

Laser Annealing Experiments with Niobium (for SRF2005 @ Cornell)

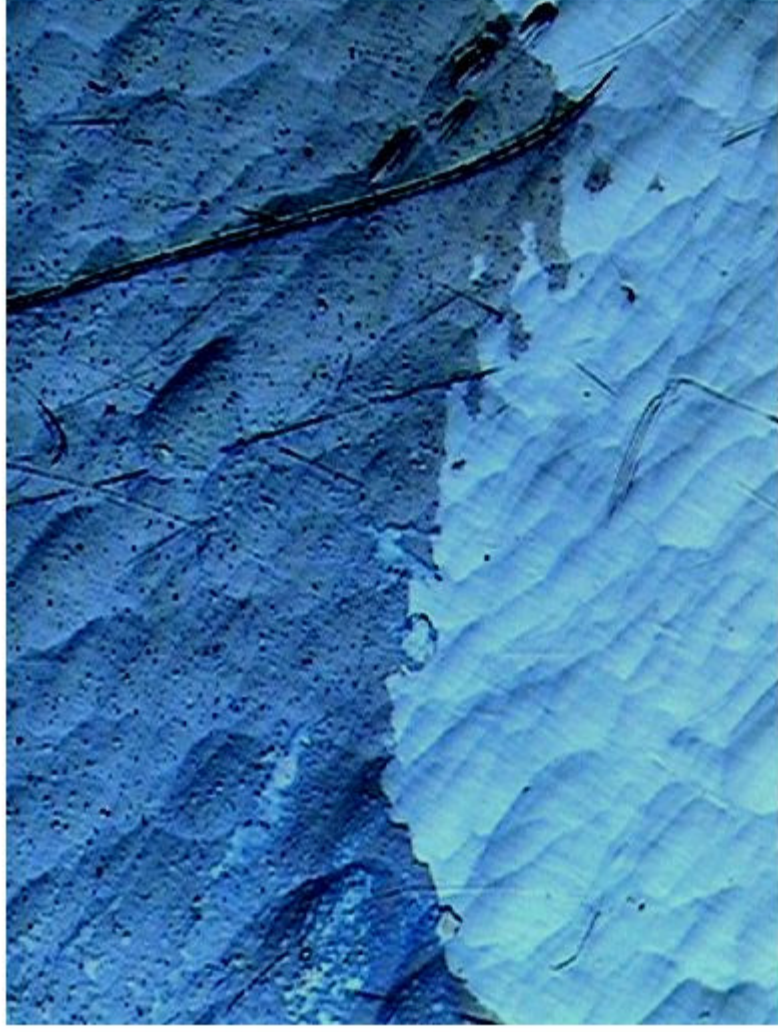
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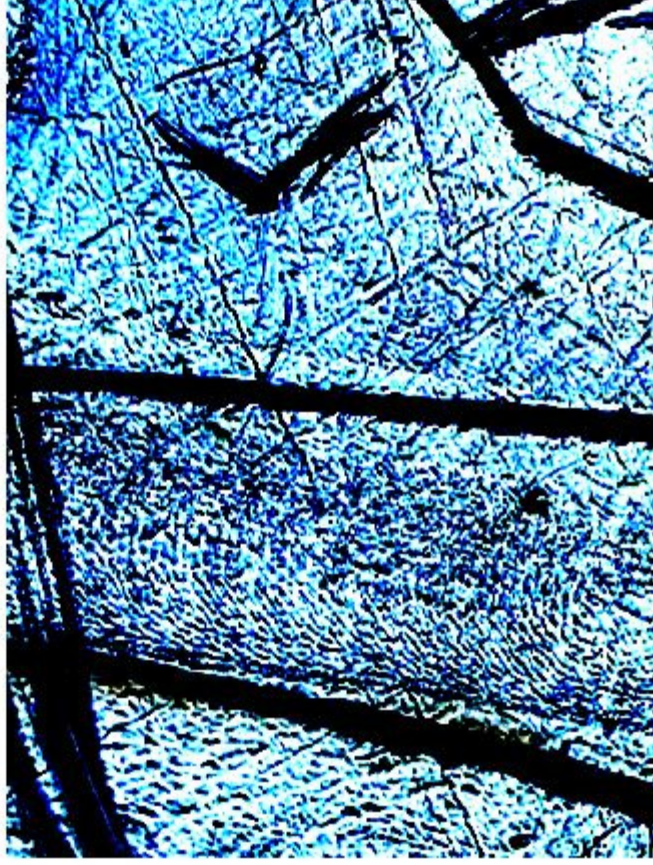
Abstract

- We describe our recent pulsed laser annealing studies on small samples of bulk niobium. We compare the effects of annealing over a wide range of single pulse energy density, and in three gas environments: air, nitrogen, and argon. Our Nd:YAG laser allows annealing studies with 266 nm UV light, and with 533 nm green light. We examine the sample surface for structural changes by SEM (Scanning Electron Microscope) and by MicroXAM (a surface mapping microscope).

