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NIOBIUM CHRONICLES: SURFACE QUALITY INVESTIGATION AND RECOVERY DURING MATERIAL PROCUREMENT FOR THE PIP-II HIGH BETA 650 MHz CAVITIES

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Abstract

The surface quality of high-purity niobium for superconducting radiofrequency cavities, produced by a reputable manufacturer, experienced a sudden and significant decline in 2021. The recovery process and root cause analysis were challenging due to a variety of factors such as COVID-19 travel restrictions, cultural differences, and bureaucratic processes. Effective open communication was crucial to resolving the issue, especially with direct vendor oversight being impossible.

INTRODUCTION

The United Kingdom Research and Innovation Science and Technology Facilities Council (UKRI-STFC) Daresbury Laboratory is responsible for procuring and testing 20 High Beta 650 MHz cavities for the Proton Improvement Plan II (PIP-II) accelerator project at Fermi National Accelerator Laboratory (FNAL). Niobium stock was ordered from a well-known supplier (a sales representative of The manufacturer) and production started in Spring 2021. Most of the 225 disks for half-cells productions were to be delivered to Deutsches Elektronen-Synchrotron (DESY) for Eddy Current Scanning (ECS). Material for beam tubes, flanges, and the rest of the components was to be delivered to UKRI-STFC Daresbury Lab in the UK.

The production started in May 2021, but delivery was delayed due to an extra layer of difficulty to agree the contract terms following the UK exit from the European Union. The logistical challenges of coordinating this process, particularly during the pandemic, were significant. The first two batches of disks were delivered at DESY by August, but ECS did not start until early September due to staff and equipment availability.

SUDDENLY - DEFECTS

On September 29th, 2021, DESY informed that a major surface quality problem had been observed during Eddy Current Scanning (ECS) of the material provided by the same supplier for a different project. DESY's team randomly sampled our disks – all failed ECS inspection due to the same problem. Both delivered batches — 80% of the disks! — and, potentially, the entire order, were unusable.

The problem manifested as a very high level of noise observed during scanning, indicating an enormous number of irregularities on the surface. The defects appeared as pits, uniformly distributed on both sides of the disks, Fig. 1 most with dimensions ranging from 10 to 40 µm in depth and 40 to 70 µm in width, Fig. 2, and some reaching the depth of 70 µm.

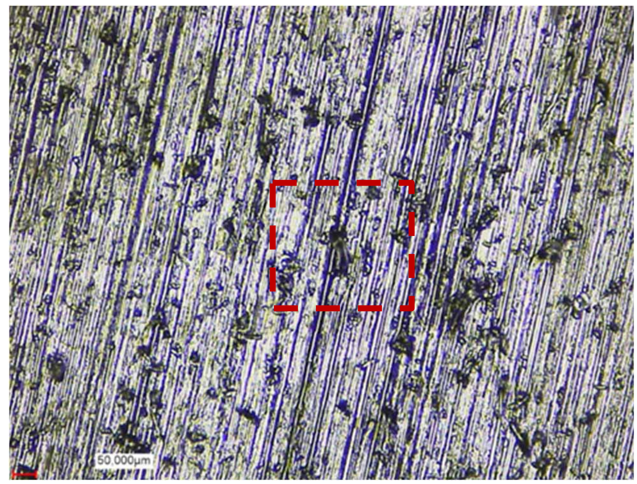
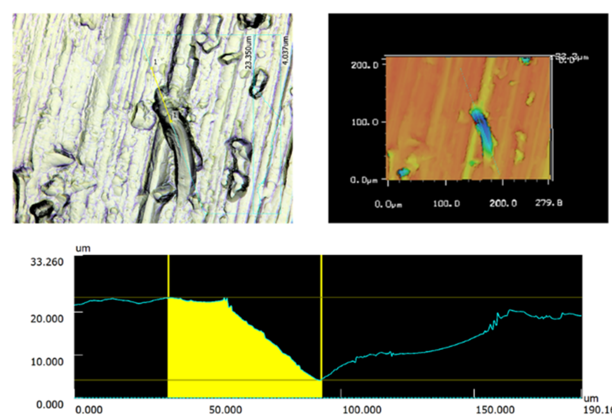


Figure 1: Half-cell material for PIP-II HB650 MHz cavities as received.

