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Subsequent Titanium Coating of RF Input Coupler Windows

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INTRODUCTION

In 2014 after four years of interruption the refurbishing and conditioning of RF input couplers (Fig.1) for PETRA III cavities were started again. Refurbishing means glass bead blasting, etching, cleaning and baking-out at 200 °C and 10^{-6} mbar vacuum pressure. Conditioning means processing at 500 MHz RF power up to 300 kW. The couplers to be refurbished had been in operation at PETRA II, HERA or DORIS III before.

Unexpectedly, the success of RF conditioning was very poor. The former success rate could not be reproduced. Only 13 out of 28 conditioning processes were successfully completed (46 %). In contrast, 31 out of 34 couplers had been successfully conditioned in the period between 2006 and 2010 (91%). The search for the causes proved difficult. All variations of the refurbishment process were not as successful as expected. Although after the 19th attempt more couplers reached the minimum target of 250 kW transferred RF power with a maximum window loss of 0.2%, most showed bluish glow effects on their window surface. Due to the assumption that multipacting is the cause of the bluish glowing the idea grew to coat a coupler window with a thin titanium layer. Because the ceramic window is brazed into the coupler construction, a way had to be found to coat the ceramic window in-situ with titanium.

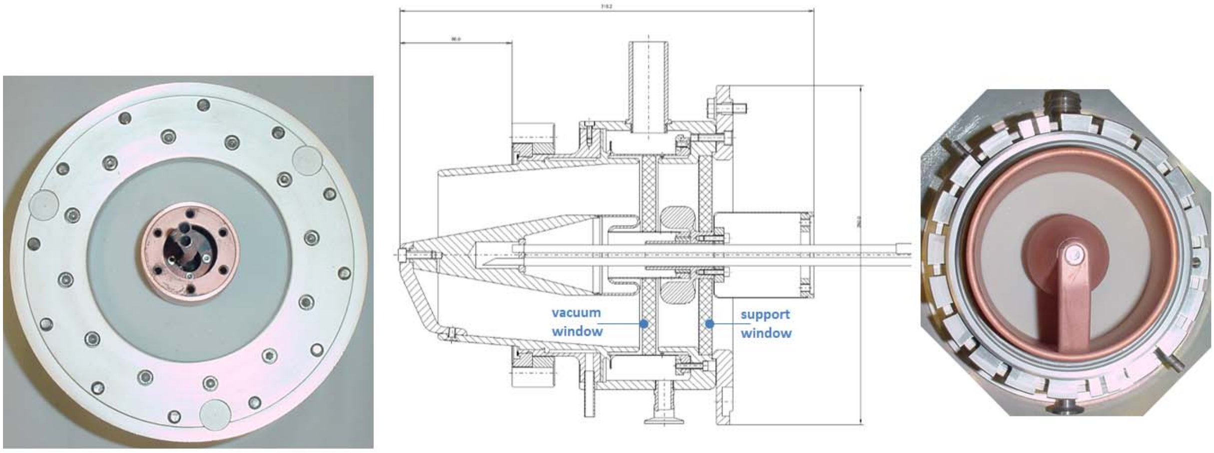


Fig. 1: RF input coupler type PETRA. **Left:** Air-side support window. **Center:** cut-view of the coupler. **Right:** vacuum-side of the coupler with coupling loop and vacuum window

EVAPORATION SET-UP

Readily available components should be used to demonstrate the basic feasibility. A used VARIAN Ti sublimation cartridge removed from a vacuum pump was found in a scrap box (Fig. 2). As recipient a suitable vacuum cross chamber was found in a shelf of the vacuum group. Together with some UHV flanges, a RF input coupler evaporation apparatus was assembled (Fig. 3). In Fig. 4 a draft of the evaporation apparatus is shown.

