Analysis of Flux Pinning Variability with Niobium Stock Material for LCLS-II

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Contribution list - Plots

- Material Plots by Jacek Sekutowicz DESY/LCLS-II, and Gigi Ciovati JLab.
- NX EBSD and tensile strength analysis for LCLS-II material studies completed Roy Crooks – Black Laboratories LLC Virginia USA - using NASA Langley equipment.

Preface

- Tokoyo Denkai (TD) material is not necessarily better than Ningxia Orient Tantalum Industry Co ltd (NX) material. Please do not just go out and buy TD material, or any other manufacture for that matter from this data set.
- The LCLS-II recipe changes came at a risk, cavities are very soft. Do not just go out and copy LCLS-II recipes without further research or risk analysis. – please go to Hot tops session this afternoon
- Given a fixed non-ideal flux expelling material, forcing grain growth **will** make the flux expulsion better at a cost of softening the niobium (from cavity results). **Maybe** – please go to hot tops session this afternoon
- All Data analysis and LCLS-II recipe modification were done mid-production, with less than ideal and limited data – See Gonnella Thursday for RF results.

LCLS-II Material

Parameter	Unit	**E-XFEL / LCLS II**
Year	-	2007&2010 / 2014
RRR	-	> 300
CHEMICAL COMPOSITION		
Ta	% or weight ppm	≤ 0.05 %
W	% or weight ppm	≤ 0.007%
Ti	% or weight ppm	≤ 0.005%
Fe	% or weight ppm	≤ 0.003%
Si	% or weight ppm	≤ 0.003%
Mo	% or weight ppm	≤ 0.005%
Ni	% or weight ppm	≤ 0.003%
H_2	% or weight ppm	≤ 2 w-ppm
N_2	% or weight ppm	≤ 10 w-ppm
O_2	% or weight ppm	≤ 10 w-ppm
C	% or weight ppm	≤ 10 w-ppm
MICROSTRUCTURE		100% recrystallized. Uniform size and equal-axed grains
Grain size	ASTM µm	Predominantly ≥ 6 ≤ 45
Maximal grain size	ASTM µm	> 4 < 90
MECHANICAL PROPERT.		
Tensile strength, Rm *	N/mm^2	> 140
Yield strength, Rp0.2 *	N/mm^2	50 < Rp0.2 < 100
Elongation, AL 30 *	%	≥ 30
Hardness, HV (min. load 10 N)	-	≤ 60

- 26 Total heat lots from Tokyo Denkai (TD) material **- 3155 sheets**
- 16 heat lots from Ningxia Orient Tantalum industry co ltd (OTIC or Ningxia or NX) - **2745 sheets**

Compiled by Jacek Sekutowicz DESY/LCLS-II

How is material specification defined/tested

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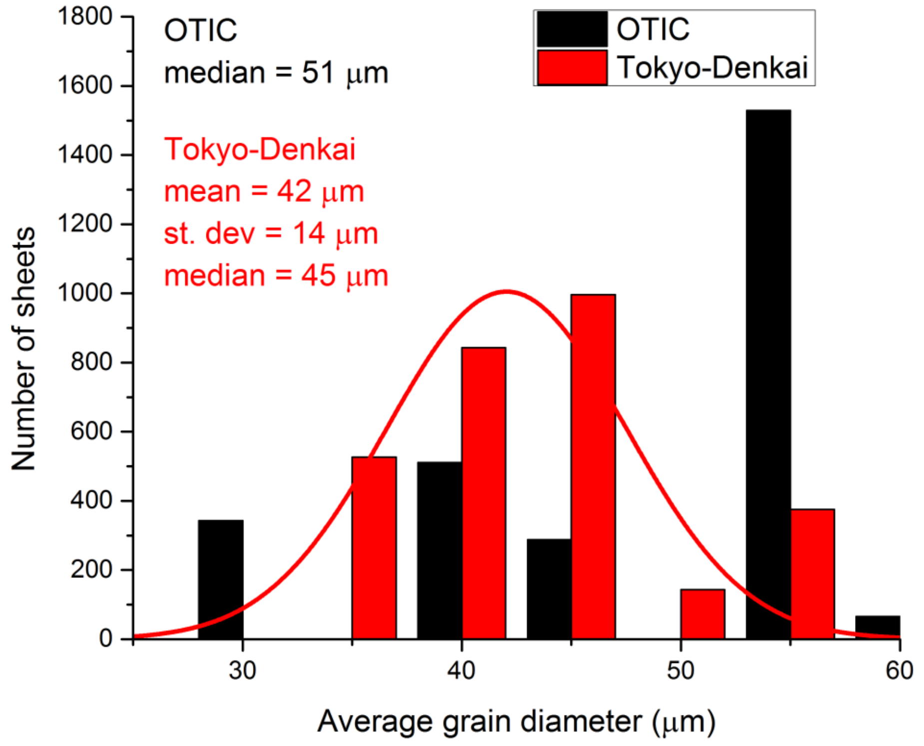
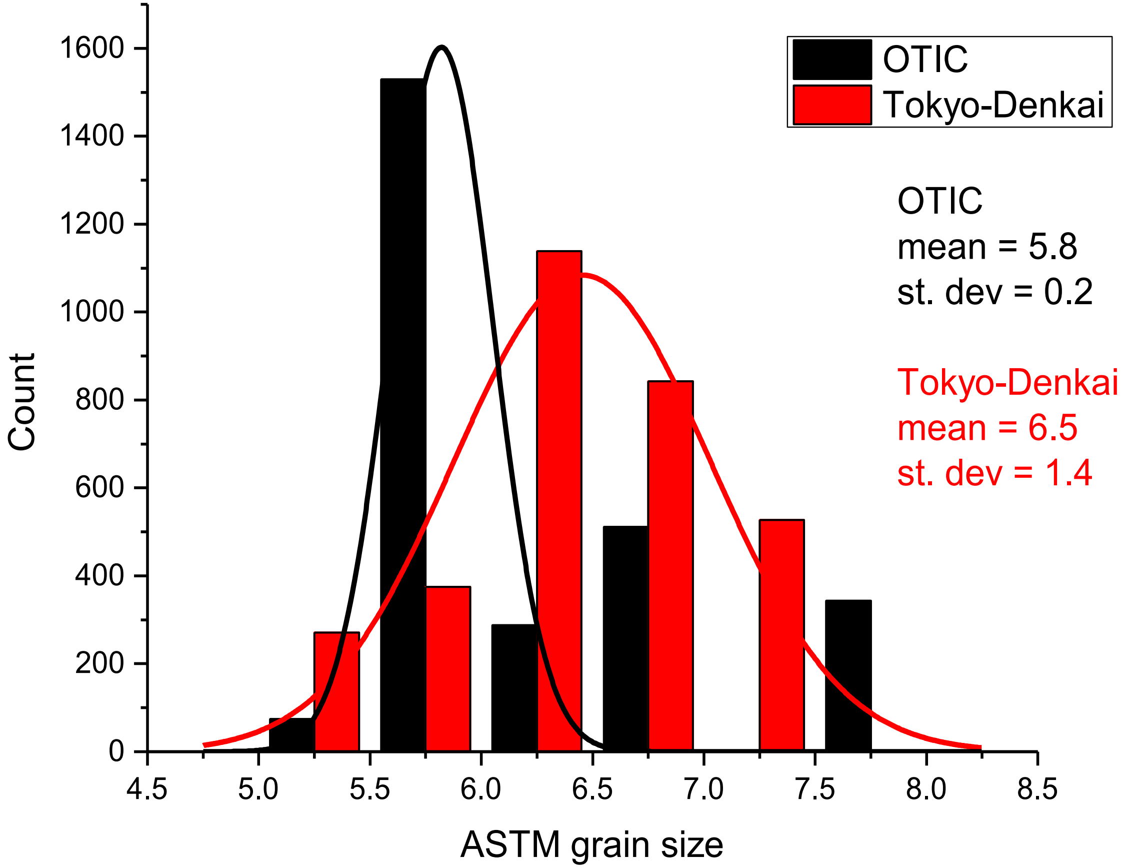
Elongation, AL 30 *	%	≥ 30
Hardness, HV (min. load 10 N)	-	**≤ 60**

