AN X-RAY FLUORESCENCE PROBE FOR DEFECT DETECTION IN SUPERCONDUCTING 1.3 GHz CAVITIES

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Abstract

The aim of this project is to develop a system for defect detection by means of X-ray fluorescence (XRF) analysis. XRF is a high sensitivity spectroscopy technique allowing the detection of trace element content, such as the few microgram impurities, responsible for low cavity performances if embedded in the equatorial region during cavity manufacturing. The proposed setup is customized on 1.3 GHz TESLA-type niobium cavities: both the detector and the X-ray excitation source are miniaturized so to allow the probe to enter within the 70 mm iris diameter and aside of the HOM couplers. The detection-excitation geometry is focused on cavity cell equator surface located at about 103 mm from the cavity axis, with an intrinsic spot-size of about 10 mm. The measuring head will be settled on a high angular resolution optical inspection system at DESY, exploiting the experience of OBACHT. Defect position is obtained by means of angular inner surface scanning. Quantitative determination of defect content can be carried out by means of the so called fundamental parameters technique with a Niobium standard calibration.

INTRODUCTION

Inner cavity inspection for defect localization is nowadays performed resorting to optical techniques, but a more effective method for elemental characterization of trace element inclusions limiting the cavity performances is needed so to correctly locate the defect position and, when possible, remove them with a guided repair procedure (e.g. mechanical grinding and local etching). Main limitations to defect detection and suppression are due to small defect size and inclusions embedded in the cavity material.

As an estimation of minimum defect radius causing thermal breakdown in a Niobium cavity operating in the XFEL working conditions, assuming f=1.3 GHz, and a 20 MV/m value for accelerating gradient, a value of 40 μm is obtained for copper (R_s=10 mΩ) [1], corresponding to 2.3 μg critical defect mass. Similar results are obtained for other elements like Fe, W, etc., that can contaminate the cavity usually involved during manufacturing.

Aiming to locate such a tiny amount of material, we need an high sensitive elemental technique. In the same time we must exploit a small size instrumentation that can be settled and moved within the cavity bore. XRF spectroscopy technique fulfills the need for high sensitivity, although the size of already existing XRF setups for industrial and academic applications is still too big for cavity inner analysis.

Given that niobium X ray absorption cross section is high for low energies (<100 keV), the excitation radiation will penetrate inside the bulk for a depth of about 10 μm [2]. So the XRF analysis has to be considered as a surface analysis. Nevertheless we consider that X ray will cover the characteristic RF penetration depth in niobium [3].

CHARACTERISTICS OF XRF

XRF technique allows a simultaneous acquisition of the whole sample spectrum in a very short time, detecting low concentration values up till a part per million. The fluorescence spectrum lines offer a unequivocal determination of sample elements.

Furthermore, it is a non-destructive technique, namely it leaves the sample completely unaltered after the measurement. It must be however pointed out that light elements (Z<12) are detected with difficulty. In the cavity case, a successful detection of carbon impurities can be obtained resorting to destructive techniques like SEM-EDS or Auger spectroscopy [4].

We performed a preliminary check in order to assess the possibility of detecting 100 μm size metal defects embedded on a pure niobium surface. Furthermore, we checked the possibility of a lateral XRF scan for an accurate defect localization when dealing with a irradiated area greater than defect dimension.

Feasibility of Defect Detection

A first successful preliminary test has been performed using a low-performance XRF portable setup, operating with a low power X ray tube (i=200 μA, V=25 kV, P= 5 W) with a 2.5 mm diameter spot and a Molybdenum target anode (E=17.6 keV), and a EDS Si detector with Peltier cell cooling. Defects are simulated with a copper salt deposition on a flat niobium surface.

Few μg of copper are thus placed on a 1 mm diameter zone. The sample XRF spectrum for a 60 s acquisition time.is shown in Fig.1

Cu K_α fluorescence line (E = 8.047 keV) corresponding to a 1 μg impurity is clearly visible. The choice of molybdenum anode has the advantage of avoiding fluorescence from niobium matrix, given that Nb absorption edge (18.9 keV) is greater than Mo K_α energy, and so increasing sensitivity in 1-15 keV spectral range.

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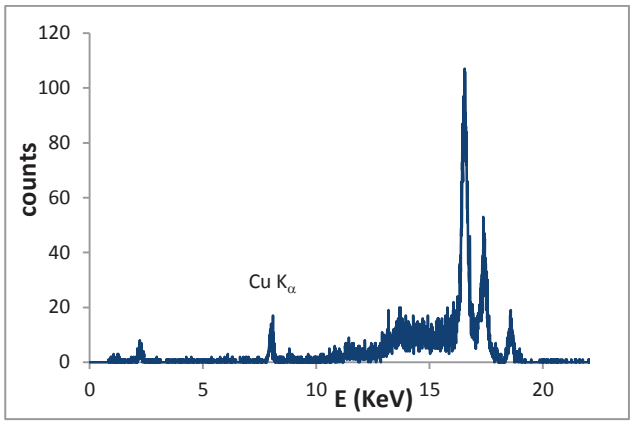

Figure 1: XRF Spectrum of Nb sample with Cu µg impurity.