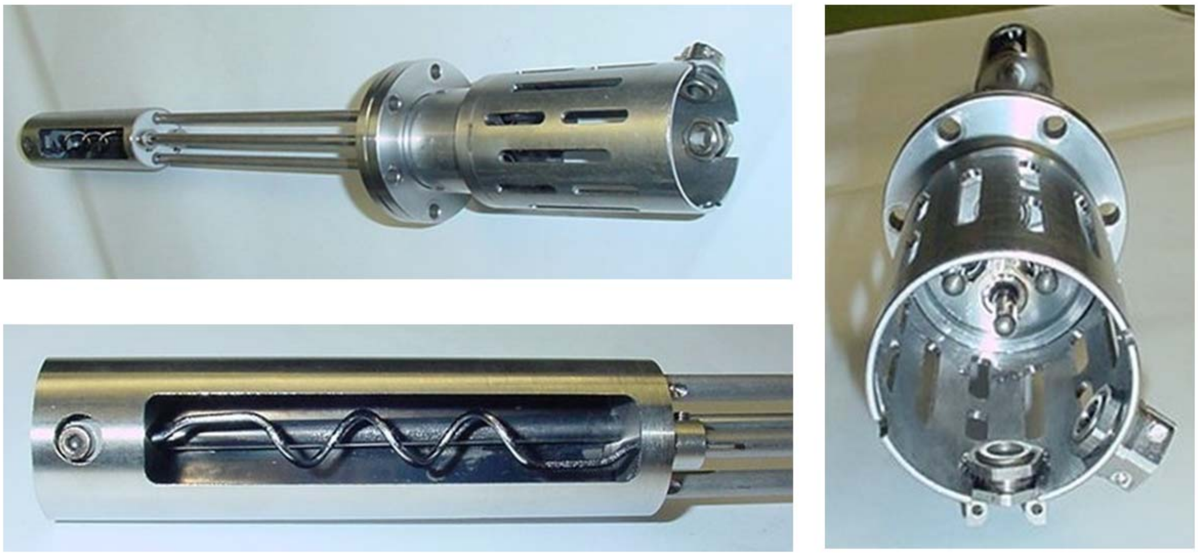


Fig. 2: VARIAN Ti sublimation cartridge (removed from a vacuum pump)

Fig. 3: Vacuum cross-chamber with the VARIAN Ti Sublimation Cartridge inserted

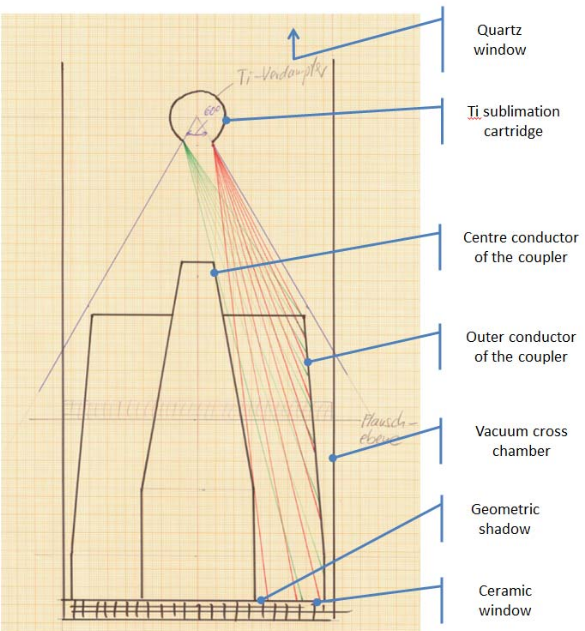


Fig. 4: Draft of evaporation set-up.

The green and red traces of two virtual titanium source points try to give a rough impression of the Ti vapor density (Fig. 4). Unfortunately, more titanium would reach the inside of the outer conductor than the ceramic to be coated. In addition, the window area close to the center conductor is in shadow. With some extra time and effort this problems could have been solved. But we decided first to try out the given geometry. In a next step suitable sublimation parameters (filament current over time) had to be explored. First, the required sublimation rate and time for 10 titanium atomic layers were estimated on the basis of a given sublimation rate curve (Fig. 5).

ESTIMATION OF THE LAYER THICKNESS

The thickness of the Ti coating should be as thin as possible, because a thick layer will increase the window loss or even will reflect the incident RF power. In order to suppress multipacting a thickness of 10 to 100 atomic layers should be sufficient. The sublimation rate of the VARIAN Ti sublimation cartridge is specified as shown in Fig. 5.

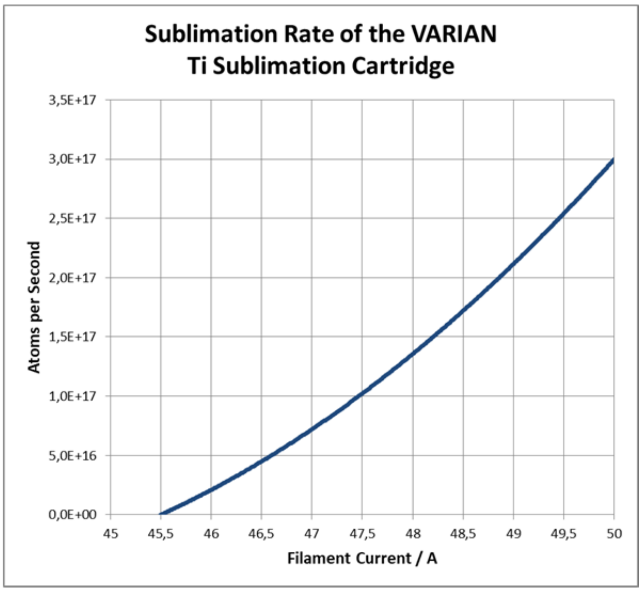


Fig. 5: Sublimation rate versus filament current

A Sublimation rate of $3 \cdot 10^{17}\, {atoms}/{s}$ or $18 \cdot 10^{18}\, {atoms}/{min}$ is obtained for a filament current of 50 A. But only about 1/6 of the sublimated atoms can leave the cartridge case because of the slotted jacket of the filament (Fig. 2). Another limitation was given by the geometrical arrangement shown in Fig. 4. It was estimated that only about 1/4 of the atoms escaped from the cartridge will reach the DUT (device under test). Thus, the number of atoms reaching the DUT per minute is

$$\frac{N_{a_{eff}}}{t} = 18 \cdot 10^{18}\frac{atoms}{min} \cdot \frac{1}{6} \cdot \frac{1}{4} = 7.5\ \cdot 10^{17}\frac{atoms}{min}$$

