

## Material Deposition and Composition of Walls

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### 2. OBJECTIVE

In the framework of this task a Laser Induced Breakdown System (LIBS) is proposed to be manufactured for in situ investigation of the wall composition, including material deposition, without breaking the vacuum. The system has to be calibrated with the specific materials which are supposed to be on the tokamak wall, that is W, Mo, C.

The role of the MEdC Association is to provide coated samples with various compositions of these materials using the Combined Magnetron Sputtering and Ion Implantation (CMSII) deposition technique. The CMSII technology is applied for 10-15  $\mu\text{m}$  and 20-25  $\mu\text{m}$  W coatings of  $\sim 1,800$  CFC tiles for the new JET wall and for a few hundreds of Fine Grain Graphite (FGG) tiles for the ASDEX Upgrade wall.

The depth profiles of the concentrations for the coating constituents will be measured by Glow Discharge Optical Spectrometry (GDOS) technique, which is currently used for quality control of the W layers deposited by CMSII technology.

### 3. RESULTS AND DISCUSSION

A number of four EURATOM Associations (ENEA – Italy, CEA – France, TARTU – Estonia and IPPMF – Poland) have LIBS equipments and they needed reference samples in order to calibrate the laser systems.

In the first phase of the project 35 tungsten coated samples with a Mo interlayer were prepared, analyzed and sent to the associations. They are listed in Table 1. The substrate was Ti.

Table 1 Distribution of the coated samples to the associations

| No. | Association      | Number of samples | Identification of samples   | GDOS profiles                                       | Coating thickness ( $\mu\text{m}$ )                          |                          |
|-----|------------------|-------------------|---|---|--|--------------------------|
|     |                  |                   |   |   | W  | Mo                       |
| 1   | CEA France       | 15                | IU-90-18; IU-90-19;<br>IU-90-20; IU-90-21;<br>IU-90-22; IU-90-23;<br>IU-90-35; IU-90-36;<br>IU-94-12; IU-94-13;<br>IU-94-14; IU-94-15;<br>IU-94-16; IU-94-17;<br>IU-94-18 | IU-90-17-1<br>IU-90-23-1<br>IU-94-7-1<br>IU-94-18-1 | 10.8 $\pm$ 1<br>11.5 $\pm$ 1<br>12.8 $\pm$ 1<br>13.1 $\pm$ 1 | 2.0<br>2.2<br>2.4<br>2.5 |
| 2   | ENEA Italy       | 10                | IU-90-13; IU-90-14;<br>IU-90-15; IU-90-16;<br>IU-90-17; IU-94-5;<br>IU-94-6; IU-94-7;<br>IU-94-8; IU-94-9;  | IU-90-17-1<br>IU-90-23-1<br>IU-94-7-1<br>IU-94-18-1 | 10.8 $\pm$ 1<br>11.5 $\pm$ 1<br>12.8 $\pm$ 1<br>13.1 $\pm$ 1 | 2.0<br>2.2<br>2.4<br>2.5 |
| 3   | TARTU<br>Estonia | 5                 | IU-90-1; IU-90-3;<br>IU-90-4; IU-90-5;  | IU-90-17-1<br>IU-90-23-1                            | 10.8 $\pm$ 1<br>11.5 $\pm$ 1                                 | 2.0<br>2.2               |

|   |                 |   |   |                          |                  |            |
|---|-----------------|---|---|--------------------------|------------------|------------|
|   |                 |   | IU-90-6;  |                          |                  |            |
| 4 | IPPLM<br>Poland | 5 | IU-90-11; IU-90-31;<br>IU-90-32; IU-90-33;<br>IU-90-34; | IU-90-17-1<br>IU-90-23-1 | 10.8±1<br>11.5±1 | 2.0<br>2.2 |

These samples were used to identify the presence of W, Mo and Ti and to see their depth variation in accordance with number of laser pulses. The LIBS spectrum is recorded for each pulse. The optimum wavelengths for recording W, Mo and Ti were identified as well. Typical depth profiles of the concentrations for the coating constituents, measured by GDOS technique, are shown in Fig.1.

