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**ENERGY**

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# Specifications and Notes for QA QC

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# Outline

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- Nb55Ti
  - Adapt ASTM B884 for composition
- Niobium
  - XFEL/007 Niobium 40
- SRF grade niobium
  - FNAL 371073-C
- Specification summaries
  - QA/QC notes specific to specs
  - Exceptions vendors are likely to request

## General goal of raw material specifications

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- All cavity fabrication and processing steps have margins for uncertainty around the nominal baseline
- Raw material specifications should protect these downstream margins without adding undue cost
  - A dime saved on a lower material grade might result in a dollar spent on a final performance limit
  - We have statistics only for standard material grades

## Nb55Ti alloy

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- Adapt ASTM B884, which applies to Nb47Ti
  - Well understood by vendors for Tevatron, LHC, and MRI wire
- Modify chemical requirements per table below
- Keep other requirements for properties, workmanship, sampling, etc.

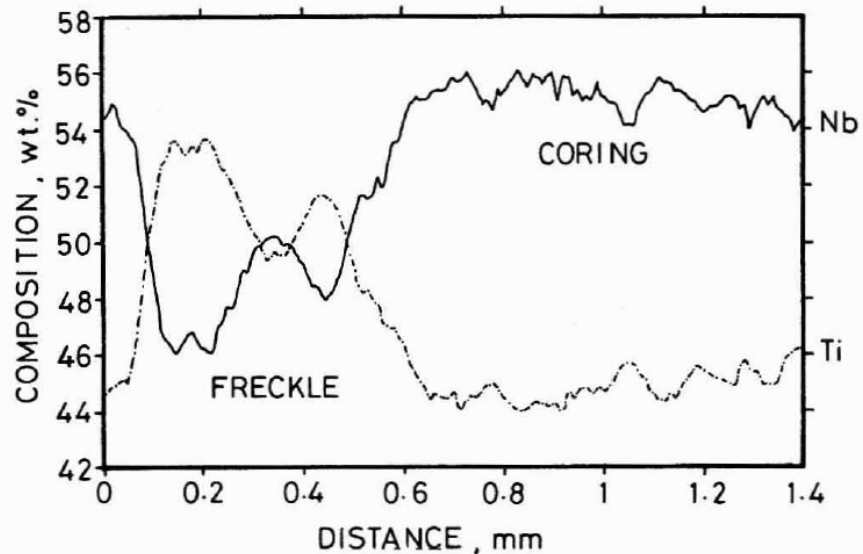
Element	Ingot Max, ppm
Al, Cr, Cu, Ni, Si	100 ea.
Fe	200
Ta	2500
O	1500
N	150
C	200
H	45
<b>Ti</b>	<b>54 to 56 % by mass</b>

Property	Spec
Hardness	170 DPH
Heat	Annealed
Grain size	2.5 for parts over 2", 4.5 for smaller parts

## Nb55Ti Alloy - 2

- Require flash radiograph to detect lack of control over melt
  - Several vendors understand and can provide this ingot QA
    - <http://fs.magnet.fsu.edu/~lee/pubs/smrg116flashradiographstd.pdf>
  - Ti freckles will show up if melt is not controlled
    - Recent experience with Nb55Ti flange
    - Risk for welds and vacuum: Martensite is possible if Ti too rich

P. J. Lee, and D.C. Larbalestier, in *Composite Superconductors*, ed. K. Osamura, T. Matsushita, P.J. Lee, and S. Ochiai, pub. Marcel Dekker Inc., New York, pp. 238-258, 1994



# XFEL Niobium 40

- XFEL/007 is nearly equivalent to ASTM B393 Grade 1
  - Chemical compositions of XFEL spec are tighter – too tight?
  - No other concerns – both specs are understood very well, quite stable in manufacture, and used daily by vendors (ITER)

Element	Ingot Max, ppm – XFEL	Ingot Max ppm - ASTM
Mo, Fe, Ni, Si	(each) 50	50; Mo=100
Ta	1000	1000
Ti, Hf	50	200
W	100	300
O	100	150
N	50	100
C	50	100
H	15	15

Property	XFEL Spec
RRR	$\geq 40$
Heat	95% Rx
Elongation*	$>20\%$ @ $10^{-3}/s$ transverse
Hardness	$<100$ HV10
UTS	$>125$ MPa
YS (0.2%)	$>75$ MPa

