NEG thin film coatings: from the origin to the next-generation synchrotron-light sources

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Outline

1. Historical perspective
2. TiZrV thin film coating for the LHC
3. MAX IV
4. Future developments
From NEG strips and SC-RF cavities to NEG thin-film coatings

- Cris Benvenuti first technical note about linear pumping by NEG strips in **June 1977**.
- At that time, the study was not directly motivated by the LEP.
- At that time, linear ion pumps had been used.

- Cris Benvenuti and the very first Nb coating team in 1983.
- The study was triggered by Emilio Picasso, the former leader of the SC cavities development then LEP project leader.
- At that time only bulk Nb was considered for SC-RF cavities.
NEG linear pumping and Nb coated SC-RF cavities: two technological achievements for the LEP

First prototype of the LEP dipole beam pipe with the St101 NEG strip in the antechamber (1983).

Installation of a LEP2 cryomodule with Nb coated RF cavities (1995)
The merging: first indirect trials

Cris Benvenuti, private communication:

- Cris had the first idea of coating the inner wall of vacuum chamber by NEG materials in the early Eighties.
- He tried to sputter a St-101 strip in a LEP chamber (Al alloy AA 6060).
- The ultimate pressure after sputtering was excellent, but the re-activation impossible.

Cris Benvenuti et al. LEP2-Note-94-21
Copper coatings for the main couplers of the LEP2 superconducting RF cavities

Jordi Fraxedas (EU fellow) performed ESD measurements of Cu and Ti coated stainless-steel RF coupler extensions. For Ti coating, he measured an abrupt decrease of the desorption yields after heating at 350°C (2 hours).

This measurement was not considered as unexpected since the ‘activation’ of Ti bulk was already known from previous experience in the ISR. The ‘activation’ had been recorded at 350°C (24h) for Ti experimental vacuum chambers.
Intertwisted Ti and Zr elemental wires

It became clear that NEG thin-film coatings would have been used in the LHC only if materials with lower activation temperature could be found.

Stainless steel $\leq 350^\circ$C
Copper alloys $\leq 250^\circ$C
Aluminium alloys $\leq 200^\circ$C
Beryllium $\leq 200^\circ$C

In the 1962 edition of the Lafferty, it is written that Ti-Zr alloys are known to activate after heating at $300^\circ$C. It was a common practice in vacuum technology.

We looked for wires or rods made of Ti-Zr alloys.

January 1996: faster activation with Ti and Zr, one wire each

ESD
1 mA
500 V
December 1997: sometime wrong ideas give great results, TiZrV

01-1996: Ti-Zr  
09-1997 Pd/TiZr  
12-1997 TiZrV

During an informal meeting (V. Rouzinov, J-M. Cazeneuve, F. Cicoira, and myself) we decided to add a wire of an element of the 5th group in Ti-Zr braid. If positive, the result would have been our **Christmas present for Cris**.

V was selected because it has the largest O diffusivity, so it could have increased the mobility of O in TiZr…
ESRF stainless steel undulator vacuum chambers & change of EU regulation in matter of RP

IVC-1998 in Birmingham

Presented at EVC-1999 Lyon (France)

Effective desorption yield [molecules photon\(^{-1}\)]

Dose [photons m\(^{-1}\)]

\[\eta = 0,38 \, \text{D}^{-1}\]

\[\eta = 4,2 \times 10^{-5} \, \text{D}^{-0,38}\]

\[\eta = 6.1 \times 10^{-6}\]

Dose [mA h]
A more systematic study started in 1998

‘The new alternative technology for flat screen displays, namely Field Emission Displays require UHV to work. It could be possible to use NEGs to provide pumping in the small space allowed in between the two panels.’

(see The Economist, September 12th-18th 1998, p.96-97)
Quick activation: yes

30 40 50 60
2θ (degrees)

TiZrV Substrate

STM

100 nm

Optimization of the deposition parameters: the role of the substrate temperature

After activation, TiZrV has a low SEY: a solution to ecloud in LHC

Installation in accelerators: the production of undulator vacuum chambers started in 1999 for the ESRF

EPAC 2000 and AVS-2000 Boston

PERFORMANCE OF A NARROW-GAP, NEG-COATED, EXTRUDED-ALUMINIUM VACUUM CHAMBER AT THE ESRF

R. Kersevan, ESRF, BP 220, F-38043 Grenoble, France

Abstract

When a narrow-gap insertion device (ID) vacuum chamber is installed in a straight section of the ESRF, its conditioning time is found to affect the operation of the storage ring mainly in two ways. The first one is the effect of the local pressure bump on the global average.

