Microscopic Investigation of Flux Trapping Sites in Bulk Nb

Martina Martinello, Zu-Hawn Sung, Sam Posen, Jaeyel Lee, Anna Grassellino, Alex Romanenko

International Conference on RF Superconductivity

2 July 2019

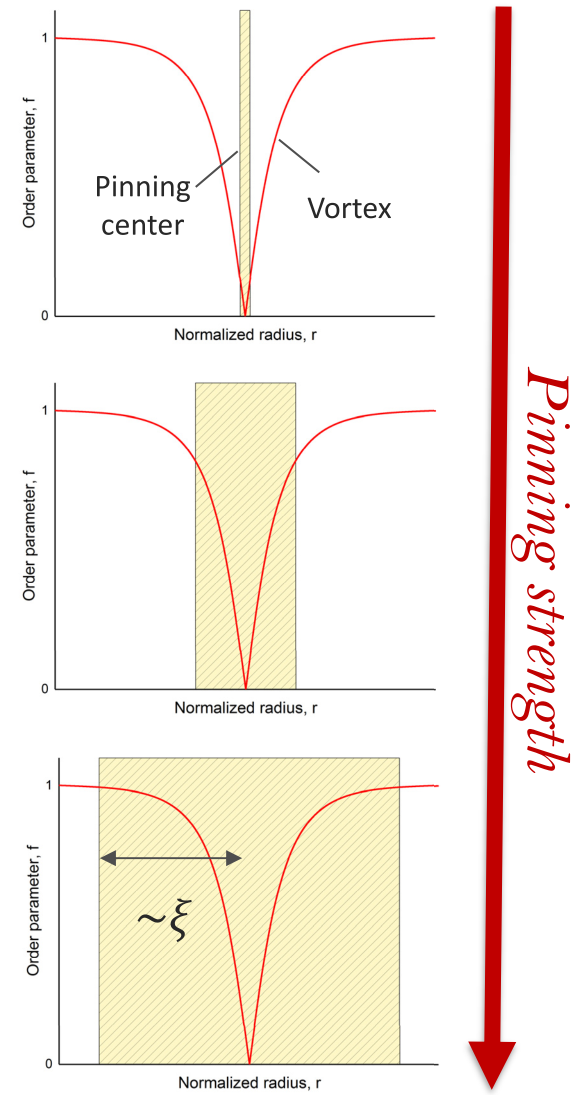
Outline

- **Introduction on pinning**
- Insights from flux expulsion studies in Nb cavities
- Microscopy studies of Nb "as-received" materials with different flux expulsion properties
- Microscopy studies of cavity cut-outs with different flux expulsion properties
- Conclusions

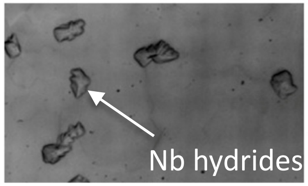
What pinning means and why it occurs

Pinning $\Rightarrow$ *minimization of the system energy*

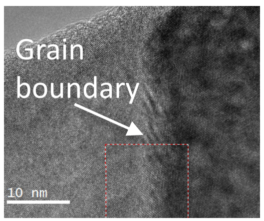
- Vortex lowers locally the condensation energy of the SC (within length $\sim 2\xi$)
- Defects also lower locally the condensation energy of the SC, and, depending on the type of defect, the change may be more or less dramatic
- <u>When vortices are pinned in defects the overall loss in condensation energy of the SC is minimized</u>
- The most efficient minimization of energy happen when pinning centers have dimension comparable to $\sim 2\xi$ (for niobium in clean limit $\xi \cong 38\,nm$ at 2K and $\xi \cong 150 - 300\,nm$ close to Tc)

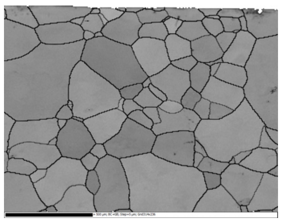


Possible pinning sites in Nb



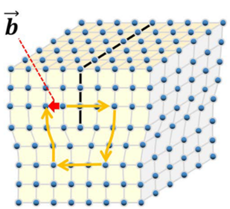
- <u>Normal-conducting and dielectric inclusions</u>: 3-D defects that introduce large κ variation (ex: nano-hydrides in the near-surface area)



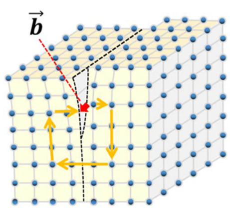


- <u>Grain boundaries</u>: 2-D defects in the crystal structure, they define the interface between 2 grains.
  - <u>Low-angle GBs</u>: the misorientation between the two grains is <15 degrees

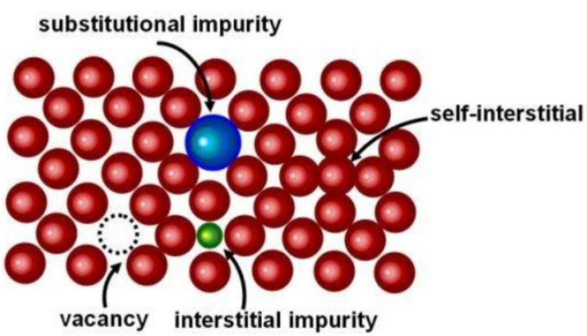
Edge dislocation



Screw dislocation

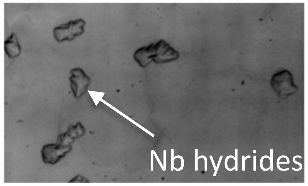


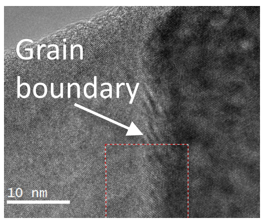
- <u>Dislocations</u>: areas were the atoms are out of position in the crystal structure.
  - <u>Tangles</u>: after plastic deformation very small grain forms (cells) that are surrounded by tangles of dislocations

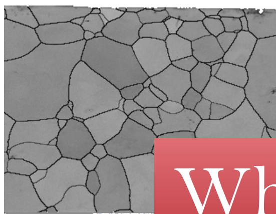


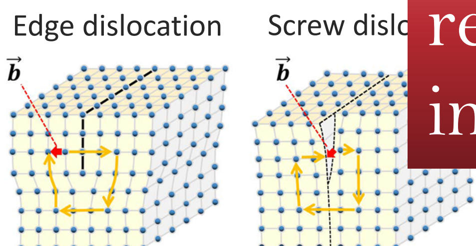
- <u>Local disorder</u>: 1-D defects (ex: impurities, vacancies)

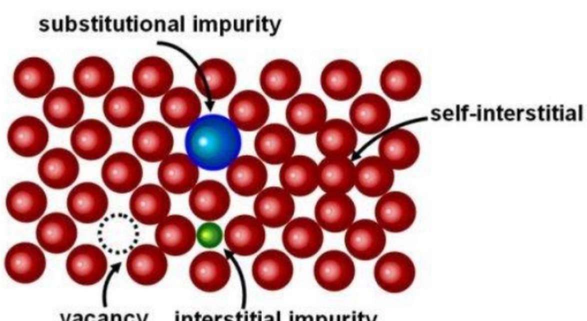
Possible pinning sites in Nb











- Normal-conducting and dielectric inclusions: 3-D defects that introduce large κ variation (ex: nano-hydrides in the near-surface area)

- Grain boundaries: 2-D defects in the crystal structure, they define the interface between 2 grains.

  ation between the

  oms are out of

  - Tangles: after plastic deformation very small grain forms (cells) that are surrounded by tangles of dislocations

- Local disorder: 1-D defects (ex: impurities, vacancies)

Which pinning sites are responsible for flux trapping in Nb SRF cavities?

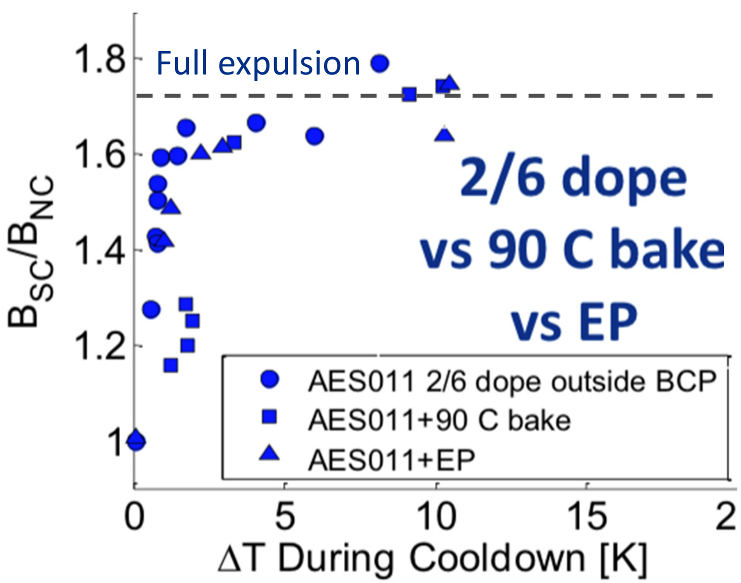
Fermilab

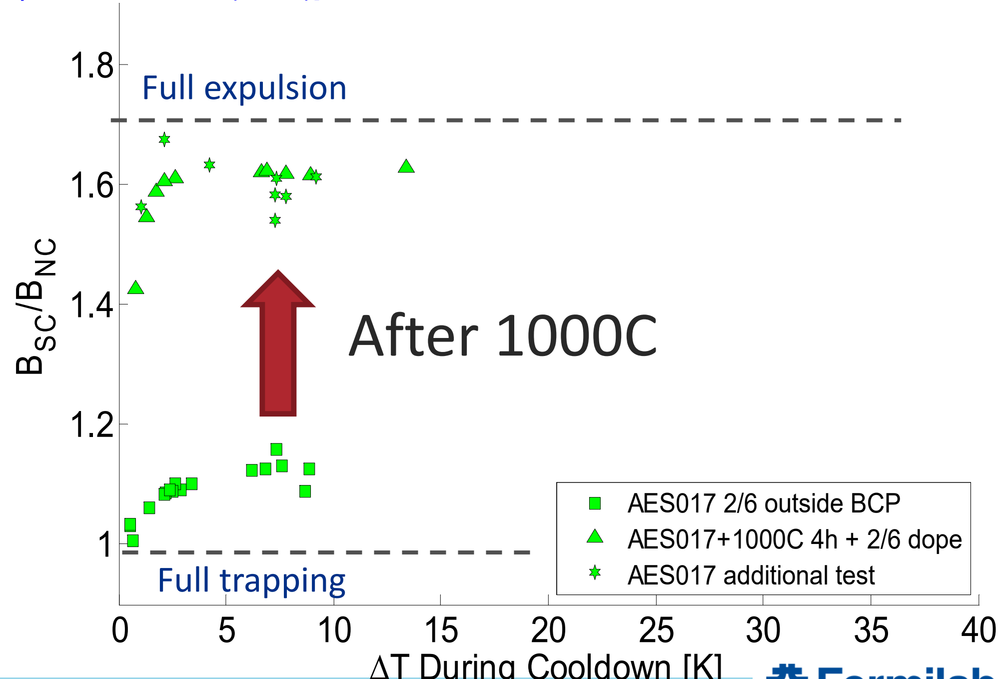
Outline

- Introduction on pinning
- **Insights from flux expulsion studies in Nb cavities**
- Microscopy studies of Nb "as-received" materials with different flux expulsion properties
- Microscopy studies of cavity cut-outs with different flux expulsion properties
- Conclusions

Facts about flux expulsion

- Flux expulsion in Nb cavities is promoted in presence of large thermal-gradient (fast cool-down) during SC transition [A. Romanenko et al., Appl. Phys. Lett. **105**, 234103 (2014); A. Romanenko et al., J. Appl. Phys. **115**, 184903 (2014)]
- Flux expulsion is a **bulk property** → does not depend on surface treatment [S. Posen et al., J. Appl. Phys. **119**, 213903 (2016)]
- Not all materials show good flux expulsion, even with large thermal gradient during the SC transition → high T treatments allow to improve materials flux expulsion properties [S. Posen et al., J. Appl. Phys. **119**, 213903 (2016)]

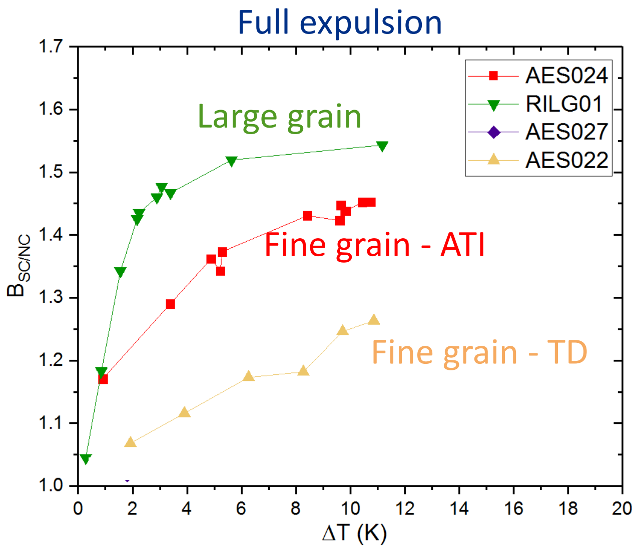




Considerations on cavities with different level of flux expulsion

Flux expulsion studies in Nb cavities:

- All data shown after baking at 800C for 3h



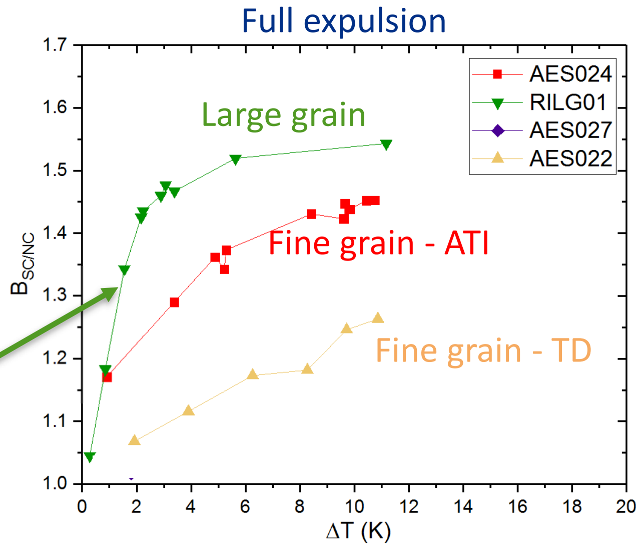
Considerations on cavities with different level of flux expulsion

Flux expulsion studies in Nb cavities:

- All data shown after baking at 800C for 3h

Large grain cavity **do not reach full flux expulsion**

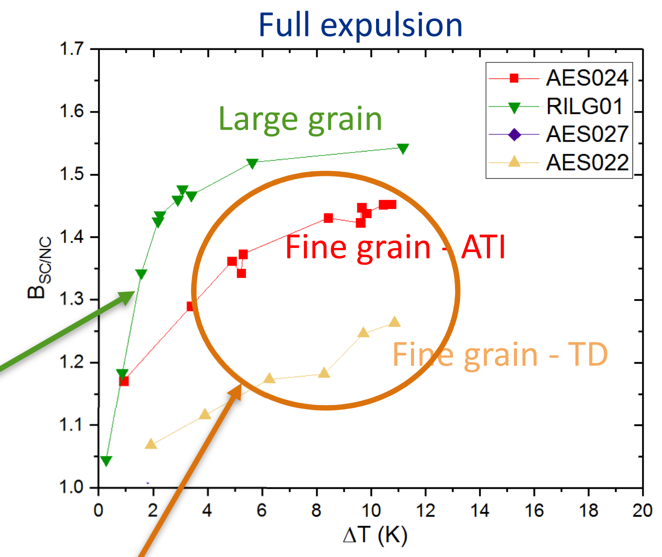
➢ GBs cannot be the only flux trapping sites in Nb cavities



Considerations on cavities with different level of flux expulsion

Flux expulsion studies in Nb cavities:

- All data shown after baking at 800C for 3h



Large grain cavity **do not reach full flux expulsion**

- ➢ GBs cannot be the only flux trapping sites in Nb cavities

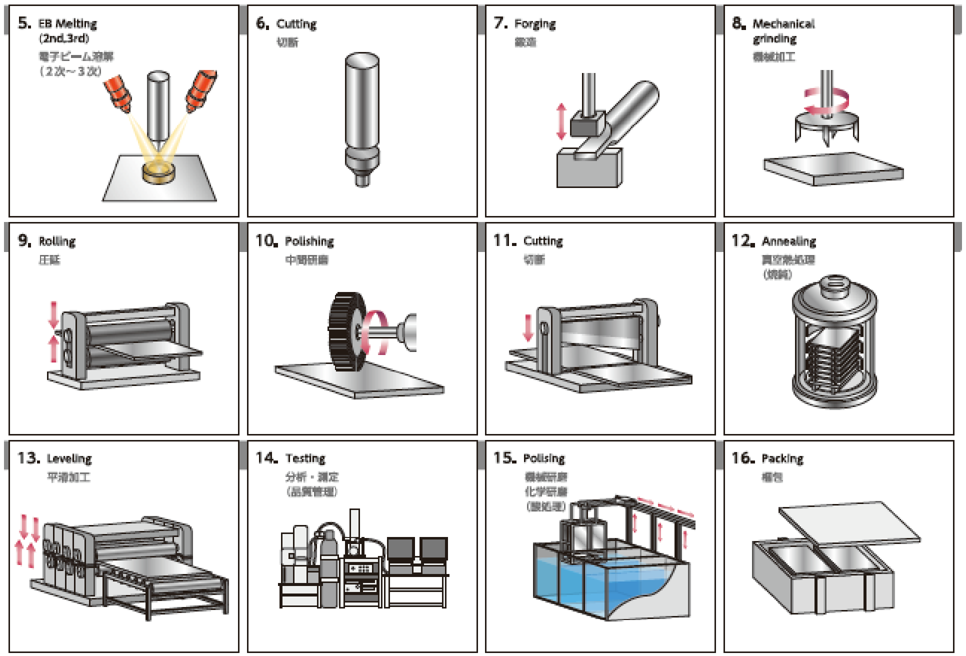
Cavities made out with materials coming from different Nb vendors show different flux expulsion properties

Outline

- Introduction on pinning
- Insights from flux expulsion studies in Nb cavities
- **Microscopy studies of Nb "as-received" materials with different flux expulsion properties**
- Microscopy studies of cavity cut-outs with different flux expulsion properties
- Conclusions

Overview Nb cavity sheet fabrication

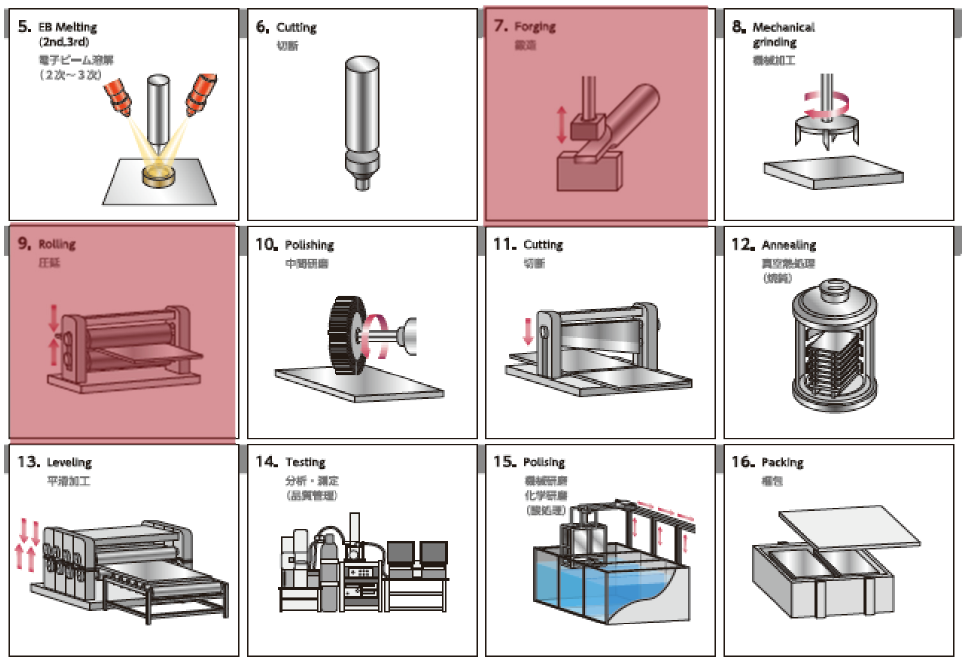
At Nb vendors



From Hiroaki Umezawa, CTO
Tokyo Denkai Co., Ltd. – TTC 2017 MSU

Overview Nb cavity sheet fabrication

At Nb vendors

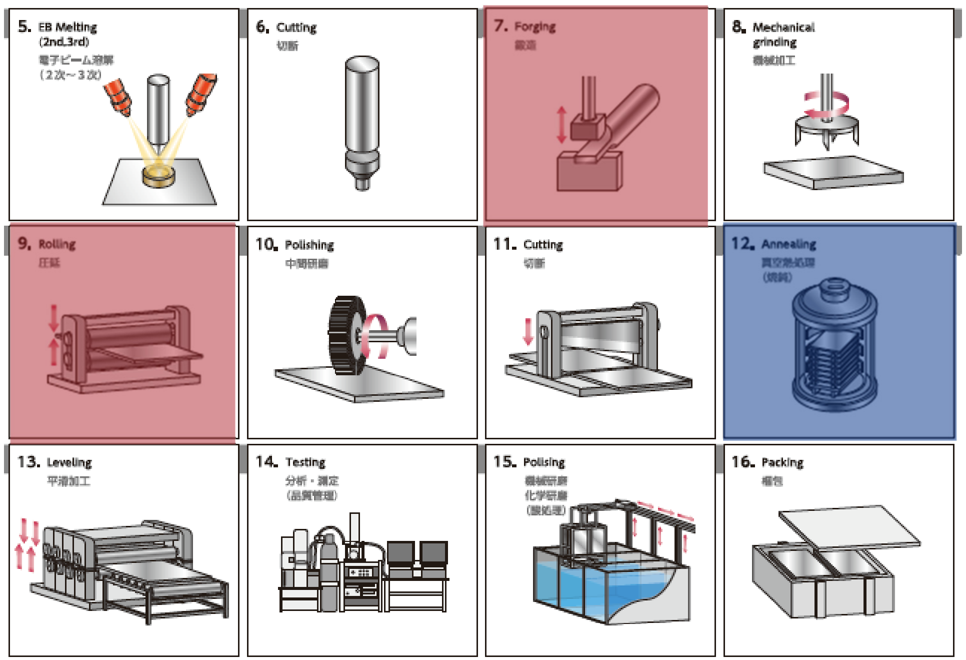


Cold work → introduces dislocations and dislocations tangles

From Hiroaki Umezawa, CTO
Tokyo Denkai Co., Ltd. – TTC 2017 MSU

Overview Nb cavity sheet fabrication

At Nb vendors



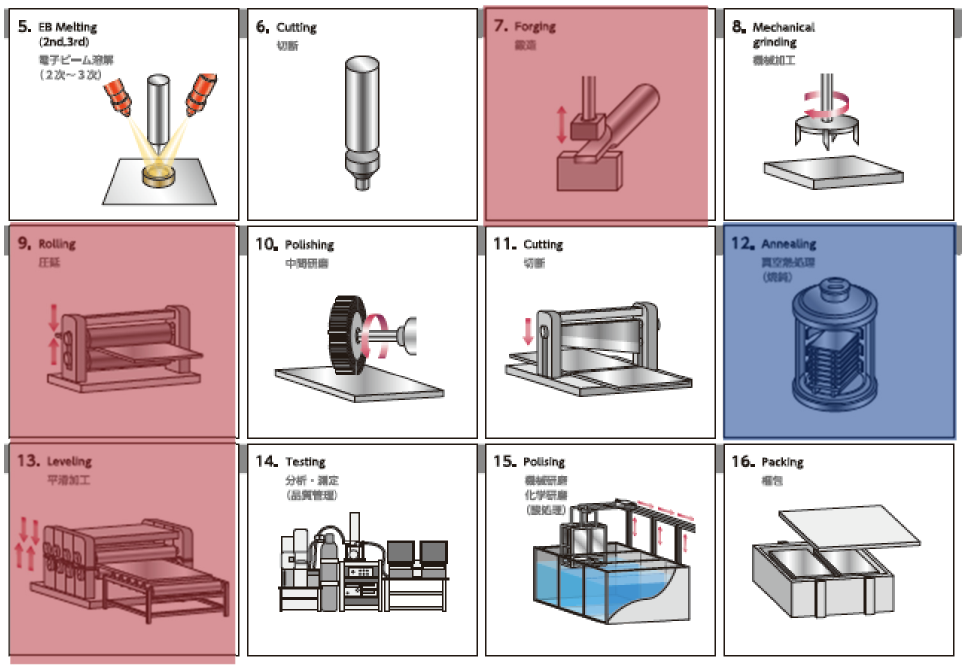
From Hiroaki Umezawa, CTO
Tokyo Denkai Co., Ltd. – TTC 2017 MSU

Cold work → introduces dislocations and dislocations tangles

Annealing → re-crystallization (nucleation of new grains), followed by grain growth

