

XAFS investigation of metal-doped carbon films

C. Adelhelm and M. Balden

Max-Planck-Institut für Plasmaphysik, Boltzmannstr. 2, 85748 Garching, Germany

Carbon (in form of CFC) is considered as plasma-facing material in future fusion devices like ITER [1]. The reactivity of carbon against hydrogen species (chemical erosion) is the main drawback, causing life-time problems of the components and formation of hydrocarbon layers in hidden parts of the reactor vessel. This is of safety concern if radioactive tritium – the fuel for fusion – is used. Doping of carbon with transition metals strongly reduces chemical erosion, which is the motivation for the development of new doped graphite and CFC materials [2]. To investigate the underlying mechanism of the reduced chemical erosion well-defined deuterium ion beam experiments were performed with metal-doped amorphous carbon films (a-C:Me) [3]. In order to evaluate and understand the influence of their micro- and nanostructure on the erosion process, it is important to characterize these coatings detailed on different length scales.

Hydrogen-free a-C:Ti films were produced by dual-source magnetron sputter deposition [4]. Annealing of the films (at 700 K – 1300 K) induced structural ordering, as carbide grain formation and growth, and changes of the carbon matrix. Using grazing incidence XRD, TiC crystallite formation and growth (some nm in size) could be observed for 7.5 % Ti doped a-C:Ti after annealing to ≥ 900 K, but not for lower temperatures. A sample with only 1 % Ti appeared x-ray amorphous even after annealing to 1300 K. Therefore, Ti *K*-edge XAFS measurements were performed at beamline C to study the atomic environment of Ti in the films by qualitative comparison with TiC standard. Spectra were acquired by detecting the emitted fluorescence light. Electron yield detection was applied for a spectrum of TiC powder. Data processing was performed using the program ATHENA [5]. The edge energy E_0 was determined from normalized spectra by taking the energy of the half edge step intensity.

Fig. 1 shows XAFS spectra and $\chi(R)$ for **7.5 % Ti a-C:Ti** annealed up to 1300 K and TiC standard. After annealing to 900 K, clear correspondence to TiC is observed. The lower intensity of the second Ti neighbour indicates a very small particle size with a large fraction of Ti atoms at the surface of TiC crystallites, thus having no complete second Ti shell. With increasing annealing temperature, the Ti-C interaction increases, with increasing electron transfer from Ti to C. This is evident in the developing of E_0 , as shown in Fig. 2a.

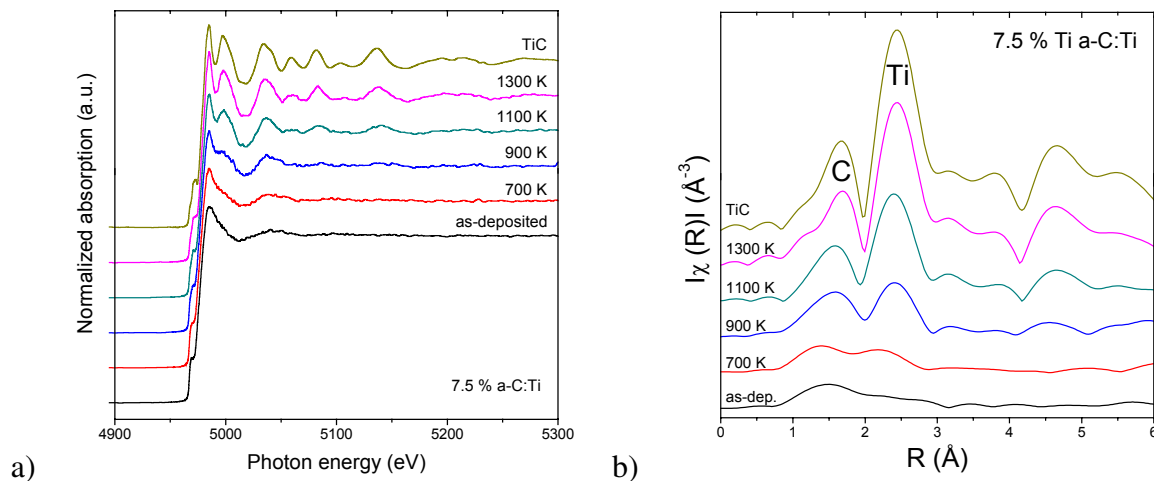
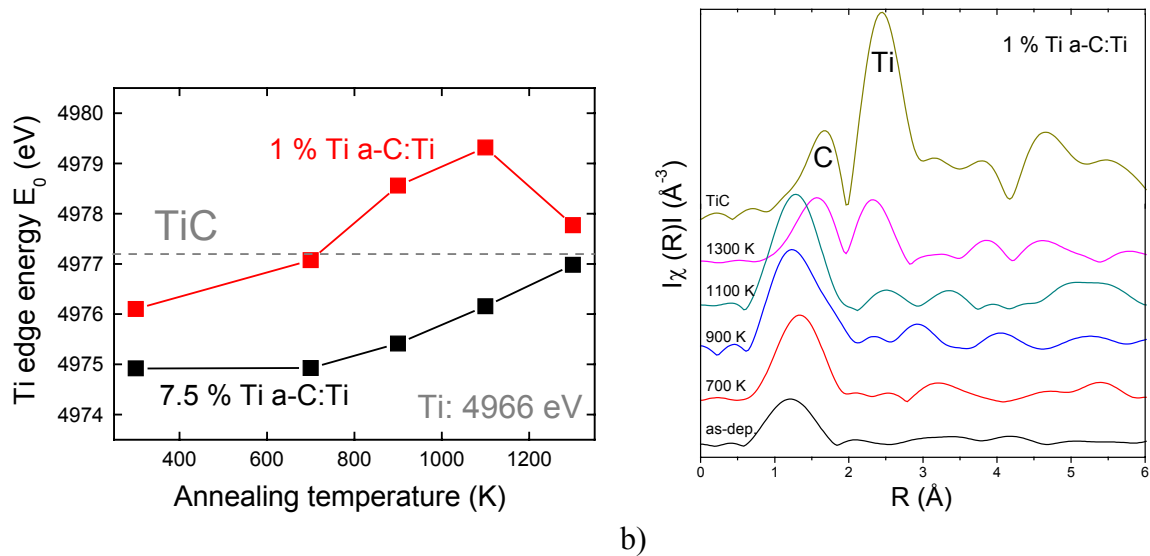


