

Effects of Grain Size and Interstitial Content on Recrystallization in Nb After Cold Rolling

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Abstract—A fully recrystallized microstructure with a fine grain size can improve the performance of superconducting radio-frequency cavities produced from high-purity Nb (ASTM B393–18 Type 5 Nb) while providing mechanical strength. Recrystallization depends on impurity content, initial microstructure, deformation state, and annealing conditions. To better understand how fine-grained, fully recrystallized microstructures may be produced, the recrystallization behaviors of Type 5 and Type 2 Nb materials were studied. Type 5 Nb specimens were produced with both fine and coarse initial grain sizes. All were cold rolled and then annealed under vacuum for one hour to determine the rolling reductions and temperatures required for recrystallization. The recrystallized fraction exceeded 95% in fine-grained Type 5 Nb rolled to a 30% or greater thickness reduction and then annealed at 800°C or higher. The coarse-grained Type 5 Nb required greater rolling reductions to produce any recrystallized grains at 800°C. The higher interstitial content of the Type 2 Nb required a temperature of 1000°C or higher to reach a recrystallized fraction greater than 95% in specimens rolled to a 60% reduction. Recrystallization is more easily achieved during annealing at a given temperature for a set time by increasing rolling reduction, decreasing interstitial content, and decreasing initial grain size prior to cold rolling. To avoid grain growth following recrystallization, which increases grain size, the minimum time and temperature necessary for complete recrystallization should be applied.

Index Terms—Heat treatment, niobium, superconducting materials, superconducting radio-frequency (SRF).

I. INTRODUCTION

NB serves as both a functional and structural material in the construction of Nb superconducting radio-frequency (SRF) cavities. SRF cavity performance is assessed by efficiency, i.e., quality factor, at high accelerating electric field

strengths. Improving Nb SRF cavity performance while maintaining mechanical strength presents a metallurgical processing challenge. SRF cavity performance can be improved by eliminating impurities and lattice defects, such as dislocations and grain boundaries [1], [2], [3]. Dislocation density accumulated during cavity forming operations may be reduced by recovery (RV) and recrystallization (RX) of the microstructure during annealing, with RX offering the greatest reduction [4], [5]. Mechanical strength, necessary to sustain pressure loads and prevent damage that may occur during handling, requires a fine grain size [6]. This is because other approaches to strengthening, such as alloying or cold working, cannot be applied without sacrificing cavity performance. An ideal SRF cavity microstructure is one that is completely recrystallized with a sufficiently fine grain size to meet specifications for mechanical strength. Because the transition from RX to grain growth is often gradual, and recovered grains often persist, we consider microstructures with a recrystallized fraction greater than 95% to be *completely recrystallized*.

To better understand how to produce ideal SRF cavity microstructures, this study examines how interstitial impurity content and initial grain size affect the specific processing conditions necessary to completely recrystallize Nb. Recent studies suggested that RX in Nb is sensitive to both interstitial content [7] and deformation state [8]. Interstitial impurities, such as C, O, and N, tend to raise the annealing temperature required for RX. Deformation state, which depends on the initial microstructure, imposed plastic strain path, and deformation temperature, can strongly affect the annealing conditions required to achieve RX and the resulting grain size.

II. EXPERIMENTAL PROCEDURES

Two grades of unalloyed Nb were used in these experiments. The first is a high-purity ASTM B393–18 Type 5 Nb sheet material [9] produced by Tokyo Denkai for superconducting applications. The as-received microstructure was recrystallized with an equivalent lineal intercept grain size of 39 μm ; this is a relatively fine grain size for such a high-purity metal. The residual resistivity ratio (RRR) and upper bounds on the interstitial O and N contents reported by the manufacturer are presented in Table I. These upper bounds are near the detection limits for measuring interstitials, and the actual O and N contents are probably lower. The second material grade is commercial-purity ASTM B393–18 Type 2 Nb sheet that was remelted in a

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TABLE I
IMPURITY CONTENT AND RRR OF Nb MATERIALS

ASTM B393–18 Grade	O (wt. ppm)	N (wt. ppm)	RRR
Type 5 Nb	<10	<10	393
Remelted Type 2 Nb	370 ± 19	70 ± 5	~10

vacuum arc melter to produce specimens of a convenient size. This material contains greater concentrations of interstitials than Type 5 Nb, as measured using inert gas fusion analysis and reported in Table I. The RRR of the remelted Type 2 Nb was estimated from the measured interstitial concentrations to be on the order of 10 using the approach of Singer et al. [10]. The microstructure of the remelted Type 2 Nb was recrystallized to an equivalent linear intercept grain size of 140 μm .