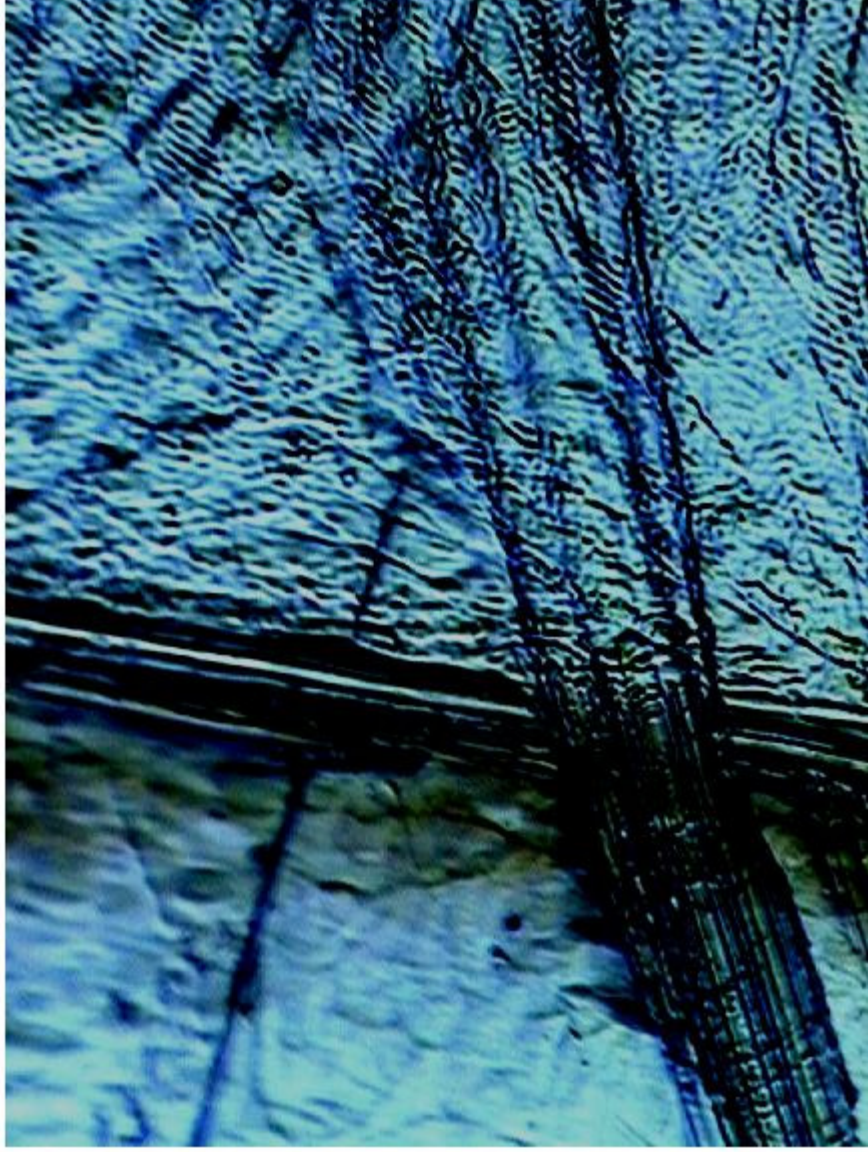


A few optical photographs... This is annealing on a high RRR sample of bulk niobium, which had been used for mechanical and electropolishing tests.

