# Development of X-band accelerating structures for high gradients<sup>\*</sup>

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**Abstract:** Short copper standing wave (SW) structures operating at an X-band frequency have been recently designed and manufactured at the Laboratori Nazionali di Frascati of the Istituto Nazionale di Fisica Nucleare (INFN) using the vacuum brazing technique. High power tests of the structures have been performed at the SLAC National Accelerator Laboratory. In this manuscript we report the results of these tests and the activity in progress to enhance the high gradient performance of the next generation of structures, particularly the technological characterization of high performance coatings obtained via molybdenum sputtering.

Key words: high-gradients, soft X-ray FEL, RF cavities for particle accelerators, RF sputtering

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#### 1 Introduction

The next generation of linear accelerators is highly demanding in terms of maximizing accelerating gradients, to minimize the overall machine length and cost. Experience shows that obtaining high gradients with a normal-conducting structure requires operation at a relatively high frequency. However, the accelerating gradient of normal conducting accelerating structures is mainly limited by RF breakdown [1].

Currently, researchers face many challenges in improving the performance of X-Band accelerating systems. The current main thrust is to characterize high gradient RF performance in various structures made with different materials. In particular, INFN is currently working on SPARC/SPARX (Sorgente Pulsata Autoamplificata di Radiazione Coerente/Sorgente Pulsata Autoamplificata di Radiazione X) SASE-FEL [2,3] projects and there is a promising proposal of an X-Band option for beam acceleration from 0.3–0.5 GeV to 1 GeV and beyond. An intense activity of design, construction and experimental tests of short 11.424 GHz high power standing wave (SW) sections started at Frascati in the framework of a collaboration with SLAC (Stanford Linear Accelerator Center) and KEK (Kō EnerugĪ Kasokuki Kenkyū Kikō) laboratories. The goal of the collaboration is to determine the maximum sustainable gradient in normal-conducting RF powered particle beam accelerators with an extremely low probability of RF breakdown.

In Fig. 1 we show the structures designed and manufactured at Laboratori Nazionali di Frascati (LNF) by INFN using vacuum brazing and electroplating procedures. These SW structures have been manufactured by the Co. Me. B. S. r. l. Company in Italy.

The SW structures are made of three cells with

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Fig. 1. Photographs of the three manufactured X-band cavities: (a) Cu brazed; (b) Mo brazed and (c) Cu electroformed.

the shape shown in Fig. 2. Structures are designed to maintain a higher gradient in the middle cell, while the two outer cells have half the gradient of the middle cell. The geometry of the middle, high gradient cell is based on the geometry of a periodic accelerator structure cell with an aperture radius of 5.65 mm and an iris thickness of 4.6 mm [4]. The surface field profile is shown in Fig. 3. These surface fields are normalized to 10 MW of RF losses. Data of the cavity characteristics were calculated using the 2D finite element code SLANS [5]. Both calculations and measurements for the on-axis field for the vacuum brazed structure are shown in Fig. 4.



Fig. 2. Shape of the 1C-SW-A5.65-T4.6 structure as calculated by the 2D finite element code SLANS.

Additional details, mechanical drawings and assembly information regarding these structures are available in Refs. [6, 7]. High power tests of both Cu and Mo brazed structures have been performed at the SLAC's ARD that is carrying out advanced high gradient research for future colliders. In this contribution we will present the results of the high power tests of the vacuum brazed copper section. Results of a molybdenum brazed section and of a copper electroplated section will be discussed in a forthcoming publication.



Fig. 3. Surface electric field (blue solid line) and magnetic field (red dashed line) vs. distance along the surface for the  $\pi$ -mode of the 1C-SW-A5.65-T4.6-Cu structure calculated by the finite element code SLANS. The fields are normalized to 10 MW of RF losses.



Fig. 4. Designed on-axis electric field vs. distance along the axis for the π- mode of the 1C-SW-A5.65-T4.6-Cu structure (blue line) and bead-pull data (red crosses) for the 1C-SW-A5.65-T4.6-Cu-Frascati-#2 structure. Data are normalized to maximum values.

