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DIAGNOSTIC OF DEFECTS IN HIGH PURITY NIOBIUM

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The investigation of the quench area of TTF cavity D6 confirms a necessity of a quality control of the Nb sheets. A small cluster with high Ta content was detected in D6 by means of X-ray micro radiography. The identification and estimation of Ta concentration was done via Synchrotron Radiation Fluorescence Analysis (XAFS, SURFA).

Some methods of nondestructive diagnostic of Nb sheets (X-ray radiography, neutron radiography, neutron activation analysis NAA, XAFS, SURFA, ultrasonic-, eddy current inspection, microhardness measurement) were analysed for check of efficiency.

The eddy current method was chosen as most suitable for detection of defects (rather fast, sensitive to different sorts of defects, has a high resolution). An eddy current scanning system (with probes specially developed for this purpose) is created and about 700 Nb sheets are tested. The penetration depth of the signal is about 500 μm.

SURFA and NAA are applied for supplemental nondestructive identification and investigation of detected defects. The first method is more efficient for analysis of layers close to the surface (with a penetration depth between few μm and few hundred μm), NAA delivers the information about bulk Nb and demonstrates very high sensitivity to Ta inclusion in Nb.

Some results are discussed. For example some small iron spots probably imbedded during rolling were detected in one Nb sheet with the help of the eddy current system. The sort of the inclusion and its three dimensional profile was determined by SURFA.

Keywords: Superconductivity; Radiofrequency; Cavities

1 FOREIGN MATERIAL INCLUSION IN TTF CAVITY D6

Traditionally the quality control of high purity niobium deals with three aspects: purity, workability and surface quality. Another

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problem, which becomes more important for high gradient cavities, is the nonhomogeneous distribution of foreign elements in Nb.

The cold test in the vertical cryostat of cavity D6 before and after post purification with Ti has shown, that the dependence of Q on field E almost does not change and is limited at 13 MV/m. The worst performance shows the cell 5. The application of a rotating T–R mapping system, which is applied at DESY for diagnostic of the hot spots in TTF 9-cell cavities, detected the sharp temperature increase in the definite area of the 5th cell[1](Figure 1).

The eddy current inspection of this cavity from outside with an extremely sensitive probe was done at the BAM (Bundesanstalt fuer Materialforschung und -pruefung, Berlin). A signal deviation at the same area was found. At the same time a careful inspection of the inner surface by means of an endoscope system did not demonstrate any kind of disturbance.

The dumb-bell was cut out from cavity D6 for further non-destructive investigations. The X-ray micro radiography with area detector and high spatial resolution (about 10 μm), that was done at BAM, allows to discover a black spot on the photograph of the analysed Nb area (Figure 2). The cross section of the spot is about 0.2–0.3 mm and the shadow indicates a foreign material inclusion with higher density and atom number in comparison with Nb.

The next step was the nondestructive identification of the inclusion. First of all the thermal neutron radiography facility Gentra-3 of

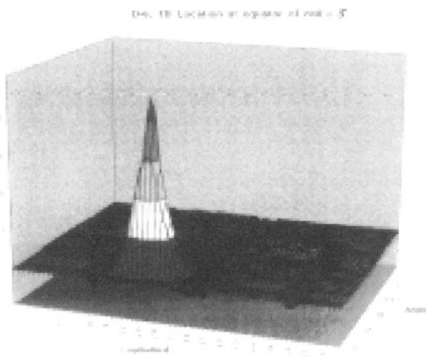

FIGURE 1 Temperature distribution in cell 5 of cavity D6 during the RF test.

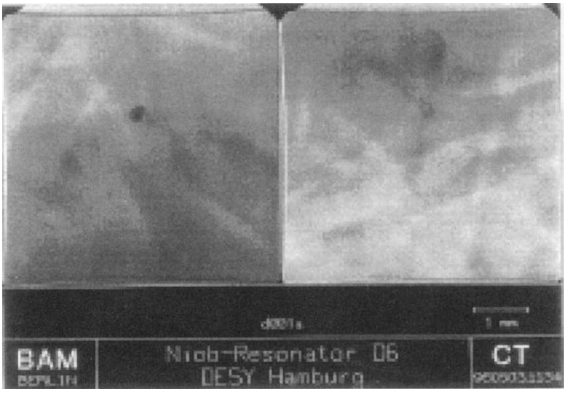

FIGURE 2 Spot area of a dumb-bell of cavity D6. Positive print of a X-ray radiograph.

the GKSS (Forschungszentrum Geesthacht) was used, which is designed for the examination of large objects. The attenuation of the beam is due to the interaction of neutrons with nuclei. The efficiency of the method depends on the absorption coefficient of the object and it differs from X-ray method. In any case some elements can be a strong absorber for X-ray but they are transparent for neutrons and vice versa. Unfortunately the absorption coefficient of the inclusion material is close to Nb (how it turns out later) and therefore this irradiation test was without success. Figure 3 shows a negative print of the spot area of the dumb-bell from cavity D6.

