SUMMARY OF THE SYMPOSIUM ON INGOT Nb AND NEW RESULTS ON FUNDAMENTAL STUDIES OF LARGE GRAIN Nb*

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Abstract

The First International Symposium the on Superconducting Science and Technology of Ingot Niobium was held at Jefferson Lab in September 2010. Significant activities are taking place at laboratories and universities throughout the world to address several aspects related to the science and technology of Ingot Nb: from ingot production to mechanical, thermal and superconducting properties. The ingot Nb technology has been fully proven in the linear accelerator FLASH at DESY. A summary of the results presented at the Symposium is given in this contribution. New results on the superconducting properties and interstitial impurities content measured in large-grain Nb samples and cavities are briefly highlighted.

INTRODUCTION

R&D activities on large-grain/single-crystal ingot Nb technology have been pursued worldwide, since initial developments at Jefferson Lab in 2005. Following the International Workshop on Single Crystal-Large Grain Niobium Technology held in Araxá, Brazil, in 2006 [1], a two days International Symposium on this subject was organized at Jefferson Lab in September 2010. Fifty-seven participants from fifteen research laboratories and universities and from nine industrial companies attended the Symposium. The latest global developments on both technological and scientific aspects of ingot Nb related to SRF cavities were brought into focus at the Symposium. Proceedings of the Symposium have been published by the American Institute of Physics in 2011 [2].

This contribution provides a concise summary of the developments on ingot manufacturing technology, cavity fabrication and test results and fundamental studies on samples. In addition, highlights from recent work on large-grain cavities and samples at Jefferson Lab will be given in the last section of this article, prior to some final conclusions.

INGOT FABRICATION

CBMM in Brazil, W. C. Heraeus in Germany and Tokyo Denkai in Japan are pursuing R&D on different topics related to Nb ingot production for SRF cavities. CBMM focused on the development of an improved process to produce high-purity ingots by investigating the effects of several parameters such as Ta content, feeding rate, atmosphere, getter and number of melts. As a result,

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high purity ingots with oxygen, nitrogen, hydrogen and carbon contents lower than 10 wt-ppm were obtained [3]. W. C. Heraeus is pursuing the development of suitable parameters to produce approximately 2 m long ingots with a single-crystal with diameter larger than 150 mm in diameter in the center of the ingot. This would allow avoiding necking and tearing at the iris during deep drawing of a disc. In addition, a multi-wire slicing process was developed to cut multiple Nb discs from an ingot for mass production purposes in a cost effective way [4]. The same process was pursued by Tokyo Denkai where a multi-wire saw was installed which allowed slicing of 150 2.8 mm thick Nb discs from an ingot in 48 h. The accuracy in the discs' thickness was better than achieved with standard rolled sheets by about a factor of five. In addition, they used an ultrasonic tomography method to identify different grain orientations being formed during the electron beam melting of ingots [5]. Additional activities in collaboration with KEK include attempting to grow single-crystal ingots from a single-crystal Nb seed inside the electron beam melting furnace.

CAVITY FABRICATION AND TEST RESULTS

Europe

DESY in Germany has been pursuing the large-grain Nb technology in collaboration with W. C. Heraeus and Research Instruments (RI), formerly ACCEL [4]. Eleven 9-cell 1.3 GHz cavities of the TESLA shape were fabricated by ACCEL out of large-grain Nb of different purity. After fabrication it was found that, although the accuracy of the half-cells' shape was within tolerances, larger deviations resulted from discs with (100) orientation in the central crystal than from discs with (211) or (221) orientations. After fabrication the cavities surface treatments consisted of 100 µm material removal by Buffered Chemical Polishing (BCP), heat-treatment at 800 °C for 2 h under vacuum, 20 µm etching by BCP, high-pressure water rinsing (HPR) and baking in ultrahigh vacuum (UHV) at 125 °C for 48 h (LTB). The RF tests at 2 K (Fig. 1) showed accelerating gradients (E_{acc}) up to 25 - 30 MV/m, corresponding to peak surface magnetic field (B_p) values of 110 - 130 mT, with Q_0 values above 1×10^{10} , exceeding the specification for the XFEL project. Further results from the high-power RF tests at cryogenic temperatures showed that:

- No significant dependence of the maximum $E_{\rm acc}$ -value on the Nb residual resistivity ratio (*RRR*) in the range 150 500, as shown in Fig. 2.
- The Q_0 -values at 1.8 K is almost a factor of two higher than for standard fine-grain Nb cavities,

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indicating that lower residual resistance can be achieved using large-grain Nb.

