



**AFRL-RQ-WP-CP-2023-0004**

## **INTENTIONAL IRON DIFFUSION IN BETA-Ga<sub>2</sub>O<sub>3</sub>**

**Kevin Bray  
UES Inc.**

**Jacob Lawson  
University of Dayton Research Institute**

**Joseph Neil Merrett  
Air Force Research Lab**

**FEBRUARY 2019  
Interim Report**

**DISTRIBUTION STATEMENT A. Approved for public release. Distribution is unlimited;  
AFRL/PA-2019-3792; Cleared 07 August 2019.**

**AIR FORCE RESEARCH LABORATORY  
AEROSPACE SYSTEMS DIRECTORATE  
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7542  
AIR FORCE MATERIEL COMMAND  
UNITED STATES AIR FORCE**

## NOTICE PAGE

Using Government drawings, specifications, or other data included in this document for any purpose other than Government procurement does not in any way obligate the U.S. Government. The fact that the Government formulated or supplied the drawings, specifications, or other data does not license the holder or any other person or corporation; or convey any rights or permission to manufacture, use, or sell any patented invention that may relate to them.

This paper was cleared for public release by AFRL Public Affairs, AFRL/PA and is available to the general public, including foreign nationals.

Copies may be obtained from the Defense Technical Information Center (DTIC)  
(<https://discover.dtic.mil>).

AFRL-RQ-WP-CP-2023-0004 has been reviewed and is approved for publication in accordance with assigned distribution statement.

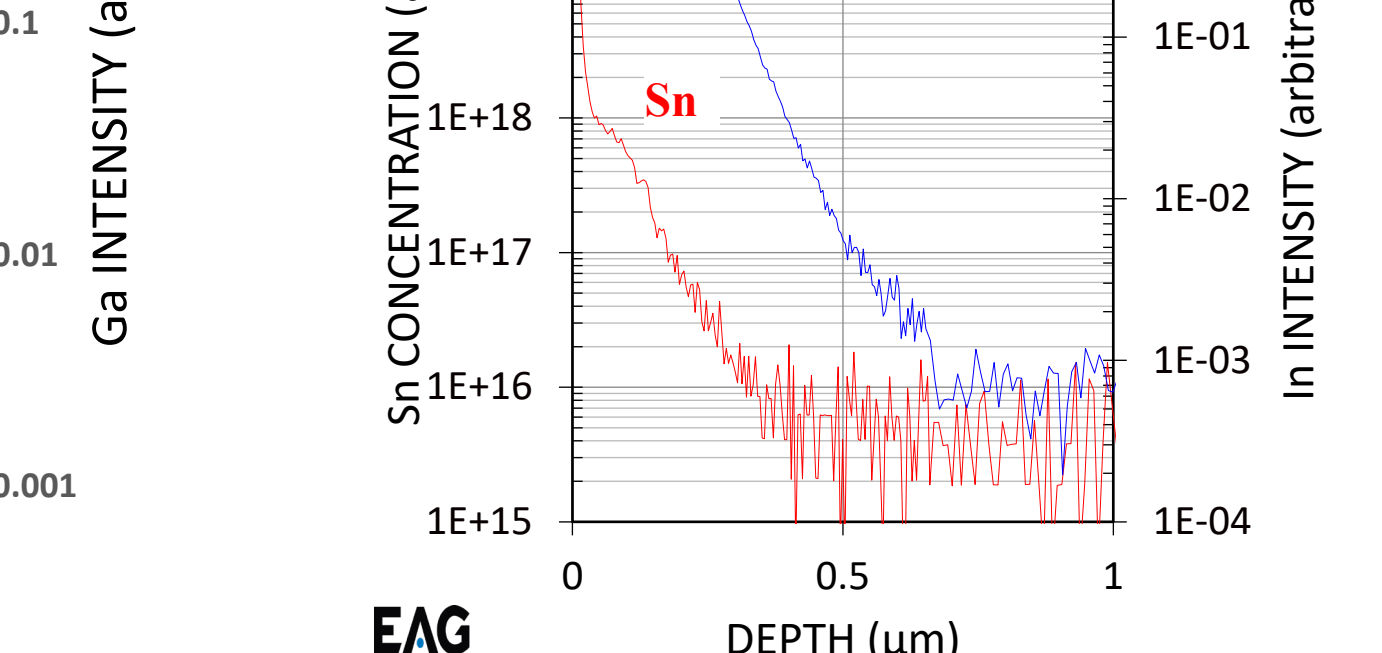
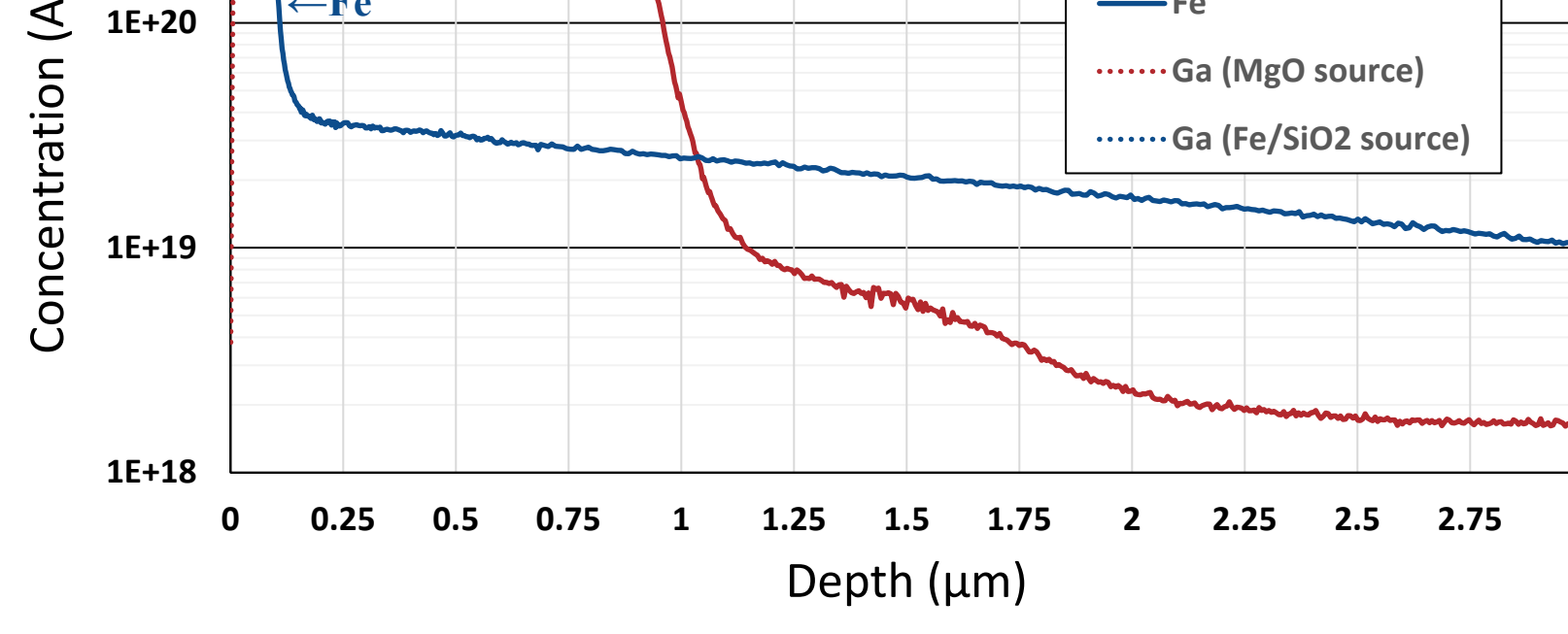
This paper is published in the interest of scientific and technical information exchange and its publication does not constitute the Government's approval or disapproval of its ideas or findings.