Identification of the inclusion was done at DESY in Hamburger Synchrotronstrahlungslabor HASYLAB. The synchrotron radiation produced at Hamburger storage ring DORIS can be used for identification of very small inclusions of different chemical elements, thanks to high intensity. The most important method for this is the fluorescence analysis. Fluorescence appeared during spontaneous returns of exited atoms or molecules to the basic status. Owing to the fact that the synchrotron radiation has a high spread of energy (from visible light till the hard X-ray) the tunability of synchrotron radiation allows the selective excitations of elements.

There are two variations of the fluorescence method. In synchrotron radiation fluorescence analysis SYRFA the excitation is done with the white beam and a semiconductor detector analyses the energy

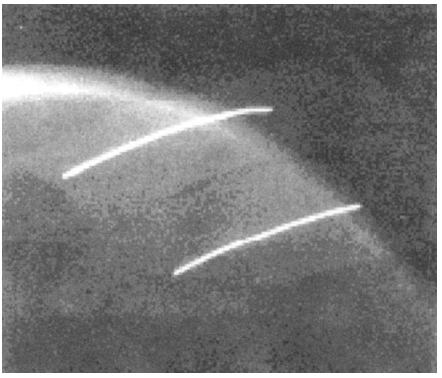

FIGURE 3 Spot area of a dumb-bell of cavity D6. Negative print of a thermal neutron radiograph.

of the fluorescence. In the XAFS topography method the energy selection occurs in the primary beam. One observes the absorption edge of definite elements.

The advantage of the fluorescence method is high surface resolution (few micrometers) and a very high sensitivity (sometimes few particles per billion). The disadvantage is the small penetration depth (between few tenth and a couple of hundreds of micrometers). This allows the detection of element traces close to the surface only.

Both versions of the fluorescence method were applied in our case. The XAFS method has shown, that the fluorescence takes place in the area that is interesting for us at energies close to $L_3 = 9{,}881$ keV, $L_2 = 11{,}136$ keV, $L_1 = 11{,}682$ keV, which corresponds to the L lines of tantalum. Certainly the Nb reflexes are also presented.

The SURFA method has allowed more detailed investigation of the Ta inclusion. The fluorescence experiment was carried out in a wide energy range from 0 to 80 keV. At first a spectrum far away from the spot was recorded, then the second one (dotted line) in the middle of the spot. Two spectrums of K-lines synchrotron radiation fluorescence can be seen in Figure 4, that lay on top of each other.

Both spectrums display the Nb lines and in addition Ta lines (Ta-Kα1 = 57,532 keV, Ta-Kα2 = 56,277 keV, Kβ1 = 65,223 keV) according to the vendor specification. The content of dissolved Ta in Nb is roughly 200–300 ppm. This Ta is responsible for the reflex

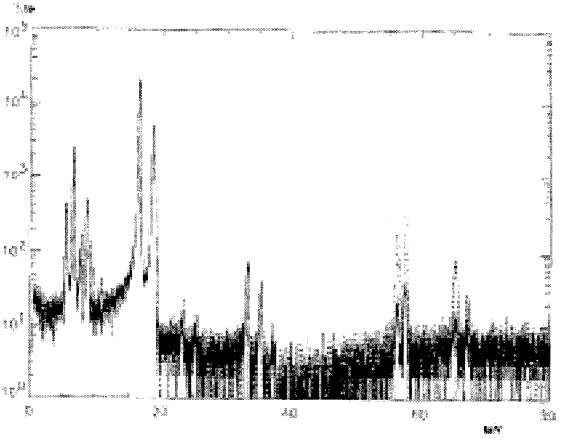

FIGURE 4 Spectrum of K-lines synchrotron radiation fluorescence in the Ta spot area (dotted line) and far away of it (full line).

obtained away from the spot (Ta background). But in the middle of the spot the Ta signal increases by a factor of 10. This means that Ta is not completely dissolved and this area represents a cluster of Nb–Ta alloy with a Ta content close to the surface of about 2000–3000 ppm, which is uncommonly high. The spot area was scanned in two perpendicular directions (Figure 5). It turns out, that the spot has an oval shape with a size of about 0.5 mm in one and 1 mm in another direction.

