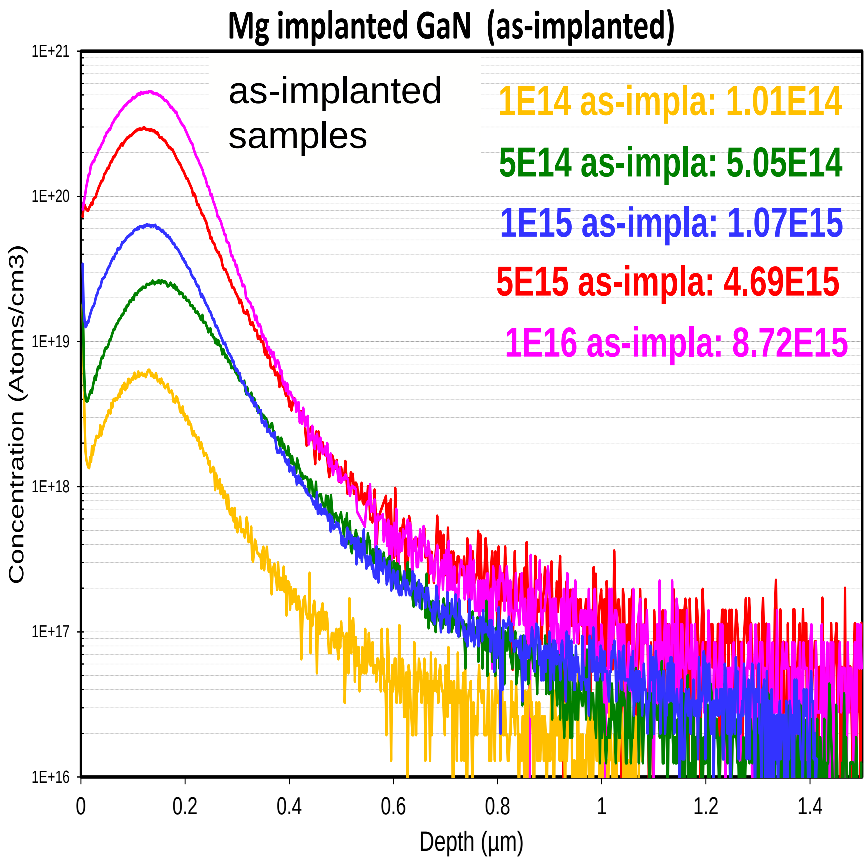
- c plan GaN substrate
- Implanted with room temperature
- 150keV
- five types of implanted dose

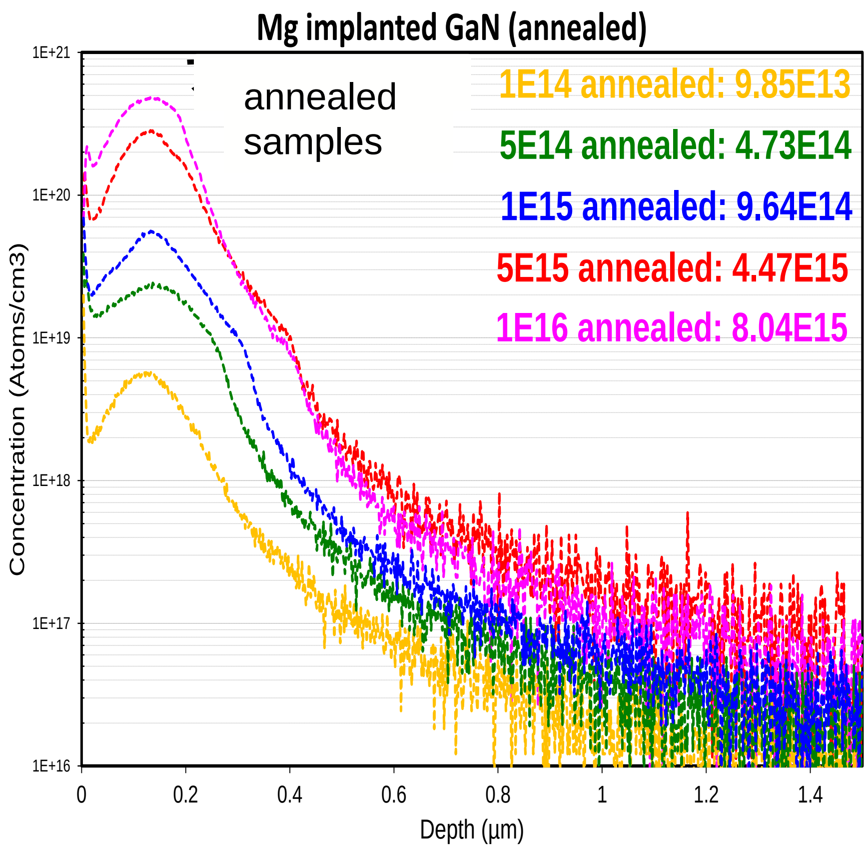
	Dose (atoms/cm2)	Annealed
S1	1e16	
S2	1e16	○
S3	5e15	
S4	5e15	○
S5	1e15	
S6	1e15	○
S7	5e14	
S8	5e14	○
S9	1e14	
S10	1e14	○

Annealing condition：1230° C _1min_Nitrogen ambience, with SiN cap

Pre-test : Mg depth profile by SIMS

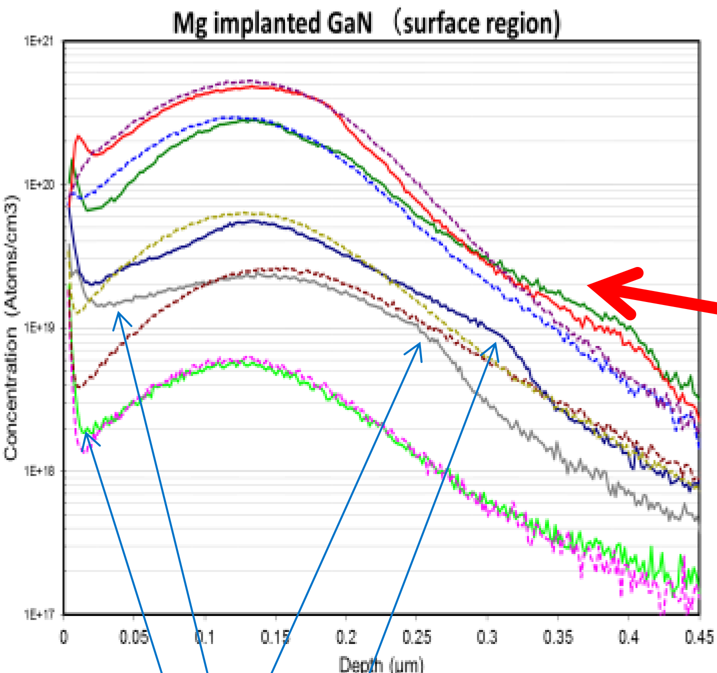
- **At the beginning, SIMS analysis was performed to confirm Mg depth profile.**
- **MgCs+ secondary ion was detected using 3keV,Cs primary ion bombardment.**

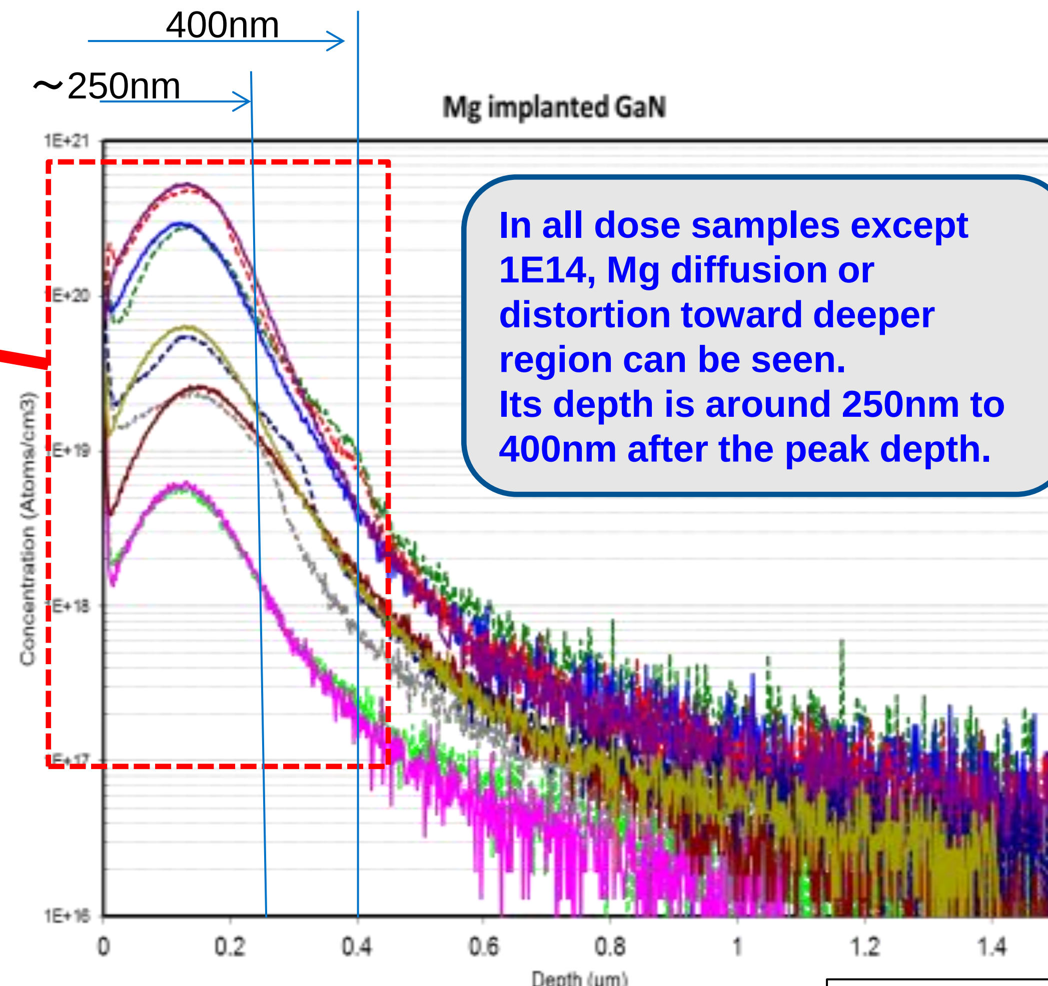




- **The amount of Mg dose was reduced around 2~9% after annealing.**

Pre-test : Mg depth profile by SIMS





In all dose samples except 1E14, Mg diffusion or distortion toward deeper region can be seen. Its depth is around 250nm to 400nm after the peak depth.

Mg migration toward surface after annealing is seen. And 5E14 annealed showed a strange profile.

- **As-implanted samples show Gaussian like profile.**
- **Annealed show distortion in Mg profile**

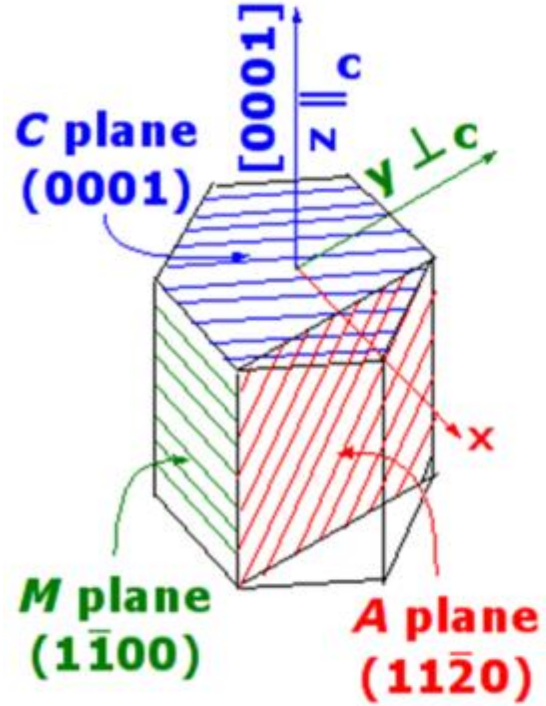
What is the cause？

Check crystallinity by RBS

Experiment 1:RBS analysis (Rutherford Backscattering Spectrometry）

Instrument：NEC accelerator &End stationRBS-400
Incident ion：He^{2+}
Incident energy：2.275MeV
Incident plane：c plane（0001）
Detector angle；160° ,100°
Measurement：random & align (channeling)

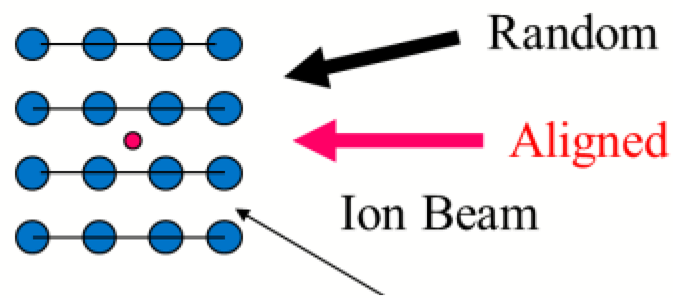
＜GaN crystal＞



About cChanneling method

Basic Concept of Channeling

. When an ion beam is aligned along a major crystal axis or plane, ion-atoms interaction probability is significantly reduced and this results in the large reduction of scattering events and ions penetrate deeper into the crystal.

