Material, RRR of niobium sheet and weld, other QA

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Nb ordered on 02-NOV-06 from Wah Chang

- Batch 1 delivered spring 2006, had poor formability
- "Batch 2" better formability due to different production and anneal

- 120 Nb squares 10.433 x 0.110 inch
  - Reference Sheets FAB-200224 and FAB-200227

- 1 sheet 17 x 66.25 x 0.118
  - Batch FAB-200336
- 1 plate 7 x 12 x 0.4
  - Batch FAB-200806
- 1 sheet 14.13 x 54 x 0.118
  - Batch FAB-200337
- 1 plate 1.875ϕ x 25
  - Batch FAB-200401
- 1 Ring 5.315 x 2.953 x 1.063
  - Batch FAB-200397
- 1 Rod 0.940ϕ x 18
  - Batch FAB-200400
- 1 Rod 0.5ϕ x 10
  - No batch #

Composition of INGOT

Impurities	Spec of ppm in weight*	Stated by vendor
Ta	≤ 500	370 – 390
W	≤70	< 30
Ti	≤ 40	< 40
Fe	≤ 30	< 25
Mo	≤ 50	< 30
Ni	≤ 30	< 20
H	≤ 2	< 3
N	≤ 10	< 20
O	≤ 10	< 30
C	≤ 10	< 20

*Multiply interstitial wt. ppm by 5 to get mol ppm; divide Ta wt. ppm by 2 to get mol ppm

Not checked by Fermilab

RRR > 300

Batch	Vendor test	Fermilab test
Batch FAB-200224	415, 397	406
Batch FAB-200227	412, 422	469
Batch FAB-200336	426, 426	427
Batch FAB-200337	416, 422	390

Batch 1 sheet 217 RRR was 409

Mechanical property 1

Tensile strength Spec: > 14000 psi

Batch	Vendor test	Fermilab test
Batch FAB-200224	23800-24200	27847
Batch FAB-200227	23900-24600	27267
Batch FAB-200336	23100-23800	25091
Batch FAB-200337	23400-23700	27412

Yield strength Spec: > 7000 psi

Batch	Vendor test	Fermilab test
Batch FAB-200224	7400-9000	10587
Batch FAB-200227	8000-8900	10732
Batch FAB-200336	8000-8600	13053
Batch FAB-200337	8600-9800	13778

Mechanical property 2

Elongation Spec: > 35 %

Batch	Vendor test	Fermilab test
Batch FAB-200224	58-66	65
Batch FAB-200227	60-66	66
Batch FAB-200336	62-65	65
Batch FAB-200337	64-66	66

Strain hardening coefficient:

Batch	Vendor test	Fermilab test
Batch FAB-200224	0.34-0.44	0.36
Batch FAB-200227	0.36-0.38	0.39
Batch FAB-200336	0.35	0.26
Batch FAB-200337	0.34-0.37	0.27

Mechanical property 3

Hardness: < 60 HV10

Batch	Vendor test	Fermilab test
Batch FAB-200224	45-48	
Batch FAB-200227	50-51	
Batch FAB-200336	44-45	
Batch FAB-200337	42-45	

Grain size: Batch 1 spec ASTM#6 global, #5 local Batch 2 ASTM#5 global

Recrystallization > 90

Batch	Vendor test	Fermilab test
Batch FAB-200224	100	100
Batch FAB-200227	100	100
Batch FAB-200336	100	100
Batch FAB-200337	100	100

Grain size: finer than ASTM 5

Batch	Vendor test*	Fermilab test
Batch FAB-200224	6-6.5	5.5
Batch FAB-200227	6.5	5
Batch FAB-200336	6.5	5
Batch FAB-200337	6	5