- Each sheet is tested for hardness – must be less than HV 60, if harder they are sent back to Manufacture for annealing again.
- The hardest and softest sheet from each lot then goes to analysis (Done by DESY) for LCLS-II.
- Lots are defined by **heat lots**, not by ingot - TD uses more than one ingot per heat lot while Ningxia uses one ingot per heat lot.

LCLS-II Material Distribution

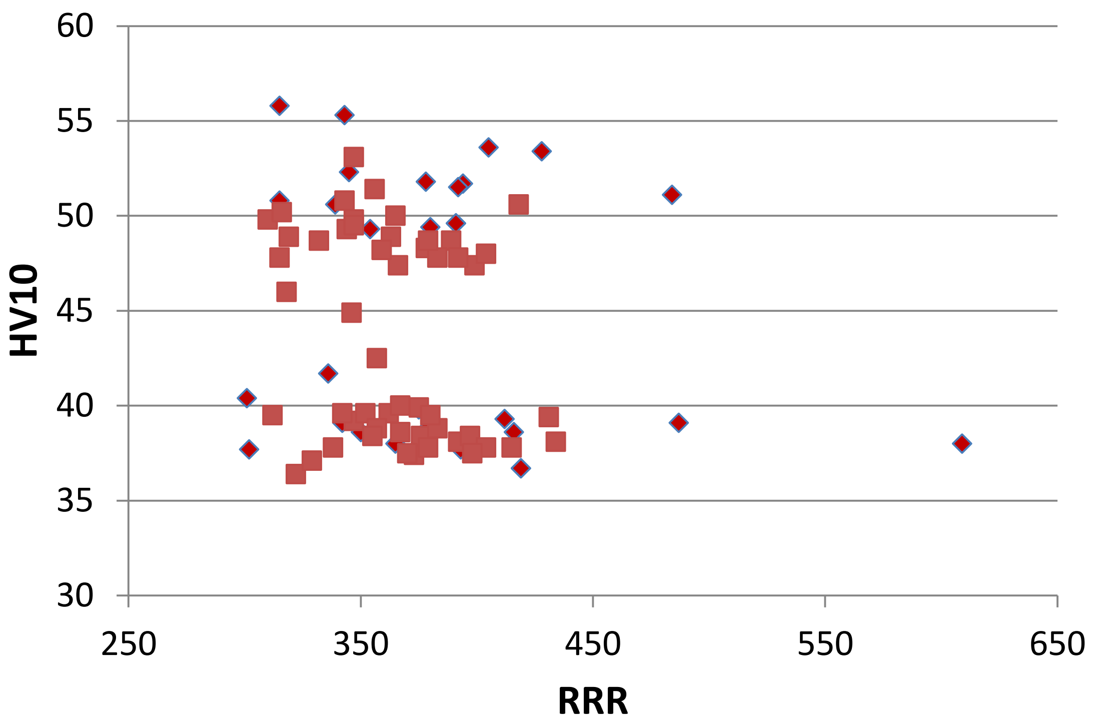
Gigi Ciovati - JLab plots

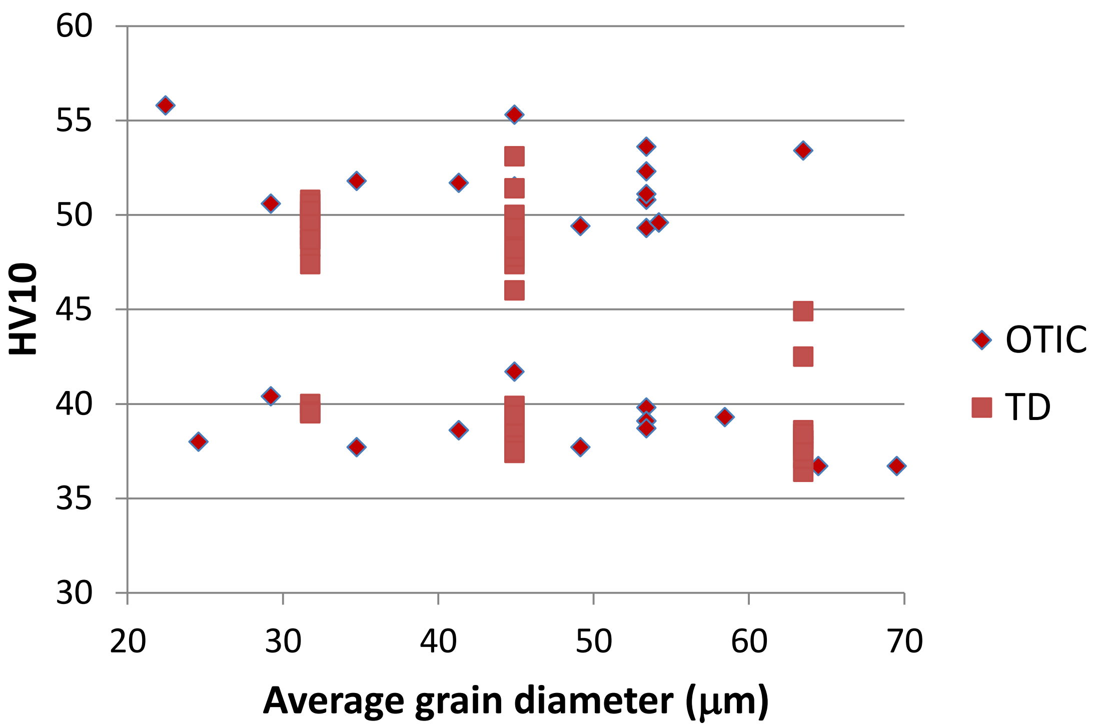




- TD distribution is more expected "Predominately ASTM 6.0"
- OTIC/NX in predominately "ASTM 5.8" but strange distribution

