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Title: Characterization of BOR-60 Irradiated 14YWT-NFA1 Tubes

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Appendix E
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QA program which meets the requirements of

☒ DOE Order 414.1 ☐ NQA-1-2000

This Deliverable was subjected to:

☒ Technical Review	☐ Peer Review
Technical Review (TR)	**Peer Review (PR)**
Review Documentation Provided	**Review Documentation Provided**
☐ Signed TR Report or,	☐ Signed PR Report or,
☐ Signed TR Concurrence Sheet or,	☐ Signed PR Concurrence Sheet or,
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Clarissa A. Yablinsky [signature] 1/31/2017

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Characterization of BOR-60 Irradiated 14YWT-NFA1 Tubes

Fuel Cycle Research & Development

Prepared for
U.S. Department of Energy
NTR&D Campaign

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1/31/2017
M3FT-17LA020202079

SUMMARY

Tubes of FCRD 14YWT-NFA1 Alloy were placed in the BOR-60 reactor and irradiated under a fast flux neutron environment to two conditions: 7 dpa at 360-370 °C and 6 dpa at 385-430 °C. Small sections of the tube were cut and sent to UC Berkeley for nanohardness testing and focused ion beam (FIB) milling of TEM specimens. FIB specimens were sent back to LANL for final FIB milling and TEM imaging. Hardness data and TEM images are presented in this report. This is the first fast reactor neutron irradiated information on the 14YWT-NFA1 alloy.

TABLE OF CONTENTS

FIGURES

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INTRODUCTION

Cladding materials for next generation nuclear reactors require resistance to high doses of fast neutron flux. Work through the Department of Energy's (DOE) Nuclear Technology Research and Development (NTR&D) Program's Advanced Fuel Campaign has identified 14YWT, a nanoferritic oxide dispersion strengthened alloy as a promising alloy and has made a 50 kg batch of material, FCRD-NFA1, with nominal composition Fe-14Cr-3W-0.4Ti-0.2Y in weight percentage. Plates were extruded from milled powder at Oak Ridge National Laboratory and then electro-discharge machined (EDM) into two sizes of tubes (Table I) at Los Alamos National Laboratory (LANL). The OD of the tubes were polished to remove the EDM recast layer. Twenty-four of these tubes were placed into the BOR-60 reactor, a sodium fast reactor, at the RIAR lab in Dimitrovgrad Russia. Irradiation conditions depended on exact location within the reactor, but the two samples in this study (L108 and L202) were irradiated to 7 dpa at 360-370 °C (Sample L108), and 6 dpa at 385-430 °C (L202). More detailed temperature models are pending for sample L202. Post irradiation examination took place at both LANL and the University of California, Berkeley (UCB). Facilities used at LANL (CMR Wing 9 Hot Cells, Electron Microscopy Lab (EML)) are part of a pending NSUF partner facility application, while the work at UCB was completed under a Nuclear Science User Facility, Rapid Turnaround Experiment Proposal (NSUF-RTE).

Table I. Tube dimensions, dose and irradiation temperature.

	L108	**L202**
Length (mm)	30.99	26.67
Outer Diameter (mm)	4.57	4.57
Inner Diameter (mm)	3.30	4.01
Wall Thickness (mm)	1.27	0.56
Dose (dpa)	7	6
Irradiation Temp (°C)	360-370	385-430

RESULTS

The irradiated tubes were received from RIAR at LANL's CMR Wing 9 hot cells in a 110 gallon lead shielded drum. The tubes were unpacked in the hot cell corridors where they were decontaminated to remove extensive loose alpha contamination. An ultrasonic cleaner with sodium metasilicate (as a coagulant) was used to complete this cleaning. Once the tubes were cleaned of alpha contamination, they were moved into the hot cells and cut into 2mm lengths and then quartered to make ~2x2mm tube sections. These tube sections were shipped to UCB where they were prepared for nanohardness measurements by mounting and polishing. [1] Berkovich nanohardness was measured with a Micro Materials NanoTest Vantage system. Hardness data was taken at indentation depths of 350, 500 and 750nm, as well as with multiple-load-cycle indentations. Results were fairly consistent between 500 nm, 750 nm and multi-load indentation experiments. A summary of nanohardness at 500 nm depth versus radial distance along tubes can be seen in Fig. 1. The thinner tube, (L202), has some hardening at the outer diameter, possibly due to remaining EDM recast layer. Results are consistent with irradiation conditions, with lower temperature irradiation (L108) displaying a higher hardness than the higher irradiation temperature (L202).

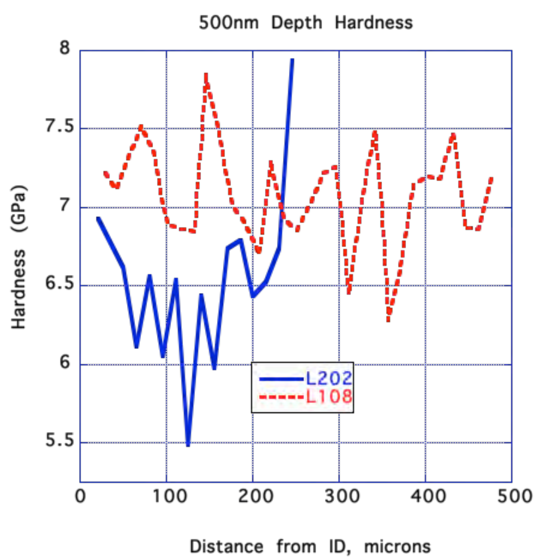

Fig. 1. Nanohardness at 500nm indent depths

Small TEM blanks were subsequently cut from the tubes using a focus ion beam (FIB) and shipped back to the EML at LANL for TEM analysis. Final thinning was done on a FEI Helios Nanolab 600 dual beam FIB and TEM analysis on a FEI Tecnai F30 TEM operating at 300 kV. Initial TEM results showing damage microstructure and appearance of small voids are shown in Fig. 2 and 3 respectively for Tube L202, and Fig. 4 and 5 for Tube L108. Notably tube L108 displays both spherical and elongated voids as seen in Fig. 5. Based on TEM analysis of voids, swelling percentage is shown in Table II. Tube L108 displays a slightly higher swelling percentage in the radial direction than Tube L202, however the overall swelling percentage is quite low at these dpa, with a maximumum of 0.03% swelling. Additional and larger TEM images can be seen in Appendix A.

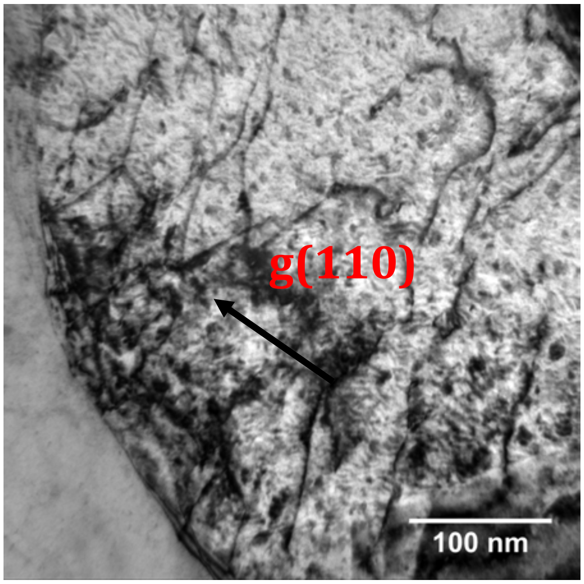

Fig. 2. TEM micrographs of Tube L202 showing radiation damage microstructure.