Overview Nb cavity sheet fabrication

At Nb vendors



From Hiroaki Umezawa, CTO
Tokyo Denkai Co., Ltd. – TTC 2017 MSU

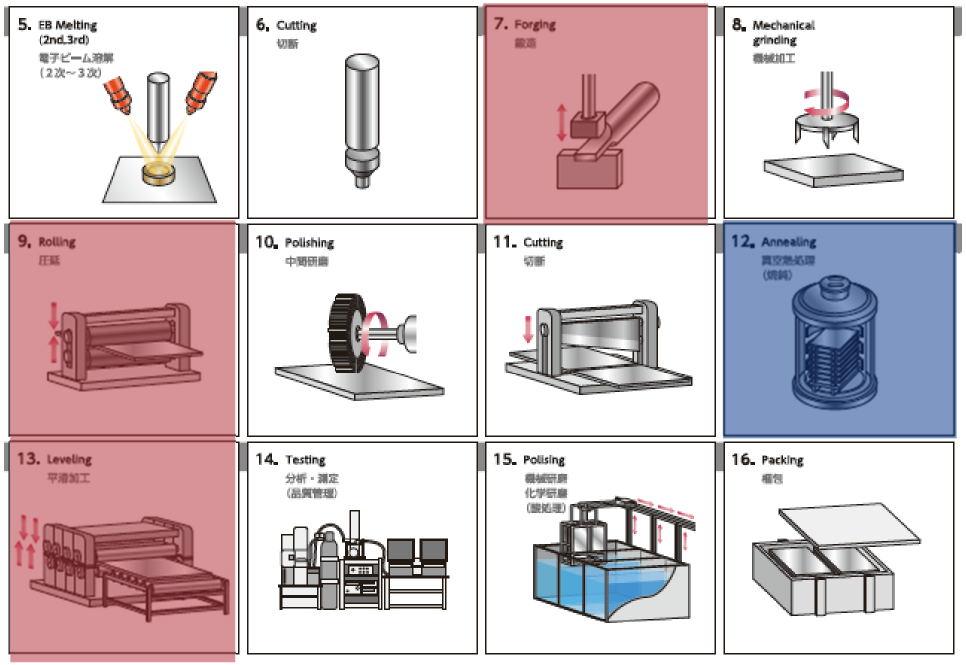
Cold work → introduces dislocations and dislocations tangles

Annealing → re-crystallization (nucleation of new grains), followed by grain growth

Additional cold work → introduces again dislocations and dislocations tangles

Overview Nb cavity sheet fabrication

At Nb vendors



From Hiroaki Umezawa, CTO
Tokyo Denkai Co., Ltd. – TTC 2017 MSU

Cold work → introduces dislocations and dislocations tangles

Annealing → re-crystallization (nucleation of new grains), followed by grain growth

Additional cold work → introduces again dislocations and dislocations tangles

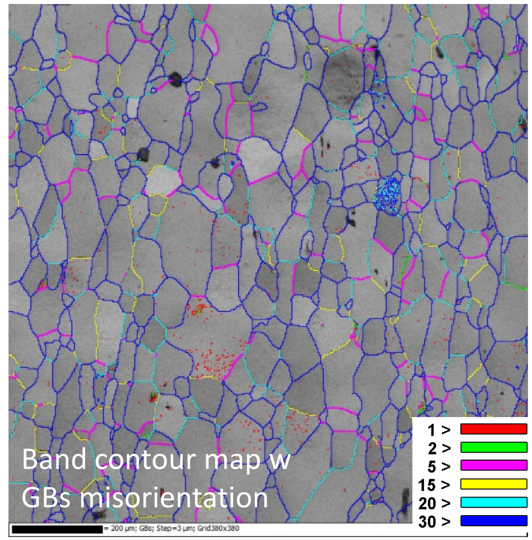
"as received" material

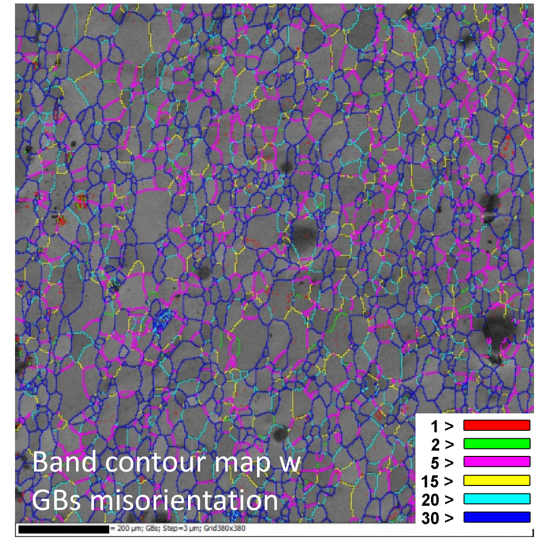
Analysis of “as received” materials (cross-section)

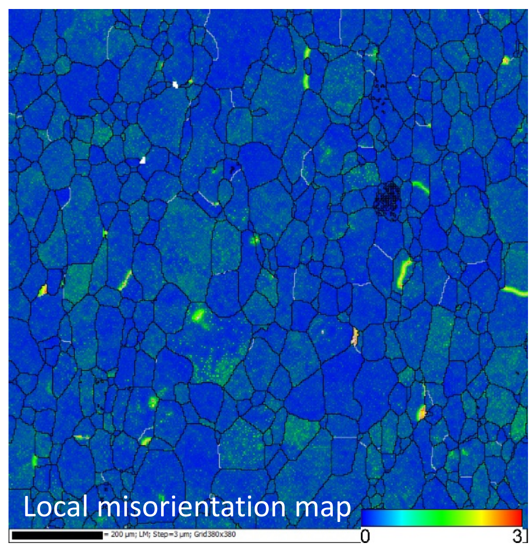
- Material that shows good flux expulsion properties (after annealing at 900C) has larger grain in the “as received” condition

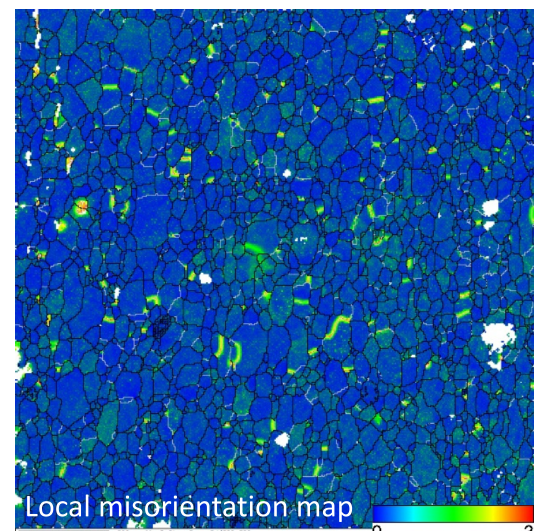
ATI -good flux expulsion-

Ningxia -bad flux expulsion-









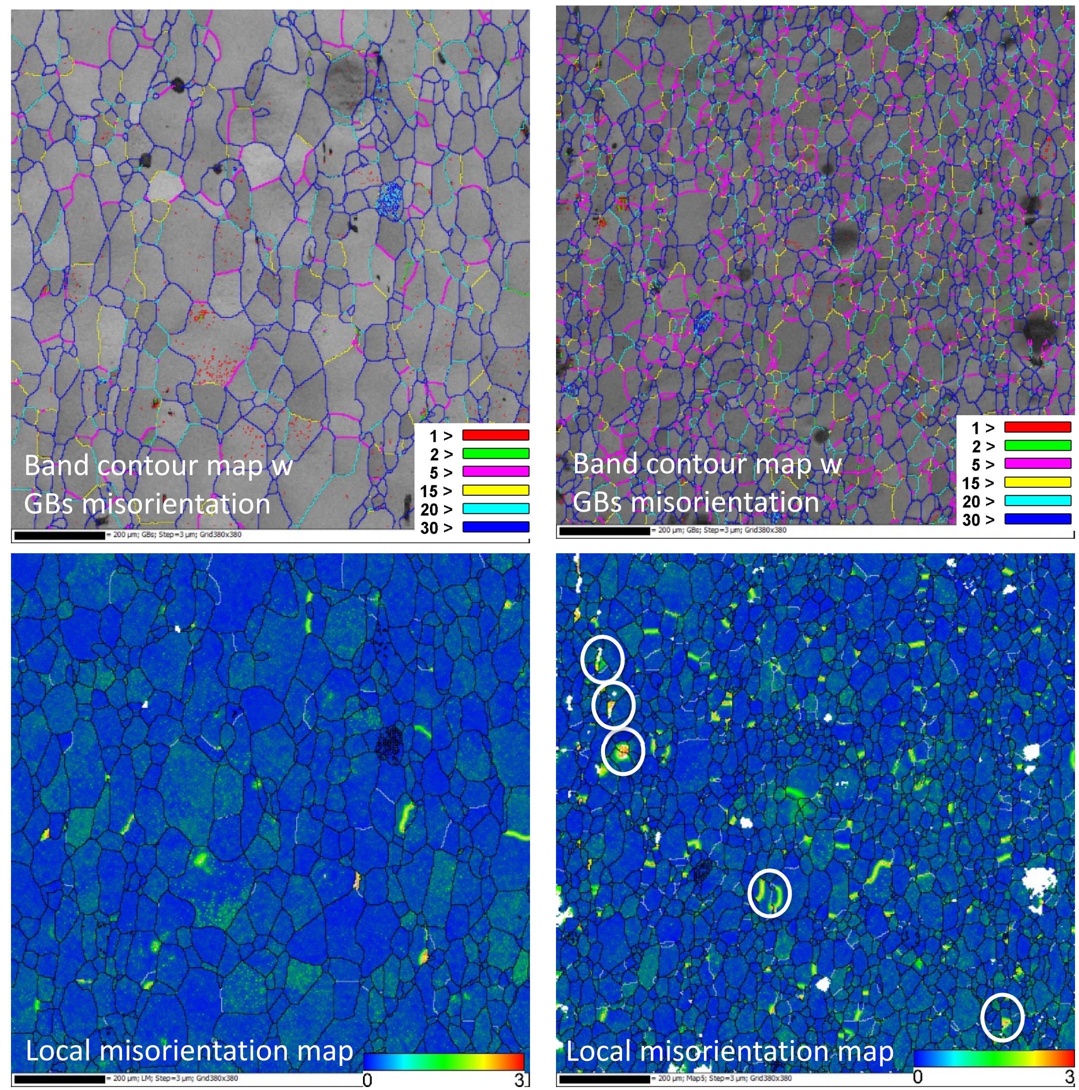
Analysis of “as received” materials (cross-section)

- Material that shows good flux expulsion properties (after annealing at 900C) has larger grain in the “as received” condition

- Material with bad flux expulsion properties shows small grains, many with high level of local misorientation

ATI -good flux expulsion-

Ningxia -bad flux expulsion-

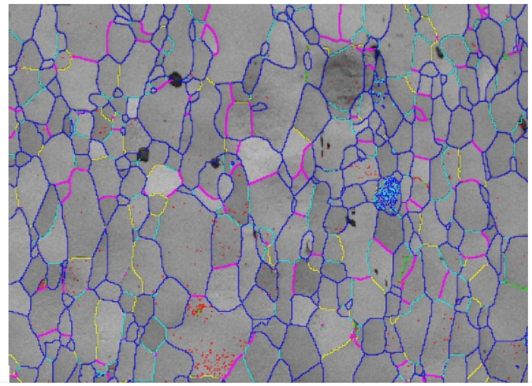


Analysis of “as received” materials (cross-section)

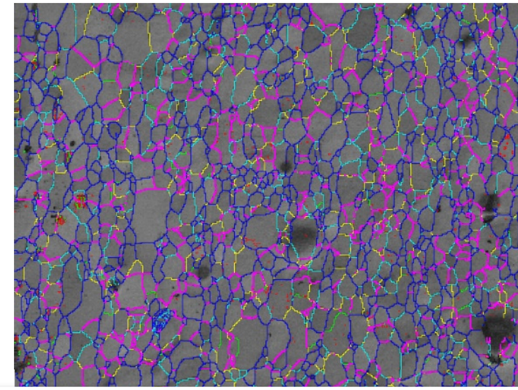
- Material that shows good flux expulsion properties (after annealing at 900C) has larger grain in the “as received” condition

- Material with bad flux

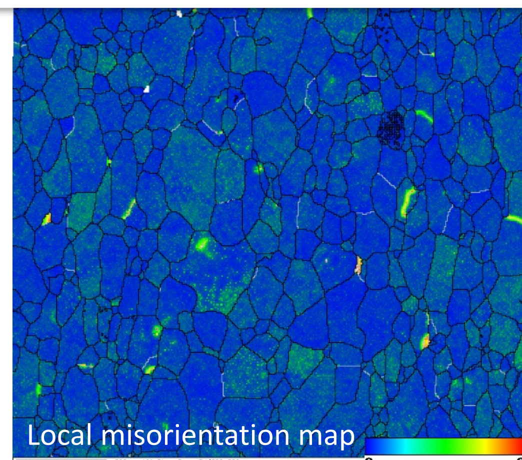
ATI -good flux expulsion-

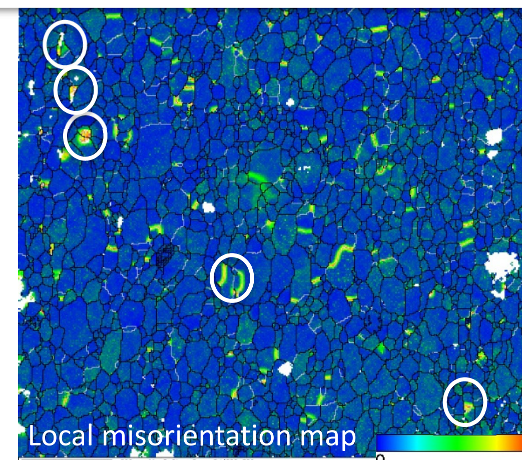


Ningxia -bad flux expulsion-



Which types of defects are present in grains with high level of local misorientation?



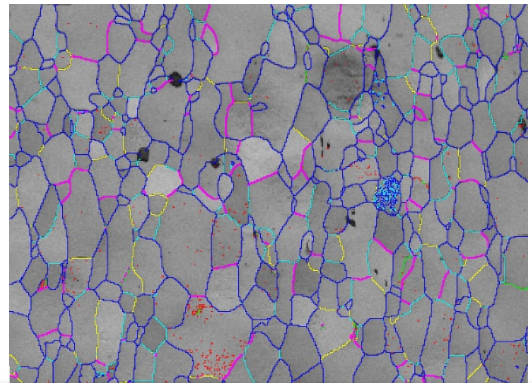


Analysis of “as received” materials (cross-section)

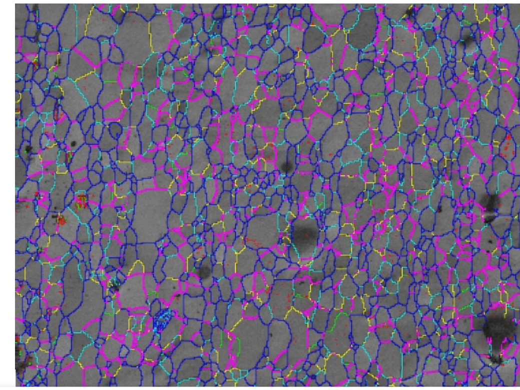
- Material that shows good flux expulsion properties (after annealing at 900C) has larger grain in the “as received” condition

- Material with bad flux

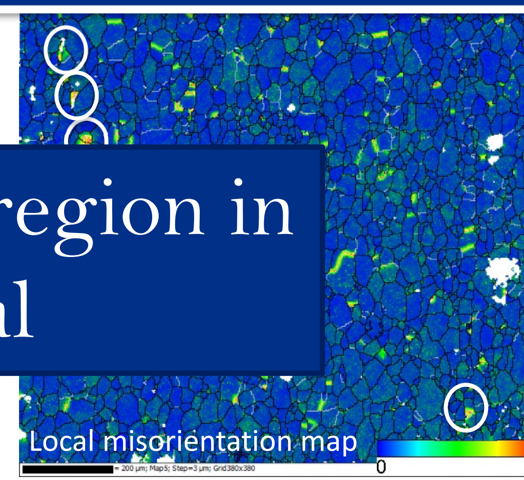
ATI -good flux expulsion-



Ningxia -bad flux expulsion-



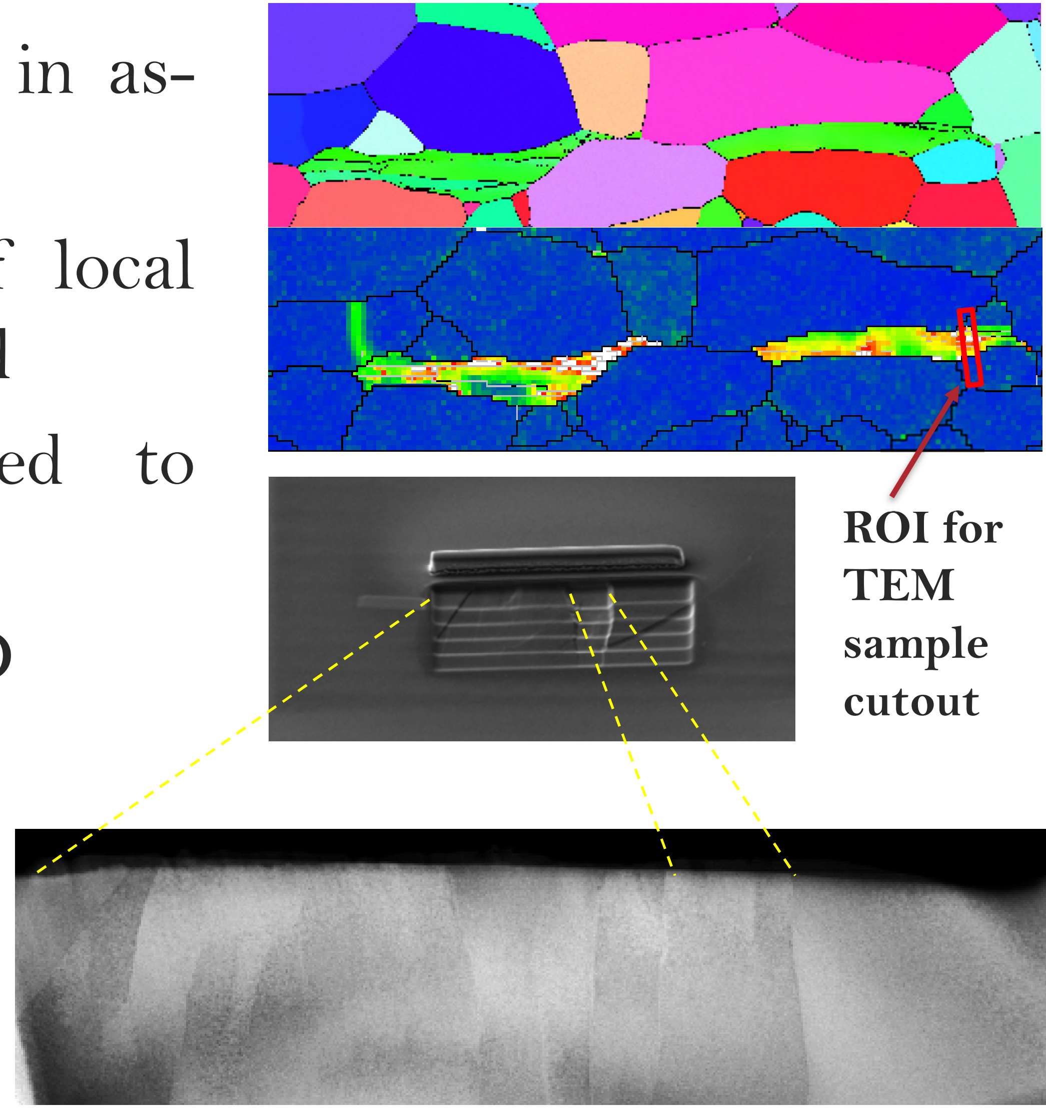
Which types of defects are present in grains with high level of local misorientation?



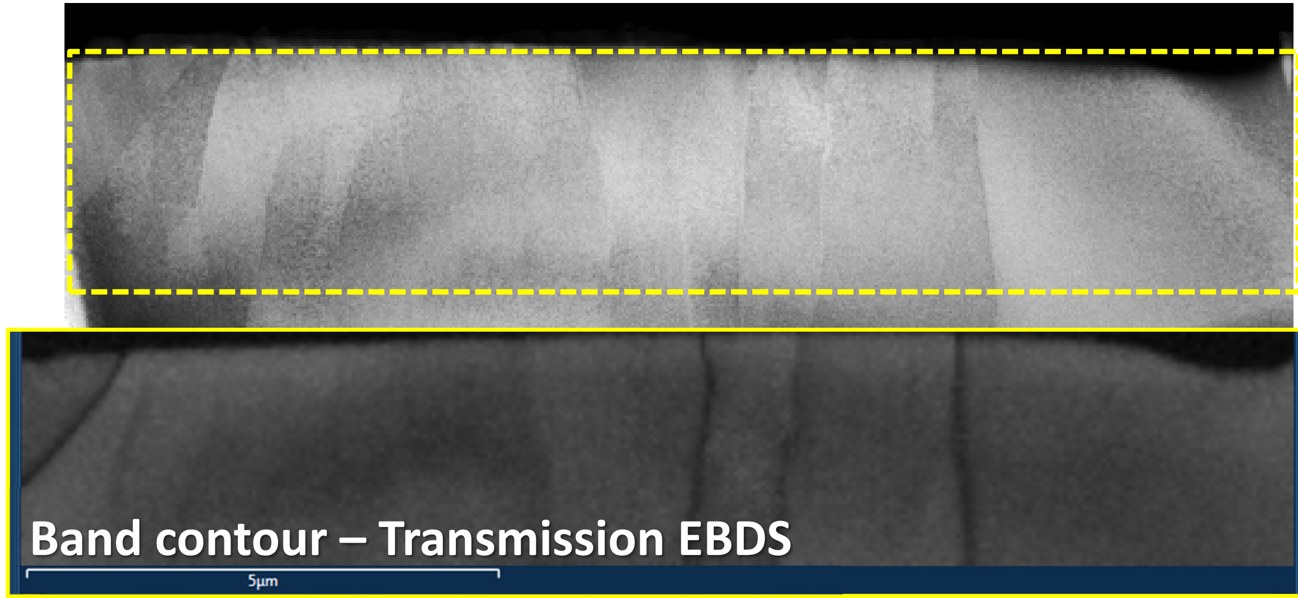
STEM studies of defective region in “as-received” material

Study of highly defective areas on "as-received" material

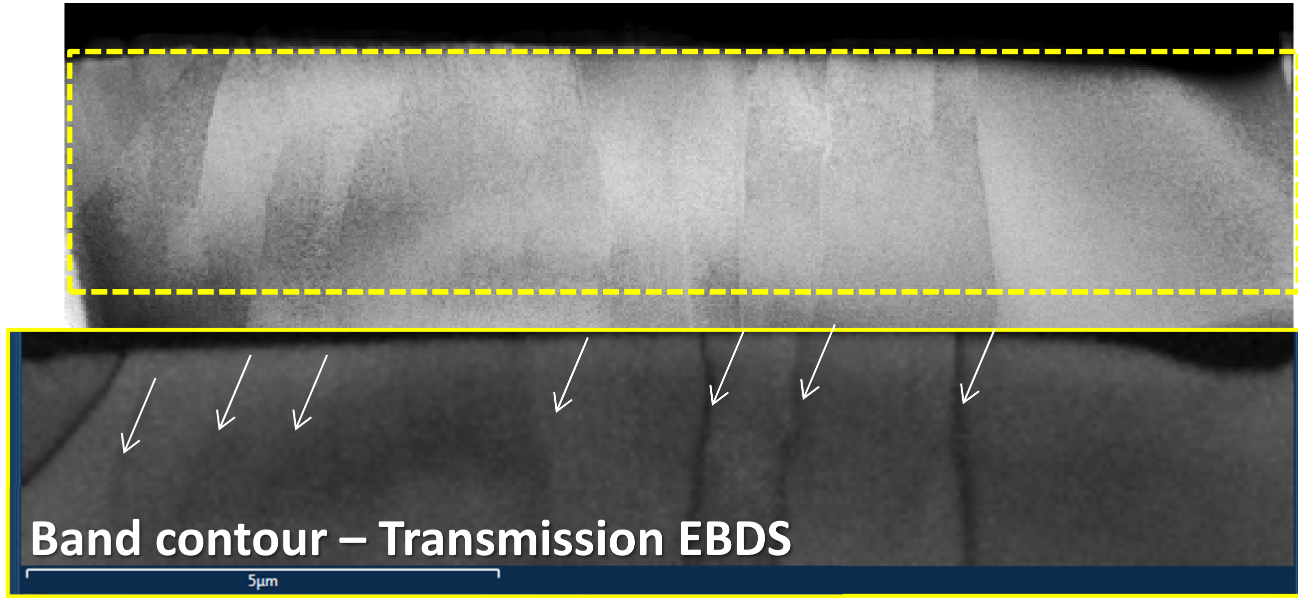
- High resolution EBSD in as-received material
- Area with high level of local misorientation identified
- TEM lamella prepared to study the area in details
    - Transmission EBSD
    - BF-STEM
    - HR-TEM



