CHARACTERIZATION OF VACUUM CHAMBER SAMPLES FOR SUPERCONDUCTING INSERTION DEVICES

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Abstract

One of the key components of a superconducting insertion device is the ultra-high vacuum (UHV) chamber. In order to reach the UHV specifications of the ANKA storage ring, it is very important to control the surface chemical composition and find proper cleaning procedures. To keep the geometric and resistive wall losses caused by the electron beam of the accelerator as low as possible, it is essential that the top few μm of the surface exhibit low roughness and good electrical conductivity at temperatures between 4-30 K.

A 300 μm thick 316L stainless steel foil, galvanized with a 30 μm electrodeposited copper layer, is used for the next superconducting undulator demonstrator developed in collaboration between ANKA (Ångstrom source KArlsruhe at the Karlsruhe Institute of Technology, KIT) and Babcock Noell GmbH (BNG). In this paper, we report on a characterization of the copper surface by x-ray photoelectron spectroscopy as well as on residual resistivity ratio RRR measurements after different cleaning procedures and annealing at various temperatures for different periods of time.

INTRODUCTION

A new superconducting undulator demonstrator is currently under development in a joint collaboration of ANKA and BNG. The 1.5 m long ultra-high vacuum (UHV) chamber ("liner") is a crucial component of such an insertion device. To achieve the maximum magnetic field on axis, the gap between the superconducting coils has to be as small as possible, meaning that the thickness of the liner itself must be minimized. Furthermore, the liner has to fulfil the UHV specification of the ANKA storage ring. In addition, a high residual resistivity ratio (RRR) $\geq$ 100 and a smooth surface with a maximum roughness σ of few μm are required to minimize resistive wall heating by image currents and/or geometric discontinuities at low temperatures.

In this contribution, we report on studies performed on samples prepared for the superconducting undulator demonstrator liner. The samples were made of a 300 μm thick stainless steel sheet, electrodeposited with a 30 μm Cu, and with a 2-5 μm Ni buffer layer in-between. X-ray have been applied to study the chemical composition of the surface after different cleaning procedures and annealing at various temperatures for different periods of time.
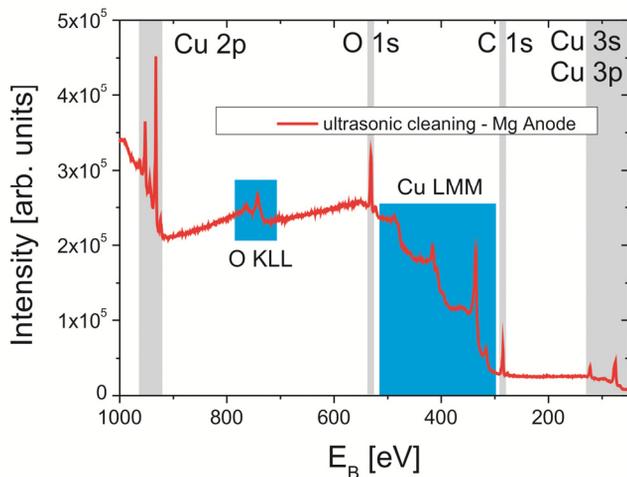
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Figure 1: XPS survey spectrum of the as-cleaned Cu foil using Mg K_α excitation. The major Auger (blue boxes) and XPS peaks (grey boxes) are labeled.

XPS EXPERIMENTS

In order to examine the surface properties of the 30μm Cu layer, XPS was used to identify and quantify possible impurities. 15x15 mm^2 samples were cut and cleaned with isopropanol in an ultrasonic bath. Subsequently, the liner foil was inserted into the UHV analysis system. XPS was performed using Mg K_α excitation and a Specs PHOIBOS 150 MCD electron analyzer. The sample was measured under different conditions: 1 day after introducing the sample into the UHV chamber, after 15 min annealing (approx.. 100 °C), after 1 h annealing (approx.. 250 °C), and after 15 min Ar$^+$ sputtering with E = 3 keV and an Ar pressure of 6.5×10^{-5} mbar. In Fig. 1, the survey spectrum of the as cleaned copper foil before annealing is plotted. In addition to the expected XPS and Auger lines of Cu we can observe signals from O and C contaminations.

