CHARACTERIZATION OF CARBON COATINGS WITH LOW SECONDARY ELECTRON YIELD

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Abstract

Amorphous carbon (a-C) coatings can reliably be produced with a maximum secondary electron yield (SEY) close to 1 at room temperature. Measurements at low temperature (LHe) are in progress. Analysis by X-ray Photoemission Spectroscopy (XPS) shows a correlation between the lineshape of C1s spectrum in XPS and the maximum SEY of the investigated samples. The initial level of oxygen on the surface of the various samples does not seem to be related to the initial maximum SEY value. However, the increase of the SEY with air exposure time on each individual sample is related to the amount of oxygen containing adsorbates. Storage in different environments has been investigated (static vacuum, aluminium foil, dry nitrogen and desiccators), and shows significant differences in the "aging" behaviour. Aging is very moderate when storing samples wrapped in aluminium foil in air. Samples which have undergone aging due to inappropriate storage can be recovered nearly to the initial value of their SEY by surface treatments such as conditioning by electron beam, annealing under vacuum and ion bombardment. However, an enhanced sensitivity to air exposure is observed for most of these curing methods.

INTRODUCTION

Carbon coatings prepared by magnetron sputtering deposition exhibit very low Secondary Electron Yield (SEY). They represent a very promising solution to mitigate electron cloud if applied as coatings on the inner wall of the vacuum chamber of particle accelerators. In particular the low SEY is obtained without activation or bake-out treatment in UHV. Their performance was demonstrated in the Super Proton Synchrotron (SPS) [1], [2]. The results presented in the following show that their application can be extended to cryogenic systems. Most of the coatings have initially a maximum of the SEY (δ_{max}) close to 1, measured after air exposure of 1-2 hours, with a very narrow distribution. Deviations remain in the interval 0.85-1.15 and are partly due to change in coating parameters. The minor differences in composition measured with X-ray Photoemission Spectroscopy (XPS) do not correlate with the SEY data. An alternative approach based on the XPS carbon C1s lineshape is therefore proposed. Particle accelerators operate for tens of years and the functional lifetime of such a coating must be sufficiently long to avoid frequent maintenance interventions. Aging in terms of progressive increase of SEY is known to occur for all metal surfaces exposed to air and a characterization from this point of view is necessary also for the carbon coatings. Along the same line the possible techniques to recover the initial SEY value were also investigated.

EXPERIMENTAL

All investigated carbon samples were prepared by magnetron sputtering in different geometries, with several types of cathodes all made of graphite. Further details are given in [3]. The SEY as a function of the primary electron energy (range 50–1800eV) at normal incidence has been measured in two different systems, both working in 10^{-9} mbar pressure range. Both instruments measure the SEY by acquiring simultaneously the absorbed current on the sample and the secondary electron current on a collector, which is coaxial with the electron gun. All SEY measurements were performed by limiting the electron dose impinging on the surface: for a full curve the dose was below 10^{-6} C/mm^2. The accuracy of the SEY measurement is ±0.03 at room temperature (RT) and ±0.05 at 4.7K. The system operating at RT is coupled through UHV with an XPS spectrometer (MgK_α, non-monochromatized source, 45° emission angle). It enables the sample exchange through a fast entry load-lock. The system is also equipped with a flood gun for electron irradiation with 500 eV electrons, with an ion gun (Ar+, 3 keV) and a sample heater. The system which operates at cryogenic temperatures is described in more details in [4].

RESULTS AND DISCUSSION

The SEY of the a-C coating after bake-out at 150^{0}C has been measured at RT and at 4.7 K in the cryogenic system (Fig. 1). The value of δ_{max} is 1.12 and no significant difference is detected considering the experimental accuracy of the measurements. This behaviour is expected, since metals do not show relevant dependence of SEY as a function of temperature and the a-C coating is an electrical conductor. More in general the excitations producing secondary electrons are of the order of eV whereas the changes in the electron distribution in the density of states occur in a range of kT or tens of meV. The SEY measured for various coatings has been correlated with the shape of the C1s line measured in XPS. In particular, Fig. 2 shows four carbon spectra for coatings having different SEY. The maximum intensity was normalized after subtraction of a constant, calculated as the average value in the region 278-281 eV binding energy. The intensity in the region 287-290 eV is correlated to the δ_{max}, as shown in Fig. 3, for 289 eV binding energy. Higher intensity at 289 eV corresponds to lower δ_{max}. The correlation is not due to the presence of other elements, which could give intensity in this region as a consequence of their bonding to carbon. In particular the amount of oxygen measured on the samples by XPS

after production (between 0 and 16%at) is not related either with the δ_{max} or with this intensity.