A model of the inclusion can be imagined under consideration of X-ray experiments. It consists of a nuclei inside the Nb with a rather high concentration of Ta, that can be registered by means of X-ray. There is a halo around the nuclei with less Ta concentration. The halo is spread rather widely and reaches the surface. This is confirmed by synchrotron fluorescence. Figure 6 represents schematically the described picture. The impurity distribution in the spot area is typical for incomplete dissolution of the components during melting. This event is a bit surprising because the Nb ingot was melted few times in the EB furnace.

The Nb–Ta alloys create a solid state solution in the whole concentration region within 0–100%, which is well known from its phase diagram.[2] One should expect the complete dissolution of the Ta

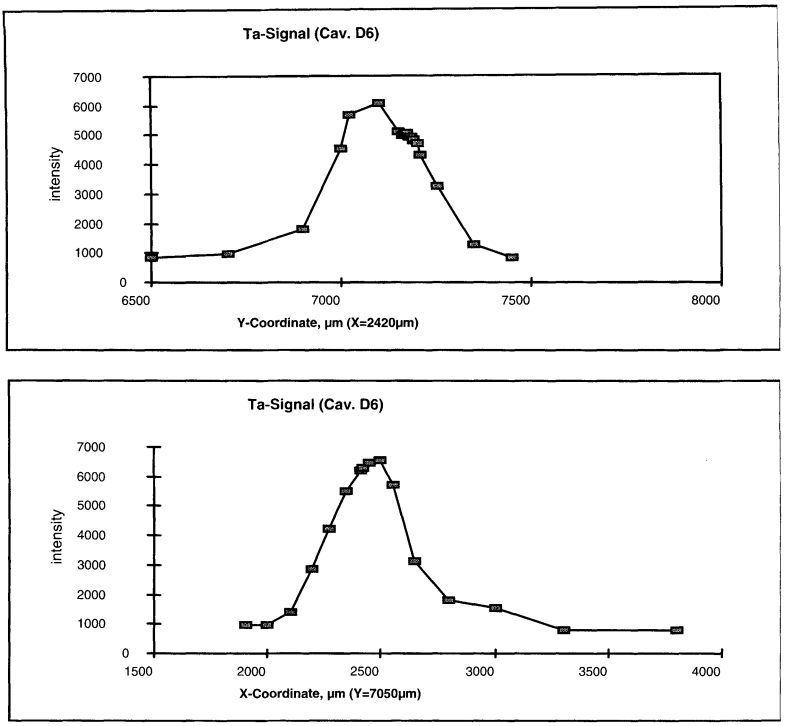

FIGURE 5 Sizes of Ta spot in cavity D6.

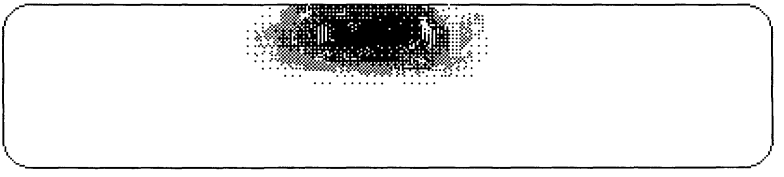

FIGURE 6 Imagination of Ta inclusion in the cavity wall.

component. Nevertheless the melting point of Ta is roughly 500°C higher than that of Nb. If the temperature was not sufficiently high during the melting or the melting time was too short this phenomena can happen in principle.

The obtained content of Ta in the spot gives the opportunity to estimate the RRR with the help of the empirical formula, which

TABLE I Comparison of RRR values for Nb in the spot and outside

	O (μg/g)	*N* (μg/g)	*C* (μg/g)	*Ta* (μg/g)	*RRR* (calc.)
Nb-300					
Before solid state gettering	2	1.5	1.5	200	335
After solid state gettering	0.04	0.45	0.45	200	475
Ta-spot					
Before solid state gettering	2	1.5	1.5	2000	56
After solid state gettering	0.04	0.45	0.45	2000	59

describes the influence of different impurity elements on RRR.[3,4]

$$RRR = \frac{R(300\,\mathrm{K})}{R(10\,\mathrm{K}) + \sum_{i=1}^{4} \frac{\partial R_i}{\partial C_i} C_i}$$

