DEVELOPMENT OF AN X-RAY FLUORESCENCE PROBE FOR INNER CAVITY INSPECTION

M. Bertucci*, P. Michelato, L. Monaco, M. Moretti, INFN Milano - LASA, Segrate (MI), Italy
C. Pagani, Università degli Studi di Milano & INFN Milano - LASA Segrate, Italy
A. Navitski , DESY, Hamburg, Germany

Abstract

The development of an x-ray fluorescence probe for detection of foreign material inclusions of the inner surface of 1.3 GHz tesla-type Niobium cavities is here presented. The setup dimensions are minimized so to access the inner cavity volume and focus on the surface of equator. Preliminary tests confirmed the system capability to detect and localize with good precision small metal inclusions of few micrograms. The results obtained from the inspection of some 1.3 GHz XFEL series production cavities are also pointed out.

INTRODUCTION

The ability to detect performance-limiting defects on the inner surface of superconducting radiofrequency (SRF) niobium cavities, which lead to low quality factor Q_0 factor, thermal breakdowns (especially at the equator welding seams and the surrounding area), and X-ray radiation (mainly due to sharp geometric defects on the irises) provides a tool of quality control (QC) and failure reason clarification. Detection of failures and defects, especially in early production steps, would significantly reduce repetition of quite expensive cryogenic RF tests and retreatments of the cavities. Inspection of the inner cavity surface by an optical system is an inexpensive and useful means for surface control and identification of dangerous or suspicious features [1, 2]. It does not provide, however, information about material content in the defect region, which is required for sorting out the cavities with foreign inclusions and for the localisation of a contamination source in the production cycle. Preliminary diagnostic is usually performed during the QC of niobium sheets resorting to several non-destructive techniques, e.g. eddy current scanning [3].

X-ray fluorescence (XRF) analysis is widely used for elemental and chemical analyses, particularly in the investigation of metals. This technique, already employed during the QC of niobium sheets [4], appears to be entitled for development of a diagnostic tool for the detection of trace element inclusions on the cavity surface. Preliminary feasibility tests [5] performed with a low-performance XRF setup, demonstrated that low amounts (some μg) of different metals could be easily detected when embedded in the niobium matrix. These encouraging results have been the first step towards the development of an XRF tool for the QC of the inner surface of 1.3 GHz SRF cavities. The complicated shape of the cavities and hidden inner surface require, however, development of a special device.

* michele.bertucci@mi.infn.it

CHARACTERISTICS OF XRF SPECTROSCOPY TECHNIQUE

XRF spectroscopy is well known non-destructive elemental analysis technique based on the detection of characteristic X-ray radiation emitted from a material that has been excited by a high-energy primary X-ray source. It allows simultaneous acquisition of the whole sample spectrum in a very short time, detecting low concentration values up to a part per million. The fluorescence spectrum lines offer an unequivocal determination of sample elements. The XRF setup consists of an X-ray tube for primary excitation of the material to be investigated and a detection unit for energy dispersive spectroscopy. The excitation spectrum of the tube is given by bremsstrahlung radiation produced by electron-bombardment of the target along with characteristic fluorescence line of the target material. The X-ray tube radiation in its turn excites the characteristic radiation of sample elements (X-ray fluorescence spectrum). A schematic overview of an XRF setup is shown in Fig. 1.

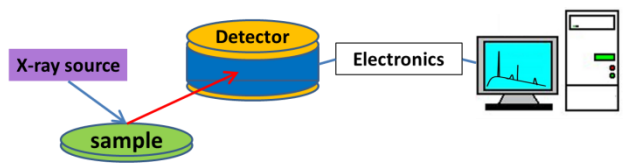

Figure 1: Schematic overview of an XRF setup.

Typical benchtop XRF instruments do not require a specific sample preparation, although both X-ray source and detector must be as close as possible to the sample surface to maximize detection efficiency and avoid spurious signals in the detected spectrum. Aiming to develop an inspection tool for the cavity inner surface, the X-ray source and detector must enter the inner cavity space.