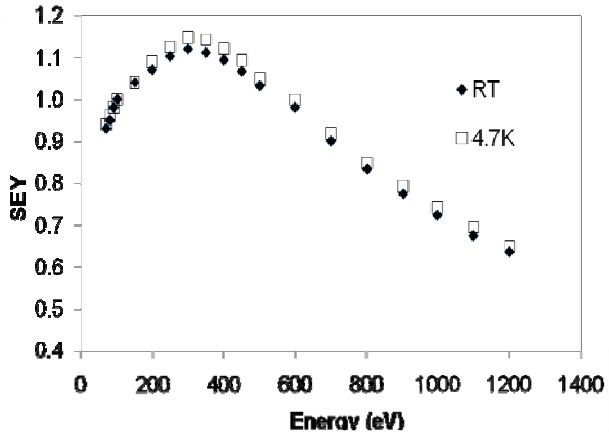

Figure 1: SEY of a-C coating at RT and 4.7 K.

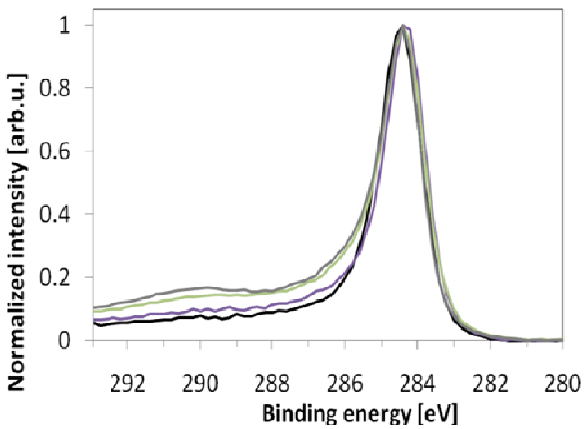

Figure2: C1s normalized intensity. The curves correspond to samples with δ_{max}= 1.15, 1.07, 0.99, 0.88 (bottom to top).

The change of SEY with time (aging) has been studied by storing the samples in different environments: in air wrapped in aluminium foil (up to 14 months), in air in a non-sealed polystyrene box (up to 14 months), in stainless steel vessel filled with N_2 (up to 9 months; 1 mbar after evacuation), in a stainless steel vessel in static vacuum (up to 4 months; before closing the vessel, the sample was pumped down to 10^{-7} mbar) and in desiccators. The worst case is found for the storage in a polymer box (Fig. 4), for which the SEY increases well above the possible statistical uncertainty. It is remarkable that just wrapping the samples in Al foil prevents the aging. This method can easily be applied to vacuum chambers, by closing them at both ends with Al foil. All other cases are similar to the samples wrapped in Al foil. The cause of the aging is not yet identified, but a correlation could be established between the increase of SEY and the amount of oxygen detected on the surface by XPS, independently of the storage type. This behaviour is shown in Fig. 5, where the samples from three different coating runs and all types of storage are included. It could be an indication that oxygen or oxygen related species – as water or hydrocarbons carrying oxygen containing groups – are responsible for the increase of the SEY with time of storage. However, the increase of oxygen with time could also be only a side effect, simultaneous with the accumulation of hydrocarbons on the surface. In XPS it is difficult to distinguish the adosorbed hydrocarbons from the carbon substrate, but a clear intensity in the spectrum region of C to O bonds (288eV) indicates at least their presence. It is important to reemphasize that before aging no correlation has been found between the initial level of oxygen measured by XPS and the SEY.

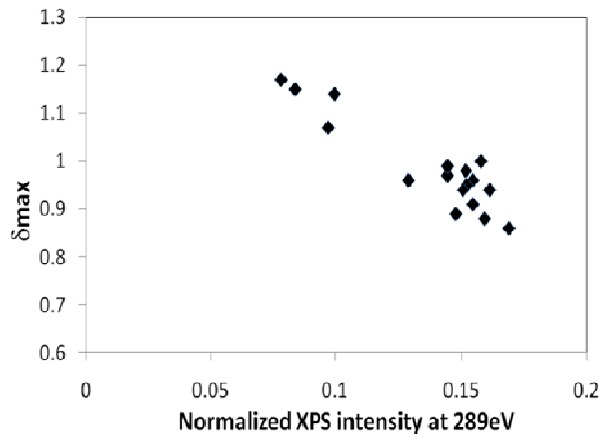

Figure 3: δ_{max} as a function of the C1s XPS intensity.

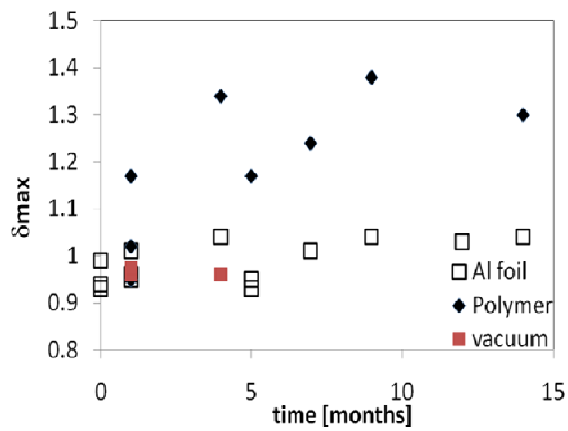

Figure 4: Change of the SEY as a function of time and storage.