$i = 1(=\text{oxygen}), i = 2(=\text{nitrogen}), i = 3(=\text{carbon}),$
$i = 4(=\text{tantalum})$

$R(300\,\mathrm{K}) = 1.46 \cdot 10^{-5}\,\Omega\,\mathrm{cm}, R(10\,\mathrm{K}) = 8.7 \cdot 10^{-9}\,\Omega\,\mathrm{cm}$

The values $\partial R_i/\partial C_i$ have been found by using pure and doped[4] samples: for N: $3.49 \times 10^{-9}\,\Omega\,\mathrm{cm/wt.\,ppm}$; C: $3.33 \times 10^{-9}\,\Omega\,\mathrm{cm/wt.\,ppm}$; O: $2.64 \times 10^{-9}\,\Omega\,\mathrm{cm/wt.\,ppm}$; Ta: $0.12 \times 10^{-9}\,\Omega\,\mathrm{cm/wt.\,ppm}$, respectively. The results of calculation can be extracted from Table I. The Ta contribution in the spot determines the RRR value. It is about 60 before post purification (much less than 300). Unfortunately the solid state gettering is not in a position to reduce the Ta concentration in Nb. Therefore even after post purification, when the content of oxygen, nitrogen and carbon is significantly reduced, the RRR value remains almost the same.

2 ANALYSIS OF METHODS APPLICABLE FOR A NONDESTRUCTIVE DIAGNOSTIC OF Nb SHEETS FOR TTF

The example with cavity D6 shows, that a small spot only in one sheet damages the complete performance of the cavity. It is evident, that a total quality control of a Nb sheets is necessary in order to achieve further improvement of cavity performance.

Embedded inclusions, voids, cracks and scratches deeper than 15 μm are not tolerable in the Nb sheet. Surface control of Nb is usually made by visual inspection, anodization and looking for discoloration, water soaking and looking for rust traces. It should be taken into account, that removing 100–200 μm of material from the surface occurs during cavity preparation. Inner defects that locate close to the surface will be uncovered. This means the quality control should be done both at the surface and inside of the Nb in the areas adjoining to the surface area.

What kind of quality control is necessary for Nb sheets? Some important aspects of quality control can be pointed out:

- it should be nondestructive;
- it should be total. At least one side of the sheet should be scanned. The penetration depth of the signal should be not less than 0.3 mm;
- it should be fast. The scanning time of one sheet should be not longer than 1 h;
- it should have a high resolution, defects with a size of 100–500 μm should be detectable;
- it should have a high sensitivity to clusters with small deviation of impurity content.

The analysis of the advantages and of the disadvantages of different methods of quality control is demonstrated in Table II. The consideration of negative and positive aspects of different methods of nondestructive inspection for identification of nonhomogenates in Nb points out, that the most suitable method for our aim is the eddy current control.

This procedure allows to control the surface and the areas adjoining the surface area. The penetration depth can be changed due to the frequency choice.

Modern eddy current facilities can scan large areas with rather high speed. High resolution can be achieved by optimising the electrical and mechanical parameters of the probe. It is possible to detect defects of a hundred micrometers size in the depth of few hundred micrometers.

TABLE II Methods of defects diagnostic in Nb-sheets (265 × 265 × 2.8 mm)

Method	*Principle of the method*	*Penetration depth*	*Resolution*	*Destructive or nondestructive (test time of the sheet)*	*Remarks*
X-Ray radiography	Difference in X-ray absorption (defects and Nb)	Complete	Depends on the test part size (10 μm– 1 mm)	Can be nondestructive (about 30 min)	"Shadow picture" depends on the difference in density and atom number
Neutron radiography	Difference in neutron absorption (defects and Nb)	Complete	Depends on the test part size (10 μm–1 mm)	Can be nondestructive (about 60 min)	"Shadow picture" depends on specia quality of isotopes. Good detection of light elements
Ultrasonic	Reflection of sound waves at interface	Complete (need a coupling)	Up to 50 μm	Nondestructive (about 30 min)	Nonhomogenates in metals are difficult to inspect
Eddy current	Electro-magnetic induction	Depends on frequency (μm–mm)	Up to 100 μm	Nondestructive (about 30 min)	
Neutron activation analysis	Irradiation with thermal neutrons. Measurement of γ-spectrum	Complete	Detection of clusters with sizes up to 100 μm	Nondestructive (about 15 h for 1 sheet)	Very sensitive to Ta inclusion. Some ppm of Ta in Nb can be detected
Synchrotron fluorescence analysis SYRFA	Excitation by white beam (analysis of fluorescence energy spectrum)	1–100 μm	Up to 1 μm	Can be done nondestructive (it needs some hours for inspect. of area 20 × 20 mm)	K-Lines (Energy about 0–80 keV). Sensitivity: up to few ppm of impurity content