# REPORT DOCUMENTATION PAGE

PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ORGANIZATION.

<b>1. REPORT DATE</b> 05022019		<b>2. REPORT TYPE</b> Interim		<b>3. DATES COVERED</b>	
				<b>START DATE</b> 02052017	<b>END DATE</b> 08072019
<b>4. TITLE AND SUBTITLE</b> Intentional Iron Diffusion in Beta-Ga <sub>2</sub> O <sub>3</sub>					
<b>5a. CONTRACT NUMBER</b> In-house		<b>5b. GRANT NUMBER</b>		<b>5c. PROGRAM ELEMENT NUMBER</b> 62203F	
<b>5d. PROJECT NUMBER</b> 3145		<b>5e. TASK NUMBER</b>		<b>5f. WORK UNIT NUMBER</b> Q11S	
<b>6. AUTHOR(S)</b> Kevin Bray, Jacob Lawson, and J. Neil Merrett					
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> UES Inc                      University of Dayton Research Institute      AFRL/RQQE 4401 Dayton-Xenia Rd.      300 College Park                      Fifth Street Dayton, OH 45432-1894      Dayton, Ohio 45469                      WPAFB, OH 45433				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b>	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> Aerospace Systems Directorate Air Force Research Laboratory Air Force Materiel Command Wright-Patterson Air Force Base, OH 45433-7542			<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b> AFRL/RQQE	<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b> AFRL-RQ-WP-CP- 2023-0004	
<b>12. DISTRIBUTION/AVAILABILITY STATEMENT</b> DISTRIBUTION STATEMENT A. Approved for public release. Distribution is unlimited. Cleared 7 August 2019.					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> This report details efforts to selectively dope Beta-Ga <sub>2</sub> O <sub>3</sub> . Doping was achieved through diffusion source layers containing Mg, Fe, Sn, and indium. SIMS analysis is presented to show the depth of diffusion of the different species. Experiments showed that diffusion is a viable method to selectively dope gallium oxide.					
<b>15. SUBJECT TERMS</b> Beta gallium oxide, iron diffusion, selective area doping,					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>		<b>18. NUMBER OF PAGES</b>
<b>a. REPORT</b> Unclassified	<b>b. ABSTRACT</b> Unclassified	<b>c. THIS PAGE</b> Unclassified	SAR		4
<b>19a. NAME OF RESPONSIBLE PERSON</b> Joseph Neil Merrett				<b>19b. PHONE NUMBER (Include area code)</b> (937) 952-9181	

Sample	Substrate	Orientation	Process	Temp (°C)	Time	Notes
Sn/SiO <sub>2</sub>	10/500	Fe-doped (0,1,0)	RF sputter	1240	15*	~2 min BOE, ~1 min 1:3 HCl:H <sub>2</sub> O
Sn/ITO	10/1500	Fe-doped (0,1,0)	RF sputter	1240	15*	~5 min 1:3 HCl:H <sub>2</sub> O
MgO	2000	UID (0,1,0)	e-beam RF sputter, co-dep, 20% O <sub>2</sub>	1240	15*	~2 min H <sub>2</sub> SO <sub>4</sub>
SiO <sub>2</sub> :Fe	2000	UID (0,1,0)		1240	15*	~2 min BOE, ~1 min 1:3 HCl:H <sub>2</sub> O



1240 °C Conclusions: Sn possible with Sn/ITO or Sn/SiO<sub>2</sub>, but not very efficient. Fe and Mg showed promise with Fe. Source layer concentration appears to be too high – possible ternary formed with Mg and Fe.