- No significant difference in the performance of cavities whose grain boundaries had been grinded during fabrication was observed, compared to the other large-grain cavities.
- The average maximum E_{acc} -value of BCP-treated large-grain cavities was about 5 7 MV/m higher than fine-grain ones treated in the same way.



Figure 1: $Q_0(E_{acc})$ measured at 2 K at DESY for 11 1.3 GHz 9-cell cavities manufactured by ACCEL and treated by BCP. The Q_0 degradation at high field for cavities AC158 and AC112 is due to field emission and Q-drop (AC112 was not baked at 120 °C), respectively. The star symbol indicates the requirement for the XFEL project. The figure is from Ref. [4].



Figure 2: Maximum E_{acc} measured in fine-grain (blue diamonds) and large-grain (red squares) cavities treated in a similar way, as a function of the *RRR* of the Nb. The figure is from Ref. [4]

Three cavities were subsequently treated by Electropolishing (EP), removing about 50 - 100 μ m, followed by HPR and LTB. The maximum E_{acc} -value improved by about 10 MV/m in two of the cavities while it degraded by about 50% in the third one. Temperature mapping and optical inspection showed the presence of large areas with small craters present in the "hot-spot" regions. Finally, the two electropolished large-grain cavities have been integrated with He vessel, high-power couplers and installed in two cryomodules in the FLASH accelerator at DESY. The cavities are operating at an E_{acc} -value of 33 MV/m and 27.5 MV/m in the accelerator,

with beam, therefore providing a complete demonstration of the SRF cavity technology based on large-grain Nb.

Americas

Extensive RF testing at Jefferson Lab of 1.3 GHz and 1.5 GHz single-cell cavities made from large-grain Nb from different manufacturers (CBMM in Brazil, Ningxia in China and W. C. Heraeus in Germany) resulted in excellent, reproducible performances at 2 K: average B_p -values of about 140 mT with Q_0 -values at the highest gradient of about 1×10¹⁰ [6]. The cavity treatments prior to RF testing consisted of BCP, heat treatment at 800 °C for 2 h or 600 °C for 10 h, HPR, LTB and post-purification at 1250 °C with Ti as getter. This work provided the basis for pursuing large-grain Nb R&D activities in other parts of the world.

Eight multi-cell prototype cavities were built at JLab of different frequency, shape and number of cells. CBMM ingots with different Ta content were used mostly for the multi-cell cavity fabrication. The main issue during fabrication was the recurrence of "holes" produced during at least one of the equator electron-beam weld (EBW) of each cavity, which had to be repaired with Nb "plugs". The surface treatments were similar to those applied to the single-cell cavities. The average maximum (quench) B_{p} value of all multi-cell cavities measured at 2 K is 100 \pm 18 mT, significantly lower than obtained from single-cell cavity tests [7]. EBW repairs are certainly good candidates for causing premature quenches below 100 mT, as it was confirmed on one cavity tested with thermometry. In addition, features such as etch pits and "grooves" were observed on the inside surface of some of the cavities.

A positive result from the multi-cell cavities was the confirmation of maintaining high Q_0 -values (greater than 1×10^{10}) up to $B_p > 100$ mT, as it was measured on single-cell cavities. The values of the residual resistance were deduced from the Q_0 -values at low field, as described in [8], and are lower than those obtained on fine-grain cavities at the same frequency, by a factor of ~2, as shown in Fig. 3, and consistent with the results at DESY.



Figure 3: Residual resistance values obtained from the low-field $Q_0(2.0 \text{ K})$ for fine-grain (FG) [8] and large-grain (LG) cavities at different frequencies. The figure is from Ref. [7].