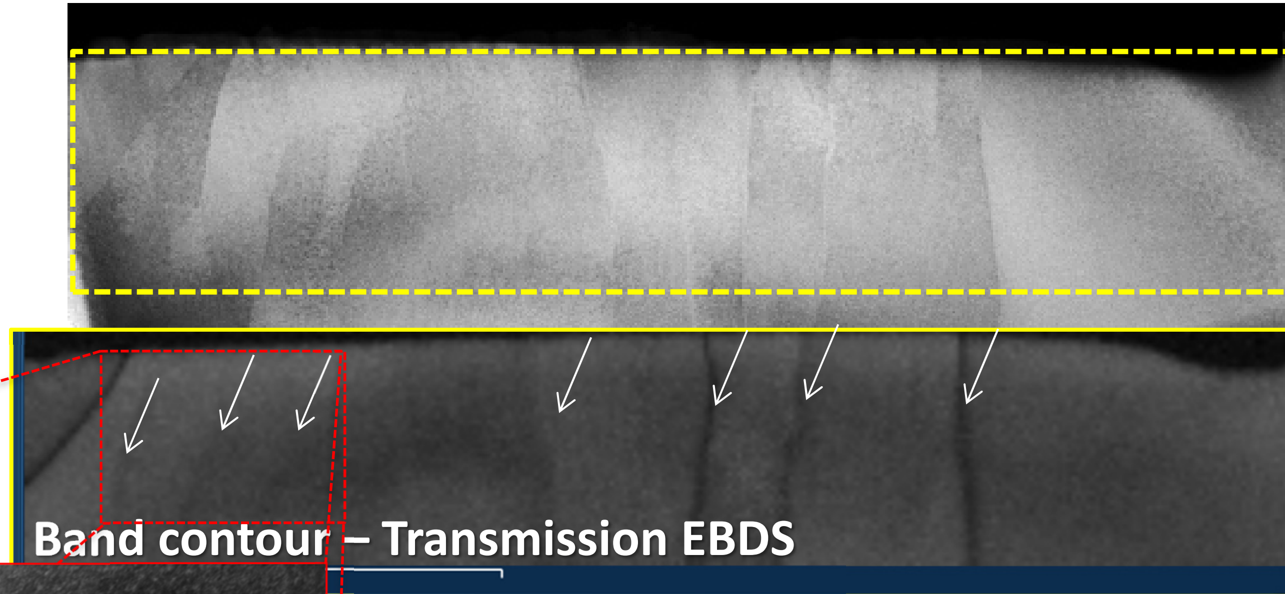
Study of highly defective areas on “as-received” material

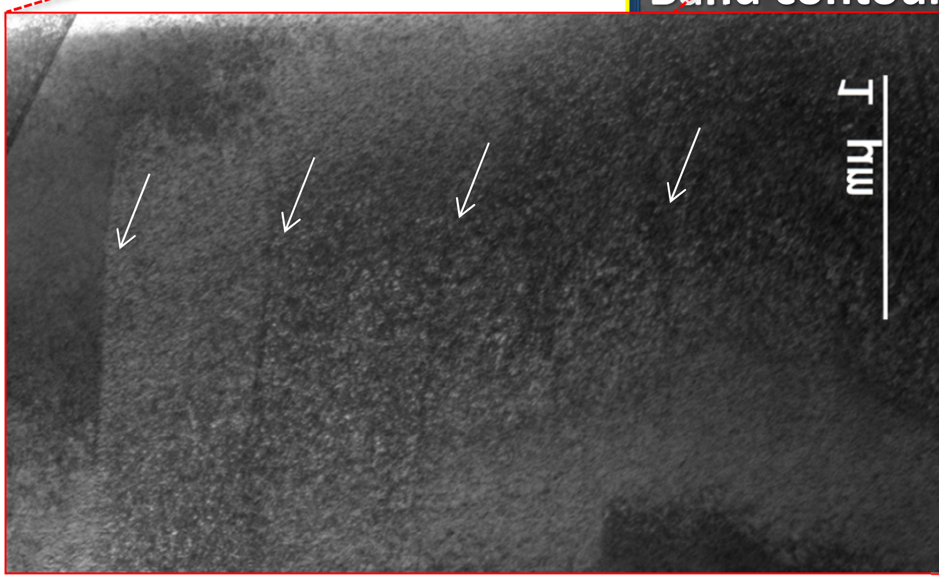


Study of highly defective areas on "as-received" material

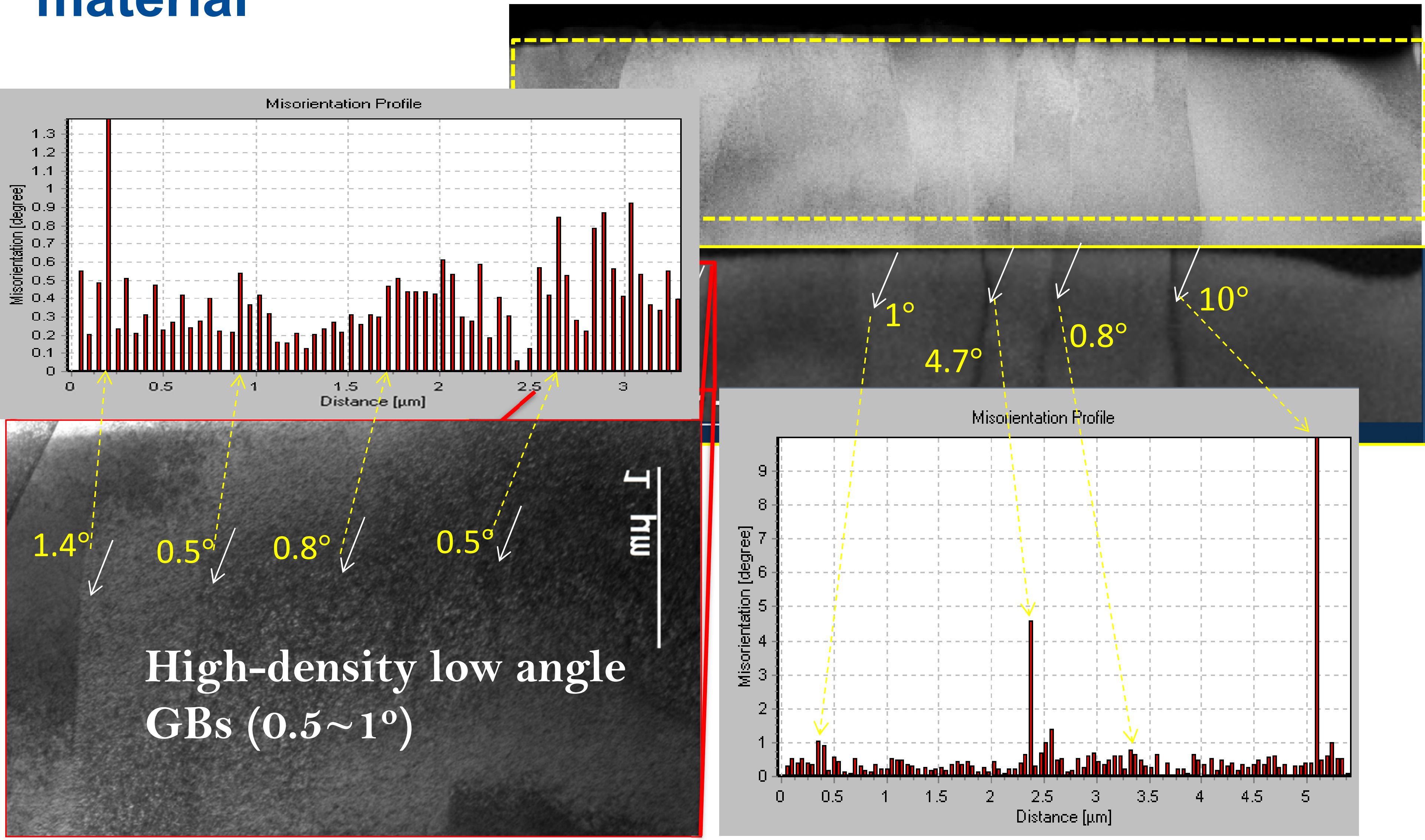


Study of highly defective areas on “as-received” material

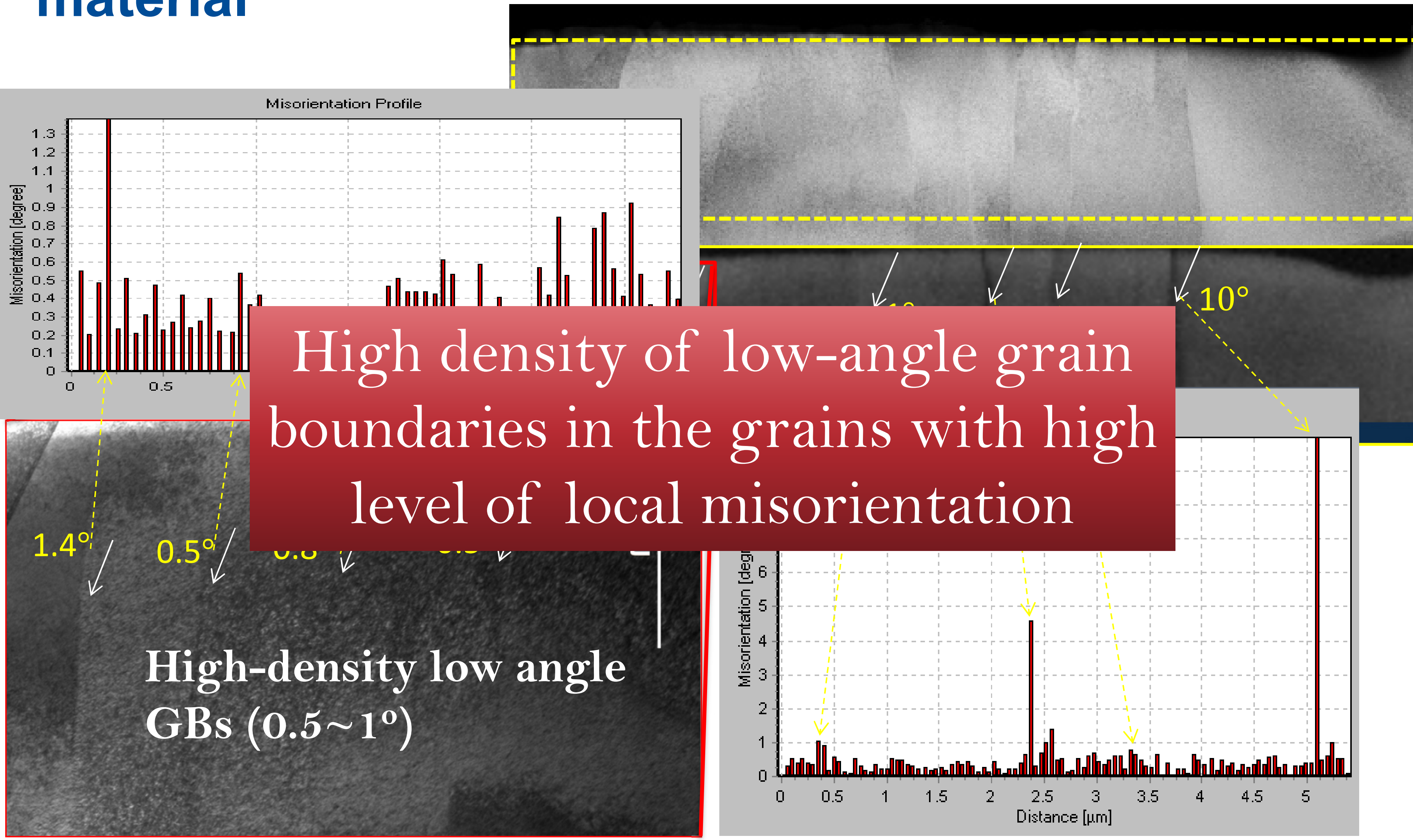




Study of highly defective areas on "as-received" material

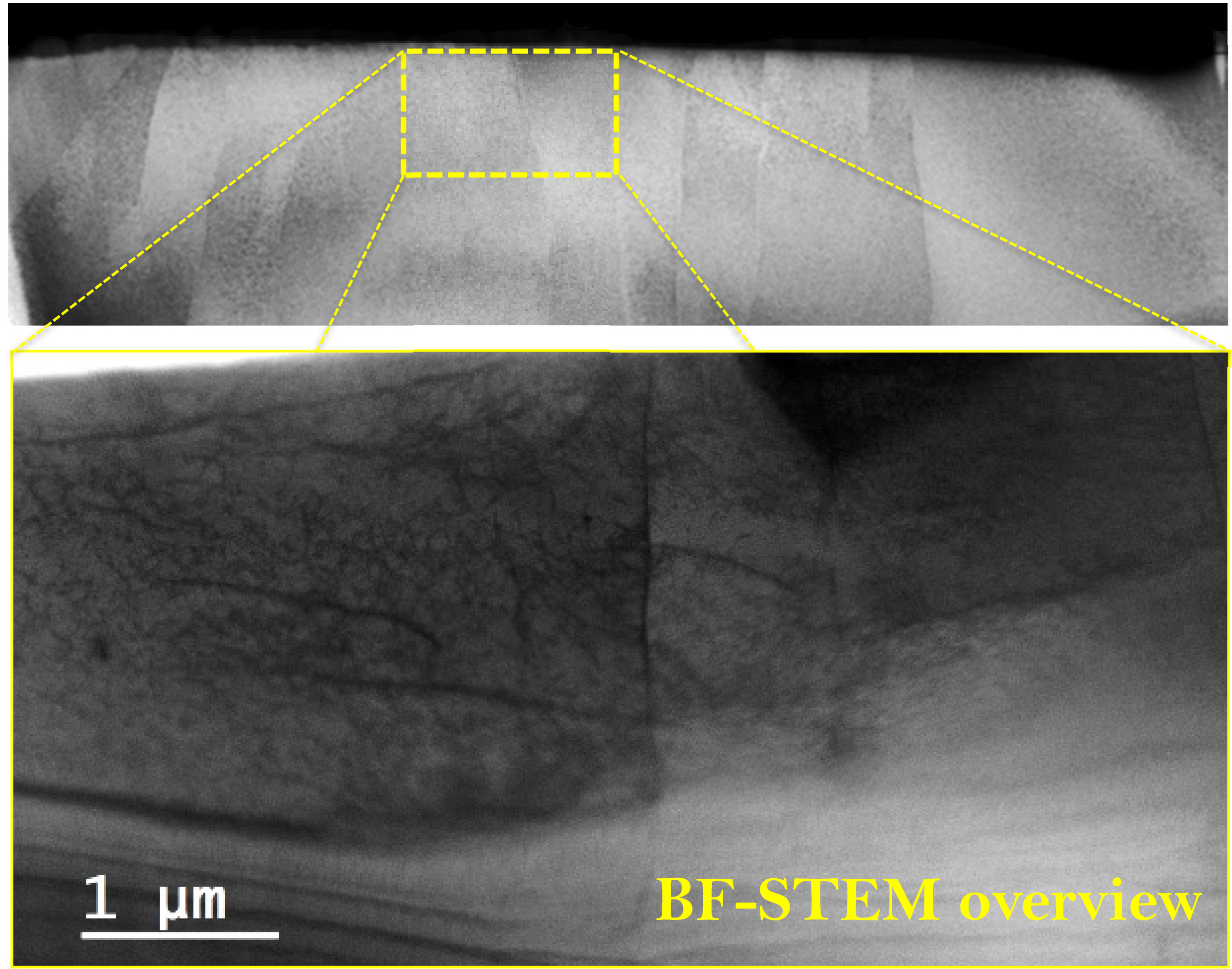


Study of highly defective areas on "as-received" material

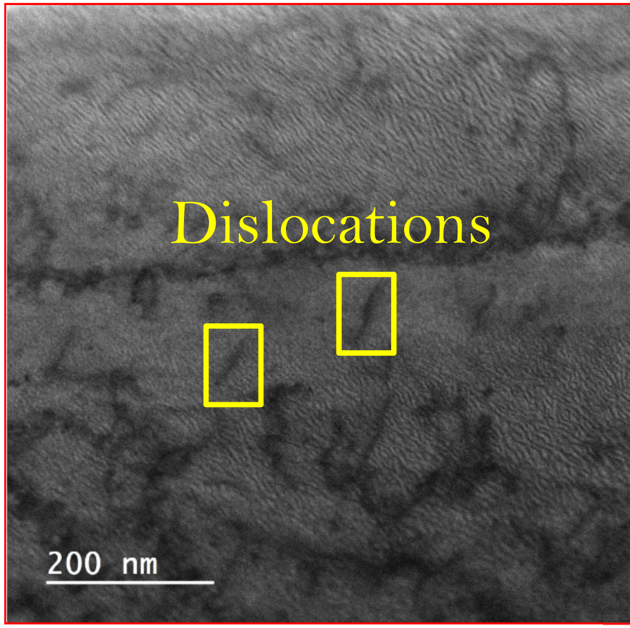


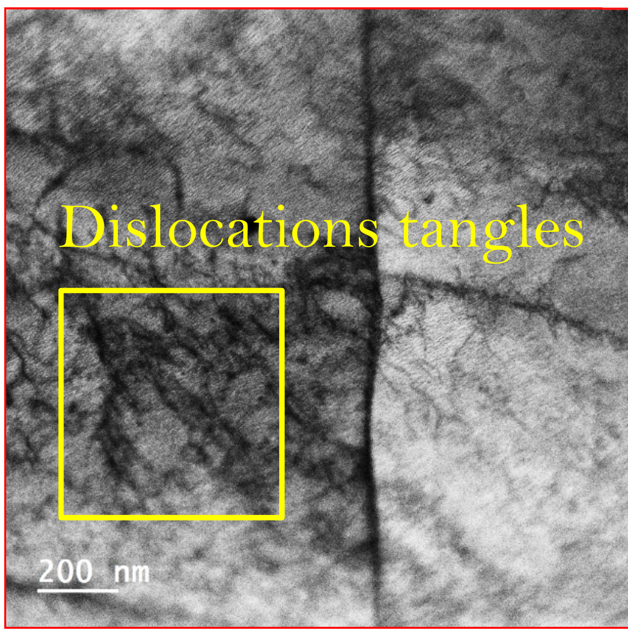
High density of low-angle grain boundaries in the grains with high level of local misorientation

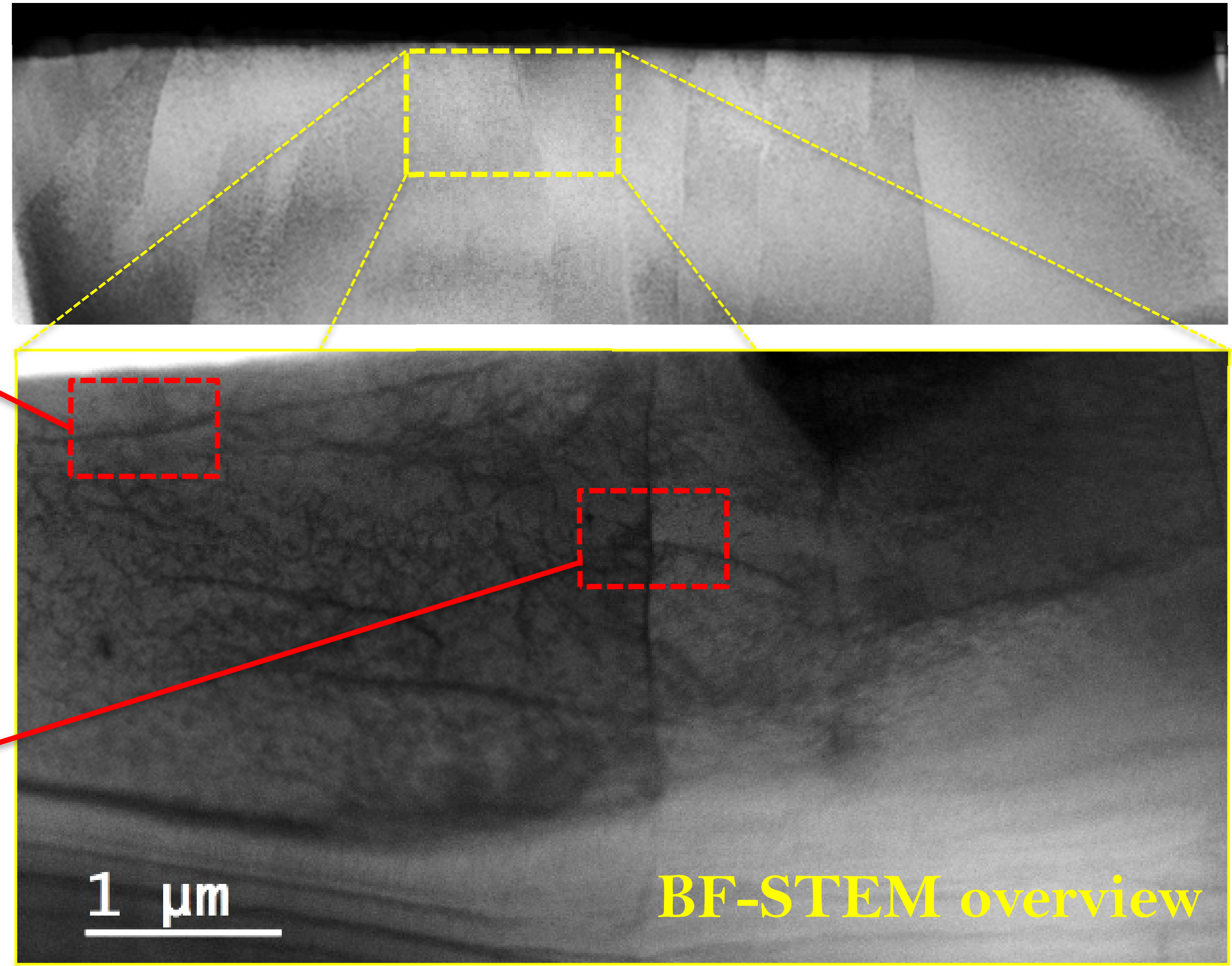
Study of highly defective areas on “as-received” material



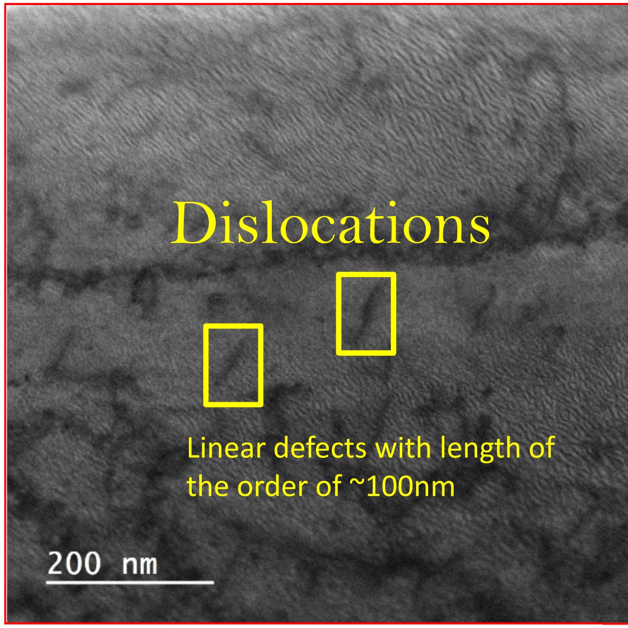
Study of highly defective areas on “as-received” material