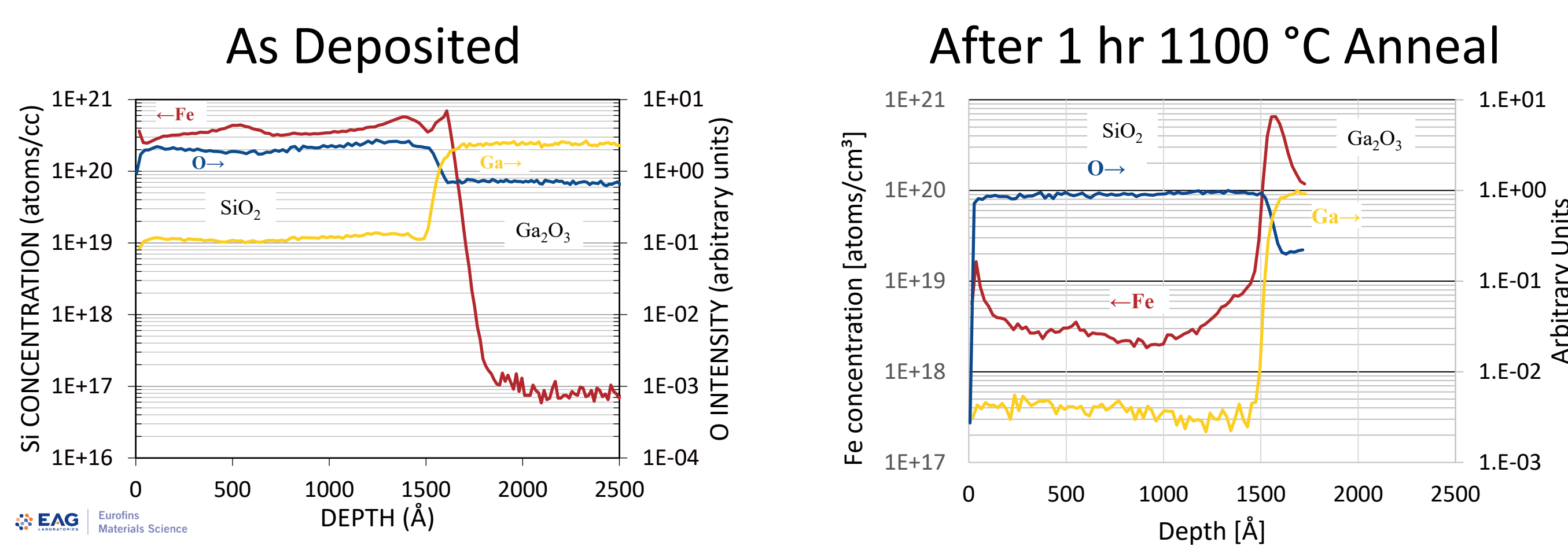
2<sup>nd</sup> Pass: Focus on Fe diffusion without alloying with surface

- reduce Fe in source layer: targeted 4e20 Fe/cm<sup>3</sup> in SiO<sub>2</sub>
- reduce temperature to reduce stress on furnace and sample

Experimental details

- Deposited source layer on Sn-doped (0 1 0) and (-2 0 1)
- Wet etched source layer from ~ half of sample surface
- Four anneals performed in purified Ar with one of
  - 1100 °C for 1 hr
  - 1100 °C for 3 hr
  - 1175 °C for 3 hr
  - 1175 °C for 6 hr

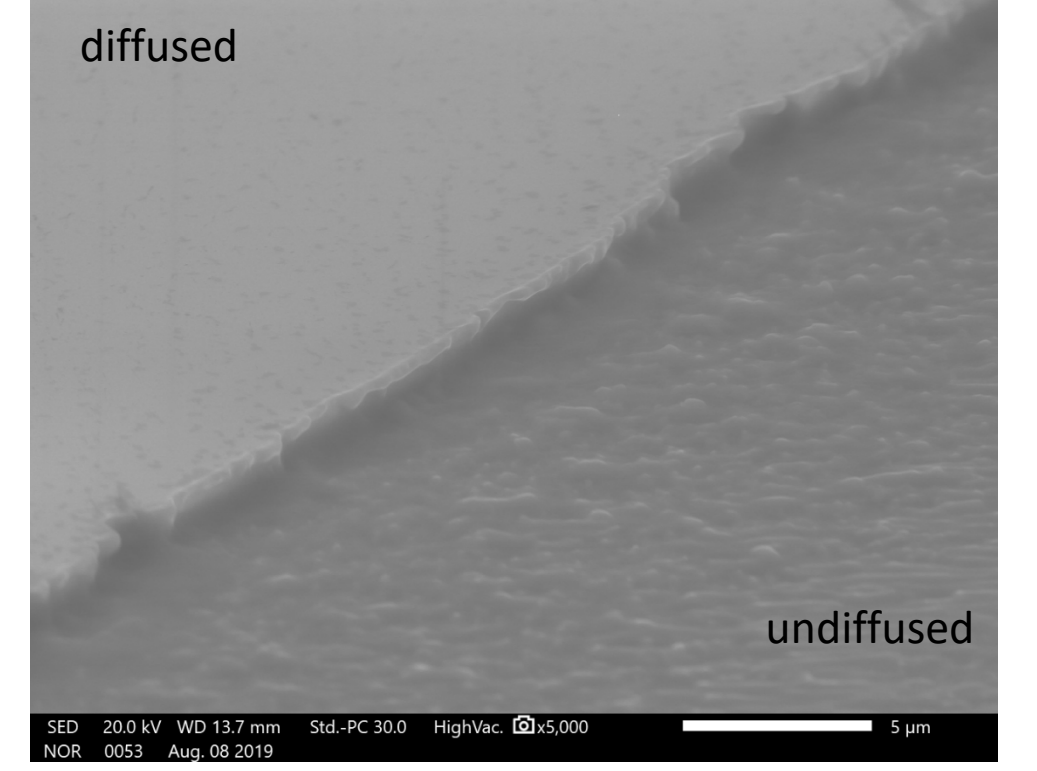
SIMS of Fe-doped SiO<sub>2</sub> source layer