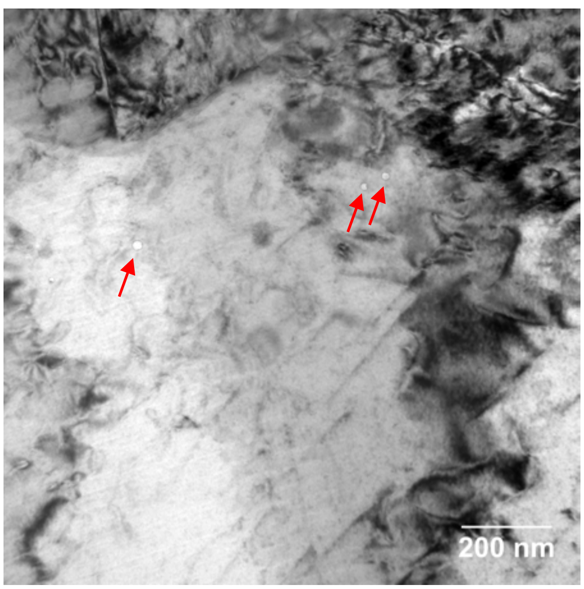

Fig. 3. TEM micrographs of Tube L202 showing the presence of small voids in underfocus conditions. Arrows point out the voids.

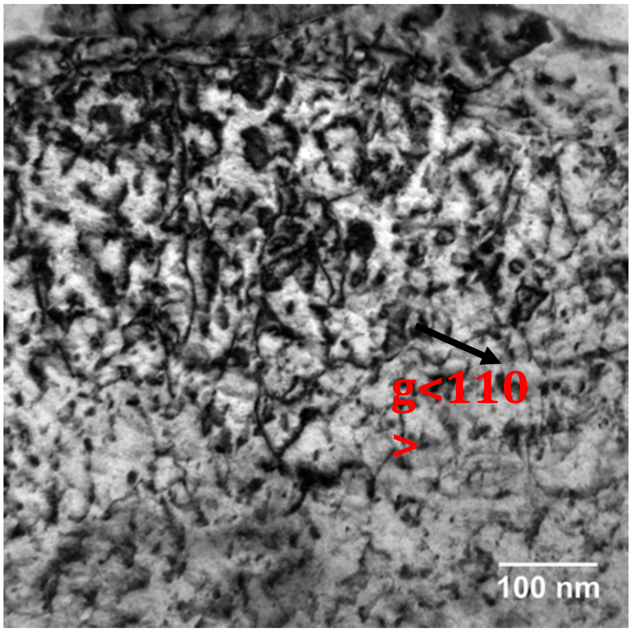

Fig. 4. TEM micrographs of Tube L108 showing radiation damage microstructure.

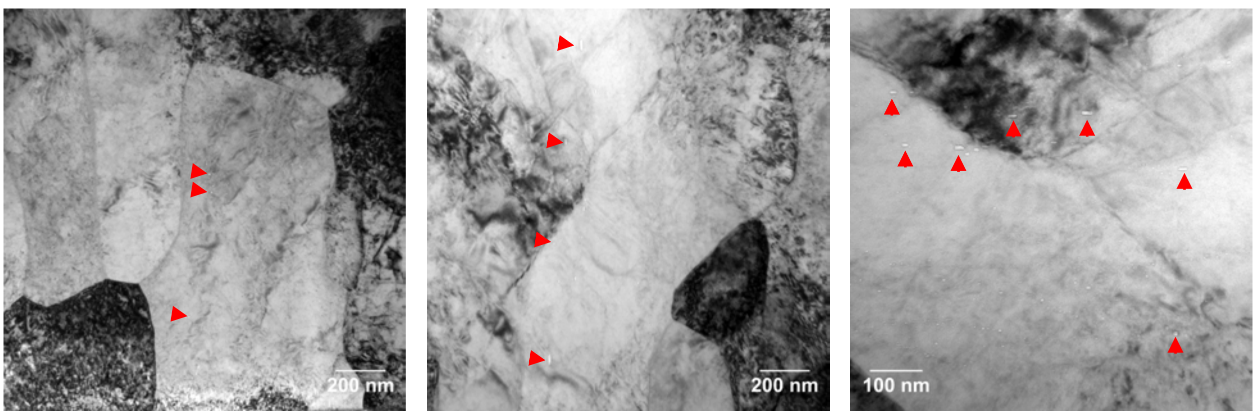

Fig. 5. TEM micrographs of Tube L202 showing the presence of small voids in underfocus conditions. Arrows point out the voids.

Table II. Swelling percentage from TEM analysis.

	L108 Thick	L202 Thin
Radial Swelling %	0.015	0.005
Azimuthal Swelling %	0.030	0.026
Dose (dpa)	7	6
Irradiation Temp (°C)	360-370	385-430

CONCLUSIONS AND FUTURE WORK

The results presented here are the first data from fast neutron irradiated FCRD-NFA1 14YWT alloy. This was a successful use of multiple NSUF facilities to obtain nanomechanical properties and TEM images from irradiated, activated, and contaminated material. Initial results are consistent with irradiation conditions. Nanohardness and TEM work on control material is ongoing at both LANL and UCB. Preparation for tensile ring pull tests for bulk mechanical properties are underway at LANL.

