TiN COATING OF RF POWER COMPONENTS FOR THE EUROPEAN XFEL

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Abstract

Thin TiN layers on surfaces of RF components have the ability to reduce secondary electron emission and multipactor effects. A new equipment was designed and built for mass production at DESY to generate TiN films by deposition of Ti vapour in low pressure ammonia ambience. This new setup was already transferred to industry for the XFEL RF coupler production. The technical layout of the apparatus and results of SEM/EDX surface analysis and depth profile analysis are shown.

INTRODUCTION

Field emission and multipacting effects often limit the performance of RF power components. In consequence the application of RF power leads to excessive heating and thermal stress and in case of ceramic windows, in melting of the brazed seam and cracking of the window itself. Together with special design based on avoiding electron resonance conditions, a thin layer with reduced secondary emission yield (SEY) suppresses multipacting. Titanium nitride is the most commonly used material because of its low SEY of 1,2 to 1,5 compared to 3 – 5 of Aluminium Oxide and the good stability in high RF fields [1][2][3].

PROCESSING METHOD AND GENERAL SETUP

A Titanium wire of 1mm diameter with a purity of not less than 99.8% is used in our setup as sublimation source. A sufficient sublimation rate is reached at a wire temperature above 1700 °C attended by deposition of Ti vapour on the substrate, here a ceramic coupler window. Conversion to TiN takes place in low ammonia atmosphere, in the range of 5 x 10^{-4}mbar, after deposition. In various tests we found out that a current of 23 A applied for 60s to the wire get the best coating results. To ensure a better deposition a preheating of the substrate sometimes is needed. This preheating is done with a moderate heating of the wire up to 1000 °C. During the heating impurities desorbs from the wire as well, while the sublimation rate of Ti is still negligible.

After deposition the coated components are stored in the vessel under ammonia atmosphere with a pressure in the range of 15mbar to 20 mbar for at least 24 hours. A resistivity of the TiN layer up to 10 Mohm/sq was reached then and enhanced to 1Gohm/sq after 1 day under normal air. Table 1 shows some characteristic coating parameter [4].

Table 1: Characteristic coating parameter

Purity of material (Ti)	99.8%
Heating current	23 A
Duration	60s
NH_3 pressure, coating	5 x 10^{-4}mbar
NH_3 pressure, post processing	15 – 20 mbar
Wire temperature, preheating	1000 °C
Surface resistivity after 24 h	10 Mohm/sq
Surface resistivity, after 72h	1 Gohm/sq

A schematic layout of the setup is shown in Figure 1.

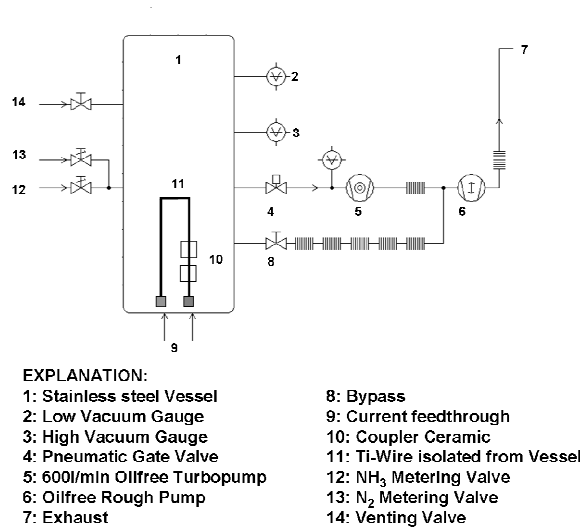

Figure 1: Schematic layout.

To afford a batch production of coated coupler windows, a complex inner assembly was constructed at DESY whereas BESSY/Berlin supplied most of the semi-finished products due to a cooperation. With this setup, embedded in a cylindrical stainless steel vessel of 0,7m diameter and approximately 1m height, it is possible to coat up to 20 ceramics simultaneously from the inner and outer side and in addition a face side coating as well. Special attention has to be paid to the flexible suspension of the wires made of copper strings. This secures the wires from stretching during the intensive heating. Figure 2 and 3 show a detailed CAD drawing and a picture of the inner assembly.