## 2 High gradient tests of the INFN brazed SW structure

The high power tests of the INFN brazed SW structure are a part of a complex set of research efforts on the fundamental physics of RF breakdowns [4]. Characterization of the RF breakdown performance of short SW structures is one of the main test-vehicles in this research. Tests of the SW structures have been performed at SLAC at two dedicated RF power stations. Up to now, more than 30 structures

have been tested, including the three made by INFN. To identify structures we use abbreviations derived from the names of the corresponding periodic structures including the manufacturer's name and the serial number. As an example, the initials 1C-SW-A5.65-T4.6-Cu-Frascati-#2 refer to the high-gradient cell 1C with a 5.65 mm aperture radius (A5.65) and a 4.6 mm thick iris (T4.6) manufactured by INFN at LNF. Finally, #2 indicates that this is the second structure manufactured.

After several tests of different SW structures we

found that the RF breakdown performance of structures with the same geometry and materials is reproducible and almost independent of initial surface conditions or manufacturers [4]. We also point out that calculations of the breakdown probabilities are obtained after the initial processing, when the breakdown rate does not change over hours. Tests were performed with a 60 Hz RF pulse repetition rate. After installing the structure in the setup test we increased the RF power gradually and recorded the breakdowns.





Fig. 5. RF pulse shaped simulating beam loading by a multibunch beam: (a) measured input and reflected RF power together with the reflected power calculated using an equivalent circuit; (b) the peak surface electric field and the peak surface pulse heating calculated using the same RF input pulses. The charging time is  $\sim 170$  ns and the flat part of the pulse is  $\sim 150$  ns.



Fig. 6. The breakdown probability for the soft copper structure 1C-SW-A5.65-T4.6-Cu-Frascati-#2 vs. (a) gradient; (b) peak pulse heating; (c) peak surface electric field; (d) peak surface magnetic field. The structure is powered with a shaped RF pulse to simulate beam loading with a charging time of  $\sim 170$  ns and a flat part of: 1) 600 ns (red diamonds), 2) 150 ns (black circles).

The test pulse shape simulates the multi-bunch beam loading in a SW structure. As an example, Fig. 5(a) shows the pulse shape together with the peak surface electric field and the calculated peak pulse heating (Fig. 5(b)).

Figure 6 shows the breakdown probability at two different pulse lengths of the INFN brazed structure: 1C-SW-A5.65-T4.6-Cu-Frascati-#2).

The breakdown probability increases with the pulse length and it is highly correlated with the calculated peak pulse heating temperature, but also the gradient and the peak surface electric field and the peak surface magnetic fields. When we compare the latter structure with other brazed devices, the results are in accordance with those obtained with structures of the same geometry manufactured at KEK as shown in Fig. 7. Fig. 8 shows the two halves of the Frascati structure after their sectioning for observation after the RF tests. We observed negligible



Fig. 7. (color online) Comparison of the breakdown probability for three soft-copper 1C-SW-A5.65-T4.6-Cu structures: 1) Frascati-#2 (blue circles); 2) KEK-#2 (red diamonds); 3) KEK-#4 (green pyramids). The structures are powered with a shaped RF pulse to simulate beam loading with a charging time of ~170 ns and a flat part of ~150 ns.



Fig. 8. Photo of the 1C-SW-A5.65-T4.6-Cu-Frascati-#2 structure sectioned after high power tests for diagnostics.

damage due to the breakdowns, a behavior typical of these copper structures.

One of the main results of the test is that the manufacturing technology of high gradient accelerating structures is mature and devices manufactured independently by INFN, KEK and SLAC show comparable performances. As a consequence, we may consider high temperature brazed Cu structures as a reference for studies devoted to the improvement of accelerator performances by reducing the breakdown rate at high gradients.