ACKNOWLEDGEMENTS

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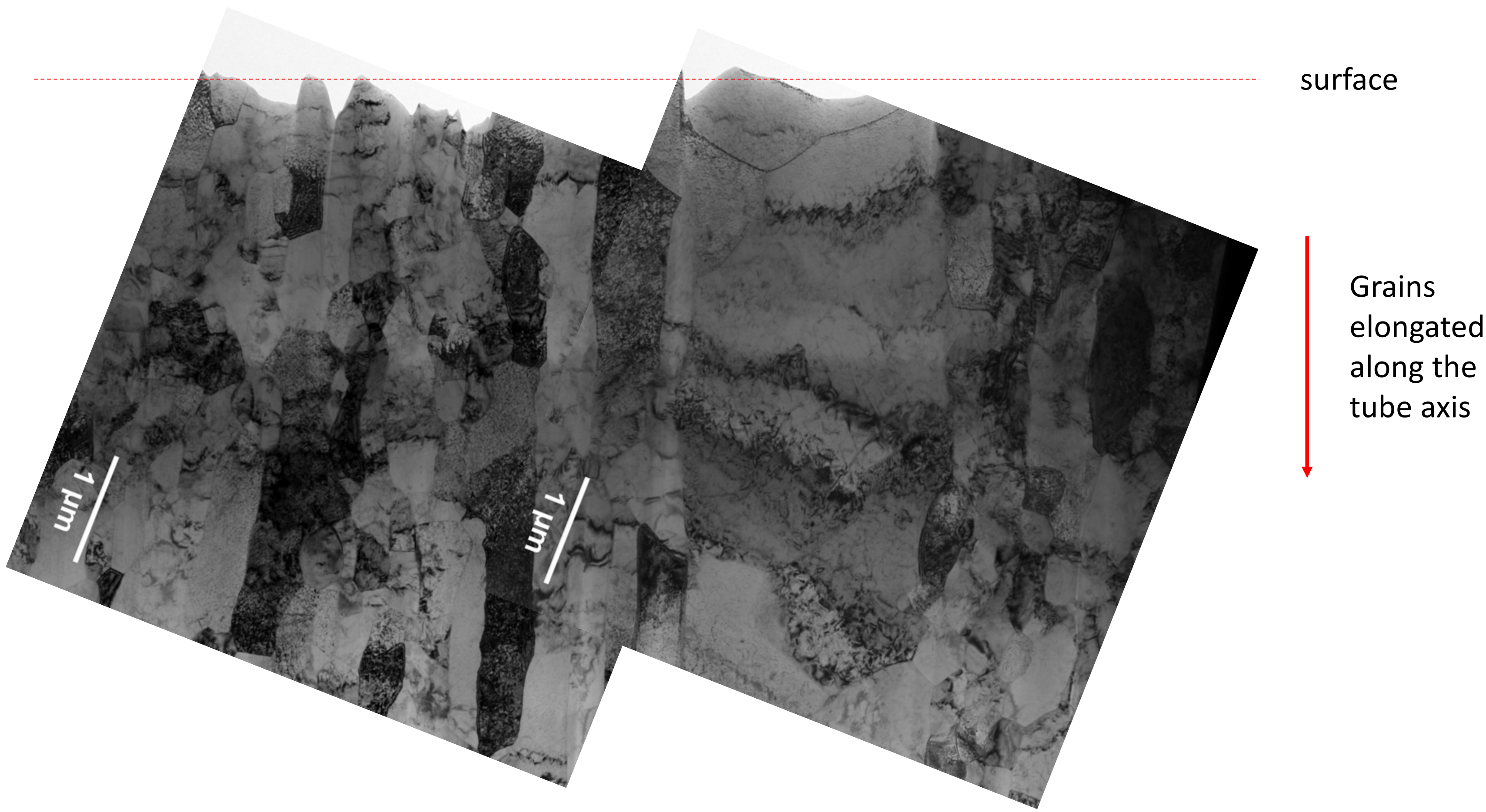
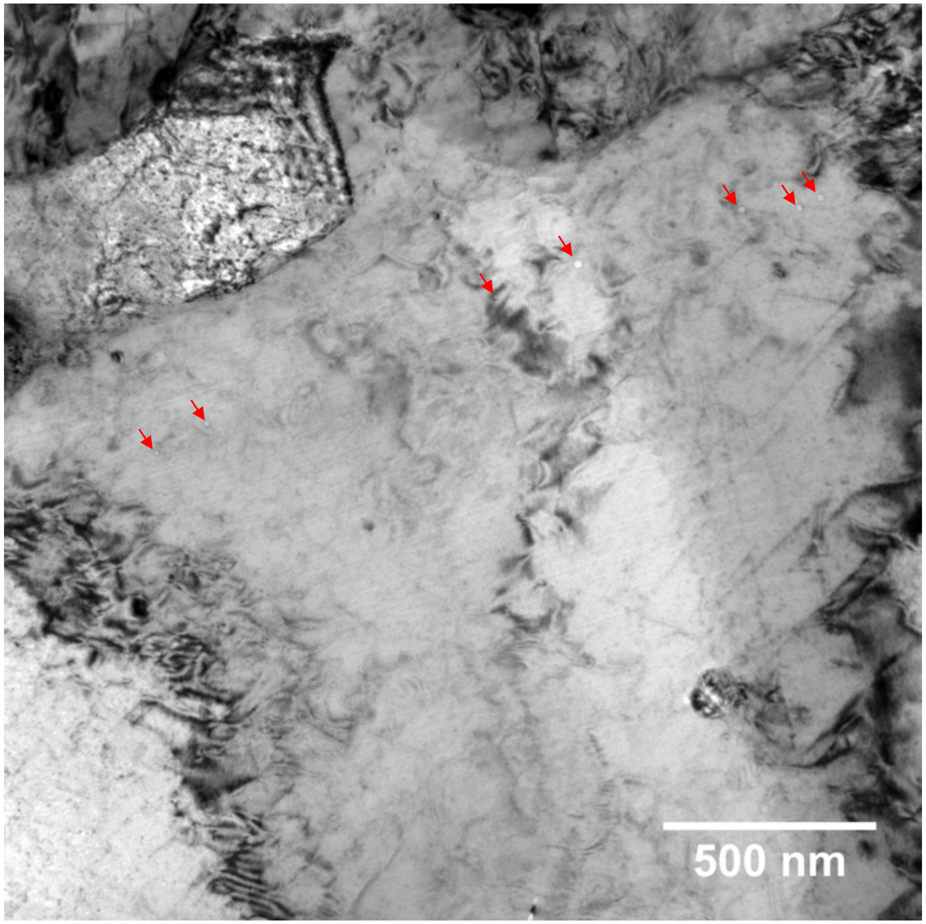
1. Krumwiede, D.L., Abad, M.D., Saleh, T.A., Maloy, S.A., Odette, G.R., Yamamoto, T., Hosemann, P. Initial studies on the correlation of nanohardness to engineering-scale properties of neutron-irradiated steels (2016) International Congress on Advances in Nuclear Power Plants, ICAPP 2016, 1, pp. 224-229.

Appendix A

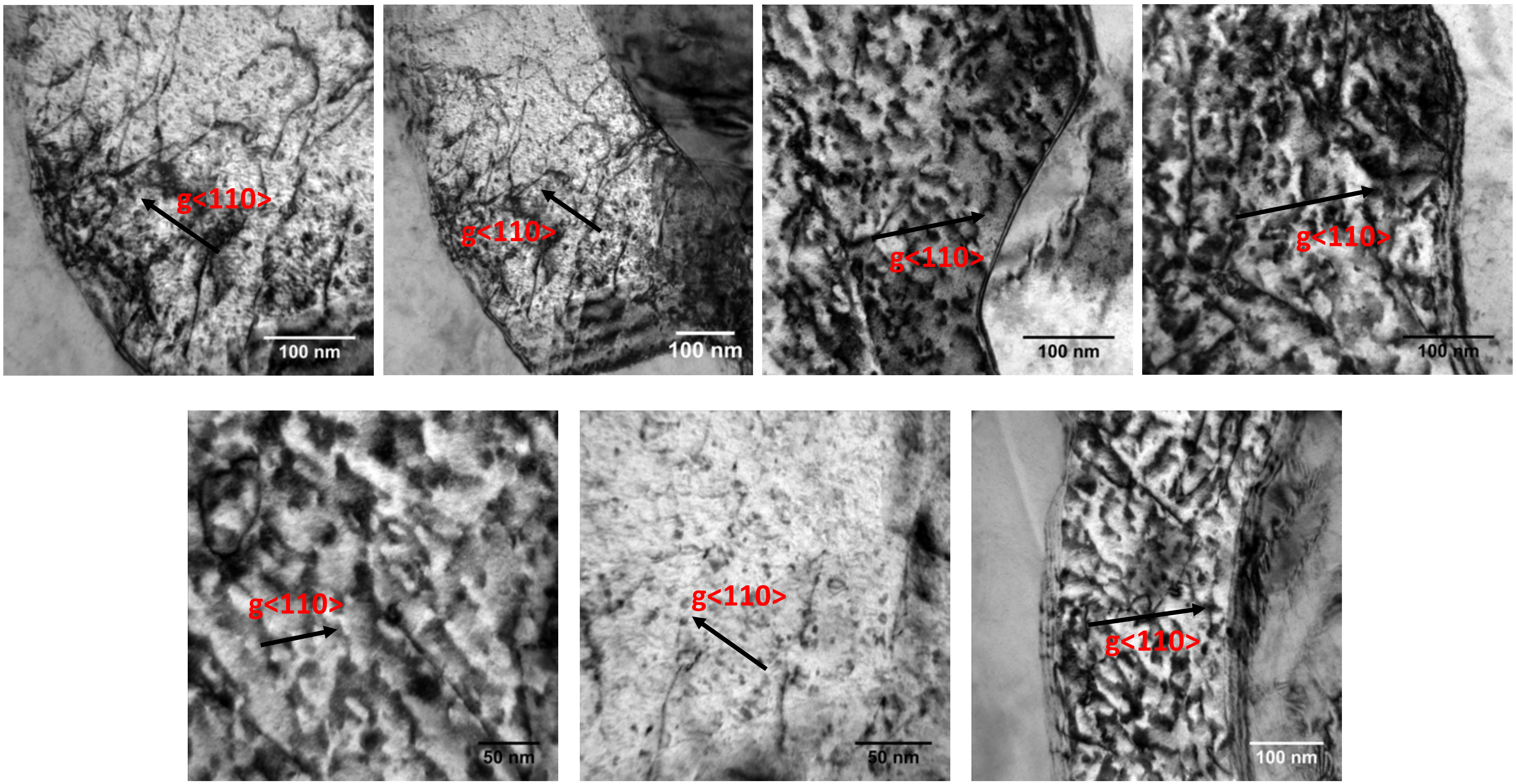
L202 – THIN
6 dpa at 385-430 C

Thin-Foil 1 (radial direction)





Some voids were observed at 3 µm under-focus.