TABLE II (*Continued*)

Method	*Principle of the method*	*Penetration depth*	*Resolution*	*Destructive or nondestructive (test time of the sheet)*	*Remarks*
Synchrotron fluoresence analysis XAFS	Energy selection in the primary beam. Observation of the absorption edge	About 10 μm	Area 12 × 12 mm can be tested in one step	Can be done nondestructive (5 h for inspection of area 150 × 100 mm)	L-lines (Energy about 0–10 keV) Sensitivity: up to few ppm of impurity content
Microhardness	Intruding of the diamond pyramid	Depends on loud value. For Nb normally about 10 μm	About 50 μm	Conditionally nondestructive (damage layer will be removed)	Sensitivity depends on the hardness differences

3 EDDY CURRENT SCANNING SYSTEM

A new eddy current system was set up by DESY. The conceptual design, development of the probes, software and the scanning system was done by BAM. Schema of the eddy current scanning system for TTF Nb sheets can be seen in Figure 7.

ELOTEST PL.E (Rohmann GmbH) was chosen as device for receiving eddy current signals. The normally used frequency is about 100 kHz, that corresponds penetration to a depth in Nb of about 0.5 mm. The small probes usually are very sensitive to variation of the distance between probe and scanning surface. An air pressure pillow principle was applied in order to avoid at least the main consequences of this problem. Besides the elimination of the friction between probe and scanning surface brings an additional advantage. The probes contain two coils with sizes of about 3–5 mm created on the basis of absolute measurement.

The scanning system allows to reach a maximal probe speed of 1000 mm/s. The data acquisition takes place when the probe is

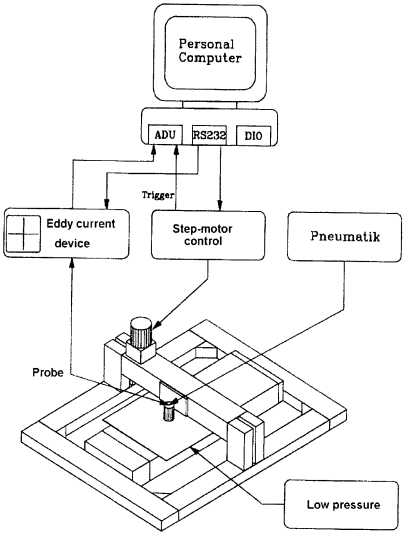

FIGURE 7 Scheme of the eddy current scanning system.

moving in both directions. The main software consists of two separate parts, first one for the measurement and the second for estimation of the dates. The Nb sheet is pressed to the stretching table after having pumped out the air under the sheet. This principle allows to make the sheet plane and to reach the required accuracy.

First of all the system was tested on holes drilled in the Nb sheets with 2.8 mm thickness. The depth of the holes was about 1–2 mm, the diameter within 0.1–1 mm. The measurement from both sides of Nb sheet allows to check the eddy current signal not only from the open holes, but also from the covered with Nb holes. It turned out that some probes reach the required purpose to detect the pores with a diameter 100 μm. However, in this case the signal was comparable with the noise level. The detection of holes with a diameter of 0.2 mm and higher can be done without any problem.

In order to pay attention to the experience with cavity D6 a test sheet with artificially imbedded Ta inclusions was created. The drilled holes in the Nb sheet were filled out with Ta wire, then those locations were melted with electron beam and finally the complete sheet was polished. The sizes of the Ta defects and their distribution together with the results of the eddy current scan of the Ta test sheet can be seen in Figure 8. The sensitivity and resolution of the system is sufficient for a reliable registration of Ta defects.

At the same time the sensitivity of the created eddy current system is so high, that even rather small geometrical deviation on the surface within 50–100 μm (local grinding marks, rolling imprints and so on) can be very clearly registered. In principal the geometrical deviations are harmless for cavity performance.

A microhardness measurement is a good way to separate quickly the sheets with material inclusions from the sheets with geometrical deviations on the surface. The application of this method in our case can be considered as nondestructive, because the intruding depth of the diamond pyramid is less than 10 μm and the diamond damaged layer will be etched away during later preparations. The efficiency of the microhardness measurement is demonstrated in Figure 9, where the line through one of the Ta inclusions in the Ta test sheet was measured.

