Characterization of the Internal Surface of Superconducting 352 MHz Accelerating Cavities using a dedicated Scanning Auger Spectrometer

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Abstract
The energy upgrade project of the Large Electron Positron (LEP) collider at CERN from 55 to about 90 GeV requires the use of about 250 superconducting four–cell 352 MHz cavities. The majority (over 200) of these cavities, which are being manufactured by three European industrial firms according to a procedure developed at CERN, are made of copper, coated internally with a 1.5 µm thick niobium film deposited by magnetron sputtering. Contamination and defects, which may be produced during the manufacturing process, have detrimental effects on the cavities' RF performance. For this reason, a dedicated computer controlled surface analysis instrument, incorporating Auger Electron Spectroscopy, Scanning Auger Mapping and secondary electron imaging, has been designed and built at CERN, to perform elemental and microscopical characterization of the LEP-type cavities surface.
In this paper a study of the inner surface of a few of these cavities is presented, including the characterization of the defects found. In addition, the variation of the surface composition of two copper cavities, prior to niobium deposition, after bakeout cycles at temperatures in the range from 120°C to 200°C is reported.

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1. Introduction

The energy upgrade project of the Large Electron Positron (LEP) collider at CERN from 55 to about 90 GeV requires the use of about 250 superconducting (SC) radio frequency (RF) 352 MHz four-cell cavities [1], which are being progressively installed in the existing LEP machine. The majority (over 200) of these cavities, which are being manufactured by three European industrial firms according to a procedure developed at CERN [2], are made of copper, coated internally with a 1.5 µm thick niobium film deposited by magnetron sputtering. Coated cavities provide several advantages with respect to bulk niobium cavities, especially in terms of thermal stability and cost. Contamination and defects which may be produced during the manufacturing process have been shown to have detrimental effects on the RF performance of the cavities [3,4]. For this reason, several diagnostic techniques have been developed and implemented to characterize the inner surface of SC cavities [5], including scanning thermography [6] and scanning electron microscopy [7]. A dedicated surface analysis instrument, incorporating Auger Electron Spectroscopy (AES), Scanning Auger Mapping (SAM) and Secondary Electron (SED) imaging, has been recently designed and built at CERN [8, 9], to perform elemental and microscopical characterization of the LEP-type cavities surface, possibly providing a direct link between the nature of the local surface defects and/or contamination and their effects on the RF operating conditions.

2. Results and discussion

The cavities, produced in industry, are accepted or rejected by CERN following RF tests. About 160 cavities have been accepted to date (September 1995). The rejected cavities are systematically inspected by means of a video camera set-up on a computer controlled optical inspection bench [10]. The results of the optical inspection and of temperature mapping measurements provide, in most cases, an explanation for the inadequate performance of the cavity. In some cases, however, further investigations using the surface analysis dedicated instrument are necessary to provide information on the nature of the defects in the Nb film.

Six Nb coated cavities have been analysed to date. In general, the non defective areas of the Nb film are oxydised (Nb$_2$O$_5$) [11] and present very low carbon contamination. The majority of the surface defects consist of regions where the Nb film is missing; the substrate Cu surface is then visible, usually oxydised and contaminated with C (examples in Fig. 1a, 1b, 1c). This kind of defect was found 12 times, with linear dimensions of the exposed Cu area ranging from ~50 µm to ~1 mm. In one particular case, an area of approximately 15 mm x 4 mm of the film was missing. In three cases, besides Cu, Ni and Fe were also detected in the defective area (example in Fig. 1d, 1e, 1f). In general, it has been possible to correlate the number and the total surface area of these non superconducting defects with the unsatisfactory RF performance of the corresponding cavity.
Fig. 1: Examples of typical film defects. The convention used for the Auger maps is that higher concentrations of the detected element correspond to lighter points; a) SED image of a ‘blister’ type defect; b) Nb SAM corresponding to a); the film is broken and the Cu substrate is visible (dark area); c) Cu SAM of a large defect where the Nb film is missing; d) SED image of a defect; the corresponding SAM of Nb (e) reveals inhomogeneities of the Nb concentration; the Auger scans taken at points A and B, shown in f, reveal the presence of Cu (A) and of Ni and Fe (B); g) ‘etching’ pits, SED image; h) SED image of a ‘stain’ of the Nb film; the corresponding Cu SAM is shown in i. The Nb film is not missing (as seen from AES measurements) but some Cu is detected.
Other types of defects were found, such as:

- “Etching pits” (Fig. 1g), attributed to gas bubbles retained on the Cu surface during the chemical polishing [4]; in one of the inspected cavities hundreds of these pits were found in one of the cells; however, they seemed to be uniformly coated with the Nb film.
- “Stains” on the Nb film (Fig. 1h, 1i), where besides Nb a small Cu signal is detected via AES, indicating either partial film peeling off or diffusion of Cu from the substrate; this kind of defect was found 3 times.
- Carbon granules, where a strong, local C contamination was found (4 times).
- Dust particles, detected as charging particles by the instrument (~10 times).

This classification is not exhaustive but is representative of the kind of defects of the Nb coating encountered to date in the characterization of industrial cavities. It can be concluded that the main problems are related to the film adhesion; the careful preparation of the Cu substrate seems to be of crucial importance, since residuals of chemical products or foreign metal particles can endanger the adhesion of the film and result in unsatisfactory RF performance of the cavity.

For this reason, a study of the residual surface contamination of an oxygen free copper cavity prior to niobium deposition, before and after bakeout cycles at different temperatures (120_C < T < 200_C) was carried out. Outgassing measurements were performed during each bakeout cycle using a calibrated Residual Gas Analyser. The number of outgassed molecules per square centimeter was calculated for the most important gas species desorbed. The same experiment was performed on a Cu cavity from which the Nb coating had been chemically removed (‘stripped’), to compare the results with the never coated (‘new’) cavity and to get an indication of the role of the film in “gettering” impurities from the Cu substrate.

Average numbers of outgassed molecules during each bakeout cycle are shown in Fig. 2a and b for H_2, H_2O and CO, CO_2, respectively. These gas species account for the largest part of the total number of molecules desorbed.

![Figure 2: Number of outgassed molecules during each bakeout cycle; first bakeout at 120_C, followed by three bakeouts at 200_C. a) H_2 and H_2O; b) CO and CO_2.](image)

The largest outgassing, especially in the new cavity, takes place during the first bakeout at 200_C. The decrease of the number of oxygen containing molecules released, between the first and second bakeout at 200_C, is more important in the new cavity. The hydrogen outgassing is higher in the new cavity and shows little change between the bakeouts at 200_C for both cavities. The outgassing of CO and CO_2 during the first bakeout at 200_C is much higher in the case of the new cavity; during the following two bakeouts it
is lower for the new cavity than for the stripped one. This agrees with the observation that the O concentration (measured by AES) after the first bakeout at 200_C is lower in the new cavity; the probability to form CO and CO_2 molecules is therefore smaller.

The main results of the AES measurements can be summarized as follows:

i) Before the bakeouts, the carbon content was higher and the oxygen content lower for the stripped cavity; the overall Cu concentration was similar for the two cavities.

ii) The new cavity presented an important chlorine contamination, which decreased after each bakeout cycle at 200_C, whereas in the case of the stripped cavity very little Cl was found. Such a high Cl contamination was not observed during AES measurements performed on Cu samples using a 'traditional' spectrometer. The origin of this contamination, and its possible correlation with Cl outgassing measurements is currently under investigation.

iii) The Cl and C contamination level increased after the first bakeout at 120_C and decreased after subsequent cycles at 200_C in both cases. The oxygen level decreased after each bakeout cycle.

iv) The impurity content on the surface of the new Cu cavity was not uniform. In particular, the Cl concentrations presented large variations; in some points very high C concentrations were detected. This could possibly endanger the local adhesion of the Nb film. The surface of the stripped cavity was generally more uniform.

v) Some sulphur segregation was observed in both cases, increasing slightly after each bakeout cycle at 200_C.

3. Conclusions

Further experiments are planned using the dedicated surface analysis instrument according to the needs of the cavity manufacturing and maintenance programme. The consequently growing statistics of typical surface defects will hopefully help establishing correlations between these and the cavities RF performance.

4. References