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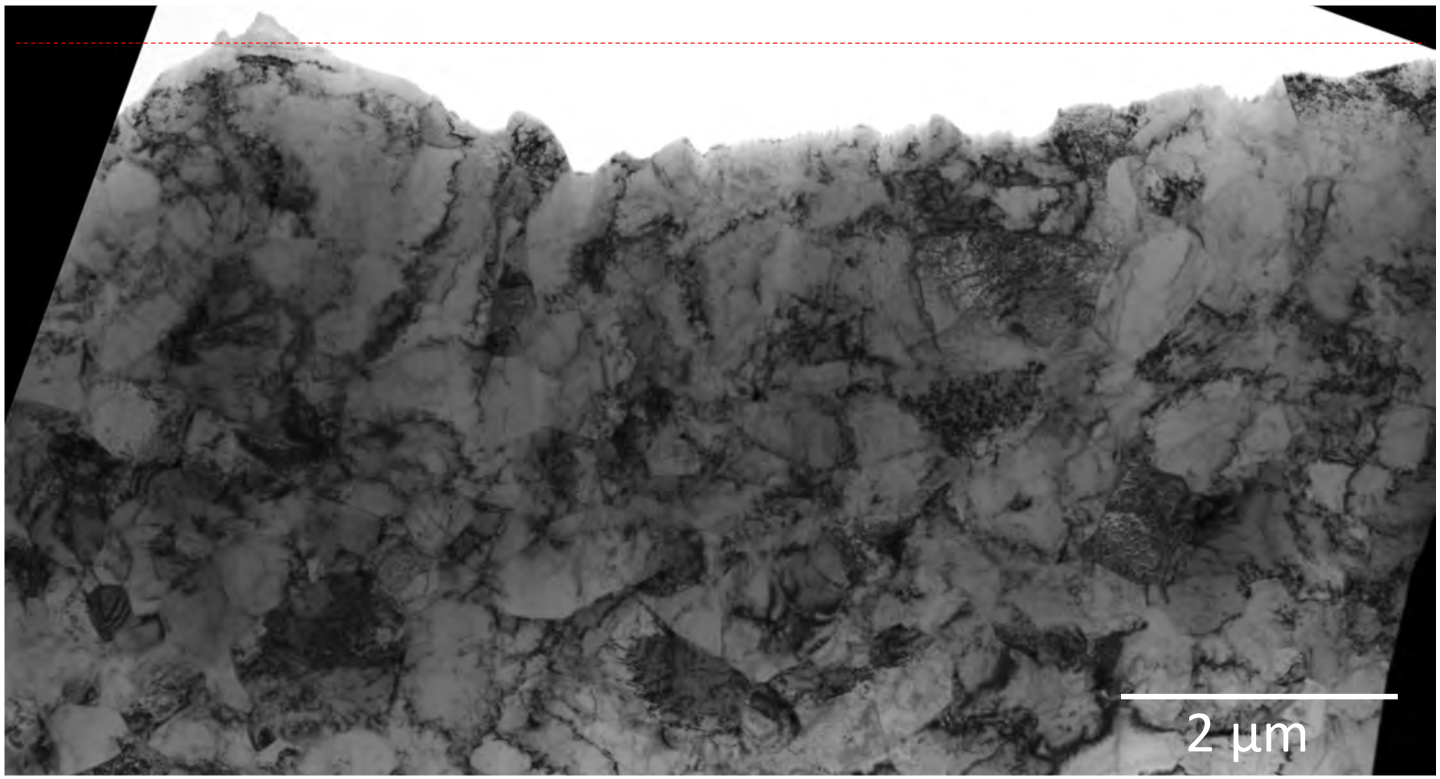
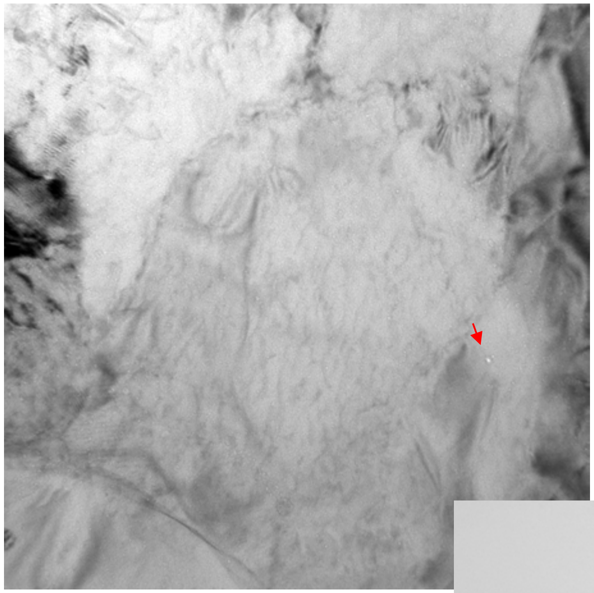
Thin-Foil 2 (azimuthal direction)





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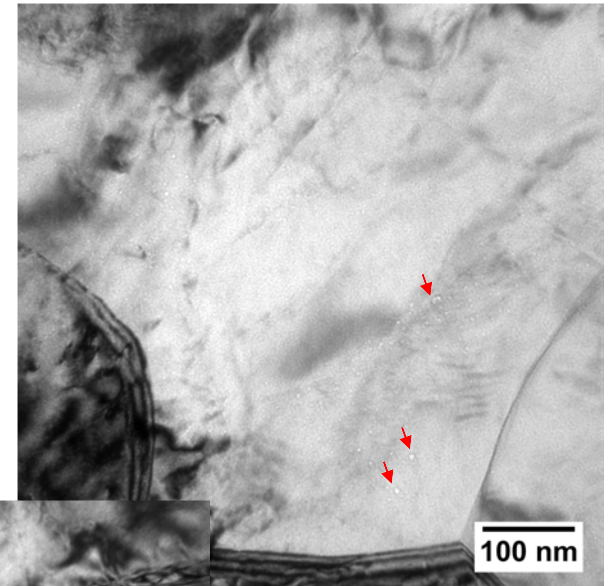