Detection Depth

Since the niobium X-ray absorption cross section is high for energies below 100 keV, the excitation radiation will penetrate inside the bulk for a depth of about 68 μm when employing an X-ray tube with the molybdenum anode. The penetration depth corresponds to an inverse of attenuation linear coefficient of niobium μ_{Nb} of 146 cm^{-1} at the energy of Mo K_α characteristic line of 17.4 keV [6].

ISBN 978-3-95450-178-6

This means that the XRF analysis has to be considered as a "surface" analysis technique. Nevertheless, since RF penetration depth in niobium is just around 36 nm, the XRF technique should cover the whole depth of interest.

Sensitivity

The instrument should be sensitive enough to detect metal impurities with a size as small as a critical defect size causing thermal breakdowns at relevant accelerating gradients (for example at least 23.6 MV/m for the European X-ray Free-Electron Laser (EXFEL) [7] cavities). Using a simple model [8] of heat propagation, critical defect size can be evaluated for different metals and compared to a minimum detection limit (*mdl*) [9], namely a minimum detectable concentration for a given element (*i*). *mdl* is a concentration corresponding to three times the standard deviation of the background intensity I_B in the spectral zone of a chosen X-ray characteristic line. Under the assumption of a low concentration of a given element *(i)*, *mdl* can be calculated as:

$$mdl_i = 3\sqrt{I_B}/S_i, \quad (1)$$

where I_B is the background intensity and S_i is the element sensitivity, namely the ratio between the X-ray intensity at the characteristic line and the element concentration. Aiming to analyse an entirely metallic sample with a nearly monochromatic radiation, we expect to have a high sensitivity for elements with atomic number Z above 25 due to a high photoelectric cross section for excitation energy and low continuous background due to low scattering cross section for residual bremsstrahlung. Benchtop XRF setups easily attain a ppm level of the *mdl* for elements with characteristic K lines lying in the spectrum zone from 5 to 15 keV [10].

Detection Window

There are several issues limiting the detection-capability of an XRF system. Elements lighter than sodium (Z of 11) are difficult to quantify unless background and very comprehensive inter-element corrections are performed. For a standard semiconductor detector, the efficiency is highest in the energy range from 6 to 11 keV. Reduction for low energies is mainly due to the absorption by the beryllium window of the detector and by air whereas for higher energies it is due to a lower photoelectric absorption cross section in the intrinsic active region of the detector-crystal. Moreover, every element with the absorption edge higher than the excitation energy of the tube cannot be ionized and produce X-ray fluorescence. The emission of L lines can be exploited instead of K lines in the case of heavy elements (e.g. gold, lead, and tungsten) since L absorption edge is lower than K one. However, the corresponding characteristic lines must lay in the high efficiency-range of the detector. As a consequence of these limitations, a detection window, namely which elements can be detected independently of their concentration, can be defined for every XRF setup. Almost all elements of the most probable inclusions on the cavities like iron ($K_{\alpha 1}$ line at 6.4 keV), copper ($K_{\alpha 1}$ at 8.05 keV), titanium ($K_{\alpha 1}$ at 4.51 keV), tungsten ($L_{\alpha 1}$ at 8.4 keV), and tantalum ($L_{\alpha 1}$ at 8.15 keV) are in the detection window. On the other side, such elements like magnesium ($K_{\alpha 1}$ at 1.25 keV), aluminium ($K_{\alpha 1}$ at 1.49 keV), and silicon ($K_{\alpha 1}$ at 1.74 keV) are difficult to detect.