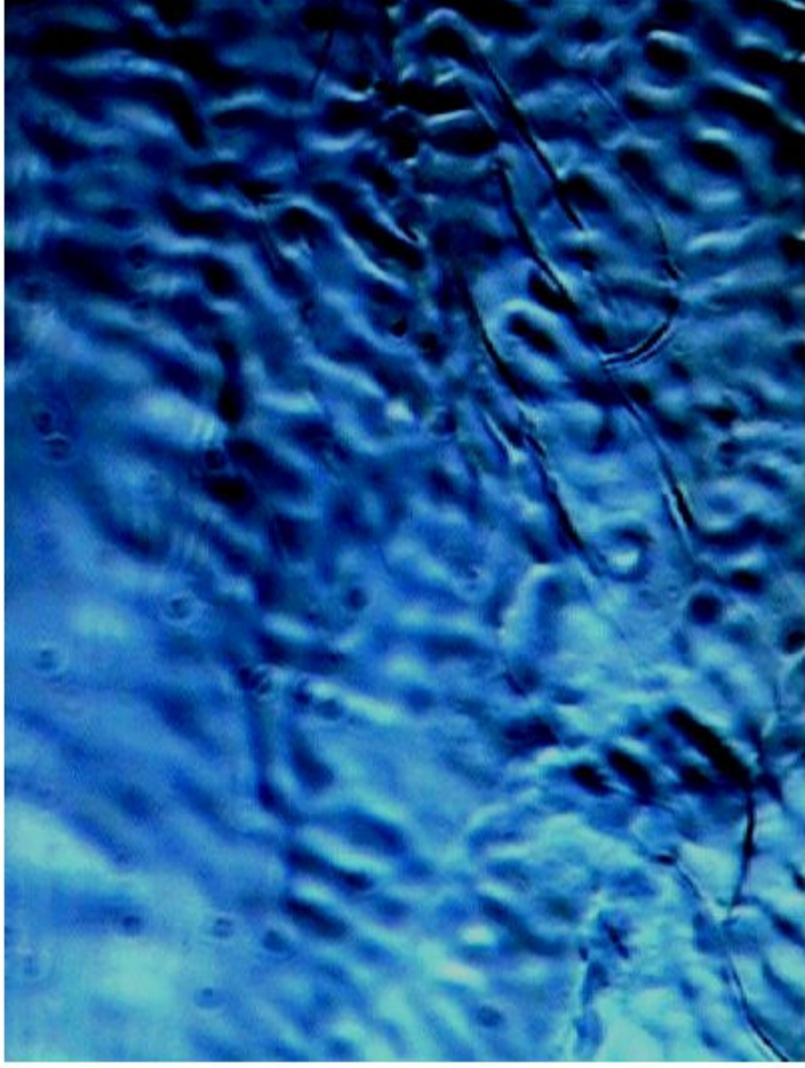
An annealing pass between and across
mechanical polishing score marks



Smoothing of the crossing score mark

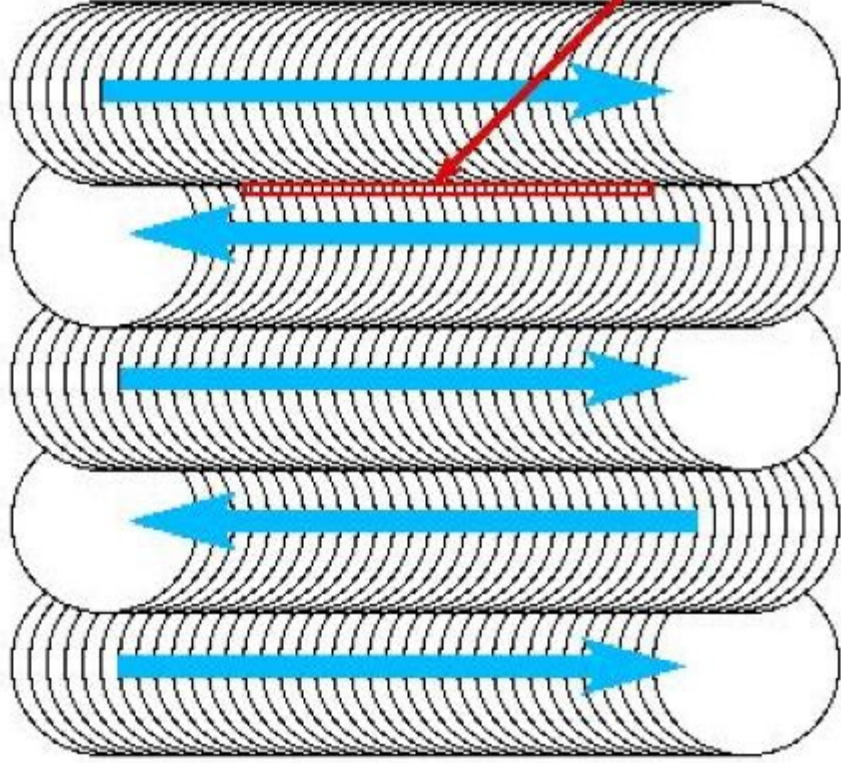


Within the annealed region, much smoothing, but new ripples from the annealing process.



- Two initial tests: 0.25 mm rolled foil from Goodfellow.
Third test: high RRR 3 mm DESY sample.
- Nd:YAG laser's maximum power: from 600 mJ/pulse in IR(1064 nm) to 40 mJ/pulse in UV (216 nm).
- 10 Hz in 4 ns pulses. No cumulative heating. The surface is cool again before the next pulse arrives.
- Surface melt condition was readily achieved in air with either UV or green light over a large range of single pulse energy density.
- Melts down surface structure, but tends to leave a ripple structure on scale of beam spot size.