The size of the surface to be coated is $17400\ mm^2$. The diameter of a titanium atom is $294\ pm$.
The number of atoms required for one atomic layer is

$$N_{a_{layer}} = \frac{17{,}4 \cdot 10^{-3}\ m^2 \cdot 4}{\pi(294 \cdot 10^{-12}\ m)^2} = 2.6 \cdot 10^{17}$$

Thus, the sublimation time for one atomic layer is

$$T_{evap} = {N_{a_{layer}}}\Big/{\frac{N_{a_{eff}}}{t}} = \frac{2.6 \cdot 10^{17}}{7.5\ \cdot 10^{17}\frac{atoms}{min}} = 0.34\ min$$

This result should be understood as a guideline based only on rough estimates. It means that in a first experiment the sublimation time for about 10 atomic layers should be in the range of a few minutes.

PILOT TESTS TO DETERMINE THE SUBLIMATION TIME

For the first evaporation tests an air-side support window of the same size and made of the same material as the vacuum window was used as DUT. The air-side support window is only clamped to the coupler and not brazed as the vacuum window (see Fig. 1, center). This means that it can be easily removed for coating and reinstalled for subsequent RF power loss measurements. Fig. 6 shows filament current and vacuum pressure versus sublimation time of the pilot tests. The results are shown in Tab. 1.

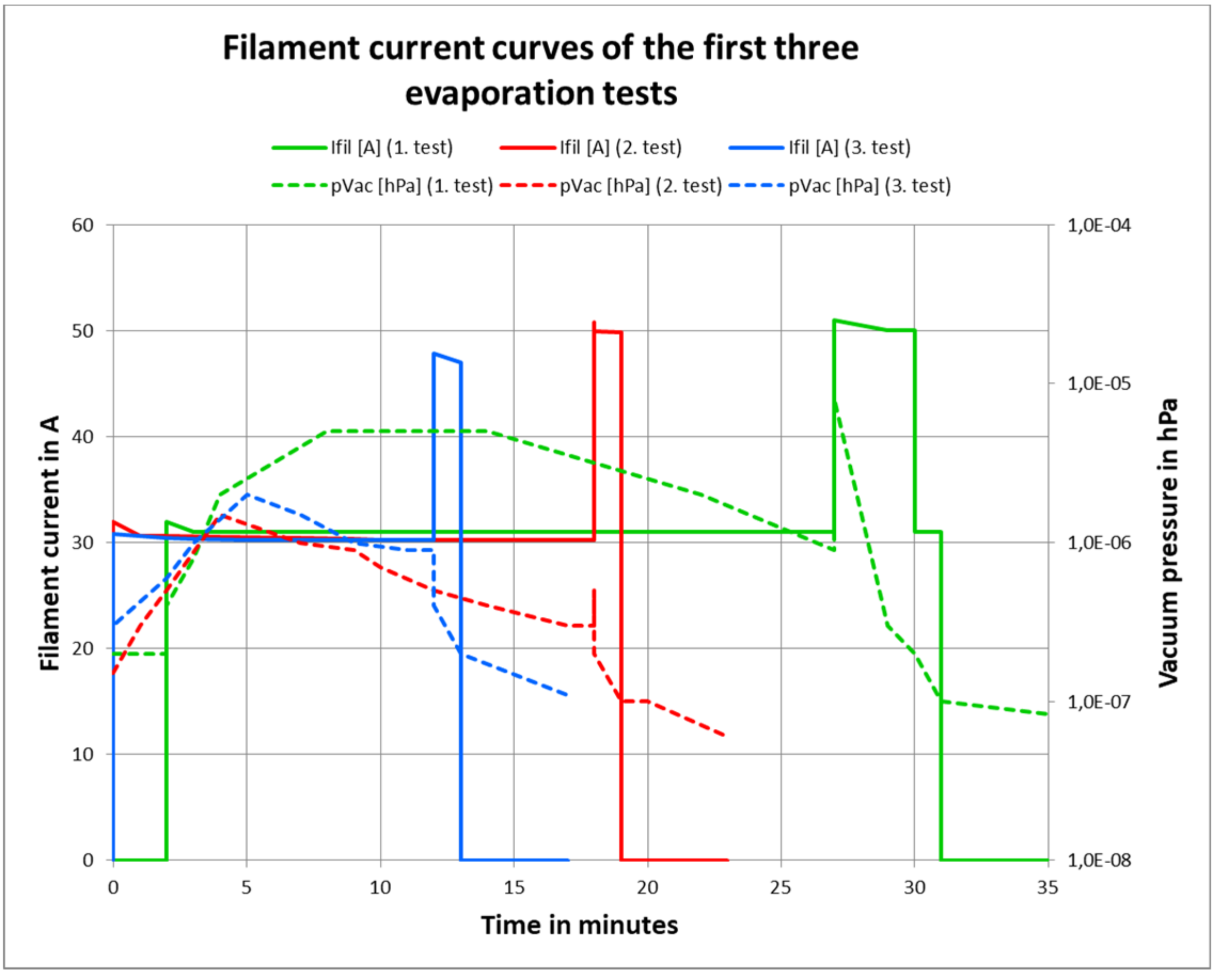


Fig. 6: Filament current and vacuum pressure versus sublimation time of the pilot tests

Control of the thickness of titanium deposition by means of resistance measurement

The thickness of the titanium deposition was controlled by resistance measurements. For this purpose, the circumference of the DUT was electrically contacted by the outer conductor connection flange of a coupler and the center by a brass ring (Fig. 7).