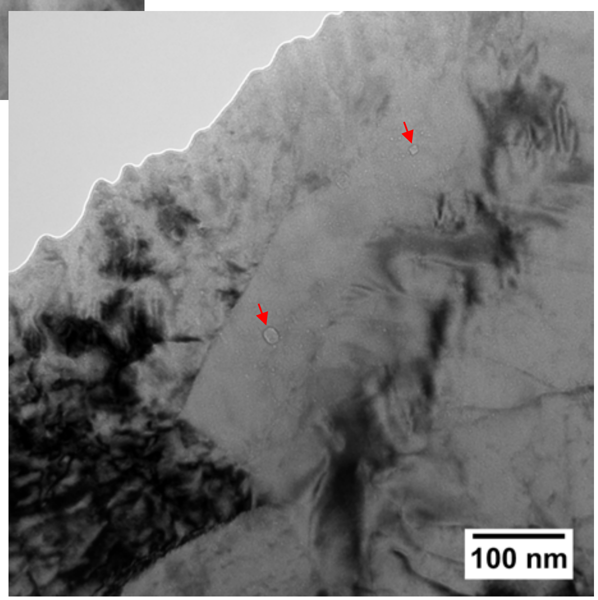

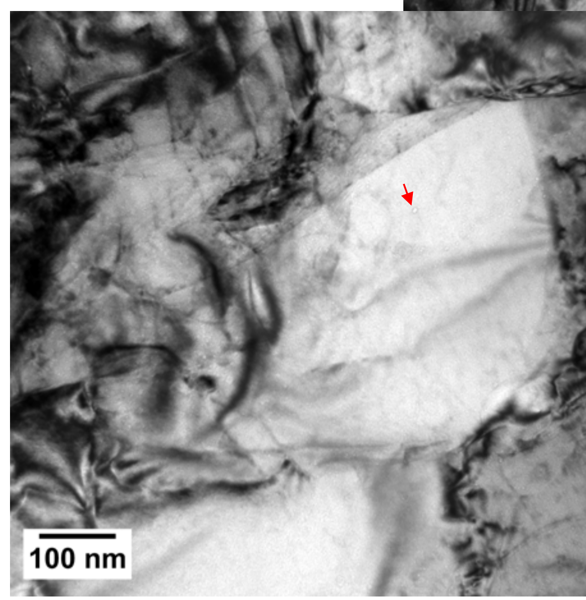

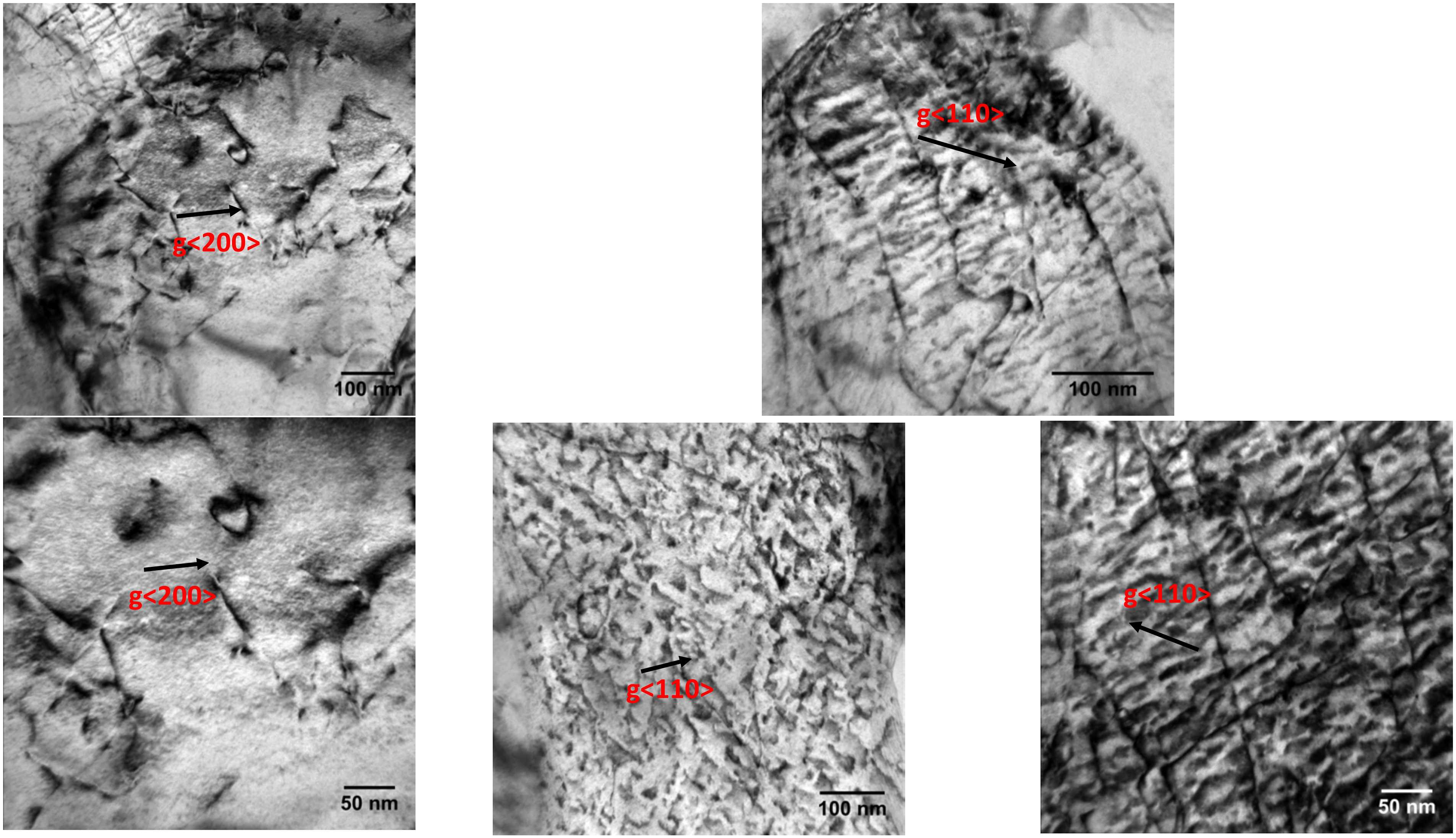
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L108 – Thick Tube
7dpa at 360-370C

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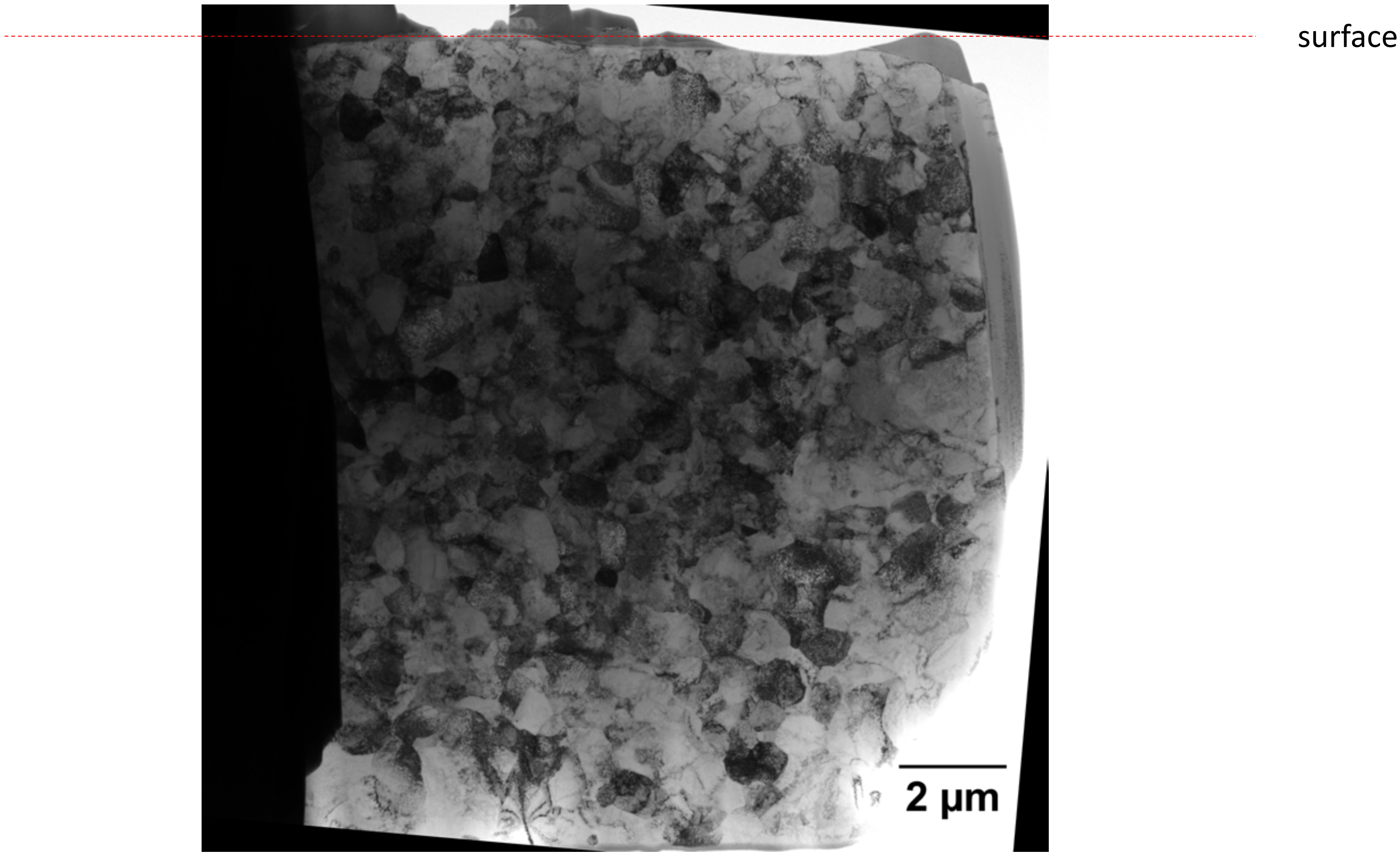

