Nb-RRR SHEET INSPECTION BY MEANS OF ULTRASONIC MICROSCOPY

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Abstract

Nb-RRR sheet material is one of the key components of super-conducting linear particle accelerator projects (e.g. XFEL, ILC). The high quality requirements led to sophisticated quality systems in the manufacturing line. A major aspect is the development of non-destructive inspection methods for the detection of surface defects, delaminations, pores, inclusions and impurities. Up to now the standard inspection technologies for quality assurance of Nb-RRR sheet material are based on electromagnetic techniques, e.g. SQUID and eddy current. For these methods the detection limit is in the range of 0.1 mm.

Ultrasonic microscopy (USM) is a well established and economic technique for non-destructive surface inspection. For such applications strongly focused ultrasonic waves are applied. For volume inspection of sheet material the focal length of the ultrasonic transducer needs to be increased in order to enable constant defect resolution throughout the whole cross section of the material under inspection. For Nb-RRR sheets with typical thickness of 2.8 mm a detection limit of 0.1 mm is expected. First results of USM on Nb-RRR sheet material are presented.

INTRODUCTION

Sheet material made of ultra-high-purity Niobium (so called Nb-RRR grade) is the key component for future linear accelerators, based on the superconducting RF technology. One is the XFEL (X-ray Free Electron Laser) whose construction started in 2007 at the German Electron Synchrotron in Hamburg. Based on the same technology is the ILC (International Linear Collider) which is currently in the design phase and will be the next major accelerator project in particle physics after XFEL realization.

Since 2004 Plansee SE (A) worked on the qualification as material supplier for the XFEL project. In co-operation with W. C. Heraeus (DE), which covers the raw material competence, Plansee SE established the industrial production of products made of Nb-RRR in various geometries and sizes. In December 2007 the qualification procedure at DESY could be successfully finished and Plansee SE was qualified as supplier for Nb-RRR 300 sheet material.

To be prepared for large production scale quantities for the XFEL project, activities for the installation of a Quality Assurance (QA) management system are ongoing. A major quality criterion for Nb-RRR sheets is the surface quality. For inspection of surface quality visual inspection and eddy current testing (ET) are established for pre-series production and future project realization as baseline. Both methods are commonly used for detection of defects and inclusions at the surface. For volumetric inspection SQUID (Superconducting Quantum Interference Device) was investigated during the last years and activities for establishment of a test standard are in work [1-4].

With ultrasonic microscopy (USM) an equivalent test procedure is available which allows a standardized surface inspection on industrial scale. For USM an automized visualization of surface defects and establishment of standardized test records is possible [5]. With adaptation of test setup the possibility of volumetric testing was assessed and first results will be presented.

ULTRASONIC MICROSCOPY

Ultrasonic microscopy (USM) is established for the inspection of thin films in various industrial applications, e.g. electronics [6]. USM uses ultrasonic frequencies from 50 MHz to 2 GHz which is much higher compared to conventional ultrasonic NDT operating frequencies using about 1 to 20 MHz. This leads to enhanced defect resolution since the wavelength is reduced to the µm regime. An additional feature is the strong focussing of the ultrasonic wave which leads to lateral resolution in the magnitude of microns in the focused plane. For high resolution inspection in the volume the focus of the transducer is moved below the surface [7]. For inspection of the whole sample volume several scans need to be performed.

For the application described a device of the type Krämer Scientific Instruments, Vario III B is used. The inspection is performed in water immersion applying the pulse echo technique. A scanning mechanism moves the transducer and enables the generation of a C scan image. A sketch of the setup is given in Fig. 1.