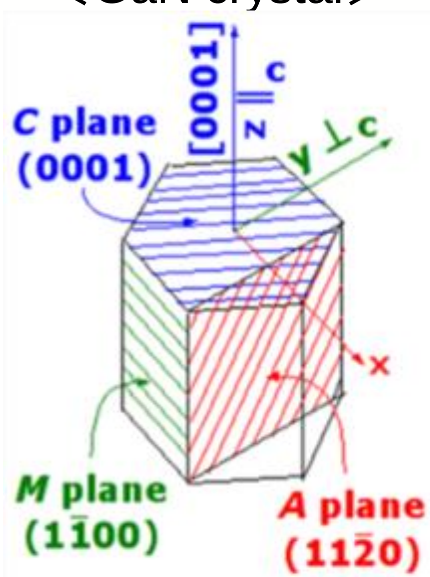


Channel: column of crystal lattice

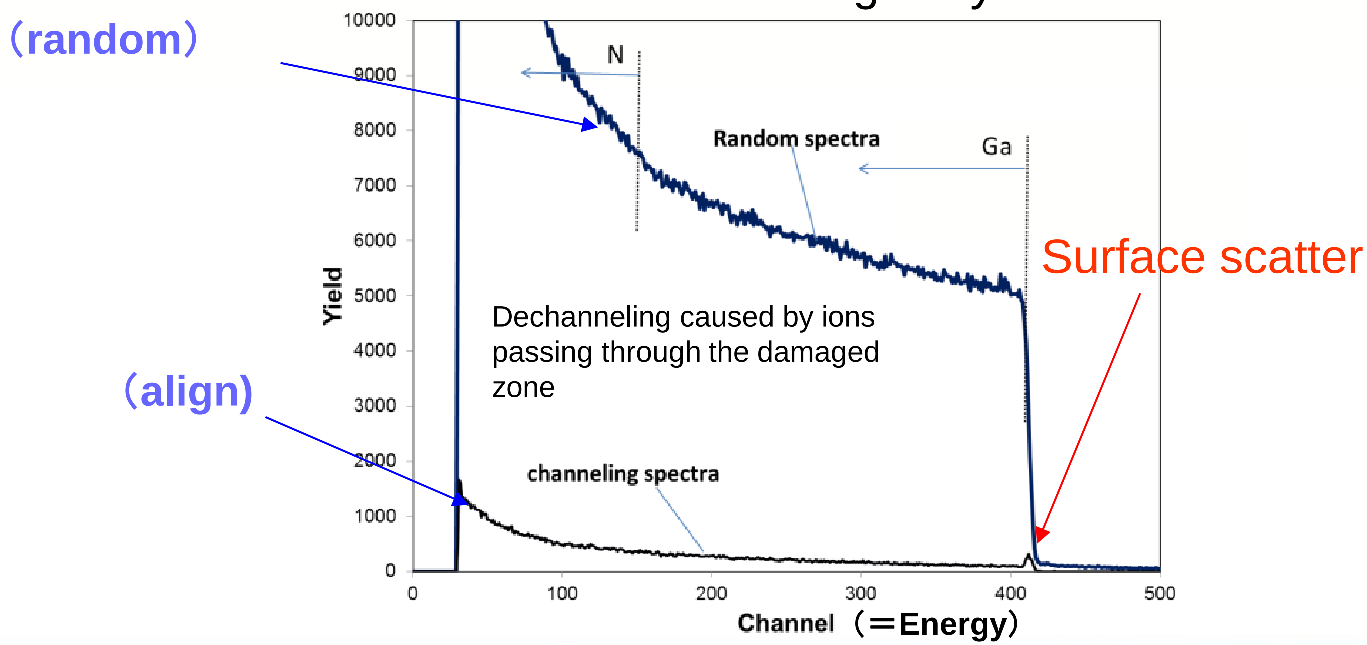
●Evaluation of crystallinity

used to study the crystal damage, defect concentration after implantation（displacement of lattice atom）

＜GaN crystal＞

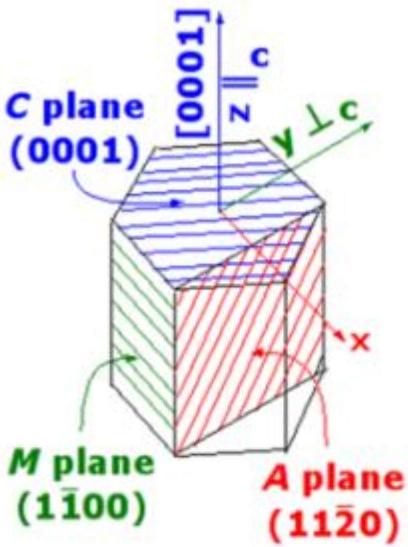


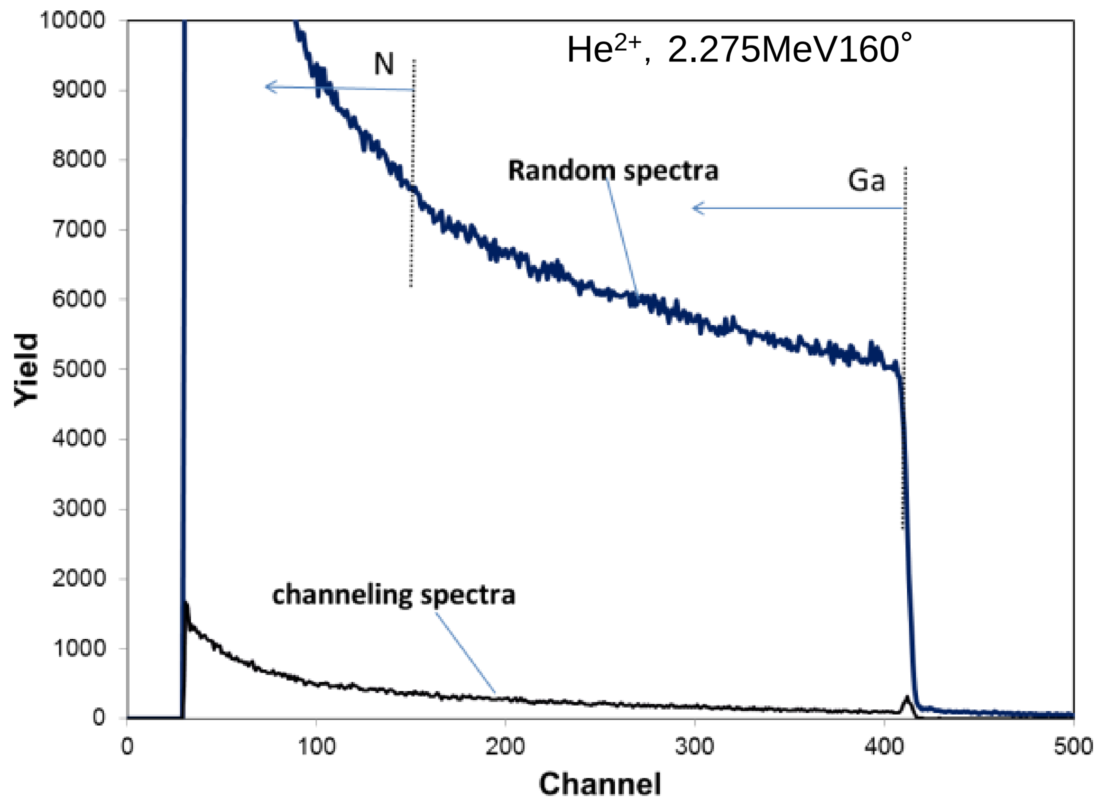
Data of GaN single crystal



Result :
GaN single crystal（Reference)

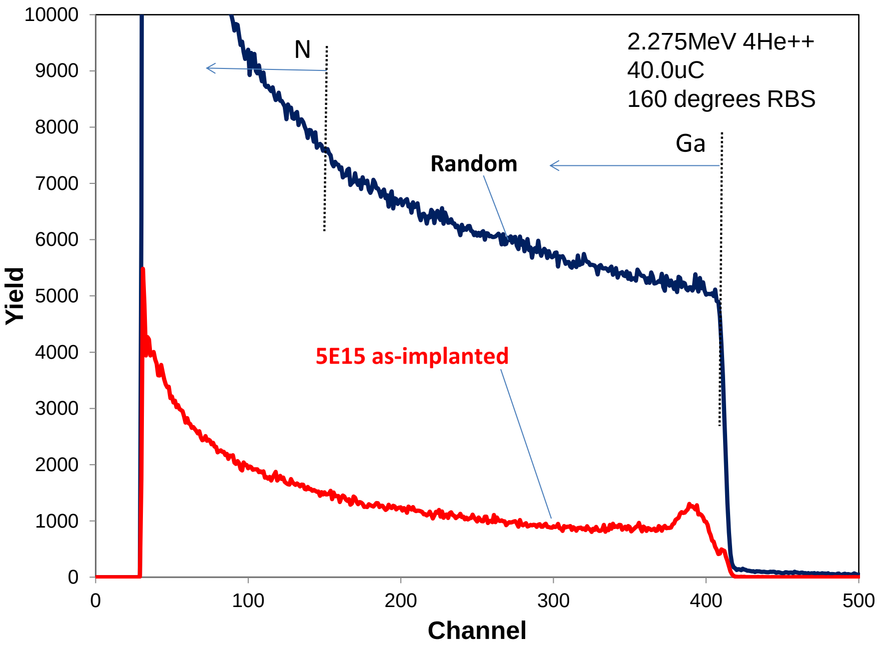
Ga atom displacement from crystal lattice on c-axis is measured.

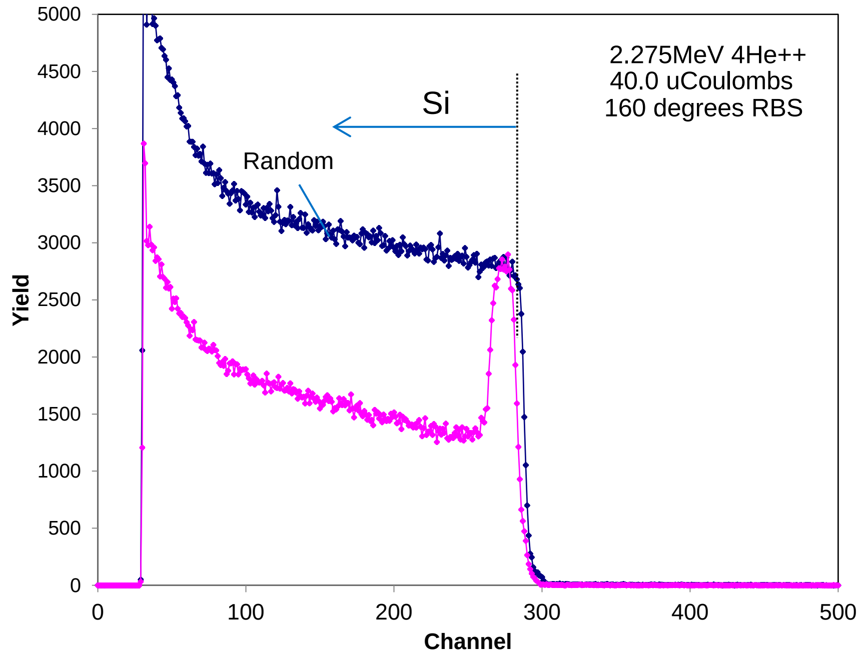




Result:

5E15 Mg implanted into GaN and Si for comparison



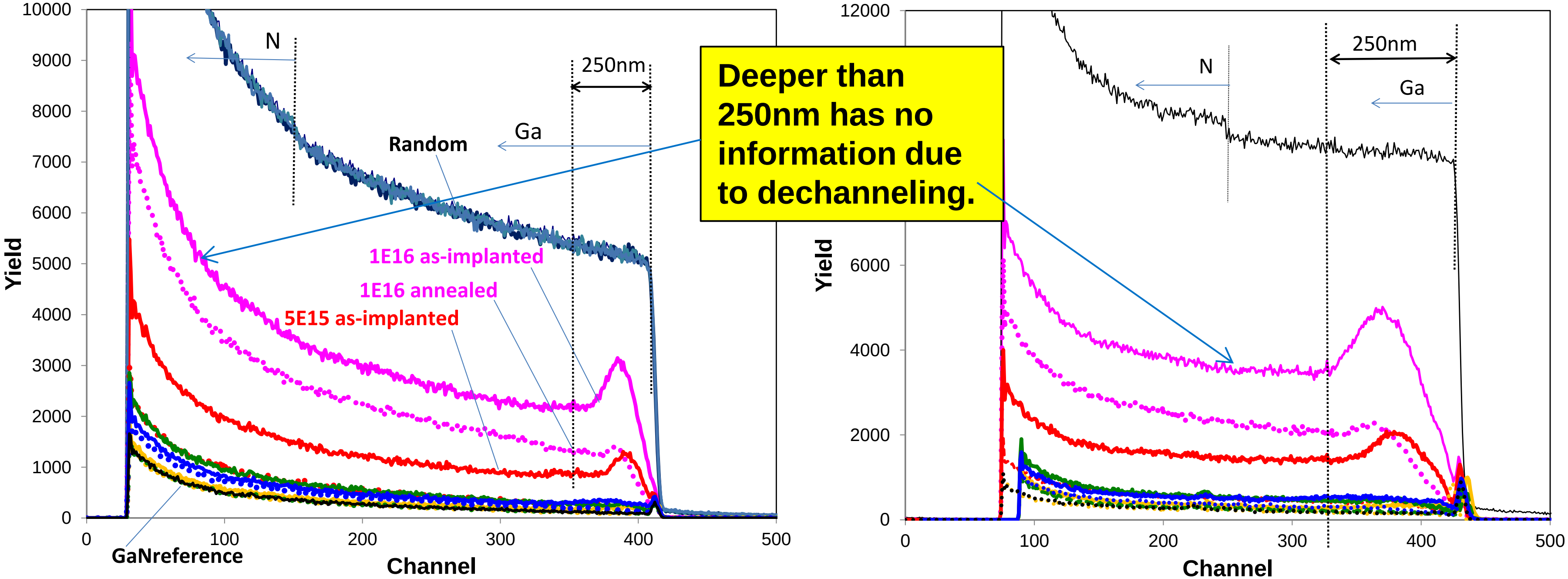


Damage in GaN crystal ; ~8%

Damage in Si crystal ; 100%

Result:
Comparison of channeling spectra

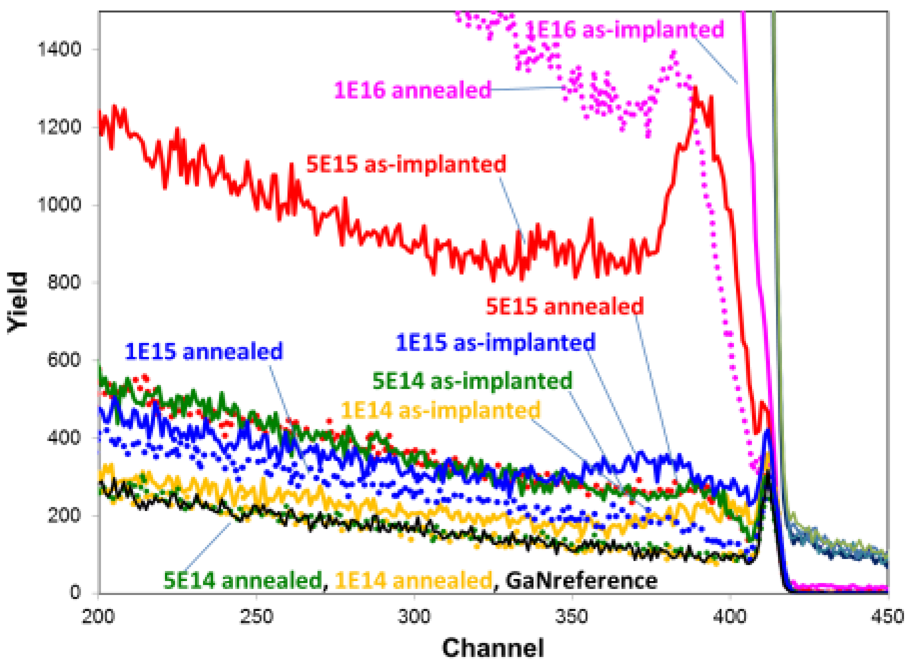
160° detector angle　　100° detector angle



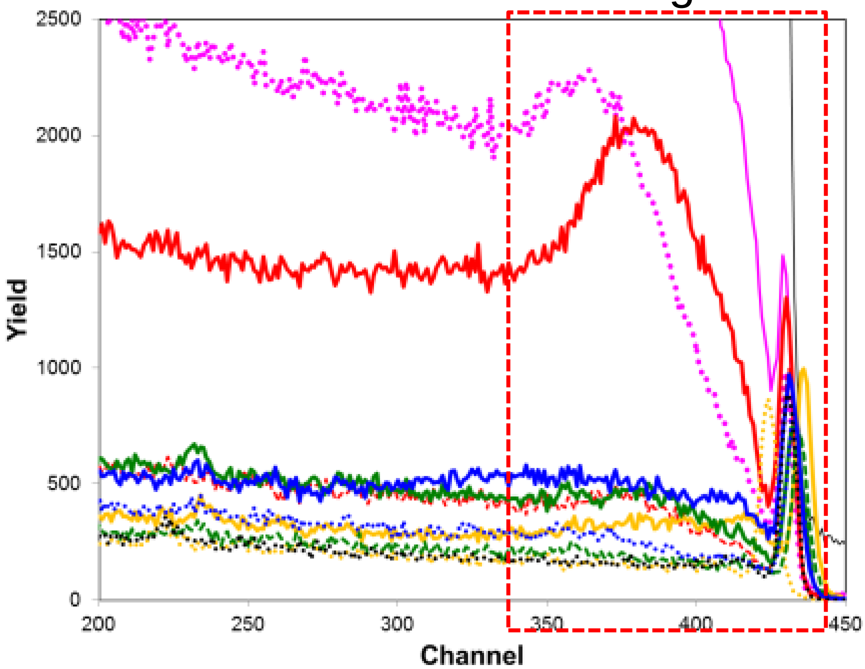
- **100° spectra shows crystallinity of surface in detail.**
- **Reduction of intensity is detected in annealed samples of all dose samples. It indicates recovery of damage by annealing.**
- **RBS can measure crystal information until only 250nm from surface.**

Result:
Comparison of channeling spectra (expanded)

160˚ detector angle



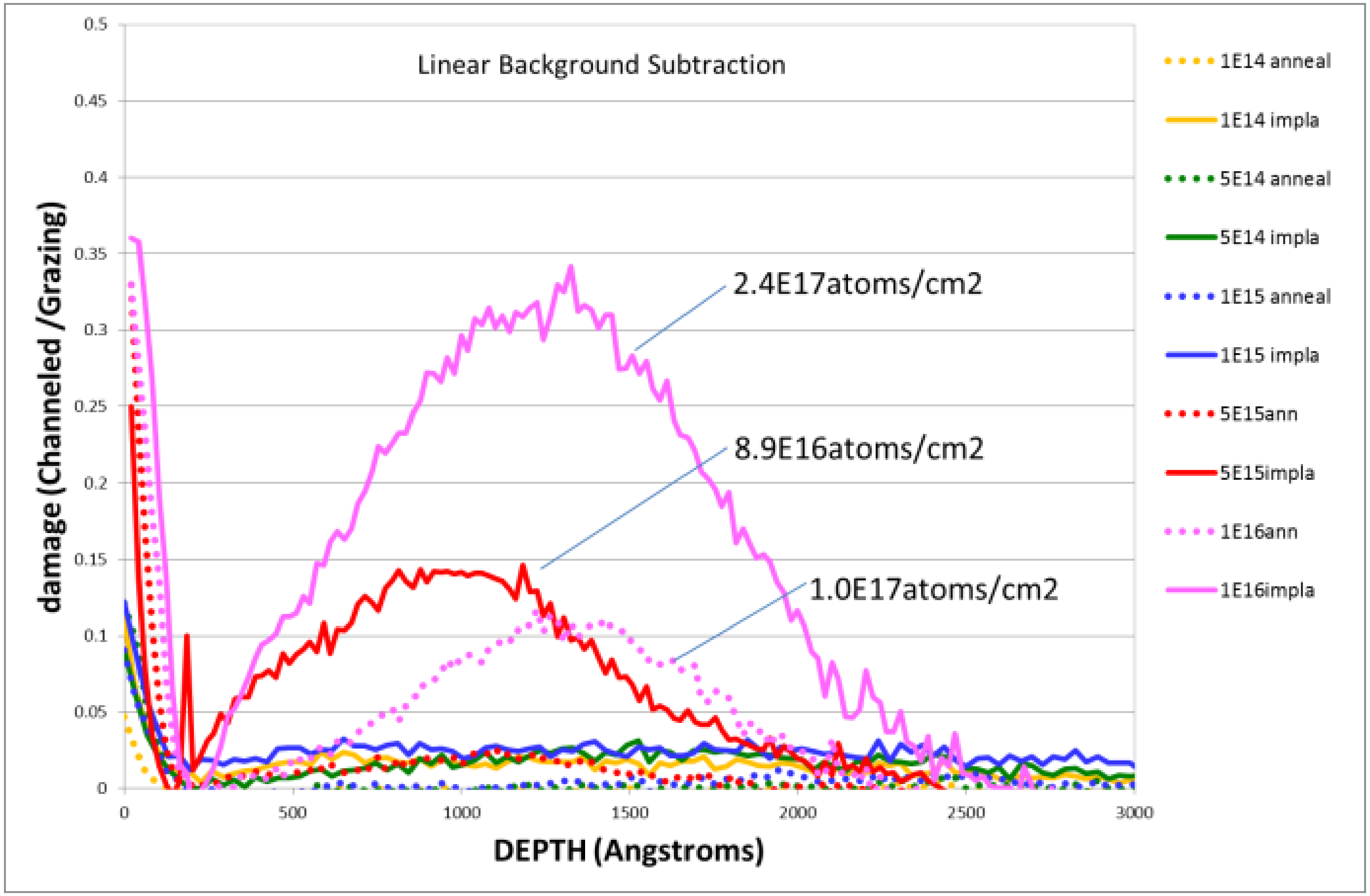
100˚ detector angle



- **Reduction of intensity is detected in annealed samples of all dose samples. It concludes recovery of damage by annealing.**
- **1E14 & 5E14 annealed samples detect no significant displacement. (same as reference data)**

Result:
Damage depth profile

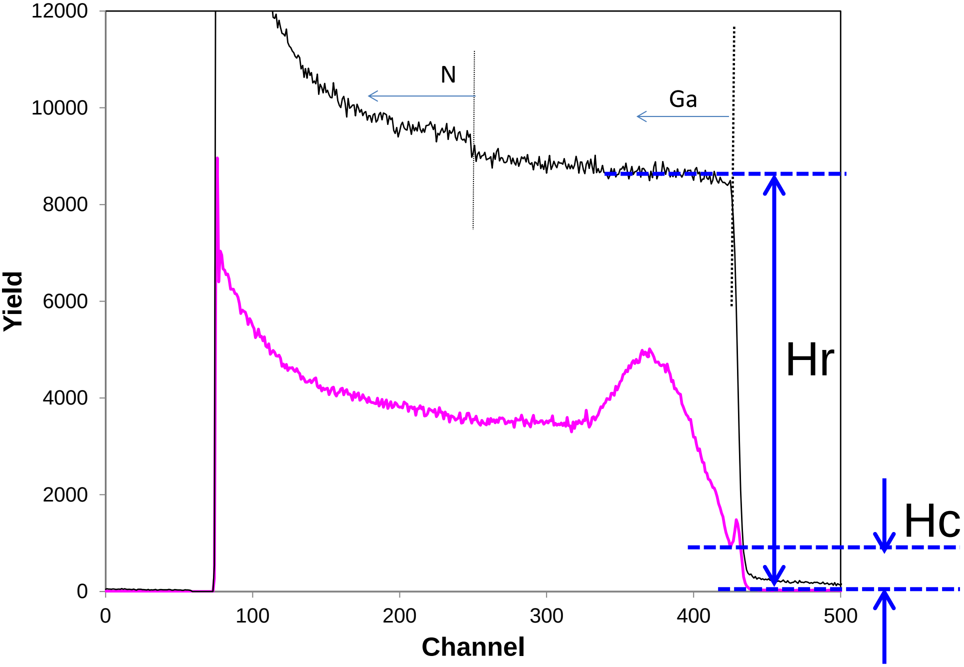
＜damage depth profile is converted from 100° spectra＞

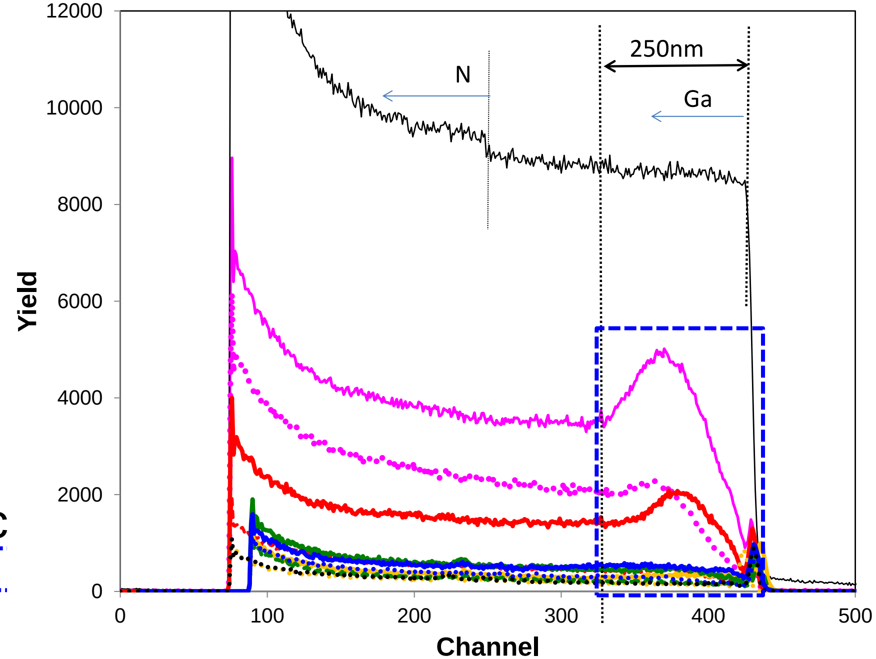


This damage profile is converted from 100° spectra, correspond to the red rectangle region shown in the previous slide.