Tab. 1: Measured resistances of the evaporated Ti layers

Evaporation test	**1st**	**2nd**	**3rd**
Degassing	27 min @ 31 A	17 min @ 30 A	15 min @ 30 A
Sublimation	3 min @ 50 A	1 min @ 50 A	1 min @ 47.5 A
Measured resistance	300 Ω	5.6 kΩ	150 kΩ

The measurement results are shown in tabular form in Tab. 1 and as a diagram in Fig. 8.
The power function fitted to the measured values is

$$\frac{R}{k\Omega} = \frac{10^7}{\left(N_a/10^{17}\right)^e}$$

Where R is the surface resistance and N_a is the number of sublimated atoms.

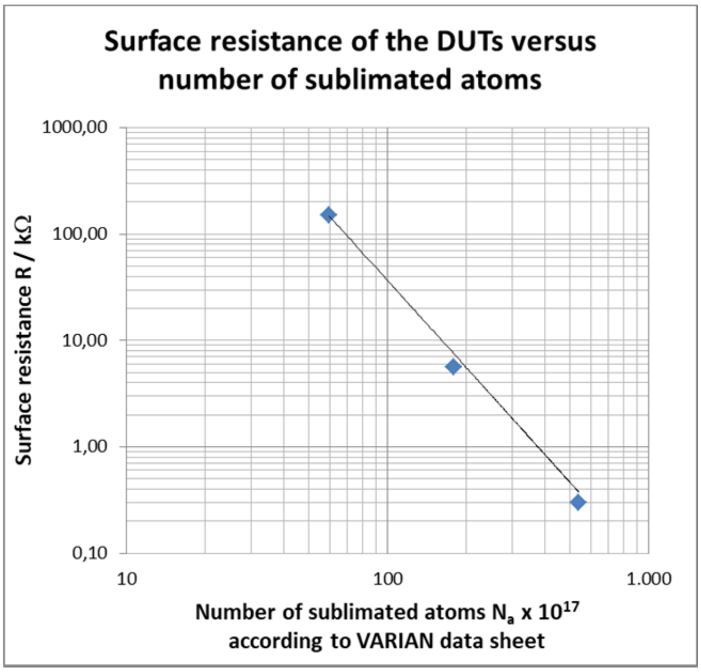


Fig. 8: Measured surface resistances of Tab. 1 versus sublimation rate (Fig.5) times sublimation time

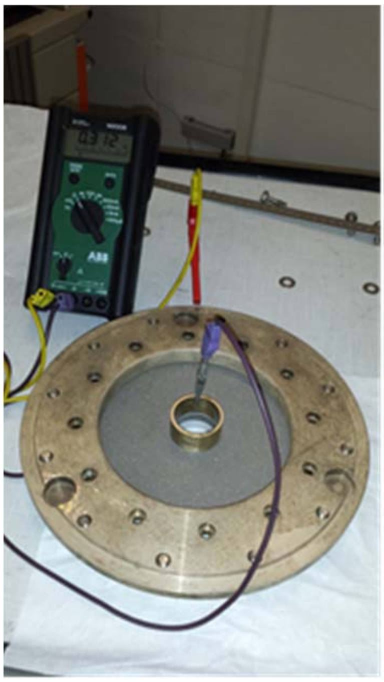


Fig. 7: Control of the layer thickness of a DUT by means of resistance measurement

When trying to calculate the thickness of the evaporated Ti layer δ using the formula for a disc-shaped coaxial resistor

$$\delta = \frac{\rho_{Ti}}{2\pi R} \cdot ln\left(\frac{D_a}{d_i}\right),$$

where ρ_{Ti} is the specific resistance of titanium, R is the resistance measured according to Fig. 7, D_a is the outer diameter of the RF window and d_i is the diameter of the hole for the center conductor, the results are incredible. For example, the measured resistance of $R = 300\ \Omega$ gives a thickness of the titanium deposition of $\delta = 330\ pm$. This corresponds approximately to the diameter of one titanium atom and is incredible. For higher R values the formula even gives fractions of an atomic diameter. A possible explanation for the non-credible results could be that the formula used is not valid on an atomic scale and R is primarily determined by the macroscopic surface roughness and less by the thickness of the Ti layer.

Power test of the titanium coated windows

The titanium coated window from the 1st evaporation test was not tested at high power because it seemed to make no sense due to the high conductivity of the thick Ti layer. The titanium coated window from the 2nd evaporation test was installed as an air-side support window in the input coupler of our coupler test-cavity. The window power dissipation of this input coupler was previously measured with uncoated windows. The comparison with the coated window should now show what effect the titanium layer has on the window losses. Unfortunately, the coated window of the 2nd evaporation test already failed before the first measurement could be carried out. Shortly before reaching the 50 kW measuring point a clear clicking sound was heard close to the coupler. The power test was interrupted to find out the cause. It turned out that the DUT was broken (see Fig. 9 and 10).

