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INSIGHT INTO DESY'S TEST LABORATORY FOR NIOBIUM RAW MATERIAL AND SEMI-FINISHED PRODUCTS

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Abstract

DESY has started setting up a test laboratory for niobium more than 20 years ago. The initial application was to assure required surface quality of niobium sheets before its forming to half cells for the 1.3GHz SRF TESLA shape cavities. As a first test equipment DESY developed a basic eddy current test device which was refined continuously.
Since that time the laboratory grew with the requirements on R&D activities for niobium raw material and its semi-finished products.
To be able to assure the quality of niobium products needed for the European XFEL series cavity production, the lab's infrastructure was updated significantly. Now the capabilities of the test laboratory cover the investigation of the fundamental physical properties of various materials including for example mechanical properties, surface, microstructure and chemical composition analysis.
The Quality Assurance for the European XFEL was performed successfully on an outstanding level and in the meantime the laboratory was used for several other projects like LCLS-II and ESS.

We present DESYs test infrastructure as well as applied methods for the Quality Assurance and R&D activities and we report about experiences.

INTRODUCTION OF THE EXISTING TEST INFRASTRUCTURE

The test laboratory was created to provide the ability to qualify the material in scope of European XFEL and other projects. The qualification implies itself the destructive and non-destructive investigation of fundamental physical properties of various materials not only for SRF application but also for related material structures including joining seams.

Eddy Current Testing at DESY

For a detection of surface imperfections and defects in the niobium sheets like foreign material inclusions, scratches, pits, etc. we are operating two industrial eddy current scanning devices.

The method was initially developed for SRF applications by DESY together with the Bundesanstalt fuer Materialforschung (BAM) and the industry in the early 2000 [1]. A lab device was used successfully to assure the quality of niobium sheets for cavities which are operated in DESYs FLASH accelerator until today. To be able to fulfil the requirements on the large series production of niobium sheets for the European XFEL, two industrial fabricated machines (Fig. 1) have been ordered and operated successfully.

For the European XFEL more than 15,500 sheets were inspected.

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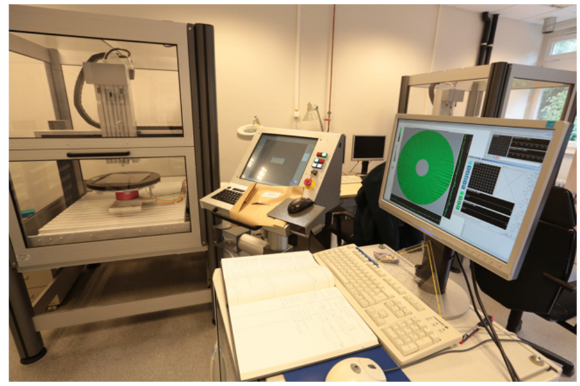

Figure 1: Eddy current scanning devices. Turn table and eddy current sensor in a safety chamber on the left, control equipment on the right.

Purity Analysis / Analysis of the Chemical Composition

Element composition analysis The suspicious areas on the Nb sheets detected by eddy current scanning are to be analysed subsequently by using the available X-Ray fluorescence element analyser (Fischerscope XDAL).

Analysis of gases contents and sulfur For an analysis of the purity of the niobium and other related compounds we are using two analysers from companies LECO and HORIBA with good experiences.

With these devices we are able to perform the so called "COHNS" measurements. Contents of carbon, oxygen, hydrogen, nitrogen-gases and sulfur can be measured by 'hot extraction method' with accuracy down to 0.1ppm.

RRR measurement Controlling the residual resistivity ratio (RRR) of high purity niobium products is the key issue for the fabrication of superconducting accelerating cavities and to fulfil the electrical requirements [2].

Our test equipment for measuring the RRR value was set up for many years; it was upgraded to the highest technical standard and is used for external services and DESYs R&D issues continuously. The measuring of RRR values is carried out mostly by temperature extrapolation method with accuracy about 3%. If required, the measurements can be performed with higher accuracy down to 1% by the magnetic field extrapolation method.