Result :
Quantification of damage

Two methods of evaluation for damage
Xmin and Ga displacement





- Xmin=Hc／Hr

- **Hc：lowest intensity after surface peak**
- **Hr：random intensity at the energy of Hc**

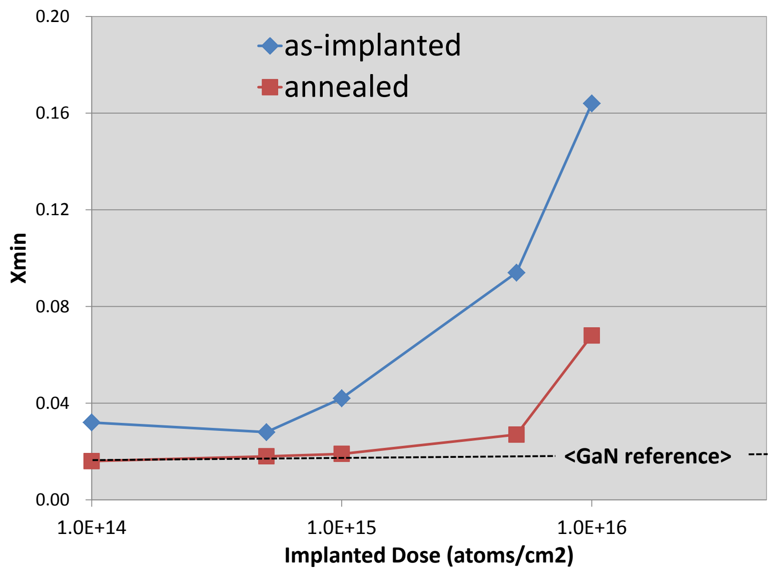
- **Ga displacement：[atoms/cm²]**
  Ga atom density by 250nm from surface
- **Ga displacement ratio:**
  Ga atom density in channeling／Ga atom density in random

Result；quantification of crystallinity (damage)

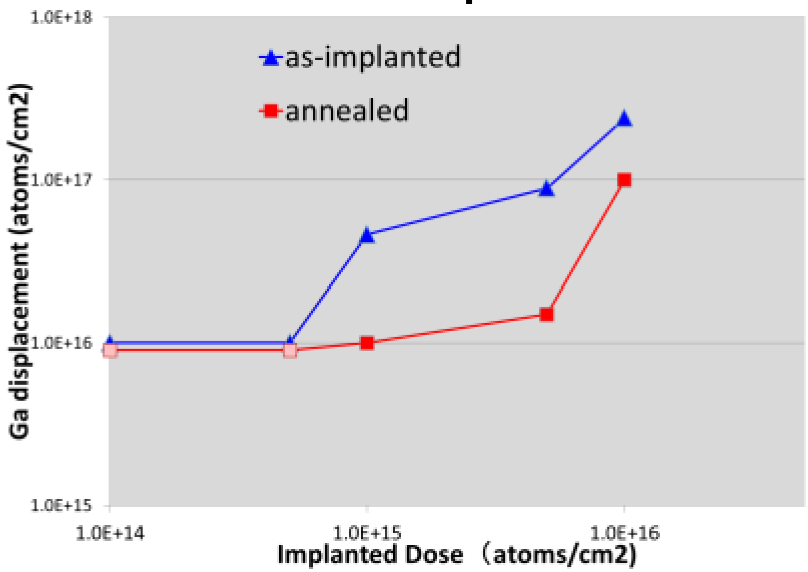
X_{min} and Ga displacement [atoms/cm2]

Sample	Xmin	Ga displacement	Ga displacement ratio(%)
1E14 as-implanted	0.03	1.0E+16	<=1%
1E14 annealed	0.02	<1E16	NA
5E14 as-implanted	0.03	1.0E+16	<=1%
5E14 annealed	0.02	<1E16	NA
1E15 as-implanted	0.04	4.6E+16	4%
1E15 annealed	0.02	1.0E+16	1%
5E15 as-implanted	0.09	8.9E+16	8%
5E15 annealed	0.03	1.5E+16	1%
1E16 as-implanted	0.16	2.4E+17	22%
1E16 annealed	0.07	1.0E+17	9%
GaN reference	0.02		

Relation between X_{min} and Dose



Relation between Ga displacement and Dose



Result; Summary of RBS analysis

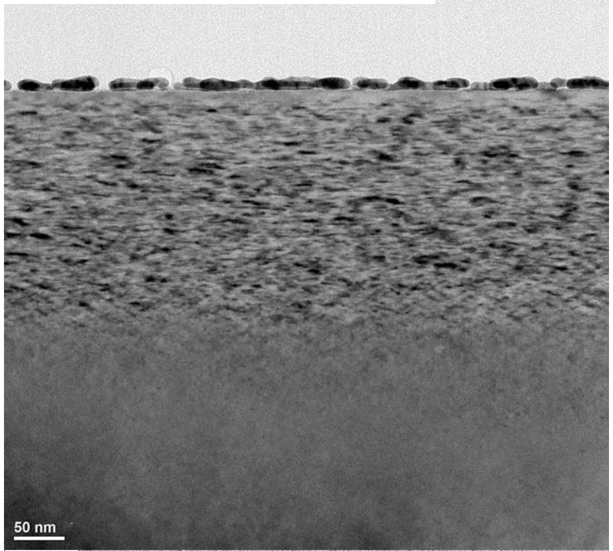
1. The amount of GaN crystal damage by Mg implantation was evaluated using the concentration of Ga displacement.
2. Ga displacement was detected in all as-implanted samples of 1E14～1E16.
3. The reduction of Ga displacement was seen in all annealed samples. It suggested recovery of crystal for annealed samples.
4. No detectable damage was seen in annealed samples for 1E14, 5E14.
5. Small amount of damage in deeper region than 250nm was not able to be evaluated by RBS due to de-channeling effect.

Experiment 2: TEM observation

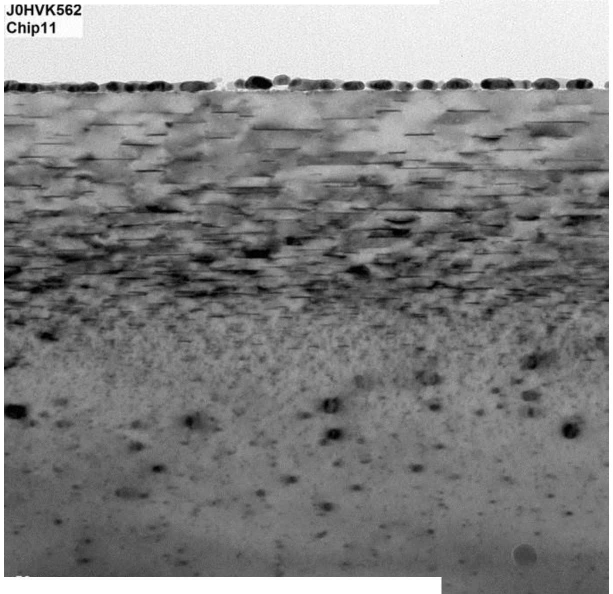
Sample preparation：cross sectional foil by FIB
Observation plane：（11-20）
TEM voltage：200kV
Instrument：Osiris （FEI）

Result :high dose implanted samples(S1～S4)

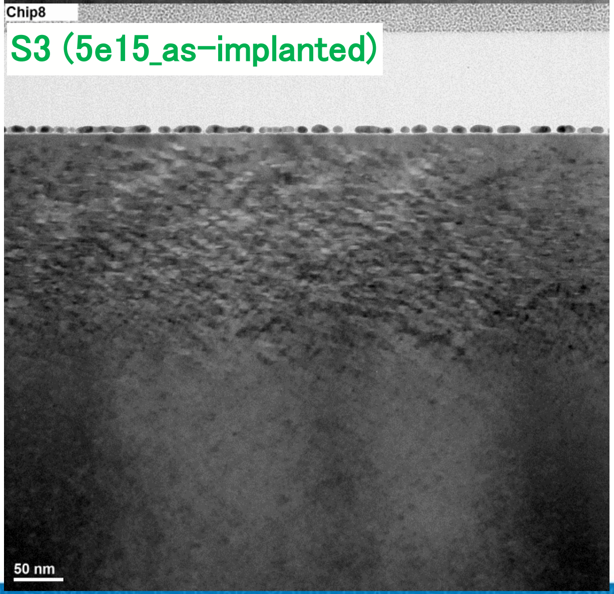
S1 (1e16_as-implanted)



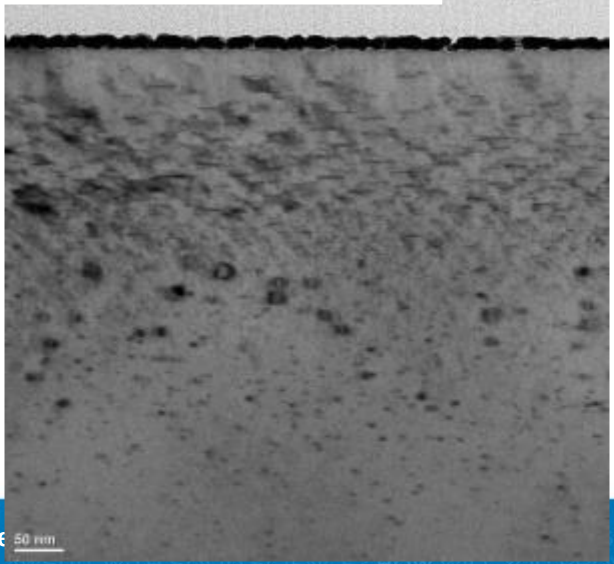
S2 (1e16_annealed)



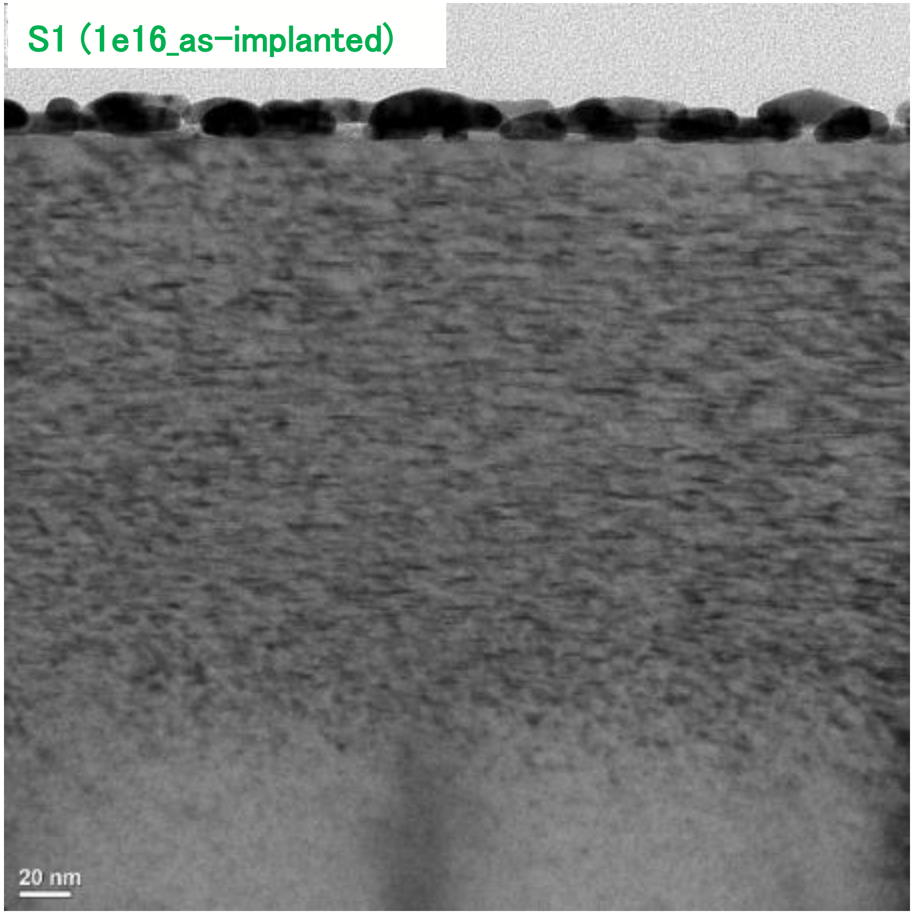
S3 (5e15_as-implanted)