#### 3 New technological researches

In order to improve the performance and reduce the manufacturing cost of the accelerator cavities for the next generation of accelerators, in many laboratories an extensive manufacturing technology and R&D activity is in progress. In particular, researchers are exploring the improvements in sputtering techniques to optimize homogeneous deposition of materials with the attempt to better characterize the achieved coatings. In particular, we recently focused at Frascati on the possibility of growing thin reproducible coatings via molybdenum sputtering.

Molybdenum is a Group 6 chemical element with an atomic weight of Z=42 g/mole. Pure molybdenum is characterized by a high melting point, hot strength and creep resistance. The free element has a Mohs hardness of 5.5 and the sixth-highest melting point (2623 °C) of any element. It readily forms hard, stable carbides. For this reason it is often used in highstrength steel alloys, although in nature it does not occur as a free metal. Molybdenum burns only at temperatures above  $600 \,^{\circ}\mathrm{C}$  and although it has one of the lowest coefficients of thermal expansion among commercially used metals it behaves harder than Cu. As a consequence, Mo is an interesting material for accelerator components and a stimulating option for RF linear accelerating structure with low breakdowns at high RF power. At CERN, dc breakdown rate tests are in progress with materials such C, Cu, W, Nb, Mo, Cr, V and Ti. For application in high gradient accelerating structures, the results of the Mo breakdown rate and of its electric field are very promising if compared with Cu materials [9]. However, small levels of impurities can impair molybdenum properties, and molybdenum is very difficult to machine with a low surface roughness (its typical roughness is  $\sim 350$  nm). Actually, this may limit the performance of accelerating sections due to RF breakdown.

This phenomenon has been also observed at SLAC during the high power tests of a brazed molybdenum structure [10].

To overcome these problems, and to improve RF breakdown performance, we started a feasibility study of various sputtering techniques. Using low surface roughness ( $\sim$ 70 nm) Cu cylindrical samples, 2 cm in diameter and 3 mm in height, we deposited on top thin Mo coatings of various thickness ranging from  $\sim$ 180 to  $\sim$ 600 nm by sputtering.

Using atomic force microscopy (AFM) we characterized the surface morphological quality of the Mo coatings. The original undulations at the Cu surface were  $\sim 700$  nm, with many overlapping spikes, due to the lathe step when machining the Cu cells. The spikes disappear when a layer of  $\sim 100$  nm of Mo is deposited. In general, the results confirm that the Cu machined surfaces have an initial low roughness that is preserved after sputtering. The molybdenum on a copper surface acts like a smooth layer, actually improving the original roughness. In Fig. 9 we show the copper three-cell structure layout to be sputtered with molybdenum.



Fig. 9. Three cells  $\pi$ -mode standing wave Xband structure.

Since Cu and Mo have different thermal expansion coefficients, the Mo layer deposited on top of a Cu surface is not stable under variable temperature conditions. Molybdenum changes its crystalline structure around 900 °C and it is necessary to investigate and control the diffusion mechanisms. We studied the deposition of thin Mo layers followed by thermal treatment to fix the film on the substrate. We compared a set of different oxygen-free-high thermal conductivity (OFHC) Cu samples sputtered with variable Mo coatings whose thickness ranged from 180 nm to 600 nm followed by thermal treatments from 600 °C to 750 °C for 10 minutes. The results were also compared with molybdenum samples with a triangular shape of a few square cm surfaces, 8 mm thick machined only on one side with a roughness of 300 nm, and with and without a thermal treatment at 855 °C. Fig. 10 shows a comparison of the representative coated surfaces. These Scanning Electron Microscope (SEM) images have been collected at the KEK laboratory after heating at 600 °C and 2 h of annealing of the whole sample. They are magnified views of the OFHC Cu samples surfaces with a sputtering of molybdenum (from ~180 nm to 600 nm) followed by 10 min of treatment at different temperatures.