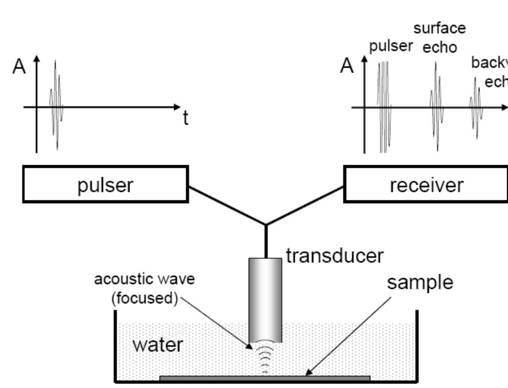

Figure 1: Sketch of the measurement chain of the USM.

EXPERIMENTAL WORK

Nb-RRR Sheet Material

Nb-RRR grade material is achieved by electron beam melting under high vacuum. Starting from premelted blocks with low content of metallic impurities (esp. Ta content) the material is remelted several times to reduce the impurities step by step. Especially the interstitial dissolved non-metal elements (C, O, N, and H) must be reduced to levels below 10 ppm to achieve RRR values > 300 on the semi finished products.

The melted cylinders were processed to plates by a multi-stage conversion process followed by sheet rolling. During processing the parameters must be carefully controlled to prevent reaction with the atmospheric gases. Due to the softness of the material and the cold-welding tendency of niobium a careful handling during processing is necessary to prevent surface defects like scratches, pinholes or metallic inclusion. To keep the high purity level over the whole production route, all processing steps must be well defined and controlled.

After sheet rolling to typical thickness of 2.8 mm, surface treatment and tailoring according customer specification is made. Small defects or inclusions on the surface can lead to severe problems in cavity performance. Therefore, beside the visual inspection, a test method which allows a standardized detection of surface defects is necessary for establishment of a QA management system for industrial production.

Test Results Surface Inspection

For maximum resolution of the surface inspection the ultrasound frequency needs to be as high as possible. Fig. 2 compares the C scan of a defect indication obtained from a 75 MHz and a 150 MHz transducer detected on a Nb-RRR 300 sheet. The grey value code in the C scan gives the locally measured amplitude of the surface echo.

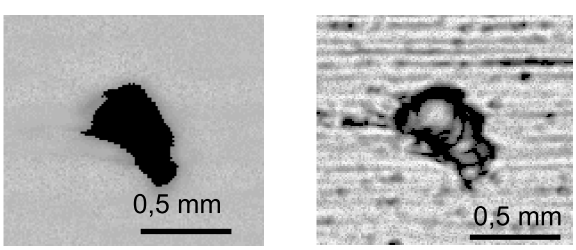

Figure 2: Comparison of the C scan obtained from a 75 MHz (left) and a 150 MHz (right) transducer showing a surface defect.

From Fig. 2 it is seen that the 150 MHz transducer displays the surface roughness which is within the specification (e.g. typical measured value: Rmax. = 17 µm). To allow an automized surface inspection the resolution needs to be reduced in order to discriminate the indications from the surface structure. This is possible by application of the 75 MHz transducer with a focal length of 8 mm; see Fig. 2 (left side).

The testing procedure was applied to 15 Nb-RRR sheets with dimension 265 x 265 x 2.8 mm. The material was produced under production conditions. USM testing was performed applying the 75 MHz transducer. An example for a C scan of the surface inspection is given in Fig. 3. The image is free of artefacts and enables a clear defect representation (see marked defect indication in Fig. 3).

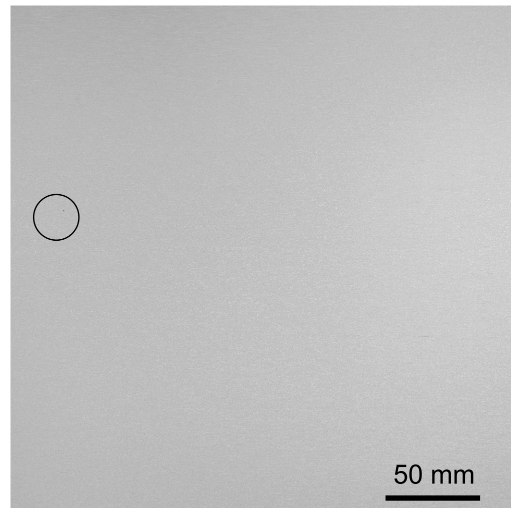

Figure 3: C scan of the surface inspection of a Nb-RRR sheet (265 x 265 x 2.8 mm, part no. C4 2.1-11). The location of a small defect is indicated by the black circle (detail see Fig. 3).