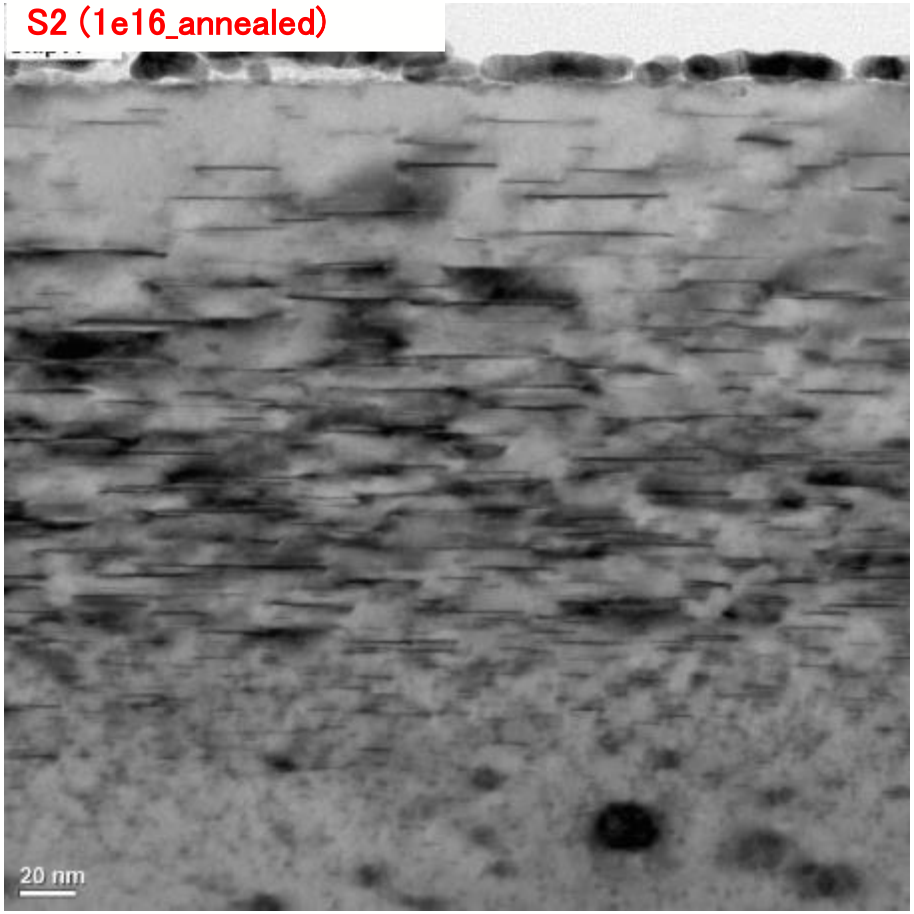


S4 (5e15_annealed)



Result; Expanded data

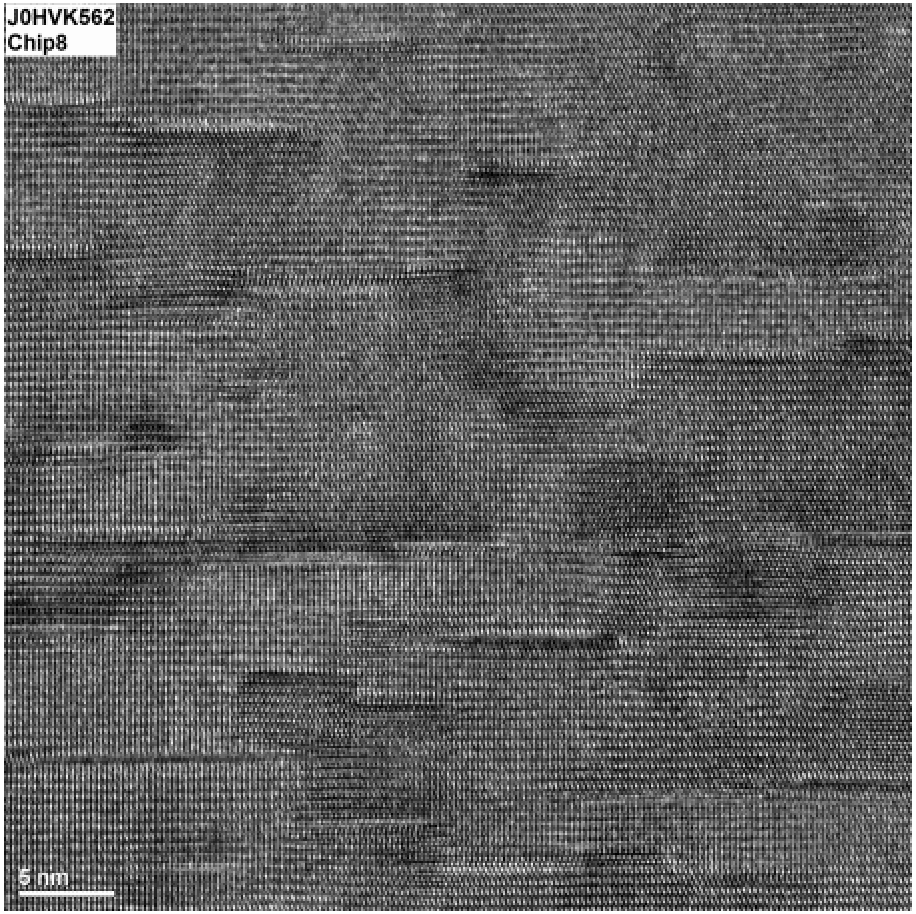


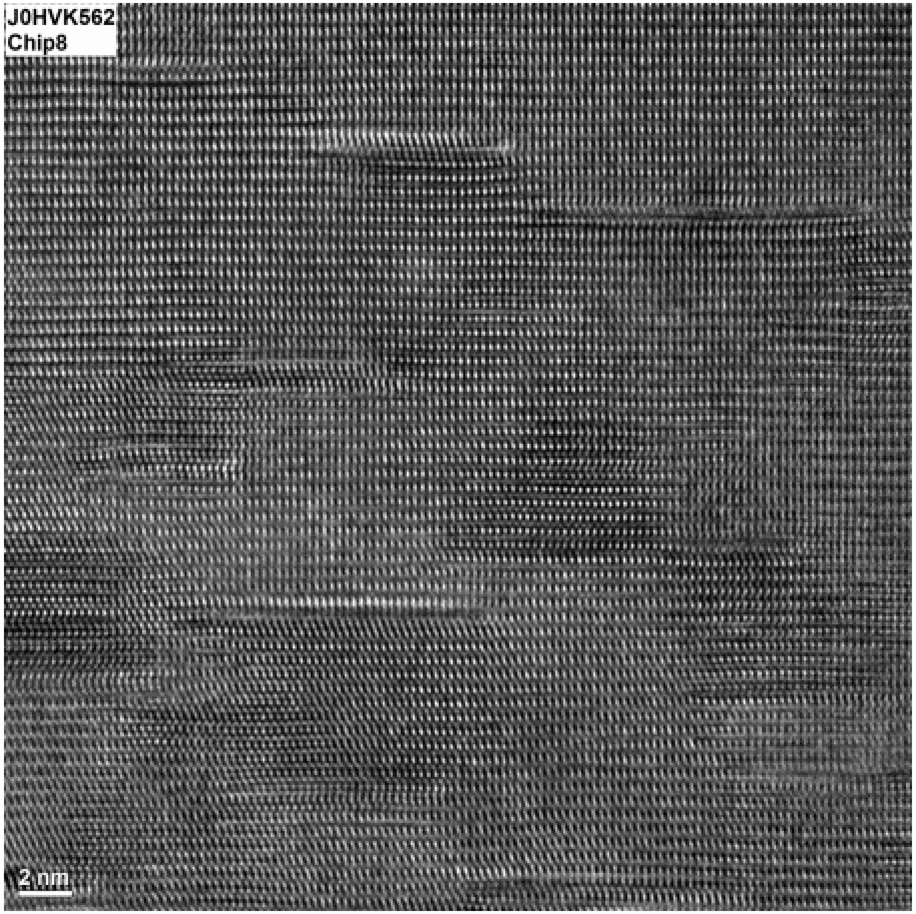


In as-implanted samples, many of small defect can be observed. In annealed samples, some crystalline recovery is seen, but longer defects can be observed clearly. At deeper region, dot defects clearly exist in annealed samples.

Result: High Resplution-TEM

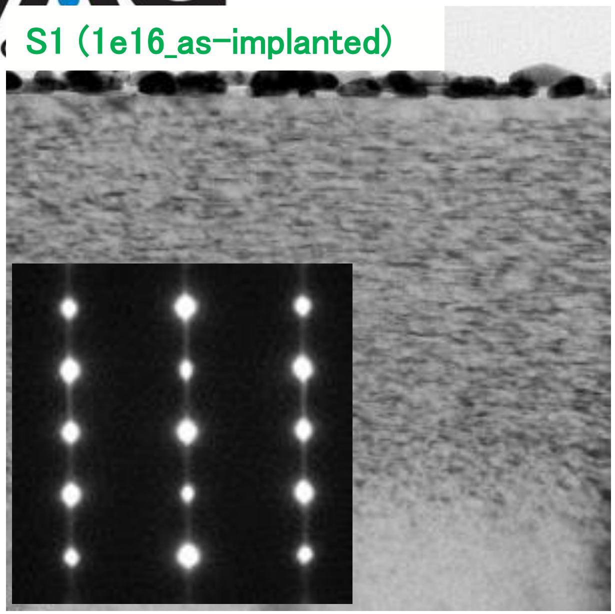
S3 (5e15_as-implanted); high resolution TEM observation

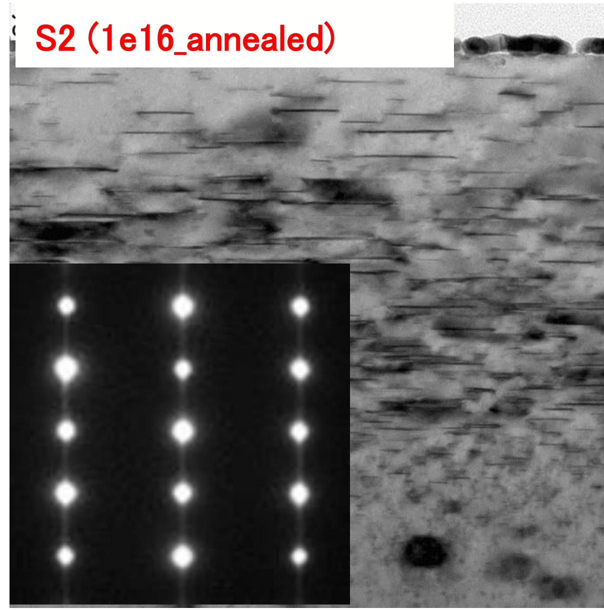




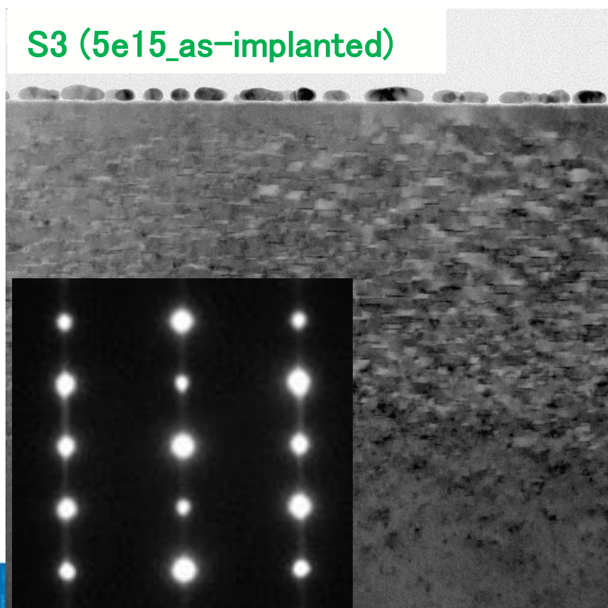
Result: Electron diffraction

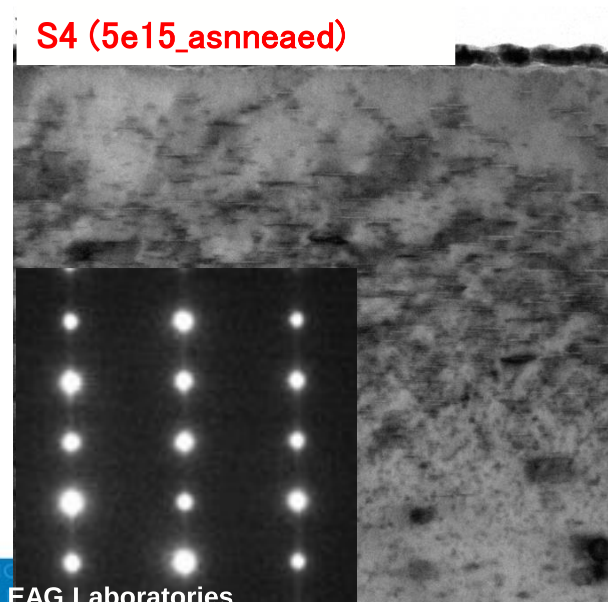
Higher dose (S1～S4)

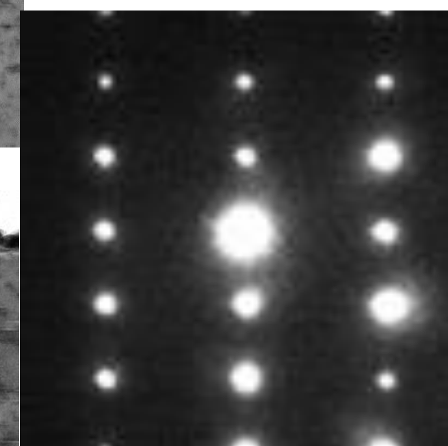




In the implanted region, streak is observed.