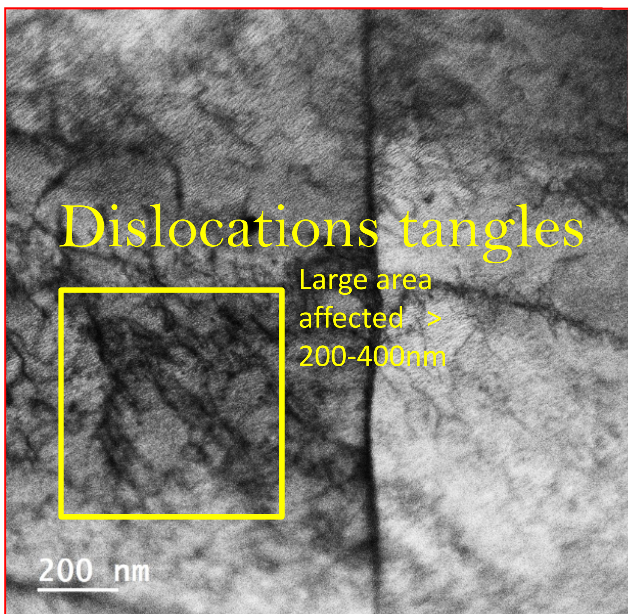


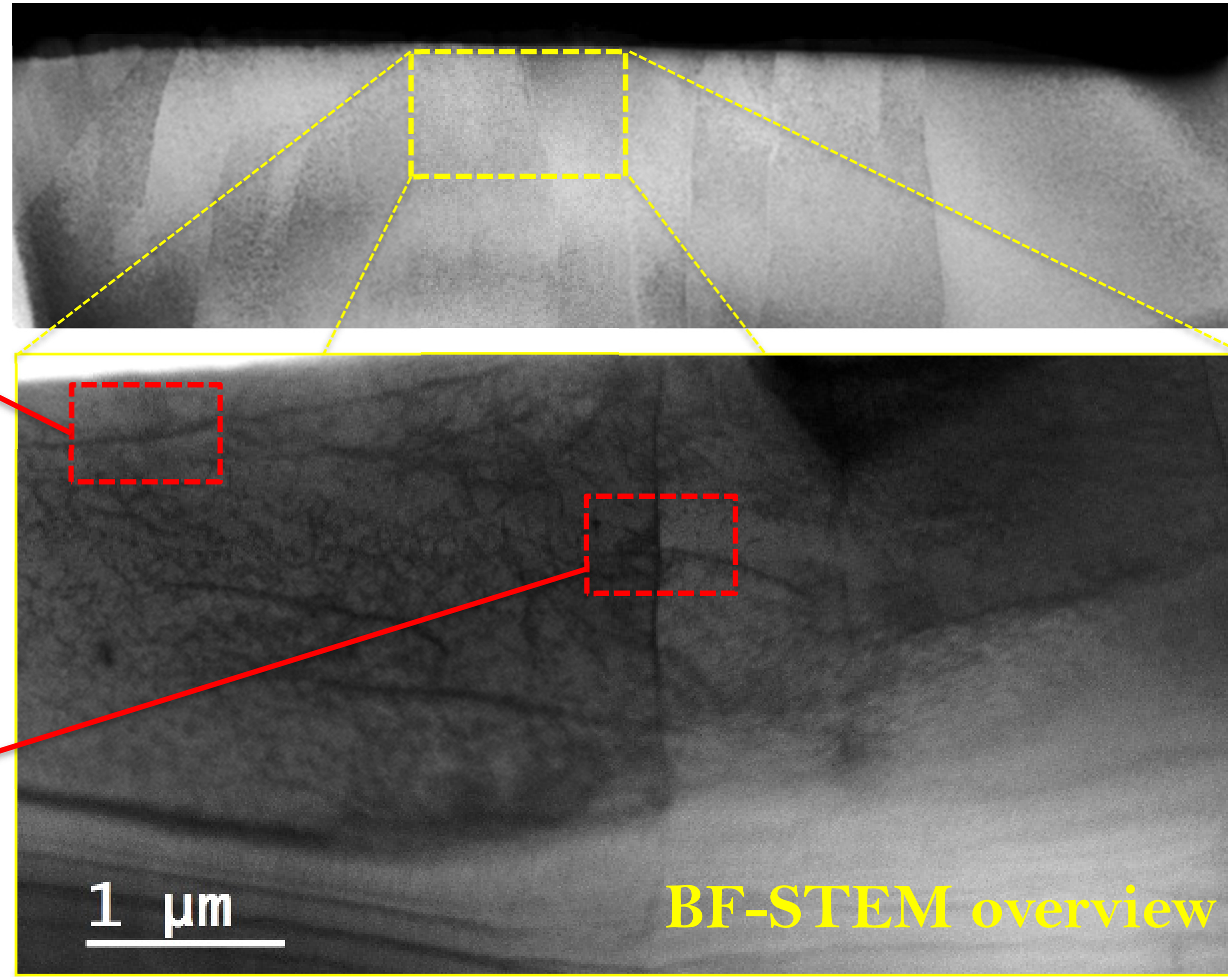




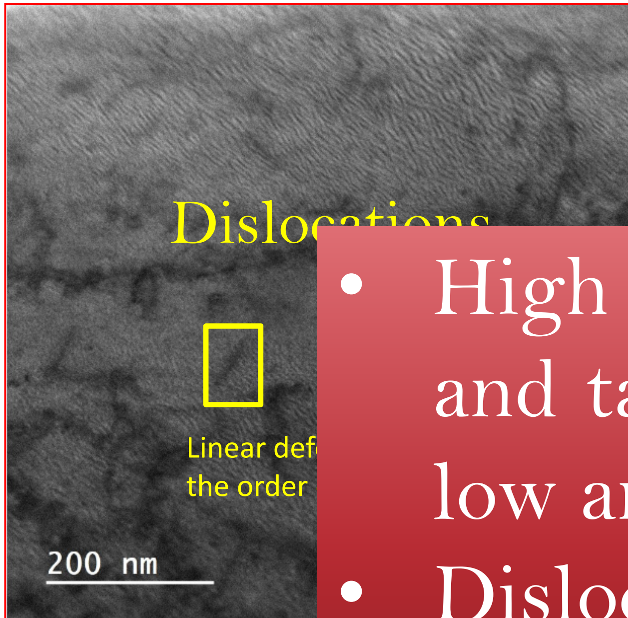
Study of highly defective areas on “as-received” material

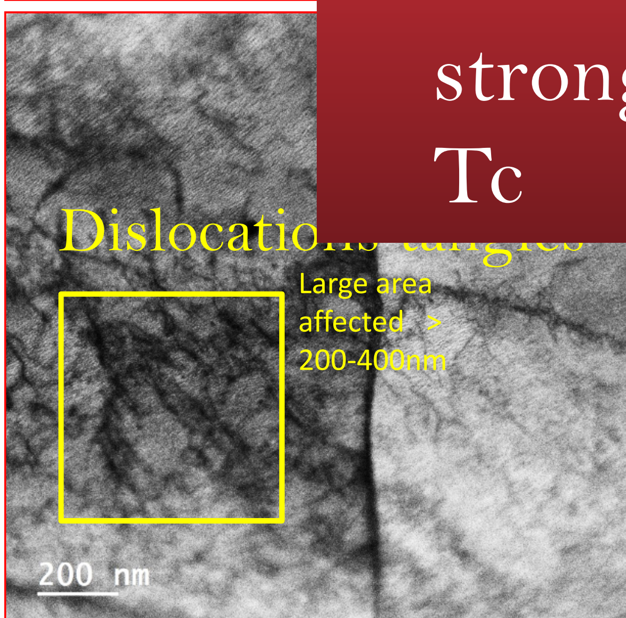


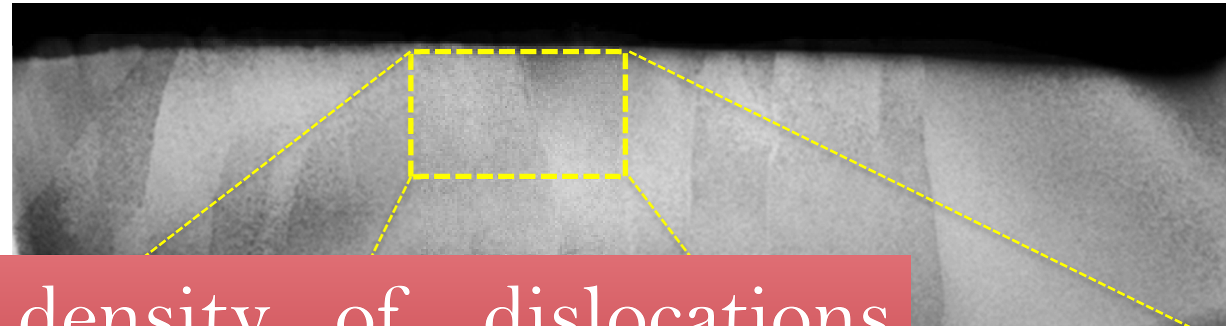


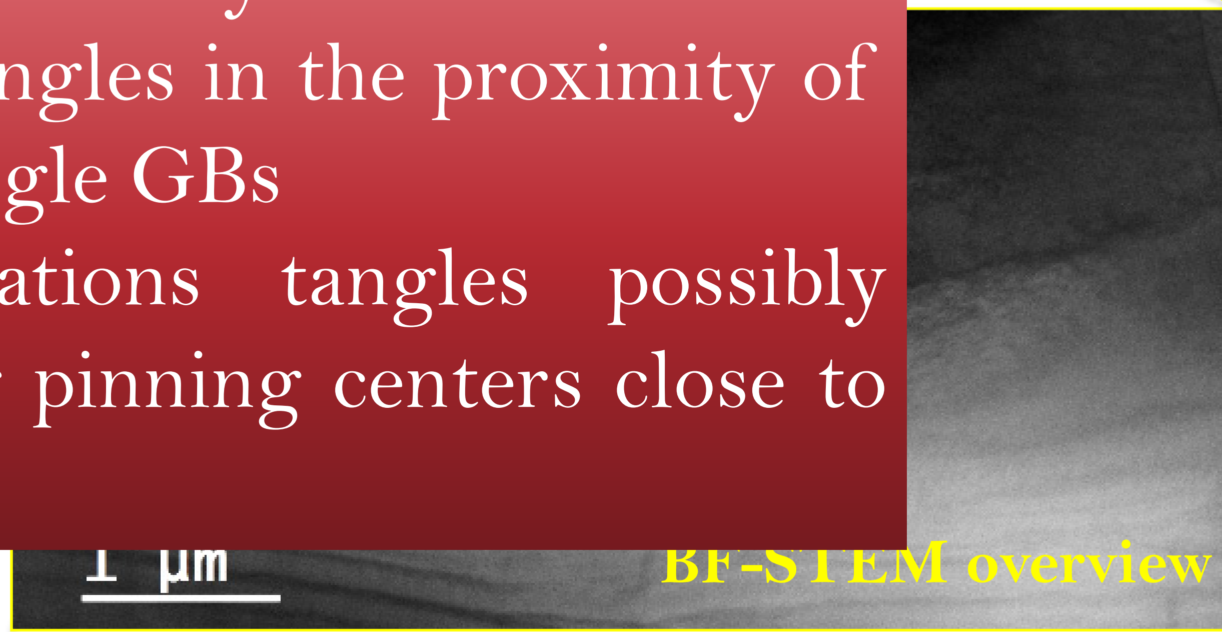


Study of highly defective areas on “as-received” material



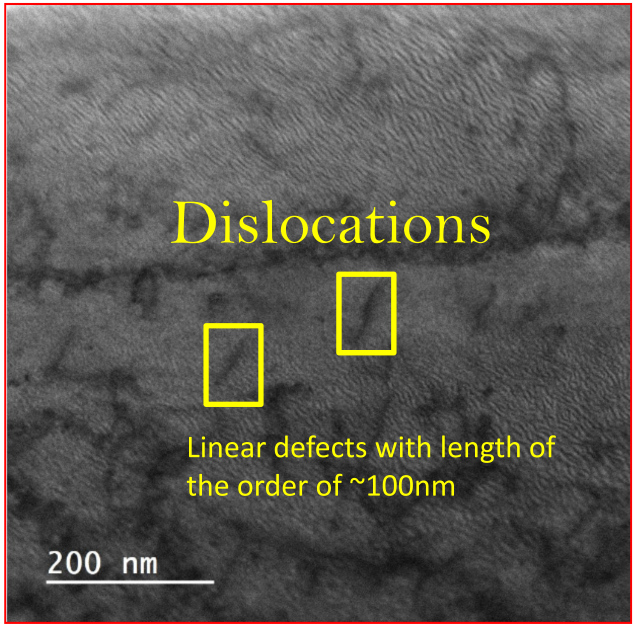


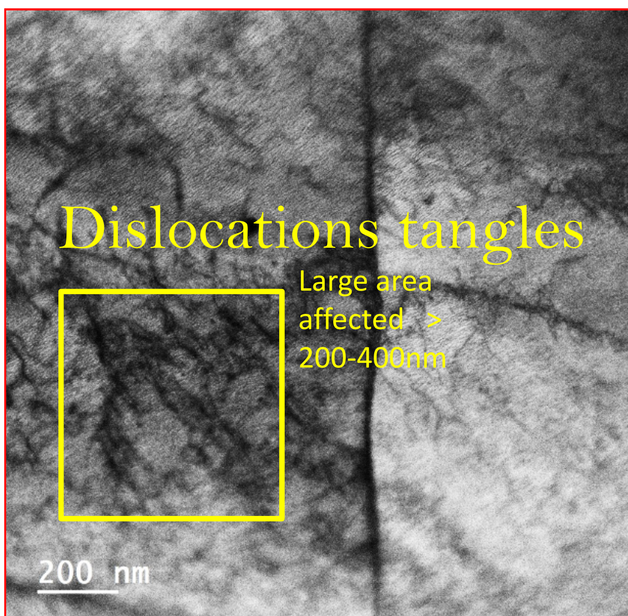


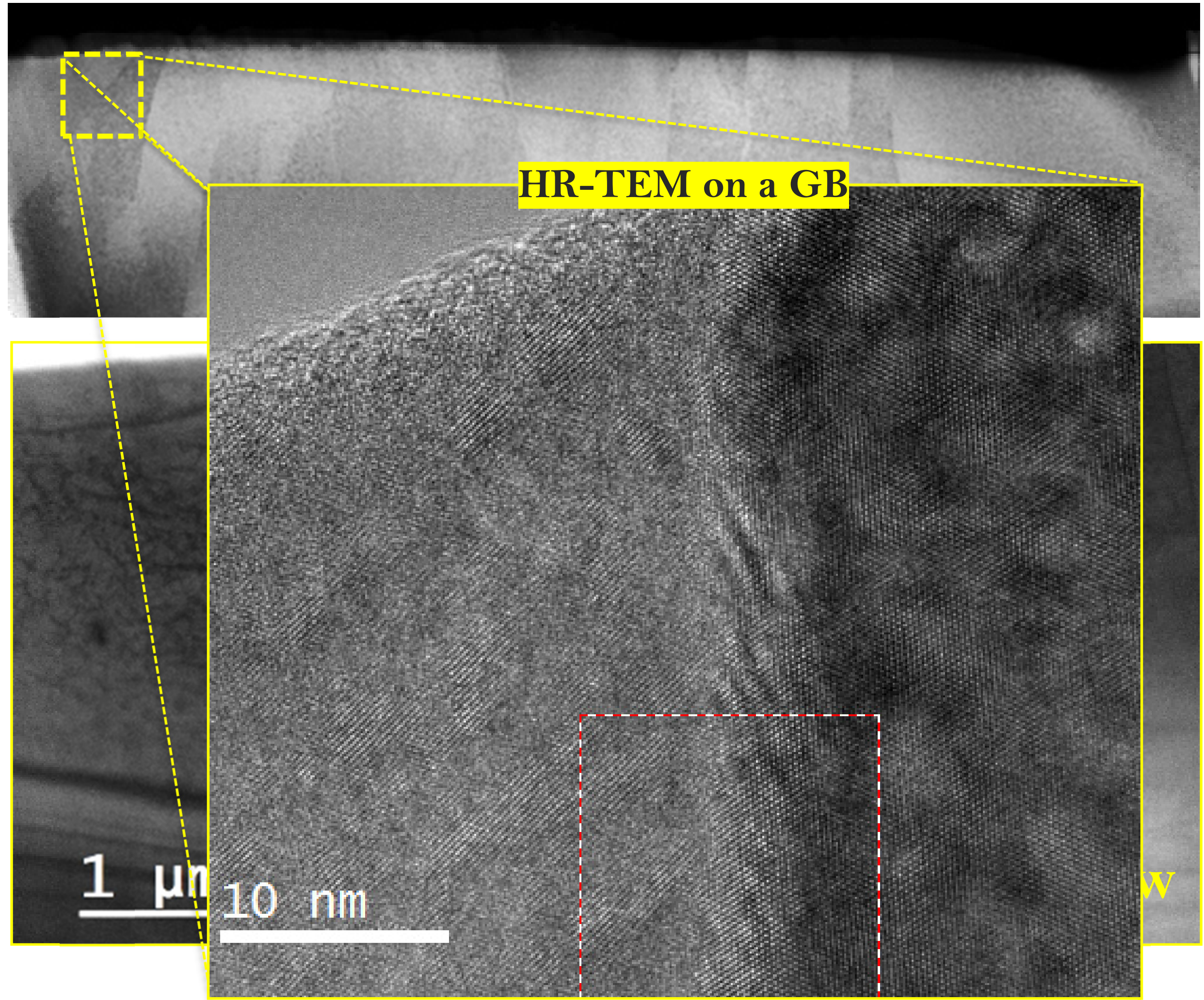


- High density of dislocations and tangles in the proximity of low angle GBs
- Dislocations tangles possibly strong pinning centers close to Tc

Study of highly defective areas on “as-received” material

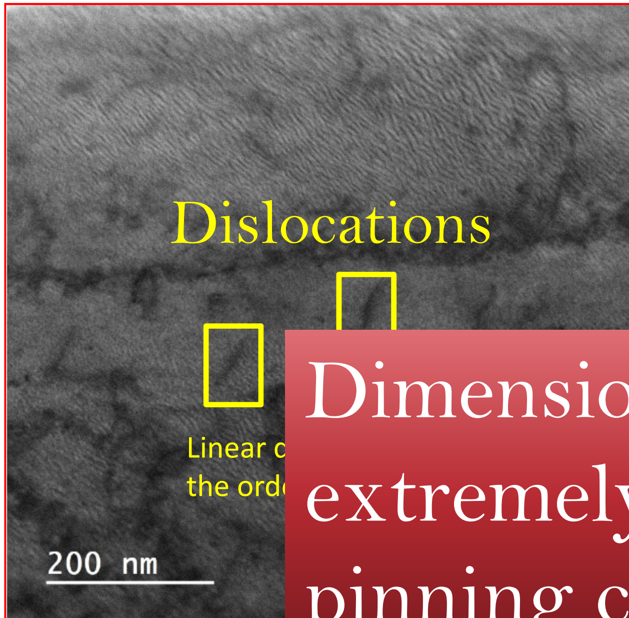


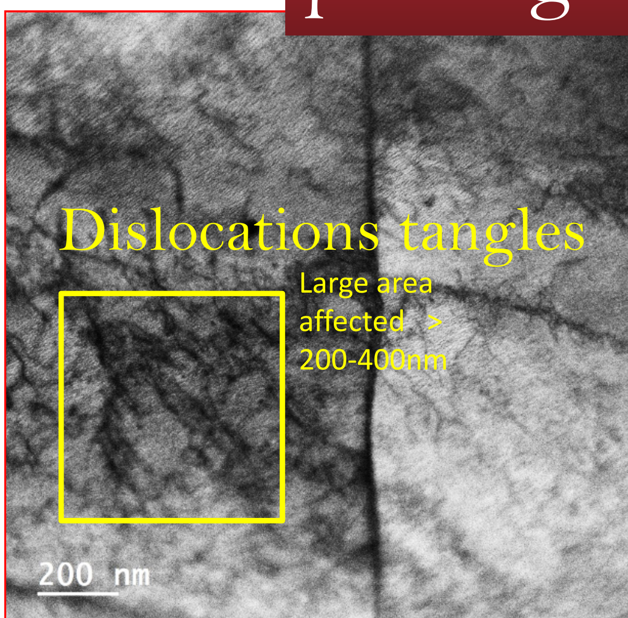


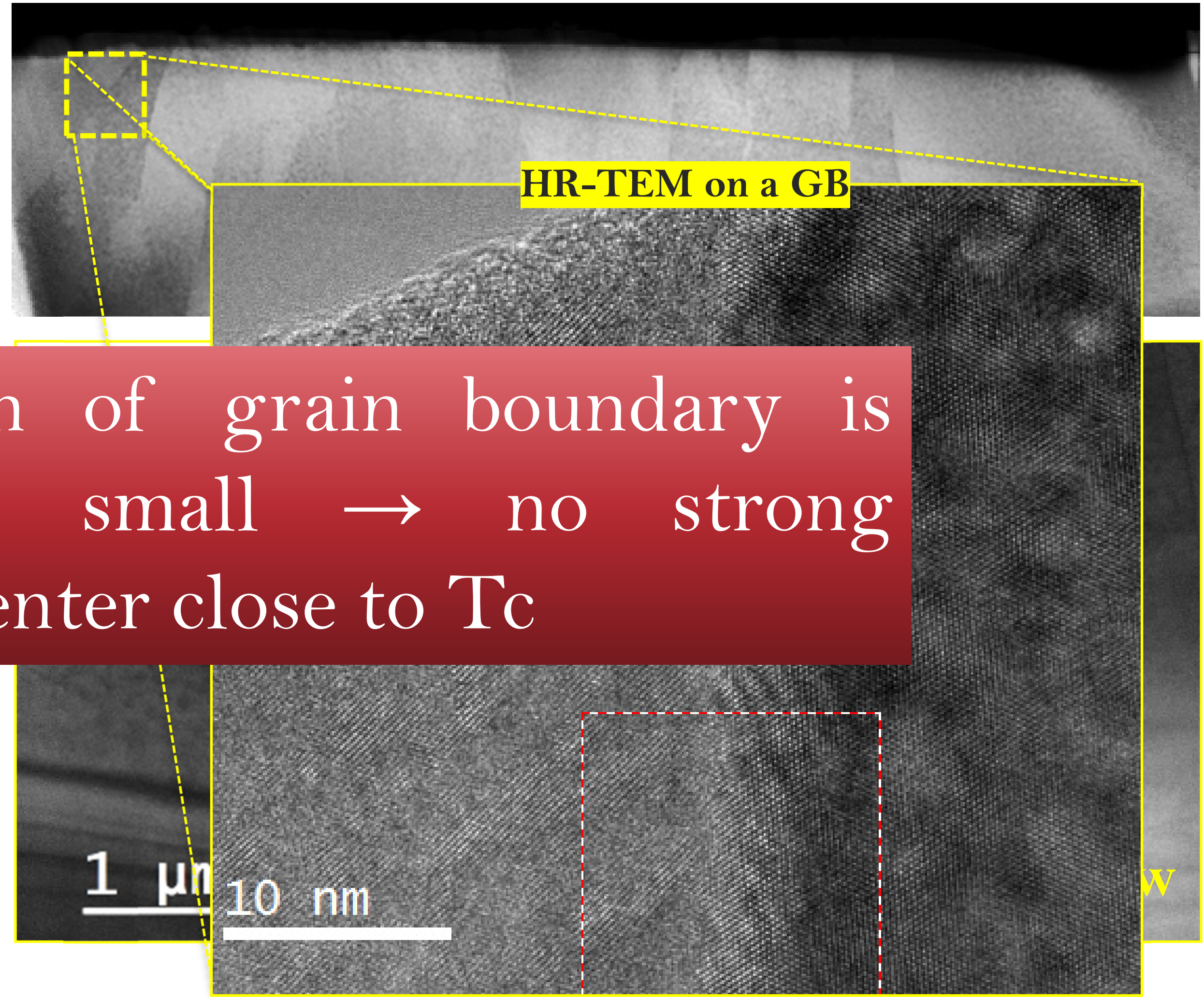


- The width of the GB is extremely small (<1 nm)

Study of highly defective areas on “as-received” material







Dimension of grain boundary is extremely small → no strong pinning center close to Tc

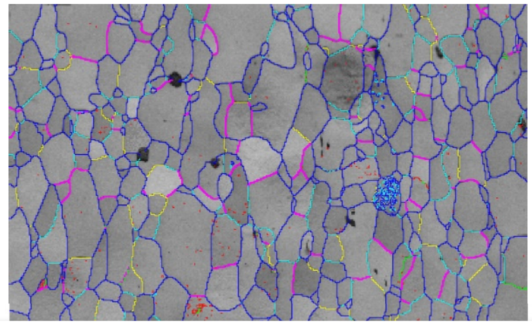
- The width of the GB is extremely small (<1 nm)

Analysis of “as received” materials (cross-section)

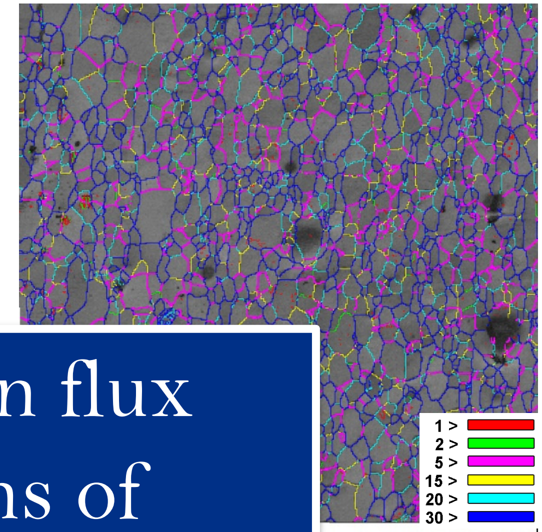
- Material that shows good flux expulsion properties (after annealing at 900C) has <u>larger grain</u> in the “as received” condition

- Material
  expulsion
  <u>small grai</u>
  <u>level of lo</u>

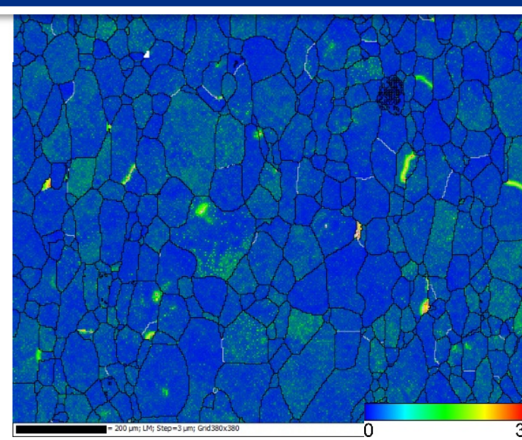
ATI -good flux expulsion-

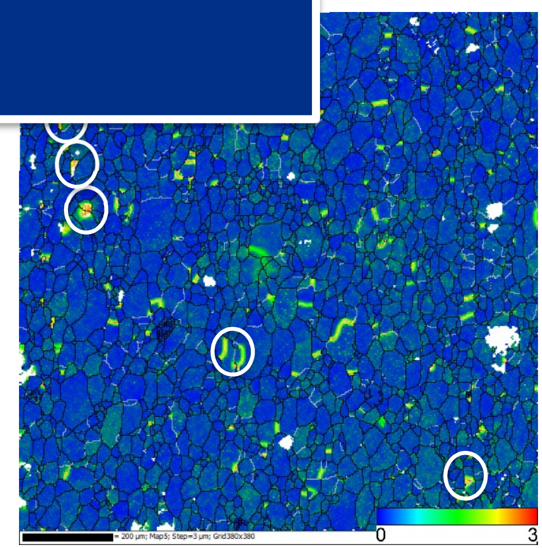


Ningxia -bad flux expulsion-



So…why relation between flux expulsion and dimensions of grains?