By November 2021, at least 500 sheets from various customers had been checked at DESY for surface defects. The results were very concerning - all orders placed with this supplier since May 2021 were affected. Sampling non-cell material at Daresbury supported the findings Fig. 3.



Profile1	Horz. dist.	Hght. diff.	Hght. ave.	Angle	C.S. length	C.S. area
Seg.1	57.150um	19.154um	16.386um	18.529°	66.985um	940.138...

Figure 2: 3D profile of one of the pits shown in Fig. 1.

The defects were not visible by a naked eye, the surface just appeared subjectively rougher, but when compared side-by-side with a known good material, the grinding grooves looked noticeably deeper, Fig. 4.

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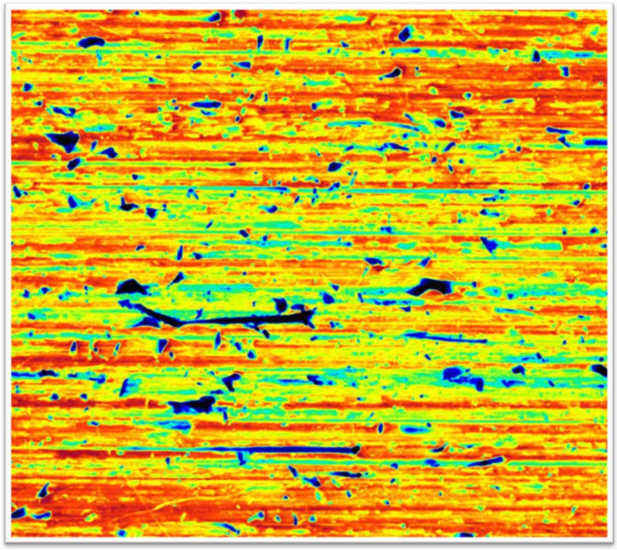

Figure 3: Beam tube material for PIP-II HB650 MHz cavities.

Figure 4: Surface of one of the PIP-II disks appears normal without magnification.

RECOVERY

First, the production was halted; this ensured that 10 of the remaining 45 discs were not subjected to the same treatment. UKRI-STFC, DESY, and FNAL, collaborated to develop a recovery plan: return all the delivered disks to the manufacturer for additional grinding and etching, and conduct a thorough investigation to identify the root cause of the problem, working with both the supplier, the manufacturer, and the other affected customers.

DESY attributed the problem to issues during the final processing steps – global grinding and the following etching. The pits were slightly larger than the particles of the final grinding media (320 mesh, 36 μm), suggesting that the etching acid was trapped under embedded polishing particles, making the pits larger instead of smoothing the surface, Fig. 5. The initial conclusion from the manufacturer in October 2021 was "insufficient etching" due to staff misinterpreting the procedure and the suggested remedy was more etching.

Several etching procedures were evaluated via 3D microscopy by the manufacturer, Fig. 6. The results were unsatisfactory for PIP-II – many pits were still present and would generate a similar level of noise during ECS. The samples were sent to DESY despite recommendations against it, and subsequently failed the inspection in December 2021. Recovery efforts for other projects were ongoing and included surface treatment tests conducted by the cavity manufacturers as well.

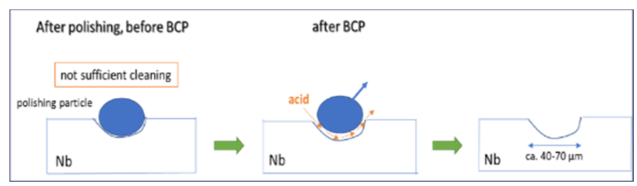

Figure 5: Defect formation schematic, by DESY.