* Grains no larger than ASTM 4

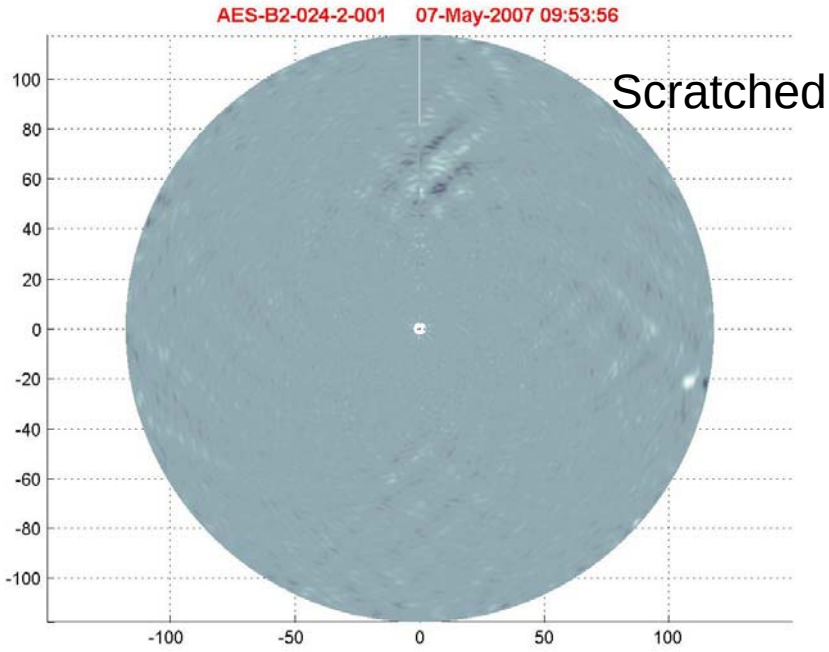
Eddy current scans – few defects if any

Eddy Current Scanning Summary Wah Chang material for AES cavity batch 2

Disk side	Vendor Batch #	RF OK	Unfiltered	Filtered	Visual Inspection	Inpected by
001_1		Yes	BMP file	JPG file	Light crease OR-279	O.F.
001_2		Yes	BMP file	JPG file	Clean	O.F.
002_1		No	BMP file	JPG file	Clean w. tiny pit MR-120	O.F.
002_2		Yes	BMP file	JPG file	clean	O.F.
003_1		Yes	BMP file	JPG file	Clean	O.F.
003_2		Yes	BMP file	JPG file	Clean	O.F.
004_1		Yes	BMP file	JPG file	Clean	O.F.
004_2		Yes	BMP file	JPG file	Clean	O.F.
005_1		Yes		JPG file	Clean, light mark, MR-205	O.F.
005_2		no		JPG file	Clean	O.F.
006_1		Yes		JPG file	Clean	O.F.
006_2		Yes		JPG file	Clean	O.F.
007_1		Yes		JPG file	Clean	O.F.
007_2		Yes		JPG file	Clean	O.F.
008_1		Yes		JPG file	Clean	O.F.
008_2		Yes		JPG file	Clean	O.F.
009_1		Yes		JPG file	Clean	O.F.

Sheets 2, 5, 11, 24, 32, 36, 55, 56, 57, 62, 64, 72, 75, 76, 79, 80, 82, 83, 90, 91, 95, 102, 108, 120 have minor flaws on one side