RRR and Hardness Values by Heat Lots





- Max harness of TD is on average less
- No ASTM 7.5 or 8 in TD material
- We think RRR NX outliers are not real (RRR from manufactures) – DESY verified >300 spec all lots
- Very hard 50-60 micron NX sheet might suggest more dislocation compared to TD

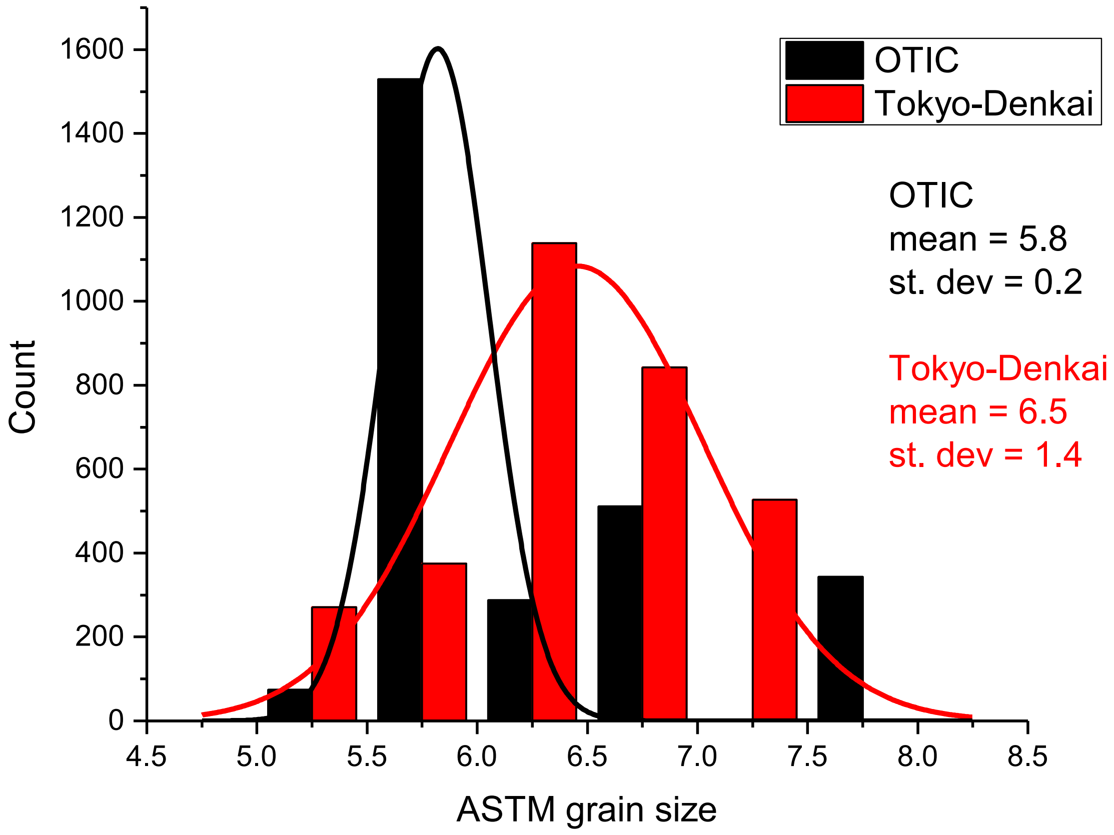
Gigi Ciovati - JLab plots

OTIC/NX Material sorting based on ASTM crystal size – this was a guess

Gigi Ciovati - JLab plots

- “Lot A” ~600-700 from softest/largest crystal sheets (ASTM 5 to <6)
- “Lot B” ~400-500 from the middle sheets (ASTM 6 to>7)
- “Lot C” ~150-200 from the largest/smallest crystal sheets (ASTM 7 to 8)

TD material was not sorted or actively tracked



Material Studies

- Tensile measurements of TD and NX samples

- EBSD NX (JLab)

- See EBSD from Sam's talk on TD to 900°C cross-section

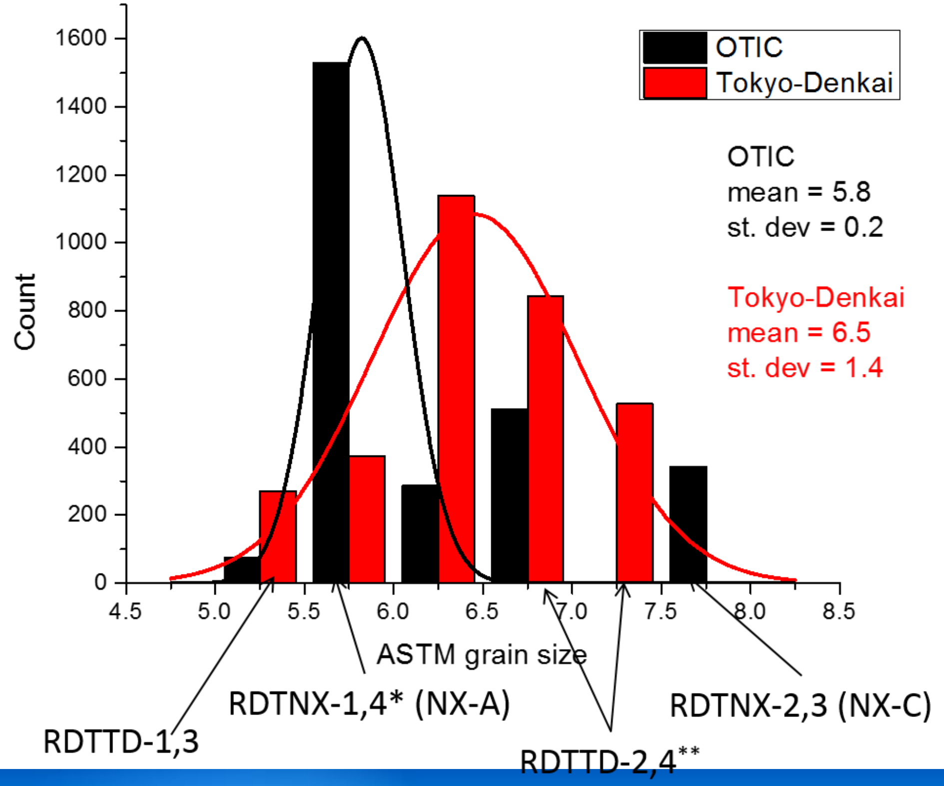
- Looking for changes that could correlate to flux expulsion.
- Goal, can we find a temperature to heat treat material to obtain good flux expulsion without the cavities getting too soft all while in production. 9 cell prototypes were tested for field flatness after shipping tests based on the data.