ESRF: 10 chambers already inserted within two year
ELETTRA: 1 chamber installed (11/2000)
NSRRC: 1 prototype chamber coated, test in August 2002
DIAMOND: 1 prototype chamber coated (11/2000)
SOLEIL: under consideration

First chamber produced for other Institutes: PSI June 1999
First ESRF Al-alloy vacuum chamber

Cross section of the TiZrV coated ESRF’s vacuum chamber (length= 5.5 m)

No effect on the machine global impedance was observed.
**ESRF** was the first institute/firm to be licensed by CERN (2000). The support of Jean-Marc Filhol was important.

**SAES** was the first industrial partner (January 2001)

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**Memorandum**

To: Mme. E. Regat / FI
C: Mlle H. Pessard / EST, C. Benvenuti / EST-SM
From: P. Chiggaito / EST-SM-DA

Subject/Object: Facturation chambre 5-m avec couche NEG

Je vous serais reconnaissant de facturer à:

M. Roberto Keservan,
ESRF European Synchrotron Radiation Facility,
Polygone Scientifique Louis Neel, BP 220,
F-38043 Grenoble CEDEX

les travaux suivantes:

1. Etude et déposition d’une chambre ESRF en Al

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**Other licensees:**
- DESY, GSI, LNLS
- VARIAN (now Agilent), FMB
First hypothetical application in the LHC: TiZrV in a beam screen without holes

- The coating of a tight beam screen was inquired by L. Evans, the LHC project leader.
- Heating would have been possible by 4 molybdenum heaters isolated by alumina tubes.
- The main advantages would have been:
  - No need for beam scrubbing in the LHC arcs.
  - He leaks from the capillaries would have been less critical.
  - Possible heating of the beam pipe.

However:
- The pumping efficiency of TiZrV at 20 K was not confirmed.
- Pumping of CH₄?
- The cryopumping of the cold bore would have been lost.
- It was too late to start a parallel study of integration.
- Additional costs for coating and cabling.

Management decision: Forget beam-screen and be ready for the coating of experimental and long-straight section beam pipes.
TiZrV thin film coating for the LHC

- 6 Km of beam pipe
- About 1400 vacuum chambers
- Experimental vacuum chambers
In 2003 the team focused on the facility for the LSS series production

- The development and the construction of the coating facilities started at the end of 2002.
- Pre-series of LSS standard chamber: November 2003
The new set-up for the coating of the LSS chambers

Slide prepared for the Department Head

The cathodes

Manifold and chambers on the bench

The solenoids

The series production started in February 2004
The coating system

3mm wires of Ti, Zr and V
The LSS standard vacuum chamber

L max = 7 m
ID = 80 mm
thickness = 2 mm

316LN st. steel: allows high T treatments without softening

OFE copper: high conductivity, reliable brazing with st. steel

OFS copper: high conductivity, better mechanical performance than OFE at high temperature

Courtesy of S. Atieh
October-November 2003: modification of surface treatment for copper tubes

The adhesion of the TiZrV film on copper was probed by high-pressure water rinsing and/or ultra-sound agitation in deionized water.
October-November 2003: modification of surface treatment for copper tubes

Flake with copper on one side and NEG on the other
Meeting LSS – 07/11/2003

Present: J-P. Bacher, P. Chiggiato, P. Costa Pinto, A. Lasserre, G. Arnau Izquierdo, I. Wevers

The following treatments were presented:
1. Standard cleaning + High pressure water rinsing
   The surface appears very rough in the affected zone. This method should be easy to implement.
2. SulfoNitric acid (4µm) + final chromic acid passivation
   The surface aspect is also rough (holes have a clear edge) in the affected zone but the rest is smooth. The application of this method is very difficult and hazardous.
3. Ammonium persulfate (35µm) + final chromic acid passivation
   The affected zone can still be distinguished from the rest but appears to have smooth bumps instead of 'holes' like the former 2 methods. This method should be feasible to apply. The effect on the brazing should be checked.
The coating team in September 2004
2004: Ultrathin heaters for vacuum chambers inserted into magnets

INTRODUCTION

Beam stay clear requirements combined with limited space inside magnet yokes initiated the development of the heater. Additional reasons are the small separation between the two LHC beams and transparency for experiments. Table 1 lists all normal conducting LHC magnets which will be equipped with the new heater. The heater is also foreseen for the following areas with space constraints: recombination chambers, experimental chambers and chambers adjacent to collimators. Special requirement arise also from the fact that all chambers are NEG (TiZrV) coated which requires homogeneous activation temperatures beyond 200°C.