Next, the manufacturer re-analyzed changes to the surface treatment steps since 2011, new conclusion – "excessive etching". Turns out, many changes were introduced in the recent years, including increasing the etching time in response to another customer's request related to surface cleanliness after polishing. This information was not initially shared, leading to misunderstandings and delays in resolving the issue.

Multiple combinations of grinding and etching were tested on the samples from both the original delivery, and 35 newly produced disks were evaluated via ECS at DESY.

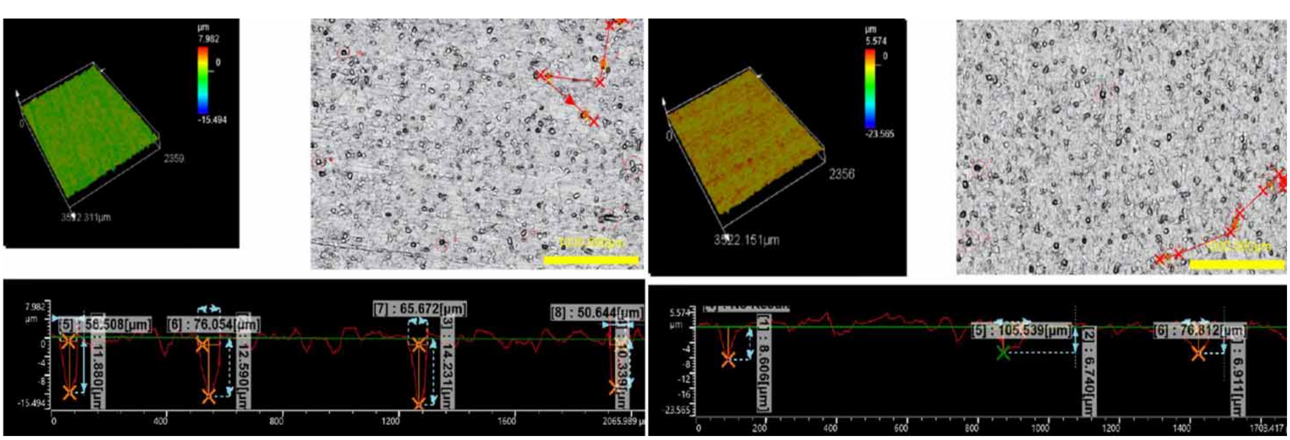

Figure 6: Surface quality after two different etching trials.

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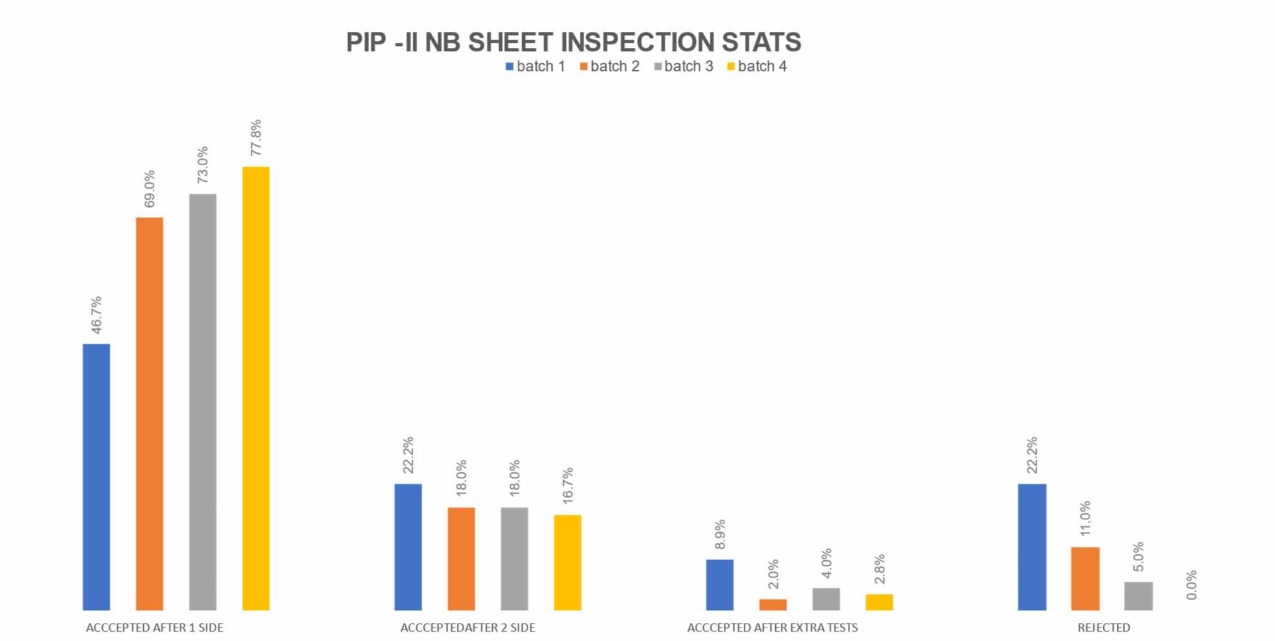