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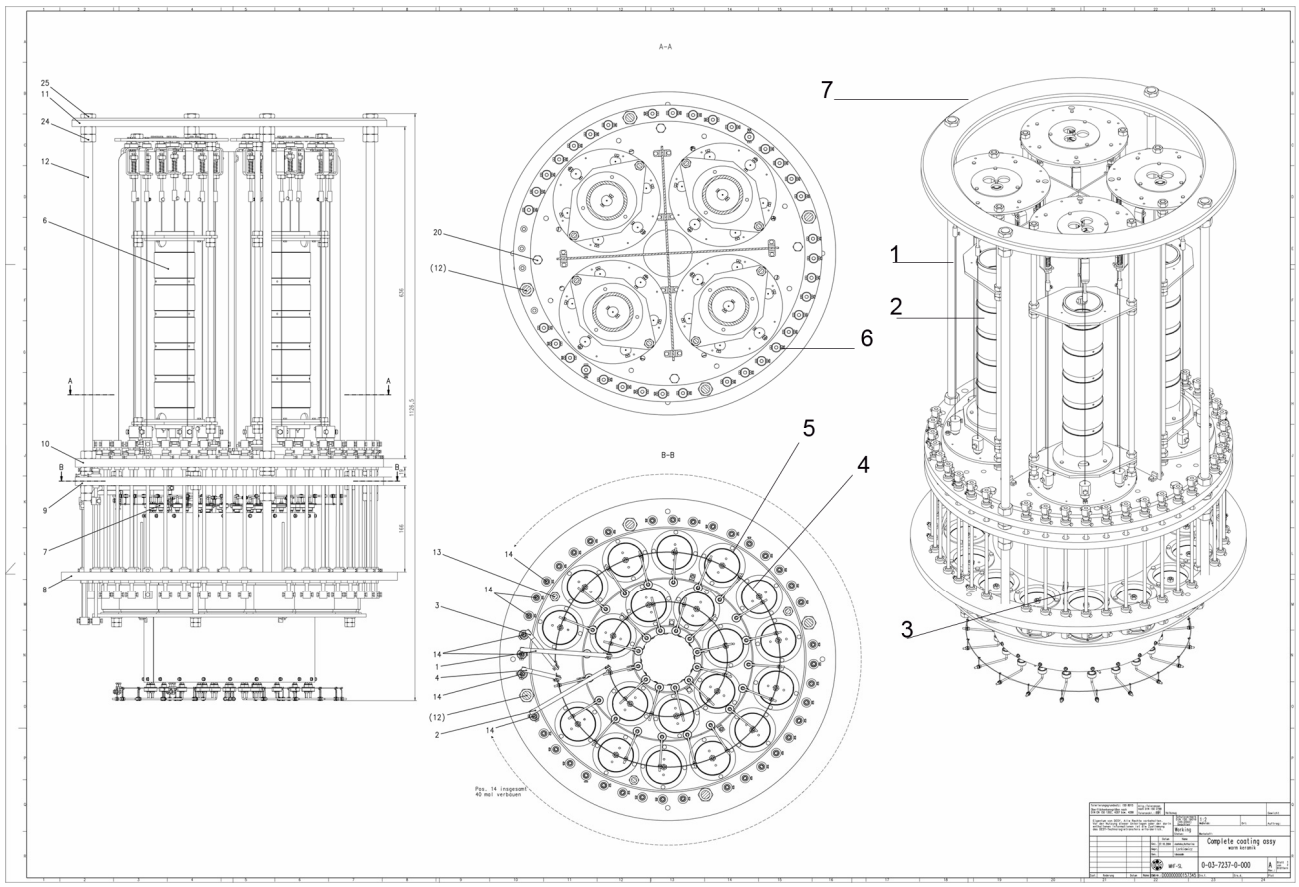

Figure 2: Detailed CAD drawing of inner assembly. 1: outer Ti wire, 2: coupler ceramics, 3: current supply, 4: Ti wire for front side coating, 5: front side ceramics, 6: isolation, 7: support frame.