Stepping pattern for laser spot.



As the spot advances, the laser pulses, and each pulse tends to melt down the previous, so leaving a pattern of stern wave ripples spaced by 25 microns.

In these tests we used a very heavy overlap, and we find a tendency to produce hairline cracking near the edge of the region where one pass overlaps the previous pass.

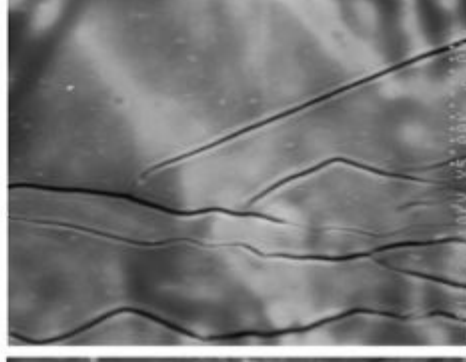
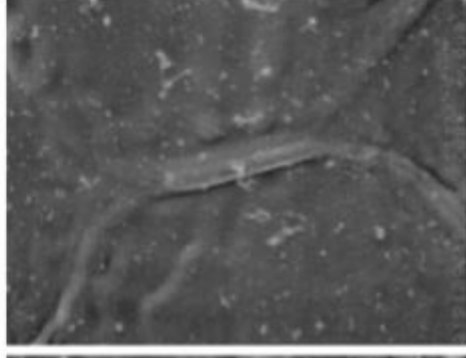
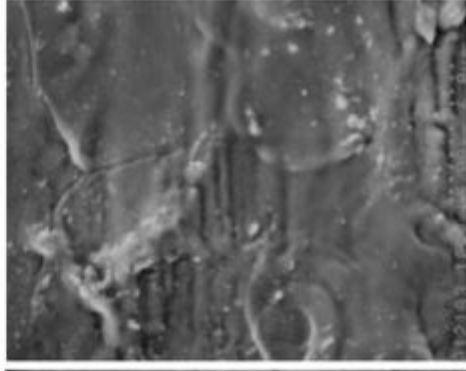
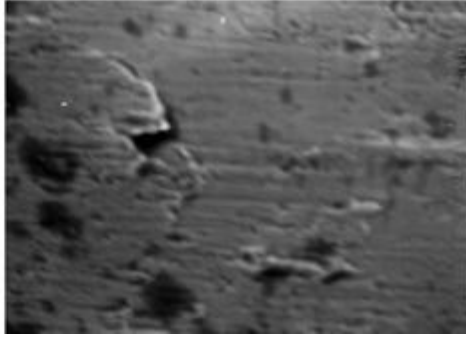
Hairline horizontal cracking in and near the overlap region.

- Surface melt condition was readily achieved with either UV or green light over a large range of energy density per pulse.
- Tendency to leave a ripple structure on scale of beam spot size. Higher pulse energy density => higher ripple amplitude.
- Note hairline surface cracks in these SEM photos at X5K.
(More serious at 2 degrees K?)

533 J/cm²

72 J/cm²

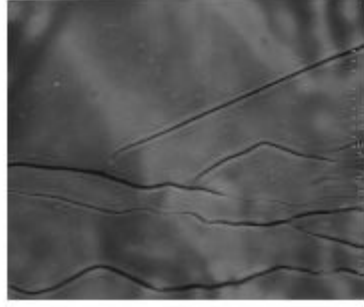
15 J/cm²



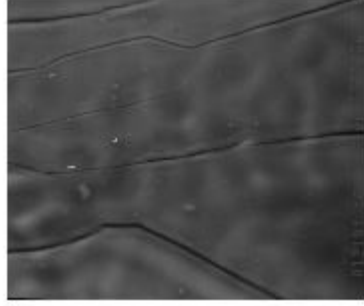
Unannealed UV, focused Green, defocus to 40 microns to 40 microns 200 microns

Ambient gas environment ? No effect?

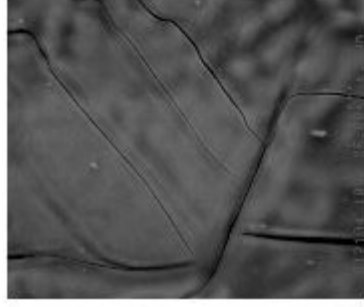
SEM photos show hairline cracks at X5K and ripples at X1K