Sheets 98, 100, 109, and 119 were withheld due to defects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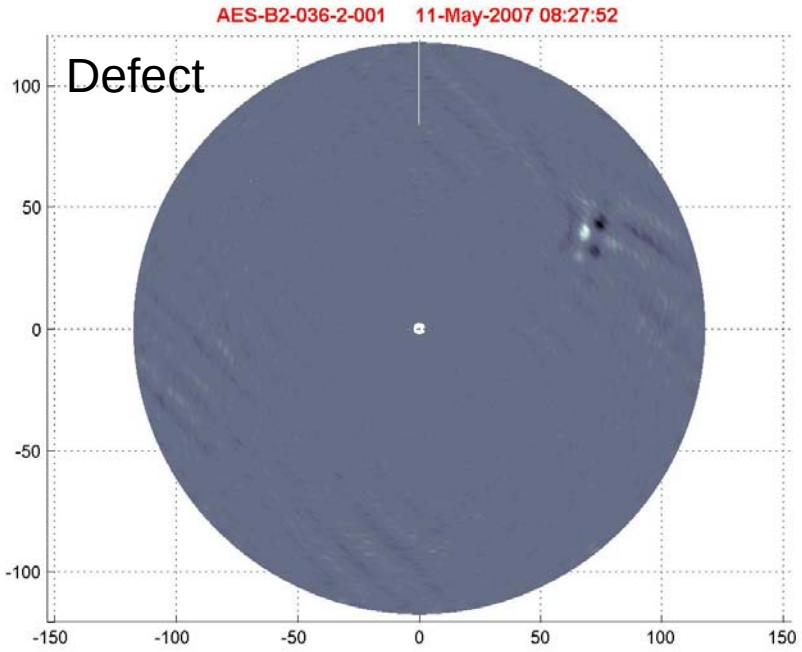
Scratched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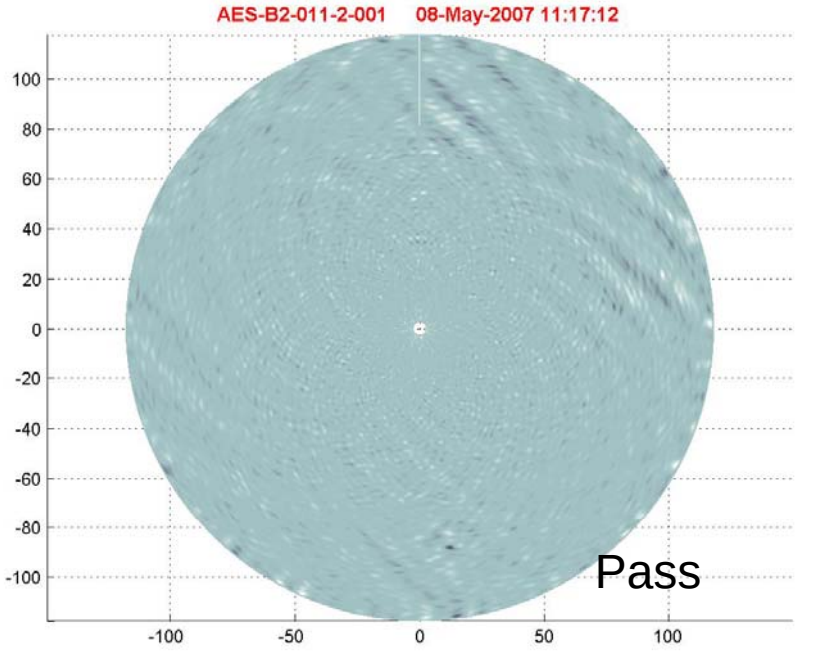
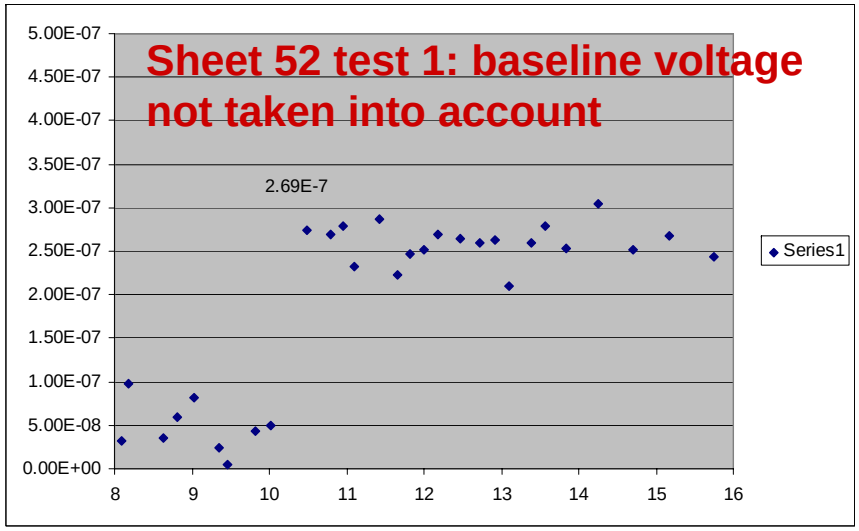
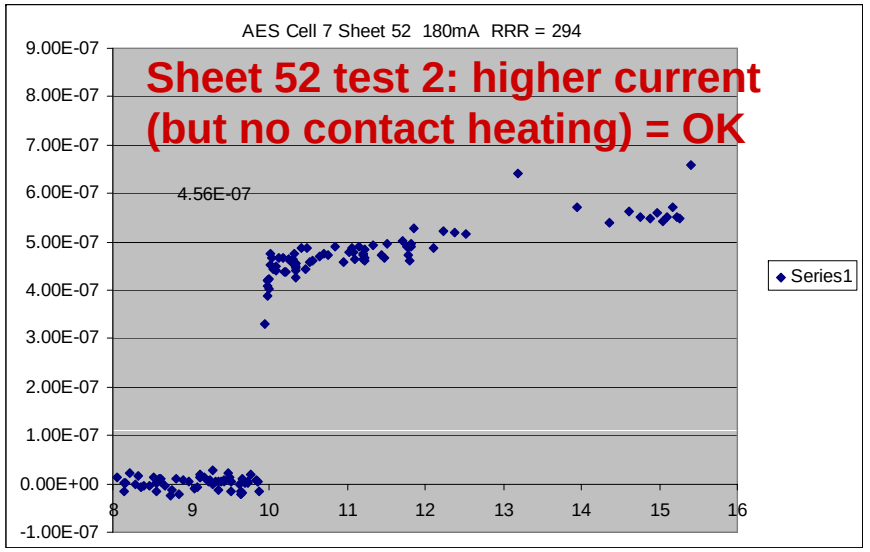
Defect