Asia

KEK in Japan is pursuing the combination of largegrain Nb with BCP treatment as a cost-effective highgradient cavity production technology, in alternative to the standard fine-grain Nb with EP [9]. Studies are being done on 1.3 GHz single-cell cavities of the Ichiro shape to determine the optimum amount of material removal by BCP which maximizes the E_{acc} -value. It should be noted that, unlike other laboratories, centrifugal barrel polishing (CBP) is routinely used at KEK to remove material after cavity fabrication. Three 9-cell, 1.3 GHz cavities of the Ichiro shape were built from large-grain Nb discs from Tokyo Denkai. During fabrication it was found that fewer defects were present in the equator areas if EBW was done from the inside, rather than the outside surface and two of the three multi-cell cavities were built using this technique. The surface treatments consisted of CBP, 10 um removal by BCP, heat treatment at 750 °C for 3 h, 50-100 µm BCP, HPR and LTB. Initial RF tests of two 9-cell cavities at 2 K resulted in a quench at $E_{acc} = 27 \text{ MV/m} (B_p$ ≅ 100 mT).

Activities on large-grain Nb cavities development are also ongoing at BARC, India, where they built a $\beta = 0.49$, 1050 MHz single-cell cavity [10] as part of the R&D on a CW accelerator for subcritical nuclear reactors.

IHEP in China built a 1.3 GHz, 9-cell cavity of the Low Loss shape using large-grain Nb from Ningxia [11]. The cavity was treated at IHEP by CBP, BCP, heat treatment at 750 °C/3 h and LTB. The cavity was tested at 2 K at KEK and was limited by field emission and quench at $E_{\rm acc}$ = 20 MV/m ($B_{\rm p} \cong$ 72 mT). Optical inspection of the quench location identified by thermometry revealed defects at the equatorial EBW.

SAMPLE STUDIES

Superconducting Properties

Superconducting properties such as the field of first flux penetration, $H_{\rm ffp}$, the upper critical field, H_{c2} , the surface critical field, H_{c3} and the critical temperature, T_c , have been measured at JLab for hollow Nb rods machined from large-grain ingot of different purity from CBMM. The samples' treatments were similar to the cavity treatments and included BCP, heat treatment at 600 °C for 10 h, additional BCP, LTB and EP [12, 13]. The main results can be summarized as follows:

- No significant dependence of H_{ffp} and H_{c2}, measured by both low-frequency and DC techniques, on *RRR*value or Ta content
- No significant variation of DC H_{ffp} (~189 mT at 0 K) and H_{c2} (~435 mT at 0 K), after LTB was done to the EP-treated samples
- The low-frequency $H_{\rm ffp}$ and $H_{\rm c2}$ are lower than the DC-ones by about 10-20% for EP treated samples, while they are ~35% lower than the DC-ones for BCP-treated samples (for example $H_{\rm ffp}$ at 2 K is ~170 mT as obtained from DC magnetization, while it is ~110 mT as obtained from the AC penetration depth)

• The low-frequency $H_{\rm ffp}$ -value at 2 K is significantly lower in BCP-treated than EP-treated samples (~110 mT compared to ~170 mT)

The differences in the $H_{\rm ffp}$ -values for EP, BCP and LTB treatments measured from the AC penetration depth, sampling a depth of ~30 µm from the surface, are consistent with the changes in the onset of the high-field Q-slope observed in SRF cavities and therefore suggest differences in the surface barrier and magnetic flux penetration as likely mechanisms involved in the high-field Q-slope phenomenon.

DC magnetization studies were also carried out at RRCAT in India, on $2 \times 2 \times 2$ mm³ Nb samples cut from large-grain ingots from CBMM and the results confirmed that no significant difference in terms of $H_{\rm ffp}$ was found with Ta content ranging between ~150 and ~1300 wt-ppm [14]. Measurements were done on pristine, "as cut", conditions as well as after BCP and heat treatment at 600 °C for 10 h.

Magneto-optical imaging studies were carried out at NHMFL/Florida State Univ. on fine-grain Nb samples, fine-grain samples which had been electron beam welded and therefore had ~mm size grains, and bi-crystal samples cut from large-grain Nb discs. The samples were treated by BCP, mechanical polishing, heat treatment at 750 °C and LTB. The results of their study showed that [15]:

- Topological features such as grain boundary (GB) steps of height greater than ~10 μm found on the fine-grain welded samples can induce preferential flux penetration in those regions.
- Preferential flux penetration at a GB was clearly observed in the by-crystal samples when the grain boundary plane is close to parallel with the applied magnetic field (Fig 4).