\*ASTM B393 requires  $10^{-4}/s$  through yield point

## SRF grade niobium

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- FNAL 371037-C is very close to XFEL/007 and ASTM B393
  - Vendors know these specs well and have specific manufacturing activities to comply with them
- FNAL 371037-C has evolved to address changes in vendor patterns and cavity performance statistics
  - Vendors appear to have found **at least 2 paths** to comply with sheet specifications
    - These paths produce different half-cell appearance and different response to processing, although both still qualify for Q(E)
  - High values of yield strength and hardness, and low values of elongation, were found to correlate with increased spread in cavity performance distribution
    - Increased spread can instigate more reprocessing steps

## Changes in vendor patterns

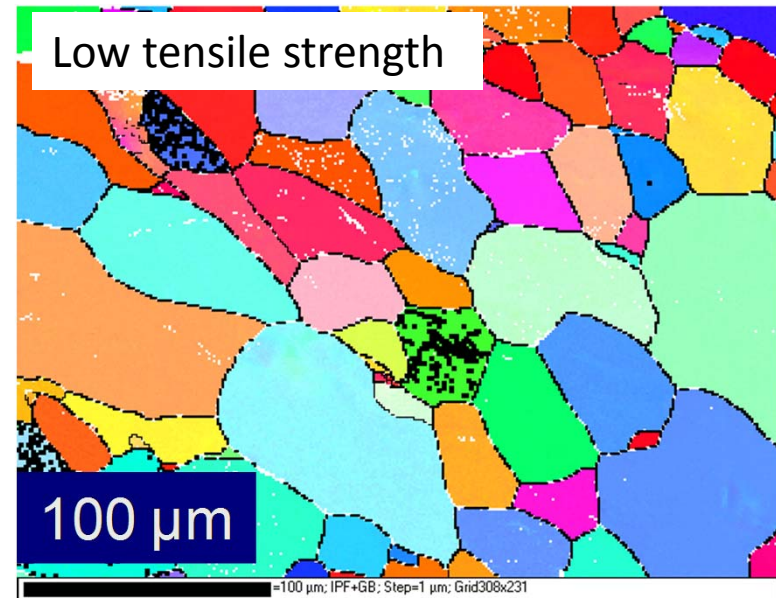
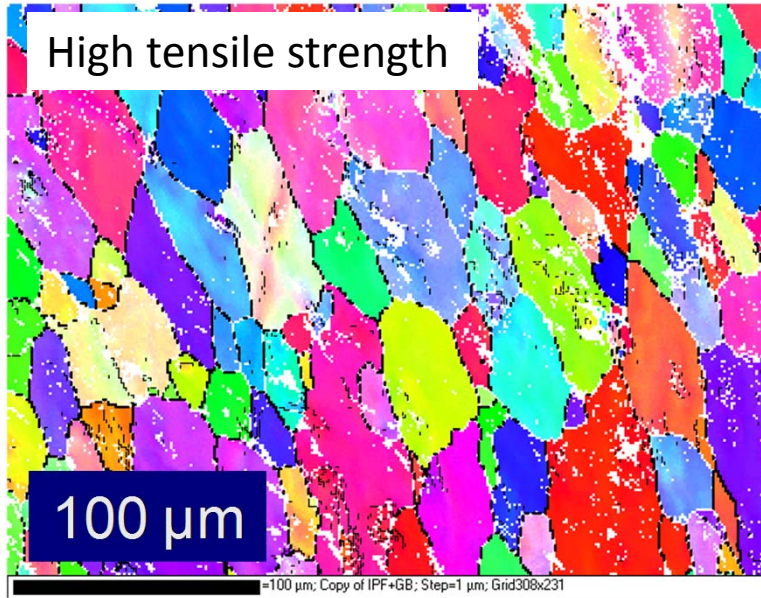
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- Two methods to meet sheet spec:
  - Start with RRR 300, forge + roll, anneal back to 300
    - (+) mechanical properties
    - (-) grain size blows up, exceptions requested
  - Start with RRR 400, forge + roll, matrix anneal with rolling
    - (+) RRR and grain size simultaneously met
    - (-) local variations of mechanical properties, cold work residues
    - Exceptions requested under 371037-C, **not captured by XFEL**
- 371037-C is a strategy to reduce risk
  - Well-annealed material appears to be more tolerant of variations in chemical polishing
    - Wider margins for downstream processes
  - Priority: 1) mechanical props, 2) RRR, 3) grain size