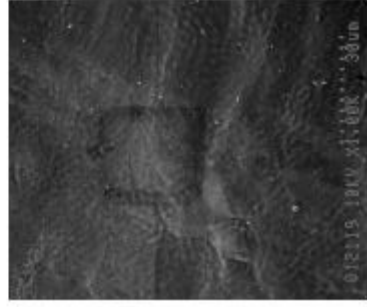
Annealed in air



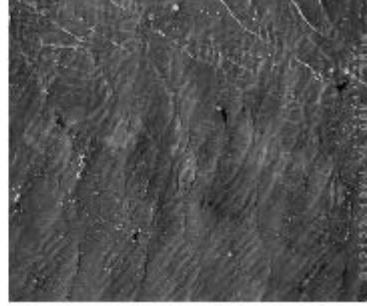
Annealed in nitrogen



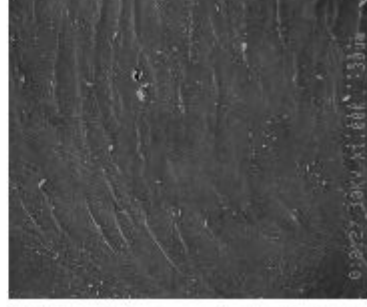
Annealed in argon



Annealed in air

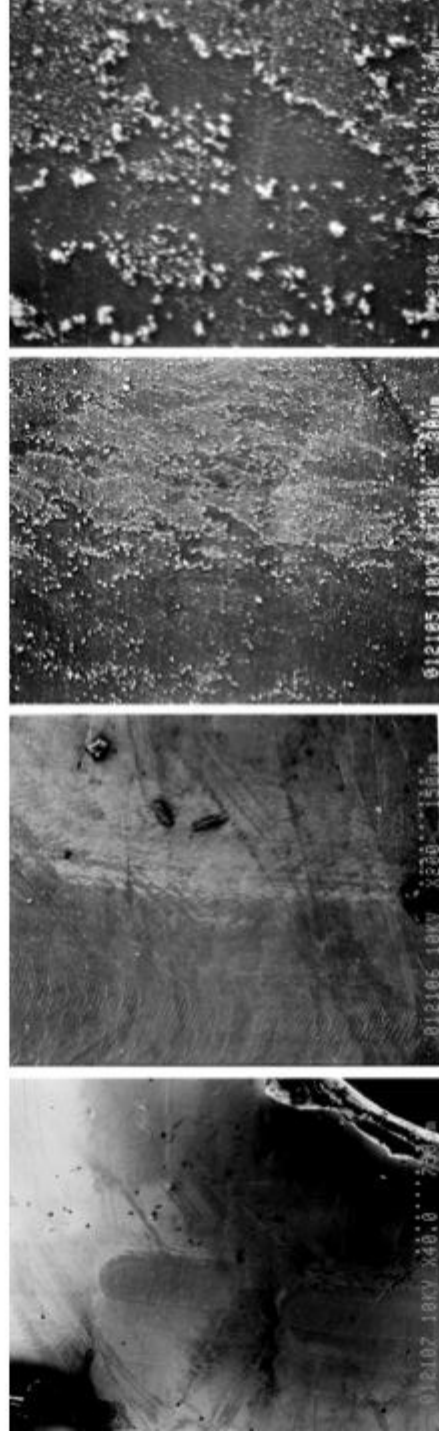


Annealed in nitrogen



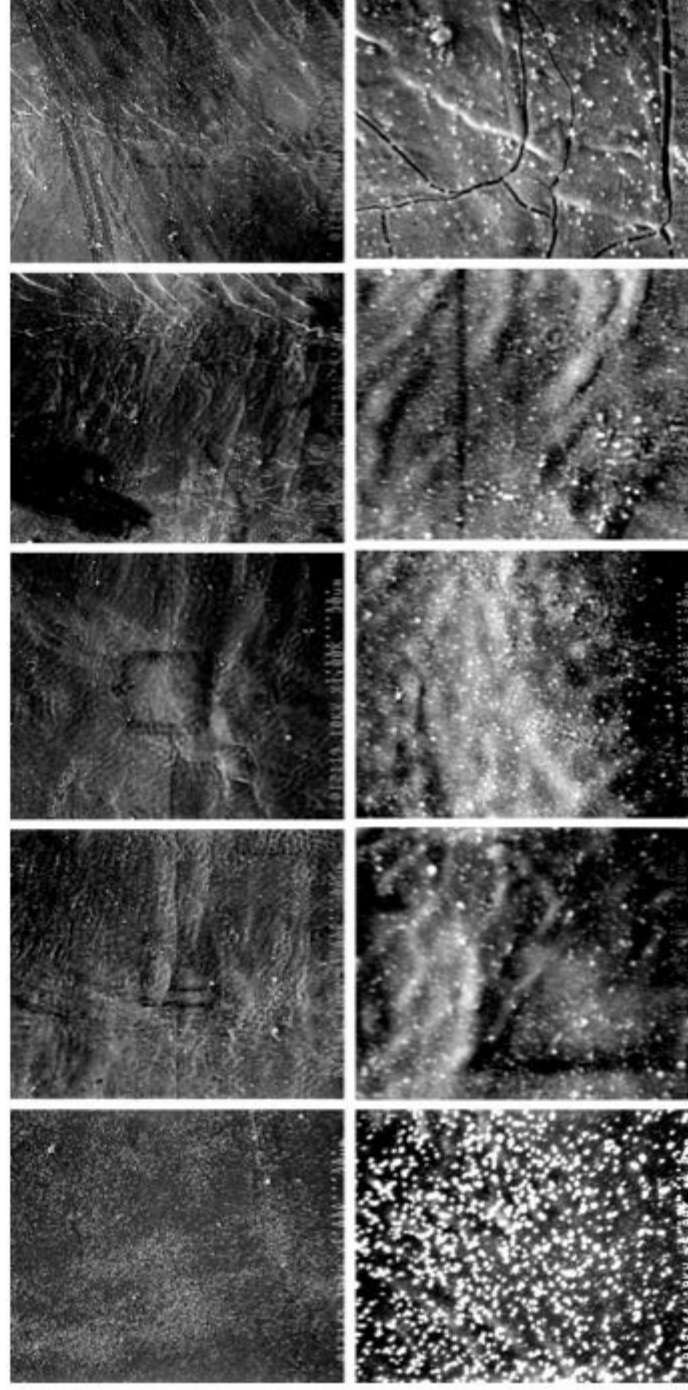