Figure 1: a) Ti *K*-edge XAFS spectra of **7.5 % Ti a-C:Ti** annealed up to 1300 K and TiC standard.
b) $\chi(R)$ after FT, k -range = 2–8.5 Å⁻¹, k^2 weighted.



a) Development of Ti-K-edge position with annealing temperature. b) **1 % Ti a-C:Ti**: $\chi(R)$ after FT, $k\text{-range} = 2\text{-}8.5 \text{ \AA}^{-1}$, k^2 weighted.

$\chi(R)$ for the **1% Ti a-C:Ti** sample is shown in Fig. 2b. For temperatures $<1300 \text{ K}$, the spectra are different compared to the 7.5 % doped film. Already for the sample after deposition a first maximum with higher intensity is evident at a lower R compared to C in TiC. It gains intensity with annealing, but no second maximum at the position of the next Ti coordination shell in TiC appears. After annealing to 1300 K, the atomic environment has strongly changed and shows similarity with TiC. The development of the edge position is shown in Fig. 2a. It indicates a stronger electron transfer to the next bonding partner compared to the 7.5 % doped sample. E_0 for 1 % Ti is at even higher energy compared to TiC for $T = 900\text{-}1300 \text{ K}$. The development of $\chi(R)$ and E_0 in the case of only 1% Ti can be explained by an O environment of Ti up to 1100 K. This is also supported by an O content of about 2 % determined by RBS. Annealing leads to a better ordered first shell and increasing Ti-O interaction, which increases E_0 above the value for TiC. After annealing to 1300 K, formation of a carbide environment starts to develop. But the lower intensity of the second coordination shell indicates a very small particle size.

Summarizing the results, the atomic environment of Ti in 7.5 % Ti a-C:Ti annealed to $<900 \text{ K}$ can be described as amorphous without formation of TiC crystallites (but with C in the first shell). At higher temperatures, formation of a TiC-like coordination is evident in $\chi(R)$. This is in line with results from XRD. For a-C:Ti with 1 % Ti, Ti is probably coordinated by oxygen up to 1100 K. Annealing leads to an increase of order in the Ti-O bonding. At 1300 K, formation of a TiC-like coordination starts to develop with probably very small TiC crystallite size.

E. Welter is acknowledged for his dedicated assistance during measurements. Part of this work has been performed within the framework of the Integrated European Project "Extremat" (contract NMP-CT-2004-500253) with financial support by the European Community. It only reflects the view of the authors and the European Community is not liable for any use of the information contained therein.

References

- [1] G. Federici, C.H. Skinner, et al., Nuclear Fusion 41, 1967 (2001)
- [2] E. Juan Pardo, M. Balden, et al., Physica Scripta T111, 62 (2004)
- [3] M. Balden, C. Adelhelm, Physica Scripta T128, 121 (2007)
- [4] M. Balden, B. T. Cieciwa, et al., Surface and Coatings Technology 200, 413 (2005)
- [5] B. Ravel, M. Newville, Journal of Synchrotron Radiation 12, 537 (2005)