EXPERIMENTAL SETUP

The XRF setup for the detection of foreign material inclusions in the SRF cavities has been considered of the similar construction as OBACHT system [2] at DESY for high-resolution optical inspection of the inner surface of the cavities. It uses a linear drive for longitudinal displacement of the cavities and a rotational drive for the camera. The XRF probe, including X-ray source and detector, is customized to enter within around 70 mm diameter of the cavity-iris and aside of the high order mode (HOM) couplers of 1.3 GHz TESLA-shape cavities. Primary goal is inspection of the equator region of the cavities, which is most affected by foreign inclusions due to high magnetic field. The excitation and detection setup is customized to focus on the equator surface located at about 103 mm from the cavity axis with an intrinsic spot-size of about 10 mm. More precise localization of the inclusions is foreseen by longitudinal and angular surface scanning. A low-size X-ray source XRT-30® from PROTO Manufacturing Inc. with diameter of around 30 mm and 125 mm length has been chosen due to the space limitations. This is a fine focus X-ray source employing molybdenum target-anode, operating at up to 30 kV accelerating voltage, 10 mA beam current, and 300 W beam power, and is water cooled. A silicon drift detector (SDD) from XGLab S.R.L. [11] has been chosen. It offers a good performance even with a very small size and equipped with an external amplifier. The external multichannel amplifier (MCA) unit can be placed far away from the detector minimizing the volume entering into the cavity. X-ray tube and detecting unit are combined into a single measuring head (see Fig. 2) focusing on to surface at around 100 mm distance with around 30° tilted detector.

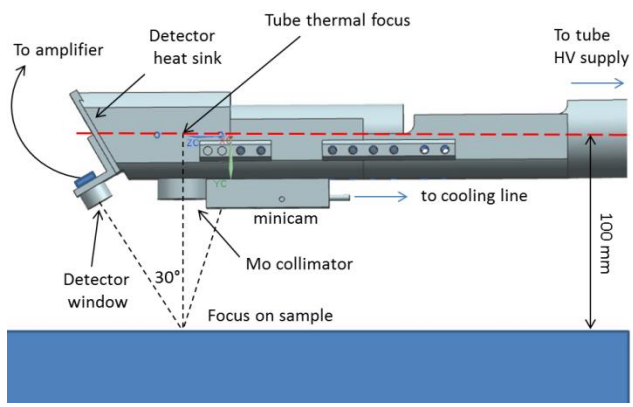

Figure 2: Schematic sketch of the XRF setup.

A collimator made out of molybdenum is mounted in front of the window of the X-ray tube. A mini-camera is placed beside the collimator and allows controlling the correct position of the detector system in the cavity. A picture of the experimental setup entering a 9-cell 1.3 GHz SRF niobium cavity is shown in Fig. 3.

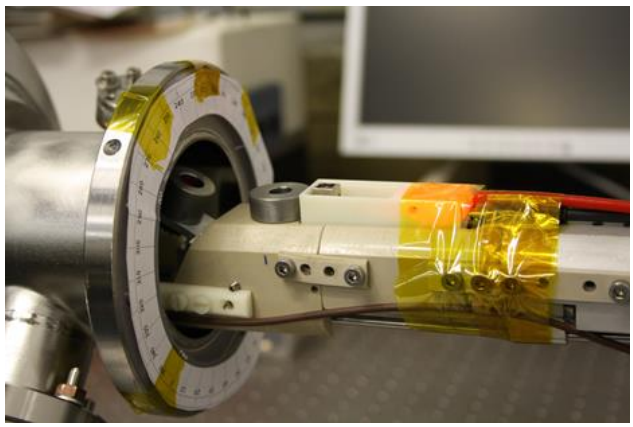

Figure 3: XRF probe entering the inner space of a 9-cell cavity.

Performance Test of the XRF Setup

Initial tests performed so far with the XRF setup confirmed the expected instrument capability [12] with the minimum detection limit as low as few µg for metallic impurities with characteristic X-ray lines laying in the spectrum zone from 5 to 10 keV. Table 1 summarize the results of the detection limit measurements compared with the critical mass for thermal breakdown at 20 MV/m for 1.3 GHz Nb cavities, calculated using the model reported in [8]. The XRF instrument is able to detect the most probable inclusions with masses below (or around, as for pure titanium) the critical values for thermal breakdown except for aluminium, whose sensitivity coefficient is significantly lower.

Table 1: Minimum detection limit and critical mass for different materials: electrolytic tough pitch copper (ETP Cu), oxygen-free copper (OF Cu), stainless steel (316L SS), aluminium alloy (6061-T6 Al), pure tantalum (Ta-pure), commercially pure titanium (Ti-pure), and tungsten carbide (WC).