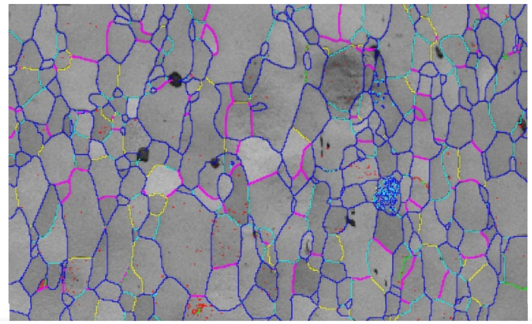


Analysis of “as received” materials (cross-section)

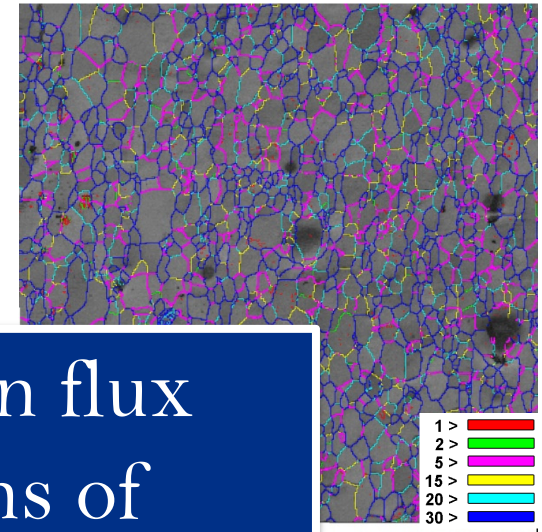
- Material that shows good flux expulsion properties (after annealing at 900C) has larger grain in the “as received” condition

- Material expulsion small grai level of lo

ATI -good flux expulsion-



Ningxia -bad flux expulsion-



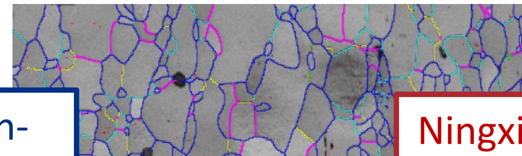
So…why relation between flux expulsion and dimensions of grains?

GBs block dislocations movement, therefore grains with high density of dislocations are found even after annealing

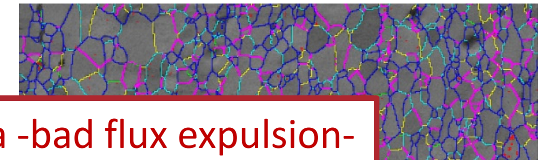
Analysis of “as received” materials (cross-section)

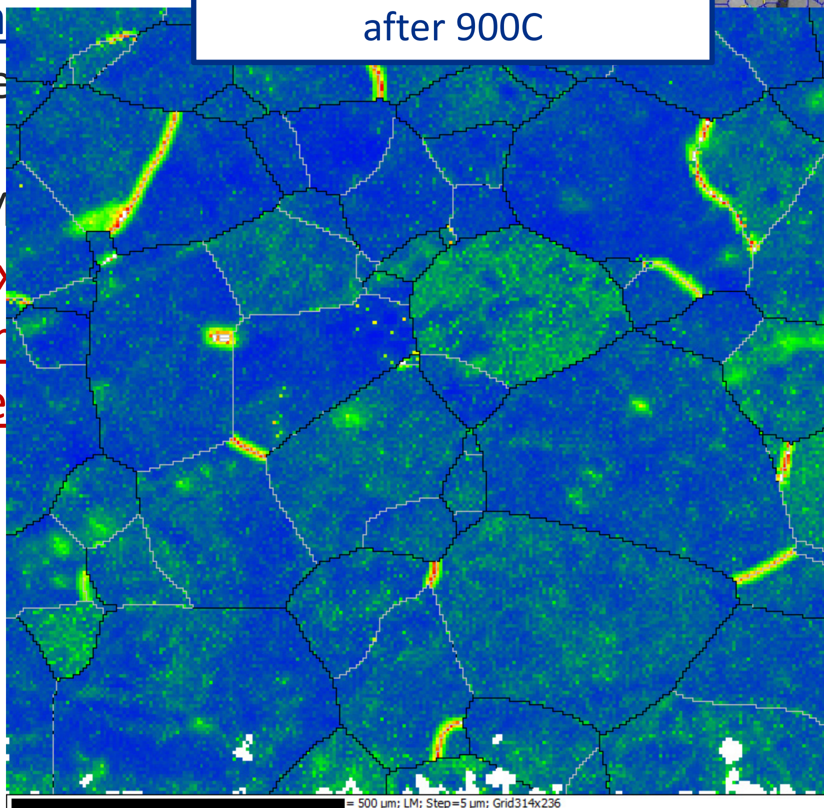
- Material that shows good flux expulsion properties (after ann
  la
  re
- M
  ex
  sr
  le

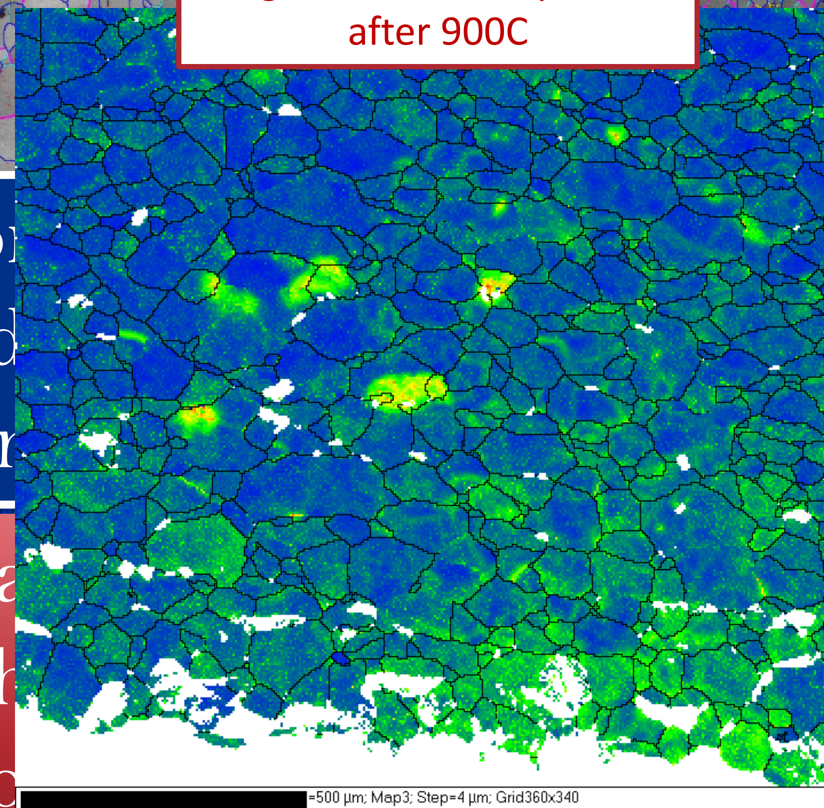
ATI -good flux expulsion-



Ningxia -bad flux expulsion-

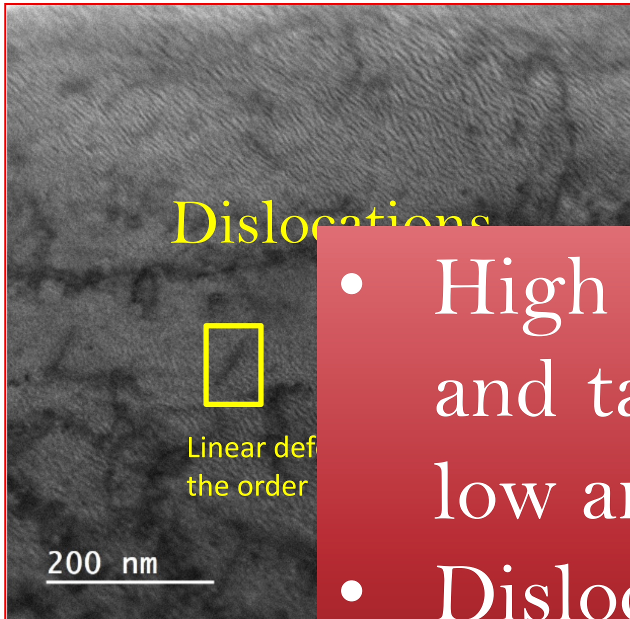


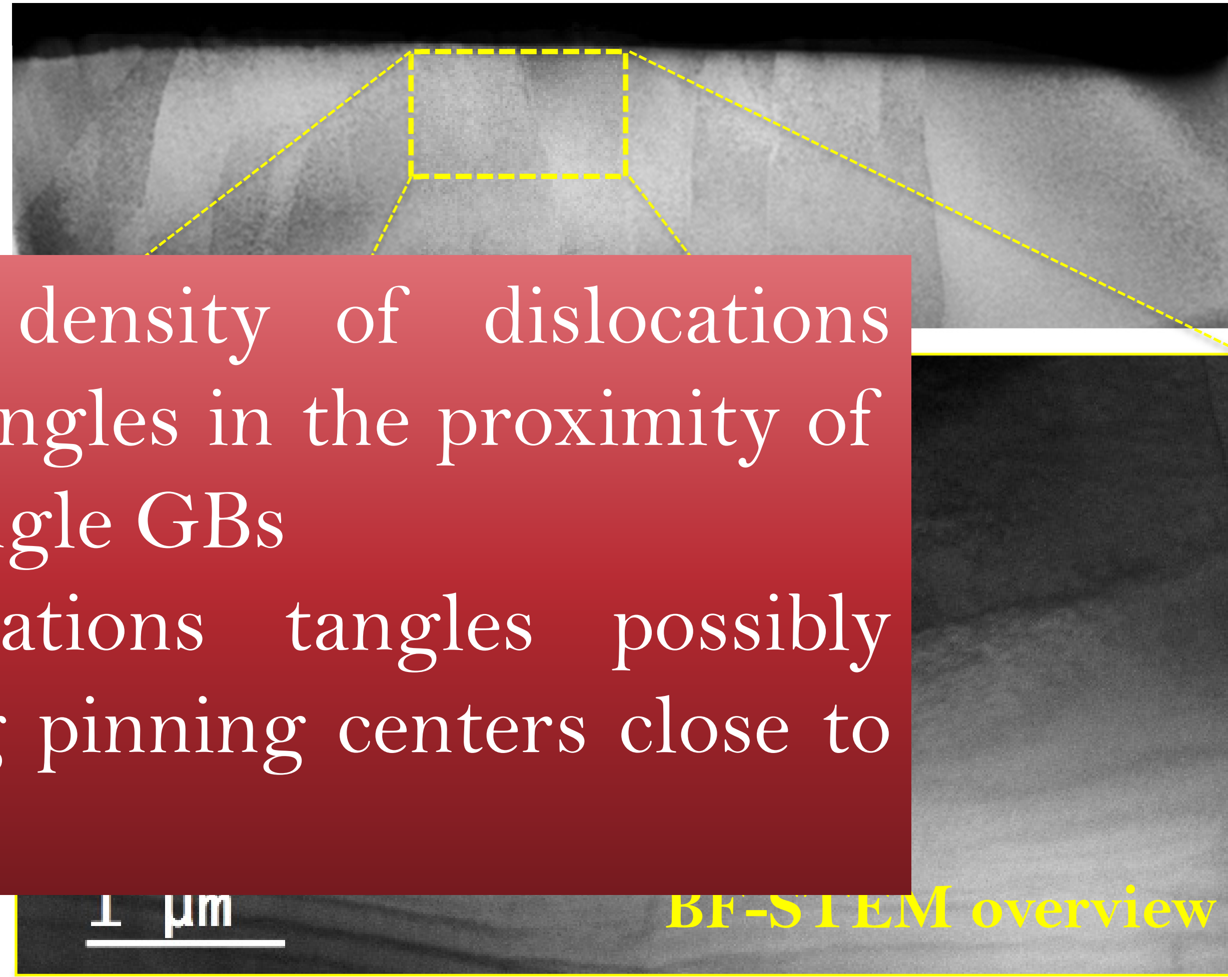




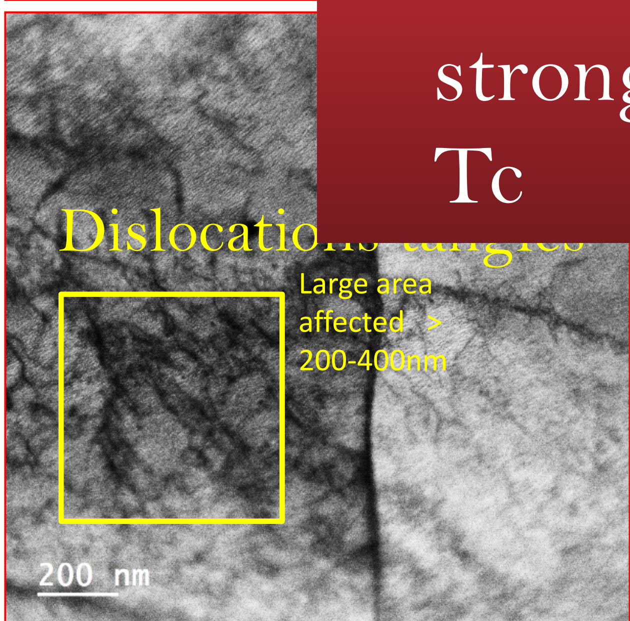
annealing

Study of highly defective areas on "as-received" material

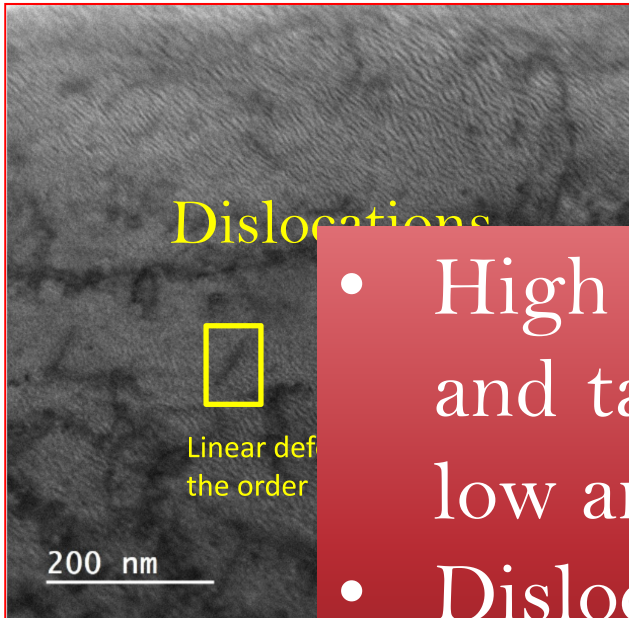


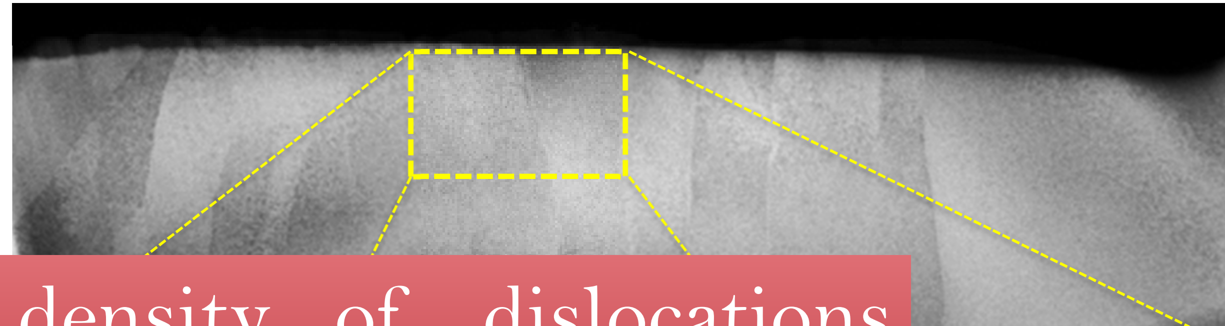


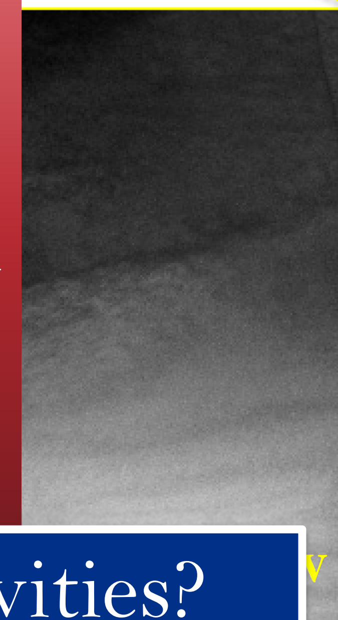
- High density of dislocations and tangles in the proximity of low angle GBs
- Dislocations tangles possibly strong pinning centers close to Tc

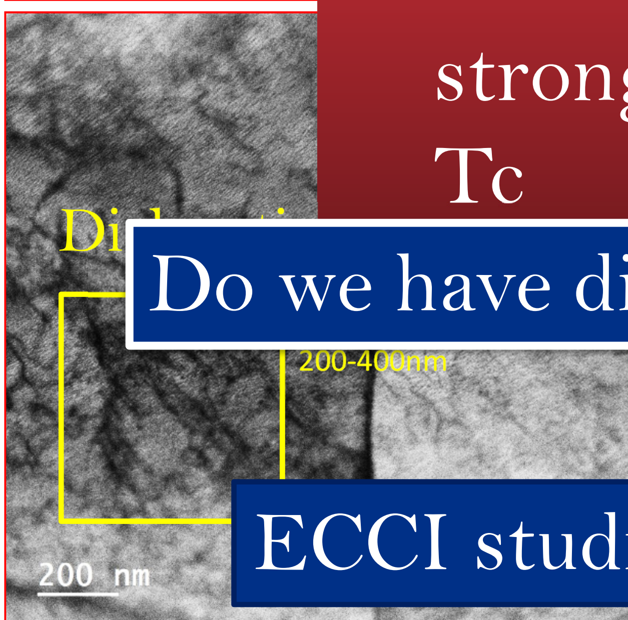


Study of highly defective areas on “as-received” material









- High density of dislocations and tangles in the proximity of low angle GBs
- Dislocations tangles possibly strong pinning centers close to Tc

Do we have dislocation tangles in Nb cavities?

ECCI studies in CAVITY CUT-OUTS

Outline

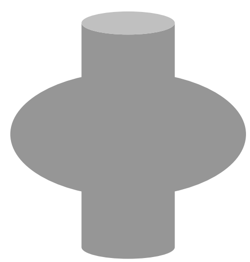
- Introduction on pinning
- Insights from flux expulsion studies in Nb cavities
- Microscopy studies of Nb "as-received" materials with different flux expulsion properties
- **Microscopy studies of cavity cut-outs with different flux expulsion properties**
- Conclusions

Overview cavity fabrication, processing and test

"As-received" material

At cavity vendors

Sheet deformed and welded into cavity shape



At research institutes

CBP/BCP/EP
+
High-T treatment (800-900C)

Cavity test (flux expulsion, Q vs Eacc, T-map, etc)

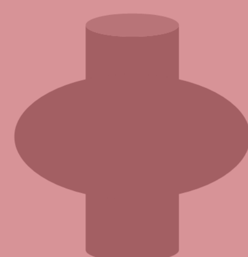
Cavity cut-outs

Overview cavity fabrication, processing and test

"As-received" material

At cavity vendors

Sheet deformed and welded into cavity shape



Introduction of dislocations and dislocations tangles

At research institutes

CBP/BCP/EP

+

High-T treatment (800-900C)

Cavity test (flux expulsion, Q vs Eacc, T-map, etc)

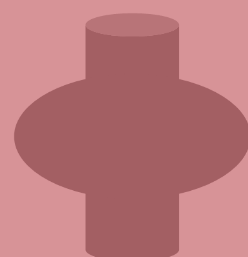
Cavity cut-outs

Overview cavity fabrication, processing and test

"As-received" material

Re-crystallization (nucleation of new grains), followed by grain growth

shape



Introduction of dislocations and dislocations tangles

At research institutes

CBP/BCP/EP
+
High-T treatment (800-900C)

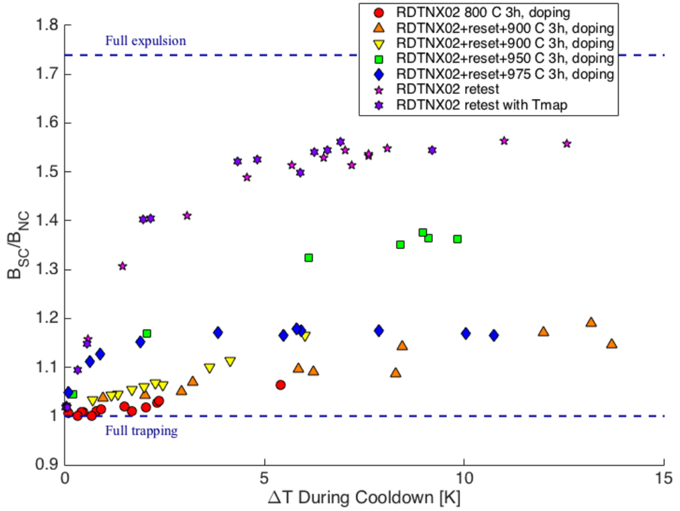
Cavity test (flux expulsion, Q vs Eacc, T-map, etc)

Cavity cut-outs

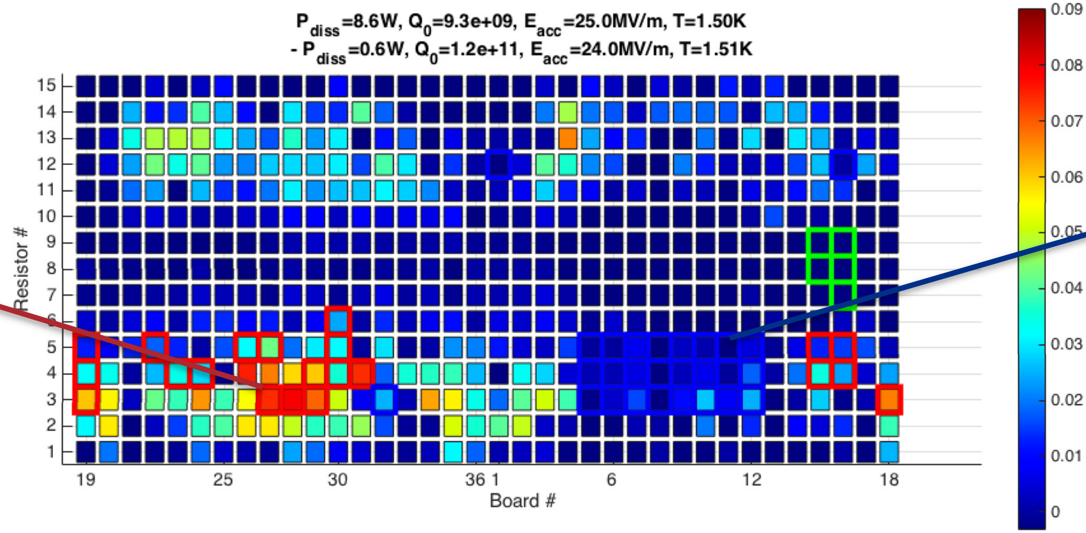
Cavity cut-outs generated from a non perfectly expelling cavity

Ningxia cavity (RTDNX02):

- Very poor flux expulsion after 3h at 800C
- Little improvement after subsequent annealing at 900C for 3h
- Some improvement after subsequent annealing at 950C for 3h
- Medium flux expulsion properties after annealing at 975C for 3h