The coating tests clearly point out that Mo coatings should really be used in high gradient accelerating structures. In Fig. 10(a) we show a Cu surface with 180 nm sputtering of Mo after annealing at 750 °C for 10 minutes. The texture and the porosity observed on top of the structure surface are due to the structure of the underlying substrate. The image points out that a sputtering thickness of 180 nm is not enough to have a homogeneous surface coating. Fig. 10(b) shows a surface with 350 nm sputtering of Mo after annealing at 600 °C for 10 minutes. The surface characters still reflect the profile of the substrate, however they show a reasonable contact force between the Cu and Mo materials. Finally, Fig. 10(c) shows the surface with 600 nm sputtering of Mo after annealing at 750 °C for 10 minutes. Due to the thermal stress induced by heating the material at 600 °C for 2 h, a clear separation of the Mo film from the substrate is observed. These photographs mainly characterize the surface of the substrate. The reasons for the separation phenomena may be associated with the low contacting force and the intense thermal stress on the thin Mo coating. Indeed, the thermal expansion coefficients of Cu and Mo are quite different, i.e.,  $1.6 \times 10^{-5}$  and  $5 \times 10^{-6}$ , respectively. We suggest that the thinly coated Mo film on the Cu substrate could be associated with the intense sheering force following the heating process. To confirm this mechanism, additional tests will be necessary with a mirror surface as a substrate.

To better understand the feasibility and the properties of copper samples with molybdenum coatings, Rutherford backscattering spectrometry (RBS) experiments were also performed at the University of Catania. RBS measures the energies of backscattered particles from a target and the compositional depth profile can be easily determined from an RBS



Fig. 10. Comparison of SEM images taken at the KEK laboratory after heating at 600 °C and 2 h of annealing of the whole sample. The images (details are in the photographs) correspond to Cu OFHC samples with: (a) 180 nm sputtering of molybdenum followed by a 10 min treatment at 750 °C; (b) 350 nm sputtering of molybdenum followed by a 10 min treatment at 600 °C; (c) 600 nm sputtering of molybdenum followed by a 10 min treatment at 750 °C.

spectrum. Actually, the elemental composition of the sample can be determined from the positions of peaks in the energy spectrum. The depth of the Mo layer can be determined from the width and shifted position of these peaks, and the relative concentration of the peak heights. We performed RBS analyses with a <sup>4</sup>He<sup>+</sup> ion beam to characterize the depth profile of Mo on the Cu substrates. The Rutherford backscattering spectroscopy is a unique and valuable tool for studying the inter-diffusion processes in multilayer structures, yielding detailed insight about the effects of reactions, diffusion and segregation on the structure and composition of different interfaces.

Figure 11 shows the typical RBS spectra of the investigated samples. In the left panel we compare the profiles obtained with a 2 MeV He beam at normal incidence and a scattering angle of  $165^{\circ}$  on Sample #1, made with a 300 nm Mo coating on Cu, as deposited (black curve) and after 10 minutes of annealing at 750 °C (red curve). The Mo surface scattering is indicated in the spectrum. The thickness of the Mo film is 294 nm with a minor contamination of argon and carbon.

After annealing, the curve shows a weakening of the Mo content in the range between channels 400– 550 and an increase around channel 380, a behavior compatible with inter-diffusion of the Mo at the Cu-Mo interface. Inter-diffusion is the process of diffusion and mixing of atoms so as to approach a homogeneous mixture at the interface. In the right panel of Fig. 11 we compare profiles obtained with a 2.5 MeV He beam at normal incidence and with a scattering angle of 165° on Sample #2, made with a 600 nm Mo coating on Cu (black curve), as deposited and after 10 minutes of annealing at 750 °C (red curve). The estimated thickness of the Mo coating is 615 nm with



Fig. 11. Comparison of RBS spectra performed at the University of Catania on two OHFC Cu samples with a 300 nm and 600 nm sputtering of molybdenum. Left panel: 2 MeV He beam at normal incidence with scattering angle of 165° on sample #1 with 300 nm Mo/Cu as deposited (black curve) and after 10 minutes annealing at 750 °C (red curve). Right panel: 2.5 MeV He beam in normal incidence with scattering angle of 165° on sample #2 with 600 nm Mo/Cu as deposited (black curve) and after 10 minutes annealing at 750 °C (red curve). Green labels indicate Mo surface scattering.

minor contamination of carbon. If compared with the thinner coating, the similar increase of the Mo signal after annealing suggests that the inter-diffusion profile at the Cu-Mo interface is the same.