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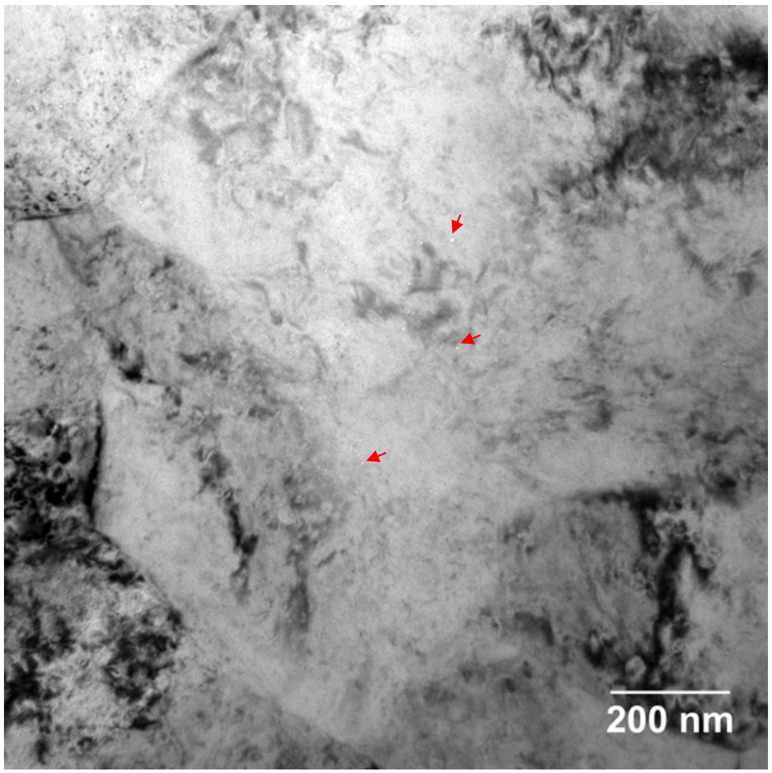

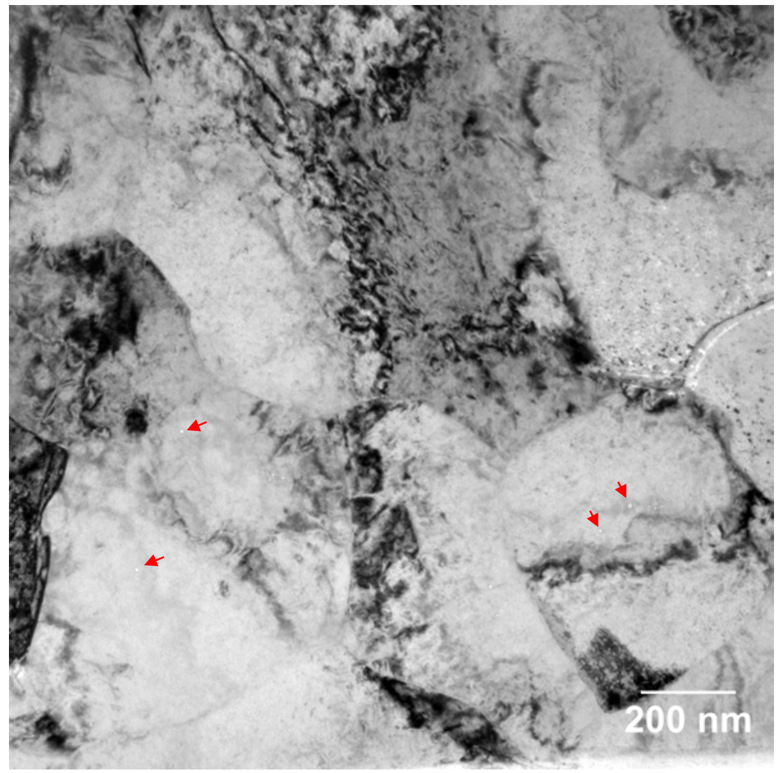

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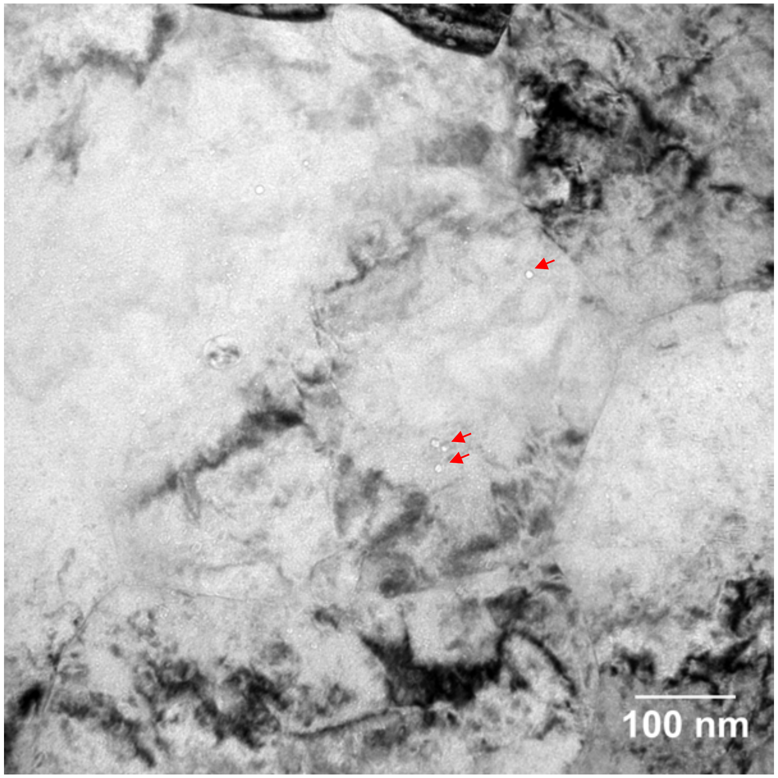