Reminder - Flux expulsion 800°C vs 900°C

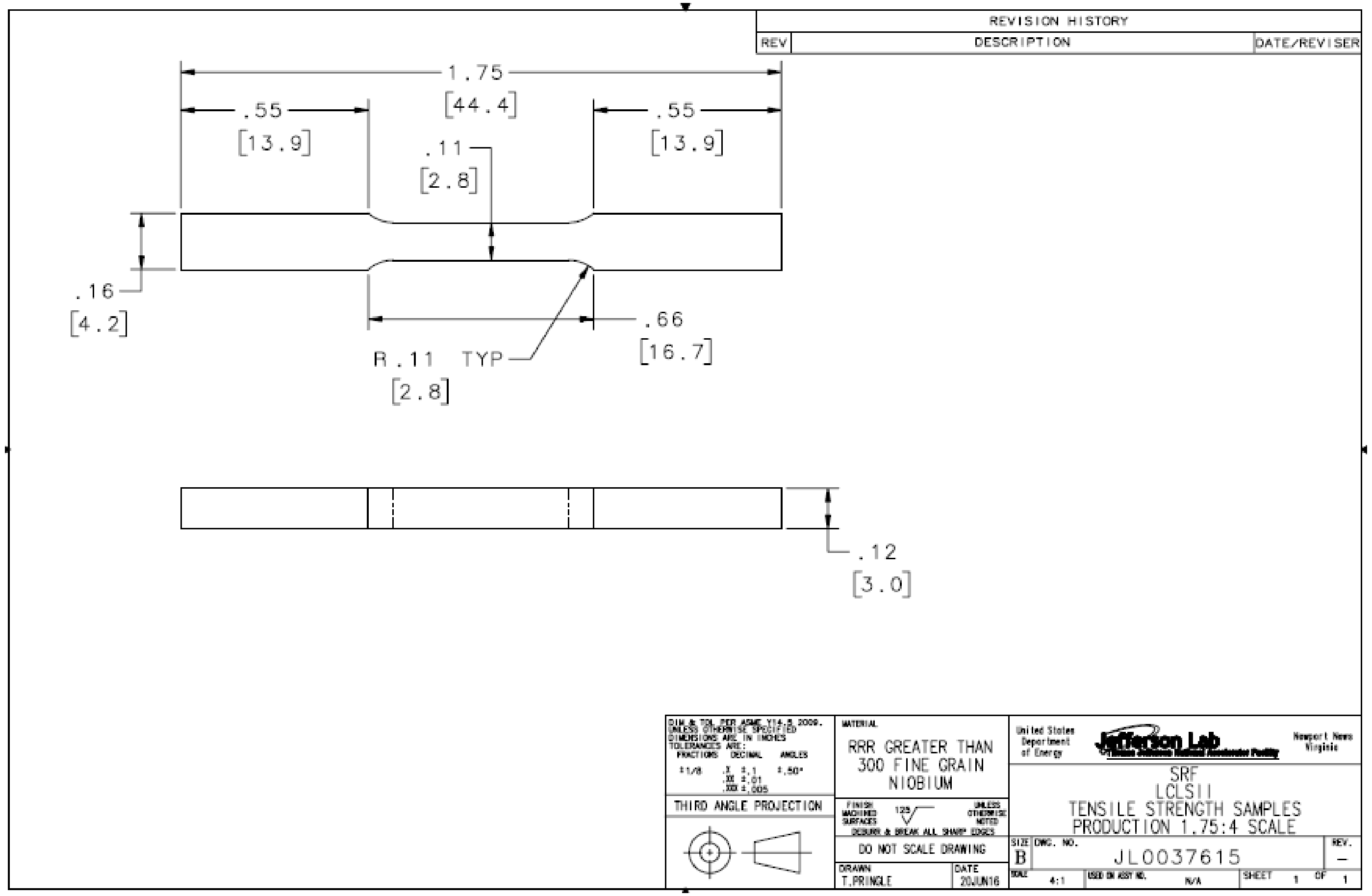
Ningxia Lot A ΔT	Flux expulsion baseline 800°C (JLAB data)	Flux expulsion post 900°C 3 hours (FNAL data)	Ningxia Lot C ΔT	Flux expulsion baseline 800°C (FNAL data)	Flux expulsion post 900°C 3 hours (FNAL data)	Tokyo Denkai Lot B-C ΔT	Flux expulsion baseline 800°C (FNAL data)	Flux expulsion post 900°C 3 hours (FNAL data)	Tokyo Denkai Lot A ΔT	Flux expulsion baseline 800°C (JLAB data)	Flux expulsio post 900°C 3 hours (JLAB data)*
2K	10%	46%	2K	2%	5%	2K	23%	69%	2K	14%	71%
6K	21%	**70%**	6K	15%	**35%**	6K	30%	**95%**	6K	26%	**90%**
10K	30%	80%	10K	20%	42%	10K	34%	100%	10K	NA	NA

- Absolute value between different labs and different test setups have not been fully calibrated, look at relative changes.
- Both TD material lots show excellent expulsion after 900°C
- NX-A material shows "ok" expulsion after 900°C, 950°C would maximize it.
- Lot "C" NX material shows minimal expulsion after 900°C, and acceptable after 975°C, 9 cell prototypes results later.

*Different test setup than 800°C data (800°C D7 and 900°C D3)
Table initially compiled by Anna Grassellino FNAL, modified here