Fig. 9: On the untreated side of the window hairline cracks were visible.

Fig. 10: On the coated side of the window, a tiny, millimeter-sized piece was broken out.

The power test of the 3rd evaporation test was successful. The titanium thickness which corresponds to a surface resistance of R=150 kΩ obviously did not increase the window losses (see Tab. 2).

Tab. 2: Comparison of the power losses of an uncoated and a one-sided Ti-coated support window. The outlet temperature rise of the window cooling air was measured as an indicator of the power loss.

RF coupler input power	Uncoated windows	2nd evaporation test, R=5.6 kΩ	3rd evaporation test, R=150 kΩ
0	**27.2 °C**	**27.2 °C**	**28.2 °C**
50 kW	33.2 °C	>44.3 °C, window broken	33.8 °C
100 kW	39.5 °C		39.1 °C
135 kW	42.8 °C		42.0 °C

The next step was to clarify whether this layer thickness is sufficient to suppress the observed bluish glow effects. For this purpose the vacuum window of a complete coupler was coated in a 4th evaporation attempt.

TITANIUM COATING OF THE 1ST RF INPUT COUPLER

The coupler S/N 014-82 was chosen for the first coupler coating attempt. This coupler was removed after the final shutdown of DORIS from the DORIS Cavity 4.1. Two previous conditioning processes had to be prematurely terminated due to intense bluish window glowing (see Fig. 11). Therefore, this coupler was likely a good candidate to demonstrate the beneficial effect of titanium coating.

Fig. 11: Coupler S/N 014-82 before titanium coating at 190 kW RF power and $5*10^{-8}$ hPa vacuum pressure

In a 4th evaporation attempt (see Fig. 12) the vacuum window of the first coupler S/N 014-82 was coated with the sublimation parameters of the 3rd evaporation test (see Tab. 1).

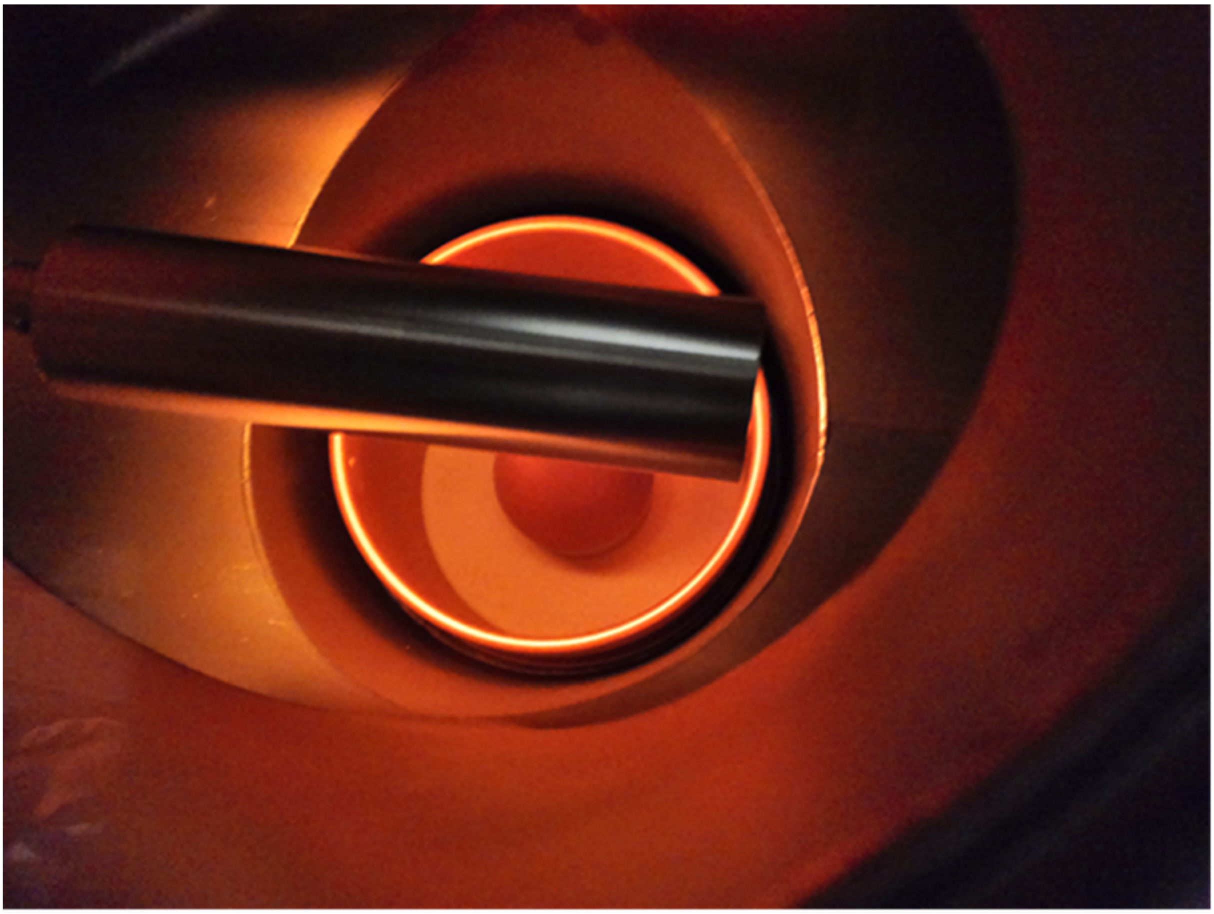
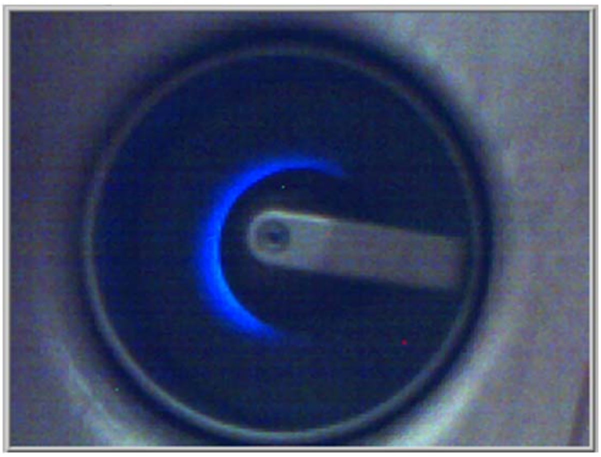
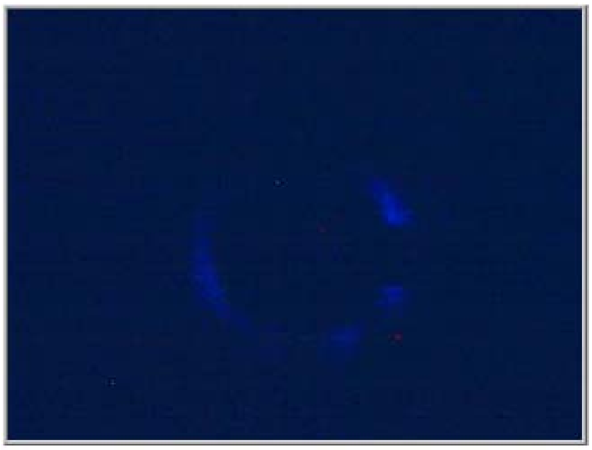