Annealed in argon

**SEM photos of the edge:
Transition from annealed to unannealed.**



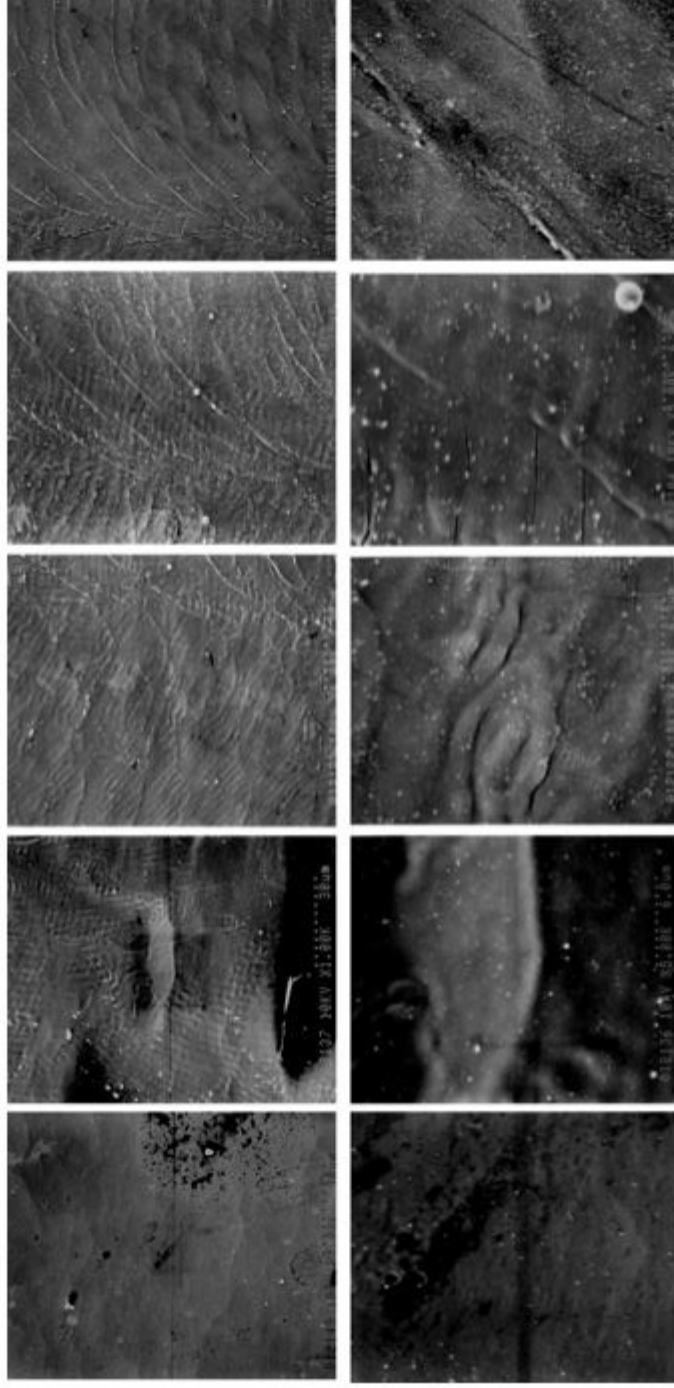
- Annealed at 12 J/cm²/pulse in nitrogen gas.
- Magnifications range from X40 to X5000.
- High RRR DESY sample
- Sample hanger hole lower right (DESY processing).