Tensile Measurements - Samples

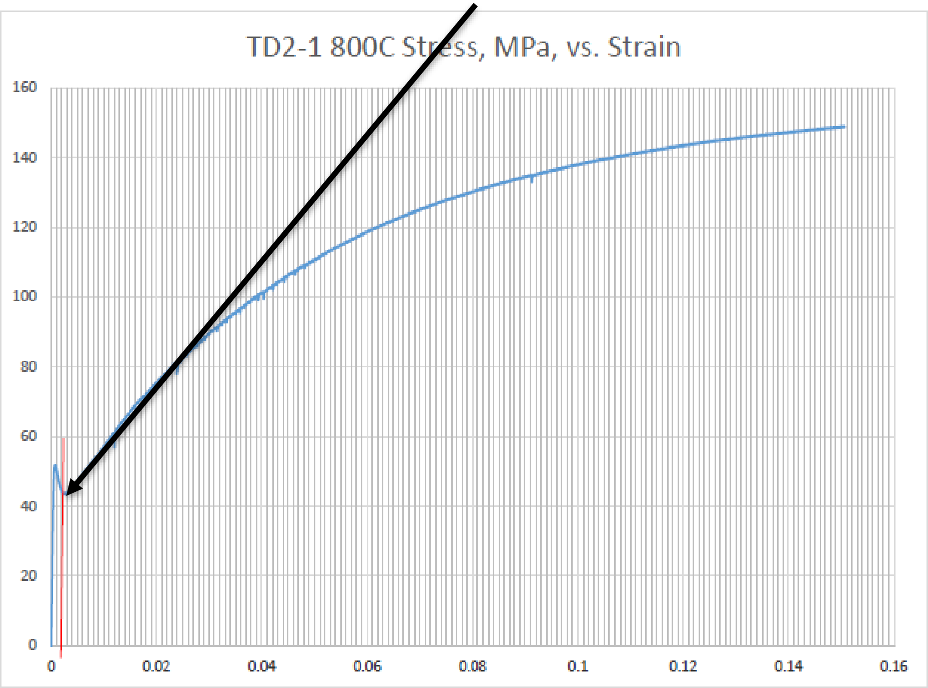


Much smaller than standard because they had to come from sheet corners.

Samples were heat treated after cutting – this may make all data invalid. (the choice of using sheet corners after cutting was a compromise because of timing.)