Test of Mechanical Properties

The laboratory is equipped with several devices to analyse the mechanical properties of metallic products.

Destructive A new tensile testing machine from company ZwickRoell® is available since a couple of month. It can be operated with a maximum test load of 20kN.

Two hardness test devices are available (Fig. 2). The Vickers-Method can be applied with a load range 5-50000 gf: HV0.005 - HV50.

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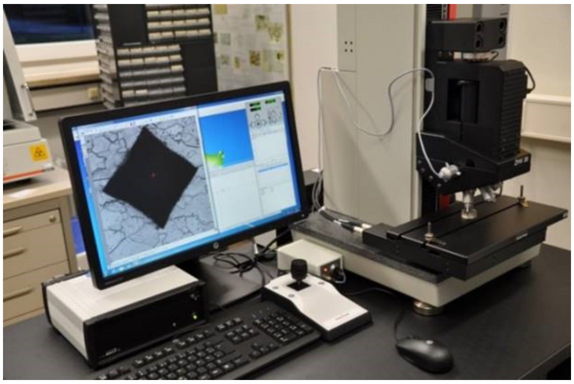

Figure 2: Hardness test device (Zwick/Roell®), sample and test head on the right.

Non-destructive An ultrasonic measuring device is available for sheet's thickness measurements, for thickness measurements of coated layers and for checking sheets and plates for potential laminations caused by the rolling process.

Surface Analysis

Surface roughness Roughness measurements can be performed on flat surfaces within a range of 350µm with accuracy down to ~ 0.08µm.

Surface profile Figure 3 shows the result of the topography of a hole that was localized roughly by eddy current scanning on a half-cell sheet. The suspicious area is observed by a 3D microscope (Keyence®) equipped with a 2.11 million pixel camera and a magnification up to 1000x.

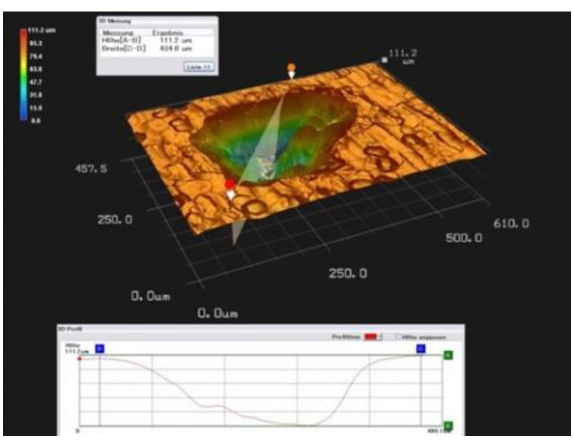

Figure 3: Result image of the topography of a defect, the diagram on the bottom shows the depth profile of the defect.

Such a 3-D profile measurement is important to be able to demonstrate that a defect is out of the specified surface condition and the affected sheet has to be rejected for re-work or replacement by its manufacturer.

Metallography By using a microscope with an extra bright 100W halogen light source and a magnification up to 1000x we are able to observe the microstructure (Fig. 4). Grain sizes can be measured by adequate accuracy to con-firm that their sizes are within the specified range and to ensure that the products can be used for fabricating cavities without any restriction.

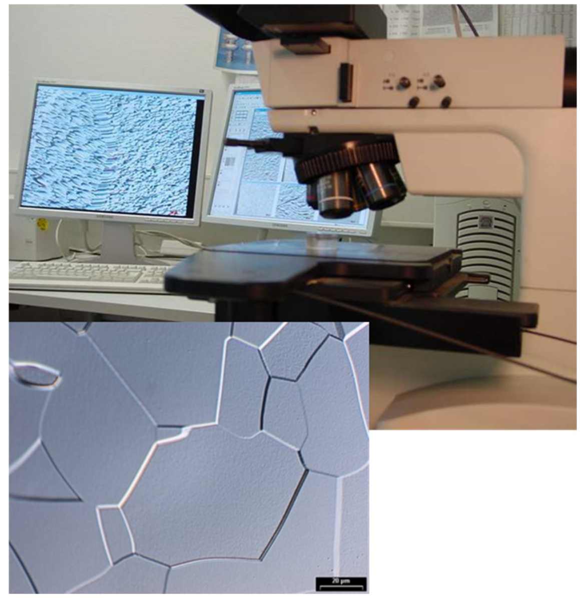

Figure 4: Olympus microscope and grain image, below a measured grain size sample.