Two types of experiments were used to probe RX behavior after unidirectional cold rolling. The first, called a *critical RX temperature* experiment, was used to determine the minimum annealing temperature necessary to achieve complete RX given a specific rolling reduction and annealing time. For this experiment, five specimens each of the fine-grained Type 5 and the Type 2 Nb materials were cold rolled to a thickness reduction of 60% and then annealed in a vacuum for 1 h at different temperatures. The second experiment type, called a *critical RX strain* experiment, was used to determine the minimum rolling reduction necessary to achieve complete RX given a fixed annealing temperature and time. For this experiment, five specimens each of the fine-grained Type 5 and Type 2 Nb materials were cold rolled to thickness reductions from 20% to 60% and then annealed for 1 h. The Type 5 Nb materials were annealed at 800 °C, and the Type 2 Nb was annealed at 1000 °C. Five additional specimens of Type 5 Nb with coarse grains were prepared and used for critical RX strain experiments by annealing the fine-grained Type 5 Nb material in a vacuum at 1300 °C for 1 h. The resulting grain growth produced grains with diameters approximately equal to or slightly less than the 3 mm specimen thickness. A vacuum of 10^{-4} torr or better (typically 10^{-5} torr) was maintained in all annealing experiments. All specimens were initially 3 mm thick and were cold rolled to their final thicknesses using a 10% reduction per rolling pass. Vickers hardness, a surrogate for tensile strength, was measured for each specimen before and after annealing treatments in both experiment types. Hardness was measured on the rolled sheet surface using an indenter load of 5 kgf with three measurements per condition.

Specimens for metallography were prepared by grinding with progressively finer SiC papers and polishing with progressively finer diamond suspensions. Rotary polishing for 2 hours with a 5:1 mixture of 0.05 μm colloidal silica to 30% H_2O_2 followed by 2 h of vibratory polishing with colloidal silica produced a suitable surface finish for observation in a scanning electron microscope. Microstructures were characterized using the combined backscattered electron (CBSE) imaging technique recently introduced by Bennett and Taleff [11]. This technique combines three grayscale backscattered electron images of the same field taken at slightly different tilt angles into a single red–green–blue

color image to better reveal grain boundaries and distinguish recrystallized from unrecrystallized regions. Average grain area in recrystallized microstructures was measured using the rectangular planimetric technique and converted to equivalent lineal intercept grain size as prescribed by ASTM E112–13 [12]. The recrystallized fraction in partially recrystallized microstructures was measured according to ASTM E562–19^{e1} [13].

III. RESULTS

In critical RX temperature experiments, fine-grained Type 5 Nb specimens cold rolled to a 60% reduction achieved complete RX during annealing for 1 h at temperatures of 800 °C and higher. The microstructures produced by annealing at 700 °C, 800 °C, and 900 °C are presented in Fig. 1. The specimen directions with reference to the sheet geometry are the rolling direction (RD), long transverse direction (LTD), and short transverse direction (STD). Remelted Type 2 Nb specimens rolled to the same 60% reduction completely recrystallized during annealing for 1 h at temperatures of 1000 °C and higher. The microstructures produced by annealing at 900 °C, 1000 °C, and 1100 °C are presented in Fig. 2. For completely recrystallized microstructures of both materials, the recrystallized grain size increased with increasing annealing temperature. This is evident in Fig. 3(a), which plots grain sizes from fine-grained Type 5 Nb specimens against annealing temperature. This suggests that grain growth occurred after RX at temperatures above the critical RX temperature. Below the critical RX temperature, the recrystallized fraction increased with increasing annealing temperature.