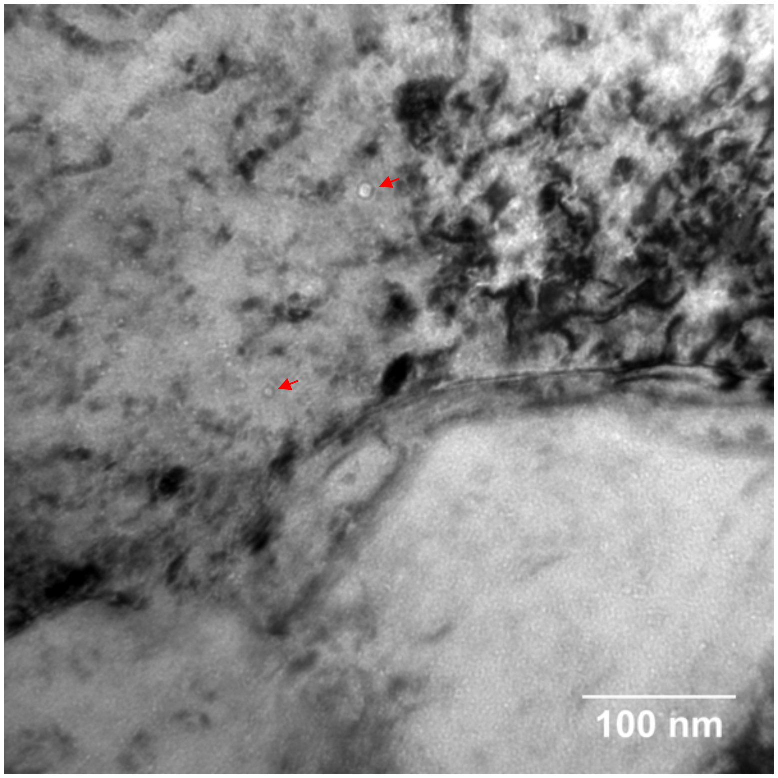

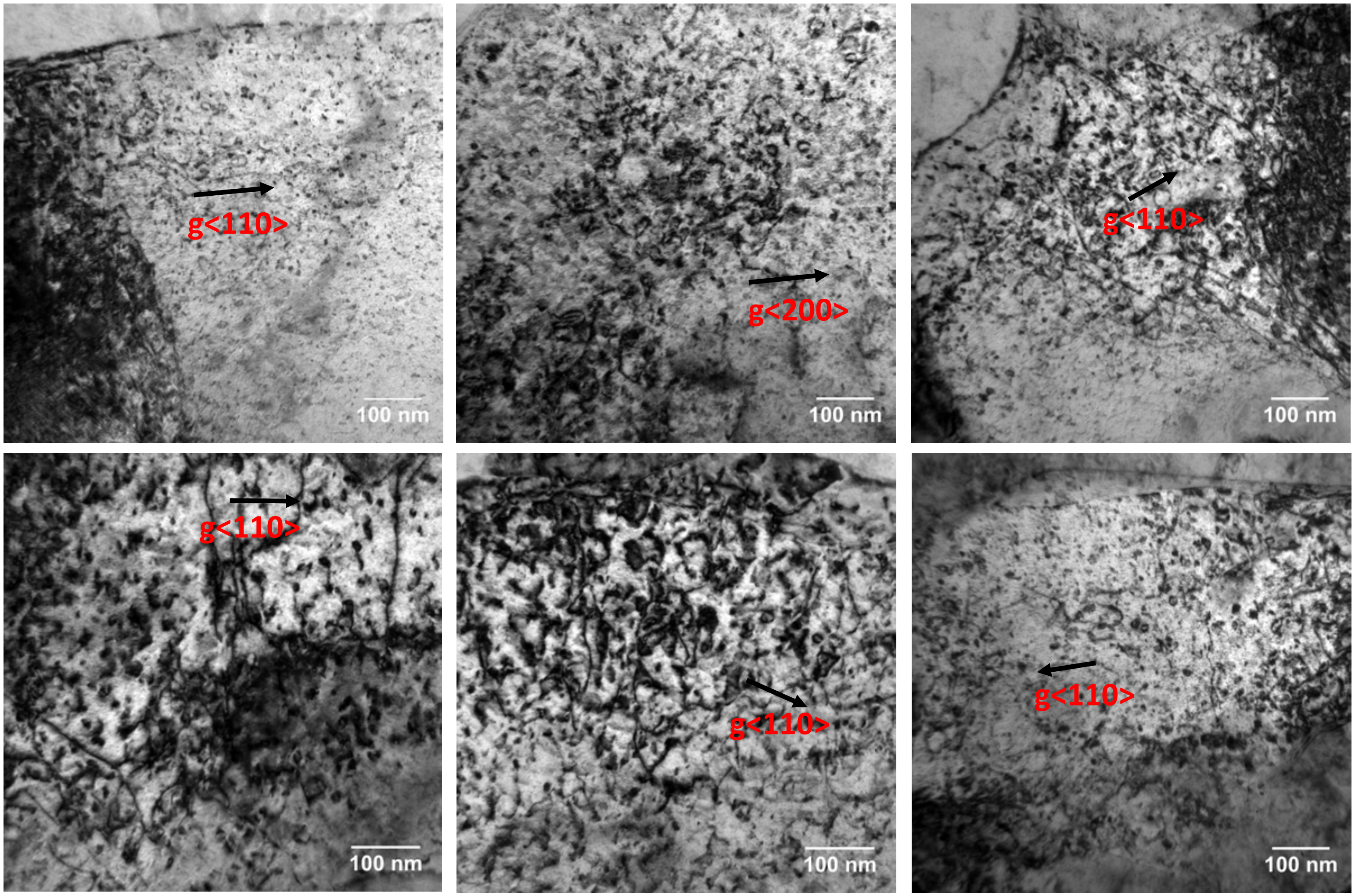
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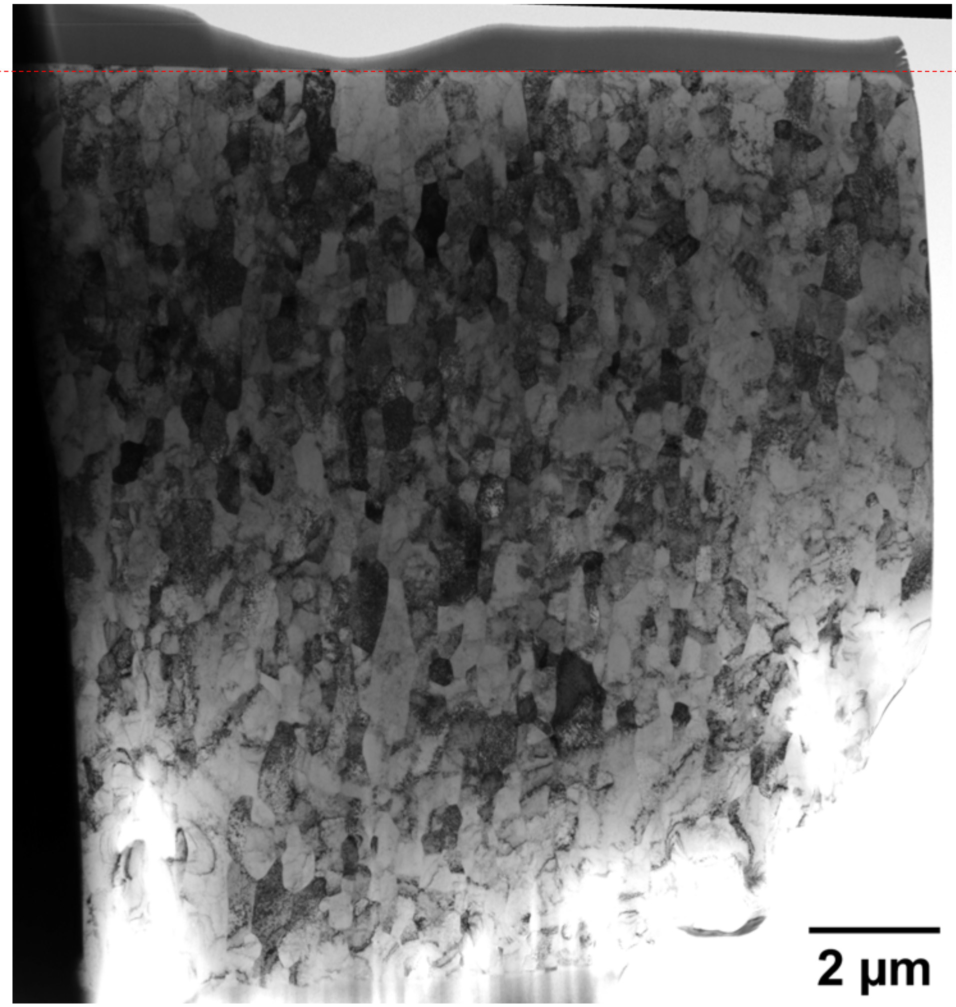
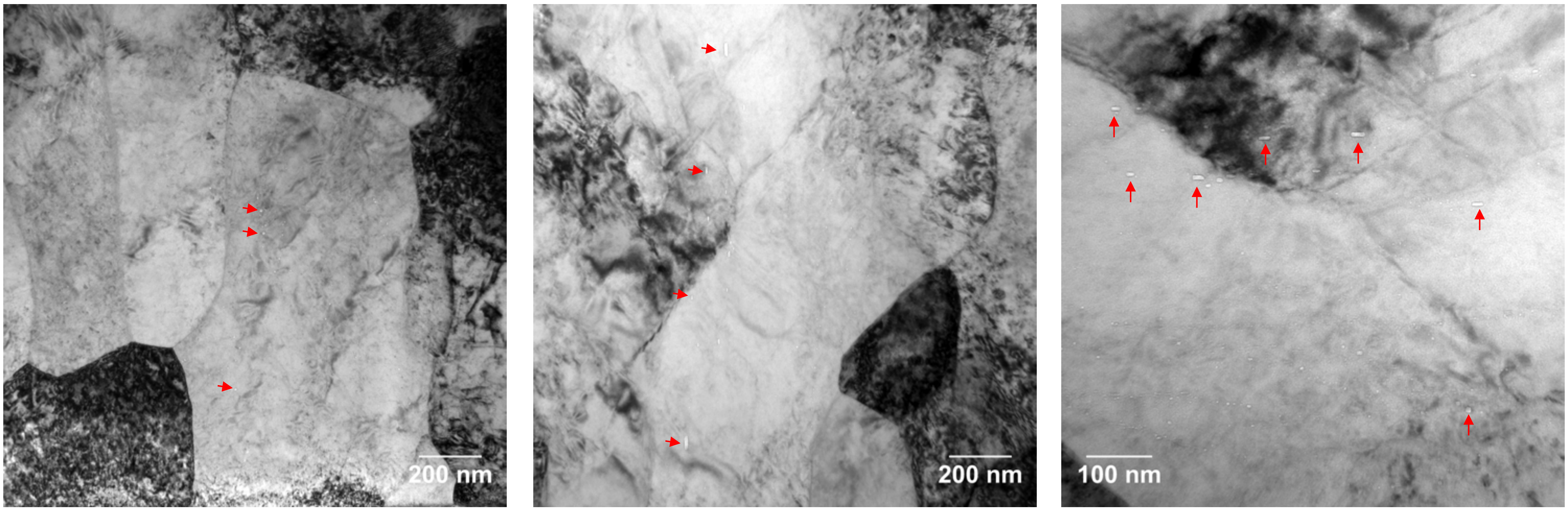
Thick-Foil 2 (azimuthal direction)