Material	*mdl* (µg)	critical mass (µg)
ETP Cu (RRR=50)	0.13	240
OF Cu (RRR>400)	0.13	4040
316L SS	0.35	2.69
6061-T6 Al (RRR=2)	3901	0.185
Ta – pure	0.26	59.9
Ti – pure	0.36	0.169
WC	0.09	0.630

An example highlighting the high instrument sensitivity is presented in Fig. 4, showing the XRF spectra of a niobium sample with and without an artificial iron impurity of 10 µg dropped onto the surface. The K_α characteristic line of iron at 6.4 keV is clearly visible in the first case.

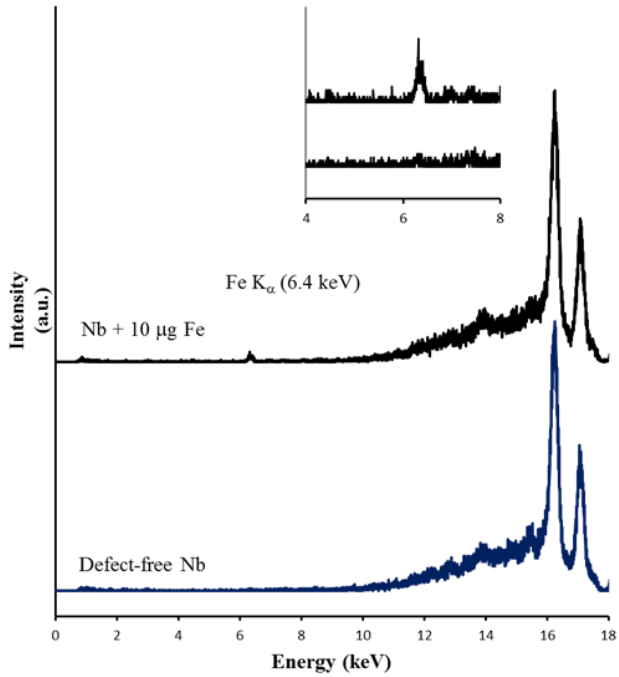

Figure 4: XRF spectrum of a niobium sample with (upper) and without (lower) 10 µg iron impurity. The magnified view shows the spectrum in the range from 4 to 8 keV (inset).

The ability of the system to detect the position of an impurity/inclusion has been tested placing a metallic particle on the equator of a single-cell niobium cavity and acquiring the XRF spectrum at different angular positions. The results indicate the accuracy of the defects localization of a few degrees, what is enough for the application of further surface mapping and repair techniques like optical inspection and local grinding or etching.

XRF INSPECTION OF CAV00811

The XRF setup has been tested on a real cavity CAV00811 from the series production for the EXFEL project. The cavity showed a poor performance during the first cryogenic RF test, having thermal breakdown at 12 MV/m. OBACHT visual inspection detected a defect (Fig. 4) on the welding seam of the equator 9 at around 180° (177.6° center point of the upper image in Fig. 5). This defect has been considered as a contamination with a foreign material before or during the electron beam welding process.

