Phys. Scr. **T128** (2007) 157–161 doi:10.1088/0031-8949/2007/T128/030

# Beryllium coatings on metals for marker tiles at JET: development of process and characterization of layers

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Received 10 October 2006
Accepted for publication 7 December 2006
Published 8 March 2007
Online at stacks.iop.org/PhysScr/T128/157

**Abstract**
Preparatory study for the operation of the JET tokamak with a full metal wall (ITER-like wall project) also comprises several activities aiming at the development of thin beryllium coatings. The purpose is 2-fold: (i) to coat Inconel® tiles of the inner wall cladding; (ii) to develop methods for production of films for so-called marker tiles in order to enable monitoring of Be erosion from limiters. Properties of the marker film must match, as closely as possible, those of bulk Be. The first step in the R&D process was to assess coating methods and the quality of layers deposited on test coupons. Smooth, dense Be films of high purity and good adhesion to the substrate were deposited with an average deposition rate of $5 \pm 0.5\,\mathrm{nm\,s^{-1}}$ to a thickness of 7.5 $\mu$m. A marker structure consisting of a 7.5 $\mu$m Be film on top of a 2.5 $\mu$m Ni interlayer deposited on a bulk Be block has been developed and characterized by means of material analysis methods. An overview of manufacturing processes and properties of the marker coatings is presented.

PACS numbers: 81.15.−z, 81.15.Jj, 81.15.Ef, 68.55.Jk, 68.55.Nq

(Some figures in this article are in colour only in the electronic version.)

## 1. Introduction

The ITER-like wall (ILW) project is a very important part of the JET programme in support of ITER. The main aim of ILW is to implement a full metal first wall in JET and to demonstrate tokamak operation with beryllium (Be) components in the main chamber and tungsten (W) in the divertor [1]. Therefore, the R&D programme also includes activities to facilitate: (i) the coverage of the inner wall Inconel® cladding with Be coatings to reduce the influx of high-$Z$ metals to the plasma [2]; (ii) measurements of Be erosion from limiters by means of so-called marker tiles. These tiles will be distributed in areas of particular interest, such as outer poloidal limiters (OPL) and inner wall guard limiters (IWGL).

The 'marker' is a Be tile coated first with a thin (2–3 $\mu$m) film of an easily detected heavy metal (e.g., W, Re, Ni acting as an interlayer) and then coated with a few micrometres (∼7–9 $\mu$m) of bulk-like Be on top of that. The idea is shown schematically in figure 1. If the Be outer layer is eroded at the same rate as bulk Be, the erosion rate can be determined by measuring the change of the layer thickness after exposure to the plasma. This would enable the assessment of erosion smaller than the original film thickness. To measure erosion greater than 9 $\mu$m there will be precise notches

[4] See the appendix to the paper of M Watkins, 21st Fusion energy Conference Chengdu China October 2006

  

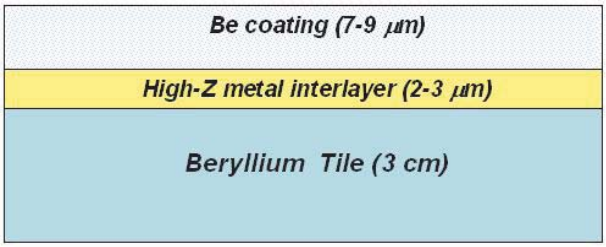


**Figure 1.** Schematic view of a marker tile.

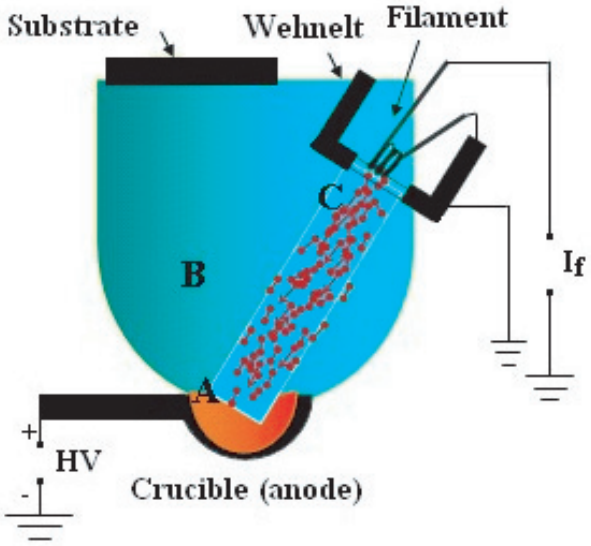


**Figure 2.** Schematic of the TVA principle: $I_f$, filament current; HV, high voltage power supply; (A), melted zone; (B), plasma created by metal vapour and (C), electron beam.

