ESRF	**Experiment title:** Thermally induced nano-structuring in metal-doped carbon films studied by means of high resolution X-ray absorption and emission spectroscopies	**Experiment number**: MA-320
Beamline: ID26	**Date of experiment**: from: 9/05/2007 to: 15/05/2007	**Date of report**: 23/08/2012
Shifts: 15	**Local contact(s)**: Marcin Sikora	*Received at ESRF:*

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Report:

Some results of the experiment are published in:
C. Adelhelm et al., Surface & Coatings Technology **205** (2011) 4335–4342.
Other part is still analyzed. It was presented during ESRF Science & Students Days, 8 - 10 October 2008, Val Cenis, France. Abstract attached below:

Nanocrystallization investigation using X-ray Emission and Absorption Spectroscopy
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Nanostructured metal-doped amorphous carbon films exhibit excellent tribological properties, high hardness and increased electrical conductivity. In the quest for optimum materials for future fusion devices like ITER, the application of carbon (CFC) together with metals as plasma facing material have been considered. Despite the formation of undesired metal-containing hydrocarbon layers during reactor operation, the metal containing carbon films show higher erosion resistance against hydrogen, which is crucial for application. In order to optimize the properties of these coatings a systematic investigation of the influence of doping on the structure and the erosion yield was studied using a model system: metal-doped carbon films produced by magnetron sputtering.

The structure of carbon films doped with low amounts of Ti, V, Zr and W was studied by XRD, RBS and EXAFS [1]. In the 'as-deposited' layers metal species have an amorphous, disordered environment which begins ordering after annealing, when a carbide and (possibly) oxide clusters appear. To monitor the course of the annealing process, and the simultaneous evolution of the local surroundings of the vanadium species, the in-situ temperature dependent measurements of V Kβ'' and $K\beta_{2,5}$ emission spectra were performed at ID26. Such measurements provide a bulk sensitive, element selective method of identifying ligands in transition metal compounds [2]. The spectra reveal a temperature correlation between the $K\beta_{2,5}$ intensity and the amplitude of EXAFS oscillations, reflecting the formation of nanocrystals. Both carbide and oxide contributions to the Kβ'' are observed in samples annealed at high temperatures (see fig.1). They appear at the temperature of the order-disorder transition, while their further evolution is qualitatively different from that of the $K\beta_{2,5}$. Since the two regions of emission spectra have a different origin and sensitivity, they may be used together to estimate the relative amounts and average size of carbide and oxide clusters formed.

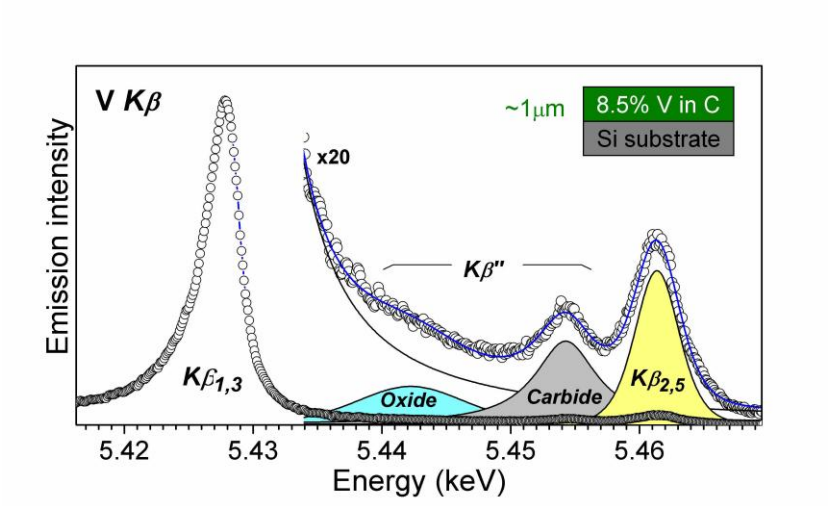

Figure 1: Vanadium Kβ emission spectrum measured at 1300K.

References

[1] - M. Balden, C. Adelhelm, M. Sikora, J. Nucl. Mater. 367-370, 1458, (2007)
[2] - S. Fazinić, M. Jakšić, L. Mandić, J. Dobrinić, Phys. Rev. A 74 (2006) 062501