The eddy current quality control of 715 new Nb sheets for TTF from three suppliers (company A,B,C) was done. The preliminary

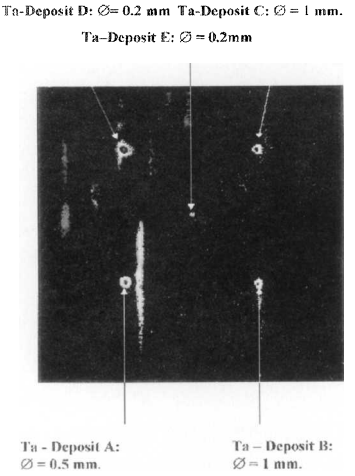

FIGURE 8 Distribution of the eddy current signal in the Ta test sheet.

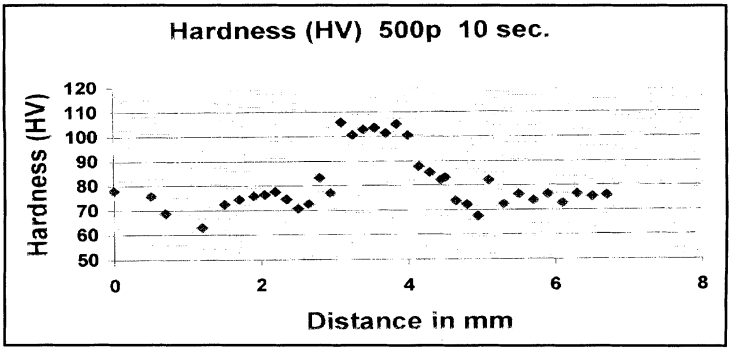

FIGURE 9 Microhardness along the line through one of the Ta inclusions in the Ta test sheet.

statistics is as follows:

Company A: 276 sheets

- 217 sheets free of defects;
- 38 sheets show a special defect structure ("dog bone") most likely due to rolling imprints (no foreign material inclusions detected by microhardness measurements, SYRFA, XAFS, NAA);
- 23 sheets show grinding marks.

Company B: 265 sheets

- 261 sheets are free of defects;
- 4 sheets show defect signals, evidently grinding marks (no foreign material detected).

Company C: 174 sheets

- 15 sheets show sharp defect signals, some are identified as iron inclusions (after chemical etching of 40 μm most of the iron signals disappeared);
- many grinding marks with defect signals still present in the ground centre.

4 NEUTRON ACTIVATION ANALYSIS (NAA) OF Nb SHEETS

The eddy current system proved itself to be a good method for detection of metallurgical irregularities on a huge surface of the test material. However, this method allows only the location of the defect and does not give information about the sort of foreign material inclusion and its concentration in Nb. The knowledge of the sort of material inclusions is very desirable especially for the Nb suppliers in order to chose prevention arrangements for future deliveries.

Different methods can be used for nondestructive identification of the defects in Nb sheets (see Table II). The application of the fluorescence analysis was described above. These methods are very sensitive but unfortunately the penetration depth of the signal in Nb is in many cases insufficient.

In opposition to the fluorescence analysis the Neutron Activation Analysis (NAA) allows to check nondestructively the Nb quality within the Nb sheet bulk. The experiments were done with the irradiation devices of Hahn Meitner Institute at the Research Reactor BER II (Berlin, Germany) and consist normally of three steps.

In the first step the Nb sheet is placed in a beam of thermal neutrons (flux density about $10^9\,\mathrm{cm}^{-2}\,\mathrm{s}^{-1}$). During this time (irradiation time t_{ir}) radioactive isotopes with characteristic half-life time are formed. The Nb sheet becomes slightly radioactive and emits gamma rays.

In the second step one measures the total radiation of the Nb sheet with a germanium detector. The distance between the sheet and the

detector is about 50 cm. Comparison of the count rates of selected photo peaks in the gamma spectrum of the sheet with those of a standard sample allows to obtain the content of definite element.

The third step allows to localize the cluster of foreign material inclusion, if it was detected during the second step. For this purpose the high sensitive image plate is put on the activated Nb sheet for 20–40 h (exposition time $t_{exp)}$. The clusters can be localised due to appearance of black spots on the image plates.