(10 and 20 $\mu$m deep) on the tiles. The major effort related to markers has been to select and optimize the most appropriate production process of dense, uniform and high-purity Be films on a high-$Z$ metal interlayer deposited on bulk Be limiters. The optimization (i.e., layer thickness, structure and purity) must ensure good adhesion and thermo-mechanical compatibility of the system. The thermionic vacuum arc (TVA) deposition process has been chosen in order to obtain high density films [3–4] for the production of markers. This paper describes results of the optimization process and broad characterization of test coupons.

## 2. TVA technique

The TVA technique is based on a high voltage (300–2000 V), low current (0.1–2 A) discharge in a pure vapour of the metal to be deposited (e.g. Re, Ni, Cr, W, Be) without using any buffer gas [3–4]. The TVA principle is presented in figure 2, where: 'A' is the melted zone, 'B' is the plasma created in pure metal vapour and 'C' is the electron beam produced by an externally heated cathode (tungsten filament). The metal evaporation takes place under vacuum conditions (less than $10^{-3}$ Pa). An externally heated cathode (W filament) produces a thermo-electron current of about 100 mA. Electrons are focused by the Wehnelt cylinder and accelerated to the anode biased to a high potential (1–6 kV). The focusing ensures easy ignition of an electrical discharge. This creates a strong local heating and, as a consequence, metal evaporation and build up of the local pressure to about 100 Pa. Ionization of the vapour initiates the discharge and plasma-expansion into the whole volume. The discharge current is limited at this stage by external resistors and the anode voltage.

Deposition rates in the range from 0.1 to 5.0 nm $s^{-1}$ can be obtained. High-energy bombardment by metal ions during the deposition ensures the formation of high density layers with bulk-like structure and good adhesion to the substrate. The discharge conditions and, therefore, the deposition process can be controlled by adjusting the filament heating current, anode voltage, anode–cathode distance and by the cathode tilt angle with respect to the anode vertical direction. With these parameters it is possible to change independently the ion flux and dose to the substrate. This is a great advantage of TVA in comparison to other film deposition technologies.

## 3. Be deposition process

The anode used for the Be film deposition was a Be cylinder placed in a tungsten holder. The upper part of the cylinder was melted and vaporised by the TVA electron gun. A bright blue colour plasma was ignited in pure Be vapour. The Be melting point is 1284 °C and a corresponding vapour pressure at this temperature is about 10 Pa. The necessary temperature for 100 Pa pressure of Be vapour is around 1550 °C. Therefore, the surface of the Be rod was locally heated up to this temperature while maintaining the lower part of the rod in the solid state. This was critical in order to obtain very pure Be films with practically no atoms coming from the crucible material, especially when using a tungsten plate support. In the TVA gun, the Be anode rod and plasma are shown in figure 3. Two kinds of Be anode cylinders were used: (i) one made from pressed flakes (NFF-Pitesti) and (ii) another made of pressed powder (Brush Wellman S65c). As proven by wavelength dispersive x-ray spectroscopy (WDS) the level of impurity species was small and almost the same for the two materials. The only difference was somewhat higher iron content in Be from NFF-Pitesti. This did not influence the coating quality thus indicating that either Be grade can be used for the production of markers.

During the Be evaporation, the intensity of the cathode filament was adjusted in the range of 40–60 A, while the applied ac voltage was in the range 15–24 V. This adjustment was necessary to keep a stable discharge. Optimal electrical parameters (i.e., voltage and current) of the discharge in Be, Ni and W vapours are given in table 1. It should be mentioned that this arc discharge cannot run without being sustained by the electron beam. Using the optimized parameters, deposition rates of $5 \pm 0.5$ nm $s^{-1}$ for Be, $1 \pm 0.5$ nm $s^{-1}$ for W and $2 \pm 0.5$ nm $s^{-1}$ for Ni were measured. To obtain good adherence of the layers, the substrate temperature was kept in the range from 350 to 400 °C for all materials. A specially designed heater with Kanthal resistive elements was used in close contact with samples in order to keep the substrate temperature constant during the whole deposition time. During the cooling phase the materials were still kept under vacuum ($10^{-3}$ Pa) in order to prevent the oxidation of the coating at elevated temperatures.