The AES Cu LMM spectra in Fig. 2, together with the Cu 2p spectra (not shown) reveal that the surface layer of the Cu foil consists of both Cu_2O and metallic Cu. The spectrum remains very similar after annealing at 120 °C, but changes significantly after annealing for 1 h at 250 °C, indicating that the Cu_2O is reduced to Cu metal. While for clean Cu_2O, no thermal reduction is reported at temperatures up to 1073 K [1], the presence of a carbon contamination layer can induce a thermal reduction already at a much lower temperature (250 °C) under the
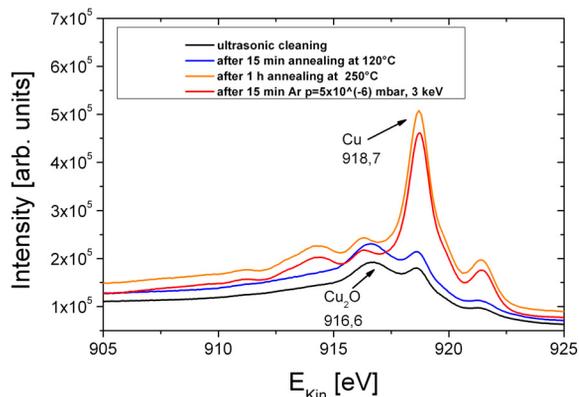
Figure 2: Cu LMM Auger spectra after different sample treatments [1]. Peak positions for Cu_2O and metallic Cu are indicated.

formation of CO_2 [2]. A detailed examination of the Cu 2p XPS spectra before annealing (not shown) reveals also a small contribution of CuO. This indicates that the topmost layer is only partially oxidized. After annealing for 15 min at approximately 100 °C, the intensity of this CuO contribution decreases. This is consistent with the observation of Lee et al. [3] that a partially oxidized Cu film was completely reduced to Cu_2O after annealing at 107 °C for 30 min at 2.7×10^{-7} mbar. The reduction is explained in Ref. [3] by an oxygen diffusion into the bulk of the Cu film, since this reaction is thermodynamically favoured compared to oxide desorption.

Besides carbon and oxygen, no additional contaminants (like P, F, or Cl) that could potentially be incorporated into the Cu layer during the electrodeposition procedure were detected. The fact that we observe a significant reduction of the C 1s signal after an Ar^+ sputter treatment of the surface indicates that most of the carbon is due to adsorbates and carbon contamination within the film is small. Summarizing the XPS results, copper oxide and the carbon contamination seem to be restricted mostly to the topmost layers and the studied Cu foil has no significant contaminations of further elements or molecules.

THERMAL DESORPTION

Besides the direct spectroscopic measurements of the Cu surface with XPS, the thermal desorption behaviour of the Cu surface was investigated. As for the XPS studies, the aim was to check the purity and the UHV compatibility of the electrodeposited Cu foil. We note that, in all the experiments presented in this paragraph, samples were mounted on a sample holder. Contaminations of this sample holder or gas trapped between the sample holder and the sample itself may thus also contribute to the total pressure. Thus, the outgassing behaviour of the foil itself is possibly less pronounced than observed in the following experiments.

The total pressure of the UHV chamber was examined as a function of time while heating to 350 °C. This experiment was done in a UHV chamber with a base

pressure of 4×10^{-9} mbar. After heating the empty sample holder, the procedure was repeated with the liner foil mounted. The time-dependence of the total pressure, measured with a Varian UHV-24 hot filament ion gauge, is shown in Fig. 3. While pressure decreases slower with mounted foil due to desorption of adsorbates, it still reaches a value of approx. 4.5×10^{-9} mbar after 50 min. This is a first indication that contaminations on the Cu surface do not appear problematic for UHV compatibility. In a further experiment, the residual gasses were monitored by a mass spectrometer (PrismaPlus™ QMS 220 from Pfeiffer Vacuum) while the total pressure was independently measured. First, the empty sample holder was heated to 600°C in a UHV chamber with a base pressure in the 10^{-11} mbar range. Second, the procedure was repeated with the liner foil mounted. The partial pressures of the released gases were monitored in the range from 1 to 64. The following masses were found to contribute to the total pressure in the UHV chamber: 1 (H), 2 (H_2), 12 (C), 13 (CH), 14 (CH_2/N), 15 (CH_3), 16 (CH_4/O), 18 (H_2O), 28 (CO/N_2), and 44 (CO_2).