Fe/SiO<sub>2</sub> cosputtered (Fe ~ 4e20), ~ 1500 Å, uniform Fe distribution

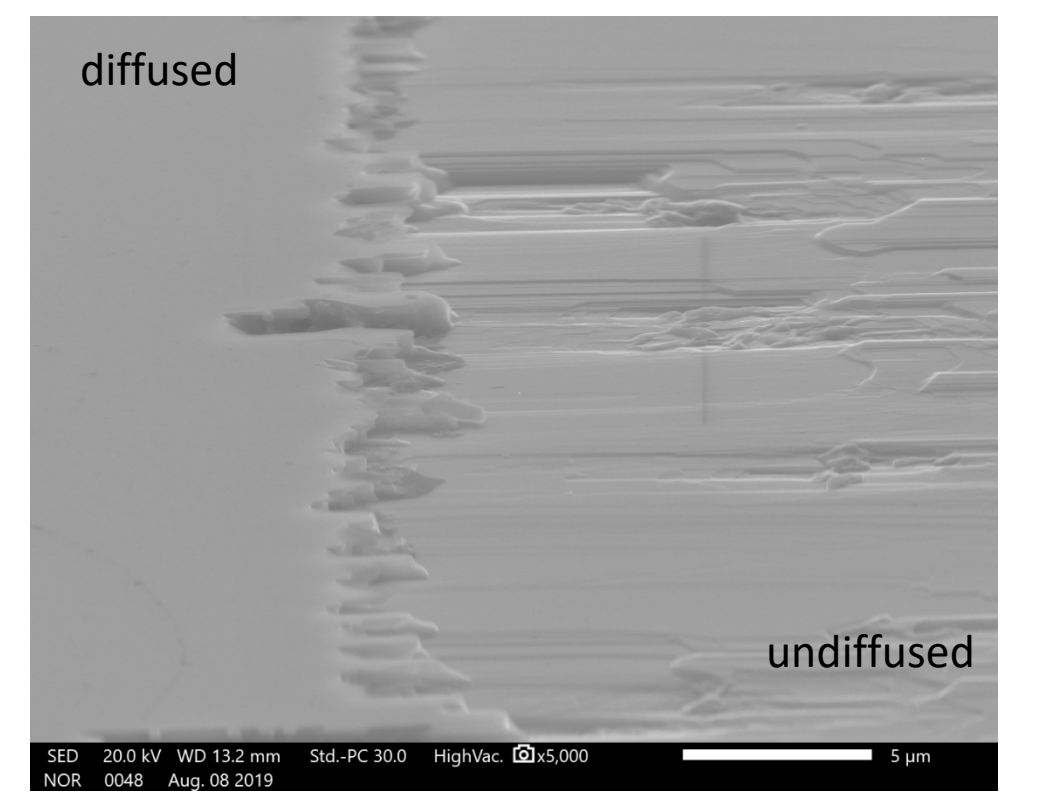
Effect of diffusion on substrate

1175 °C 6hr anneal (010)