The efficiency of the detection of definite elements depends on the intensity of their gamma rays and the correlation between the half-life time of the Nb and cluster material. For example this correlation is very profitable for detection of Ta clusters in Nb. Tantalum radioactive isotope Ta182 has a half-life $\Delta t_{Ta} = 115$ days, which is higher than $\Delta t_{Nb} = 6.2$ min. Furthermore when setting up the experiment it is important to find out the right ratio between the irradiation time t_{ir}, and the break time t_{br} (time between the end of irradiation and start of γ-spectrum measurement). The appropriate choice reduces the background signal. The NAA demonstrates a very high sensitivity, limits of impurity detection may be as low as several ppm.

The NAA of the Ta test sheet ($t_{ir} = 5$ h, $t_{br} = 2$ weeks) has shown, that on one hand about 200–300 ppm of Ta is uniformly dissolved in the sheet. This result is in good agreement with the data available from chemical analysis. On the other hand the Ta clusters can be very clearly seen on the picture of the blackening of the image plate (Figure 10).

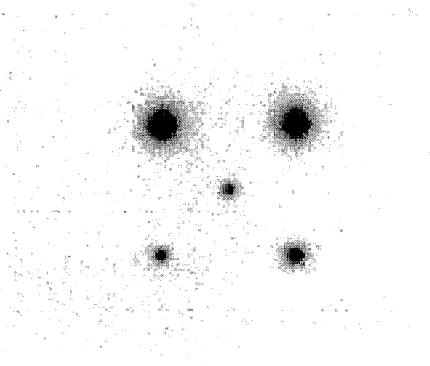

FIGURE 10 The Ta clusters detected in the Ta test sheet with NAA.

The NAA of 10 new Nb sheets for TTF from three suppliers (company A,B,C) was done. A Ta piece with a size about 0.5 mm^2 was imbedded in the angle of each sheet. All artificial Ta inclusions can be clearly seen on the image plates of NAA, but no Ta clusters are detected. These results mean that the pollution of Nb sheets with Ta clusters is probably seldom. But to be sure about this we need more statistics.

5 ONE EXAMPLE OF QUALITY CONTROL OF Nb SHEETS (IRON IN NIOBIUM)

Some rather small spots with high eddy current signal, that exceed the noise threshold X significantly, were detected in some sheets. For example a magnified eddy current picture of one of these sheet areas can be seen in Figure 11. The scope representation shows the eddy current signal in comparison with the threshold of the noises. The four lowest spots in the picture (location 1–4) were attentively investigated with SYRFA method. The tear is visible per eye in location 1. The magnified microscope picture with attached sizes is represented in Figure 12. The depth of the tear, obtained due to focusing variation

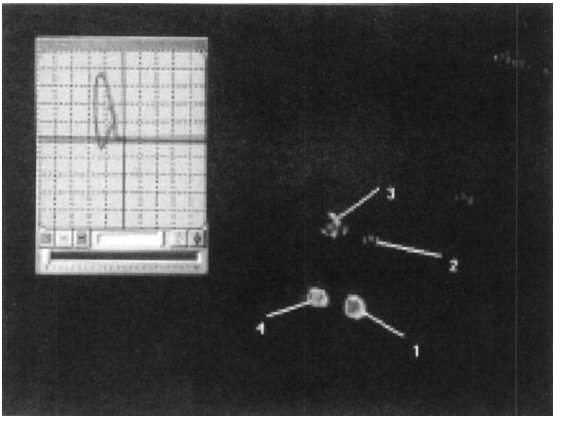

FIGURE 11 The magnified eddy current picture of the lower part of the Nb sheet T17.

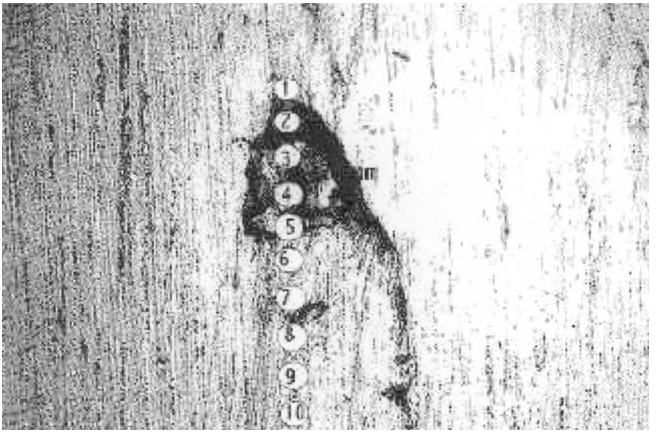

FIGURE 12 The microscope picture of location 1 of the sheet T17. The black area represents the tear.