SEM photos of surface annealed in air



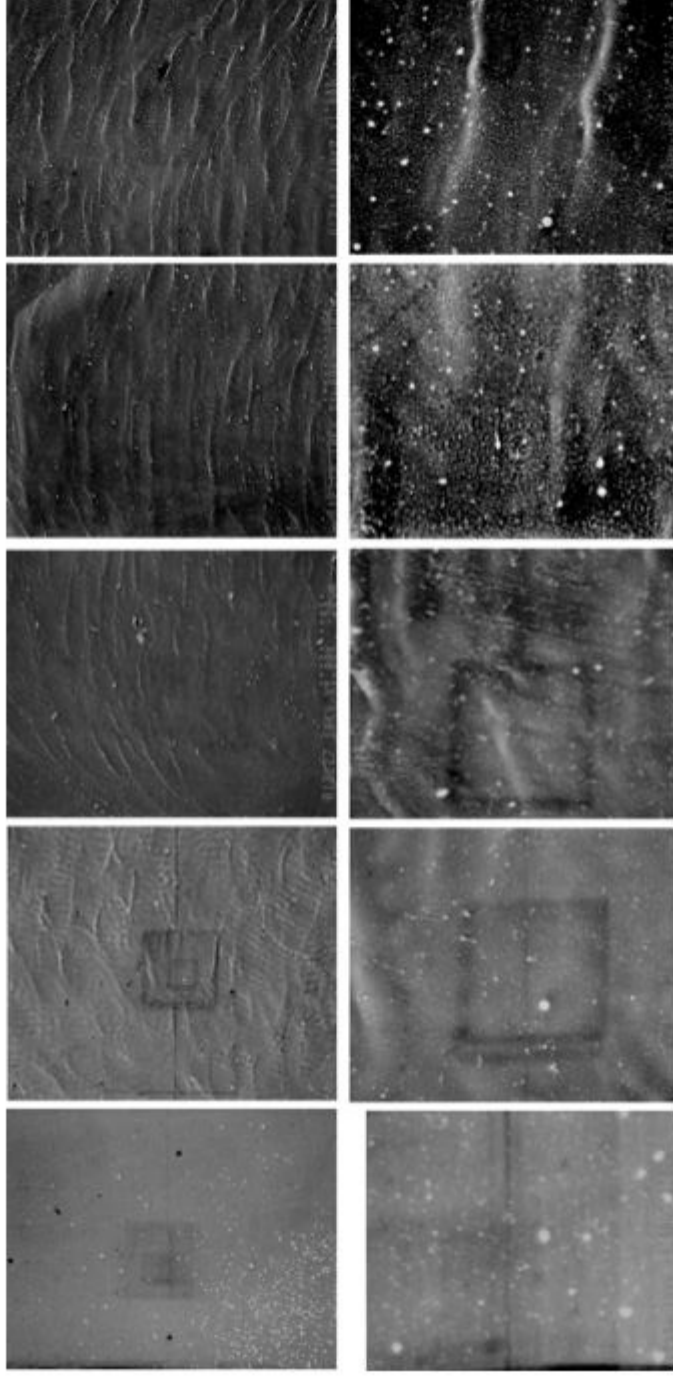
- Single pulse energy density ranges from 2.4 (left) to 12.2 J/cm² (right)
- Magnification = X1000 (upper row), X5000 (lower)

SEM photos of surface annealed in nitrogen



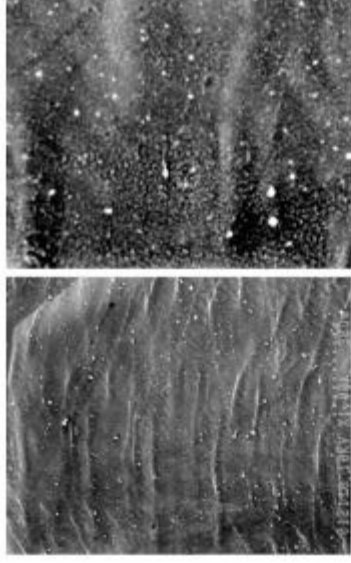
- Single pulse energy density ranges from 2.4 (left) to 12.2 J/cm² (right)
- Magnification = X1000 (upper row), X5000 (lower)

SEM photos of surface annealed in argon

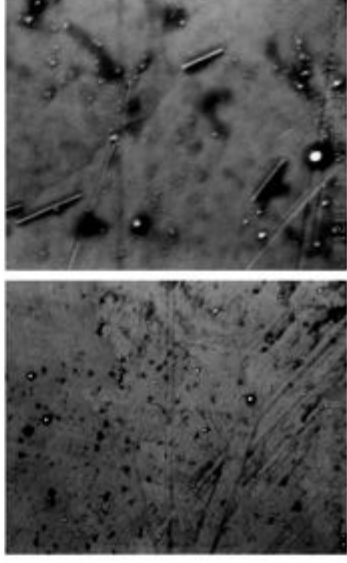


- Single pulse energy density ranges from 2.4 (left) to 12.2 J/cm² (right)
- Magnification = X1000 (upper row), X5000 (lower)

Comparison of annealed with unannealed surface



Annealed. SEM at x1000, and at x5000.



