QUALITY ASSURANCE AND ACCEPTANCE TESTING OF NIOBIUM MATERIAL FOR USE IN THE CONSTRUCTION OF THE FACILITY FOR RARE ISOTOPE BEAMS (FRIB) AT MICHIGAN STATE UNIVERSITY (MSU)

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Abstract

Niobium is the current material of choice for the fabrication of superconducting radio frequency (SRF) cavities used in SRF based accelerators. Although niobium specifications for this application have been well established, material properties of as-received materials can still vary substantially. Required for the FRIB accelerator, $13.2M of niobium materials (sheet, tube, and flange) have been contracted to several niobium vendors. The FRIB cavity designs require very large niobium sheets, increasing the difficulty in fabrication and potential for contamination. FRIB has developed and initiated plans to control niobium specifications and perform incoming acceptance checks to ensure quality is maintained. Acceptance results from several niobium shipments will be presented, looking at several production lots from the same vendor and across multiple vendors. Non-conforming results were observed and will be discussed including follow-up investigations and mitigation strategies to improve quality of future shipments.

INTRODUCTION

The Facility for Rare Isotope Beams (FRIB) at Michigan State University (MSU) is an approved $730M project funded by a cooperative agreement between MSU and The US Department of Energy (DOE) for advancement in the study of rare isotopes. The driver linac for the FRIB project is an 200 MeV/u superconducting linac with final beam power reaching 400 kW.

The FRIB linac will require the fabrication of 330 superconducting radio frequency (SRF) cavities. Two main cavity types will be used, with two accelerating beta designs for each type. Quarter-wave cavities with betas of 0.041 and 0.085 [1, 2] will be used in segment one of the linac. Half-wave cavities with betas of 0.29 and 0.53 [3] will make up the accelerator in both segments 2 and 3.

As one of FRIB raw material procurements, a large volume of niobium will be required for the fabrication of the SRF cavities. The total FRIB cavity count will require $13.2M of niobium materials. The FRIB niobium materials contracts have been awarded to three suppliers and divided among four Task Orders;

Wah Chang (Oregon, USA) – Niobium-titanium alloy
Tokyo Denkai (Japan) – niobium sheet
Ningxia (China) – niobium sheet, tube, and rod

All niobium materials will be procured and accepted using a governing material specification and acceptance criteria list (ACL). The niobium specification defines a list of material requirements including; chemical composition, surface finish, mechanical properties, metallurgical properties, and electrical properties (RRR). In addition, niobium supplies must meet specified dimensional tolerances, with tight control of thickness tolerances.

All received materials are packaged to FRIB specification, with all individual sheets numbered and tracked back to the supplier's production lots, as shown in Fig. 1. The production lot number and the number of the individual sheet within the lot are tracked through cavity fabrication and testing. The tracking allows the individual sheets, and corresponding properties, to be mapped to the fabricated cavity component. This link provides a database for trouble shooting; establishing a path from a cavity with fabrication non-conformances to the starting niobium and the material properties. The tracking also is used for tracking inventory during production.

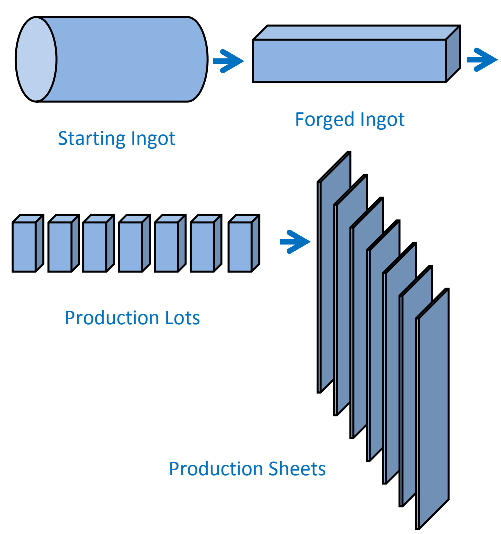

Figure 1: Niobium production tracking, linking production lot numbers to individual sheets and their material properties.

SAMPLE PREPARATION

As part of acceptance testing of incoming niobium procurements, material samples will be taken from received material and tested for compliance to the mechanical and metallurgical specifications agreed upon by MSU and material supplies, as stated in the corresponding purchase order and referenced specifications.

High RRR niobium material was received from two vendors in an assortment of sizes and thickness, meeting Task Order 1 and 2 requirements. Incoming materials are inspected and tested based on five main acceptance categories.