Hot spots: regions that trapped flux even with fast cooldown

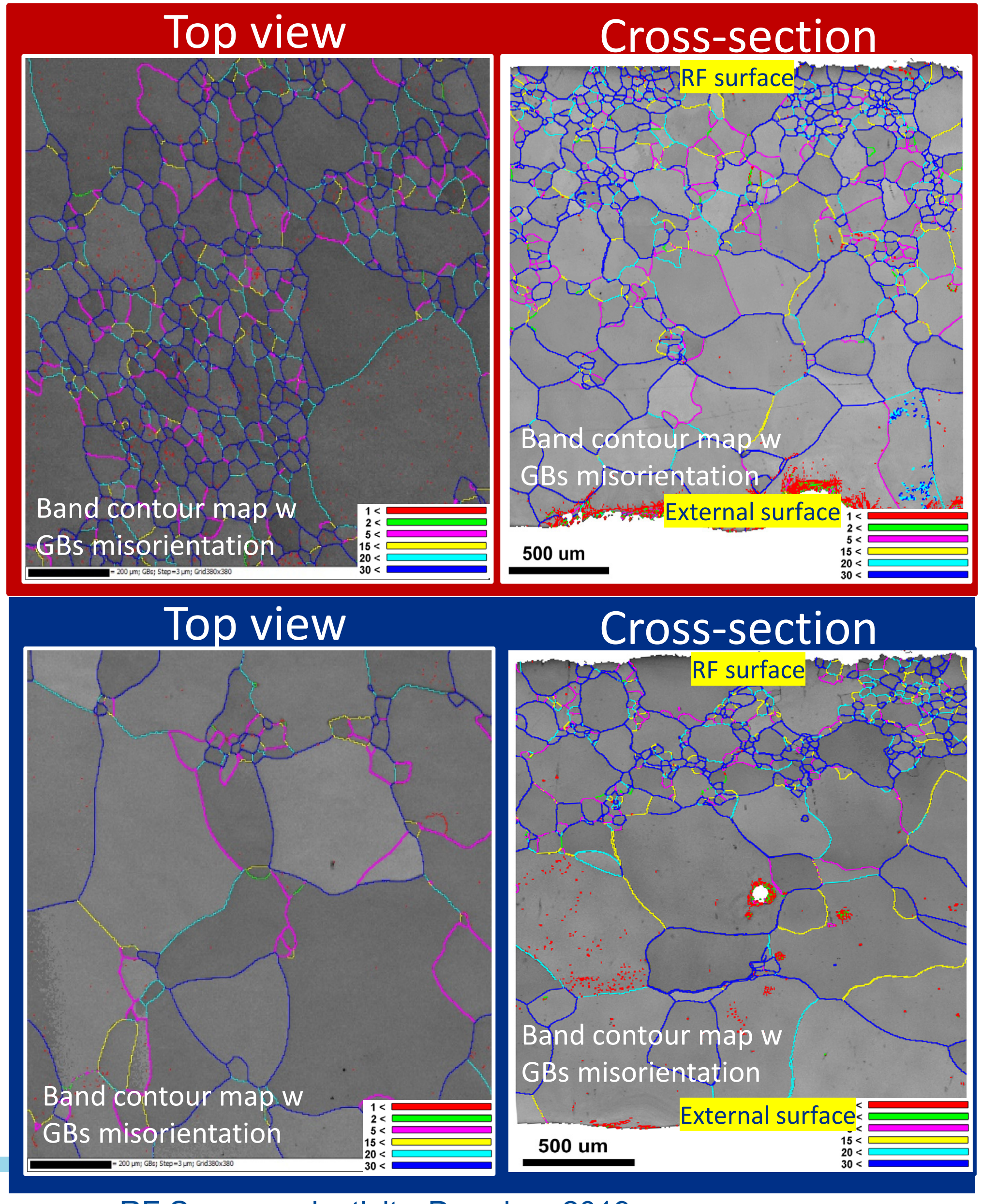


Cold spots: regions that expelled flux efficiently

For more info related to the T-map study see S. Posen talk, TTC 2018, Milan: https://agenda.infn.it/event/13791/contributions/21011/

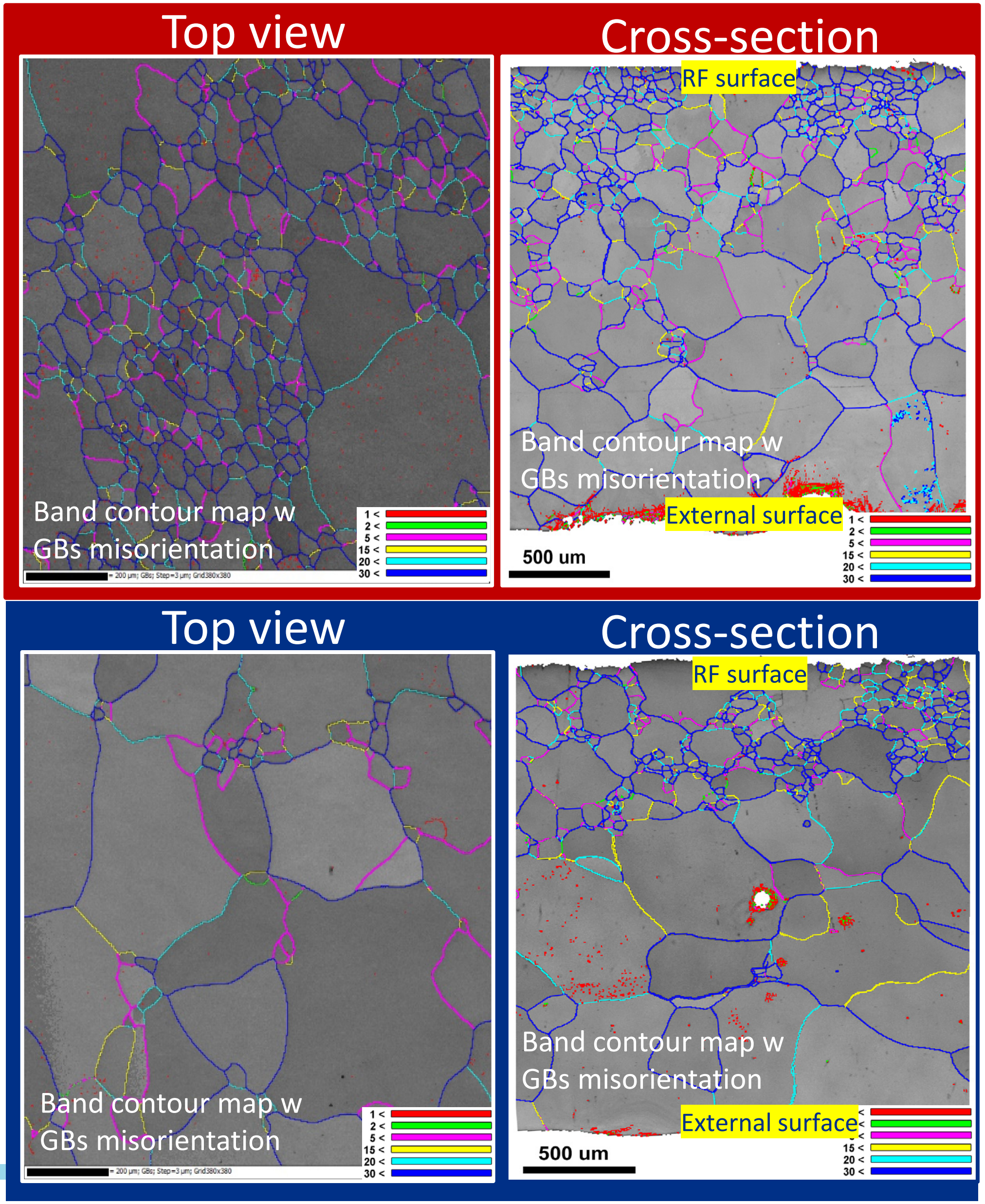
EBSD analysis of hot- and cold- spot cross-sections

- EBSD of top-view and cross-section of cavity cut-outs



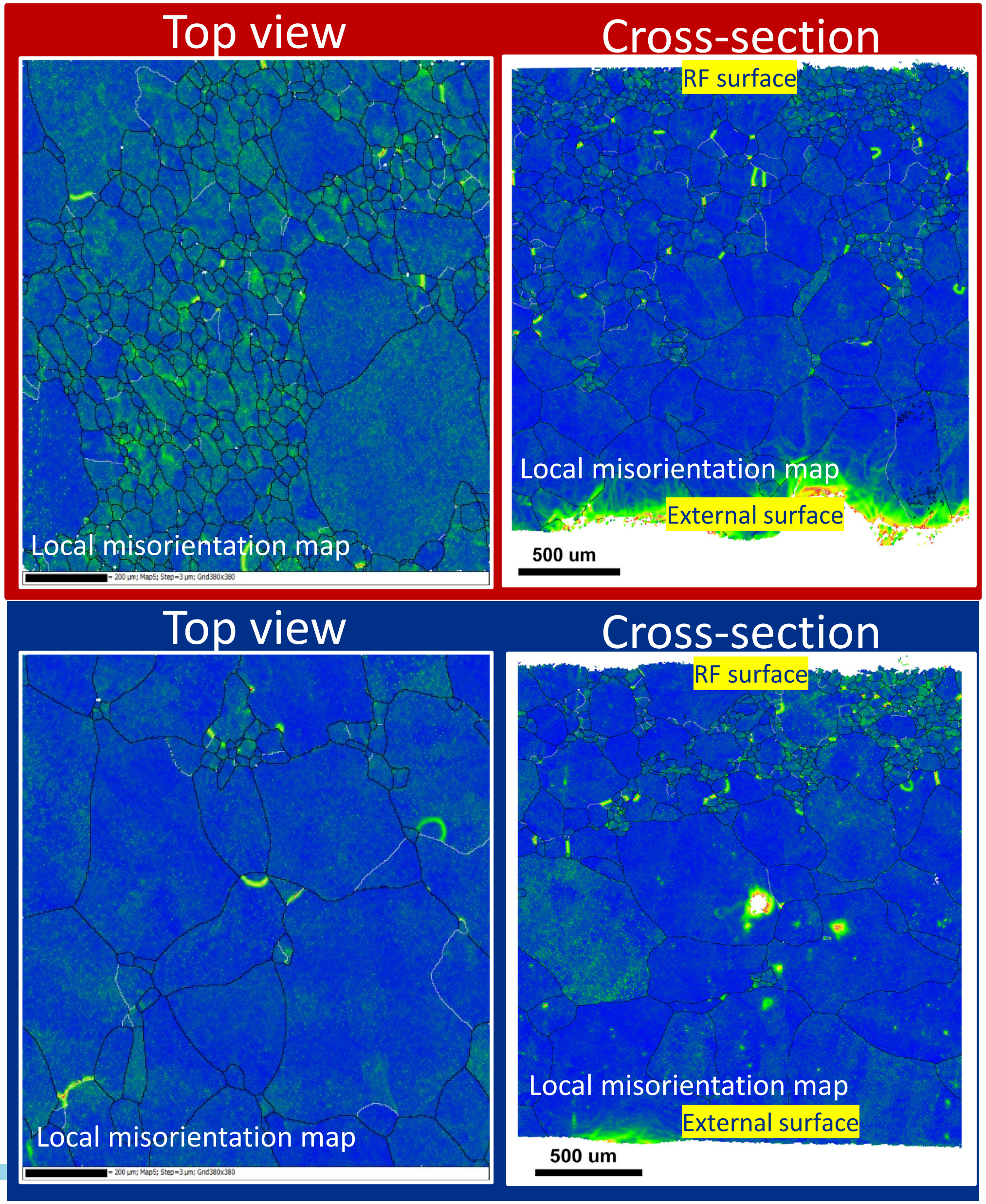
EBSD analysis of hot- and cold- spot cross-sections

- EBSD of top-view and cross-section of cavity cut-outs
- Smaller grains and higher density of low-angle GBs in hot-spot, more evident from top view than cross-section



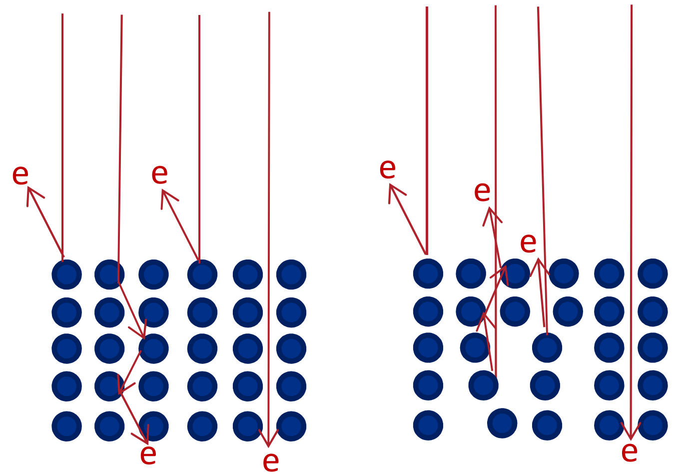
EBSD analysis of hot- and cold- spot cross-sections

- EBSD of top-view and cross-section of cavity cut-outs
- Smaller grains and higher density of low-angle GBs in hot-spot, more evident from top view than cross-section
- Higher level of local misorientation in hot-spot, more evident from top view than cross-section



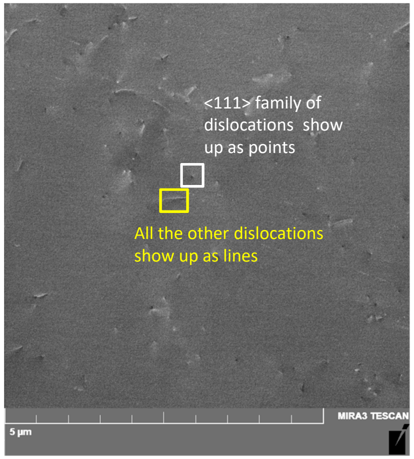
Electron Channeling Contrast Imaging (ECCI) for dislocations imaging

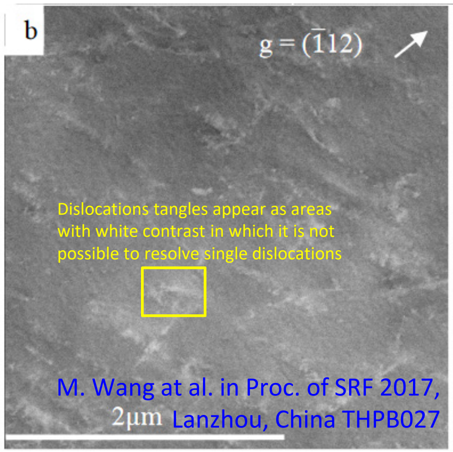
- ECCI is a scanning electron microscope (SEM) technique that makes use of the *electron channeling* mechanism.
- When electrons channel down through the crystal, **local distortions in the crystal lattice yield a modulation in the backscatter electron (BE) signal.**
  - ➢ Crystal defects such as dislocations can be imaged by ECCI
- The optimum channeling contrast in ECCI is obtained by orienting the crystal exactly into a channeling condition for a selected set of intense diffracting lattice planes
  - ➢ All images in the presentation were acquired along <200>channeling band