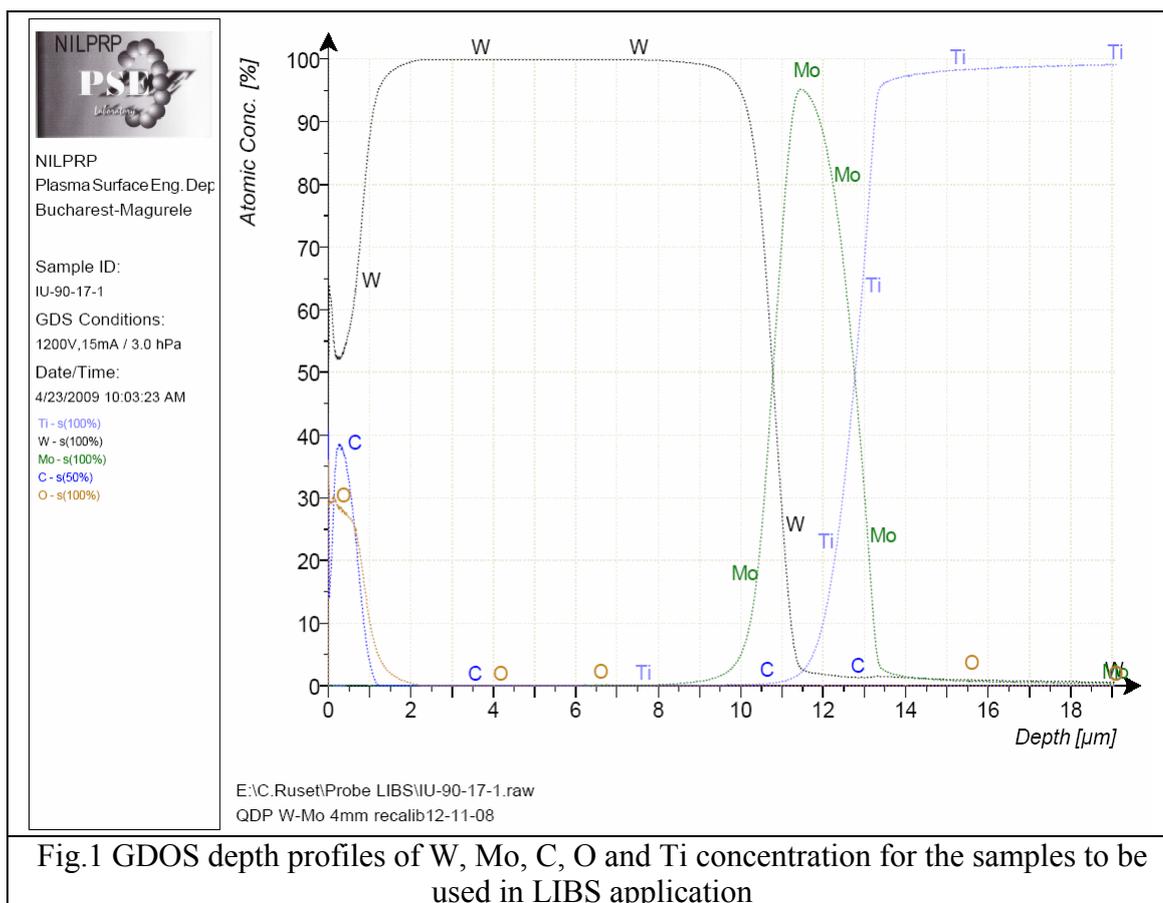


Fig.1 GDOS depth profiles of W, Mo, C, O and Ti concentration for the samples to be used in LIBS application

In the second phase of the project W – C composite calibrated coatings with a thickness of 8 – 17  $\mu\text{m}$  have produced and delivered to the associations.

Using complex magnetron targets, C-W coatings with various W concentrations were produced. Depending on the W/C ratio into the target and on the processing parameters, the coating thickness was in the range of 8 – 17  $\mu\text{m}$ . Two GDOS depth profiles for C-W composite coatings are shown in Fig.2. As it can be seen, W concentrations into the coatings is 17 at.% and 27 at.% respectively.

The coating structure was investigated by a diffractometer in Bragg-Bretano geometry using  $\text{Cu K}\alpha=1,5405 \text{ \AA}$  radiation. The XRD pattern, shown in Fig.3, reveals very wide peaks which seem to be the result of a convolution of two or more phases. These peaks might be associated with the existence of non-coherent dispersion fields produced by structures with a high level of amorphisation. Not clear peaks of W or WC have been detected.

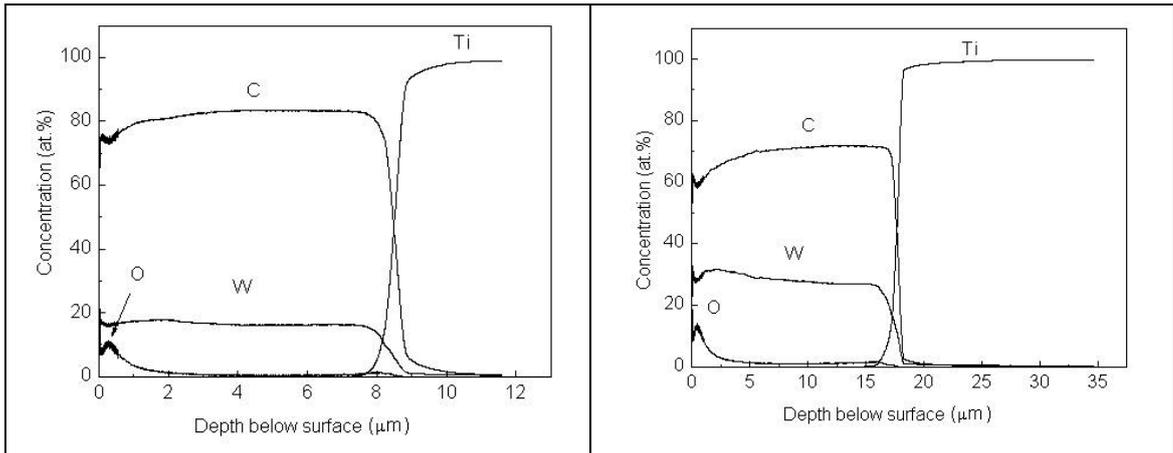


Fig.2 Typical GDOS depth profiles of C-W composite coatings produced with magnetron targets containing various W/C ratios

The coatings are compact, free of pores or cracks and have hardness approx. 2.000 HV0.025. They can be used as set up samples for LIBS experiments.