The characterization of a thin coating is challenging to analyze, although it is crucial to understand the properties of the manufactured structures. An important aspect of the characterization is the Cu diffusion into the molybdenum layer and the interdiffusion of Mo into the substrate. To clarify the process, we attempted to characterize the coating capability by investigating Mo films deposited on a 2  $\mu$ m  $SiO_2$  layer on top of a Si substrate; this makes the interpretation of RBS and electrical characterizations easier than on a Cu substrate. Fig. 12 shows the RBS spectrum collected with a 2 MeV  $^4\mathrm{He^+}$  beam at normal incidence and with a scattering angle of  $165^{\circ}$ (black line) with a simulation of the nominal sample structure. Mo and O surface scattering contributions are reported as green labels. The investigated sample has a nominal structure of 150 nm of Mo sputtered on the SiO<sub>2</sub> layer and the RBS analysis returns a Mo concentration of  $8.5 \times 10^{17}$  Mo/cm<sup>2</sup> that corresponds to a Mo coating of about 130 nm in thickness. The deposited film is characterized by a Mo concentration lower than a pure Mo film, compatible with the presence of oxide in the deposited film. The signal of the Mo film between channels 512–579 shows a  $\sim 20$  % reduction with respect to a pure Mo film. In fact an unexpected oxygen signal is observed at the surface between channels 200–238. The oxygen contamination is not uniform in the whole film. The simulation (see the red line in Fig. 12) indicates that the Mo film contains oxygen with a maximum concentration of 25%. On the latter sample we also performed an electrical characterization with a 4-point probe using the Van der Pauw configuration. With an applied voltage of 20 mV and a current of 2 mA we measured a sheet resistance of  $R_{\rm s} = (50\pm5)\Omega/({\rm sq})$ . Taking into account the thickness of the film we obtain a resistivity of about  $10^{-3}$   $\Omega$ cm compared with a sheet resistance of 0.5  $\Omega/(sq)$  of a typical 100 nm thick metallic Mo film.

The electrical measurement of the sheet resistance indicates a material resistivity higher by about two orders of magnitude compared with a pure Mo film, a difference compatible with the presence of oxides in the Mo film, in agreement with the RBS analyses. As a reference, a continuous Mo-oxide film should have a sheet resistance of ~1 M $\Omega$  [11].

To confirm the composition and check the chemical composition of the molybdenum films we also used the XPS Depth Profiling technique using the PHI 5600Ci system available at the unit of Genoa of INFN. The usual XPS depth sensitivity is  $\sim$ 5–10 nm depending on the analyzed materials. By etching the specimen under a test, by using an argon gas sputtering gun, information can be gathered about the in-depth composition of the sample. The process of sputtering and measuring is iterated in our case till all the molybdenum film is removed and the signal from the analysis area is the signal of clean copper from the deposition substrate.



Fig. 12. 2 MeV He (normal incidence, scattering angle 165°) RBS spectrum (black line) obtained on a Mo film deposited on a SiO<sub>2</sub> substrate. The simulation (red line) indicates that the Mo film contains oxygen with a maximum concentration of 25%. The film contains  $8.5 \times 10^{17}$  Mo/cm<sup>2</sup> that corresponds to a Mo coating of about 130 nm thickness. Mo and O surface scattering contributions are reported as green labels.