Figure 3: Inner assembly, with 4 mounted ceramics. 1: flexible suspension of the wire, 2: ceramic, 3: vessel flange.

In addition it is also possible to make a vapour deposition at the ceramic on 1.3 GHz waveguide windows (Figure 4). Here the coating only can take place one by one due to the larger scale of the windows. Furthermore this assembly is more or less handmade and not useable for batch production.

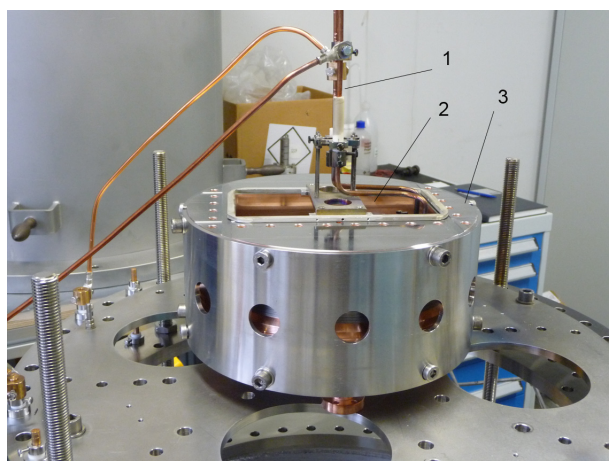

Figure 4: Setup for waveguide window coating. 1: current supply, 2: ceramic, Ti wire inside the pillbox is not visible.

SURFACE ANALYSIS

Since an in-situ measurement of the layer thickness is not possible, spot tests with applicable surface analysis methods has to be done from time to time during the batch production. Generally it can be said that a TiN layer on an almost rough Aluminium-oxide surface is hardly to determine because of the minor thickness of only 10 nm. Nevertheless we have to inspect the components which were coated by the industry periodically.

At present the only available analysis tool at DESY with fast access is a SEM/EDX device. To have a comparison we decided to commission additionally SEM/EDX and Glow Discharge Optical Emission Spectroscopy (GDOES) at the company O.f.U. Gesellschaft für Oberflächen- und Festkörperuntersuchungen/Hamburg. Both verifications showed comparable results. Energy dispersive X-ray spectroscopy (EDX) is not the commonly used method to determine the layer thickness, but it gives us an indication of the layer quality however. Using Scanning Electron Microscope (SEM) images of the breaking edge on coated ceramic samples we are able to make a statement about the thickness. Figure 5 shows an EDX spectrum of TiN coated sample with an approximately 25 nm layer measured at DESY. Figure 6 shows a SEM image taken by O.f.U. of a breaking edge of a TiN coated sample. The marked area is the thin TiN layer with a thickness of about 10 nm.

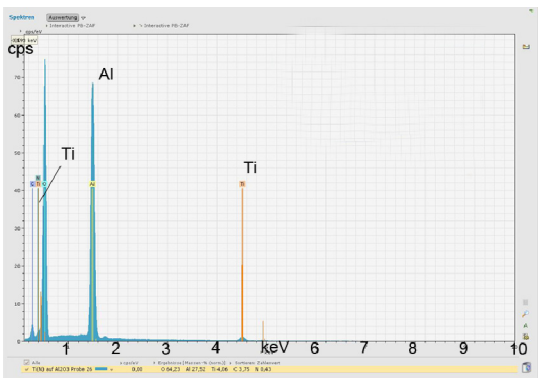

Figure 5. EDX spectrum of a TiN coated sample

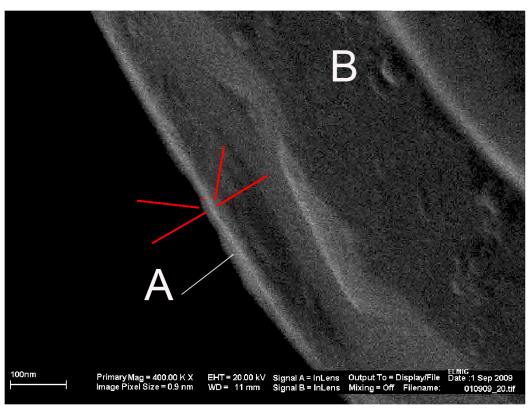

Figure 6. SEM image of a breaking edge of a TiN coated sample. The red marked area indicates the layer.