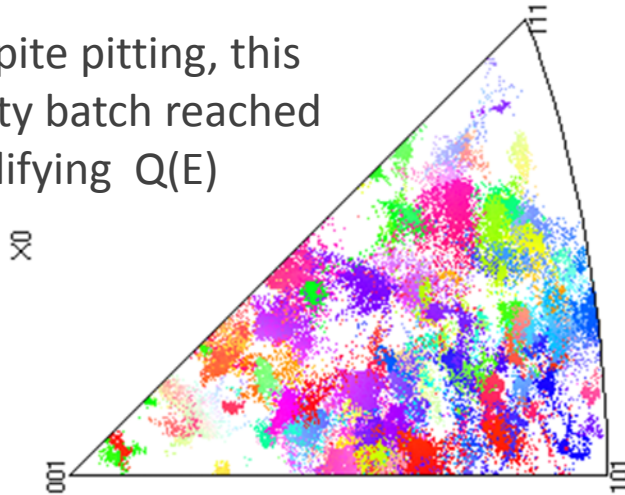


# Comparison of fabrication paths

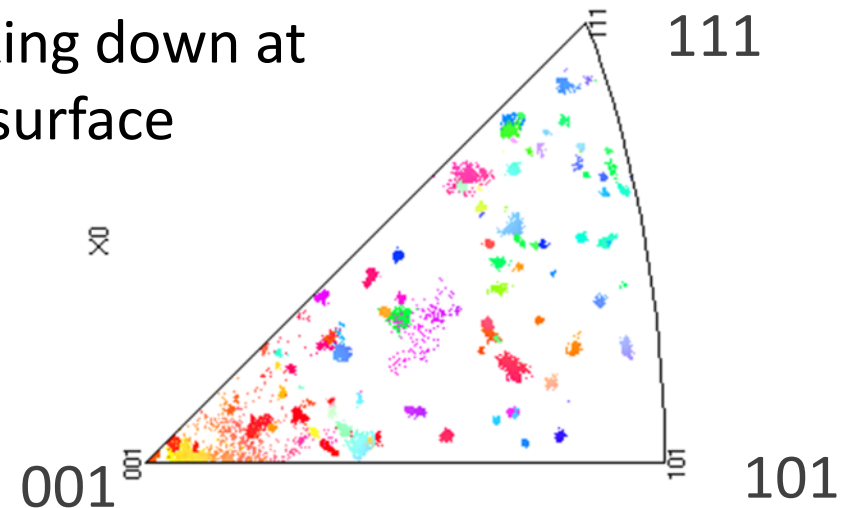
*Both are RRR 300, both are 100% Rx by optical micrograph*



Despite pitting, this cavity batch reached qualifying Q(E)