In critical RX strain experiments, fine-grained Type 5 Nb specimens cold rolled to reductions of 30% or greater achieved complete RX during annealing for 1 h at 800 °C. Remelted Type 2 Nb required a 60% reduction in thickness to achieve complete RX during annealing for 1 h at 1000 °C. Four of the five fine-grained Type 5 Nb specimens subjected to critical RX strain experiments completely recrystallized and provided grain size measurements, which are presented in Fig. 3(b). Grain size after RX decreased with increasing rolling reduction. Four of the five remelted Type 2 Nb specimens did not achieve complete RX in critical RX strain experiments.

Fig. 4(a) and (b) presents Vickers hardness measured after cold rolling (black markers) and subsequent annealing (blue markers) from fine-grained Type 5 and remelted Type 2 Nb, respectively. Remelted Type 2 Nb exhibited greater as-rolled hardness than the Type 5 Nb. As-rolled hardness increased with increasing rolling reduction, a consequence of strain hardening. Annealing produced drops in hardness for both materials, reflecting the softening expected from RV and RX. Vertical dashed lines indicate the minimum temperature or rolling reduction that produced complete RX, determined from microscopy, in each material. The hardness data do not provide a clear signal of the degree of RX in either material.

The coarse-grained Type 5 Nb specimens exhibited RX behavior distinctly different from the fine-grain Type 5 Nb specimens subjected to the same rolling reductions and subsequent annealing treatments. Of the five coarse-grained specimens

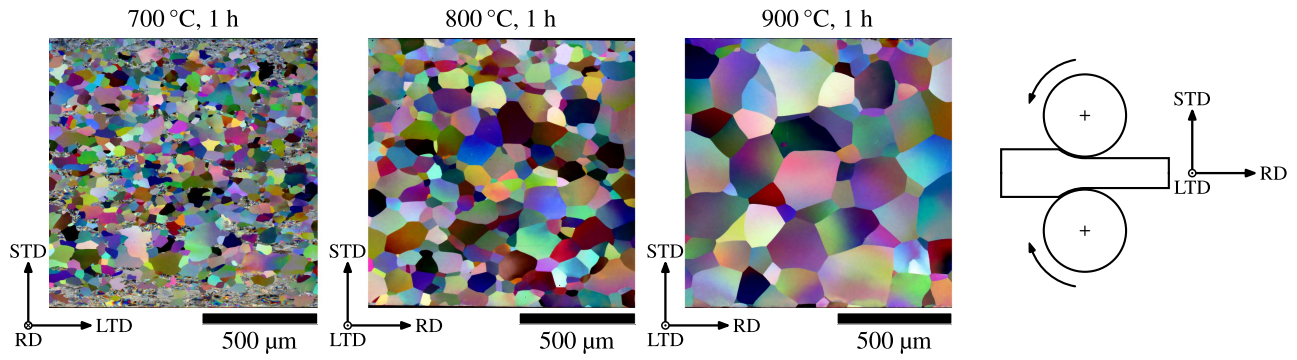


Fig. 1. Colorized CBSE images show, from left to right, microstructures produced by cold rolling the fine-grained Type 5 Nb material (RRR = 393) to a 60% reduction and then annealing at 700 °C, 800 °C, and 900 °C for 1 h. The height of each image captures the entire specimen thickness. The diagram on the right depicts a specimen passing through a rolling mill and identifies the specimen reference directions.

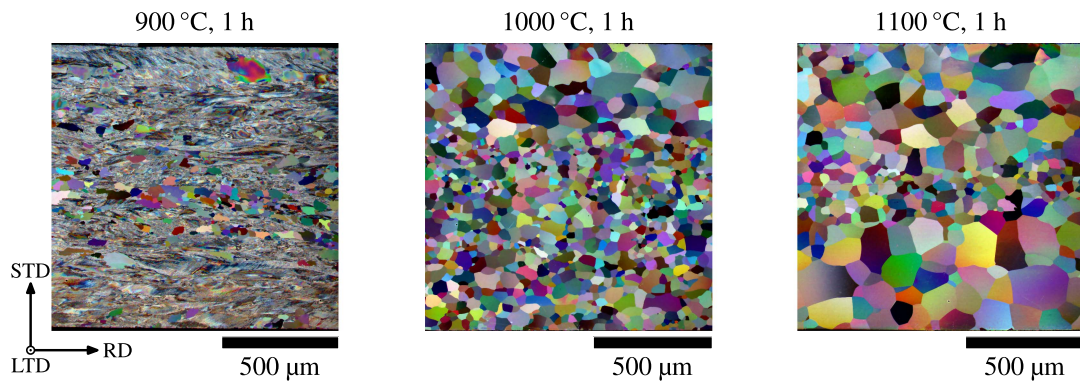


Fig. 2. Colorized CBSE images show, from left to right, microstructures of Type 2 Nb (RRR \approx 10) produced by cold rolling to a 60% reduction and then annealing at 900 °C, 1000 °C, and 1100 °C for 1 h. The height of each image captures the entire specimen thickness.