APPLIED METHODS FOR THE QUALITY ASSURANCE

DESY has developed and applied methods to assure the quality of semi-finished products of niobium and niobium titanium alloy for the fabrication of more than 800 1.3GHz superconducting cavities for the European XFEL. Almost 25000 semi-finished products of 12 different kinds have been quality tested.

The applied methods were subdivided in several phases:

- Qualification of potential vendor for sheets for the cell fabrication of cavities by testing production sample material, single-cell cavity RF tests and nine-cell cavity RF tests [3].
- Vendor qualification for the supply of products which are connected to the pressure bearing part of the cavity according to the European Pressure Equipment Directive [4]. Very important was the survey of the companies' quality management system, the examination of the production documentation method as well as the method to guarantee the traceability from the raw material to the final product.
- None-destructive and destructive tests on semi-finished products after delivery taken randomly from the delivery batches at DESYs laboratory.

Quality Testing of Sheets for Cavity Cell Fabrication

The 1.3GHz SRF TESLA shape cavity is fabricated by using 12 different kinds of semi-finished products. The type with the absolute highest amount and quality requirements is the sheet for the half cell of the cavity production. If just one out of eighteen sheets doesn't fulfil the specified requirements, the risk is very high that the whole cavity will not perform sufficiently at the end of all fabrication

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and treatment cycles which is very time consuming and cost-intensive.

Serialized testing process DESY has developed a quality testing process (see Fig. 5) that fulfils the requirements on a mass production as well as it ensures that only qualified sheets will be used for the fabrication of cavities. This process includes the examination for surface imperfections and potential foreign material inclusions. The test procedure is carried out on 100% of the sheets.

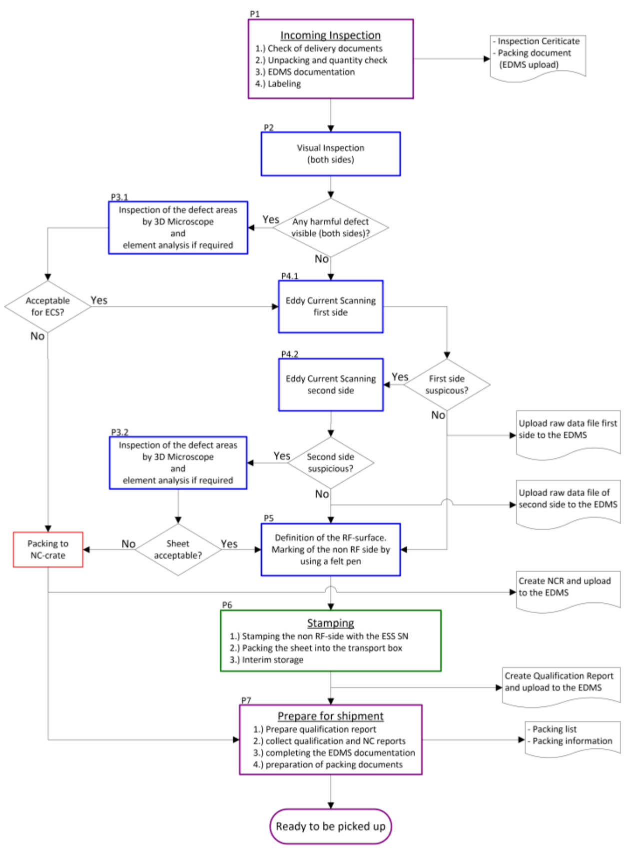

Figure 5: Work flow of the quality test process of sheets for cell fabrication.