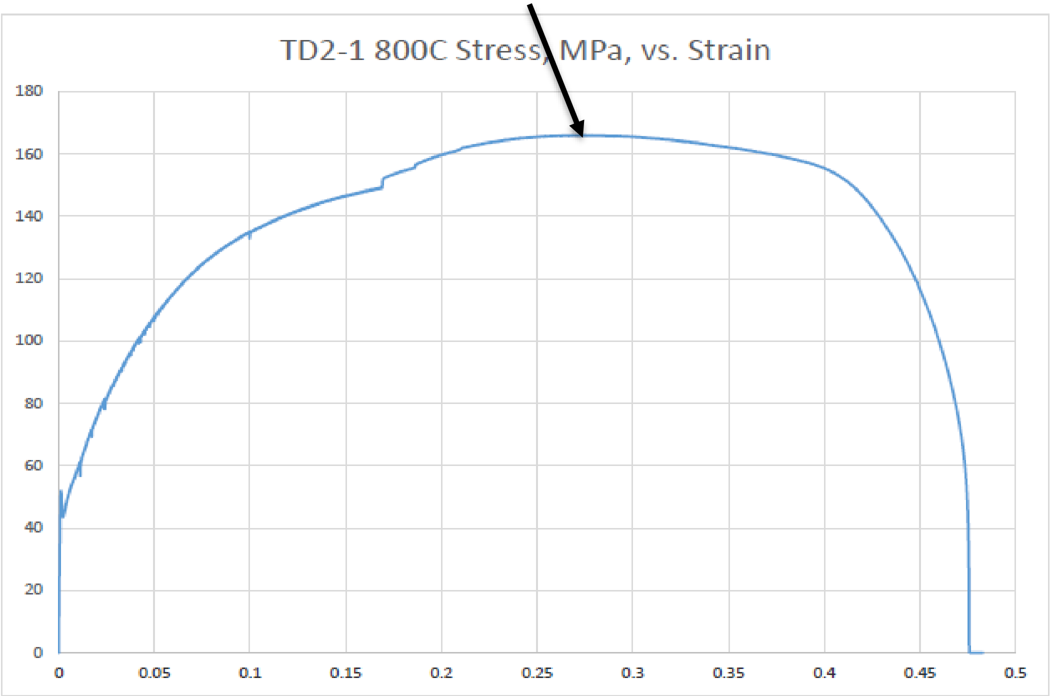
Tensile Measurements - Analysis

Yield strength (YS) = 44Mpa



$\sigma_{0.2}$ offset yield point = 44 Mpa

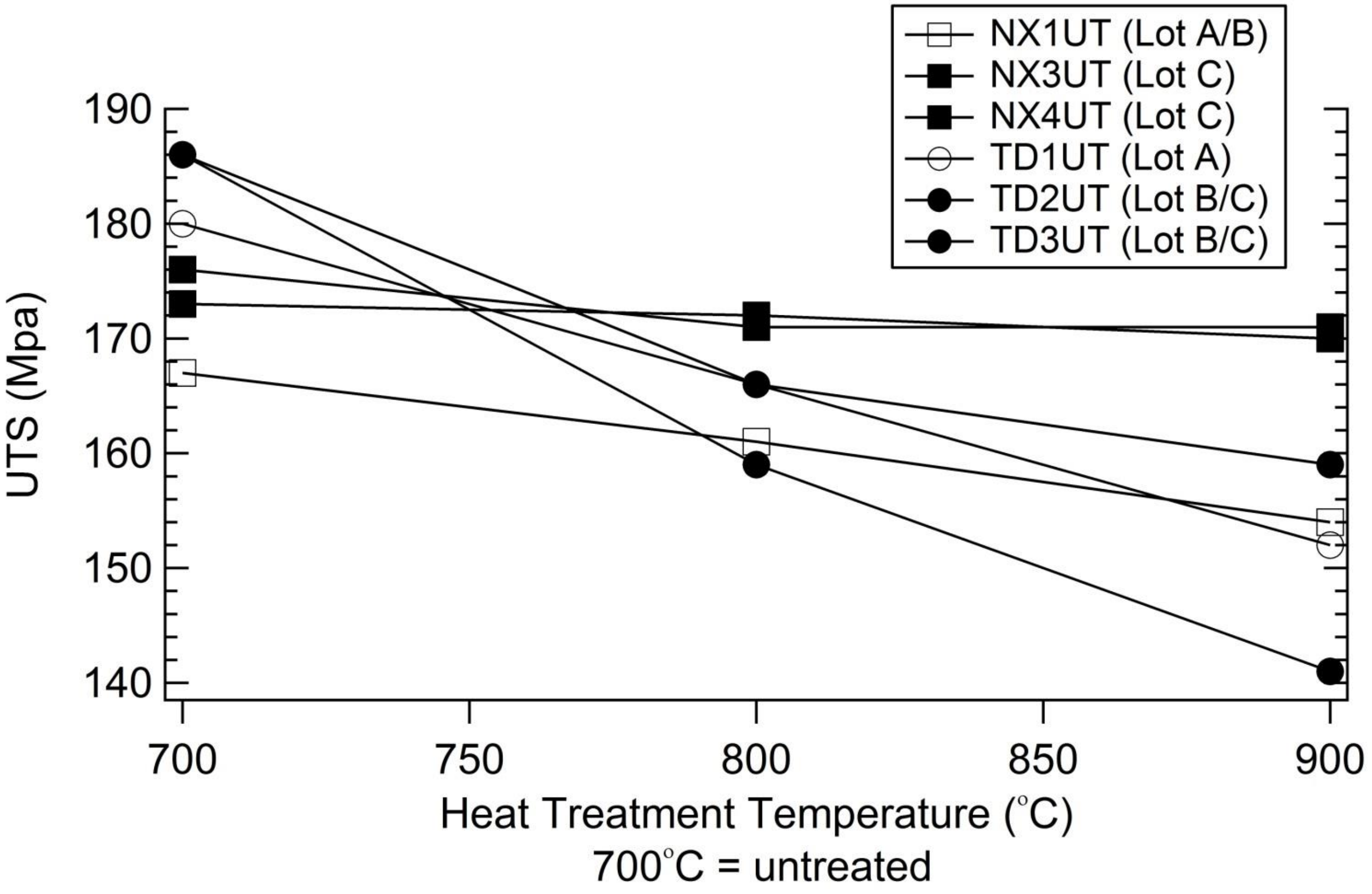
Ultimate tensile strength (UTS) 166MPa



Ultimate tensile strength = 166 MPa

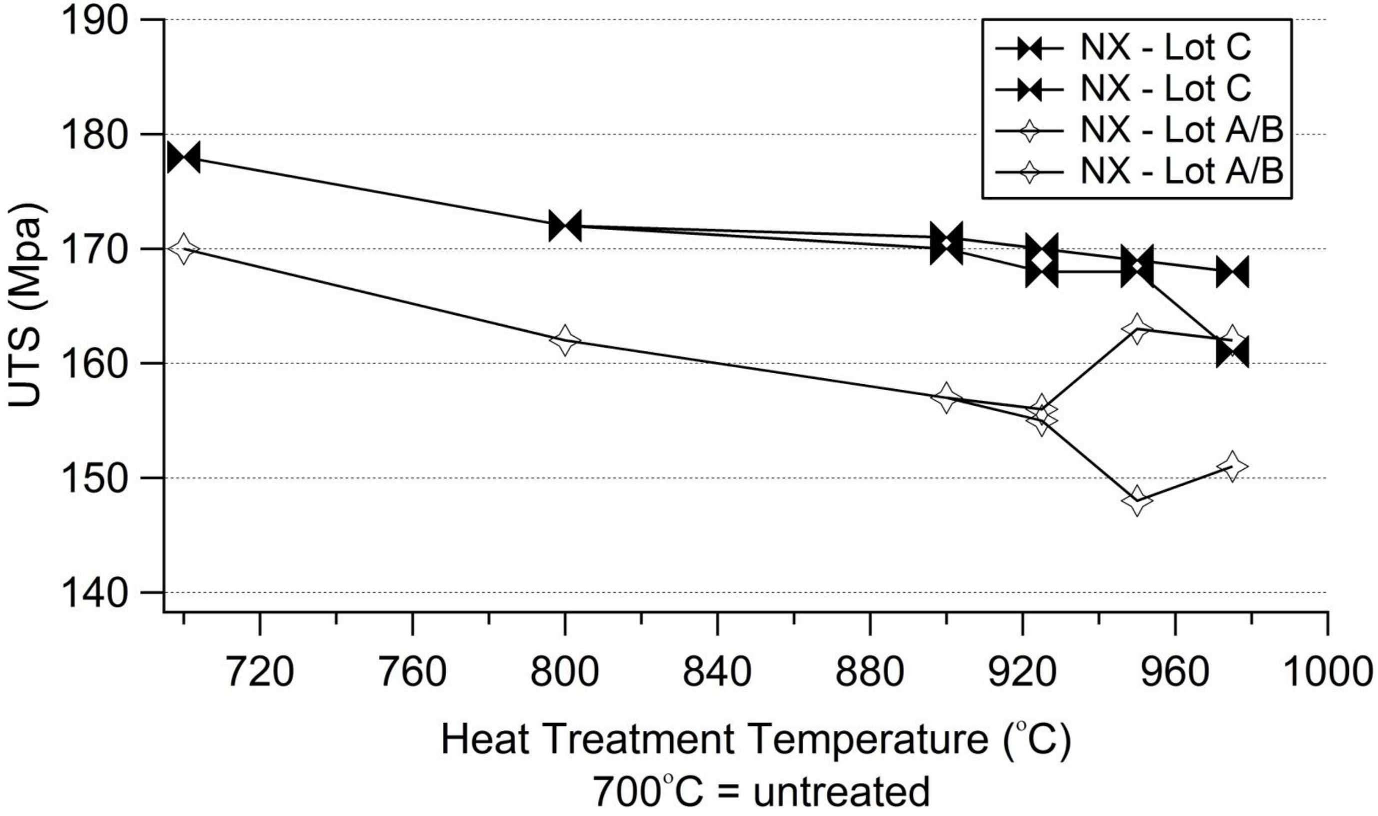
Elongation to failure: 48%

Tensile Measurements (UTS) NX and TD



- No change in Ultimate Tensile Strength for NX-C material
- NX-A slope change much less than TD
- The steep slope change from baseline to 800°C to 900°C on TD correlates with flux expulsion?

Tensile measurements (UTS) NX above 900°C

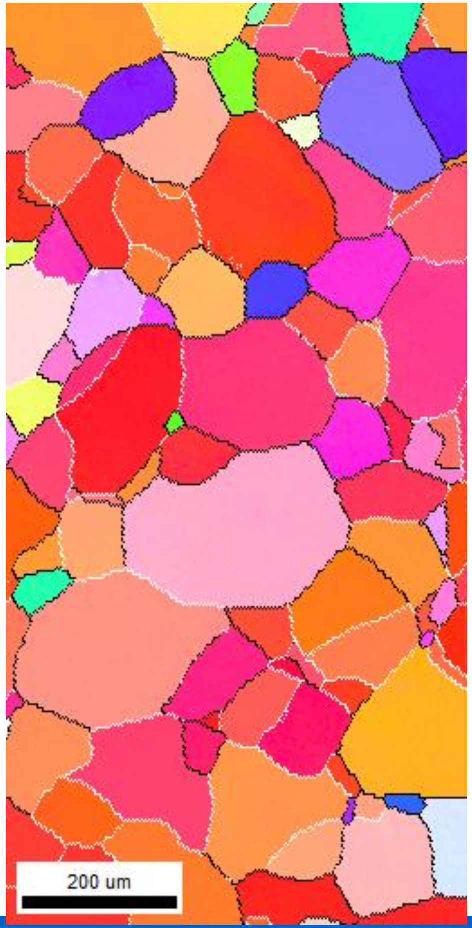


- 925°C data is strange and still under investigation – could be related to grain growth/sample size.
- Steep reduction in UTS see in TD material not seen here.

