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# The study of the colorimetry to measure the deposition thickness on the plasma-facing wall in QUEST

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## Background

- For a future nuclear fusion reactor, the characteristics of the plasmafacing wall including the information of the deposition are studied for understanding those such as tritium storage or the hydrogen recycling.
- The deposition thickness is usually measured with TEM and ellipsometer. But these two heavy devices are very difficult to be

$$\widetilde{n}_{0} \sin i_{0} = \widetilde{n}_{1} \sin i_{1} = \widetilde{n}_{2} \sin i_{2} \quad (1)$$

$$\Delta_{1} = \frac{2\pi}{\lambda} d_{1} \widetilde{n}_{1} \cos i_{1} \quad (2)$$

$$r_{1s} = \frac{\widetilde{n}_{0} \cos i_{0} - \widetilde{n}_{1} \cos i_{1}}{\widetilde{n}_{0} \cos i_{0} + \widetilde{n}_{1} \cos i_{1}}, r_{2s} = \frac{\widetilde{n}_{1} \cos i_{1} - \widetilde{n}_{2} \cos i_{2}}{\widetilde{n}_{1} \cos i_{1} + \widetilde{n}_{2} \cos i_{2}} \quad (3)$$

$$r_{1p} = \frac{\widetilde{n}_{1} \cos i_{0} - \widetilde{n}_{0} \cos i_{1}}{\widetilde{n}_{1} \cos i_{0} + \widetilde{n}_{0} \cos i_{1}}, r_{2p} = \frac{\widetilde{n}_{2} \cos i_{1} - \widetilde{n}_{1} \cos i_{2}}{\widetilde{n}_{2} \cos i_{1} + \widetilde{n}_{1} \cos i_{2}} \quad (4)$$





applied to the actual plasma-facing wall directly. Because of the convenience, the colorimetry can measure the deposition thickness on the actual plasma-facing wall easily.







Figure 1: The left is the ellipsometer and right is the colorimeter

- About the measurement of deposition thickness the colorimetry is in good agreement with the ellipsometry for a-C:H (amorphous hydrogenated carbon) dominant deposition in TEXTOR <sup>1</sup>.
- But for the metal first wall and divertor in ITER, the deposition will contain metals and will not a-C:H dominant any more. And for QUEST the first wall is stainless steel and the divertor and limiter are tungsten. After several campaigns the deposition has been formed on the plasma-facing wall. The main ingredient is the mixture of carbon and metal.







According to the equations from (1) to (7), if the incident angle  $i_0$  and the complex refractive index of the substrate  $\tilde{n}_2$  have been known (For QUEST, the substrate is stainless steel) and it is in the air which means  $\tilde{n}_0 = 1$ , there is

$$\overline{R} = f(\widetilde{n}_1, d_1) \tag{8}$$

The reflectivity  $\overline{R}$  has been measured with the colorimeter. If the complex refractive index of deposition  $\tilde{n}_1$  has been known, then the deposition thickness  $d_1$  could be derived.

## The measurement of complex refractive index of deposition on the QUEST wall with samples

Before the spring summer campaign in 2014, 6 Molybdenum samples were set up on the different positions on the first wall in QUEST. When the campaign was finished, the samples were taken out for the analysis.



**Figure 7:** The SUS samples which were taken out of QUEST after spring summer campaign in 2014

When the wavelength is 540 nm, the substrate SUS's complex refractive index is 2.67-4.03\*i.<sup>3</sup> The deposition's refractive index  $n_1$ is  $2.1 \pm 0.1$  as analyzed with Mo samples. Then the samples' deposition thickness  $d_1$  and extinction coefficient  $k_1$  could be derived from the measurement of ellipsometer.



**Figure 8:** The extinction coefficient of the deposition  $k_1$  of the SUS samples

From the Fig. 8, there are two different  $k_1$  regions apparently. On the upper wall the deposition's  $k_1$  is lower, and on the lower wall the deposition's  $k_1$  is higher.