Figure 7: Nb disk batch incoming inspection performance.

RESOLUTION

To address the surface quality issues, various improvements were implemented.

Recent changes to the surface treatment were reverted, and an extra-fine polishing and etching steps were added to the final production recipe. Multiple rework recipes were applied, depending on the condition of each disk after the unsuccessful grinding and/or etching trials. However, the acceptance criteria remained the same.

UKRI-STFC introduced new Quality Control (QC) requirements in addition to the industry-standard (grain microstructure images, mechanical and chemical properties): additional thickness measurements and 3D microscopy of 2-3 randomly selected areas 2 cm^2 each on both sides. These requirements had to be adjusted to the Manufacturer's existing equipment capabilities due to the size and weight of PIP-II disks. The final report contained 3D microscopy images and data of 20 areas of 0.6 cm^2, 10 on each side, and 4 thickness measurements performed with the calipers about 6 mm from the edge of each disk.

The resulting surface quality gradually increased with each delivery, Fig. 8, meeting and exceeding a previously set benchmark in the final batch, Fig. 8.

ALL THE BARRIERS

The supplier engagement was minimal through most of the incident, placing an extra burden on UKRI-STFC staff to manage the manufacturer on top of an already challenging technical problem. The supplier attributed their absence to the difficulties they faced due to the scale of the problem across many of their orders, and general raw material market turmoil caused by the Russian invasion of the Ukraine.

To increase supplier engagement, we requested weekly reports on the status of the disks. While the exact requirement was not fulfilled, the purpose was achieved.

Due to COVID-19, international travel was extremely limited, and so direct online calls outside of regular working hours were required as teams were unable to meet in person, thereby requiring frequent, remote interactions across 4 time zones, 5 official languages, 6 corporate cultures, and nearly a dozen individual culture and language combinations. It was the time of miscommunication, surprising findings, and email riddles to solve.

For example, independently established contract and non-disclosure agreements (NDAs) directly between the manufacturer and the testing agency (DESY) imposed restrictions on sharing findings between all involved. Cultural assumptions also led the Manufacturer to incomplete sharing of information. Detailed meeting minutes and tracked action items improved the clarity of communication.

It is worth mentioning that all sides eventually did their best to improve understanding, including bringing in new people to facilitate the meetings, and trying new-to-them communication approaches. A breakthrough was finally achieved through having difficult conversations and prioritizing honesty, clarity, and openness in all our interactions.

CONCLUSION

The investigation and recovery process surrounding the sudden and significant decline in surface quality of high-purity niobium for the PIP-II High Beta 650 MHz cavities proved to be a demanding endeavor. This paper highlights the challenges faced during the procurement process, communication barriers, and technical difficulties encountered.