1) Material quantities
2) Material dimensions and tolerances
3) Surface Finish
4) Mechanical properties
5) Metallurgical properties
6) Residual Resistivity Ratio (RRR)

As defined in FRIB Niobium Acceptance Standard Operation Procedure document, a minimum of two samples per production lot are required for acceptance testing. Samples are selected from non-usable material areas, as defined by cavity vendor cut sheet requirements. A small section of material is removed from these areas and cut into metallurgical samples to be used in acceptance testing, an example is shown in Fig. 2. The following samples are prepared and tested; standard ASTM "dogbone" sample - used for tensile and hardness testing, RRR sample - used to measure thermal conductivity or purity of the material, and (2) small squares - used in the Orientation Imaging Microscope (OIM), providing information on grain size and recrystallization. All samples are labelled and tracked back to supplier sheet number and production lot numbers.

Measurement results are documented into a niobium acceptance spreadsheet, with supporting documents, and is include as an attachment to the acceptance validation report. All non-conforming results are reported to FRIB Quality Department and feedback to suppliers.

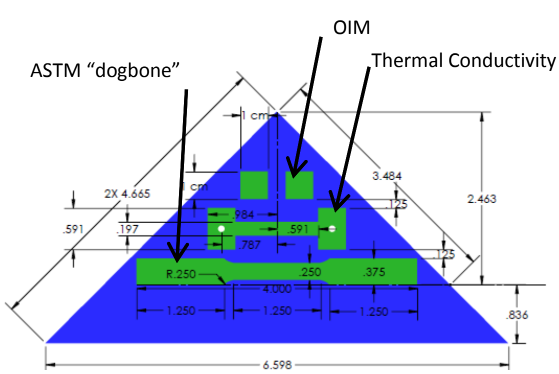

Figure 2: Concept of sample layout, cut from as-received niobium for use in acceptance testing.

ACCEPTANCE MEASUREMENTS

The ACL requirements are validated by conducting a serious of physical, mechanical, and metallurgical inspections/tests to samples removed for production sheets. The following measurements and equipment will be used to complete incoming niobium ACL.

Dimensional and Quantity Checks

All received niobium sheets will be counted and measured against tolerance specifications. Length and width requirements are + material, - zero material. Material thickness is very critical as many sheets are designed for deep drawing applications. Thickness tolerances are set for – zero, + 0.007" (0.2mm). Thickness is measured using a micrometer along perimeter and ultra-sonic thickness measurement for the interior of the sheets.

Mechanical Measurements

Standard ASTM tensile samples are Electric Discharge Machined (EDM) from as-received niobium material. Tensile samples are tested using an Instron 4302 machine at ambient conditions. A strain rate of 5 mm/min was used for all samples, running samples to fracture. Data was collected from the tests and plotted, providing an Engineering Stress verse Engineering Strain plot for each sample. Each sample plot provides three properties called out in the niobium acceptance specifications;

Yield Strength (0.2% offset): 7000 psi min.
Tensile Strength: 14000 psi min.
Elongation: 40 % min. longitudinal
Elongation: 35 % min. transverse

Hardness (Micro-hardness) (Hv 10)

Micro-hardness measurements are made on all production lot samples using a Clark Micro-hardness Tester CM-100. For each sample, five micro-hardness measurements are taken, with an average calculated. Measurements are made using a 100 gram load. The niobium specification is; Hardness, HV 10: 60 max

Metallurgical Measurements

1cm x 1cm square samples from each niobium production lot are viewed using an Orientation Imaging Microscope (OIM) to determine grain size and state of recrystallization. Samples are cleaned and etched prior to use in the microscope. For each sample, an orientation and misorientation map is generated from the OIM data. The preferred grain orientation is not a specification for FRIB niobium, but can be useful in understanding material issues if problems were to occur during cavity fabrication. The metallurgical specification is a predominantly grain size of ASTM #5 (64 µm) and >90% recrystallization.

RRR Measurements

RRR is measured from each sample lot using thermal conductivity measurements of samples and calculating

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RRR; [RRR ≅ 4 * thermal conductivity at 4.2K]. A RRR measurement system is currently being commissioned at MSU for future acceptance testing. Direct RRR measurements are faster than the thermal conductivity and the RRR system will be able to measure more than 20 samples at a time.

RESULTS

Niobium material from two vendors, supplied over two Task Orders, were sampled and measured against the FRIB acceptance criteria list. The results are present below.