The results of the depth profile analysis of a 300 nm molybdenum film are reported in Fig. 13. The plot reports the atomic concentration of the four relevant components of the deposited film as a function of the distance from the surface of the sample. It appears that the film is uniformly contaminated (in depth) by oxygen ( $\sim 20\%$  in good agreement with the RBS measurements) and carbon ( $\sim 10\%$ ). Actually, with XPS measurements, the atomic concentration of carbon is affected by a strong error (up to  $\sim 30\%$  of the measured value) because the carbon atomic sensitivity is about one third of the atomic sensitivity of the oxygen and  $\sim 1/10$  of the atomic sensitivity of the molybdenum. Moreover, one of the major problems of these measurements is the calibration of the etching rates as a function of the distance from the surface, because the sputtering yield of materials is also sensitive to the sputtering conditions.

In our experiment we took advantage of the RBS measurement to define a precise position of the molybdenum copper interface.

From the de-convolution of the 3d molybdenum XPS peak, taken at the depth of  $\sim 150$  nm, it appears that contributions to this peak are due to metallic molybdenum (227.55 eV) and to bound molybdenum (228.08 eV) corresponding to MoO<sub>2</sub> (also in agreement with the film resistivity measurement). The results of spectra de-convolution are reported in Fig. 14.



Fig. 13. (color online) Depth profile of the 300 nm molybdenum film on copper in atomic percent. The sputtering parameters are 1 A Argon Ion at 4 keV energy on a raster covering an area of 5 mm×5 mm centered on the monochromatic X Ray spot on the sample.

Atomic Force Microscopy (AFM) analyses were performed at the University of Catania on the same sample of Mo, deposited on a  $SiO_2$  substrate. The AFM analysis can detect the details of a surface at the nanometer scale revealing the morphology of the system. The AFM analyses were performed using a Veeco-Innova microscope (MSNL-10 from Veeco Instruments, with an anisotropic geometry, radius of curvature  $\sim 2$  nm, tip height  $\sim 2.5 \ \mu m$ , front angle  $\sim 15^{\circ}$ , back angle  $\sim 25^{\circ}$ , side angle  $22.5^{\circ}$ ) operating in high amplitude mode, with ultra sharpened Si tips replaced as soon as a resolution loss was observed during the acquisition. The AFM images were analyzed by using the SPMLabAnalyses V7.00 software. The AFM image in Fig. 15 shows the granular structure of the Mo deposited film, with a measured roughness in the range of 1-2 nm. The average height of the grains is  $(4.2\pm1.6)$  nm. The structure is uniform all over the deposited film. Considering that Mo is highly reactive, the AFM analyses points out that the molybdenum oxide formation is highly probable at the grain boundaries. It suggests the need for a better control of the vacuum condition and/or of the gas contamination during the sputtering deposition procedure.



Fig. 14. Deconvolution of the 3d doublet of the molybdenum spectrum. The measured peak (150 nm deep inside the 300 nm film) is well represented by two doublets corresponding to electrons from the 3d shell, the first one at 227.55 in binding energy corresponds to the metallic Molybdenum, the second one at 228.05 shows the typical chemical shift corresponding to the Molybdenum from MoO<sub>2</sub>.



Fig. 15. AFM 3D reconstruction of a Mo film on SiO<sub>2</sub>. The granular structure of the Mo deposited film has an average height of (4.2±1.6) nm with a measured roughness of 1-2 nm. The triangular shape of the grains is an artifact due to the shape of the probe.

### 4 Conclusion

A copper X-band RF structure has been manufactured at the Laboratori Nazionali di Frascati. The structure was bonded using vacuum brazing. The device has been successfully tested at high power at SLAC. Performances measured in term of breakdown probability are comparable with those of similar structures manufactured at SLAC and KEK. The technological R&D of Mo coatings on Cu performed in collaboration with KEK and SLAC is a promising technique for increasing the accelerating gradient of accelerators at higher frequencies, as pointed out in this manuscript. Further characterizations are in progress and a SW structure made of copper cells, coated with Mo will be tested in the near future.

Technological advancements are fundamental in

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this research and other coatings could be considered later to additionally improve the performance of Cubased cells.

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