

# NANOCRYSTALLIZATION IN VANADIUM DOPED CARBON FILMS STUDIED BY MEANS OF X-RAY EMISSION SPECTROSCOPY

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*Keywords:* X-ray spectroscopies, metal-doped amorphous carbon films, crystallization

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We report on an *in-situ* study of thermally induced nanocrystallization in the series of vanadium doped carbon films by means of valence-to-core X-ray emission spectroscopy.

Nanostructured metal-doped amorphous carbon films show excellent tribological properties, high hardness and increased electrical conductivity. In future fusion devices like ITER [1], the application of carbon (CFC) together with metals as plasma facing material will lead to the formation of undesired metal-containing hydrocarbon layers during operation. Compared to pure carbon they show higher erosion resistance against hydrogen. A systematic investigation of the influence of doping on the erosion process 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 [2]. It revealed that the ‘as-deposited’ layers have an amorphous, disordered surrounding which already starts to order after annealing at temperatures below 1000 K, when a carbide and (possibly) some amount of oxide clusters are created.

In order to monitor the course of annealing process and simultaneous evolution of the local surrounding of vanadium species *in-situ* temperature dependent measurements of V  $K\beta''$  and  $K\beta_{2,5}$  emission spectra were carried out. They provide bulk sensitive, element selective way to identify the type of ligand in a transition metal compound [3] since the energy separation between  $K\beta_{2,5}$  and  $K\beta''$  corresponds roughly to the energy difference between the ligand  $2s$  and  $2p$  levels (Fig. 1).

XES measurements performed at the ESRF ID26 beamline proved the feasibility of such an experiment both from a surface and buried layers. The spectra obtained reveal a nice temperature correlation between  $K\beta_{2,5}$  area and the amplitude of EXAFS oscillations reflecting the formation of nanocrystals. Moreover, both carbide and oxide contributions to the  $K\beta''$  are observed in samples annealed at high temperatures. They appear at the temperature of order-disorder transition, while their further temperature evolution (and annealing dynamics) is qualitatively different from that of  $K\beta_{2,5}$ . Since the two parts of XES spectra have a different range of sensitivity,

this technique, supported by the cluster calculations of electronic structure, may be used to derive size of nanocrystals of unlike kind.

The results of quantitative analysis of the average size of crystalline clusters of both kinds at different doping levels and annealing temperatures and their comparison to the XRD derived estimations will be presented.

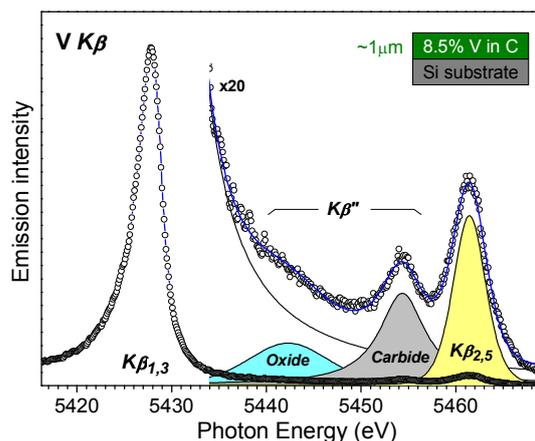


Figure 1. Vanadium  $K\beta$  emission spectrum of  $\sim 1 \mu\text{m}$  thick layer of 8.5% V in amorphous carbon upon annealing at 1300 K. The  $K\beta''$  of oxide and carbide ligands and  $K\beta_{2,5}$  peak are fitted using Voigtian profiles and are magnified by factor 20.

**Acknowledgements:** The authors acknowledge European Synchrotron Radiation Facility for provision of beamtime and B. Gorges for construction of the high temperature oven.

## References

- [1] R. Aymar, "ITER status, design and material objectives", *J. Nucl. Mater.* **307** (2002) 1-9.
- [2] M. Balden, C. Adelhalm, M. Sikora, "Thermal stability and nano-structure of metal-doped carbon layers", *J. Nucl. Mater.* **367-370** (2007) 1458-1462.
- [3] S. Fazinić, M. Jakšić, L. Mandić, J. Dobrinić, "Chemical dependence of second-order radiative contributions in the  $K\beta$  x-ray spectra of vanadium and its compounds", *Phys. Rev. A* **74** (2006) 062501.