Fig. 12: 4th evaporation attempt. Coating the vacuum window of the first coupler (S/N 014-82). The photo was taken through the quartz window during the degassing phase (for orientation, see Fig. 4)

The following day the coupler was removed from the evaporation apparatus and was installed immediately into the test-cavity. When a vacuum pressure of 10^{-6} mbar was reached a heating was activated in order to bake out the test-cavity at about 70 °C for 17 hours. After the heating was turned off and the test-cavity cooled down, the RF conditioning was started. The conditioning process went smoothly. During the conditioning, a bluish glow was observed, but significantly weaker than before without Ti coating when it glowed so bright that the camera was overdriven. At the end of the conditioning process only a weak glow could be seen at 250 kW RF power (see Fig. 13).



Bluish glow of the titanium coated coupler window at the beginning of the conditioning process at 50 kW RF power and 10^{-7} hPa vacuum pressure



Bluish glow of the titanium coated coupler window at the end of the conditioning process at 250 kW RF power and $3*10^{-8}$ hPa vacuum pressure

Fig. 13: Bluish glow of the first titanium coated coupler S/N 014-82 during the conditioning process

INTERMEDIATE RESULTS

Titanium coating of a PETRA-type cavity input coupler in a set-up as shown in Fig. 3 and Fig. 4 with sublimation parameters as shown in Tab.1, 3rd evaporation test, result a Ti layer of about 150 kΩ surface resistance, capable of mitigate but not completely suppress bluish window glow. For further evaporation attempts a Ti layer thickness corresponding to 50 kΩ surface resistance was aimed.

TITANIUM COATING OF THE RF INPUT COUPLER S/N 65-83

Coupler history

Selected was the RF input coupler S/N 65-83. This coupler was dismounted on 28.8.2014 from the HERA cavity NL-Cy 1.5 where it had been in operation at least since 1995. After refurbishing (glass-bead blasting, etching and baking) it was installed into the coupler test-cavity on 8.9.2014. On 12.9.2014 after 24 h conditioning time the coupler reached 273 kW transmitted power. But it showed bluish window glow with changing intensities over the whole conditioning process.

Additional pilot tests using air-side support windows

Due to the use of a new Ti sublimation cartridge, the sublimation parameters had to be checked first. The filament resistance of the new cartridge was about 10 % lower compared to the previously used and it accordingly drew more current. The first test with the new filament was carried out with one minute sublimation time and 55.5 A filament current (5th evaporation test). The resulting surface resistance of the DUT was 35 kΩ, which was significantly lower than the target value of 50 kΩ. For the next test (6th evaporation test) the sublimation time was reduced to 30 seconds but then the resulting surface resistance of the DUT was immeasurably high.