Table 1: LHC magnets with new heater (all dimensions in mm, chamber lengths vary between 1275 and 4150 mm; Dimensions: two external ellipse diameters and thickness; Gap: radial)

<table>
<thead>
<tr>
<th>Magnet</th>
<th>Qty</th>
<th>Dimensions</th>
<th>Material</th>
<th>Gap</th>
</tr>
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</table>

First electrical insulation layer: 2 layers of 60 μm thermoplastic PI tape (Kaneka Pilex, AST 252); currently wrapped with one single 20 mm tape and 50% overlap. The PI tape is coated on both sides with 5 μm thermosetting PI resin to keep all layers together after a heat treatment (polymerisation). II. SS heater bands of 50 μm thickness, 5 mm wide, wrapped in spaced helices; the number of helices and the spacing is used to adjust the heater resistivity. See Figure 2.

III. Second electrical insulation layer (as layer I).
IV. Optional reflective screen to reduce heat losses by radiation: One layer without overlap of 50 μm PI tape coated on one side with aluminium (customized supply, Tricon, Germany). See Figure 1.
V. Temporary layer of shrinkable polyester tape for polymerisation (117803-M11S, Fratec AG, Switzerland). The tape creates an external pressure on the chamber when heated up in order to improve the polymerisation process and to avoid delamination of the layers. The thickness of the final heater varies depending on the number of layers between 0.29 and 0.4 mm.
Functional quality control: XPS and pumping speed

Coupon samples for SEM and XPS

25-cm long vacuum chambers for pumping speed measurement
Functional quality control: XPS and pumping speed

O 1s peak area

Heating Temperature [ºC] - 1 hour duration

O 1s peak area (A.U.)

Heating Temperature [ºC] - 1 hour duration

Courtesy of M. Taborelli
Functional quality control: XPS and pumping speed

CO

Capture probability

$Q \text{ [molecules cm}^{-2}\text{]}$

$10^0$  $10^1$  $10^2$  $10^3$  $10^4$  $10^5$  $10^6$

$10^{10}$  $10^{11}$  $10^{12}$  $10^{13}$  $10^{14}$  $10^{15}$  $10^{16}$
NEG coated chambers for the LHC

- **Mar 2004 → Oct 2004**: 454 7m long standard LSS chambers
- **Jun 2005 → Jan 2006**: 650 chambers
  MBXW, MQW, MBW, MCBW, short LSS, experiments, etc

**Series production**

*Courtesy of P. Costa Pinto*
Series production

The LSS chamber was installed in the LHC tunnel in the summer 2006
Activation in the LHC tunnel

Claudio Raggi, Master Degree Thesis, 2005
Activation in the LHC tunnel

Installation of the bakeout heater and control

NEG activation monitoring.
Pressure profile during heating

 Activation of NEG Chambers

NEG Chambers at 180°C

NEG Chambers at 120°C

\[ P_{\text{Penning}} = 1 \cdot 10^{-5} \div 1 \cdot 10^{-4} \text{ [mbar]} \]

<table>
<thead>
<tr>
<th>Component</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>NEG</td>
<td>230°C</td>
</tr>
<tr>
<td>S.S.</td>
<td>150°C</td>
</tr>
<tr>
<td>Flanges</td>
<td>250°C</td>
</tr>
<tr>
<td>Sec. Valves</td>
<td>140°C</td>
</tr>
<tr>
<td>RGA + SVT</td>
<td>180°C</td>
</tr>
</tbody>
</table>

After 24h:

\[ P_{\text{Penning}} < 5 \cdot 10^{-8} \text{ [mbar]} \]

Courtesy of G. Breglioazzi
Experiments in the lab and a dedicated Monte Carlo simulation indicate that a good activation is obtained when the RGA $H_2$ signal decreases by a factor of at least 3 when opening the valve.
Average pressure, measured by BA gauges, 1 month after the end of bakeout: \( P \approx 6 \times 10^{-12} \text{ mbar } N_2 \text{ eq.} \)
The ultimate pressure was limited by the outgassing of the uncoated stainless steel module.

Degassing of the Stainless Steel Module:

With BA Gauge ON: \( Q_{\text{H}_2} \approx 4.5 \cdot 10^{-9} \text{ mbar l/s} \)

NEG Pumping Speed for \( \text{H}_2 \approx 260 \text{ l/s} \)

\[ P_{(\text{N}_2 \text{ eq})} \approx 7 \cdot 10^{-12} \text{ mbar N}_2 \text{ eq.} \]

 Courtesy of G. Bregliozi
TiZrV coating during the Long Shutdown 1: assorted geometries

Courtesy of P. Costa Pinto
TiZrV coating during the Long Shutdown 1: assorted geometries

Courtesy of P. Costa Pinto
New project: ELENA. NEG coating needed in 2015.