Test on Lateral Defect Scanning

For an effective detection of defects in a reasonably short acquisition time, a high photon flux from X ray tube is needed. This implies a high tube power and a coarse excitation beam collimation; this in turn leads to the impossibility of a fine focusing of exciting radiation. Aiming therefore to assess the possibility of a lateral XRF scan for defect localization even dealing with a large spot size, we placed a sample on a sliding support allowing a simultaneous displacement of excitation-defect focus with a 25-50 µm step (see Fig.2).

We simulated the behavior of the lateral scanning system using 70 µm diameter copper wire and 10 µg iron and tungsten single grains dropped on the niobium plate. We expect a sudden increase of peak intensity from 0 to maximum value corresponding to the passage of grain from "dark" to "bright" zone thorough a small half-light zone due to tube collimator.

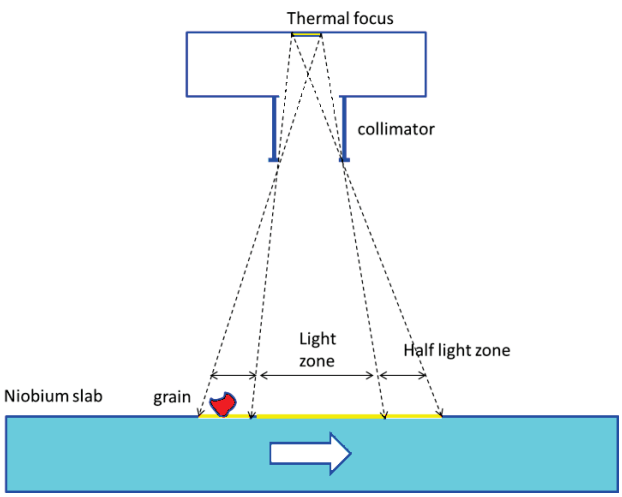

Figure 2: set-up arrangement for lateral sample scanning

Measurements have been done with the already mentioned standard XRF setup, with a 50 mm^2 niobium sample irradiated area. Experimental results are shown in Fig.3, where total number of counts is normalized with respect to maximum value.

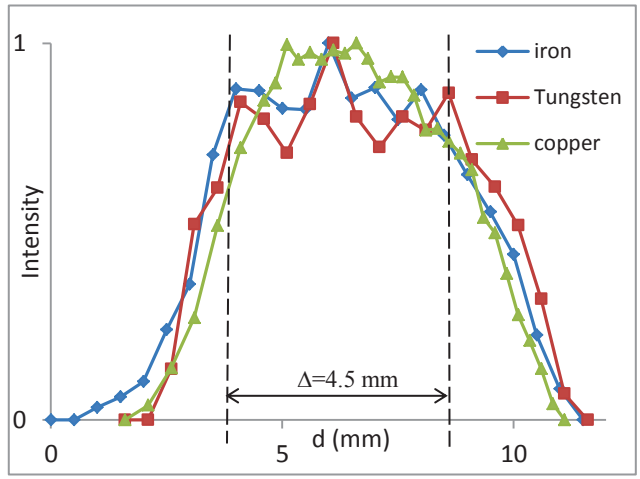

Figure 3: Peak intensity as a function of sample displacement for Fe, W and Cu defects.

We can see clearly a similar shape for tungsten and iron, where a sudden rise in peak intensity occurs due to spot brightness variation when crossing the dark – half-light-light zones, while for copper the diagram is additionally modulated by variation of sample irradiated area. The central plateau (from 4 to 8.5 mm) is consistent with nominal value for beam size (4.5 mm for a 59 mm^2 irradiated area taken from instrument datasheet). The mean value between the edges of central plateau can be taken as a first assessment of defect position.

Quantitative Measurement of Defects

Quantitative XRF analysis is based upon the fundamental parameters equations [5, 6], which express the peak intensity of a given fluorescing element (i) as a function of (i)-element atomic properties and sample element concentrations, namely $I_i = f_i(c_1, c_2, \dots, c_N)$. Once XRF line intensity values have been experimentally obtained, the unknown values of element concentration can be calculated by means of functions inversion. When dealing with the case of single small defect embedded on the niobium bulk, first order expansion of f allows to obtain a linear relation between fluorescence intensity and element concentration. Calibration constant, depending on set up geometry and tube and detector characteristics, can be calculated by measurement of a pure sample of element (i) in the same experimental conditions. As a consequence, defect mass and size can be inferred from element concentration. It must be pointed out that concentration accuracy will be affected by error on total peak counts ($\sqrt{N}$), so that the acquisition time should be extended up till a reasonable value for N is achieved.