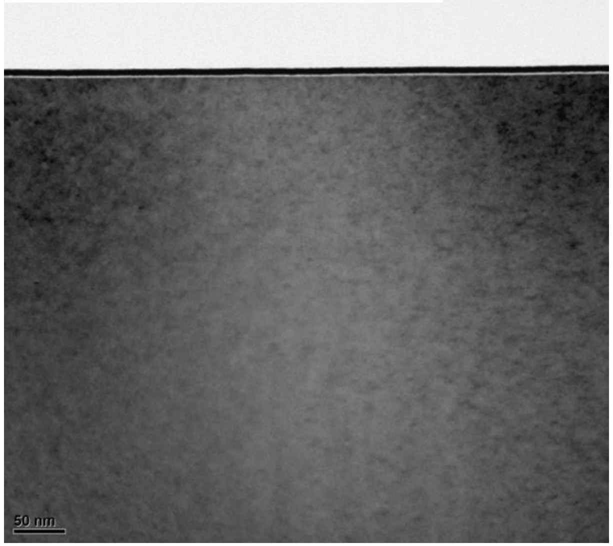




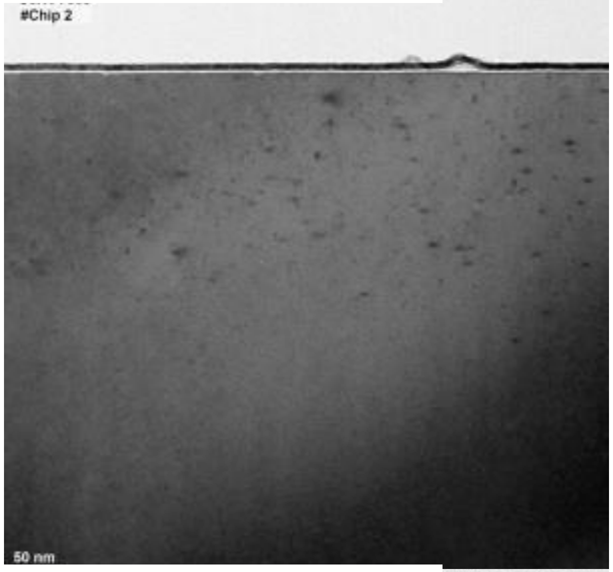
Non-implanted region don’t have streaks in electron diffraction pattern.

Result: Low dose implanted samples（S7～S10）

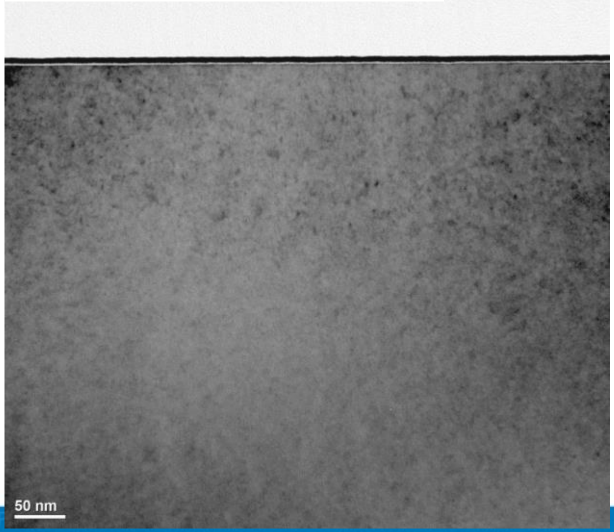
S9(1e14_as-implanted)



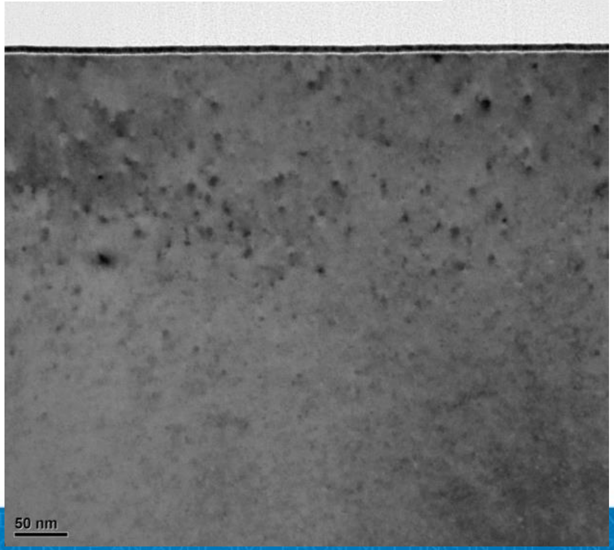
S10 (1e14_annealed)



S7 (5e14_as-implanted)

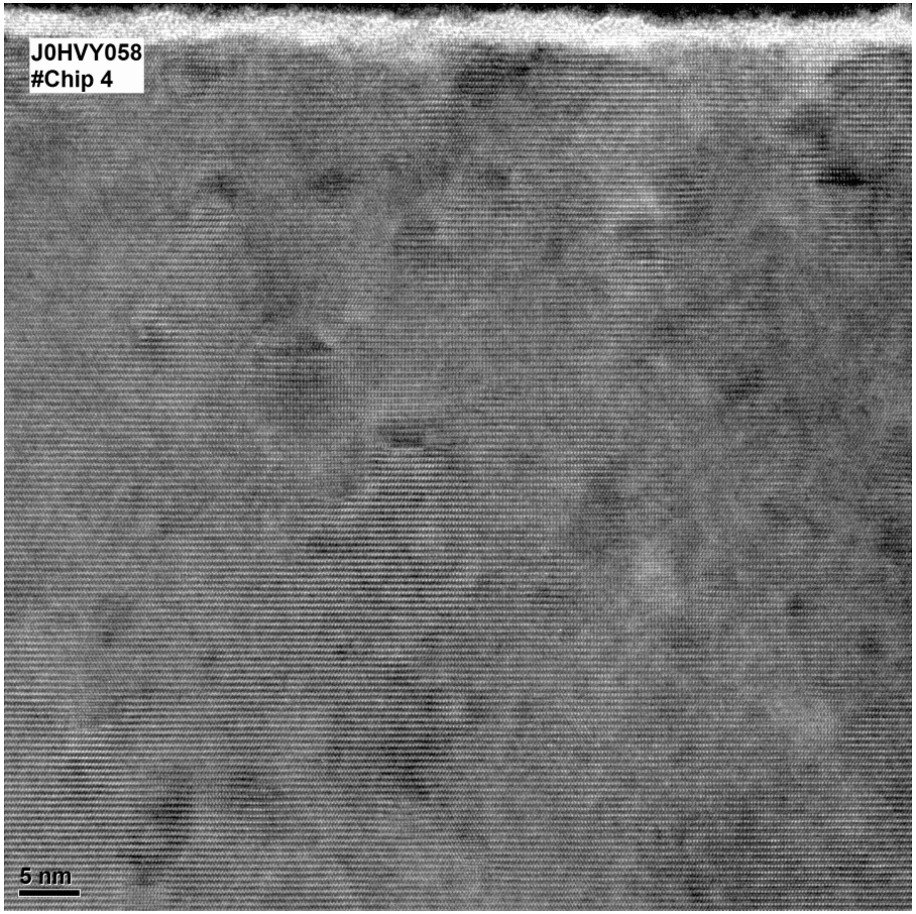


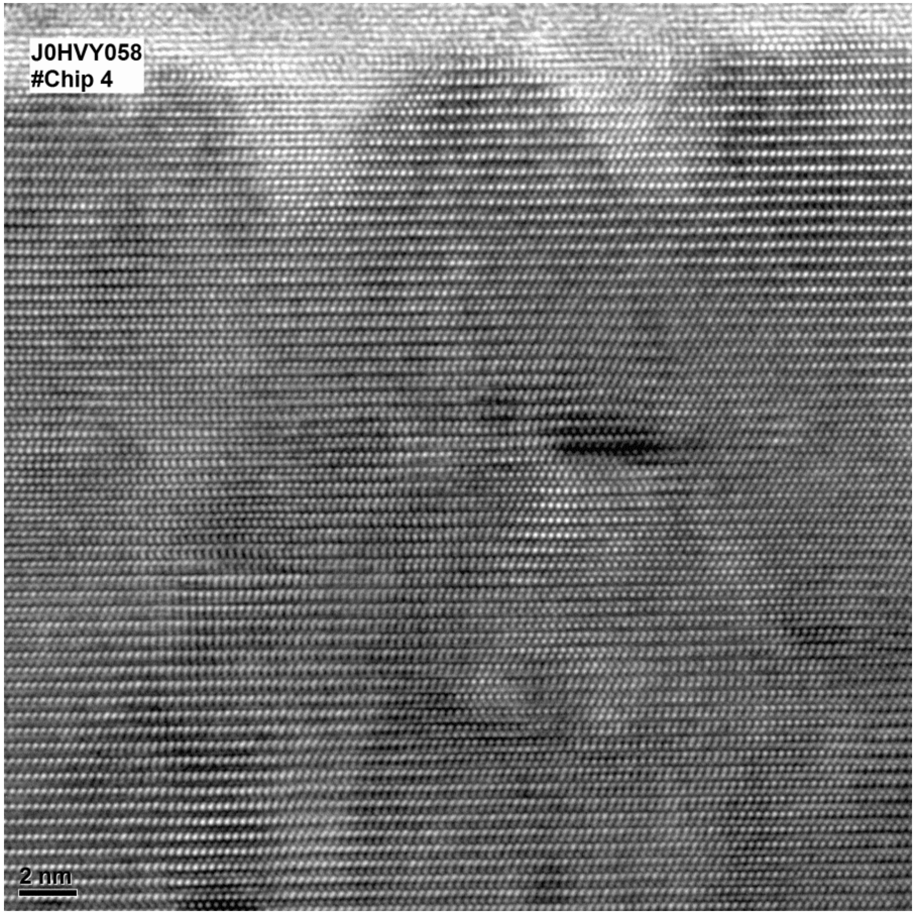
S8 (5e14_annealed)



Result: High Resolution TEM(low dose implanted sample)

S8 (5e14_annealed); high resolution observation





Result: Summary of TEM

	Dose (atoms/cm2)	Annealed	Detecting of defect by TEM
S1	1e16		○
S2	1e16	○	○
S3	5e15		○
S4	5e15	○	○
S5	1e15		○
S6	1e15	○	○
S7	5e14		△
S8	5e14	○	○
S9	1e14		×
S10	1e14	○	○

Summary of TEM observation

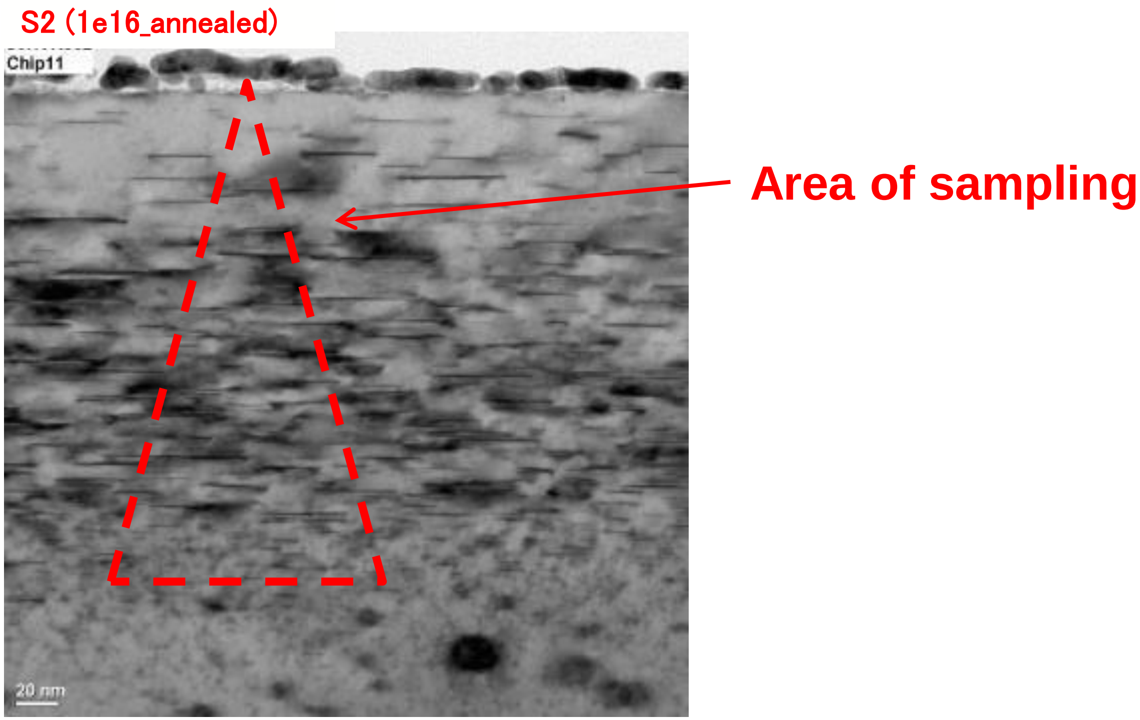
1. TEM detects damage in annealed samples even at 1e14atoms/cm2 Dose sample.
2. Overall crystallinity appears to be improved (recovered) by annealing, but also defects are seen to grow (clearly exist).
3. In the result of higher dose implantation samples, line-like defects (as plane defects) are observed, the annealing makes defects grow (longer).