Irradiation with 500 eV electrons on a sample, which has undergone aging, recovers the initial value of the SEY, as shown in Fig. 6. The conditioning down to δ_{max}=1.0 requires a dose in the 1e-3 C/mm^2 range. As a comparison, this would lead to a value of δ_{max} = 1.1-1.3 on copper conditioned in the same way [5]. To be noticed, the peak at 288 eV of binding energy related to C to O bonds vanishes upon irradiation. The fast increase of the

SEY after 24h air exposure can indicate that the surface is more reactive than the initial one, before aging.

Thermal treatment in UHV, without further air exposure as in a common bake-out procedure, was investigated on few samples as another method to lower the SEY of aged samples. Heating at 120^{0}C for a long time (42h) decreases the δ_{max} to 1.13 for a sample which had an aging up to a value of 1.33, but does not enable its recovery to the initial value of 0.97. The complete recovery is possible at 200^{0}C (sample with initial δ_{max} of 1.08) as shown in Fig. 7 for another sample.

After 10 min sputtering with Ar+ at 0.6 μA/cm^2, the δ_{max} of an aged a-C sample can be reduced to its initial value. A clear correlation between the δ_{max} and the remaining amount of O% can be seen. However, in the case of accelerator components or vacuum chambers, the in-situ treatment of surfaces is almost always excluded with the possible exception of bake-out and beam induced electrons.

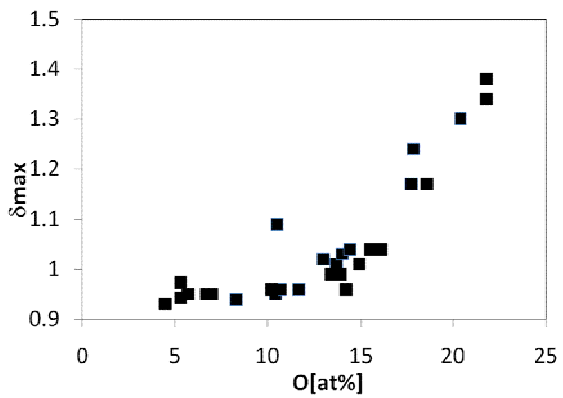

Figure 5: Maximum SEY as a function of accumulated oxygen amount on the surface.

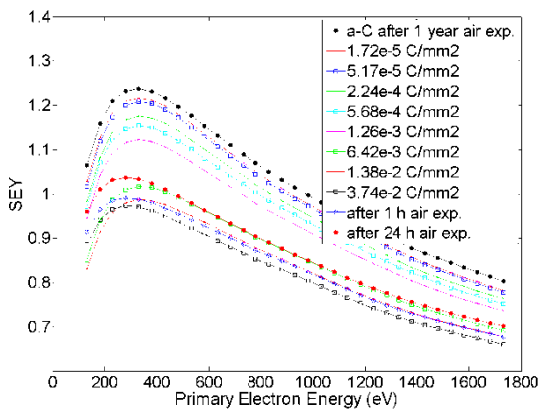

Figure 6: Evlution of SEY as a function of irradiation electron dose. The initial SEY before aging was 0.96.

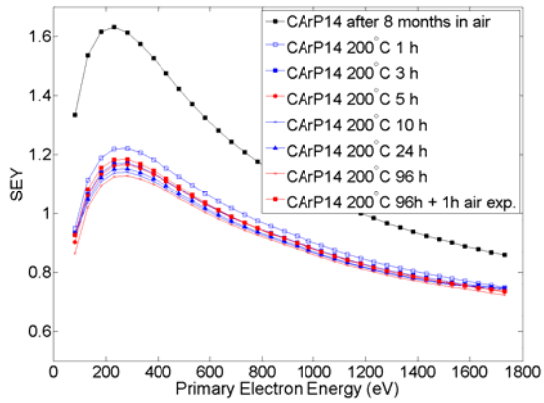

Figure 7: SEY as a function of heating time at 200^{0}C.

CONCLUSIONS

The SEY of a-C carbon coatings is independent of temperature in the range between 4.7K and RT. The SEY of different coatings changes in a small range of values around δ_{max}=1. The intensity of the C1s line on the high binding energy side, namely around 287-290 eV correlates with the measured δ_{max} in the sense that a higher intensity generally leads to a lower value of the SEY. More investigations are necessary in order to understand the origin of this relation. Instead the SEY of freshly prepared coatings does not depend on the amount of oxygen detected on the surface by XPS. Only after long term aging, the amount of oxygen is found to increase together with the SEY. Aging in air is strongly reduced by wrapping the samples in aluminium foil compared to storage in a polymer box. This protection by aluminium foil is also applicable to large vacuum chambers. Recovery of the SEY after aging can be obtained by electron irradiation, thermal treatment in UHV or ion sputtering.

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