ELENA ring:
Calculation of the pressure profile seen by the antiproton beam along one dipole magnet

MolFlow+: ray-tracing, colour-coded pressure profile, and 2000 l/s NEG lump pump

Courtesy of R. Kersevan
NEG coatings in synchrotron radiation facilities

users, in design/study
One achromat
Length 26 m

In each achromat:
- 10 BPMs
- 3 pumping ports (with ion pumps),
- 1 crotch absorber,
- 3 gate valves.

MAX IV in Lund
MAX IV in Lund: the ‘standard’ vacuum chamber

Inside diameter: 22 mm,  
Total length: 2.5 m,

Bent part:  
Arc length 1 m,  
Bending angle 30°,  
Bending radius 19 m.

Welded bellows  
Chamber body  
Ribs  
Cooling for corrector area  
Distributed cooling  
Cooling for corrector area

Courtesy of M. Grabski (MAX IV)
MAX IV in Lund: the light-e beam bifurcation chambers (VC1 and VC2)

Chamber exit

Chamber entrance

Courtesy of M. Grabski (MAX IV)
MAX IV in Lund: the light-e beam bifurcation chambers (VC1 and VC2)

VC2 with antechamber:

3rd coating – 3+1 cathodes (9e-1 mbar, 240V, 0.28A, 68W)

7th coating - 1 cathode (3.1e-1 mbar, 700V, 0.03A, 26W)

9th coating - 2 cathodes (2.7e-1 mbar, 775V, 0.06A, 47W)

12th coating - 1 cathode (3.6e-1 mbar, 650V, 0.04A, 26W)

3rd coating – 3+1 cathodes (9e-1 mbar, 240V, 0.28A, 68W)

260 Gauss, diode

360 Gauss, magnetron

360 Gauss

360 -> 270 Gauss – 1 cathode

12th coating - 1 cathode (3.6e-1 mbar, 650V, 0.04A, 26W)

360 Gauss, magnetron

9th coating - 2 cathodes (2.7e-1 mbar, 775V, 0.06A, 47W)

Courtesy of M. Grabski (MAX IV) and P. Costa Pinto
The standard coating of very small and asymmetric cross-section beam pipes:

- requires a significant amount of time for the optimization of the coating parameters;
- the quality and uniformity of the film and its performance are unmeasurable;

In addition, at present, vacuum chambers are not designed to facilitate surface treatments and coating.

July 1999

CERN quotation for ESRF

3. 5m long chambers, model 8mm high opening
   The diode sputtering method proposed for the 14mm model will not work for the 8mm high model.
   The proposed magnetron test programme for the 11mm model may demonstrate that the 8mm model can also be coated satisfactorily by the magnetron method. If this is not the case then it will be necessary to study an alternative method based on preparing two identical half chambers which will both be coated in a suitable diode sputtering chamber before being MIG welded together. We believe that MIG welding will not deteriorate the pumping characteristics of the coating and is compatible with the dimensional tolerances required. We are unable at present to establish a price estimate for this solution.
Future developments: the electroforming way

Courtesy of L. Marques Antunes Ferreira
Future developments: the electroforming way

Courtesy of L. Marques Antunes Ferreira
Future developments: the electroforming way

after electroforming

80 mm

200 mm

Courtesy of L. Marques Antunes Ferreira
Future developments: the electroforming way

Data obtained on the activation

<table>
<thead>
<tr>
<th>Samples</th>
<th>Carbide [%at]</th>
<th>Oxygen area decrease [%at]</th>
<th>Carbon area decrease [%at]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard NEG</td>
<td>&gt; 40</td>
<td>&gt; 66 %</td>
<td>No sharp limit. Typically around 50%</td>
</tr>
<tr>
<td>1TA6060</td>
<td>14,9</td>
<td>23</td>
<td>5,4</td>
</tr>
<tr>
<td>2TA1050</td>
<td>22</td>
<td>57,6</td>
<td>-56</td>
</tr>
<tr>
<td>3TA1050</td>
<td>41,1</td>
<td>67,7</td>
<td>30,7</td>
</tr>
</tbody>
</table>

According to standard NEG
not according to standard NEG

Courtesy of L. Marques Antunes Ferreira
Acknowledgments

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• José Miguel Jimenez

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