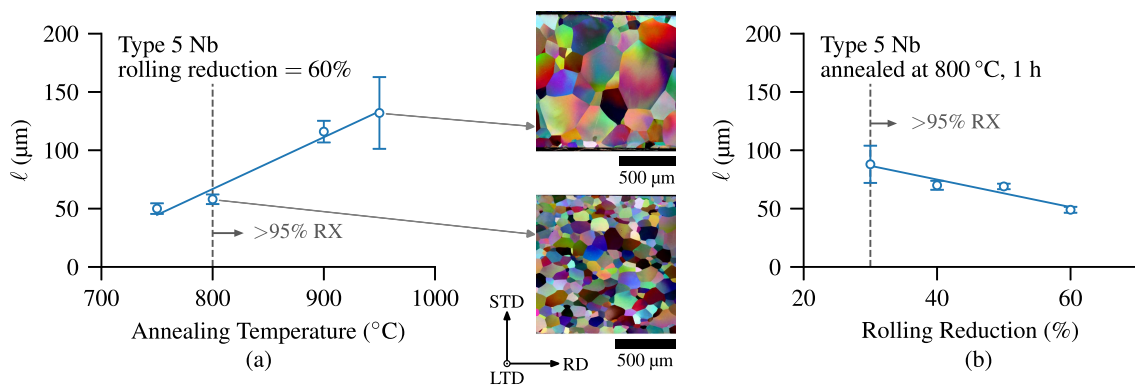


Fig. 3. Equivalent lineal intercept grain size, ℓ , from the initially fine-grained Type 5 Nb is plotted against (a) annealing temperature and (b) rolling reduction. Inset colorized CBSE images show microstructures produced by annealing at 800 °C and 950 °C. Error bars show 95% confidence intervals.

examined, partial RX was observed in only the one rolled to a 50% reduction. The remaining specimens, including the one subjected to the greatest reduction of 60%, did not exhibit RX. Unrecrystallized regions provided evidence of inhomogeneous plastic deformation in the form of shear bands. These are evident in Fig. 5, which presents microstructures of coarse-grained Type 5 Nb specimens after rolling reductions of 30%, 50%, or 60% and subsequent annealing at 800 °C for 1 h.

IV. DISCUSSION

The results of the present study demonstrate that interstitial content, initial microstructure, deformation state, and annealing conditions are all important to the RX of Nb. RX proceeds by the migration of high-angle grain boundaries, a thermally activated process, into regions of relatively high dislocation density [4], [5]. Interstitial impurities likely restrict the migration of

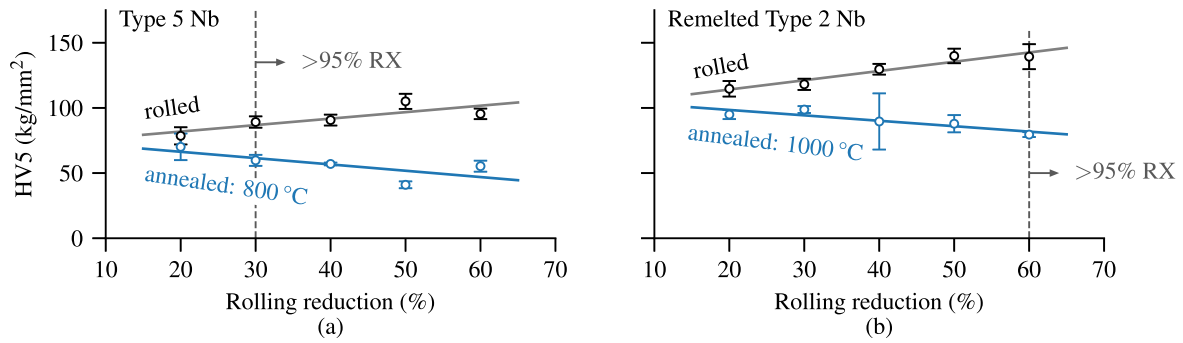


Fig. 4. Vickers hardness (HV5) is plotted against rolling reduction for (a) fine-grained Type 5 Nb and (b) remelted Type 2 Nb. Error bars show 95% confidence intervals.