The high number of products to be processed required also the development of a full process controlled documentation method which was realized by using DESY's Engineering Data Management System (EDMS) [5].

The EDMS controls work status of each single sheet through the testing process. The traceability from the sheet back to the raw material and its test results via the annealing lot is guaranteed. A paperless procure was realized.

Figure 6 describes the processes on a more simplified way. The eddy current scanning (ECS) is the central test method which detects suspicious areas of a potential defect.

After a detailed visual examination, the operators decide which side of the sheet should undergo the eddy current scanning examination first.

- A sheet is qualified when the eddy current scanning of the first side of a sheet gives no hint for a defect.
- The second side of the sheet has to be eddy current scanned if at the first side any conspicuousness was detected. The sheet is qualified when the eddy current scanning of the second side gives no hint for a defect.
- If suspicious areas were detected by eddy current signals at both sides, the sheet must be investigated by subsequent defect analysis to be able to decide if the sheet can be accepted or must be rejected finally.

Subsequent defect analysis is done by non-destructive investigation by means of optical microscopy, 3D profilometry, surface roughness measurement and element analysis if necessary.

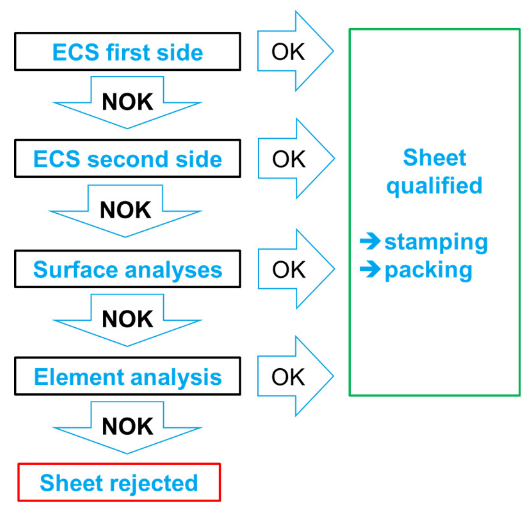

Figure 6: Simplified testing process flow.

Mechanical and electrical properties Additional tests to examine the mechanical and electrical properties of the sheet have been performed independent from the manufacturers' tests. Samples are taken from the sheet corners randomly from each annealing lot. By these tests the test result of the material properties and the correctness of the measurements performed and reported by the inspection certificate of the manufacturer should be confirmed.

EXPERIENCES

Based on the experience gained at the initial stage of the European XFEL project, a feedback to the manufacturer gave a significant improvement in the quality of the material produced later. A close contact to the niobium vendor before start of the production and within the running production phase is an indispensable tool of the quality management and one of the key requirements for a successful fabrication of high quality products.

It is undisputed that the Quality Assurance of high RRR niobium semi-finished products is essential and a main keystone to realize high gradients in superconducting RF cavities.

Since the successful activities for the European XFEL project DESY was asked to provide the quality assurance for half-cell sheets as service provision for other projects

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too. In some cases DESY operated as sub-contractor for cavity manufacturing companies.

Table 1 summarizes the number of sheets which have been tested since 2010 in relation to its project affiliation. Not taken into consideration are tested sheets which had to be/have to be re-inspected after its rejection.

Table 1: Overview of Tested Sheets

Number of sheets	Project affiliation	Testing period
15753	European XFEL	2010-2014
9158	LCLS-II	2015-2018
428	ESS (m-beta)	2018
924	ESS (h-beta)	2018-2019
24	PIP-II (prototypes)	2019 July
368	SNS PPU	2019 Q3

All test devices for the half-cell sheet inspection process are adapted to the sheet size for the European XFEL. These sheets have the dimension of 265x265x2.8mm.