Unannealed. SEM at x1000, and at x5000.

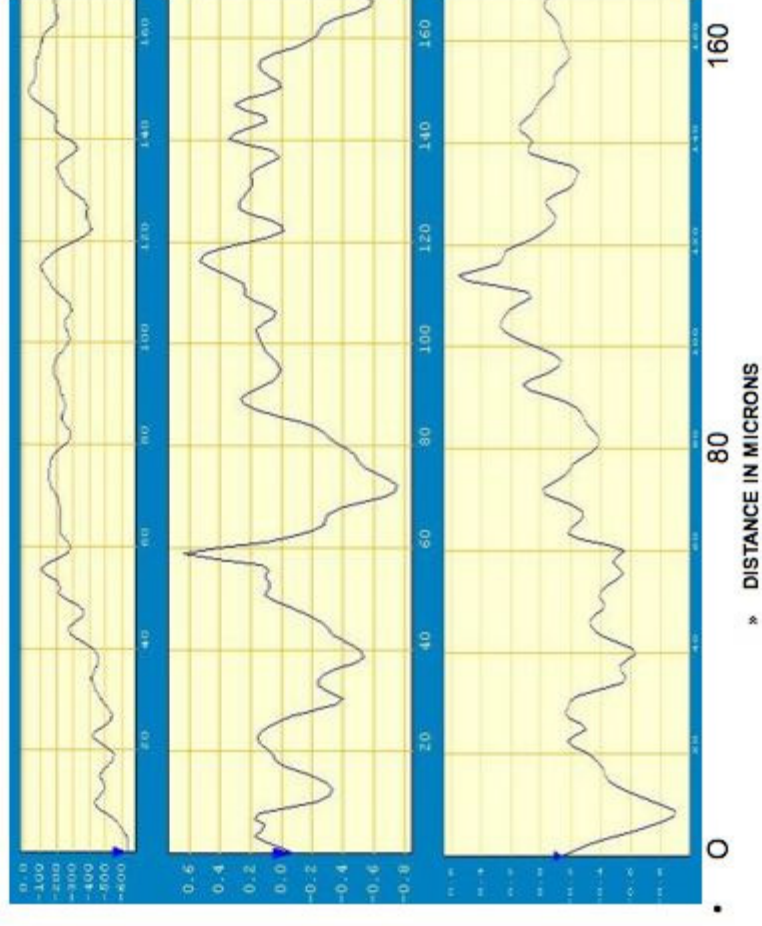
- In argon at 9.7 J/cm^2 per pulse

**STERNWAVE ROUGHNESS (deep holes are etch pits)
(COMPARING 2.4 [no visible ripples], 7.3 AND 12.2 J/CM²)**

Each line is
100 nm.

Each line is
200 nm

Each line is
200 nm



* DISTANCE IN MICRONS

Conclusions

- The SEM images show no preference for annealing in a particular gas environment. The short duration of the temperature excursion (about 50 nanoseconds) does not allow significant gas absorption.
- We have not yet found any need to use UV light, although we have not yet studied laser annealing's ability to break down interstitial niobium/light element compounds.
- Surface melt condition can be achieved over a wide range of laser pulse energy density. Our bulk studies do not find the eruptive events which limited an earlier CERN experiment annealing niobium films on copper substrates with a UV laser.
- We find a defocused laser beam provides a smoother final surface than does a small well-focused beam. Our ripple effect needs careful design of laser beam.
- Higher power melts down more surface defects, but leaves more noticeable ripples on the annealed surface.
- Annealing of niobium films is high on our list of future projects.

References

- [1] E. Radicioni et al., "Laser annealing of Nb coatings for superconducting RF accelerator cavities," NIM A 365 (1995) pp 28-35.
- [2] This was one of three samples obtained from Axel Matheisen at DESY by Bill Frisken in 1998.

Acknowledgements

- W.R. Frisken would like to thank York University's Faculty of Pure and Applied Science for the use of the SEM and the associated lab darkroom, and Ms. Karen Rethoret for cheerful instruction and assistance.
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