Test Body Preparation for Volume Inspection

For first assessment of applicability of USM to volumetric inspection of Nb-RRR sheets and determination of the detection limit a test body was prepared. Corresponding to sheet thickness for cavity production on a Nb-RRR sheet with thickness 2,8 mm different holes with diameter 0.2 mm and depth between 0.2 and 1.4 mm were drilled on one side of the sheet. Simulating punctual volumetric defects (pores, inclusions) the tip of the blind holes has an angle of 118 °. Therefore the detection of the artificial defects is more challenging than for flat bottom holes commonly used for defect resolution determination in non destructive testing. In Fig. 4 the location and dimension of the artificial defects is shown schematically.

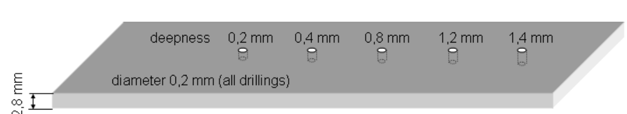

Figure 4: Schematic sketch of the test body for assessment of detection limit for volumetric defects (sheet thickness 2.8 mm).

Test Results Volume Inspection

For volume inspection of the Nb-RRR sheets a 50 MHz ultrasonic transducer is used. The use of an increased ultrasonic frequency compared to standard ultrasonic inspection increases the defect detection capability. A focal length of 50 mm was chosen. This avoids multiple scanning with different focus adjustments which would be necessary for a focussed transducer. The result obtained from the inspection of the test body described above is given in Fig. 5.

Figure 5: C-scan representation of the defect echo obtained from the test body (see Fig. 4). The white points indicate the defect echo from the 0.2 mm drillings.

The artificial defects of the test body can be recognized in the C-scan image regardless of their depth location. Therefore the capability of volume inspection with a defect resolution of 0.2 mm is proven for the setup.

The volume inspection was also performed for the 15 Nb-RRR sheets of production lot "C4" described above. No volume defects were detected.

DISCUSSION

For surface characterization Nb-RRR sheets prepared under production conditions with thickness of 2.8 mm and surface condition according to XFEL specification were used. USM was performed using a 75 MHz and a 150 MHz transducer with a focal length of 8 mm and 3.2 mm respectively. Using the 150 MHz transducer even the surface structure is displayed. Using the 75 MHz transducer the signal from the surface structure can be discriminated. Therefore this transducer is chosen for industrial application. The sensitivity of USM for surface defects is proven to be in the magnitude of 0.1 mm which is confirmed by light microscopy. To meet the specified detection limit the transducer frequency must be adapted individual according valid specification.

For determination of the defect resolution in the volume a test body of Nb-RRR sheet material was prepared. Blind holes with a diameter of 0.2 mm and a tooling angle of 118 ° were introduced in different depth. The use of a 50 MHz transducer with a focal length of 50 mm enables the detection of such point like defects through the body. Future work will focus on the detection of embedded material inclusions, e.g. Ta.

CONCLUSIONS

USM was assessed as inspection tool for surface and volume inspection of Nb-RRR sheet material. With use of specific test setups the detected surface defects stand in good correlation with results from visual inspection. Concerning volumetric defects the resolution was demonstrated by means of artificial defects. A continuation of this activity is foreseen.

By use of transducers with long focal length a multiple scanning for volumetric inspection can be avoided. Therefore, reasonable scanning times for volumetric inspection can be realized.

For large production scale a standardized rating of the material quality under inspection is demanded. USM meets this requirement by an automized establishment of test records containing a clear display of defects.

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