The presence of an edge dislocation convert an open channel to a closed one

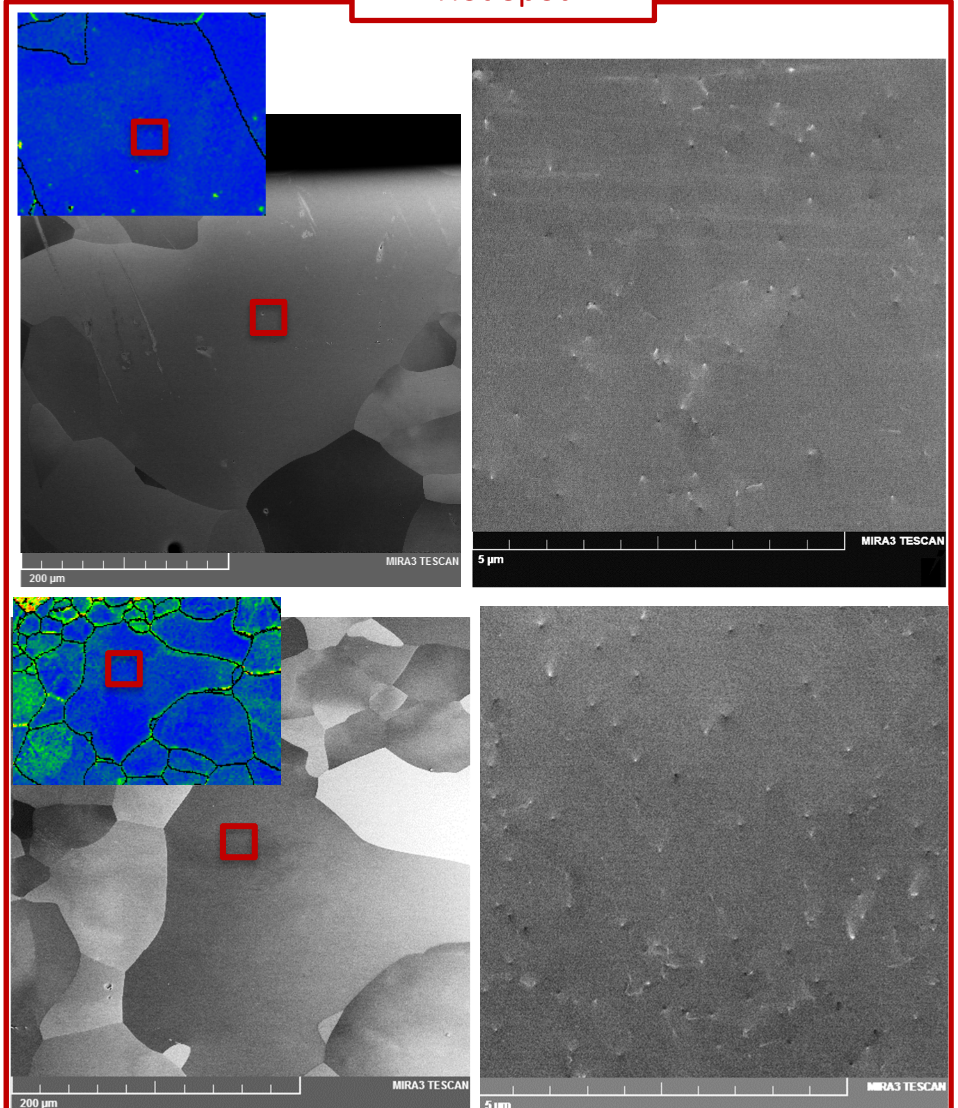
Example of ECC images



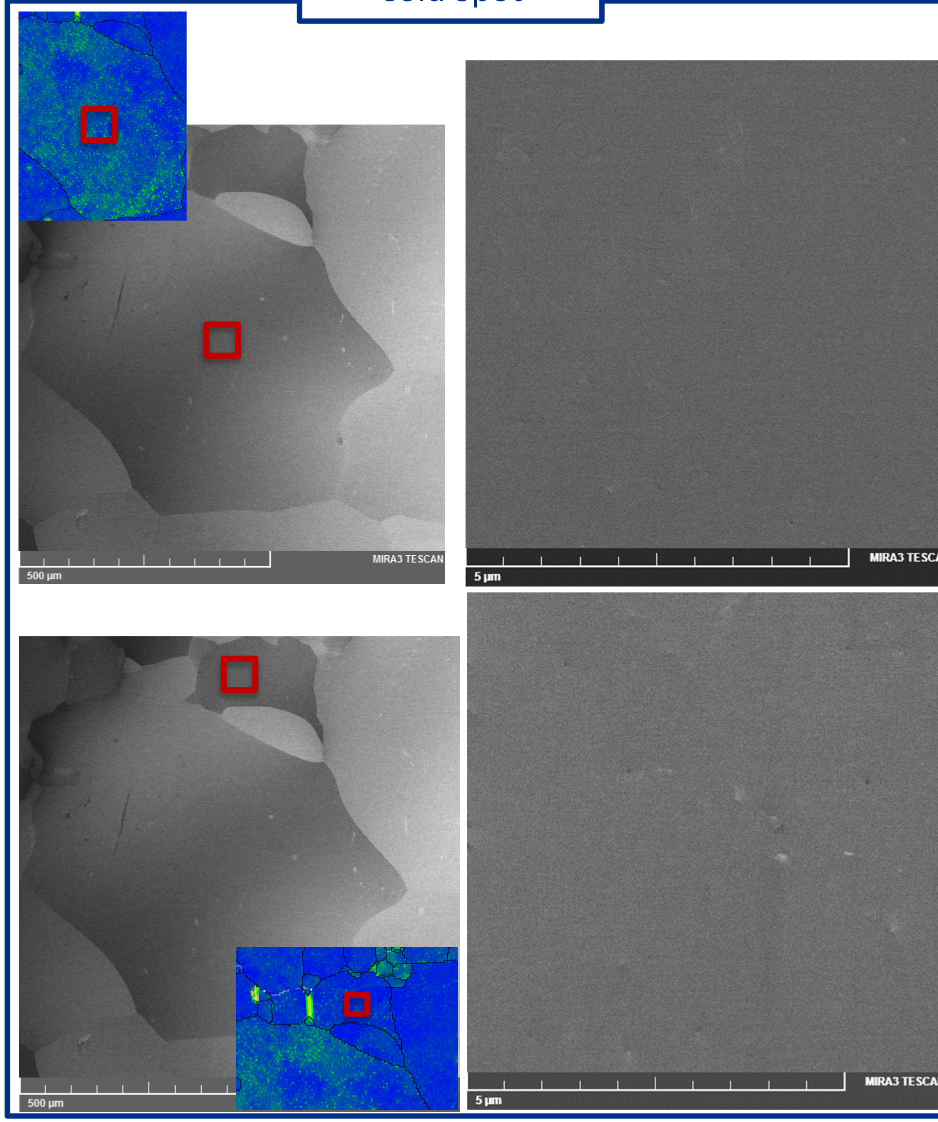


Dislocations in the middle of large grains

Hot Spot

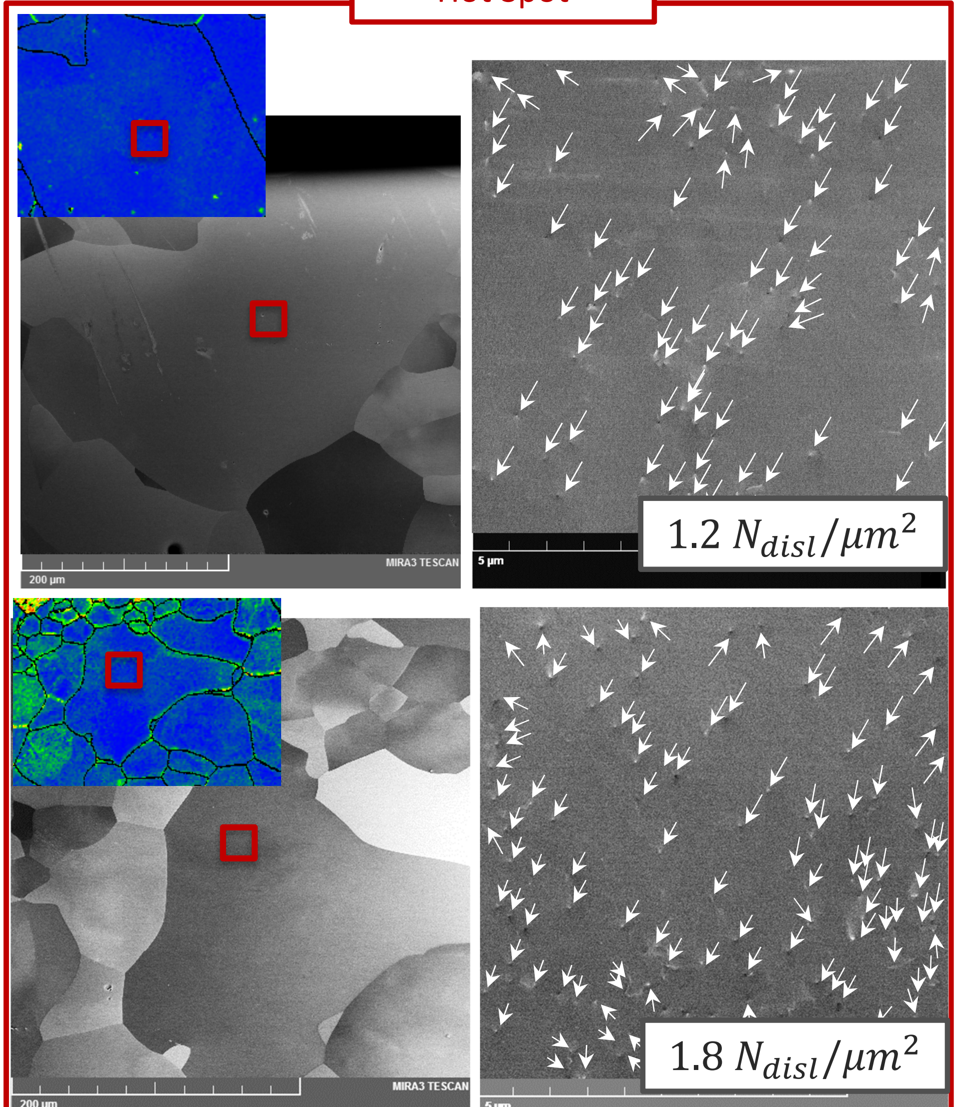


Cold Spot

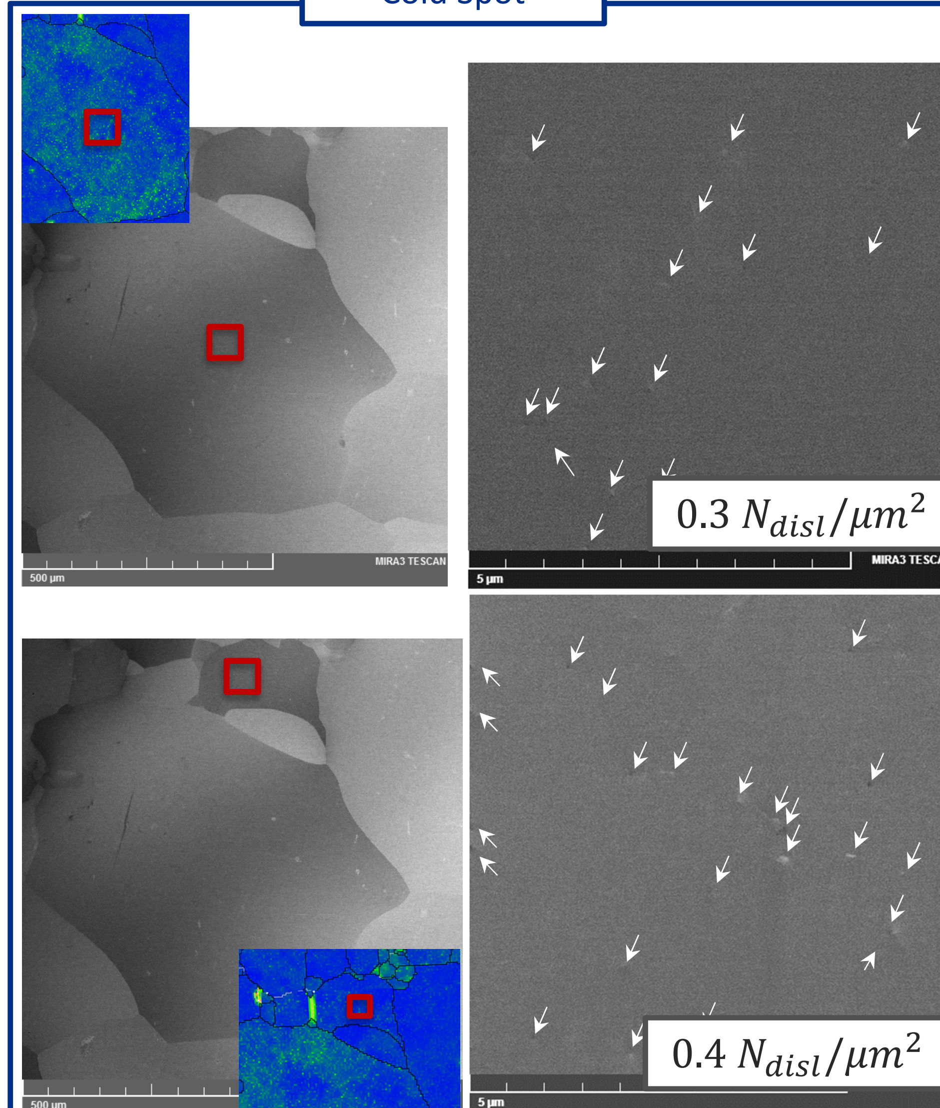


Dislocations in the middle of large grains

Hot Spot

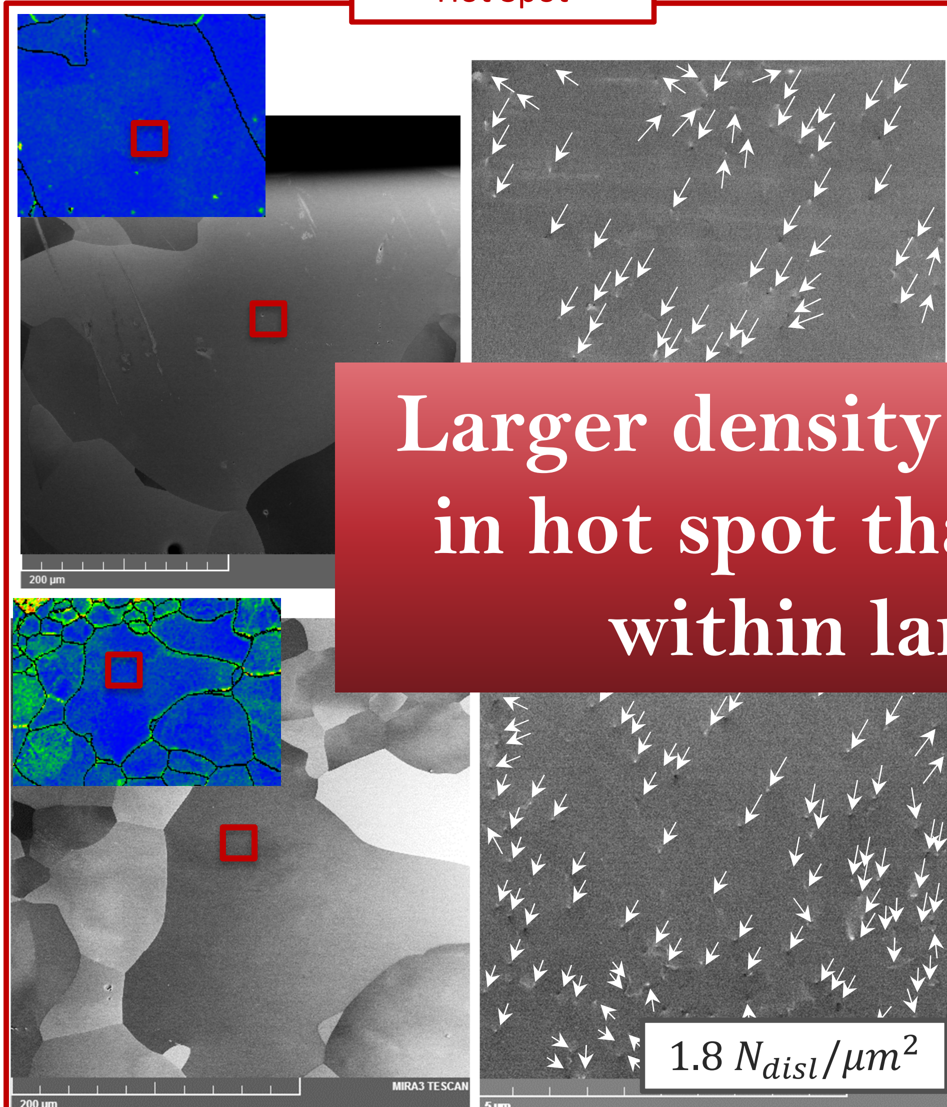


Cold Spot

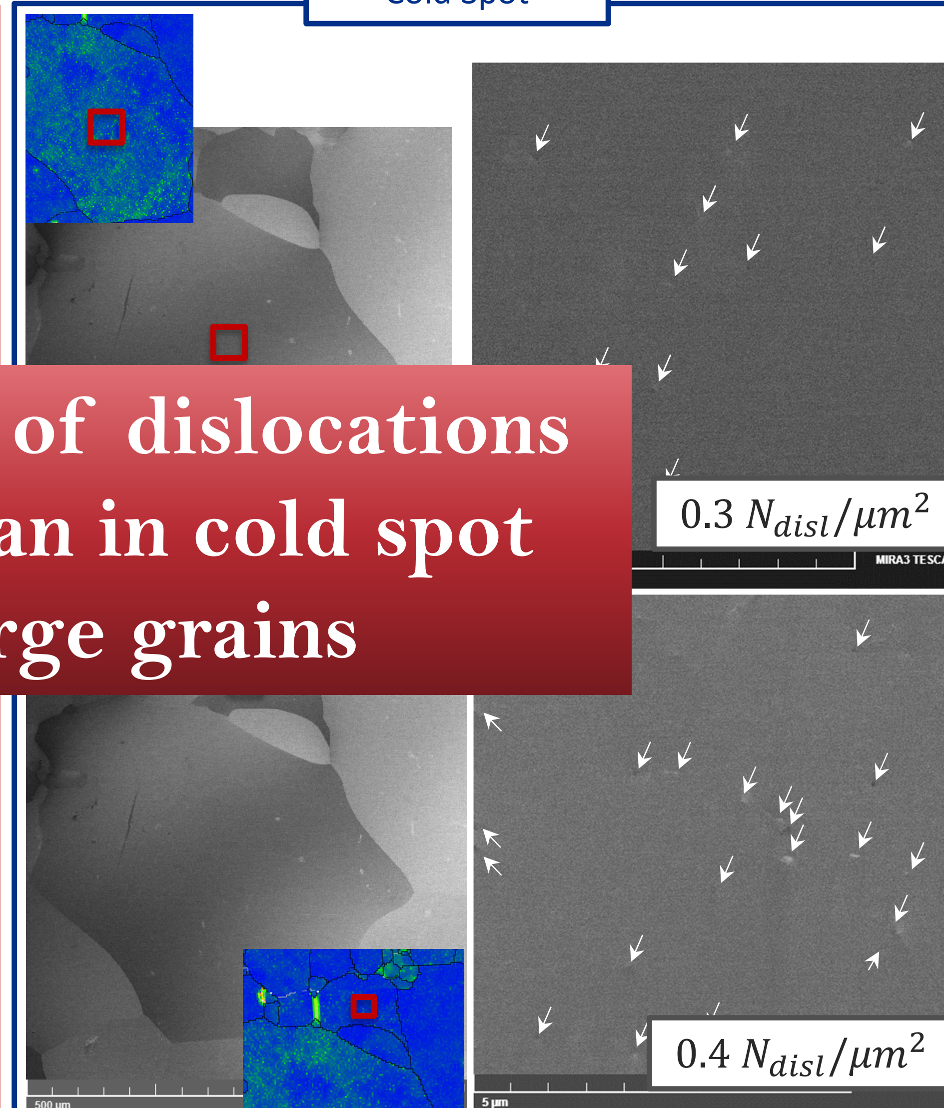


Dislocations in the middle of large grains

Hot Spot



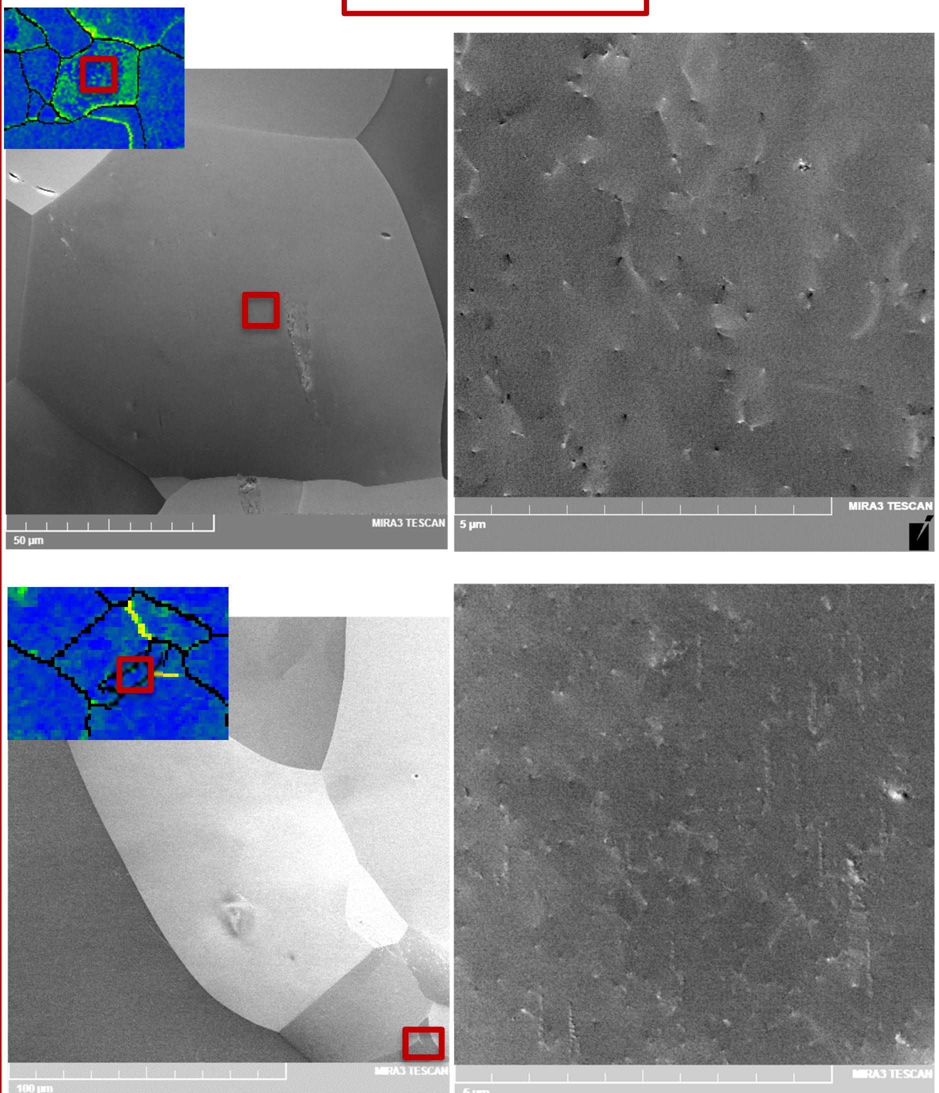
Cold Spot



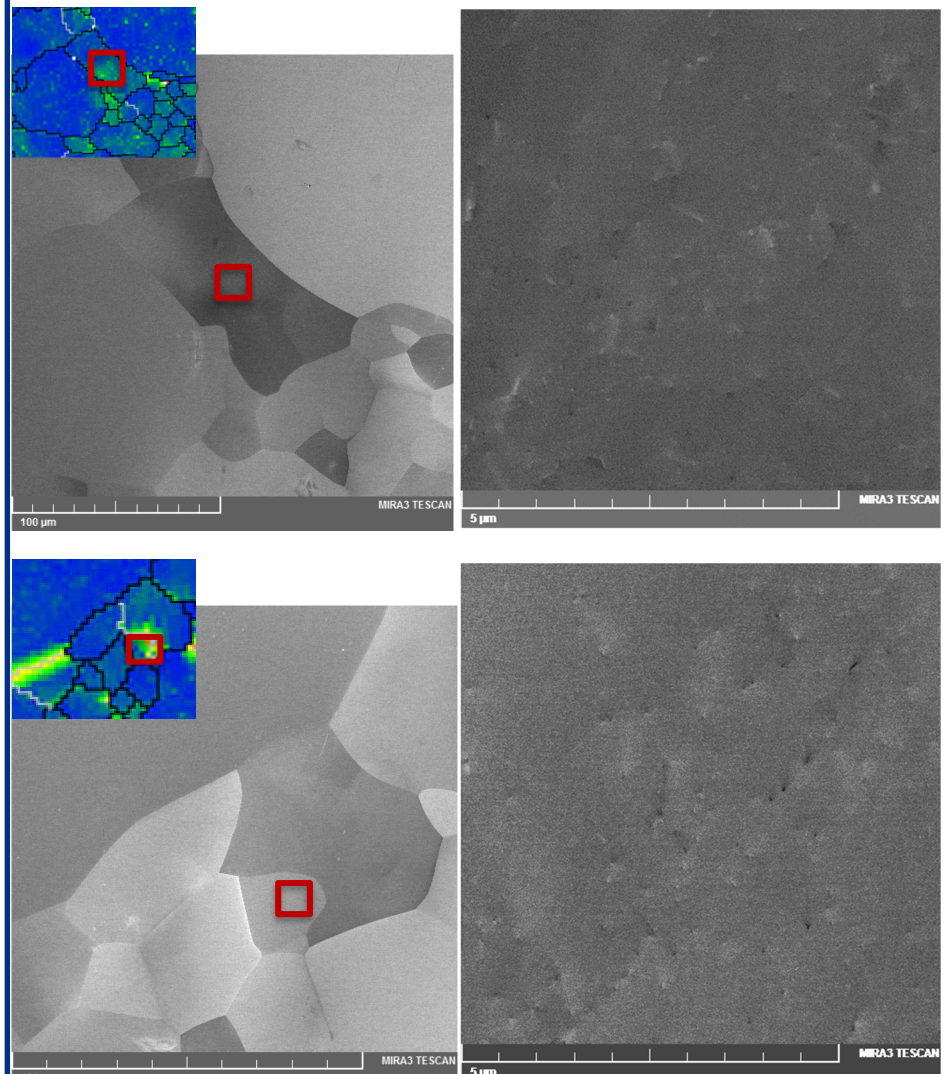
Larger density of dislocations in hot spot than in cold spot within large grains