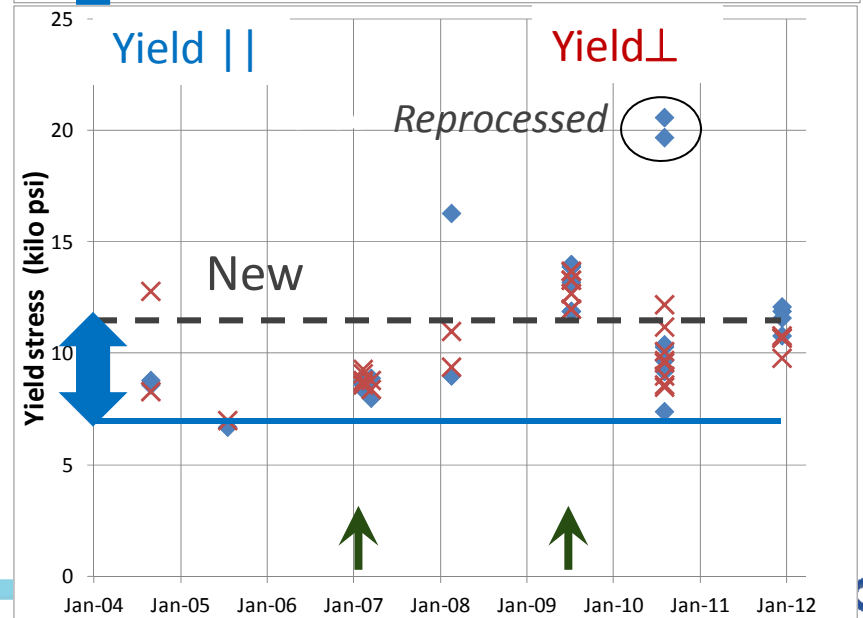
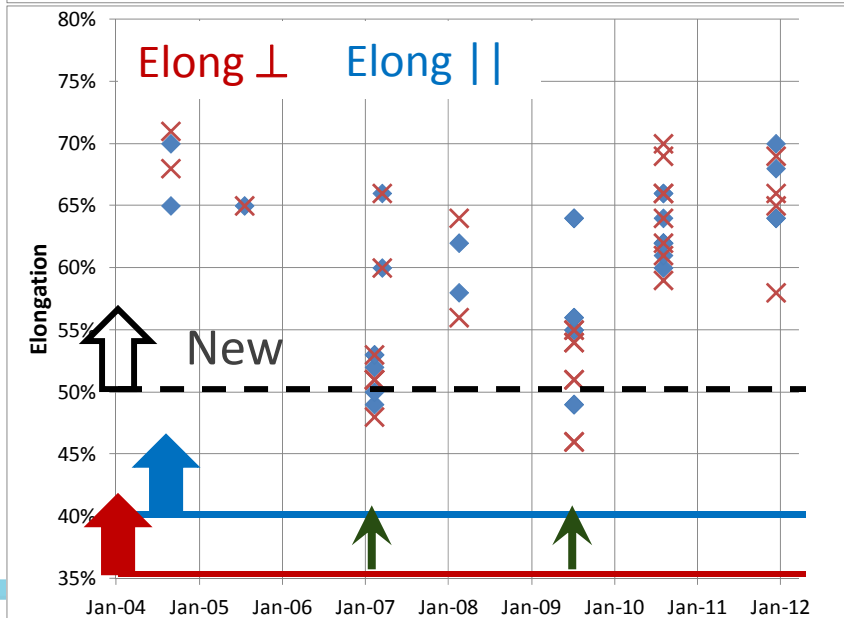
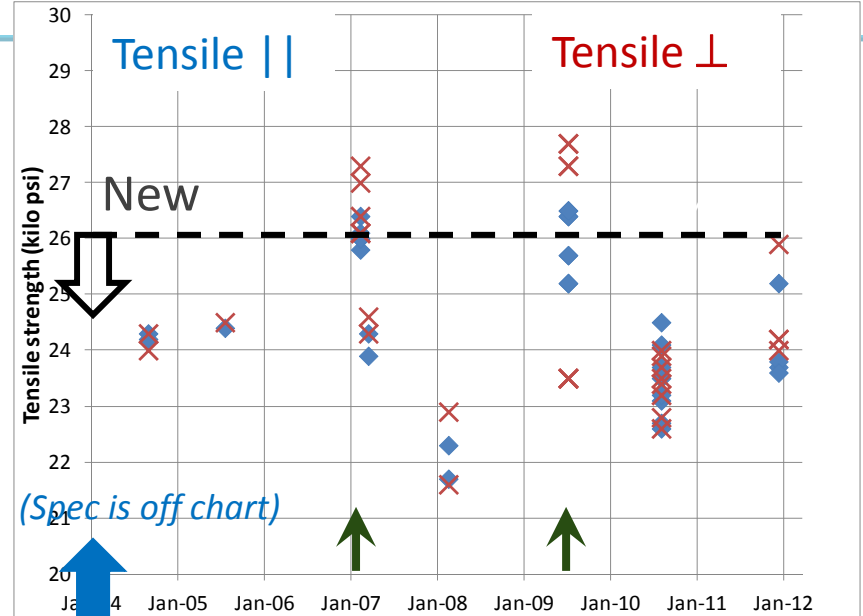
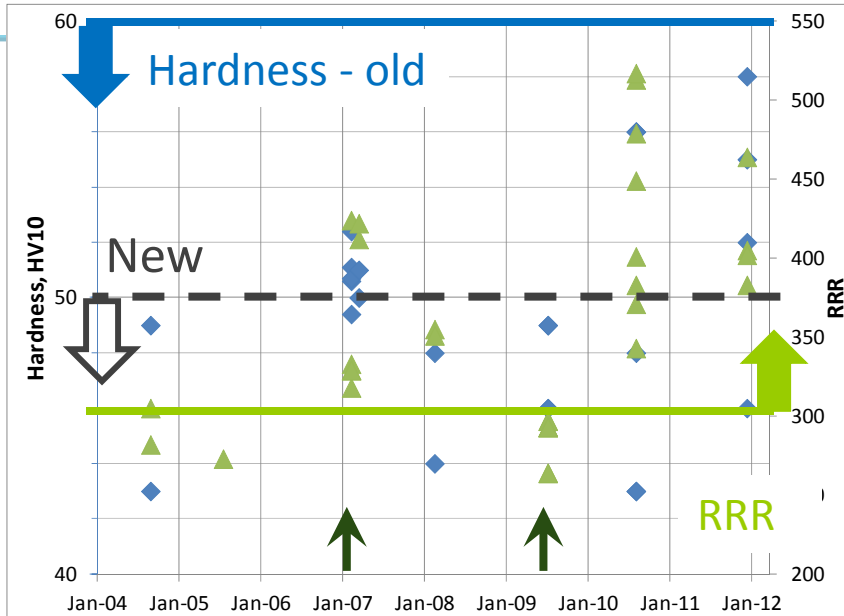