However only the TEM observation and RBS result were still difficult to explain the cause of crater bottom roughness by SIMS.
We assume Mg may be included in the defect in the considering with line defect is grown after annealing.
Therefore we proceed to the 3rd experiment to confirm it.

Experiment 3

The investigation of Mg distribution by 3D-Atom Probe

Sample：S2（Mg1e16 implanted + annealed）
Instrument：LEAP3000XSi



Result: 3D-AP analysis

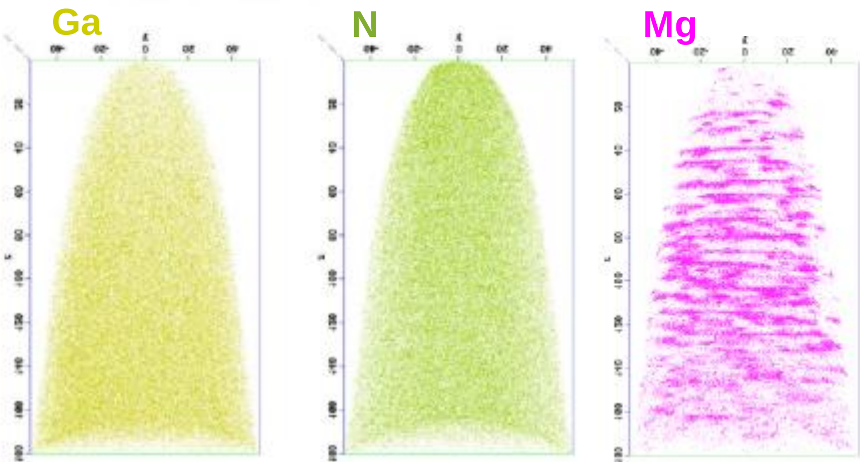
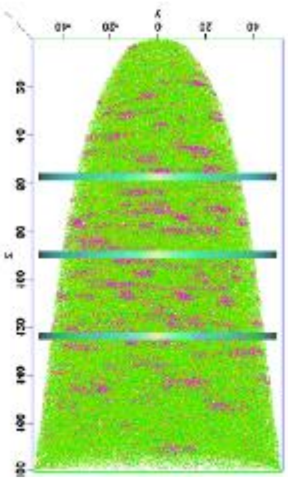


Fig 1（total mapping of each element）



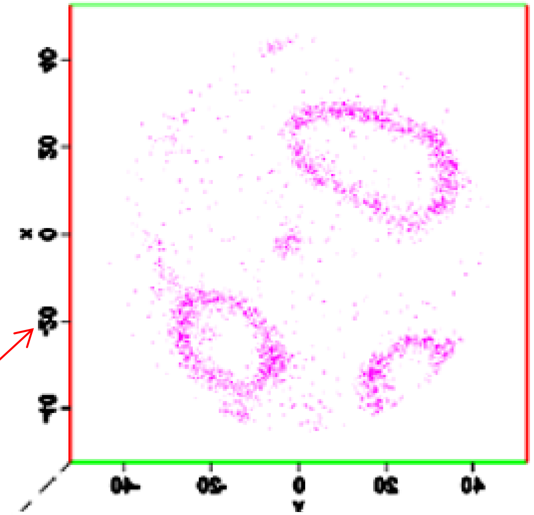


Fig2-1

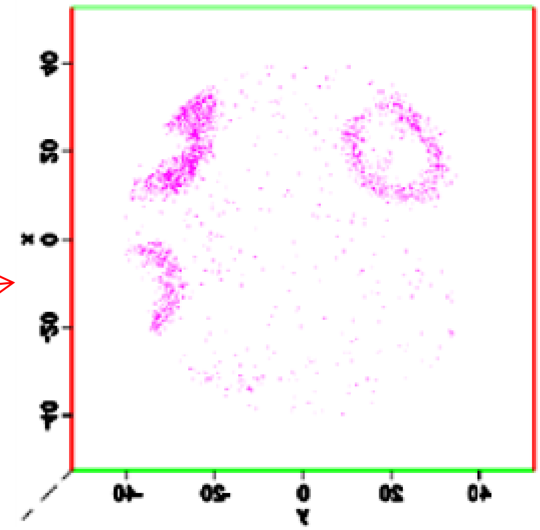
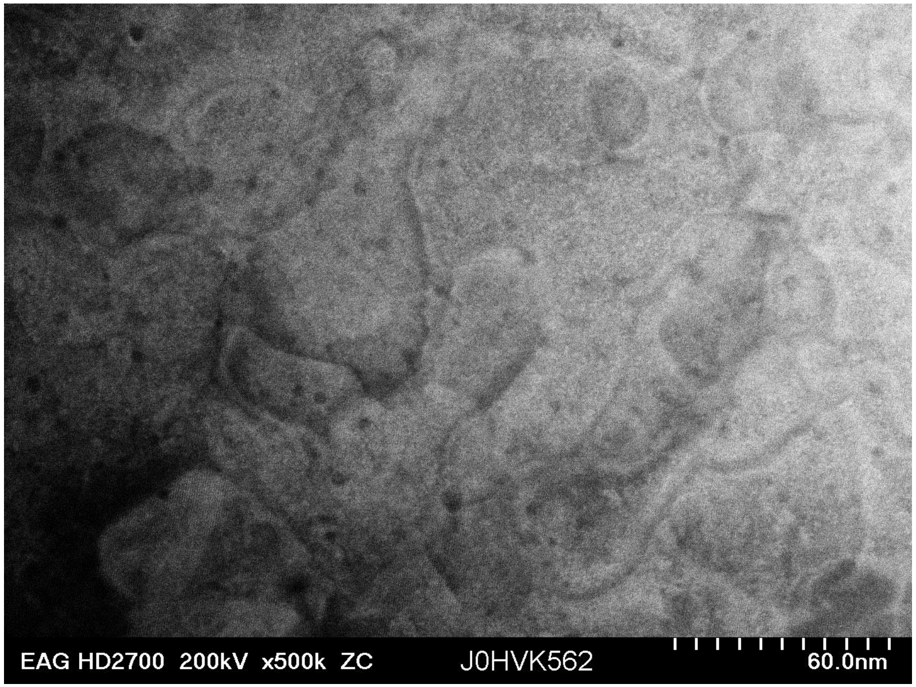


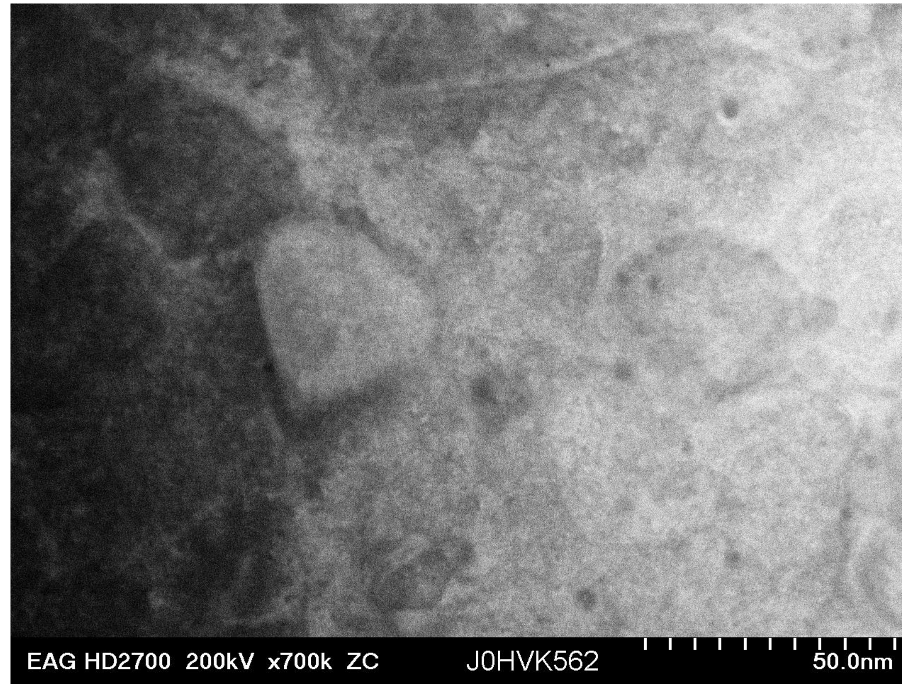
Fig2-2

Fig2-1, 2-2：lateral distribution in a slice 3nm thick

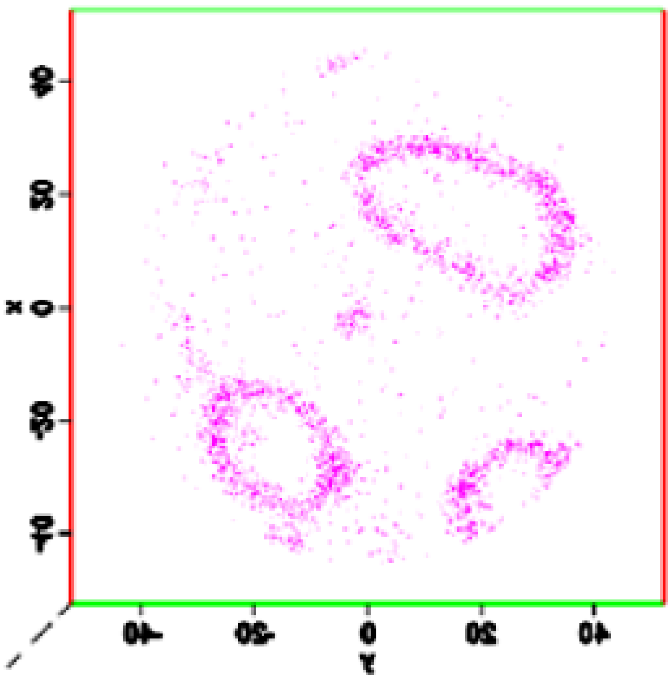
Ring-like accumulation of Mg is observed.

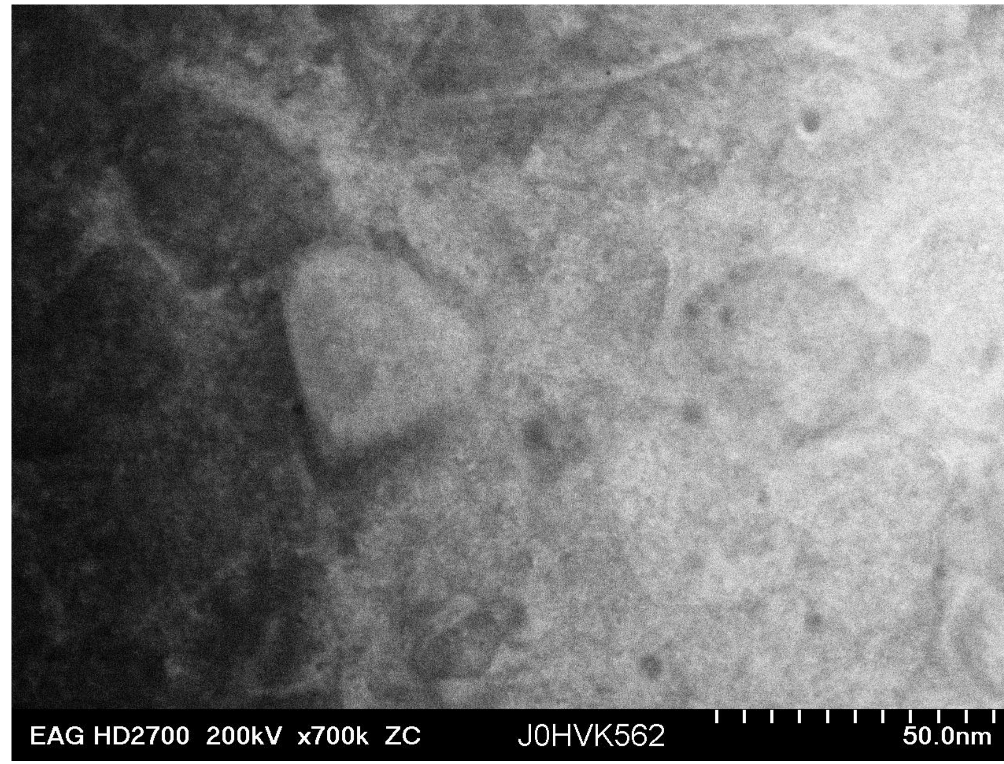
Result: TEM, plan view Observation (measured by AC-STEM (HD2700))