Dislocations in the middle of small grains

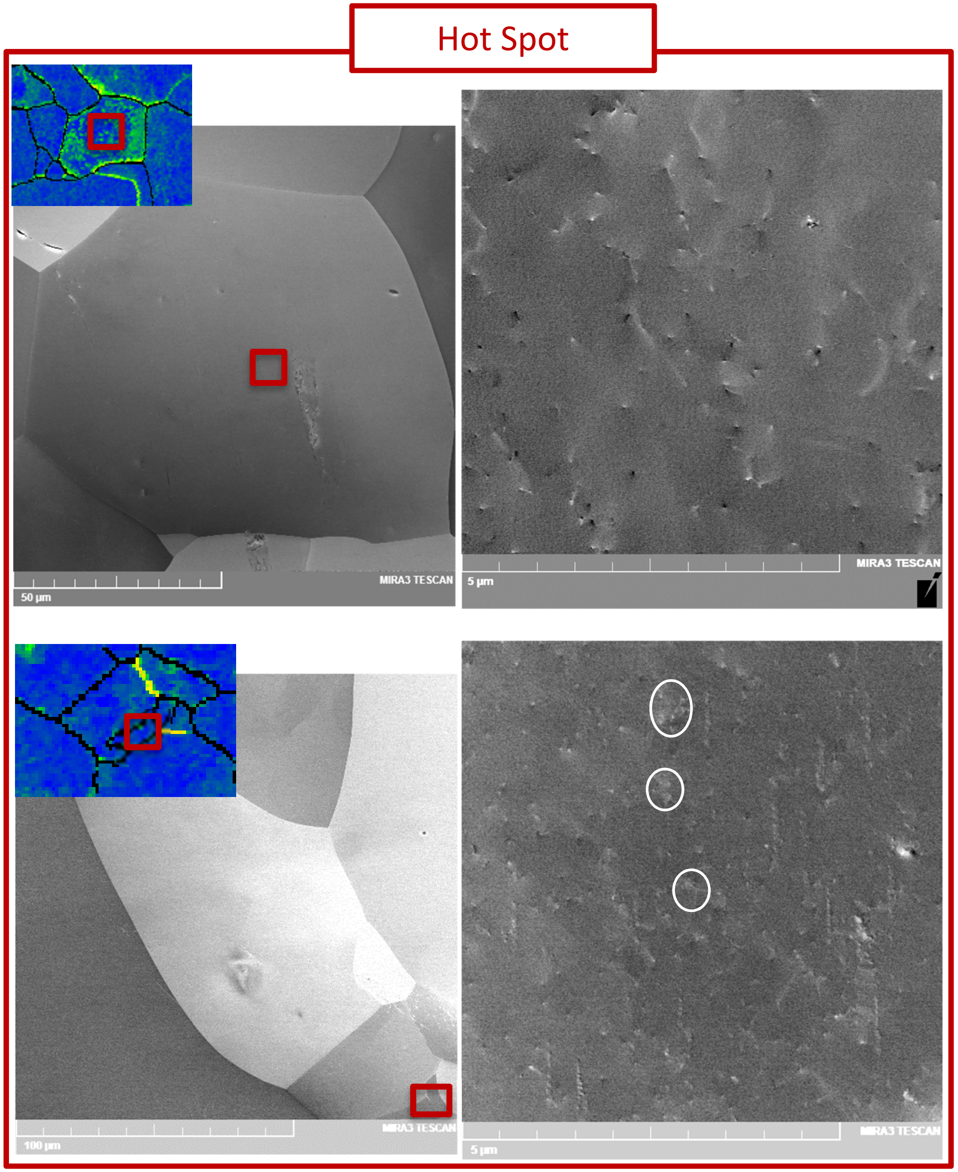
Hot Spot

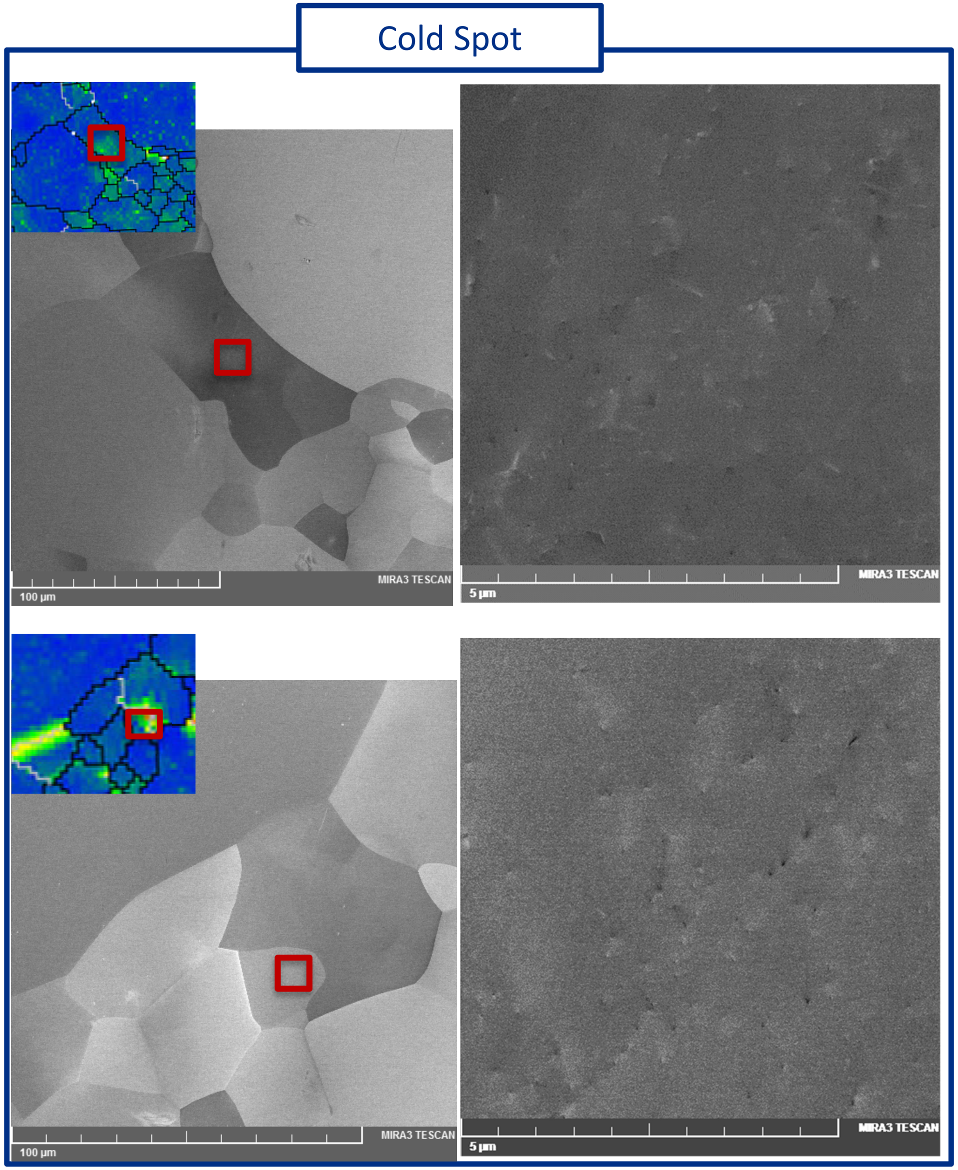


Cold Spot

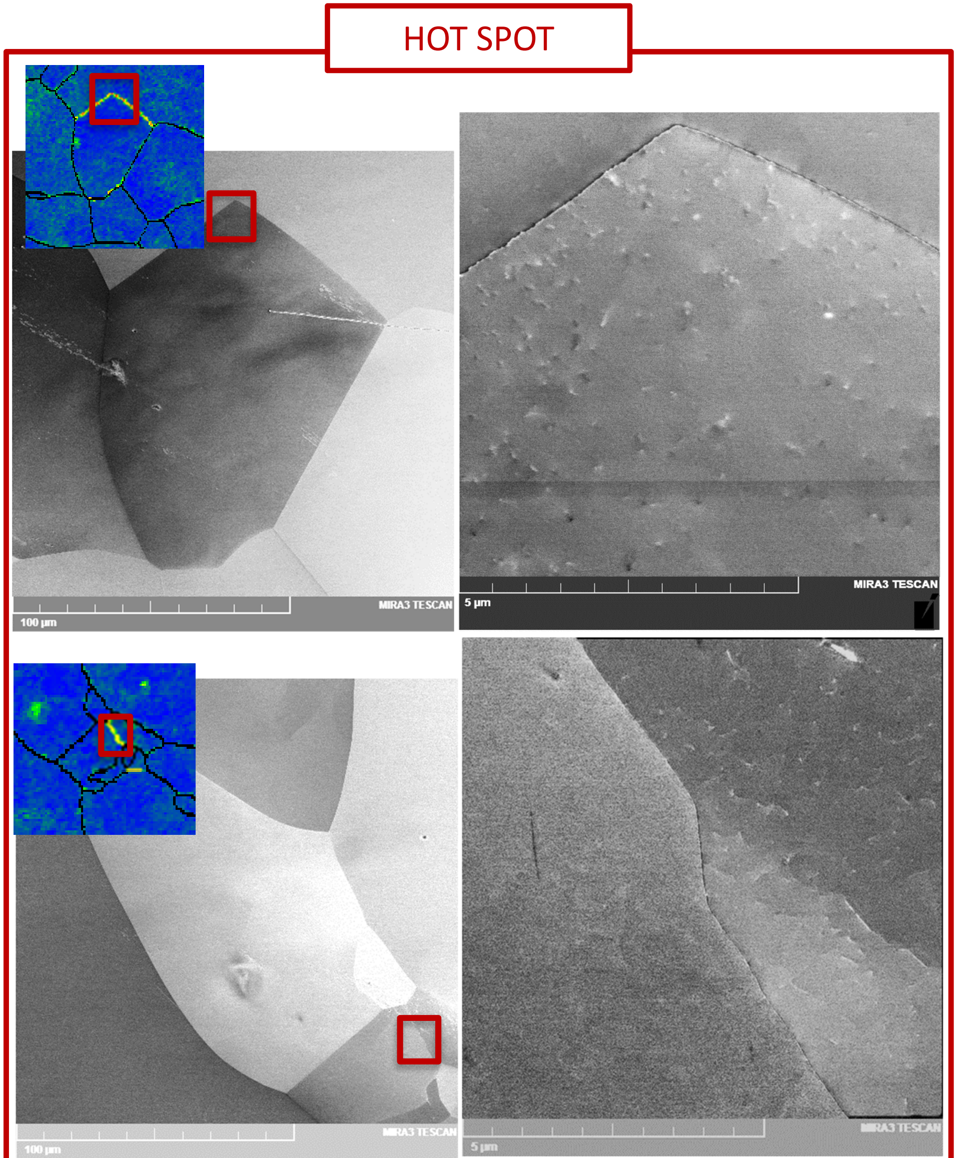


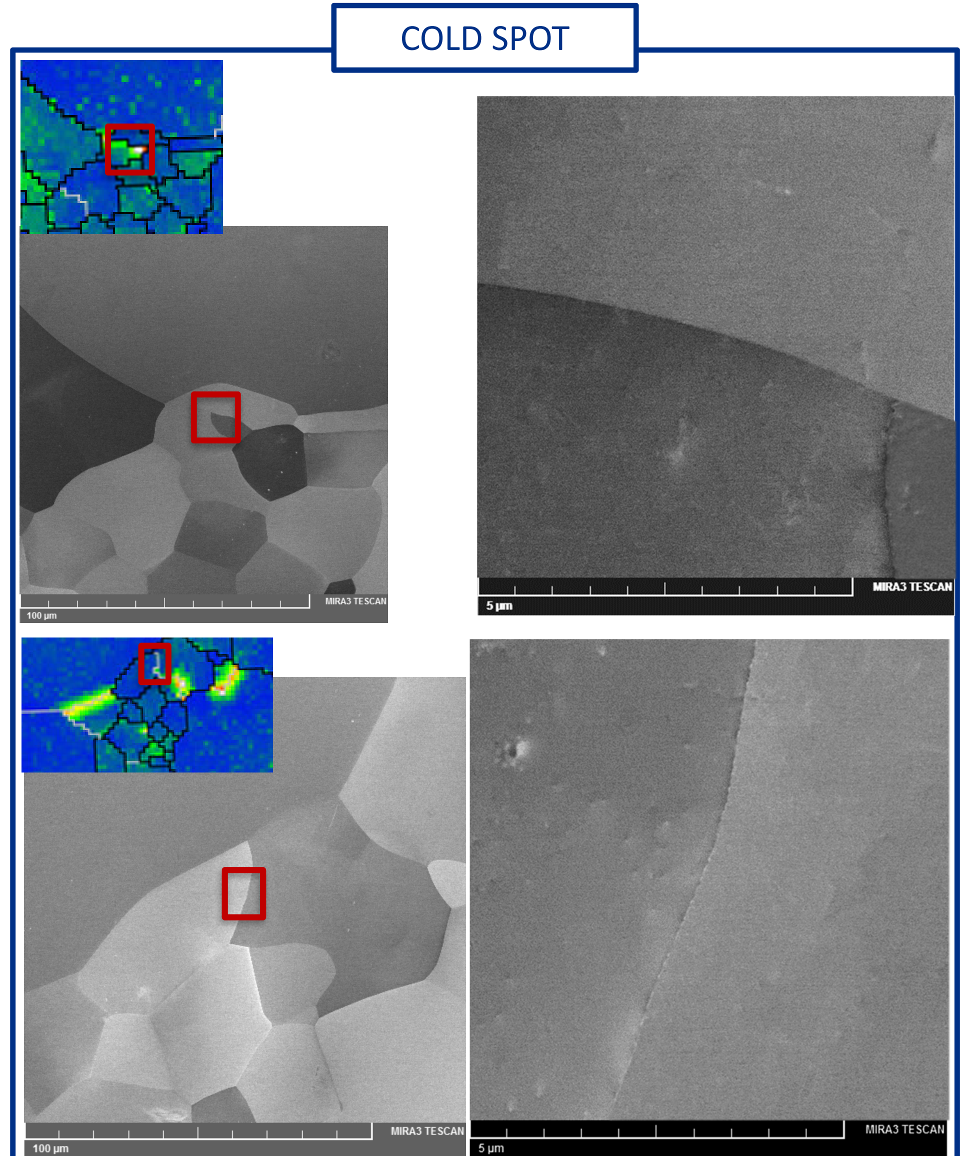
Dislocations in the middle of small grains



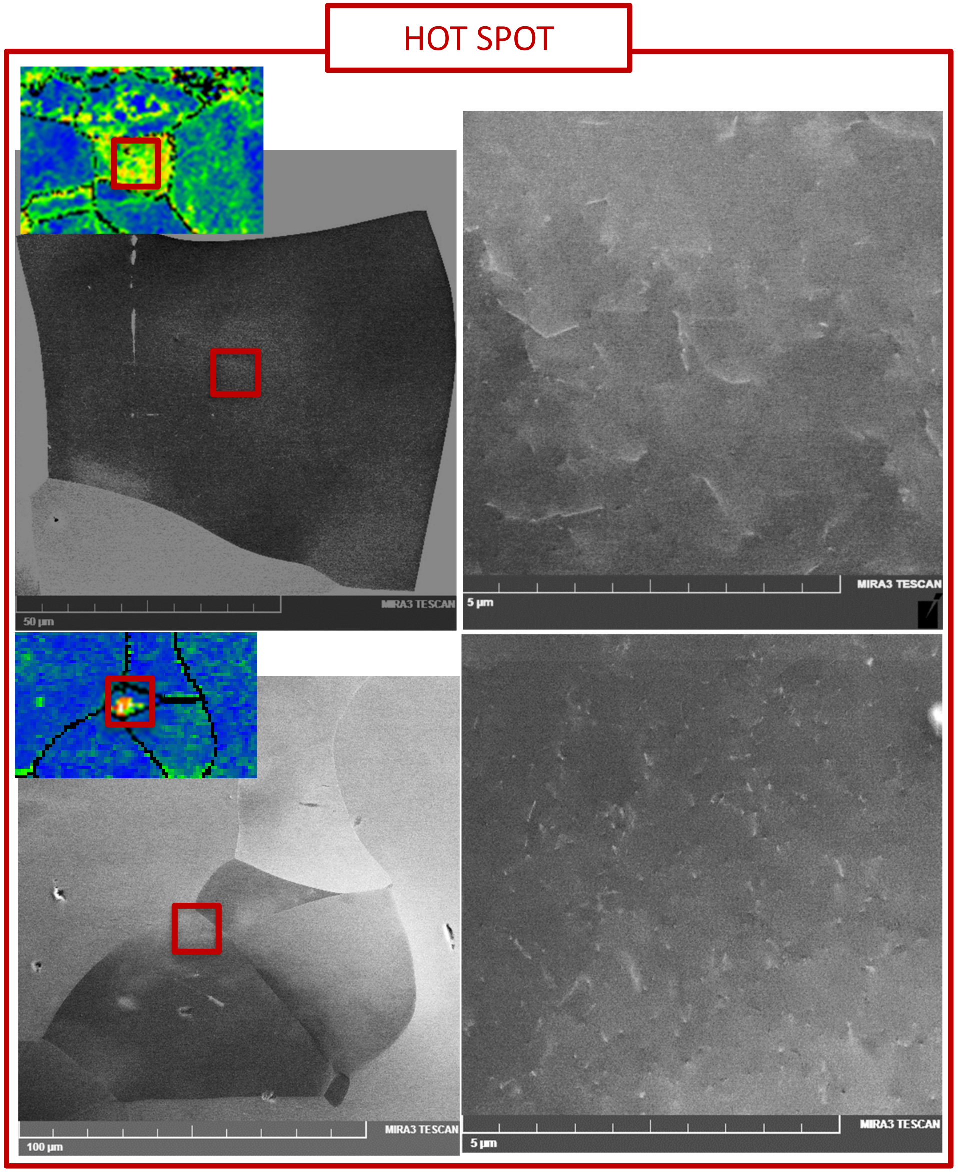


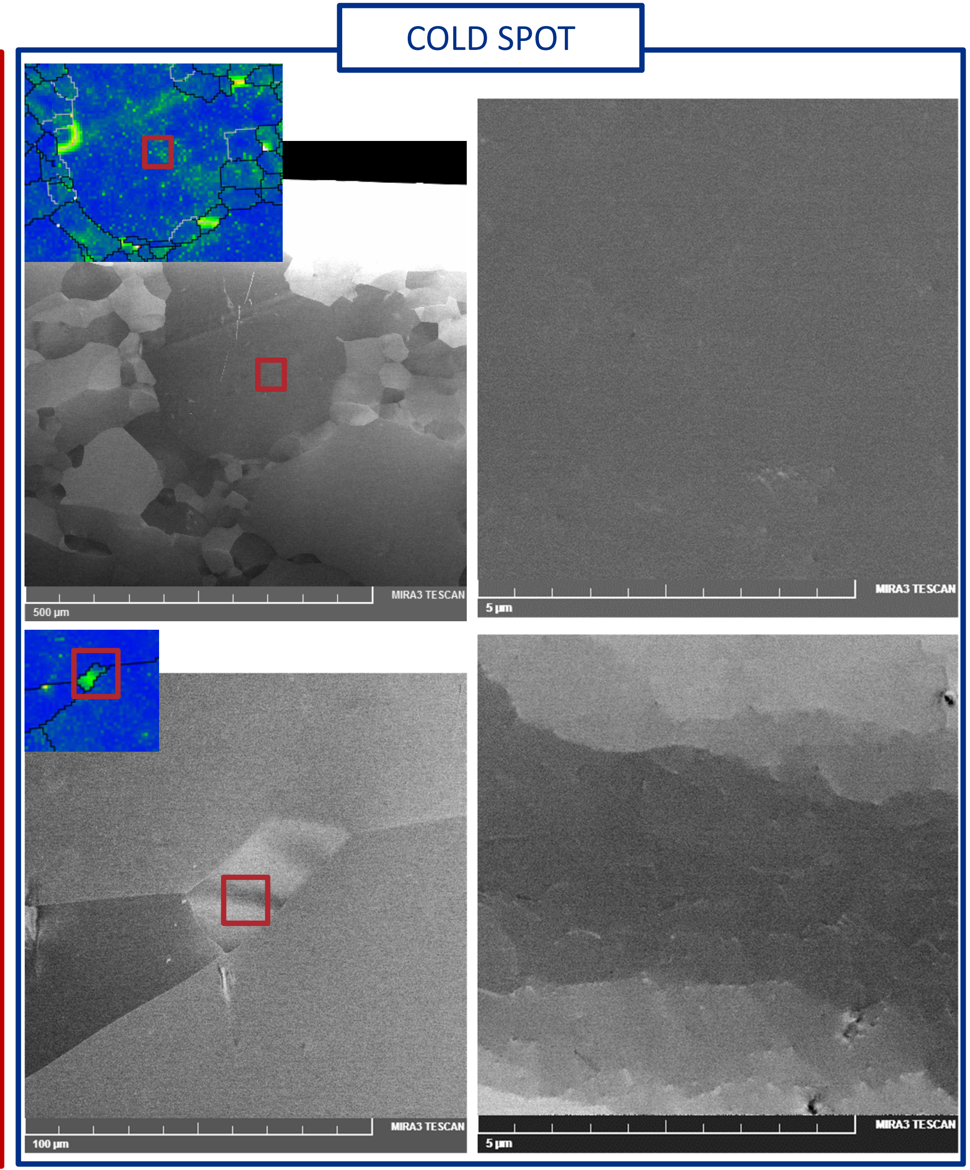
Dislocations at low-angle grain boundaries



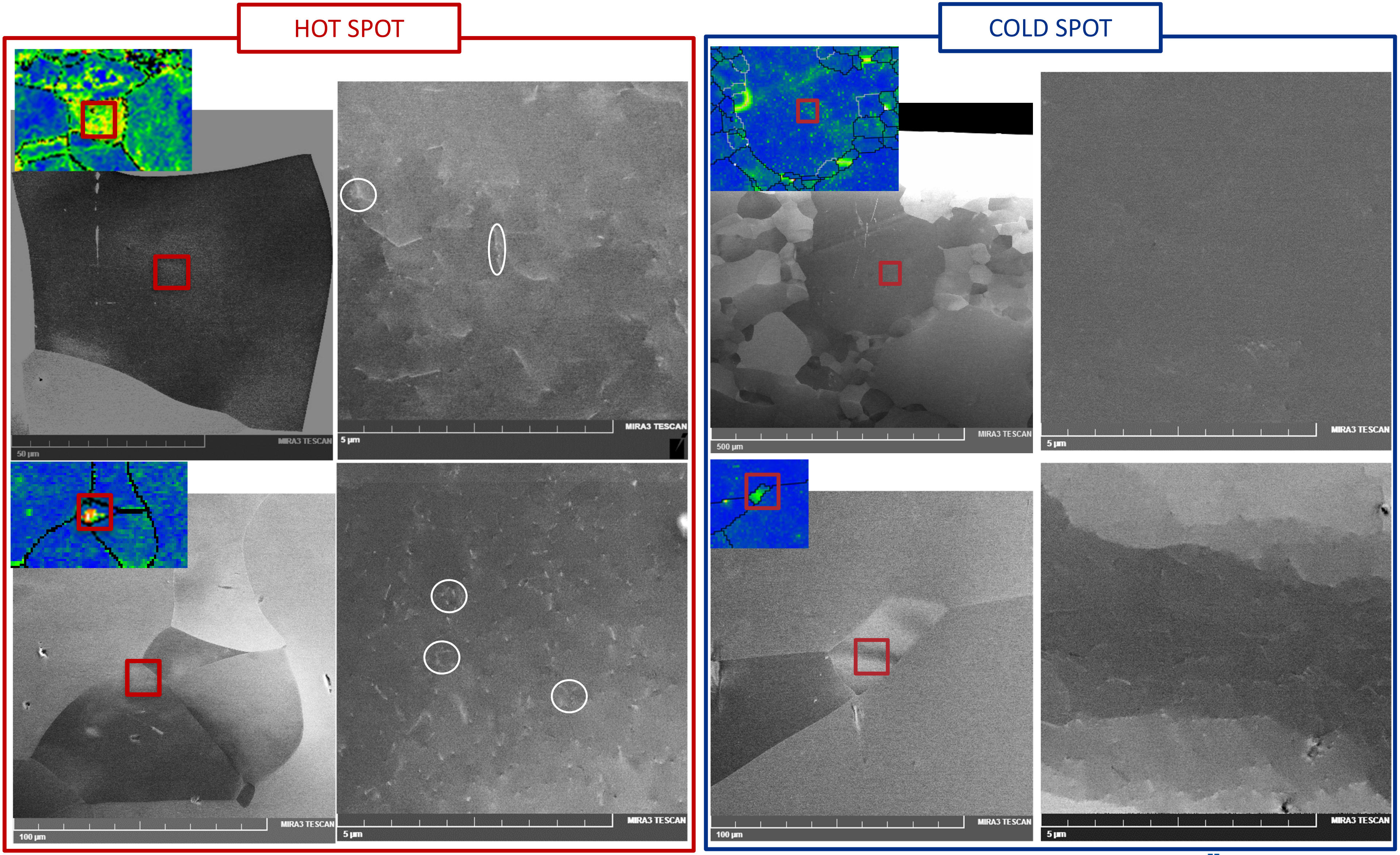


Dislocations at highly strained region



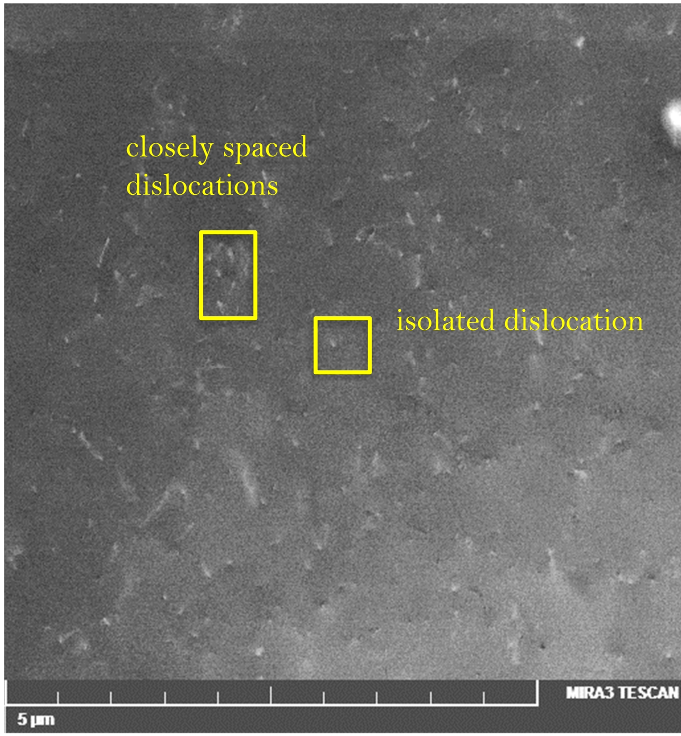


Dislocations at highly strained region



Summary results from ECCI in Hot and Cold spot

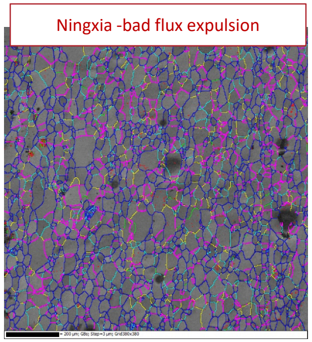
- No clear presence of dislocations tangles in cavity cut-outs
- Density of dislocations overall larger in hot spot than in cold spot
- Small grains and areas with large local misorientation show larger density of dislocations and lattice defects
- Areas in which **dislocations** are **closely spaced** to each other are **stronger pinning centers compared to isolated dislocations**

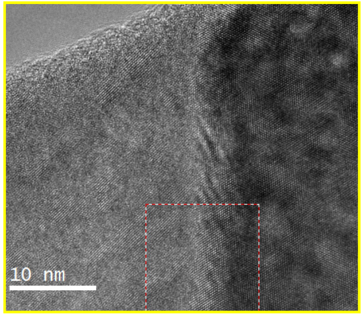


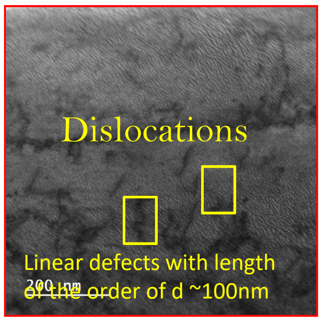
Outline

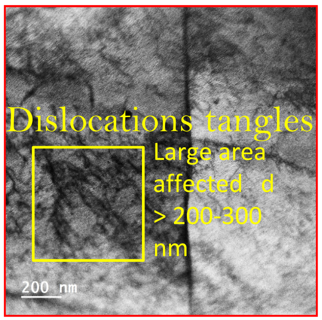
- Introduction on pinning
- Insights from flux expulsion studies in Nb cavities
- Microscopy studies of Nb “as-received” materials with different flux expulsion properties
- Microscopy studies of cavity cut-outs with different flux expulsion properties
- **Conclusions**

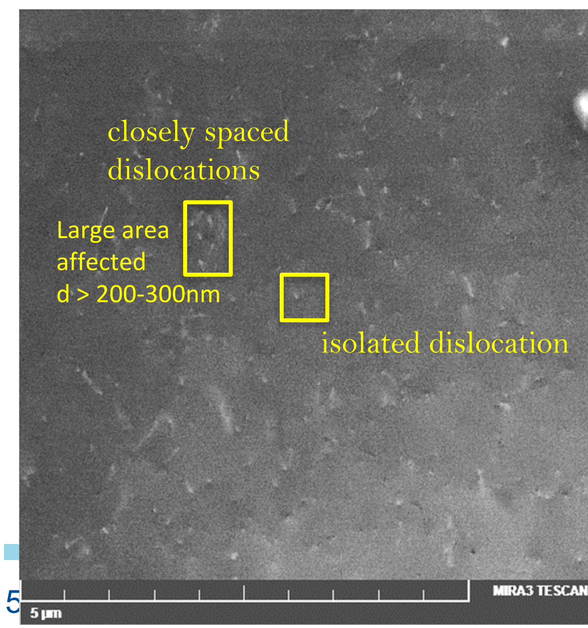
Conclusions – as received material





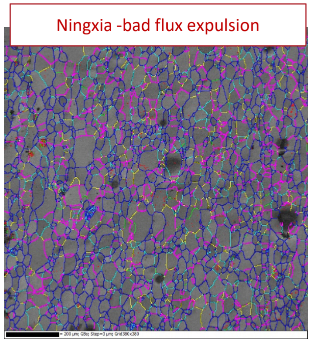


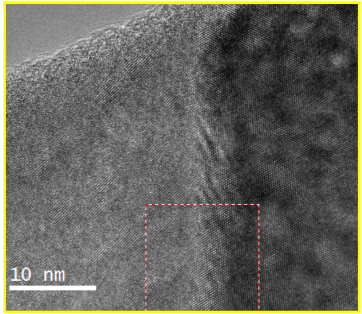


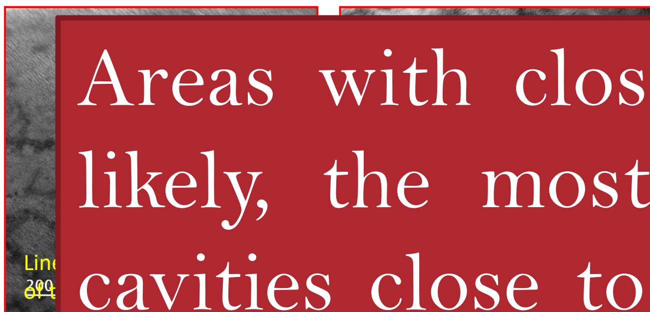


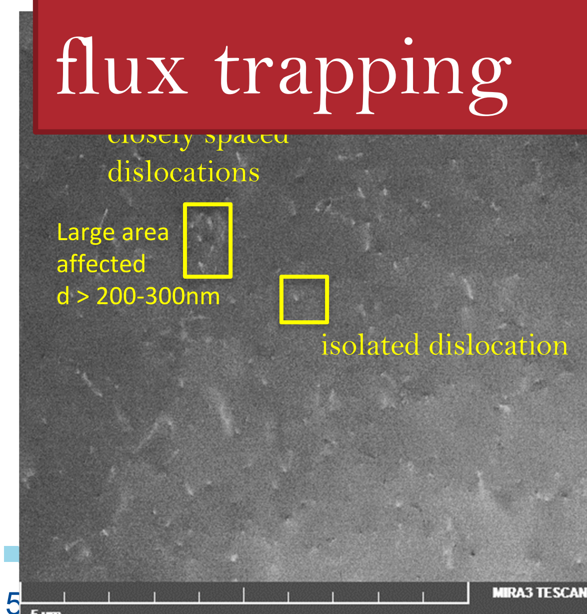
- Grain boundaries are not expected to be strong pinning center close to Tc (d << coherence length close to Tc, ξ~200nm)
- GBs block dislocations movement, therefore grains with high density of dislocations are found even after annealing → smaller grains are usually found in bad flux expelling material
- Dislocations tangles are expected to be stronger pinning center than GBs, they can affect an area as large of many 100s of nanometers (d ~ ξ)
- No clear presence of dislocations tangles in cavity cut-outs
- Presence of many regions with very high density of dislocations, affected area of ~ 200-300nm → possibly strong pinning center close to Tc (d ~ ξ)

Conclusions – as received material









- Grain boundaries are not expected to be strong pinning center close to Tc (d << coherence length close to Tc, ξ~200nm)
- GBs block dislocations movement, therefore grains with high density of dislocations are found

Areas with closely spaced dislocations are, most likely, the most effective pinning center in Nb cavities close to Tc, and therefore responsible of flux trapping

- No clear presence of dislocations tangles in cavity cut-outs
- Presence of many regions with very high density of dislocations, affected area of ~ 200-300nm → possibly strong pinning center close to Tc (d ~ ξ)

Acknowledgments

A special thanks to:

- Dr. Zu-Hawn Sung, Dr. Sam Posen, Dr. Jaeyel Lee, Dr. Anna Grassellino, Dr. Alex Romanenko, Dr. Mattia Checchin and all the FNAL SRF R&D team for experimental and intellectual contributions
- Hank Han, Minming Wang, Prof. Martin Crimp, Prof. Carl Bohelert, Prof. Thomas Bieler (Michigan State University) for helping acquiring ECCI data and for useful discussion about ECCI and dislocations

Founding agencies: DOE-HEP and LCLS-II (DOE-BES)

Thank you for your attention!