Surface Finish

All material surfaces are inspected for surface imperfections. One major issue reported during surface finish inspections was the occurrence of pitting in the sheets, shown in Fig. 3. This was observed in both vendor materials and ranged in size and distribution. The pitted areas were further analysed to determine cause of pitting and to confirm no impurities were present. Pits were random in position and found on both sides of a given sheet. The size and depth vary, but most described as 1mm and smaller in diameter and between 100-150 µm in depth.

Figure 3: Pits found during surface finish inspections of niobium sheet.

Besides general surface roughness, some concerns with the observed pits are the potential for impurities in the niobium and the possibility of additional pits just below the surface (within the depths that would be etched away during cavity fabrication and processing). To address these concerns, a series of metallurgical tests were performed on the pits and surrounding area.

The pits were examined using a Scanning Electron Microscope (SEM), model Carl Zeiss EVO LS 25. Pits were viewed, looking at tomography and surface finish of grains within the pits.

The surface within the pits varied in etched appearance. The upper rim of the pit appeared shelved and had features that looked to be shear laps, as shown in Fig. 4.

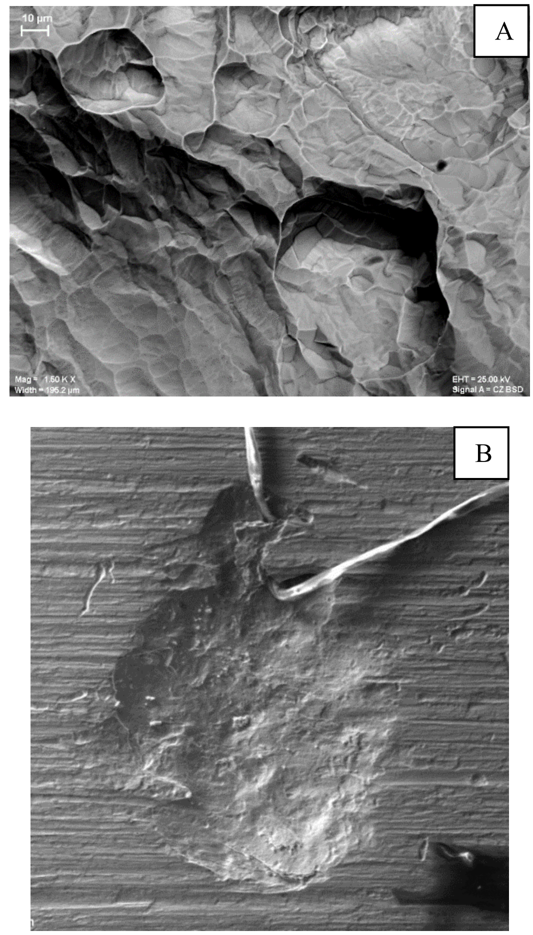

Figure 4: SEM images of observed niobium pits; A) internal surface of pit showing different etching behaviour, B) pit showing potential shear lap feature (with trapped fiber).

Within the pit, SEM images, Fig. 5, revealed particles sitting on the surface; loosely attached to the sheet material. Energy Dispersive Spectroscopy (EDS) analysis confirmed the material within the pit and the observed "attached" particles were niobium, shown in Fig. 6.

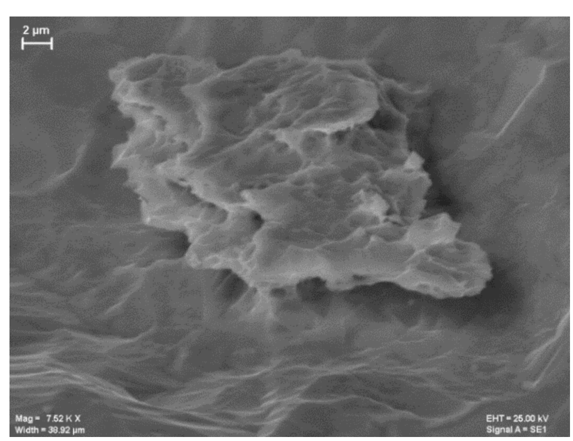

Figure 5: SEM image of particle sitting on surface of niobium pit.

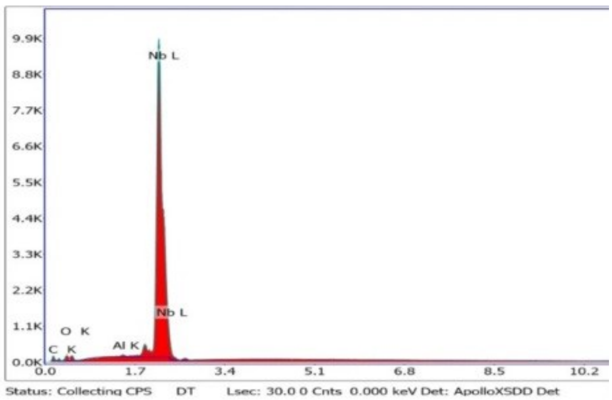

Figure 6: EDS analysis confirmed particle found in niobium pit to be niobium.

