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Incorporation of selenium into carbon films by chemical vapor deposition

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Abstract

The introduction of selenium into carbon thin films was tried by a chemical vapor deposition (CVD) from selenium and acrylic acid vapors at 900 °C on different substrates. No conclusive evidence of intercalation of selenium into the carbon films was obtained, but the materials contained a small quantity of selenium. Electrical conductivity of the film deposited on a silicon wafer was four times higher than that on a quartz glass.

Keywords: Carbon films; Selenium; Thin films; Chemical vapour deposition

1. Introduction

The graphite intercalation compounds (GICs) are a family of materials that present a variety of different physical phenomena, e.g., magnetic transitions and superconducting behavior [1]. Recently, the possibility of superconductivity at higher temperatures was reported in graphite intercalated with potassium and C_{60} [2]. Besides this, these new materials have attracted attention because of their battery performance and catalytic properties [3,4]. These facts renew the interest in finding more compounds or elements that may be intercalated into graphite to give novel physical properties.

In this paper, we report our attempts to intercalate selenium into carbon films. To our knowledge, only one report exists in this respect [5].

2. Experimental

Our method was previously used to intercalate potassium oxide [6], and consisted of thermal decomposition of acrylic acid, using a hot wall reactor. The hot wall chamber was formed by a quartz tube of 2 cm of diameter and 100 cm long, which was located in the interior of a horizontal furnace. The acrylic acid (CH_2=CHCOOH, melting point of about 140 °C) was evaporated from a tungsten crucible inside the quartz tube at the place where the temperature was around 300 °C. Selenium powder (melting point of around 217 °C) was also evaporated from another tungsten crucible placed in close proximity to the acrylic acid crucible. A flow of helium gas of around 10 sccm was introduced into the reactor to produce the drag of both vapors, selenium and carbon, through the hot zone at about 900 °C, where the deposition of the thin films took place on the substrates. The substrates used were high resistivity silicon wafers with their surface oriented parallel to the (100) plane, fused quartz and pyrolytic graphite. Thickness of around 500 nm can be obtained in approximately 15 min.

3. Results and discussion

Energy-dispersive spectroscopy (EDS) microanalysis indicated that the approximate atomic ratios of C/Se on different substrates are 98/2 on silicon, 99/1 on quartz and 99.8/0.2 on graphite.

Fig. 1 shows X-ray diffractograms of the films taken with Cu Kα radiation, deposited on the three different substrates. For the film deposited on quartz, the broad peak in the diffractogram was a superposition of two peaks from the quartz substrate and carbon film deposited on it, and so the observed peak was decomposed into two Lorentzian peaks, shown by the dotted curves in Fig. 1. The common feature in these spectra is a broad peak centered around $2\theta = 25.6°$, which is related to 002 diffraction peak of carbon with spacing around 0.347 nm. This value of the spacing is within the reported interval of values for carbon films subjected to different heat-treatment temperatures, and the broadness of the peak is indicative of a high degree of disorder in the phase [7]. These facts may be due to an incomplete graphitized state caused

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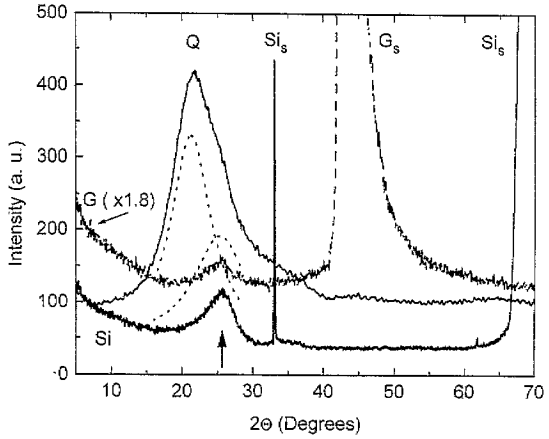

Fig. 1. X-ray diffractograms of carbon films deposited on silicon wafers (Si), fused quartz (Q), and graphite substrates (G). The vertical arrow indicates the common broad peak in the three films. The dotted curves are Lorentzian components of the X-ray signal obtained from the film deposited on the fused quartz substrate; the peak at lower angle corresponds to the substrate. The peaks marked as Si_s and G_s correspond to the silicon and graphite substrates, respectively.

by the low deposition temperature. It is important to mention that, although Grigorian et al. [5] reported on the synthesis of a pure stage-3 GIC with selenium using a similar method, we did not observe any diffraction peaks corresponding to the intercalation structure in the X-ray diffractogram.

The effect of introducing selenium into the carbon films on electrical conductivity σ is shown in Fig. 2 as a function of the reciprocal of the temperature of measurement, $1/T$. The electrical conductivity at room temperature in the films deposited on silicon is approximately four times higher than that on quartz. This may be related to the difference in selenium content in both films, mentioned before, although some uncertainty may exist in the carbon quantification. It was difficult to assign a unique activation energy to these data due to a continuous curvature in the conductivity in the whole range of temperatures from 2 to 300 K. In the case of films deposited on the graphite substrate, reliable measurements were difficult to make due to the high conductivity of the substrate.

4. Conclusions

Carbon thin films with small quantities of selenium were synthesized, but no evidence of intercalation was found. Electrical conductivity of the films appeared to increase on the introduction of selenium.

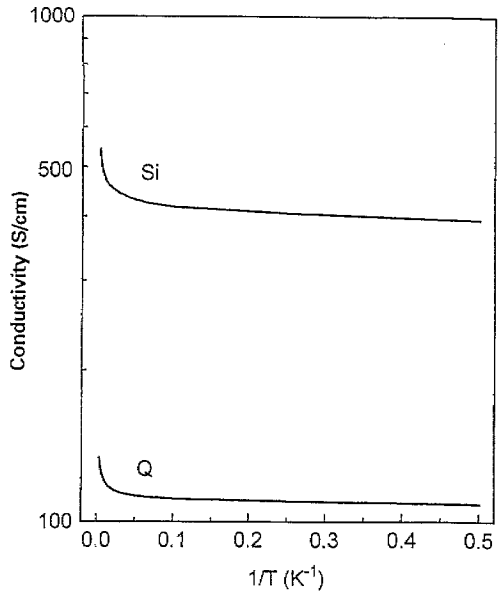

Fig. 2. Electrical conductivity vs. $1/T$ of films deposited on silicon (Si) and fused quartz (Q) substrates.

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