surface

Grains elongated along the tube axis

Some voids were observed at 3 µm under-focus.



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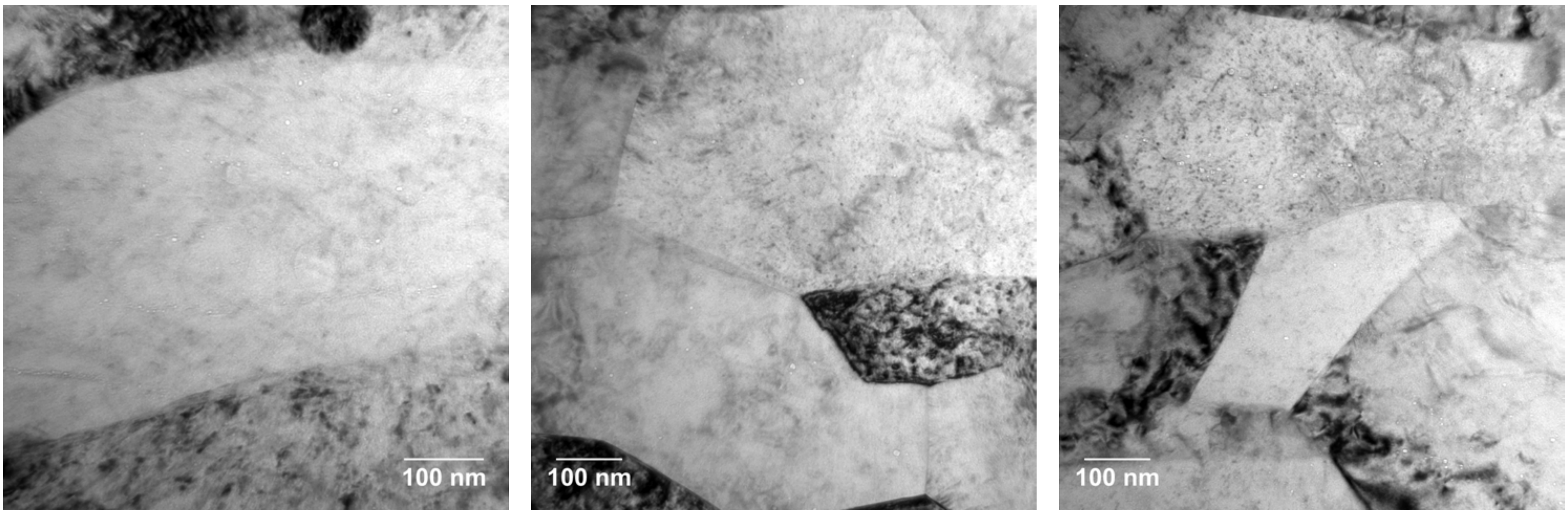

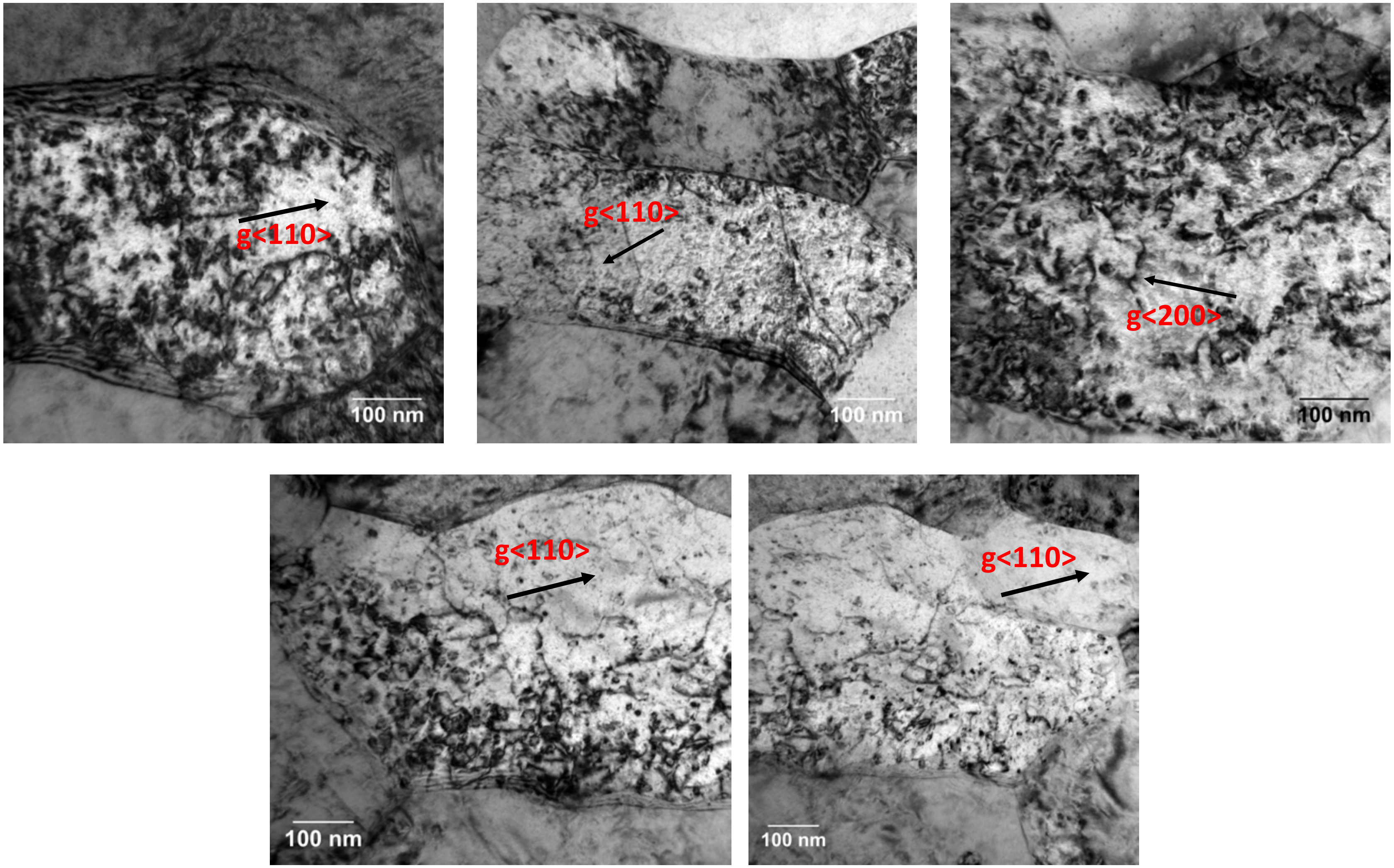
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