

Thin Glassy Carbon Coating for Protection Against Oxygen Penetration into the C₆₀ Fullerite

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ABSTRACT

Protection of fullerites against oxygen penetration is one of the main challenges for a fabrication of fullerene-based electronic devices. In this article, we report on an experimental evidence for such a protection using

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a very thin ($\sim 50 \text{ \AA}$) layer of glassy carbon produced by a low temperature pyrolysis of sucrose.

Key Words: C₆₀ films; Oxygen penetration; EPR signal; Glassy carbon.

INTRODUCTION

The C₆₀ crystal structure contains large interstitial volume that allows atmospheric oxygen to diffuse readily into this solid. A variety of experiments suggests that oxygen penetration results in a serious degradation of electronic and optical properties of C₆₀ films (for review, see Refs.^[1,2]). Recently from Ref.^[2], we demonstrated that such degradation is accompanied by the generation of recombination and/or scattering centers with deep acceptor states. The latter correlates with the appearance of a sharp EPR signal with $g \sim 2.0026 \pm 0.0001$, associated with oxygen induced C₆₀⁺ centers. Thus, it was shown that monitoring of evolution of the C₆₀⁺ EPR signal is a relevant technique for studying the interaction of C₆₀ films with oxygen and the corresponding impact on their properties.

Protection of C₆₀ films against oxygen penetration is one of the main challenges for fabrication of fullerene-based electronic devices. Here, we report experimental evidence for such a protection by a very thin ($\sim 50 \text{ \AA}$) layer of glassy carbon monitoring the oxygen penetration by EPR.

EXPERIMENTAL DETAILS

C₆₀ thin films were grown on optical glass substrates of $3.5 \times 20.0 \text{ mm}^2$ area using vacuum evaporation of pure C₆₀ powder (Hoechst AG "Super Gold Grade," $>99.9\%$). The vacuum chamber pressure was maintained at about 8×10^{-7} Torr. Detailed description of the deposition conditions is given elsewhere.^[3] The thickness of all C₆₀ films under the present study was about 200 nm. Two samples (S1 and S2) were grown under identical conditions: the deposition rate was of 6 \AA/s and the substrate temperature of 300 K. However, immediately after the vacuum C₆₀ deposition was completed, sample S2 was covered by a thin ($\sim 50 \text{ \AA}$) layer of glassy carbon produced by low temperature pyrolysis of sucrose (C₁₂H₂₂O₁₁).

EPR spectra were recorded using a Bruker EMX-220 X-band digital spectrometer at room temperature. The amplitude of 100 kHz modulations and the microwave power level were chosen as 0.1 mT and 200 μ W,



correspondingly, for preventing saturation and obtaining better signal-to-noise ratio. EPR signals' processing was done using Bruker WIN-EPR Software.

RESULTS AND DISCUSSION

In accord with our recent results,^[1,2] sample S1 in as-grown state was found to be practically EPR silent [Fig. 1(a)]. When samples were exposed to air under room light (by room light we mean a combination of daylight and fluorescent room light), a sharp ($\Delta H_{pp} = 0.124 \pm 0.005$ mT) EPR signal characterized by Lorentzian line shape and $g = 2.0026 \pm 0.0001$ appeared [Fig. 1(a)]. A weak EPR signal with $g = 2.0006 \pm 0.0001$ belongs to a standard sample containing a minute amount of thermal defects in quartz (Fig. 1). As it has been mentioned, the $g = 2.0026$ signal is associated with

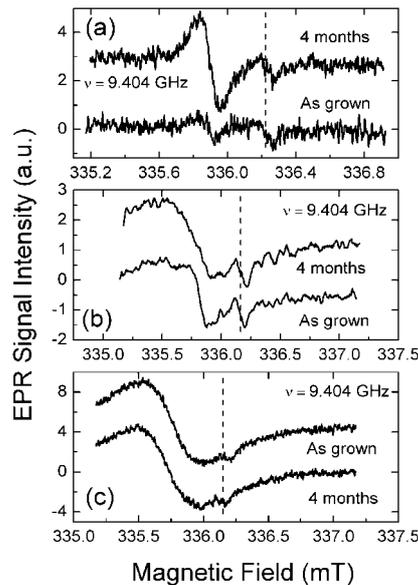


Figure 1. Room temperature EPR spectra of three film samples: (a) non-coated C₆₀ film on a glass substrate (sample S1); (b) C₆₀ film coated by protecting glassy carbon layer on a glass substrate (sample S2); and (c) protecting glassy carbon coating layer on a glass substrate. In each plot, the lower spectrum was obtained on as-grown sample and the upper spectrum on the same sample after 4 months of light/oxygen exposure. Dashed vertical lines mark the position of the weak standard sample EPR signal.

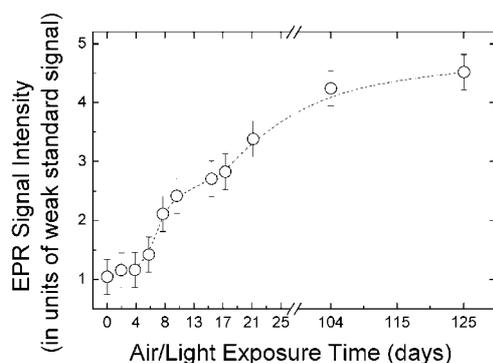


Figure 2. Dependence of the C_{60}^+ EPR signal peak-to-peak intensity on the ambient light/oxygen exposure time measured on the initial non-coated C_{60} film on glass substrate (sample S1). The signal is represented in units of the intensity of the weak EPR signal from a standard sample, see Fig. 1.

light/oxygen induced C_{60}^+ paramagnetic centers. The evolution of EPR signal that has been monitored for 4 months showed significant increase in its intensity [Figs. 1(a) and 2]. All other EPR signal parameters remained unchanged.

Contrary to the S1 sample, the S2 sample in both as-grown state and after 4 months of the exposure demonstrates the identical broad ($\Delta H_{pp} = 0.49 \pm 0.01$ mT) signal characterized by the same, like C_{60}^+ EPR signal, $g = 2.0026$ [Fig. 1(b)]. The latter was also observed in EPR spectra of pure glassy carbon film, grown on the same glass substrate, and remained unchanged during light/oxygen exposure period [Fig. 1(c)]. Signals of this type are similar to those of carbon dangling bonds^[4] and may be attributed to defects in glassy carbon. The absence of the characteristic light/oxygen induced C_{60}^+ EPR signal in the S2, sample undergone similar to the S1 sample external conditions, let us concluding that the glassy carbon layer effectively protects the fullerite film from the oxygen penetration.

CONCLUSION

Experimental evidence for the protection of C_{60} films against oxygen penetration by a very thin (~ 50 Å) layer of glassy carbon has been demonstrated using monitoring of the oxygen penetration by EPR technique.



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