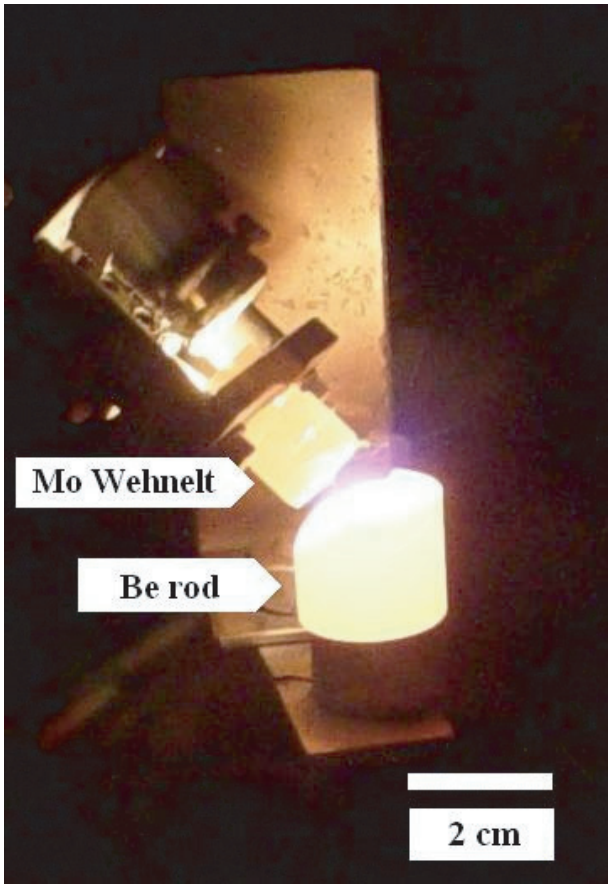


**Figure 3.** TVA gun and Be plasma.

**Table 1.** Electrical parameters of optimized discharges.

| Material | Discharge voltage | Discharge current |
|---|---|---|
| Be | $850 \pm 250$ V | $450 \pm 20$ mA |
| W | $1500 \pm 250$ V | $1000 \pm 100$ mA |
| Ni | $1200 \pm 200$ V | $800 \pm 100$ mA |

## 4. Coatings on small samples and their characterization

In the first step of the optimization process a number of small-size samples (15 mm × 15 mm) of stainless steel, graphite, glass, silicon wafer and Be discs (S65 grade from Brush Wellman, 20 mm in diameter, 3 mm thick) were coated by means of the TVA technique applying parameters described in section 3. The layer thickness was monitored *in situ* using a quartz crystal sensor. To measure the thickness greater than 1 $\mu$m (the upper limit for the quartz sensor) a special cooled holder was constructed. In front of the sensor window a disc with a 10 mm diameter hole was installed. An electrical motor working in vacuum rotated the disc (100 rpm) thus ensuring the difference by a factor of 30 between the real coating thickness and the thickness measured by the quartz monitor. After the deposition, the thickness of the layers was measured on the witness samples using a stylus profilometer. On the witness surface some steps were produced by masks or by chemical etching of a rib in the centre of the sample. The film thickness was $7.5 \pm 0.5$ $\mu$m for Be and $2.5 \pm 0.5$ $\mu$m in case of Ni layers.