AES-B2-011-2-001 08-May-2007 11:17:12
Pass

RRR measurements of 9AES001 sheets — search for a smoking gun

- August 3rd RRR measurements on sheet:
  - cell 3 sheet 49 RRR 327
  - cell 3 sheet 115 RRR 269
  - cell 5 sheet 82 RRR 278
  - cell 5 sheet 111 RRR 280
  - cell 7 sheet 9 RRR 300
  - cell 7 sheet 52 RRR 253 289, 309 upon re-test
- All sheets now measured
  - Average = 296
  - Standard deviation = 30.7





Full results

Cell / Sheet	RRR		Cell / Sheet	RRR
1 / 012	289		5 / 111	278
1 / 029	274		6 / 008	321
2 / 039	325		6 / 108	349
2 / 022	263		7 / 009	300
3 / 049	320		7 / 052	289
3 / 115	294		8 / 087	310
4 / 075	338		8 / 023	267
4 / 065	288		9 / 048	364
5 / 083	278		9 / 089	312

RRR is lower than for test pieces

Batch 1

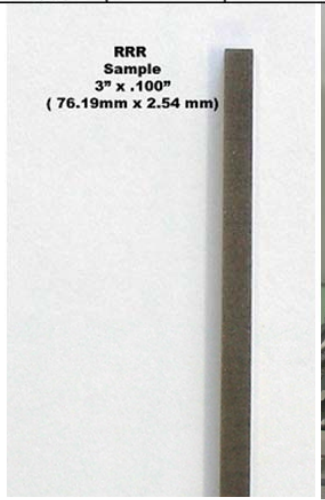
3R Measurements

Etch before HT* (microns)	0	10	40	100
Sample A	373	391	365	388
Sample B	381	388	385	380
* HT=750C,3hrs,10^-5Torr				

3R - ILC-AES b1	sample 1	sample 2	sample 3	sample 4
as received, from sheet 217	417	403		

3R of this material generally high: ~450 (spec 300)
Etching before Heat Treatment is common safety precaution – does not seem to affect 3R however

Mai 18th 2006



Batch 1 sheet weld: 303

Analysis – is there anything wrong with the material?

- Singer: RRR $\approx$ 14600 / (8.7 + 0.4 C_i)
  - C_i in mol ppm

    So, variation between 150 and 300 is a variation between 200 and 100 mol ppm,
- Koch et al: $T_c = T_{c0} - 0.93$ K / %Oxygen
  - 100 ppm oxygen makes T_c = 9.16 K instead of 9.25 K
- Ciovati et al: $B_p \sim \exp[(\Delta - \Delta_0) / k_B T_c]$
  - 100 ppm oxygen reduces B_p by only 1.3%

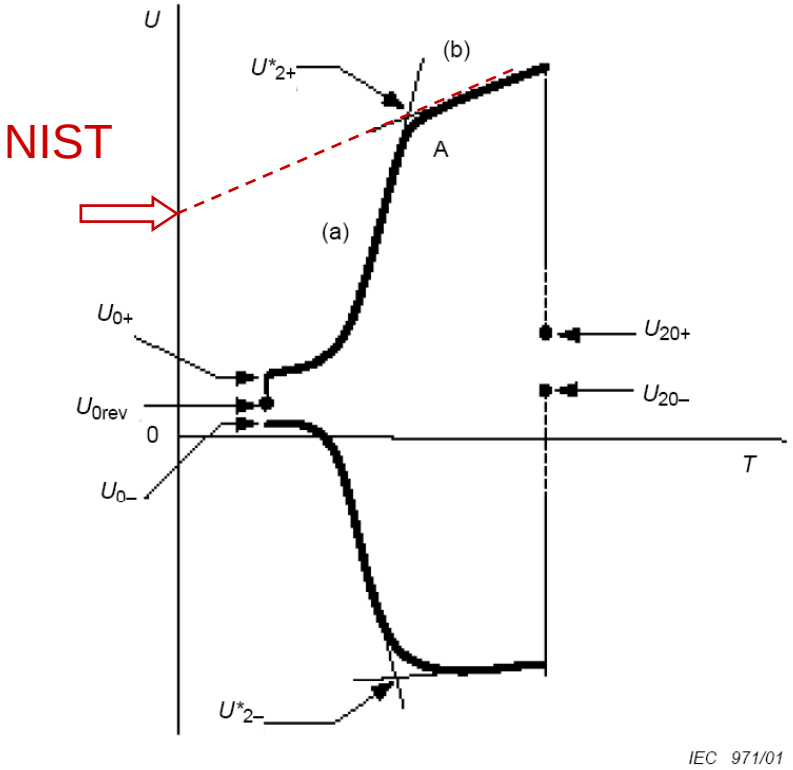
- *Implication: need unreasonably large pooling of defects to reduce B_p enough to explain results*

Why RRR = 300?