ISBN 978-3-95450-178-6

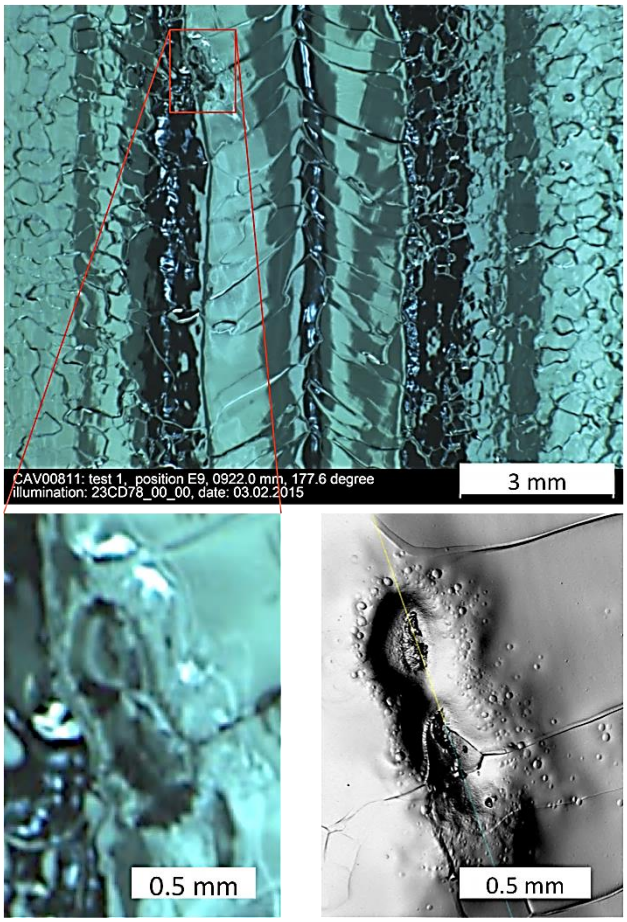

Figure 5: Defect observed by OBACHT optical inspection (marked by red square on top and magnified on lower left image) and additionally visualized by laser microscopy on surface-replica (lower right).

The XRF probe has been employed to investigate the material composition in the defect area. Current of 8 mA and accelerating voltage of 25 kV for the X-ray tube and acquisition time of 1000 sec have been chosen. Zirconium transmission filter has been employed for attenuation of the continuous background spectrum. The use of molybdenum collimators of 8 mm on the tube and 7 mm on the detector resulted in a 20 mm diameter irradiated spot on the cavity-equator. The X-ray intensity at characteristic lines for several elements as a function of the angular positioning of the cavity is shown in Fig. 6.

Fluctuations of the X-ray intensity at characteristic lines for titanium, copper, tantalum, and tungsten are comparable with the uncertainty due to the count statistics:

$$\sigma = \sqrt{I} \qquad (2)$$

In the case of iron, however, a significant increase of the intensity has been observed by passing the defect zone. Aiming at a more detailed investigation of the possible iron contamination, measurements at 1° rotation steps have been performed in the defect and surrounding area. Figure 7 shows the variation of the X-ray intensity at

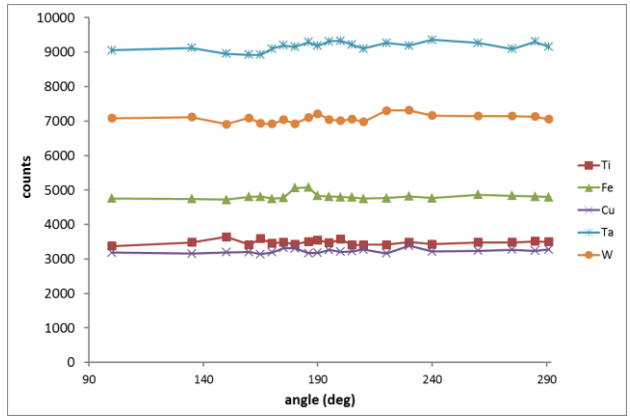

Figure 6: X-ray intensity at characteristic lines for several elements (titanium, iron, copper, tantalum, and tungsten) as a function of the angular positioning (rotation) of the cavity.

$K_{\alpha 1}$ characteristic line of iron (at 6.4 keV) as the function of the angular position of the cavity. Red line marks the average value of the background intensity, whereas the dashed line is the minimum detection limit for iron. The intensity well above the uncertainty limit confirms the presence of iron in the defect area.

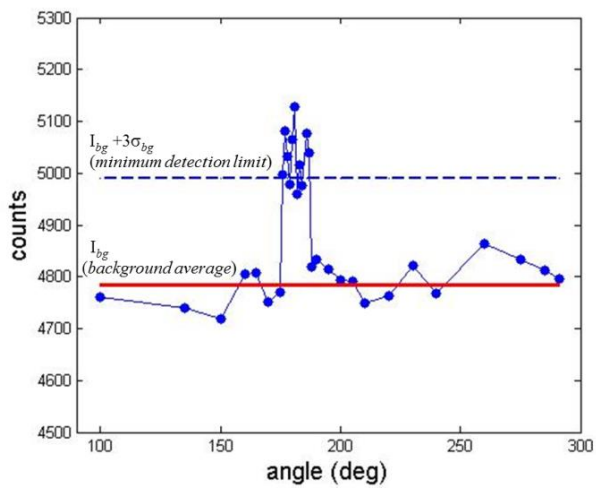

Figure 7: X-ray line intensity of iron (Kα1= 6.4 keV) as a function of angle. Background and mdl levels are indicated by a red and a dashed line respectively.

