SURFACE STUDY USING NIOBIUM SAMPLE COUPONS FOR SUPER CONDUCTING RF CAVITY

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Abstract

In order to achieve higher and more stable performance of super conducting radio-frequency (SRF) cavities, extensive effort in development and application has been done for surface treatment and conditioning methods. Those methods have been evaluated with vertical tests showing lots of remarkable results in cavity performance. However it cannot be well understood yet how surface treatment or conditioning contributed to the results and which step of process in the treatment or conditioning affected the results. In this article, we describe our try to understand those questions focusing on the surface analyses with scanning electron microscope (SEM), X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS) for electro-polishing (EP) processed niobium sample coupons.

INTRODUCTION

To develop high performance superconducting niobium radio-frequency (SRF) cavities for ILC, various studies are progressing in STF (the superconducting RF test facility) in KEK[1]. It is necessary that more than 15000 high performance 9-cell cavities of which the gradient are larger than 35 MV/m in vertical test, are produced stably for ILC. In order to achieve higher gradient and more stable performance of SRF cavities, especially, huge effort has been continuing for surface treatment and conditioning methods for niobium surfaces of cavities[2]. For example buffered chemical polishing (BCP), electro-polishing (EP), ultrasonic ultra pure water rinse and high pressure water rinse (HPR), have been developed and improved as the standard recipe at KEK. Some treatments and/or conditionings bring lots of remarkable results in vertical tests for cavity performance[3]. However it cannot be well understood yet how surface treatment or conditioning contributed to the results and which step of process in the treatment or conditioning affected the results. In fact, the gradients of treated cavities are scattered widely due to field emitters, surface roughness and/or Q-disease. Therefore we have started up a team for the fundamental surface study of niobium cavity to find the causes of limited performance of cavities with using a couple of surface analytical methods[4]. We have developed a surface analysis system for the niobium cavities with using sample coupons which can be attached to a cavity and a sample transferring system using a small UHV suitcase. In this article, we describe the development of the system and our first try for the surface analyses using the new system.

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DEVELOPMENT OF SURFACE ANALYSIS SYSTEM FOR NIOBIUM CAVITY

Sample coupons and UHV Suitcase

Niobium sample coupons were made from niobium sheet from Tokyo Denkai Co., Ltd. And the specifications of the niobium sheets are the same as that are used for 9-cell cavity fabrication at KEK. The dimention of the coupon is 8 mm in diameter and 2.0 mm in thickness as shown in Fig.1(a). A single cell cavity was drilled six holes at the characteristic positions that are “equator”, “iris” and “beam pipe” as shown in Fig.1(b) and six sample coupons were set on the holes as shown in Fig.1(c). The recipe of surface treatments for the cavity with six sample coupons was as follows: 1) the BCP with the removal depth of 10μm for each coupon, 2) the EP process with the removal depth of 20μm for the cavity with coupons (Fig.1(c)) and 3) rinsing with ultra-pure water. The cavity with coupons was moved into the clean room shortly after the rinsing and the coupons were dismounted from the cavity. After natural dry, the coupons were set in a carousel type multi sample holder and put into a small UHV suitcase (Fig.2) in the clean room. The suitcase of which base pressure is in the order of 10^{-6}~10^{-7}Pa consists of a rack-and-pinion system with a rotary drive to transfer the carousel with coupons, a gate valve, an ion pump and a battery driven power supply. By using the suitcase, the coupons are able to be carried from the clean room to the surface analysis system without contamination and dust particles.

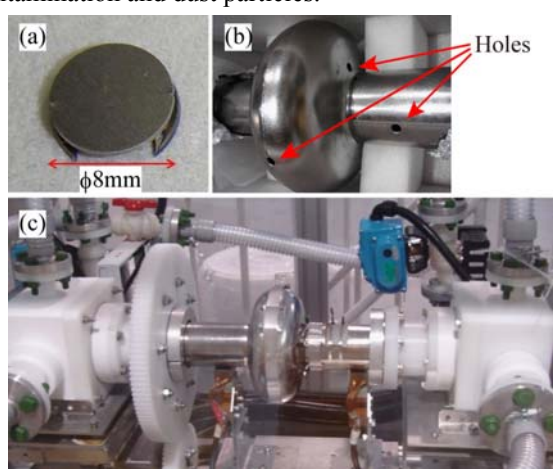

Figure 1: Niobium single cell cavity. (a) a sample coupon, (b) three pairs of holes drilled at “equator”, “iris” and “beam pipe”, (c) the cavity with samples on the EP bed.