To address concerns of potential pits lying below the surface, eddy current scanning measurements were made on several niobium samples; surveying several production lots and thickness. The eddy current scanning system is able to detect defects at the surface to a depth of ~ 150µm. The eddy current scanner clearly detected the surface pits, as shown in Fig. 7. No other defects were observed in the scanned materials.

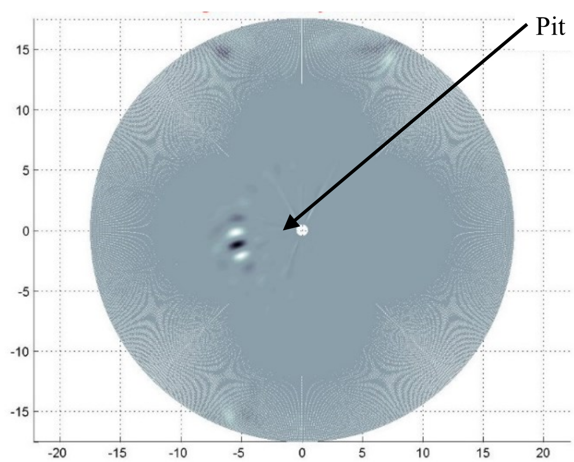

Figure 7: Eddy scans of as-received niobium sheet showing pitting at the surface.

Further analysis using OIM data showed an unusual orientation within the pit and the surrounding area. Clusters of [101] (green) orientations are not common in rolled niobium and not commonly seen in other production lots, surveyed across multiple vendors. What caused the orientation to form is curious, particularly if it relates to the pitting. If so, it is possible localized stress, caused by an embedded particle in the niobium surface or shear lap effects, could have local influence on the starting orientation of recrystallized grains. This relationship could be used as a further diagnostic of as-received sheet or subassembly fabrication in cavities.

Mechanical Properties

All samples were in the acceptance range with tensile plots following the same trends with all minimum specification values met, as shown in a typical plot Fig. 8. Several production lots were found to have a higher yield strength then typically seen. Multiple samples from the same production lots showed the same trends, verifying reproducibility in the measurement. These lots are documented and cross-checked with other acceptance testing results, such as RRR, for further understanding.

Micro-hardness

Micro-hardness results showed a large spread among the five indents and generally higher values then reported from suppliers. Discussions with niobium suppliers revealed a discrepancy in the applied load for the hardness measurement. Larger applied load can yield a lower hardness value if cluster of similarly orientated grains are sampled by the indent.

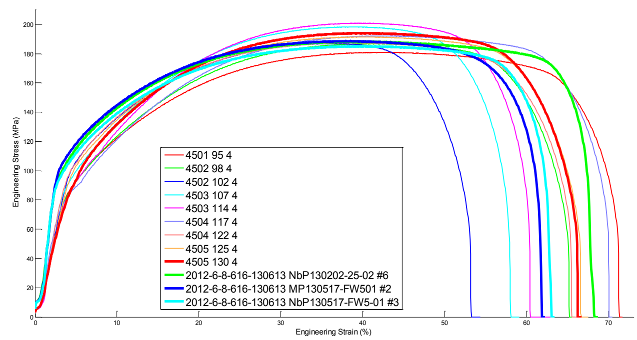

Figure 8: Stress-Strain curve generated during niobium mechanical property testing.

Grain Size

Grain size and recrystallization was measured for all sample lots. The majority of the sheet material was found to be in specification tolerance (predominately ASTM #5, 64 µm), as shown in Fig. 9. There were production lots from both vendors that had grain size non-conformance issued; one lot having slightly over size grain size and one lot having slightly under grain size. Both issues were discussed with supplying vendors; as both grain size non-conformances raise concerns; small grains can be a consequence of lower purity, suggesting the presences of impurities that can pin grain boundaries during the recrystallization annealing, and large grained material can be a cause of surface roughness during forming applications.