Tab. 3: Measured resistances of the deposited Ti layers with a new Ti sublimation cartridge

Evaporation test	**5th**	**6th**
Degassing	15 min @ 36 A	15 min @ 35.5 A
Sublimation	1 min @ 55.5 A	30 s @ 55.5 A
Measured resistance	35 kΩ	not measurable

Instead of carrying out further tests on air-side support windows, coupler S/N 65-83 was coated in the 7th attempt and 45 seconds was chosen as the sublimation time.

Titanium coating

On 10.11.2014 the coupler was dismounted from the coupler test-cavity, Ti coated and remounted one day later. The result is shown in Fig. 14. The Ti deposition is visibly uneven. The deposition was thinnest below the sublimation cartridge next to the centre conductor.

Fig. 14: 7th evaporation attempt. Vacuum window of the coupler S/N 65-83 after Ti coating. The photo was taken through the quartz window after 45 seconds sublimation time at 55.5 A filament current

Power test of the Ti coated coupler

The conditioning process showed no irregularities and took only 4 hours. In the past the typical conditioning time of those (uncoated) couplers was 26 hours on average. The coupler showed a slight bluish glowing during the conditioning process at 230 kW RF power and $1.1 \cdot 10^{-8}$ hPa vacuum pressure (see Fig. 16). At 280 kW RF power and $5 \cdot 10^{-9}$ hPa the glowing disappeared. An increase in window losses due to Ti coating was not measured.

Fig. 15: Slight bluish glow of the Ti coated coupler S/N 65-83 during the conditioning process at 230 kW RF power and $1.1 \cdot 10^{-8}$ hPa vacuum pressure

Tab. 4: Test data of the coupler S/N 65-83 before and after Ti coating compared to the average of 39 coupler-tests carried out since August 2006

Coupler S/N	**average of 39 coupler-tests**	**65-83**	**65-83 (Ti)**
Ti coating	w/o	w/o	Degassing: 15 min @ 35.5 A Sublimation: 45 s @ 55,5 A
Date of test	2006-2016	12.9.2014	24.11.2014
Conditioning time	26 h	24 h	4 h
Test frequency	498.6 to 501.27 MHz	501.26 MHz	501.26 MHz
Test power	292 kW	291 kW	285 kW
Transmitted power	289 kW	273 kW	282 kW
Reflected power	10 kW	18 kW	3 kW
Window power loss	312 W	311 W	277 W
Test-cavity vacuum		$2.4 \cdot 10^{-8}$ hPa	$5 \cdot 10^{-9}$ hPa

TITANIUM COATING OF THE RF INPUT COUPLER S/N X17

Coupler history

The coupler S/N X17 was dismounted on 28.8.2014 from the HERA cavity NL-Cy 2.6 where it had been in operation at least since 1995. After refurbishing (glass-bead blasting, etching and baking) it was installed on the coupler test-cavity on 29.8.2014. On 1.9.2014 the coupler reached 268 kW transmitted power after 16 h conditioning time. It showed a bluish window glow with changing intensities over the whole conditioning process. At some power levels during conditioning the glowing disappeared.

Additional pilot tests using air-side support windows

The coupler S/N X17 should be Ti coated in two steps. For the second coating step the cartridge should be rotated 90 ° with respect to the coupler in order to achieve a more uniform coating. Therefore another four pilot tests were carried out. The variation of the sublimation parameters and their results are shown in Tab. 5.

Tab. 5: Resistances of the evaporated Ti layers measured with additional pilot tests

Evaporation test	**8th**	**9th**	**10th**	**11th**
Degassing	15 min @ 34 A	10 min @ 34 A	10 min @ 32 A	10 min @ 31 A
Sublimation	1 min @ 51 A	2 min @ 51 A	2 min @ 48.5 A	2 min @ 50 A
Measured resistance	not measurable	59 kΩ	not measurable	110 kΩ
	DUT rotated by 90 °	No 2nd layer because resistance is already to low	DUT rotated by 90 °	No 2nd layer because resistance is in range
Degassing	10 min @ 34 A		10 min @ 32 A	
Sublimation	1 min @ 51 A		2 min @ 48.5 A	
Measured resistance	not measurable		4.1 MΩ	

The evaporation results of Tab. 5 show that the choosing the best parameters is difficult, especially in comparison with the results from the first series of tests shown in Tab. 1. The results obtained using a used VARIAN Ti sublimation cartridge are vastly different from those with a new one. The main reason is the highly dependence of the sublimation rate on the filament current. A 5 % higher filament current triples the sublimation rate (Fig. 5). Additionally the old filament had a significantly higher resistance than the new one and accordingly required less current for the same filament temperature. Actually, a few more pilot tests should have been carried out but time was running out. Therefore we decided to coat the coupler S/N X17 using the sublimation parameters of the 11th evaporation test.

Titanium coating

On 8.9.2014 the coupler was dismounted from the coupler test-cavity and was stored under vacuum. On 19.11.2014 it was Ti coated and one day later remounted into the coupler test-cavity. The coupler was evaporated twice with the parameters of the 11th evaporation test. After the first evaporation it was turned by 90 ° in order to achieve a more uniform coating.

Power test of the Ti coated coupler

The conditioning process took only 4 hours and was finished without any unusual event. The coupler didn't show any glowing during the condition process. Its window loss almost doubled due to the Ti coating but it was still in the normal range of approx. one watt power loss per kilowatt transmitted RF power.