## The comparison of the results of thickness between colorimeter, ellipsometer and TEM

In the calculation of the colorimeter, on the positions from 4 to 14 the

#### Sputtering time / s

**Figure 2:** The components of the deposition on QUEST wall It is necessary to study the feasibility of the colorimetry on measuring the metal-containing deposition thickness.

## The colorimeter

1. Measuring data RGB (Red, Green, Blue) reflectivity.

2. **Response range**: R (590~720nm), G (480~600nm), B (400~540nm).

**Maximum sensitivity**: R (615nm), G (=540nm), B (=465nm). The sensitivity versus wavelength is Gaussian distribution.

3. Sensor diameter:  $\phi$  8.1 mm.

4. Diameter of the integrating sphere:  $\phi$  47 mm.

5. Light source: white LED.

### 6. The **reflectivity** of light is output as RGB values ( $0 \sim 1023$ ).



**Figure 5:** The Mo samples on the first wall in QUEST during the 2014 spring summer campaign

When the wavelength is 540 nm, the Mo's complex refractive index is 3.79-3.61\*i.<sup>2</sup> When the different refractive index  $n_1$  of the deposition on the samples is assumed, the the different values of the deposition thickness could be derived from the measurement of the ellipsometer. Then these values were compared with those measured with TEM. From Fig. 6, in the error range of standard deviation  $n_1$  is  $2.1\pm0.1$ .



deposition's complex refractive index  $\tilde{n_1}$  is  $2.1 - (0.55 \pm 0.38) * i$ . And on the positions from 15 to 30, the he deposition's complex refractive index  $\tilde{n_1}$  is  $2.1 - (1.41 \pm 0.27) * i$ .



#### **Figure 9:** The reflectivity versus the deposition thickness $d_1$ with different $k_1$



**Figure 10:** The results of deposition thickness  $d_1$  measured with ellipsometer, colorimeter and TEM

## Discussion

The refractive index of the deposition n<sub>1</sub> on the first wall in QUEST is 2.1 ± 0.1. But the extinction coefficient k<sub>1</sub> fluctuates obviously on different positions. On the whole on the upper wall k<sub>1</sub> is low, which is 0.55±0.38. On lower wall k<sub>1</sub> is high, which is 1.41±0.27. On the upper wall the deposition is very thin and the oxygen and carbon from the air may be more concentrated, which results in the lower extinction coefficient.
On the upper wall the deposition thickness measured with the colorimeter agrees well with that measured with the ellipsometer and TEM in the error range. On the lower wall the deposition thickness of the deposition thickness. This because that for the wavelength of 540 nm the deposition k<sub>1</sub> is high enough and there may be some error of the reflectivity measured with the colorimeter.

Figure 3: The reflectivity measured with colorimeter on QUEST wall

## The principle of measurement with the colorimeter



#### Figure 4: The principle of measurement of deposition thickness with colorimeter

<sup>3</sup>Shiro Matsuda et al, Transactions of the Japan Institute of Metals 18, 1977

**Figure 6:** The above figure shows that in the measurement of ellipsometer the deposition thickness  $d_1$  could be derived from the different assumed refractive index  $n_1$ . The following figure shows the true thickness  $d_1$  measured with TEM

As shown in Fig. 7, between each two adjacent measuring points of the colorimeter there is a fixed plate (the white pieces zoned by the yellow circles) on the first wall in QUEST. After the spring summer campaign in 2014, 21 fixed plates in different positions were taken out of QUEST as the samples for the ellipsometer analysis.

## Summary

For metal containing deposition film on the first wall in QUEST, the colorimeter is a convenient tool to measure the deposition thickness on any positions on the wall. It could provide the satisfactory results for the thin deposition, and provide the values range by rough estimate for the thick deposition. For the study of tritium storage or hydrogen recycling on the first wall, the colorimeter is an effective tool.

<sup>&</sup>lt;sup>1</sup>EP. Wienhold et al, Nucl. Instrum. Methods Phys. Res. B 94 (1994) 503-510.

<sup>&</sup>lt;sup>2</sup>E. Palik, etc, Handbook of Optical Constants of Solids, Vols. 1-4 combined-AP, 1998.