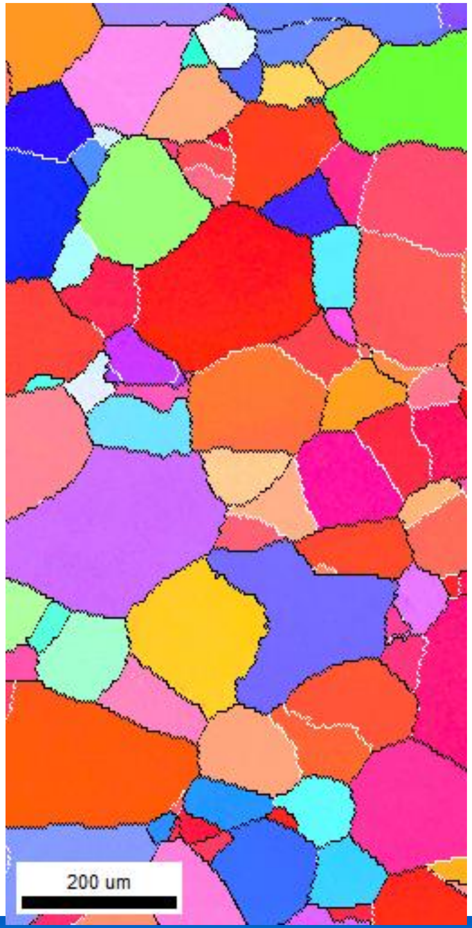
Also see S. Chen THPB028 for strange results on NX ASTM 5 samples (similar to NX-A)

Strange Grain Growth NX-A(H8) 900°C vs 975°C ~150-200µm BCP

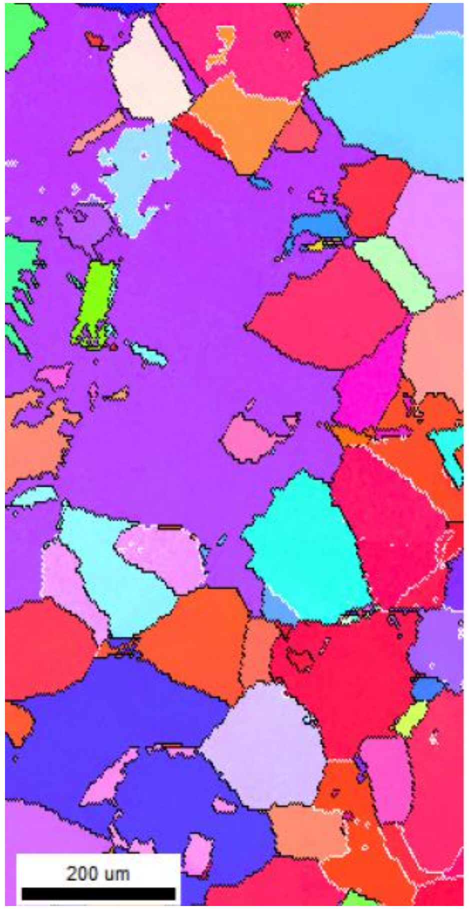
As found



900°C

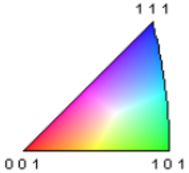


975°C



White boundaries are 5 – 15° subgrain boundaries i.e. distortion on grain boundary

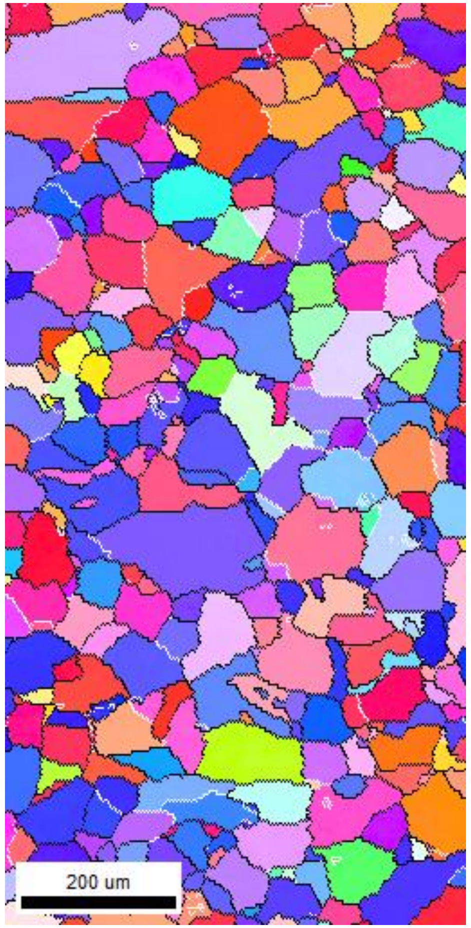
Non-random orientation shows non-uniform grain growth, did we not bcp the surface enough, are we seeing surface effects only?



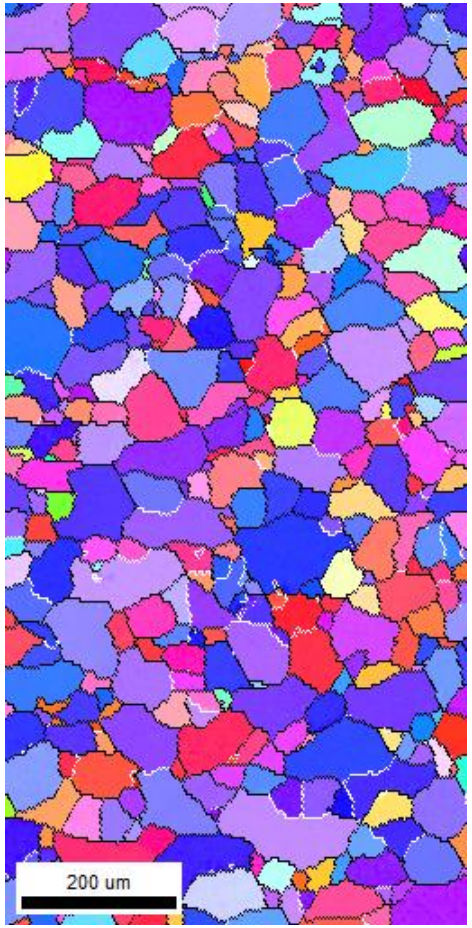
BCP may have not been enough – See S. Chen THPB028 250 micron BCP