All (0 1 0) samples showed no surface roughening in diffused region

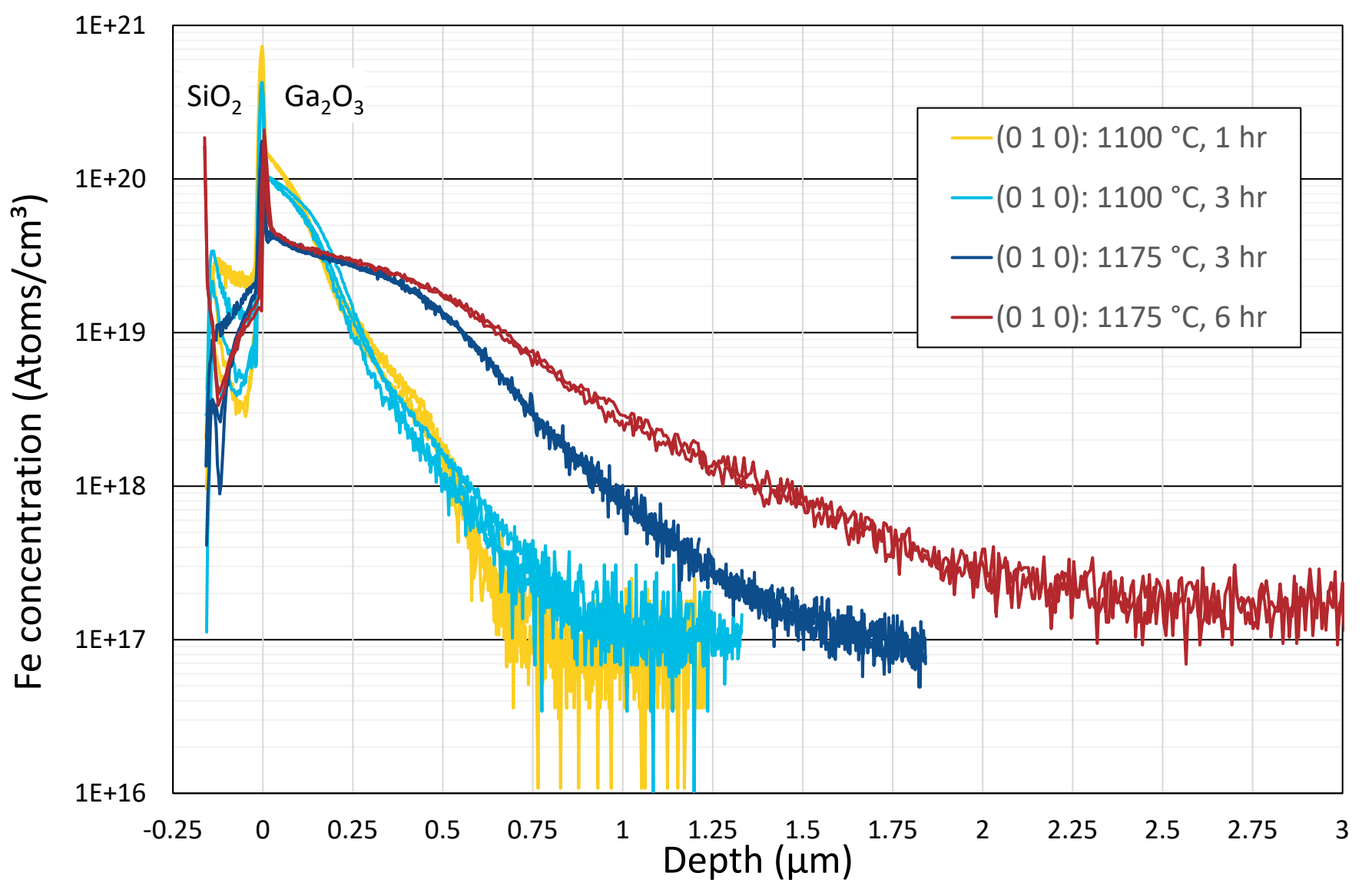
1175 °C, 6hr anneal (-201)



(-2 0 1) samples showed mostly smooth surface with some pitting in diffused region. Pitting grew larger at longer/higher temperature anneals

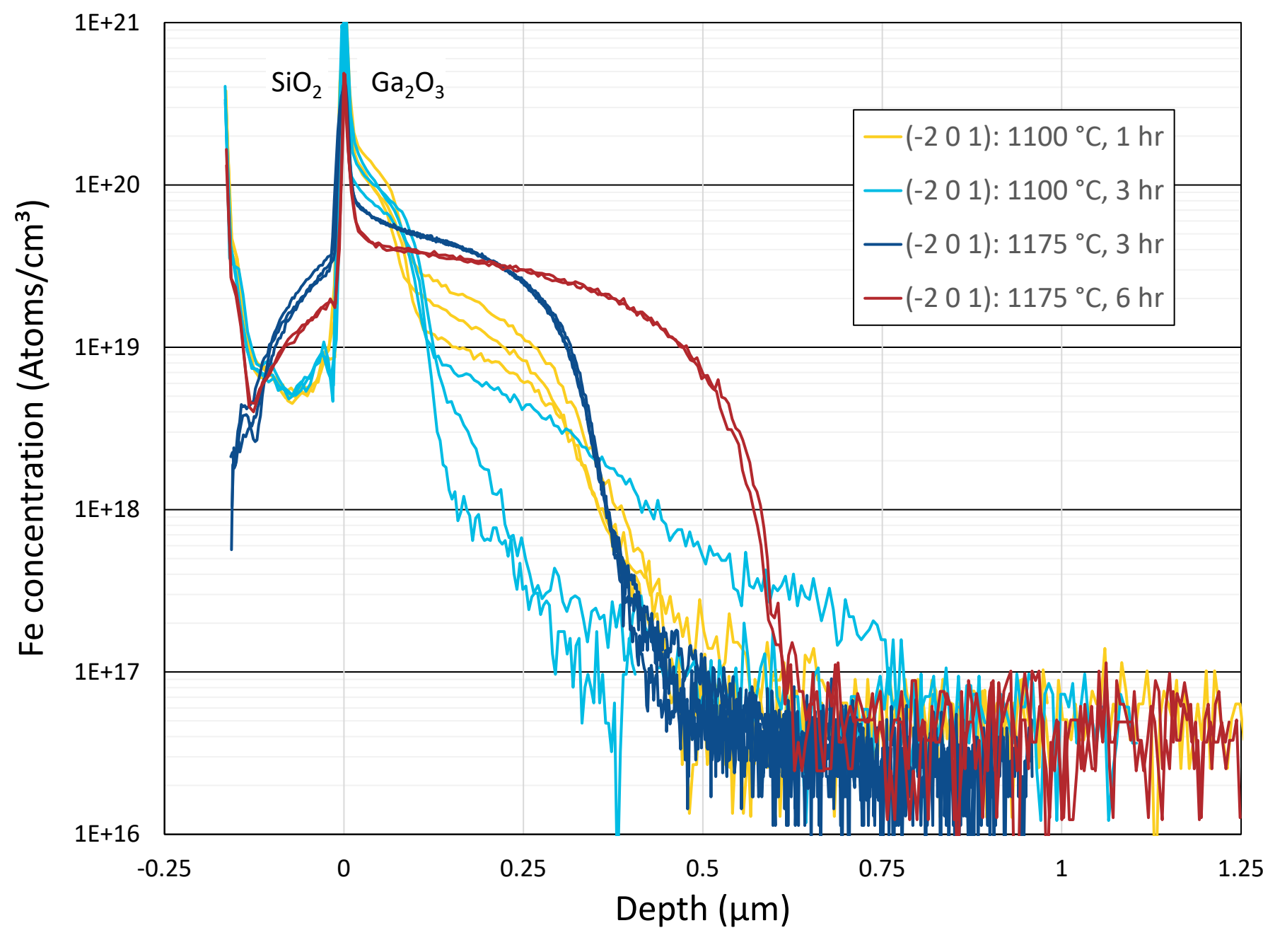
Both orientations showed roughening of the undiffused/uncapped regions