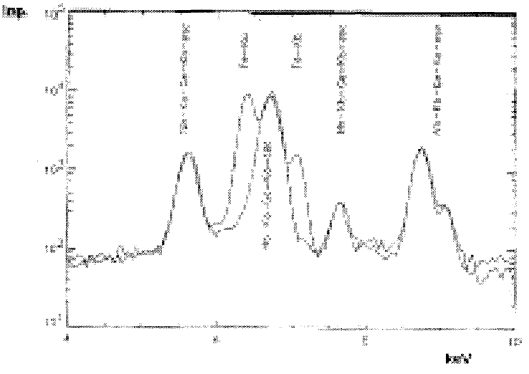

FIGURE 13 Fluorescence spectrum of sheet T17, location 1.

of microscope, is about 20 μm. The length is about 200 μm and the width about 100 μm. Figure 13 shows the combination of SYRFA signals from two different positions (far away from scratches and in their area). The additional reflexes can be clearly seen in the spectrum from the spot at 6.4 and 7 keV. These two lines correspond to the Kα and Kβ radiation of the iron.

The iron contamination was investigated in detail with SYRFA. The measurement was done in 377 points with intervals of 50 μm. The field with the size 600×1400 μm in the area of marks 1–10 of Figure 12 was scanned. On the picture of the three dimensional distribution of the iron signal (Figure 14) three maximums can be clearly distinguished. It is surprising, that the iron signal cannot be detected in the crater of the tear, the iron is disposed beneath the crater and is covered with Nb. The highest value of the iron signal is observed directly below the tear. The size of the contaminated area is roughly 400×1000 μm. The estimation of the iron layer thickness from the magnitude of the signal gives a value of about 10 μm. The depth of detected iron in Nb cannot be higher than some μm, because of the strong absorption of the iron fluorescence signal in Nb.

The locations 2 and 3 do not have a correlation (neither with eye nor with the microscope) with a defect in the sheet metal. These two positions were investigated with SYRFA in several places, no impurities could be identified.

A structure visible with the optical microscope was detected on location 4. In the microscope picture a black strip with a size of 10×100 μm has been seen. The SYRFA spectrum has shown, that an iron contamination is clearly present in this area. The size of contaminated area is about 200×600 μm. This can be seen in Figure 15 of iron signal distribution, that was acquired in 117 measurement points with intervals of 50 μm.

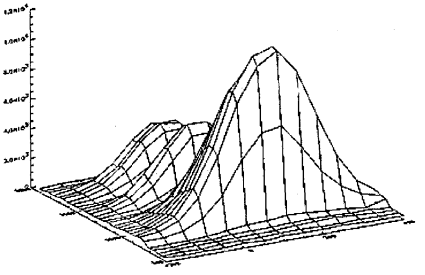

FIGURE 14 The distribution of the iron signal in location 1 of sheet T17.

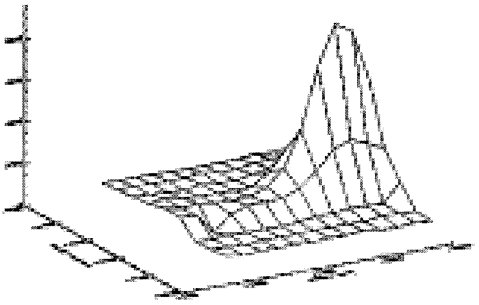

FIGURE 15 The distribution of the iron signal in location 4 of sheet T17.

It can be concluded from the sizes and shapes of the iron contamination in positions one and four, that evidently the Nb was polluted with iron particles during the rolling process.

6 CONCLUSIONS

The eddy current scanning system is proved as sensitive main instrument for quality control of Nb sheets. More than 700 sheets for TTF have been successfully tested. Some irregularities, that can be a characteristic of foreign material, are detected.

The microhardness measurement shows a good efficiency in order to separate the sheets with material inclusions from the sheets with geometrical variations in the surface.

Synchrotron Fluorescence Analysis (SYRFA, XAFS) allows to identify nondestructively and very precisely the kind of inclusion material in the areas close to the Nb sheet surface (with a penetration depth between few μm and few hundred μm).

Neutron Activation Analysis (NAA) is a reasonable method for nondestructive identification of material inclusion in the bulk of the Nb sheet. This method is especially effective in regard to searching of Ta clusters.

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