Minimal Grain Growth NX-C (H3) 900°C vs 975°C ~150-200μm BCP

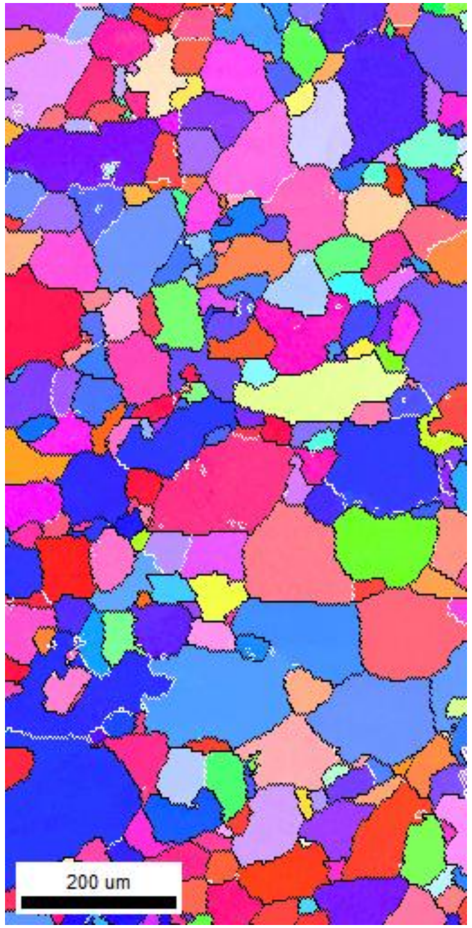
As found



900°C

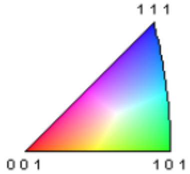


975°C



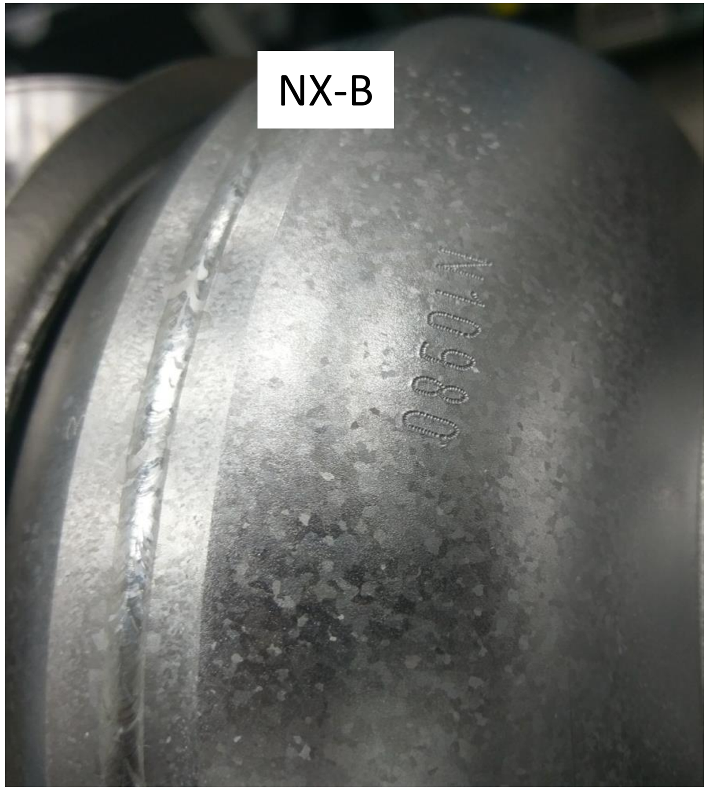
White boundaries are 5 – 15° subgrain boundaries i.e. distortion on grain boundary

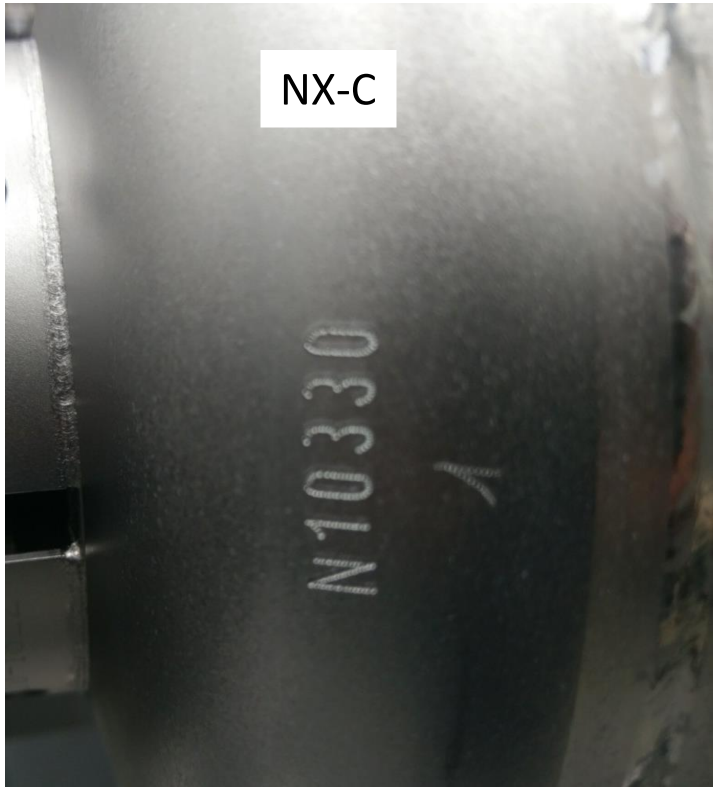
Some grain growth, but should be enough for “good enough” expulsion for LCLS-II.



BCP may have not been enough – See S. Chen THPB028 250 micron BCP

NX-B and NX-C mixed cavity @975°C - 9 cell prototype cavity





Cavity also received additional external BCP which may have removed the external damage layer
- Sam talk showed large grain growth on the center grain using cross section EBSD.

Material Studies Conclusions

- Given that a material traps flux, heat treating it until you see a reduction in UTS will reduce the flux trapping - observed.
- TD tensile strength results and EBSD from Sam's talk both suggest grain growth, but at a cost of softening of cavities compared to 800°C (additional feedback from Vendors on cavity tuning). Same for NX material but at a different temperature.
- NX-C will require up to 975°C and possibly over to reach the level of TD-900°C flux expulsion and equivalent cavity parameters for tuning. NX-C at 975°C might end up being better than TD at 900°C, stiffer cavities yet good expulsion.
- Large, non-uniform grain growth in NX-A suggest we did not do enough surface BCP, but also suggest all LCLS-II material requires large grain growth for good flux expulsion. (correlating to cavity results)

Thanks for listening – More research is needed

Material**	Recipe (HT/EP)	Vendor	Cavity count*
TD mixed	800°C/140µm	B	16
NX mixed (mostly B/C)	800°C/140µm	A	8
TD mixed	900°C/200µm	B/A	48/48
NX mixed (mostly B/C)	900°C/200µm	A	13
NX-A	900°C/200µm	B/A	28/2
NX-A	950°C/200µm	A	28
NX-B	900°C/200µm	B	18
NX-B	950°C/200µm	B/A	8/10
NX-C	900°C/200µm	B	2
NX-C	975°C/200µm	B	12
NX rework	TBD	B/A	?/?
TD rework	TBD	B/A	?/?

LCLS-II recipe by cavity vendor, material vendor, heat treatment/EP recipe by cavity counts - MOPB048