SIMS analysis of (0 1 0) samples Through SiO<sub>2</sub> layer into Ga<sub>2</sub>O<sub>3</sub>



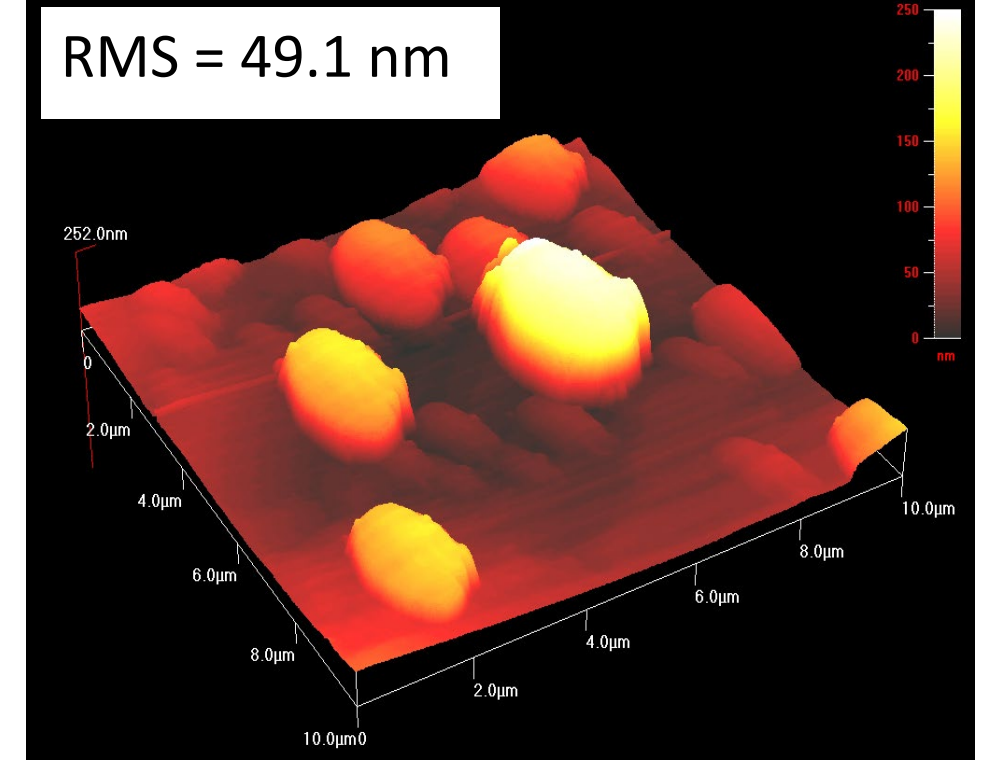
At 1100 °C, not a large difference between 1 hr and 3 hr anneals  
For 3 hr anneals, increasing from 1100 to 1175 °C increased depth of profile by almost 0.5 μm.  
At 1175 °C, increasing the time from 3 to 6 hr had a significant effect on the depth  
**Note:** These scans were not calibrated for SiO<sub>2</sub>. Therefore the Fe concentration in SiO<sub>2</sub> is unreliable.

SIMS analysis of (-2 0 1) samples Through SiO<sub>2</sub> layer into Ga<sub>2</sub>O<sub>3</sub>