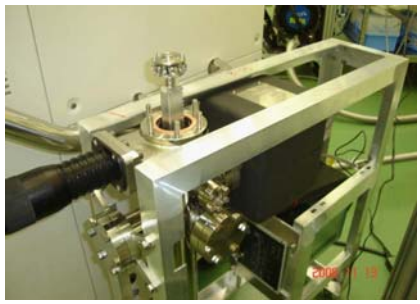

Figure 2: Picture of the UHV suitcase. Up to 12 sample coupons in the carousel can be installed.

XHV surface analysis system with loadlock chambers

We developed an extremely high vacuum (XHV) surface analysis system with three loadlock chambers to transfer the sample coupons from the suitcase to the analysis chamber and a sample storage chamber as shown in Fig.3. Each chamber is connected to the next chambers with gate valves and equipped with an individual pumping system. The 1st and 2nd loadlocks and the storage chamber have a transfer rod for each for the carousel or sample. The 3rd loadlock chamber works as a vacuum lock. The procedures to transfer the sample coupon to the analysis chamber are as follows: 1) connect the suitcase to 3rd loadlock chamber, 2) pump down the 3rd loadlock chamber, 3) transfer the carousel to the sample storage chamber (base pressure: $< 5\times10^{-8}$Pa), 4) pick up a sample from the carousel and 5) transfer the sample to the analysis chamber through the 2nd and 1st loadlock chambers.

The analysis chamber which is equipped with an electron energy analyzer, an ion mass spectrometer, an x-ray source, an electron gun, an ion gun, an extractor gauge and a residual gas analyzer, is capable of performing X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES) with Ar ion etching and secondary ion mass spectrometry (SIMS).

ANALYSES OF NIOBIUM SAMPLE COUPONS WITH VARIOUS TOOLS

XPS analyses

XPS analyses of which probing areas were less than 2 mm in diameter were carried out for sample coupons. According to XPS results, the surfaces were covered with the niobium oxide, mainly Nb_2O_5, and the atomic compositions were Nb: 10~20at%, O: 40~50at%, C: 30~50at%. Fig.4 shows a typical depth profile of the sample with Ar ion etching. The thicknesses of oxide layers that were defined as a FWHM of the amount of oxygen were 2~3 nm. There was not much difference among the three sample positions on the cavity (equator, iris and beam pipe) about the thickness of oxide layer. Sulfur which is thought as one of sources of field emission was not detected with XPS with a detection limit of 0.5at% for sulfur.

SEM observation and SIMS analyses

The sample coupons were observed with scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDXS), although there are no loadlock chambers for the suitcase. Many dots with the size of sub micro meter were found on the surfaces. In EDXS