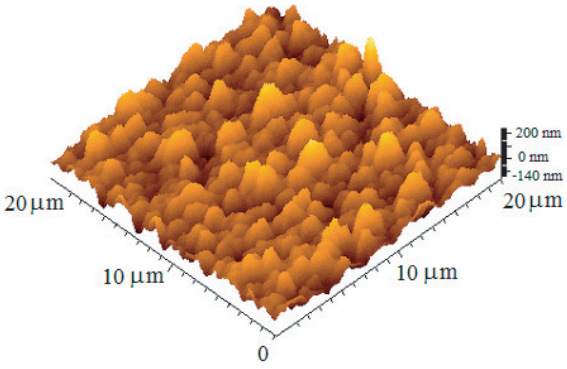


**Figure 4.** AFM image of Be coating on stainless steel.

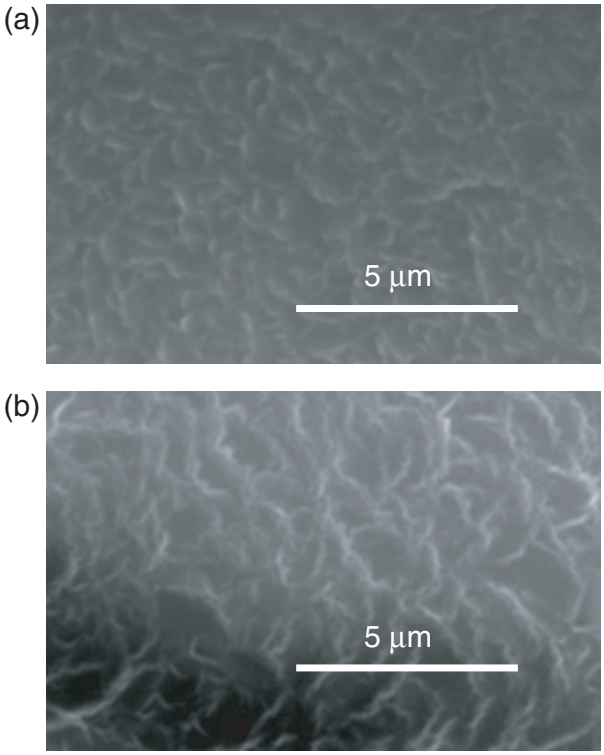


**Figure 5.** SEM images of Be coatings on stainless steel (a) and silicon (b).

A comprehensive characterization of the samples was performed using environmental scanning electron microscopy (ESEM, PHILIPS XL 30 TM), energy dispersive x-ray spectroscopy (EDX), x-ray diffraction (XRD), atomic force microscopy (AFM) and Auger electron spectroscopy (AES, PHI-Perkin Elmer 3017). The film adherence to the substrates was tested by means of an adhesive tape and a so-called pulling test. In the latter case, a 5 mm diameter rod was stuck on the Be films using a bonding resin and a pulling force was applied perpendicularly to the coating. Forces of up to 50 N (so-called detaching force) did not remove the Be films.

AFM measurements performed in a tapping mode have shown a peak-to-valley roughness of only $300 \pm 50$ nm, as shown in figure 4. Smooth and pinhole-free Be coatings on all substrate materials were observed using ESEM; the results are shown in figure 5. The crystalline structure of the deposited films has been confirmed by XRD analysis. The XRD pattern in figure 6 shows high intensity Be peaks in the coating deposited on a silicon wafer.

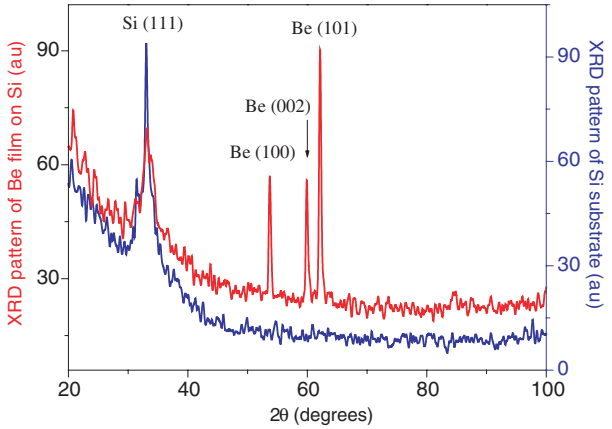


**Figure 6.** XRD pattern of the Be film deposited on a silicon wafer.

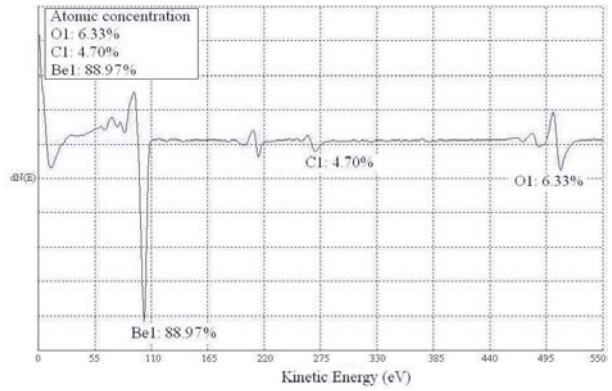


**Figure 7.** Auger spectrum of the Be film.

The surface composition analysed by AES (kinetic energy range: 0–3200 eV, resolution; 0.6%) has found a total of less than 12% of oxygen and carbon contaminants in the thin (3 nm) outermost layer. An Auger spectrum and O, C, Be concentrations are given in figure 7. The feature at around 215 eV corresponds to argon species implanted by the ion gun used for surface cleaning prior to the Auger measurement.