DC transport measurements were also done on bicrystal samples after EP and BCP to study the flux flow characteristics of grain boundaries which had different chemical treatments. The I-V curves showed that flux flow at the GB occurred at an external DC field of about 80 mT and 100 mT in the BCP and EP treated samples, respectively [16]. The flux flow dissipation is highly sensitive to the orientation of the GB and is highest when the GB plane is parallel to the direction of the external field. In addition, the degradation of superconductivity at the GB seemed to be less marked for the EP treatment than BCP. The geometric field enhancement at a grain boundary step was also measured using a micro-Hall sensor.

Mechanical and Thermal Properties

The grain orientations in ingot slices from three different manufacturers were analyzed using a Laue camera method at Michigan State Univ. (MSU). The results showed that there were no obvious commonalities among the three ingots or a preference for particular orientations [17]. Studies are in progress at NIST to measure the local strain rate of tri-crystal plates subjected to multi-axial straining, with the objective to determine

the slip systems which are locally active during the deformation [18].



Figure 4: Magneto-optical image showing flux penetration at the GB at 6 K and 80 mT magnetic field, applied nearly parallel to the GB plane (a). The optical image of the bi-crystal sample is shown in (b). The figure is from [15].

Thermal conductivity measurements carried out both at JLab [13] and MSU [19] on large-grain Nb samples from different ingots before and after heat-treatment at 600 - 800 °C for 2 - 10 h showed the presence of a large phonon peak at 2 K after heat treatment (Fig. 5), indicating a reduced phonon scattering by lattice defects, most likely dislocations. In addition, no dependence of the phonon peak on Ta content in the range 500 - 1300 wt-ppm was found.



Figure 5: Thermal conductivity as a function of temperature measured on two bi-crystal samples before and after heat treatment at 800 °C for 2 h. The Ta content is 1322 wt-ppm and 523 wt-ppm for samples 5 and 6, respectively. The figure is taken from [19].

STUDIES OF HYDROGEN IN NIOBIUM

New results on theoretical and experimental studies of hydrogen in Nb were presented at the Symposium. Depth profile measurements of C, N, O and H down to about 1 μ m from the surface were done by Secondary Ion Mass Spectrometry (SIMS) at North Carolina State Univ. (NCSU). High levels of H on the Nb surface were measured and the H concentration was significantly

reduced after heat treatment at 600 - 800 °C [20]. While it was possible to quantify the amount of C, N and O by ion implantation technique, this was not possible for either H or D because of the high mobility in Nb. Ion implantation of H and D was done successfully in a 120 nm thick oxide layer grown on the surface of a Nb sample by anodization. This allowed estimating the surface H concentration to be of the order of 2×10^{22} atoms/cm³ and confirming values obtained by other techniques, such as Nuclear Reaction Analysis. The experiments showed also that the native oxide layer is an effective barrier towards hydrogen desorption from or absorption into Nb.

A calculation of the hydrogen equilibrium fugacities for Nb, without the surface oxide layer, in contact with water or acids such as during BCP or EP was done at NIST [21] and showed that large amounts of hydrogen can be readily absorbed into the surface. Reduced hydrogen absorption is expected during EP, because of the anodic polarization applied to the Nb. The presence of grain boundaries can influence significantly the results from the calculation and it is expected the nucleation of niobium hydrides or hydrogen micro-voids to be easier at the grain boundaries of fine-grain Nb, compared to large-grain.

A new theoretical model to study hydrogen in Nb was proposed by J. Wallace of Casting Analysis, Corp [22]. In this new model the proton at an interstitial site of a BCC lattice is treated as a wavefunction in a spherical potential well or radius a and depth V. The radius a is computed from the site geometry of the host element. The well depth is the single parameter which is used to match the energy eigenvalues to the experimental activation energies for diffusion. The calculation matches the experimental data with a resolution on the order of one hundred of an eV for H, D and T in Nb. As a result of this model, interactions among proton's bound states with one another can lead to more complex electronic properties and possibly superconducting properties which need to be explored. Furthermore, the formation of a proton band within the metal can be easily expected from this model and its presence can significantly influence the magnetic and superconducting properties of the metal.

An induction reflection measurement technique was applied to study the magnetic properties of Nb foils, sputtered Nb thin films and bulk Nb samples [22]. Preliminary results showed unexpected complex changes in conductivity and magnetic permeability of the samples subjected to cooling to 77 K and heating up to 225 °C.

NEW RESULTS ON FUNDAMENTAL STUDIES OF LARGE-GRAIN NIOBIUM

In this Section, new results on fundamental studies of large-grain Nb samples and cavities will be highlighted and references to the more detailed contributions presented at this conference will be provided.