EBSD, looking down at the sheet surface



# FNAL spec was modified based on cavity statistics

Arrows are cavity batches for which Q(E) improved more slowly with chemical processing



## FNAL 371037-C and comparison to other specs

Element	Ingot Max, ppm – FNAL*	Ingot Max, ppm – XFEL	Sheet, max ppm – FNAL**	Sheet, max ppm – XFEL
Fe, Si, Ni	50, 50, 30	30, 30, 30		
Mo, W	xx, 70	50, 70		
Ta	1000	500		
Ti, Zr	50, 100	50, xx		
C	30	10	30	10
N	30	10	30	10
O	40	10	40	10
H	5	2	5	2

\*371037-C calls out chemical requirements to comply with ASTM B393

\*\* RRR requirement overrides chemical requirement

## FNAL 371037-C and comparison to other specs

Property*	FNAL	XFEL	B393-05
RRR**	>300, none < 270	>300	>260
Grain size (avg / local)	ASTM 5 / 4†	ASTM 6 / 5 none 4	ASTM 5 / 4
Heat	95% Rx, aspect < 2	100% Rx	90% Rx
YS‡ (MPa)	50 < YS < 75	50 < YS < 100	50 < YS
UTS‡ (MPa)	95 < UTS < 160	140 < UTS	95 < UTS
Elong.   , ⊥ %	>50, >50	>30, >30	>30, >30
Hardness	< 50 HV10	<60 HV10	<60 HV10

\* Properties shall not vary by more than 20% along and transverse to rolling direction

\*\* RRR = R(273)/R(10) for B393. A value of 260 is thus equivalent to 286 under the FNAL and XFEL definition. FNAL 371109 is an international standard in WG draft status.

† ASTM E3 governs metallography, ASTM E-112 governs grain size determination, and ASTM E-883 governs microscopy.

‡ ASTM E-8 governs mechanical testing, ASTM E-384 governs hardness

## Other specifications for niobium and SRF niobium

Topic	XFEL – Nb40	FNAL – SRF	Driver or Note
Defects	<15 $\mu\text{m}$	<25 $\mu\text{m}$	Taylor-Hobson profilometer; Visual inspection limit
Flatness	1 mm (3%)	6%	Sheet scanning and marking
Marking	Sheet S/N	Sheet # and 12 o'clock	Sheet and location reference to half-cell travelers
QA Sampling	2 shts. / lot	2 shts. / lot	Each lot is defined by ingot and anneal batch
QA Reporting	RRR, YS, UTS, elong, GS, chem	RRR, YS, UTS, elong, GS, chem	Mechanical tests along and transverse to rolling dir.
Bend test (optional)	1 mm radius	TBA	No orange peel
Finish	10 $\mu\text{m}$ / 10 $\mu\text{m}$	“Light” / “light”	Grind followed by pickle

## QA / QC notes

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- RRR takes precedence over particular interstitial impurity content
  - In practice,  $RRR > 300$  forces measured values to 10-15 ppm but not  $<10$ ppm
- Mechanical properties take precedence over grain size
- Risk: sheets subcontracted to equipment not dedicated to niobium
  - Many magnetic ferrites do not rust
  - ECS is excellent safeguard vs. magnetic junk
- Unknown: statistical spread in properties
  - Reduction of spread should increase downstream margins
  - 2 sheets per lot is too small of a sampling for lot statistics, even though useful for ingot-to-ingot variation
  - RRR from one ILC batch, 20 shts:  $COV = 10\%$  (i.e.  $300 \pm 30$ )
  - Half-cell corners are useful for sampling and testing
    - Mark sheet ID in all 4 corners?
  - Vendor can track sheets front-to-back on roll, “Hook ID” in furnace

## Readiness for procurement

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- Nb55Ti
  - Specification will be based on well-known, accepted, and widely used spec for MRI wire ingots.
  - Flash radiography is optional QA addition
- Grade 1 niobium
  - RRR40 spec is nearly identical to well-known, accepted, and widely used spec for superconducting wire
  - RRR40 spec has been tested for XFEL procurement
- SRF grade niobium
  - FNAL 371037-C is very close to XFEL and B393 specs, which are widely used and accepted.
  - Adjustments respond to recent vendor activity to reduce risk