Figure 7 shows an EDX spectrum of the surface layer of a TiN coated sample and a spectrum taken from the same sample, but from the breaking edge. Titanium can be clearly seen on the surface. Unfortunately the peak associated to the element Nitrogen is covered by the Ti peak due to the same emission energy. The upper spectrum was taken at point "A", the lower one at point "B" of the SEM image in Figure 6.

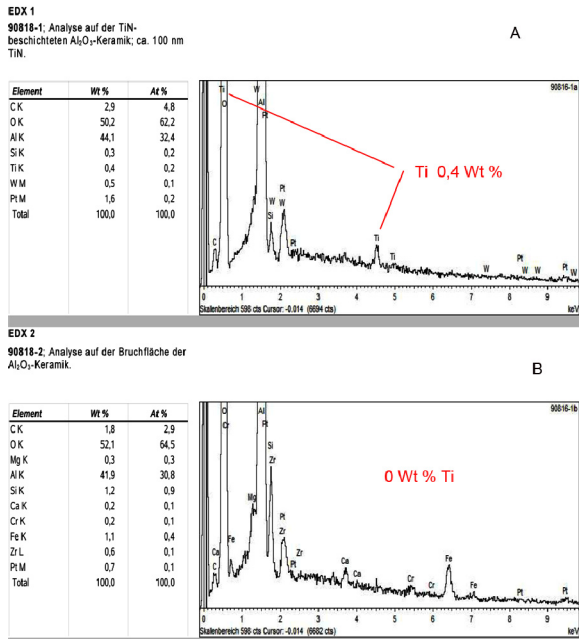

Figure 7. Spectra taken at O.f.U. from a TiN coated sample. The upper one shows a spectrum from the surface layer, the lower one a spectrum from the breaking edge.

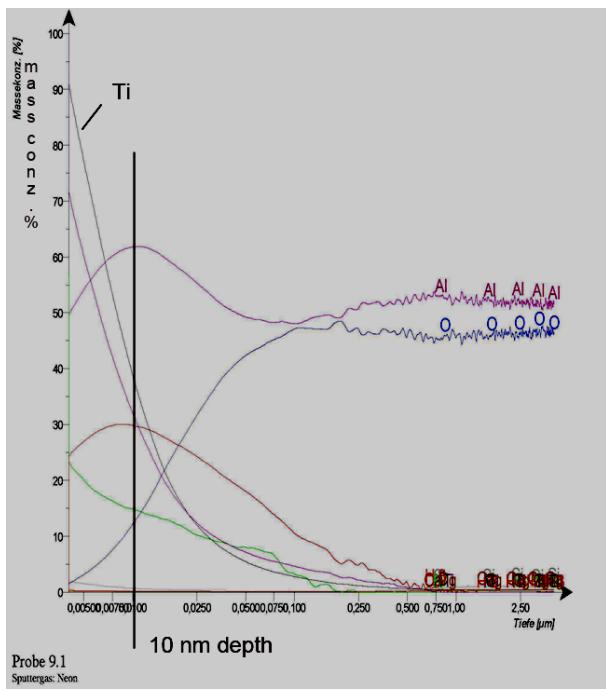

Figure 8. Depth Profile taken with GDOES

Figure 8 shows a depth profile measurement done with GDOES at O.f.U. The red arrow marks the Ti line. Up to 7 nm Ti is the most dominant element. Below 7 nm Al dominates the spectrum.

SUMMARY

With this setup it is possible to coat XFEL Coupler components in a batch production scale. Because in-situ measurements of the layer thickness are not possible, we analyse the layer after the process with SEM/EDX and GDOES to find an optimal parameter set for coating. Spot tests to control the industrial coating process with these methods are applicable but extensive. A good hint for the coating quality is the surface resistivity measurement after coating.

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