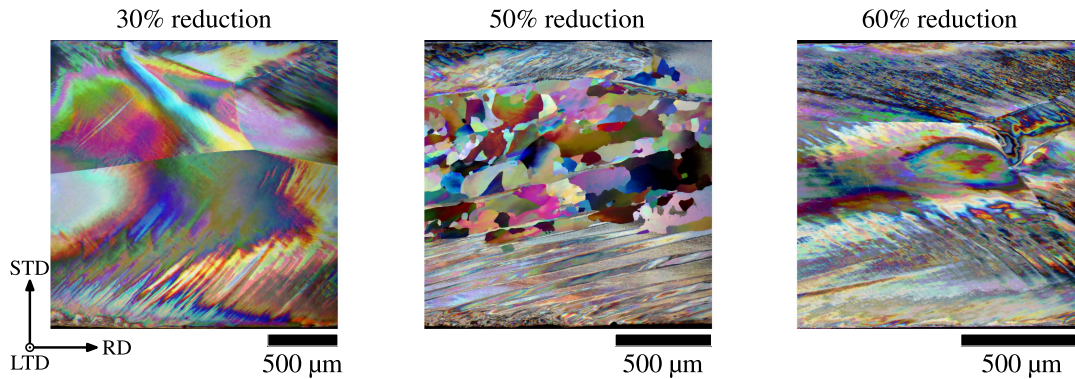


Fig. 5. Colorized CBSE images show, from left to right, microstructures of coarse-grained Type 5 Nb after cold rolling to reductions of 30%, 50%, and 60% followed by annealing at 800 °C for 1 h. The height of each image captures the entire specimen thickness.

high-angle grain boundaries and, thus, increase the temperature required for RX to occur after a given cold rolling reduction. The considerable difference of 200 °C between critical RX temperatures for fine-grained Type 5 and remelted Type 2 Nb materials indicates that small changes in interstitial content can profoundly affect RX behavior.

Initial grain size can have significant effects on local deformation state and, thus, subsequent RX behavior produced by a prescribed thermomechanical processing schedule. In coarse-grained Type 5 Nb specimens, inhomogeneous deformation, and thus inhomogeneities in stored energy, likely caused inhomogeneous RX behavior. The present results from coarse-grained Type 5 Nb are consistent with prior work on a cavity half cell formed from a slice from a high RRR Nb ingot [14]. Only one new high-angle grain boundary was found near an original grain boundary in the cavity iris after annealing for 2 h at 800 °C and no new boundaries were found in the equator after 2 h at 1000 °C. These findings suggest that coarse-grained material deformed to moderate strains cannot be reliably recrystallized.

V. SUMMARY

Optimizing microstructures in Nb SRF cavities requires understanding the effects of impurity content, initial microstructure, deformation state, and annealing conditions on RX. This study examined the effects of interstitial impurity content and

initial grain size on the RX behavior of Nb. Experimental data indicate that complete RX is more easily achieved during annealing at a given temperature for a set time by increasing rolling reduction, decreasing interstitial content, and decreasing initial grain size prior to cold rolling. This is based on the following specific findings.

- 1) Fine-grained Type 5 Nb achieved a recrystallized fraction greater than 95% during annealing for 1 h only at and above 800 °C after a 30% cold-rolling reduction.
- 2) Remelted Type 2 Nb, with greater interstitial impurity content than Type 5 Nb, achieved a recrystallized fraction greater than 95% during annealing for 1 h only at and above 1000 °C after a 60% cold-rolling reduction.
- 3) Hardness measurements did not produce a clear signal to identify the degree of RX.
- 4) After similar rolling reductions, coarse-grained Type 5 Nb was much less likely to recrystallize at 800 °C than fine-grained Type 5 Nb.

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