## 5. Manufacturing of optimized Be/heavy-metal/bulk-Be 'marker' coupons

Be coatings (7–9 $\mu$m) with a high-$Z$ metal interlayer (2–3 $\mu$m) on the bulk Be blocks were deposited on 3 cm thick Be blocks made of JET- and ITER-relevant grade (S65 grade from Brush Wellman). The thickness of these test coupons (3 cm) was similar to the thickness of real tiles being manufactured for the ILW. The similar thickness of test coupons and real tiles is essential to ensure that high-heat flux testing of the coupons will be relevant. The heat flux test will be the next step in the marker qualification process.

When optimizing the interlayer composition Re, W, Ni and Re–Ni–Cr compound films were prepared by using the same deposition technique as for Be (i.e., TVA). In the final selection of the interlayer composition, thermo-mechanical properties of respective elements and the formation of inter-metallic compounds (e.g., W–Be alloying [5, 6]) have been taken into account. Due to the best match of the thermal expansion coefficients ($\alpha$) of Be and nickel, the latter metal was chosen for the intermediate layer: $\alpha_{Ni} = 13.1 \times 10^{-6}\,K^{-1}$, $\alpha_{Be} = 11.5$–$16.5 \times 10^{-6}\,K^{-1}$ dependent on temperature: $11.5 \times 10^{-6}\,K^{-1}$, $14.5 \times 10^{-6}\,K^{-1}$ and $16.5 \times 10^{-6}\,K^{-1}$ at 293, 533 and 773 K, respectively. For other metals the coefficients are distinctly lower ($\alpha_W = 4.4 \times 10^{-6}\,K^{-1}$, $\alpha_{Re} = 6.2 \times 10^{-6}\,K^{-1}$, $\alpha_{Cr} = 6.2 \times 10^{-6}\,K^{-1}$) creating a risk of film detachment due to stresses under high thermal loads from the plasma.

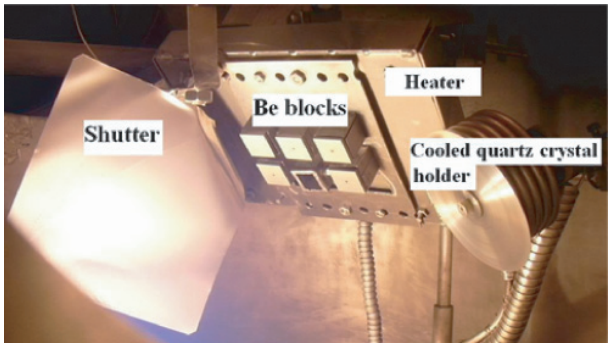


**Figure 8.** Experimental set-up for coating of Be blocks. This photograph was taken when depositing nickel layers.

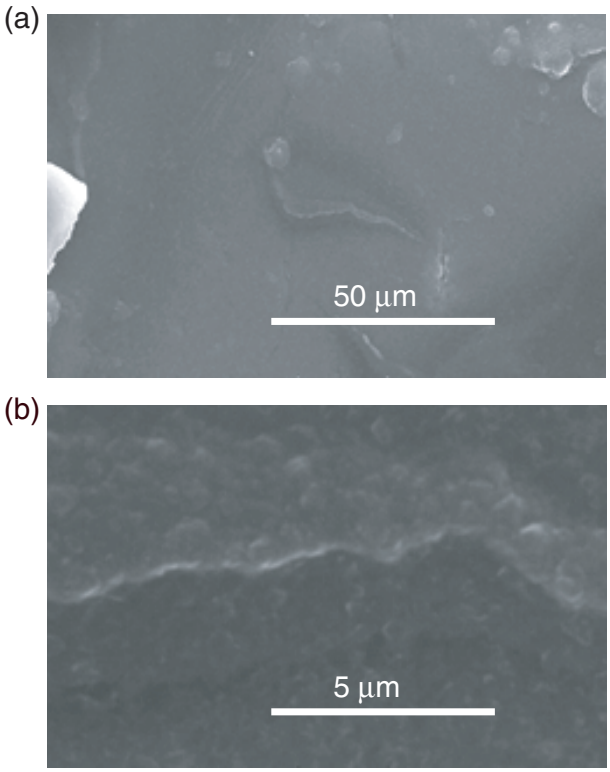


**Figure 9.** ESEM images of the Ni films deposited as interlayer on the bulk Be blocks.

Figure 8 shows the experimental set-up for coating of the Be blocks. The holder of the blocks was in close contact with an electrical heater, capable of heating the samples to 400 °C. A shutter protected the surface of the samples against impurities at the beginning of the process. The film thickness was monitored *in situ* by the quartz sensor and was also compared after deposition with the thickness measured using a stylus profilometer. SEM images in figure 9 show the topography of the Ni interlayer.