The samples were sent to associations in accordance with the Table 1.

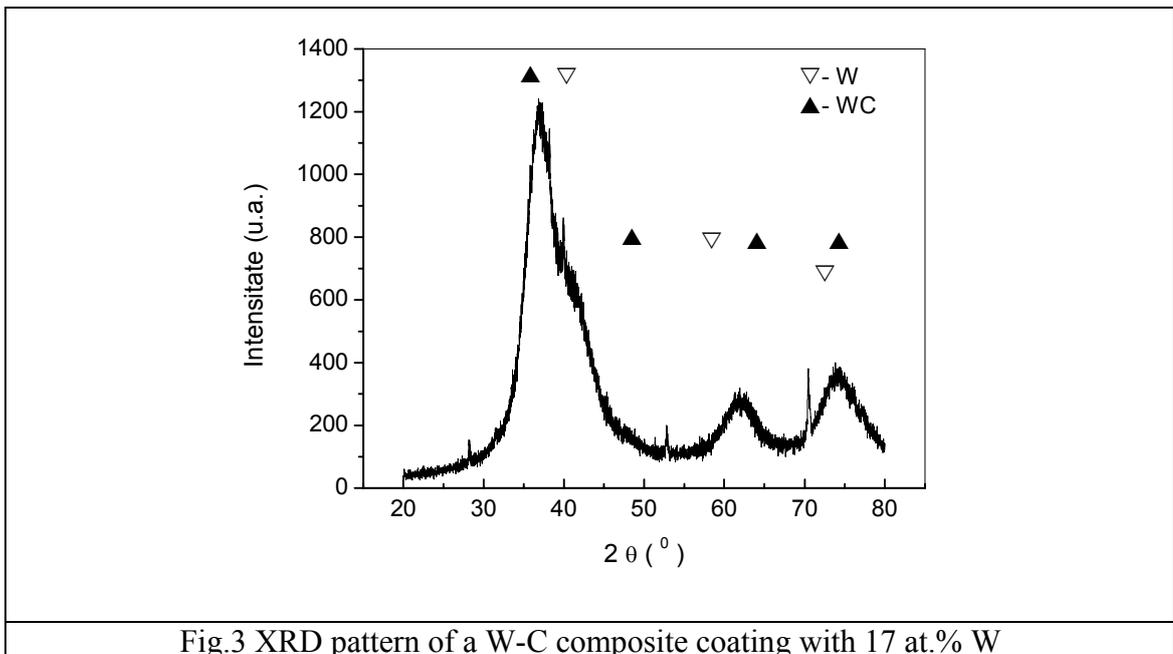


Fig.3 XRD pattern of a W-C composite coating with 17 at.% W

Table 1 Distribution of the coated samples to the associations

| No. | Association   | Number of samples | Identification of samples | Coating                     |                    |     |    |
|-----|---------------|-------------------|---------------------------|-----------------------------|--------------------|-----|----|
|     |               |                   |                           | Thickness ( $\mu\text{m}$ ) | Composition (at.%) |     |    |
|     |               |                   |                           | C                           | W                  | O   |    |
| 1   | CEA France    | 1+1               | EU-567-1                  | 17.2                        | 70                 | 27  | 3  |
|     |               |                   | EU-568-1                  | 8.5                         | ~82                | ~15 | ~3 |
| 2   | ENEA Italy    | 1+1               | EU-567-2                  | 17.2                        | 70                 | 27  | 3  |
|     |               |                   | EU-568-3                  | 8.5                         | ~82                | ~15 | ~3 |
| 3   | TARTU Estonia | 1+1               | EU-567-3                  | 17.2                        | 70                 | 27  | 3  |
|     |               |                   | EU-568-4                  | 8.5                         | ~82                | ~15 | ~3 |
| 4   | IPPLM         | 1+1               | EU-567-4                  | 17.2                        | 70                 | 27  | 3  |

|  |        |  |          |     |     |     |    |
|--|--------|--|----------|-----|-----|-----|----|
|  | Poland |  | EU-568-5 | 8.5 | ~82 | ~15 | ~3 |
|--|--------|--|----------|-----|-----|-----|----|

#### **4. DISSEMINATION OF RESULTS**

No publications were in 2009 on this subject. A publication is preparing for 2010.

#### **5. CONCLUSIONS**

The MEdC tasks in the framework of the project were accomplished. C-W samples of different compositions for LIBS experiments were produced and delivered.

#### **6. COLLABORATIVE ACTIONS**

A close cooperation between MEdC, ENEA, CEA, TARTU and IPPMF associations occurred in the framework of this project.