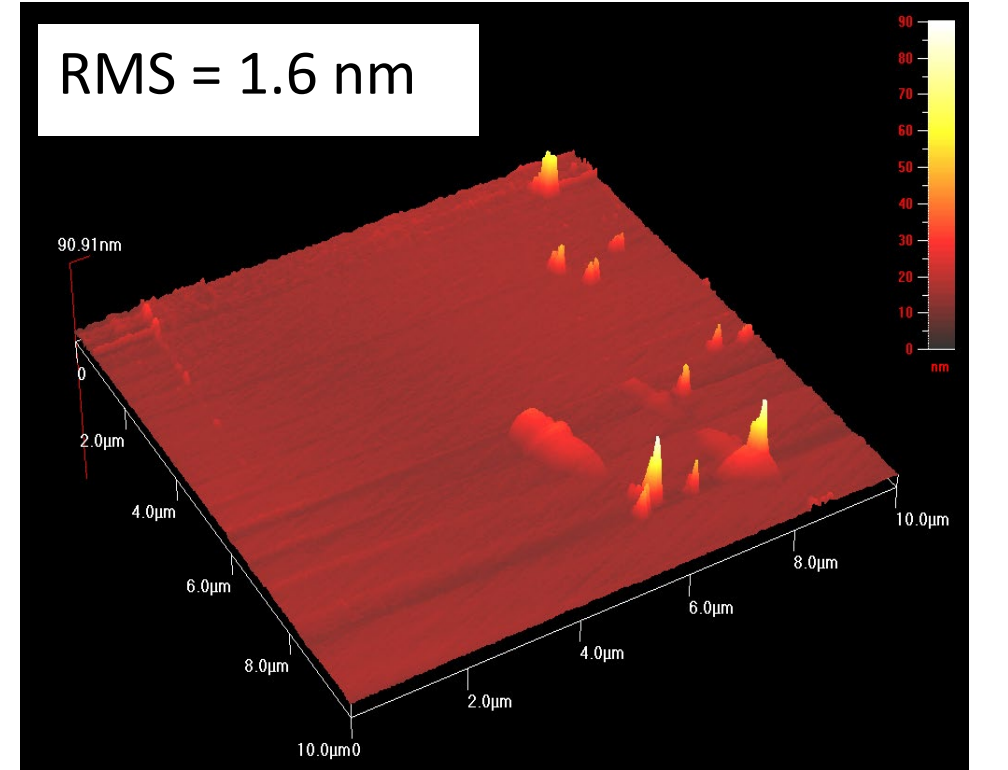


At 1100 °C, multiple scans on each sample produced varied profiles.  
1175 °C samples were more consistent. Concentration dropped sharply below 1e19.  
**Note:** These scans were not calibrated for SiO<sub>2</sub>. Therefore the Fe concentration in SiO<sub>2</sub> is unreliable.

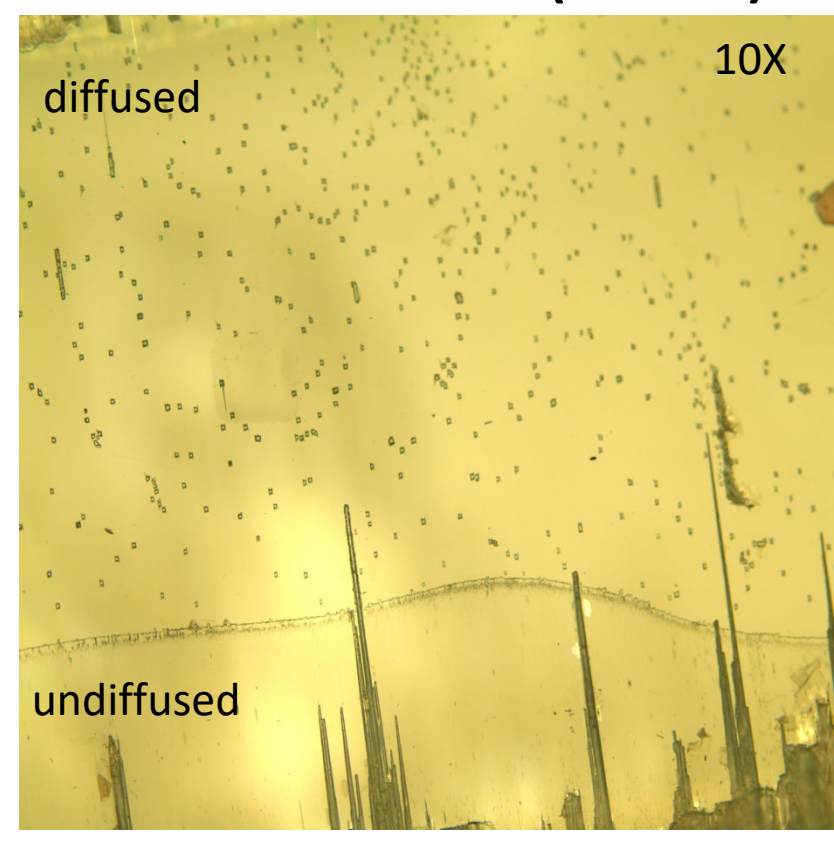
Undiffused, 1175 °C, 6hr anneal (-201)



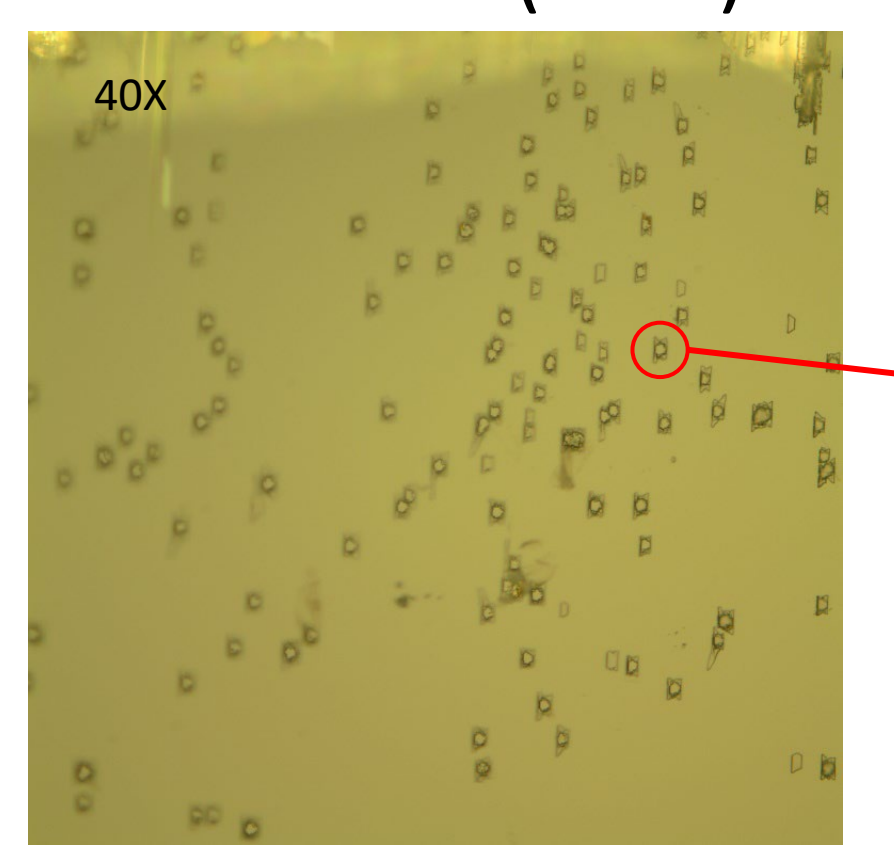
Diffused, 1175 °C 6hr anneal (-201)