DESCRIPTION OF THE EXPERIMENTAL SETUP

The X ray Fluorescence set up will settled at DESY and will be developed exploiting the experience of OBACHT system for high angular resolution optical inspection [7], which uses linear and rotational drives to displace camera inside the cavity and allows a fine optical mapping of

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inner cavity surface. The same framework will be exploited for XRF inner scanning.

A draft of the experimental setup is shown in Fig.4. Basically, the XRF probe is introduced inside the cavity. The tube – detector system must be focused on the equatorial region of the given cell. By means of a cavity rotation, the probe will scan the cell equator with a step equal to the beam size, so to analyze the whole equator circumference in a certain number of steps. This is a preliminary raw assessment of defect position. Then, if a defect is detected, a more accurate localization can be obtained by moving the irradiated zone with a smaller step within the suspect defect site. Even if beam spot size is large (25 mm), the exciting radiation intensity diminishes when moving away from the spot center, so we expect to find the defect in the zone with local maximum fluorescence intensity (corresponding to the beam spot center).

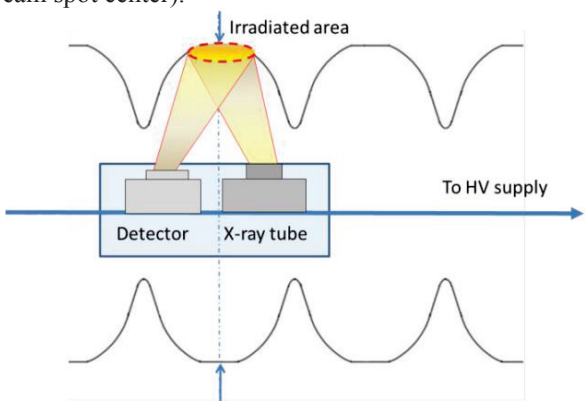

Figure 4: Draft of XRF probe.

X ray Tube

Aiming to analyze the inner zone of a 1.3 GHz Niobium cavity, whose entrance diameter is about 70 mm, we must resort to a low size X-Ray tube, that has to be introduced inside the cavity, for the whole cavity length (1.2 m, or at least half of it) together with the detection system. The tube power must be high to give certain minimum counts number in a reasonably small acquisition time.

A good choice is the ProtoXRD 30 mm Fine Focus Mo X-ray Tube (300 W, 30 kV, 10 mA, water cooled). The tube has a thermal spot size of approximately 0.5 mm × 0.5 mm. Target to window distance is 15 mm. X-ray exit window: beryllium 130 μm thick, 6 mm opening, 22°6' divergence angle. A drawing of tube is shown in Fig. 5.

Detector

We will exploit the Silicon Drift Detector developed by XGLAB srl., which offers good performances even with a very small size. The detector has a 25 mm^2 active area, 450 μm thickness, with 130 eV nominal energy resolution at Mn-K_α with an internal on-chip multilayer collimator. The detector is readout by an integrated preamplifier (CUBE) instead of standard JFET so to obtain optimal performances in terms of data throughput and energy resolution stability and allowing a low level concentration detection in very fast scanning mode. The detector is equipped with a data acquisition system providing low noise and high acquisition speed.

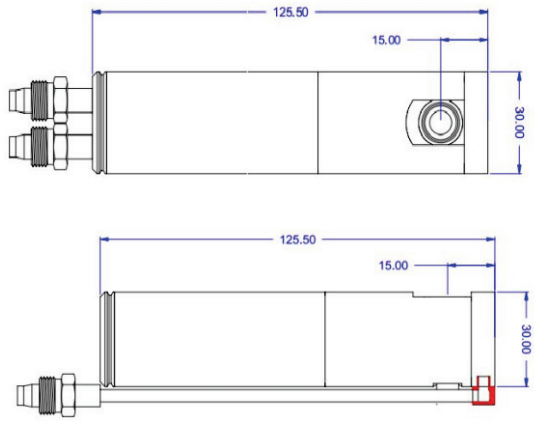

Figure 5: Linear dimensions of X-ray tube, expressed in mm. Courtesy of Proto XRD.

CONCLUSIONS

Preliminary tests confirmed the feasibility of low-size defect detection on cavity surface by means of XRF technique. The development of experimental setup is now in progress and further tests have to be done to work out the best experimental arrangement. Our final goal is the development of a new analytic tool that will extend the outreach for cavity defect diagnostics.

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