Because of the different sheets dimension for other applications (see Table 2) the devices had to be modified. The biggest adaptions are necessary for the eddy current scanning devices which have to be realized for each sheet size change. New clamping tables are to be designed and fabricated. Parameter adaptions have to be carried out in the machine control software for the scanning speed, for the surface to be scanned and for the vertical axis movement that must fit to the sheet's thickness.

Table 2: Difference in Sheet Sizes

Project affiliation	Application	Sheet sizes [mm]
European XFEL	1.3GHz Cavity (Tesla shape)	265x265x2.8
LCLS-II	1.3GHz Cavity (Tesla shape)	265x265x2.8
ESS	704 MHz medium beta cavity	460x460x4.3
ESS	704 MHz high beta cavity	D500x3.9
PIP-II	650MHz prototype cavities	OD470xID72x4.4
SNS PPU	805MHz High Beta (0.81) cavities	D410x4

RESULTS

We are describing here exemplary the results of the sheet inspection for the ESS project DESY finished recently, see Figs. 7 and 8 [6].

Some Statistics

1406 sheets/discs have been inspected by DESY excluding the re-inspection of rejected sheets. Around 92% could be accepted by eddy current scanning; almost 85% could be accepted by scanning of just one side.

On 7.8% (109 pcs) we detected suspicions areas by eddy current scanning on both sides of the sheets/discs. A detailed defect analysis was carried out subsequently. The surface in those areas in which were given strong eddy current signals was investigated by 3D-microskopy and element analysis mainly.

Finally 2.7% (36 pcs) had to be rejected; due to surface imperfections; foreign material (iron) was observed at two sheets.

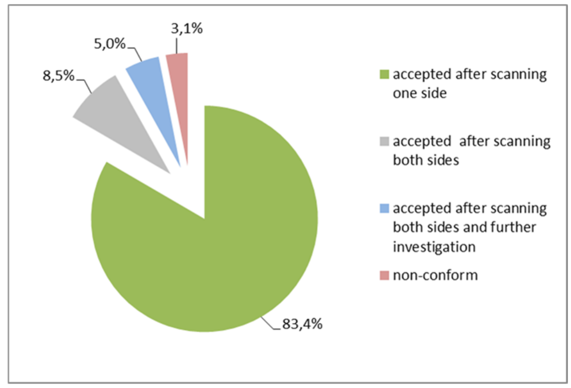

Figure 7: Inspection results of ESS square sheets.

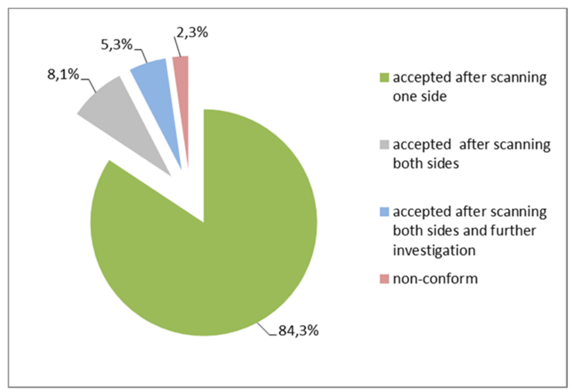

Figure 8: Inspection results of ESS round discs.

Rejected Sheets

The rate of sheets to be rejected is within the same range DESY had to reject during the inspection of half-cell sheets for the European XFEL [7]. This is in particular remarkable by taking into consideration the much bigger surface of the sheets for the ESS project.

CONCLUSION

The laboratory is operated to assure the quality of high purity niobium products for various SRF accelerator applications.

DESY implemented successfully an inspection process which fulfills the requirements on a series cavity production on an almost industrial way.

The test infrastructure is continuously upgraded for keeping it at the newest technical standards.

The diversity of lab's equipment gives the way in order to investigate the different physical properties also on alloys based on stainless steels, copper, aluminium etc.

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The qualification of complex structures including joining seams can also be carried out.

Meanwhile the laboratory with its manifold possibilities is used also for many other applications at DESY outside the SRF field.

ACKNOWLEDGMENT

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