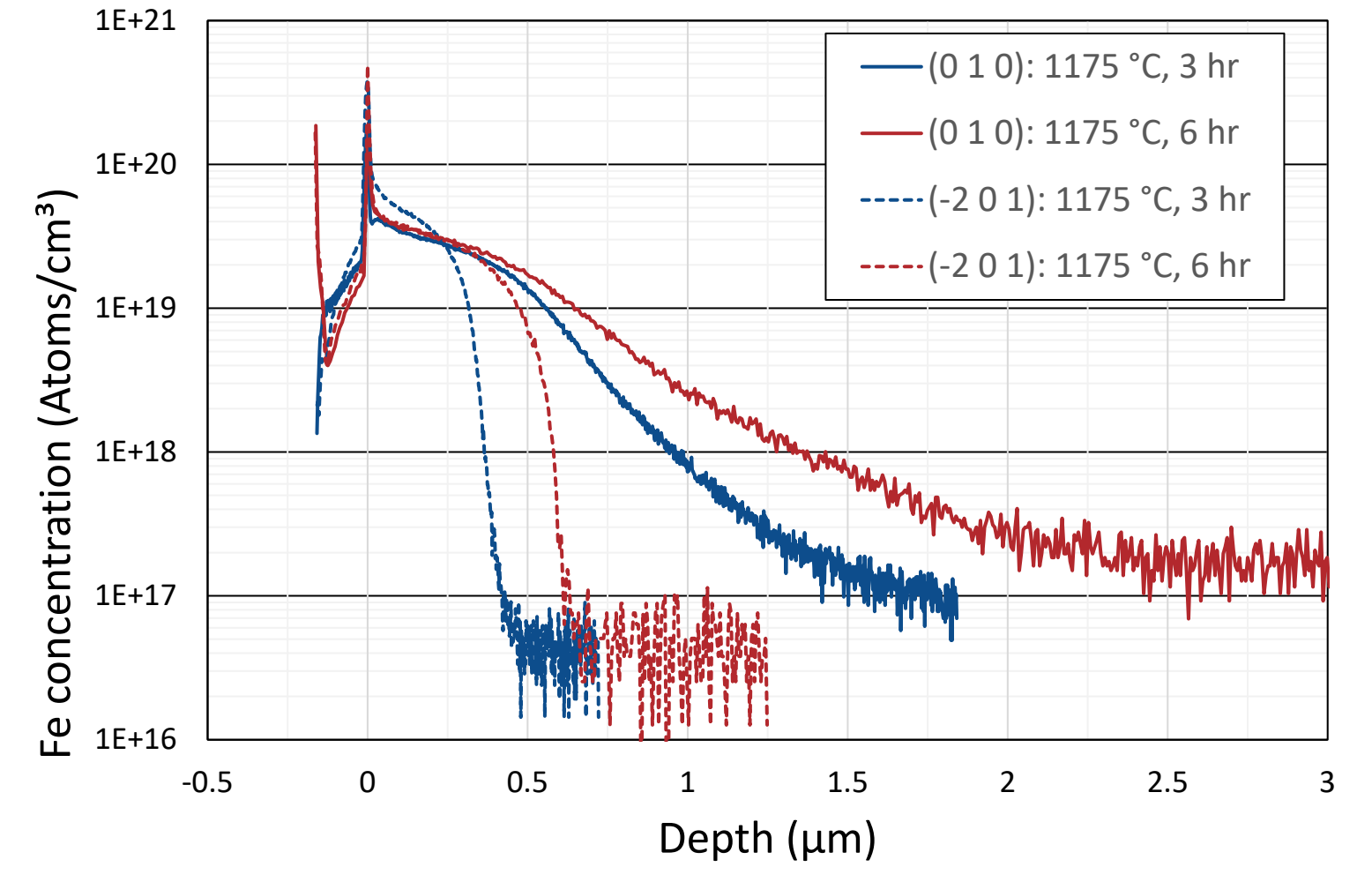
1175 °C, 6hr anneal (-201)



Diffused, 1175 °C 6hr anneal (-201)



(0 1 0) vs (-2 0 1) at 1175 °C



Conclusions

- Fe is promising material to thermally dope β-Ga<sub>2</sub>O<sub>3</sub>
- Smooth diffused surface on (010)
- Pitting observed on diffused surface of (-201)
- Roughening on undiffused regions of both orientations after 3 hr at 1100 °C (entire 1 hr samples were diffused)

Fu

- Cap un
- change
- Reduce