Tab. 6: Test data of coupler S/N X17 before and after Ti coating compared to the average of 39 successful coupler-tests carried out since August 2006

Coupler S/N	**average of 39 coupler-tests**	**X17**	**X17 (Ti)**
Evaporation	w/o	w/o	Degassing: 10 min @ 31 A Sublimation: 2 orthogonal layers 2 min @ 50 A
Date of test	2006-2016	1.9.2014	21.11.2014
Conditioning time	26 h	24 h	4 h
Test frequency	498.6 to 501.27 MHz	501.23 MHz	501.28 MHz
Test power	292 kW	289 kW	285 kW
Transmitted power	289 kW	268 kW	282 kW
Reflected power	10 kW	21 kW	3 kW
Window power loss	312 W	194 W	368 W
Test-cavity vacuum		$4*10^{-8}$ hPa	$5*10^{-9}$ hPa

EXPERIENCE WITH THE TITANIUM COATED COUPLERS IN THE PETRA III MACHINE

On 20.01.2015 the RF input coupler S/N 65-83 was installed into PETRA cavity SL-4 and the RF input coupler S/N X17 was installed to PETRA cavity SL-3. The conditioning of the cavity section vented to install the Ti coated couplers began three days later. During the conditioning of the cavity section, only those with the newly installed Ti coated couplers arced. Cavity SL-3 arced 4 times and cavity SL-4 arced 6 times during 6 days. But these arcs were most likely no coupler arcs but arcs between the cavity-gaps. The vacuum pressure was about $5 \cdot 10^{-8}\ hPa$ at this time. Before the cavity section for the coupler exchange was vented the vacuum pressure was 10 times lower. During the rest of the year of PETRA III operation the cavities SL-3 and SL-4 showed about the same arcing rate than the other cavities of the vented section. Real coupler arcs were observed only once in the cavity SL-3 (coupler S/N X17) and cavity SL-4 (coupler S/N 65-83). For comparison: the untouched cavity SL-6 coupler arced six times during this time. Since October 2015, no further coupler arc has been registered for both Ti coated couplers.

CONCLUSION OF THE EXPERIMENT

RF cavity input coupler windows of the PETRA type normally do not need to be Ti coated. In the case of that couplers cannot be RF conditioned despite a careful mechanical and chemical treatment procedure, subsequent Ti coating may be helpful. Such subsequent Ti coating of coupler windows can be carried out very easily without special knowledge and with widely available components. Pilot tests are required to achieve an optimal layer thickness, since the sublimation rates of identical Ti sublimation cartridges can be very different. The layer thickness can be controlled by measuring the surface resistance of the evaporated Ti layer. A resistance of approx. 100 kΩ per evaporation step seems to be optimal for the ceramic window of a PETRA-type coupler. For the second evaporation step the cartridge must be rotated 90 ° with respect to the coupler in order to achieve a more uniform evaporation. The very short required conditioning time of only 4 hours after the Ti coating surprised. It is not clear whether the Ti layer acted as an additional ion pump and did speed up the conditioning process due to the lower pressure, or whether it is solely the effect of secondary electron suppression. The long-time experience in the operation of two Ti coated couplers in PETRA III shows that a subsequent Ti coating as described is not harmful but can reliably suppress bluish glowing effects (multipacting assumed).

In Tab. 7 the test data of couplers before and after Ti coating compared to the average of 39 successful coupler-tests carried out since August 2006 are shown. With coupler 65-83, a higher window power loss was measured before Ti coating than afterwards. In fact, the additional losses due to the thin single titanium layer are too small to show a measurable effect. The higher power loss before the Ti coating is probably due to the higher reflected power. With coupler X17, a higher window power loss was measured after Ti coating as expected. The additional losses due to the double titanium layer show a clearly measurable effect.

Tab. 7: Test data of couplers before and after Ti coating compared to the average of 39 successful coupler-tests carried out since August 2006

Coupler S/N	**average of 39 coupler-tests**	**X17**	**X17 (Ti)**	**65-83**	**65-83 (Ti)**
Evaporation		w/o	2 orthogonal layers 2 min @ 50 A	w/o	one layer 45 s @ 55,5 A
Date of test	2006-2016	1.9.2014	21.11.2014	12.9.2014	24.11.2014
Conditioning time	26 h	24 h	4 h	24 h	4 h
Test frequency	498.6 MHz to 501.27 MHz	501.23 MHz	501.23 MHz	501.26 MHz	501.26 MHz
Test power	292 kW	289 kW	285 kW	291 kW	284 kW
Transmitted power	289 kW	268 kW	282 kW	273 kW	281 kW
Reflected power	10 kW	21 kW	3 kW	18 kW	3 kW
Window power loss	312 W	194 W	368 W	311 W	277 W
Test-cavity vacuum		$4*10^{-8}$ hPa	$5*10^{-9}$ hPa	$2.4*10^{-8}$ hPa	$5*10^{-9}$ hPa
Window power loss @ PETRA III @100mA, 40 bunches	190 W	n/a	250 W	n/a	170 W