Finally, the total pressures were monitored in a pressure versus time scan while heating up to 560 °C. Two different cleaning solutions, i.e., isopropanol (ISO) and NGL 17.40 PSP (NGL, www.ngl-cleaning-technology.com), were used to investigate a possible change in the signal. These measurements were also
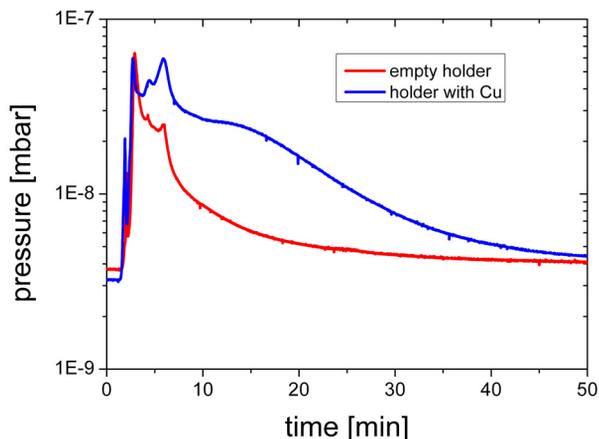

Figure 3: Total pressure of the UHV chamber as a function of time with empty sample holder at 350 °C (red curve) and with Cu liner sample at 350 °C (blue line).

repeated just with a stainless steel sheet sample (i.e., without the electrodeposited Cu layer; sample size 15 x15mm^2). We find that the total pressure recovers already after 30 min. This faster pressure decay we attribute to the heat treatment at higher temperature. We find that the Cu layer slightly increases outgassing. However, the pressure difference after 30 min is only about $1.0*10^{-10}$ mbar.

Similar to the XPS experiments discussed above, these heating experiments also suggest that the Cu liner foil

does not contain contaminations critical for the UHV conditions of the storage ring.

RRR ANALYSIS

After studying the cleanliness of the UHV chamber liner as discussed above, the low-temperature resistivity of the 30 μm thick Cu layer was measured at CEA-Irfu. Foil samples with a size of 10 x 100 mm^2 were cut and characterized by their residual resistivity ratio (RRR), which is defined as the ratio of the electrical resistivity at 295 K to the resistivity at 0 K (ρ_{295K}/ρ_{0K}). For convenience, often the resistance ratio $R_{295K}/R_{4.2K}$ is measured, but in our case the 0 K resistivity is extrapolated using the method described in [4].

Table 1: RRR results of Cu liner films. Cleaning solutions and heat treatments are given for the various samples.

Sample	Cleaning solution	heat treatment	RRR
1	ISO	bake out @100°C for 10h	167
2	NGL	bake out @100°C for 10h	205
3	ISO	100°C for 10h	197
4	NGL	100°C for 10h	208
5	ISO	150°C for 10h	196
6	NGL	150°C for 10h	153
7	ISO	325°C for 10h	147
8	NGL	325°C for 10h	159
9	ISO	as received	176
10	NGL	as received	159

If the resistance measurements are performed on sufficiently large, well recrystallized samples and at very low temperatures, the residual resistivity depends mainly on the impurity content of the sample [5]. The Cu-coated samples were cleaned either with isopropanol (ISO) or with NGL 17.40 PSP (NGL), and heat-treated at 100 °C, 150 °C, and 325 °C under UHV conditions (p_{total}(20 °C) ≤ 2*10^{-9} mbar). The results of the RRR measurements for all samples are listed in Table 1. Additionally, the RRR of a stainless steel sample of the same dimension was measured. The RRR of this sample is 3.1, i.e., a very small value. For the determination of the RRR value of the liner samples, the impact of the stainless steel foil can thus be neglected. Looking at the RRR results, two

aspects are remarkable. First, the RRR values neither depend on the temperature treatment nor on the cleaning procedure. Second, the RRR values are always in the range between 150 and 200, which is a very good value compared to only 5-20 of standard bulk Cu. In our case, the heat treatment has no significant effect on the RRR, different from the observations reported in a previous similar investigation [6]. In particular, the RRR values of our liner foils for the new superconducting undulator demonstrator do not degrade after heat treatments at temperatures up to 325°C for 10 h.

SUMMARY AND CONCLUSION

The quality of liner foils of the next superconducting undulator demonstrator for ANKA was characterized by XPS, RGA, and RRR measurements. We find the electrodeposition process to produce high quality Cu layers. No indication for contaminants (like P, F, or Cl) incorporated in the Cu layer can be found. No unexpected elements or molecules critical for UHV compatibility could be detected. Furthermore, high RRR values of up to 200 prove the high purity of the bulk Cu layer. The high conductivity at low temperatures, together with the measured roughness of the liner foil (< 2 μm), meet the requirements for a liner that minimizes heat intake from the electron beam of the storage ring.

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