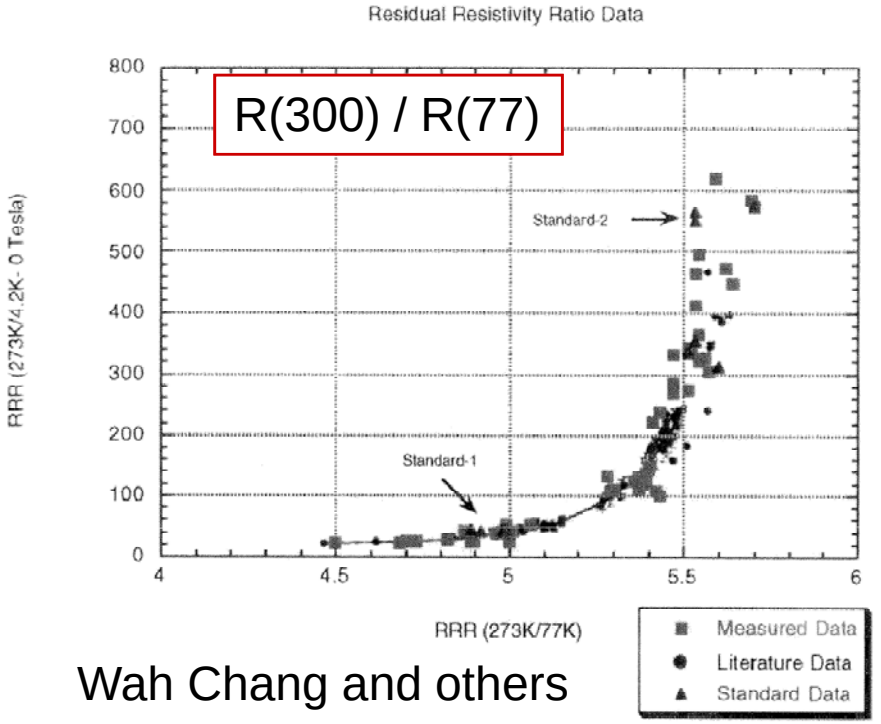
- RRR 250 to 350 makes little difference for superconductivity
- But: $T_{recrystal}$ falls as RRR increases
  - Higher purity aids removal of cold work memory at moderate (< 800 °C) temperature, which thereby improves formability and shape control
  - Gradients inside annealing furnace are not known, therefore piece-to-piece shape control is not known

Ways to measure RRR

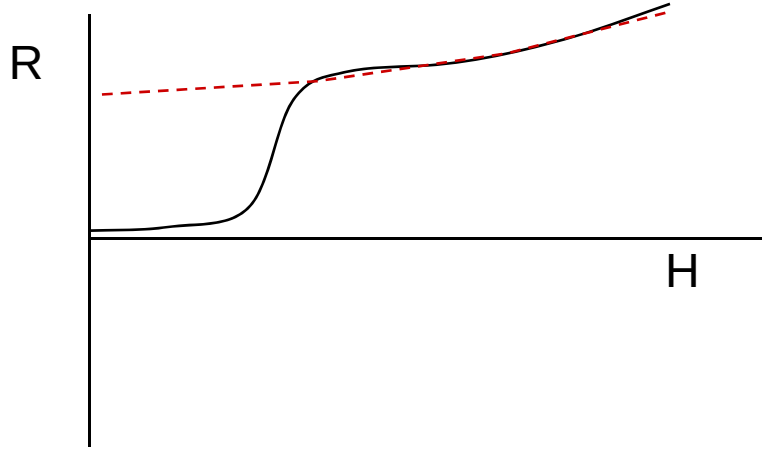
NIST



IEC 61788-4



Wah Chang and others



Recommendations and action items

- Everybody should use a (the) RRR standard
  - IEC 61788-4 Cu/Nb-Ti strand can be applied
  - Measurements and definitions vary by lab and by vendor
  - We need inter-lab and round-robin comparisons
- We should link sheet index to location in anneal furnace and to position in bulk rolled sheet
- Carry indexing thru to finished cavity (our AES001 indices are flipped from John's)
- We should work with vendors to verify annealing conditions, or establish an anneal standard
- We need to verify that "bad" sheets make "bad" cavities
  - Make a single-cell cavity out of the suspect sheets
  - Revisit past work or make a new series of "identical" cavities from sheets with a range of RRR