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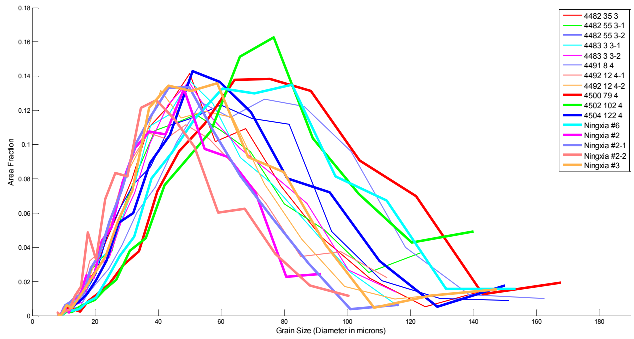

Figure 9: Grain size distribution of as-received niobium sheet samples over several production lots.

Recrystallization

Recrystallization is determined by looking at the misorientation local average among grains in the OIM maps. A high fraction of grains with large orientation gradients (within the grain) can indicate the material is not fully recrystallized, as shown by the gradient scale from blue to red in Figure 10-B.

A prefer texture is not a specification for sheet niobium production, but the information is obtained as part of the OIM measurement and is of interest in comparing fabrication and performance results in cavities. Texture is very random among production lots with a heterogeneous texture gradient in every lot. A wide distribution of banding is observed among the sheets, varying mostly between the [111] (blue) and [001] (red) orientations, normal to the surface of the sheet, as shown in Figure 10.

RRR

Calculated RRR values confirmed niobium sheets were within specification and consistent with vendor supplied measurements.

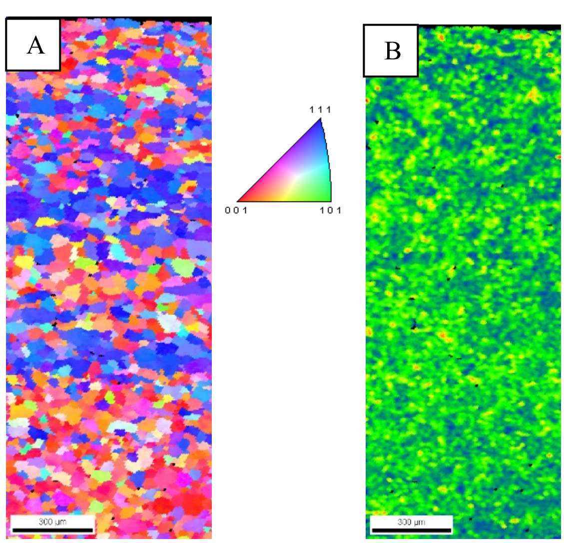

Figure 10: OIM maps of as-received niobium sheet; (A) map showing grain orientation normal to sheet surface, (B) map showing misorientation angle.

CONCLUSION

FRIB has begun receiving niobium material for the production of SRF cavities, distributed among multiple vendors and Task Orders. As-received material is subjected to acceptance testing as defined in the FRIB Acceptance Criteria List (ACL). To date, over 300 sheets of material has been received and inspected for FRIB. The majority of the material is found to be within FRIB specifications, accepted, and inventoried for FRIB production. Several non-conformances were measured and documented, resulting in the rejection of material and deviation requests. Some non-conformances were documented, but accepted for production. In these cases, non-conformance reports are discussed with vendors and solutions implemented to ensure future shipments are within FRIB specifications.

Niobium supplier visits were conducted to discuss ACL results of as-received materials, non-conformances, and strategic plans to bring production material into specifications. As discussed, pitting was a major focus of vendor improvements. Pits were examined using several metallurgical measurement techniques; concluding the pits were caused by niobium particles being embedded into the sheets during the rolling process, as illustrated in Figure 11 Vendors were very open to their production produces, including onsite discussions on how to improve the niobium sheet production. Vendors were very responsive to suggests; as demonstrated by immediate implementation of production improvements seen in follow-up visits.

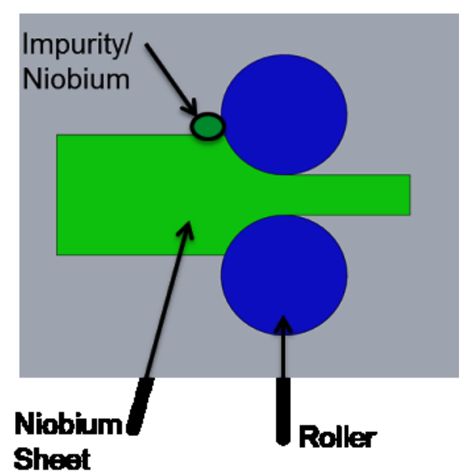

Figure 11: Illustration showing pit forming mechanism in niobium sheet fabrication.

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