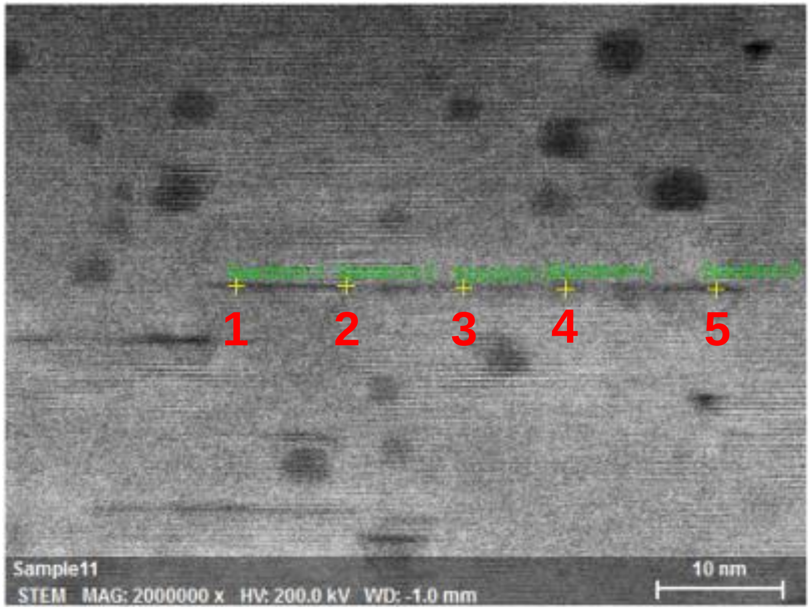
Comparison
Plane- View STEM and 3DAP



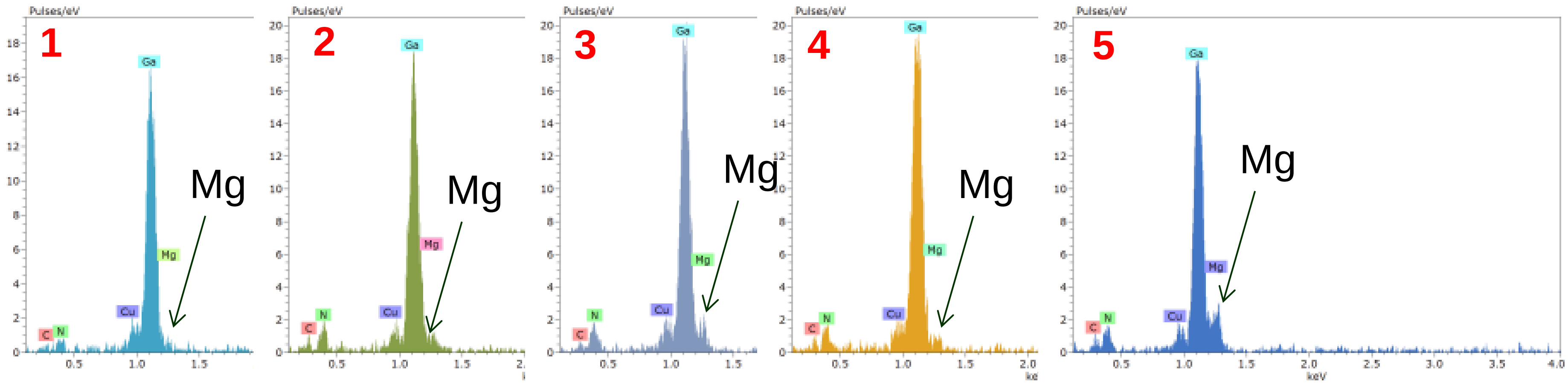


The size of ring defect by PV-TEM agree well to the size of Mg distribution by 3DAP. We conclude Mg accumulates at the fringe of ring-like-plan-defect.

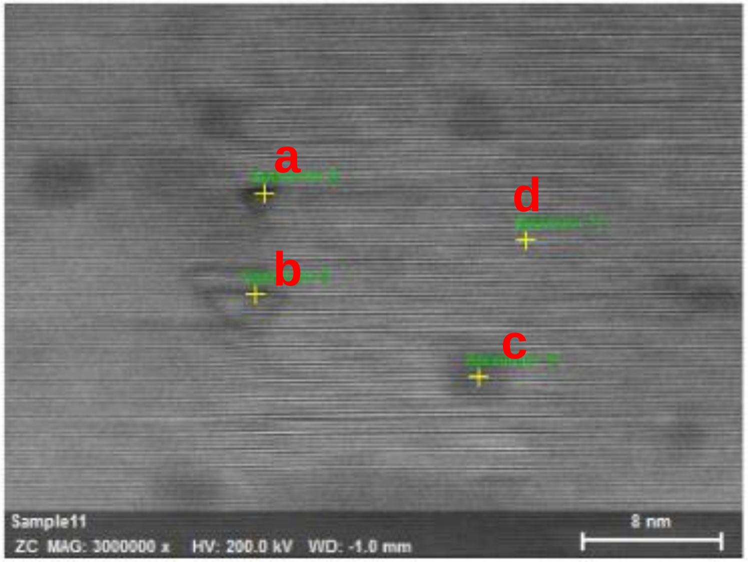
More result by STEM-EDS (by AC-STEM)



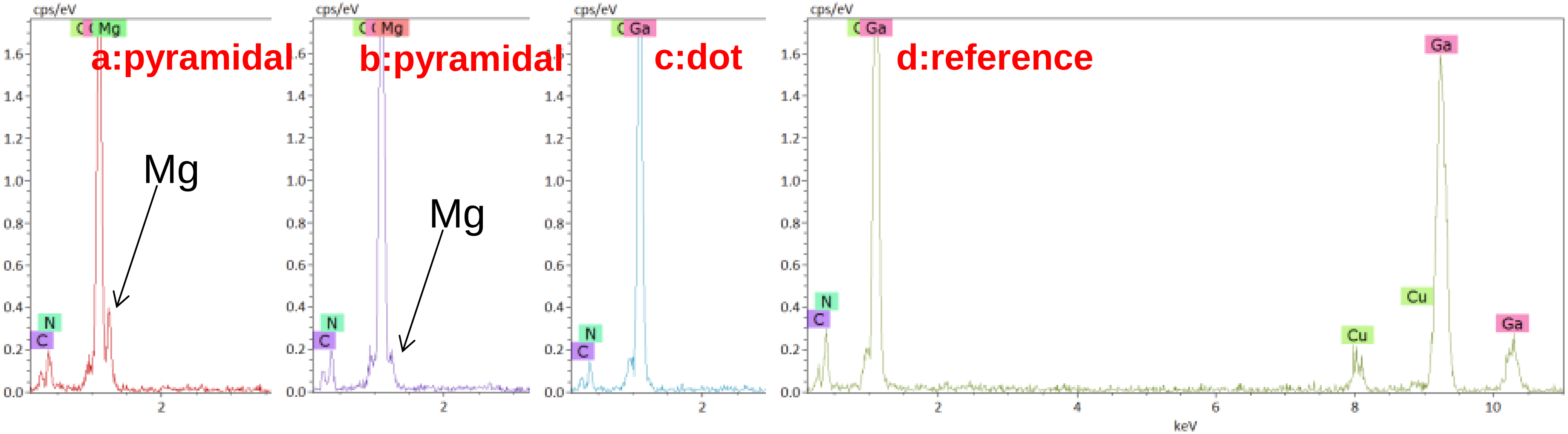
TEM-EDS analysis is done at a line defect. Mg is detected clearly along with the line defect.



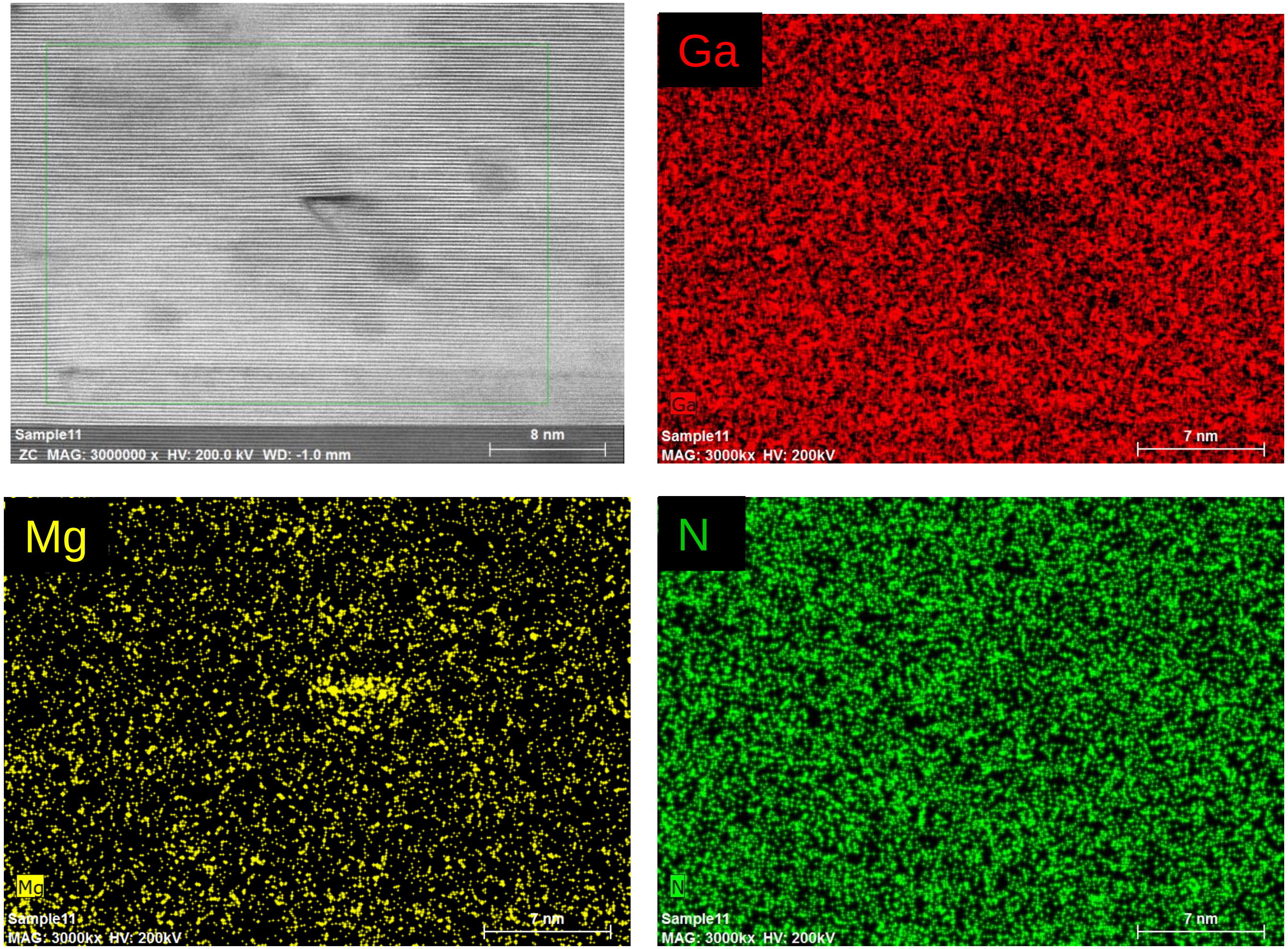
More result by STEM-EDS (by AC-STEM)



In 1E16 annealed sample, pyramidal defect and dot defect as well as line defect are observed.
Both defects of pyramidal and dot are much smaller than line defect.
Mg was detected in pyramidal and dot defect as well.
But no Mg is detected in non-damage region.



More result:
TEM-EDS mapping (by AC-STEM)



Summary

1. RBS can detect change in crystallinity before and after annealing.
2. TEM can detect Mg implanted damage in GaN after annealing, even at low dose of 1e14atoms/cm2 sample.
3. However defects observed by TEM appear to not completely agree to crystalline recovery by RBS. Here we have to consider the direction of measurement by RBS and by TEM. (RBS sees c-axis, but TEM sees a-plane.) Therefore RBS result is not entirely discrepant to TEM result.
4. It is confirmed that while annealing leads to crystalline recovery, it also make some defects grow.
5. In 1e16 annealed sample, Mg accumulation at fringe of defect is confirmed by 3DAP and TEM-EDS. This is assumed to be a cause of SIMS crater bottom roughening.
6. In this study, we confirmed Mg accumulation in the higher dose sample. We can assume Mg accumulation would occur in lower dose implanted sample in consideration with the fact that the existence of defect after annealing was seen.

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