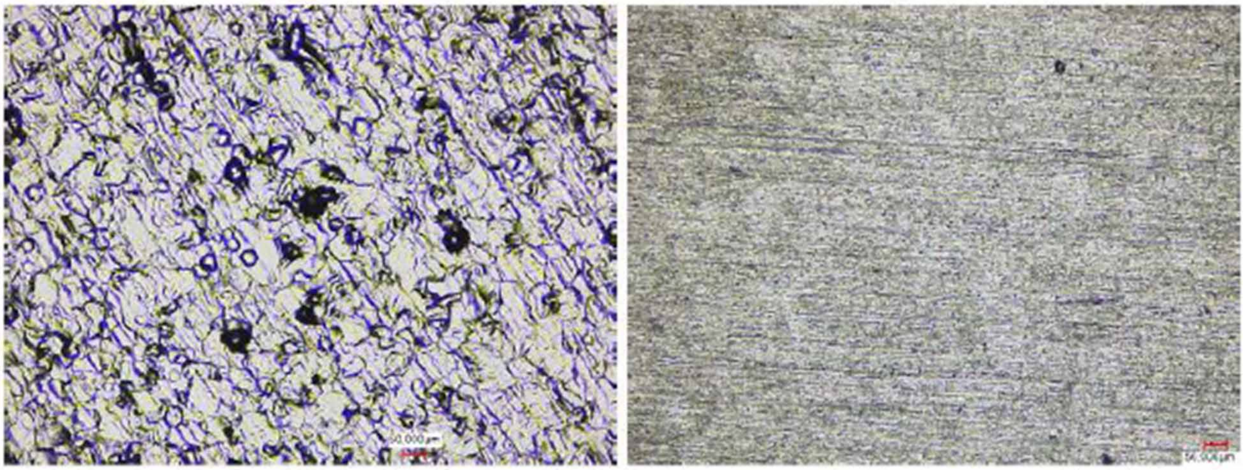

Figure 8: Surface quality after the first rework attempt (left), and the final results (right).

The root cause of the surface defects was identified as issues during the final processing steps following a recipe change, specifically, global grinding and etching. Multiple attempts were made to address the issue. The final production recipe reverted the most recent changes and introduced additional extra-fine polishing and etching steps. New pre-shipment quality control measures such as 3D microscopy and additional dimensional control were implemented. The surface quality gradually improved, ultimately exceeding the initial benchmark.

To improve future procurement processes, it is recommended to enhance communication channels and practices, and establish robust quality assurance and quality control protocols.

BUT WAIT, THERE IS MORE!

The quality problems we encountered also has potential implications on the mechanical properties and overall material behavior impacting the manufacturing process of the cavities, including shaping and welding.

Extra etching and grinding resulted in the disks narrowly meeting the thickness specification, limiting the ability to repeat cavity surface treatments, should it be necessary. Niobium thickness is also critical for meeting the cavity pressure code requirements.

Finally, some disks were rejected due to local grinding indents, and the last batch of the newly produced disks did not meet the grain size specification. The finer grain disks were accepted anyway after careful evaluation of the effect of the cavity manufacturing steps on the grain size and mechanical properties, and the risks associated with rework.

Given the history of the material, the cavity manufacturer now must take extra precautions at every step of the production, e.g., using a disk from each annealing charge and ingot combination for the pre-series cavities to verify mechanical properties. However, this decision restricts our ability to sort half-cell material to verify magnetic flux expansion properties. At the time of writing, welding of the dumbbells and end groups for the pre-series cavities is complete. We have not yet encountered any more problems.

ACKNOWLEDGEMENTS

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Information presented in this paper is based on private communications with the people mentioned above, reports provided by the supplier and the manufacturer through the duration of the contract, and my first-hand experience.