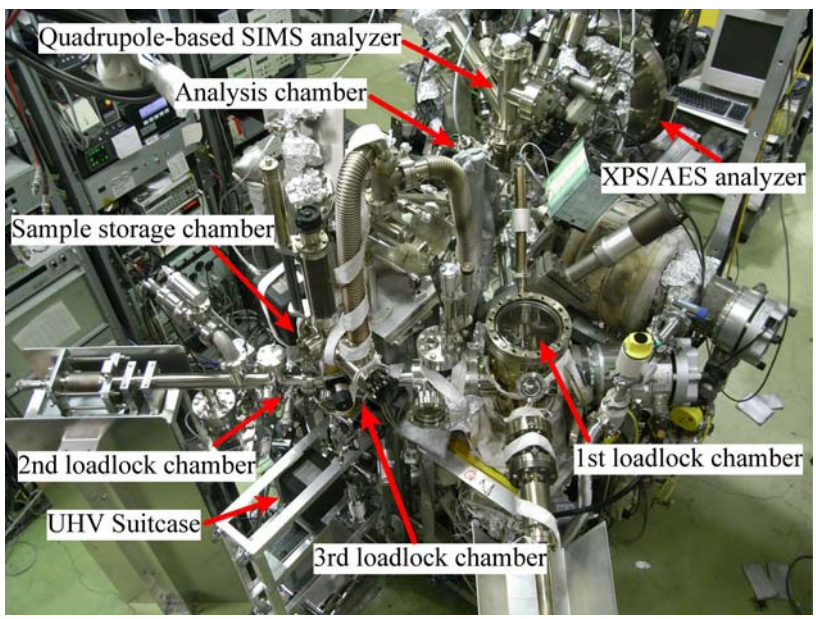

Figure 3: Overview of the XHV surface analysis system. The system equipped with XPS/AES/SIMS analyzers and the loadlock chambers to transfer the sample coupons without air exposure. The base pressure in the analysis chamber is in the order of 10^{-9}Pa.

analyses, chromium, iron and silicone with a size of several micro meters were found. However the compositions of the dots were not identified with EDXS analysis because a sensitivity of lower-Z element contamination especially formed on higher-Z material might be too small for EDXS.

We tried analyses of the samples with quadrupole-based SIMS at KEK and ToF-SIMS (time-of-flight SIMS) at ULVAC PHI, Inc. having more than 10^3 times higher sensitivity for lower-Z elements in comparison with XPS. Fig.5 shows a ToF-SIMS image (600×600 μm) with a resolution of 256×256 pixels and a color gradation of 40 steps corresponding to the ion intensity of sulfur compound. According to the image analysis, the brightest dots show sulfur concentrated locally with the size of around 20 μm. Although it is difficult to analyze quantitatively with ToF-SIMS, the sulfur composition was speculated to be several tens ~ hundreds at.ppm from the sensitivities and detection limits of XPS and ToF-SIMS.

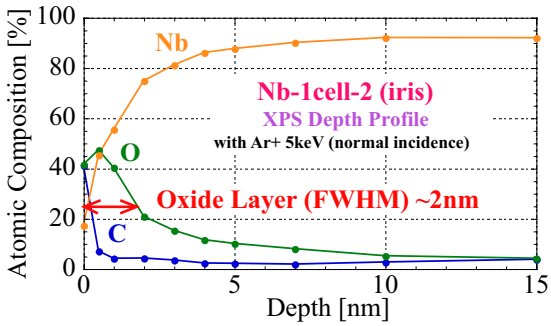

Figure 4: XPS depth profile with Ar ion etching of the sample set at the iris position. The thickness of oxide layer was around 2 nm.

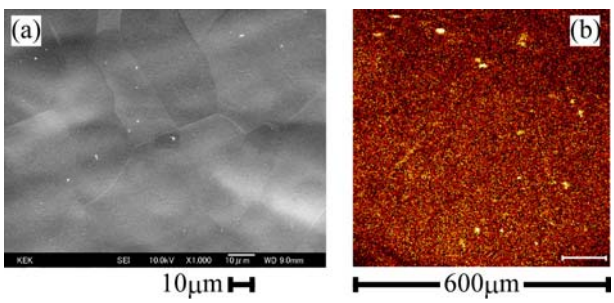

Figure 5: (a) SEM image. There were many dots but the compositions were not identified with EDX. (b) Secondary ion image for sulfur with ToF-SIMS with a resolution of 256×256 pixels and a color gradation of 40 steps. The bright spots show the concentration of sulfur compound.

CONCLUSIONS

We have started up the surface analysis team to research the production of high gradient superconducting cavities with high reproducibility. The UHV suitcase, the XHV surface analysis system with loadlock chambers, the niobium single cell cavity with sample coupons and handling procedure of the sample coupons were developed to understand well the condition of niobium surfaces processed with various treatment and conditioning methods. The EP process for the cavity with sample coupons and the subsequent surface analyses were carried out and the system was confirmed to work well. In XPS, it was found the surfaces were covered with niobium oxide with a thickness of 2~3 nm and there are no contamination elements such as sulfur within detection limit. According to SIMS results, there is sulfur dispersed on the surface and the amount of sulfur was estimated to be several tens ~ hundreds at.ppm at the surface. We are going to continue the surface studies for other recipes of surface treatments and conditionings with the system to find the cause of bad performance of cavities and its solution.

ACKNOWLEDGEMENTS

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