Since the significant increase of the intensity is observed for the angular positioning between 176° to 187°, the defect position should be at around 181.5°. These results are in a good agreement with the results obtained by OBACHT and replica surface inspection

CONCLUSIONS

New diagnostic probe based on the XRF spectroscopy for inspection of SRF cavities has been designed, developed, and tested. It allows detection of foreign material inclusions on the inner surface of the cavities as it has been confirmed by finding iron contamination on a cavity, limited by low-field thermal breakdown, from the series EXFEL production. The minimum detection limit down to a few μg for metallic impurities with characteristic X-ray lines lying in the high efficiency spectrum zone from 5 to 10 keV has been shown.

The XRF instrument is able to detect the most probable impurities with masses below the critical values relevant for thermal breakdown except for aluminium, whose sensitivity coefficient is significantly below the detection limit.

An accurate localization of defects on the surface has been demonstrated by the angular or lateral displacement of the cavity.

This analytic tool can improve the quality control of the cavities allowing identification of chemical composition of contaminations or defects discovered by means of optical inspection systems and helping to localize the failures in the production sequence.

REFERENCES

[1] T. Harden, M. Borden, A. Canabal, P. Pittman, T. Tajima, "Pre-conceptual design of automated systems for SRF cavity assembly and optical inspection", Proceedings of PAC07, Albuquerque, New Mexico, WEPMS032 (2007).

[2] F. Schlander, et al., "Quality Assessment for Industrially Produced High-Gradient Superconducting Cavities", Proceedings of the 2nd International Particle Accelerator Conference, San Sebastian, Spain, September 4-9 2011, http://jacow.org/.

[3] W. Singer, D. Proch, A. Brinkmann, *Proceedings of the 1997 Workshop on RF Superconductivity,* Abano Terme (Padova), Italy, October 6-10, 1997, edited by V. Palmieri and A. Lombardi (Istituto Nazionale di Fisica Nucleare, Società Italiana di Fisica, Università degli Studi di Pavia, 1998), p. 850.

[4] W. Singer, X. Singer, A. Brinkmann, J. Iversen, A. Matheisen, A. Navitski, Y. Tamashevich, "Superconducting cavity material for the European XFEL", Supercond. Sci. Technol., **28**, 8 (2015).

[5] P. Michelato, M. Bertucci, A. Navitski, W. Singer, X. Singer, C. Pagani and Y. Tamashevich., *Proceedings of the 16th International Conference on RF Superconductivity*, Paris, France, September 23-27 2013, edited by Claire Antoine (IRFU), Sebastien Bousson and Guillaume Martinet (IPN), pp. 736 – 739.

[6] National institute of Standards and technology, Physical Meas. Laboratory; http://physics.nist.gov

[7] XFEL Technical Design Report, DESY 2006-097, Hamburg, July 2007, www.xfel.eu/.

[8] H. Padamsee, Jens Knobloch, Tom Hays, *RF Superconductivity for accelerators*, New York, Wiley-VCH, 2008, pp. 205, 206.

[9] R. Jenkins and J. V. Gilfrich, X-Ray Spectrom, **21**, 263 (1992).

[10] R. O. Miller, *Spectrochemical Analysis by X-ray fluorescence*, New York, Plenum Press, (1972)

[11] L. Bombelli , C. Fiorini , T. Frizzi , R. Alberti , R. Quaglia, "High rate X-ray spectroscopy with 'CUBE' preamplifier coupled with silicon drift detector", proceedings of NSS/MIC 2012, Anaheim, CA, USA IEEE (2012).

[12] M. Bertucci, P. Michelato, M. Moretti, A. Navitski, C. Pagani, "Defect Detection inside Superconducting 1.3 GHz Cavities by means of X-Ray Fluorescence Spectroscopy", to be published on Rev.Sci.Instrum.

ISBN 978-3-95450-178-6