Initial studies on reducing the surface hydrogen content in SRF bulk Nb cavities by eliminating the chemical etching after the high-temperature UHV heat treatment process showed a significant improvement of the Q_0 -value after heat treating at 800 °C/3 h followed by 120 °C/12 h. A study has been done at JLab on a large-grain single-cell cavity to determine the optimum heat treatment temperature, between 600 – 1200 °C which results in the highest Q_0 improvement at 2 K and $B_p = 90$ mT. The results showed a Q_0 improvement of about 30% after heat treatment at 800 °C/3 h or 1000 °C/2 h, followed by 120 °C/12 h bake [23].

The role of trapped magnetic vortices in Nb as source of RF hot-spots was investigated on a photo-injector type single-cell cavity made of large-grain Nb at JLab. The cavity was excited in the TE_{011} mode and a thermometry array allowed localizing hot-spots on the cavity surface. By using a 10 W, 532 nm laser beam directed onto the hot-spot locations inside the cavity as a heater, changes in the temperature maps were observed after laser heating, consistent with the movement of trapped vortices [24].

Studies of interstitial impurities in bi-crystal Nb samples by SIMS at NCSU showed different hydrogen and oxygen concentrations within different crystals. Evidence for GB segregation was found only for C [25].

Superconducting properties of hollow rod Nb samples from new ingots is still being pursued at JLab. Besides DC and AC measurements, the rod samples are inserted in a pill-box cavity to tests their RF properties at 3.5 GHz. So far, the highest Q_0 of about 3×10^9 and a maximum B_p of about 55 mT were measured at 2 K on a sample which had been electropolished. The limitation at high field is due to reaching the critical heat flux for He II across the rod's cooling channel [26].

DISCUSSION AND CONCLUSION

The Symposium organized at JLab provided a venue to discuss recent present and progress on the superconducting science and technology of ingot niobium for application to SRF cavities. Good progress has been made throughout the world in the fabrication and tests of multi-cell cavities with the best results been achieved by the collaboration between DESY and RI. They fully demonstrated the competitiveness of large-grain Nb technology with two 9-cell cavities operating at highgradient in a real accelerator. Problems with equatorial welds in other laboratories prevented them to consistently reach high $B_{\rm p}$ -values in multi-cell cavities as for singlecell cavities.

Sample measurements indicated no significant dependence of superconducting or thermal properties on the Ta content. Good correlation between the near-surface $H_{\rm ffp}$ and the *Q*-drop onset in SRF cavities was found, before and after baking and with BCP or EP. Reduced superconductivity at GBs was also measured on samples being dependent on the orientation of the GB plane with respect to the direction of the applied field and on the chemical treatment. Large concentrations of H in Nb after standard chemical treatments are expected on the basis of thermodynamics calculation, they were measured by SIMS and they are reduced by high-temperature heat treatments. New theoretical studies based on quantum

mechanical principles revealed the complexity of the interaction between H and Nb. This new model indicate the possibility of changes in the superconducting and magnetic properties induced by H in Nb, depending on parameters such as temperature and concentration, which are not expected on the basis of classical models.

During the discussion sessions at the Symposium, it was debated whether ingot Nb had become the technology of choice for SRF cavities. While some believed that was the case, others argued that a significant performance benefit of this technology versus the standard fine-grain approach should be realized to gain wider acceptance. The cavity test data obtained so far indicate that higher Q_0 values have been obtained in large-grain cavities than fine-grain ones. High Q_0 -values are required in CW SRF accelerators. An R&D effort is underway at JLab to develop a cost-effective process which maximizes Q_0 .

Another issue which was discussed was that while Nb manufacturing companies are investing in ingot production studies, there isn't yet a set of specifications, for example in terms of grain orientations, distribution and impurity concentration, on which the SRF community as agreed upon. These specifications could emerge from further R&D studies aimed, for example, at investigating changes in lattice defects and impurities in crystals of different orientations during the manufacturing and processing of cavities.

In conclusion, the competitiveness of ingot Nb cavity technology has been demonstrated in a real particle accelerator. Laboratories which are relatively new to SRF cavities, such as in India and in China, are investing in this technology. Ongoing R&D projects aim at investigating various aspects of the superconducting science and technology of ingot Nb and, in particular, in exploring the potential to produce SRF cavities with high Q_0 -values.

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