Using the Be-compatible TVA facility a number of test Be coupons previously coated with the 2–3 $\mu$m Ni interlayer

**Figure 10.** Be block coated with Ni ($2.5 \pm 0.5\,\mu$m) and Be ($7.5 \pm 0.5\,\mu$m).

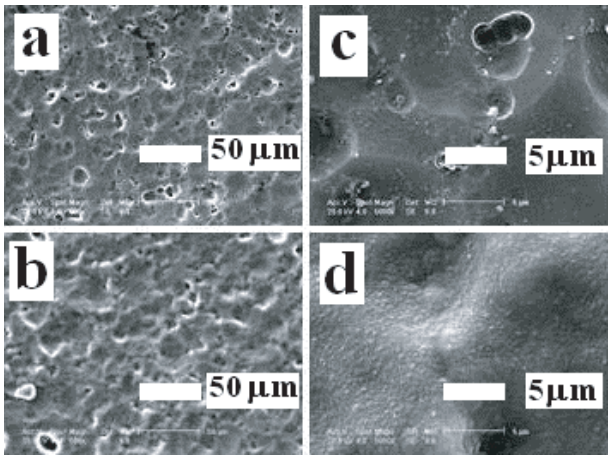


**Figure 11.** Surface morphology of a bulk Be block before and after coating with Ni and Be.

were coated over with $7.5 \pm 0.5\,\mu$m film of pure, bulk-like dense Be. Figure 10 shows the coupon coated with Ni and Be. The batch for coating was composed of four bulk Be blocks already coated with a Ni interlayer. Two locations were reserved for witness samples: stainless steel and glass substrates of the same size as the bulk Be blocks. After every deposition process, witness samples were tested for adhesion and thickness. Before and after the deposition, the Be blocks were analyzed by ESEM. Images recorded at two different magnifications are shown in figure 11. It may be noted that the surface topography of the film looks smoother than the initial surface of the block. The film was analyzed by atomic absorption spectroscopy, which showed that the impurity (oxygen, carbon) content in Be was below 0.2%.

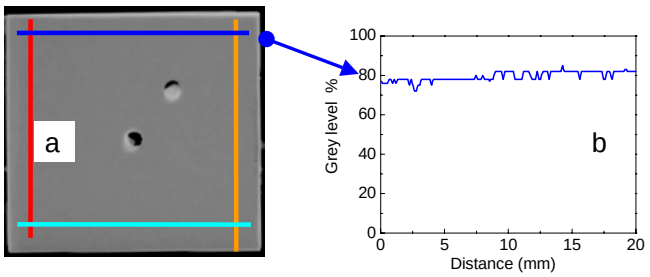


**Figure 12.** Micro-radiography of a bulk Be block. The density distribution of the layer in the indicated direction is shown.

The uniformity of the deposited films was checked by means of micro-radiotomography using a tomograph developed at NILPRP [7]. The analysis was performed under following the conditions: micro-focus x-ray source with $3\,\mu$m focus size, $U = 100\,$kV; $I = 75\,\mu$A, Cd pre-filter, a-Si flat panel detector, $100\,\mu$m pixel size. Figure 12(a) shows an image acquired by the micro-tomograph and the direction in which the density analysis was performed. The image on the right side (figure 12(b)) shows the density distribution along the direction indicated by an arrow. The results have proven high uniformity of the deposited layers.

## 6. Concluding remarks

TVA deposition technology has been successfully applied to manufacture test coupons of marker tiles. Based on testing of several different high-$Z$ metal/Be combinations nickel has been selected for the interlayer of the markers. As proven by several analysis techniques, the thin layers of nickel and Be are uniform and of high purity and good adhesion to the underlying Be substrate. The experience gathered during the R&D phase will be used in production of full size marker tiles. High heat flux-testing, the next step in the overall qualification process, will allow the determination of the durability of markers under high power loads under plasma-impact.

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