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## LETTER TO THE EDITOR

# Incorporation of potassium into graphitic films by chemical vapour deposition

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**Abstract.** In this work we report on the synthesis of potassium intercalated into graphitic thin films by a chemical vapour deposition method. We use acrylic acid as a source of carbon and a solution of KOH in methanol as the source of potassium; the vapours of both compounds are thermally decomposed at 800 °C onto fused silica substrates to form thin films. Our analysis indicates that the material obtained consists of a mixture of stages I and II of potassium–oxygen graphite intercalation compounds.

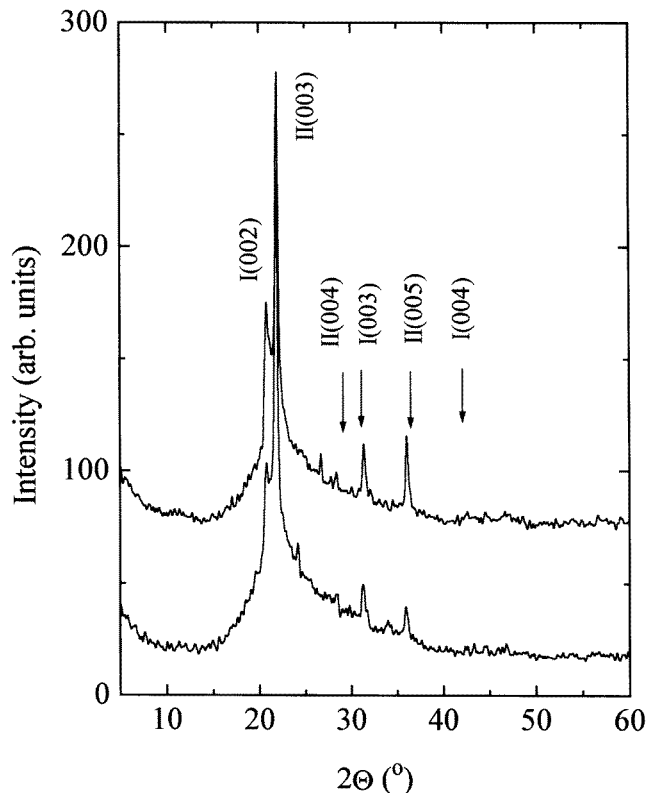
Graphite intercalation compounds (GICs) are of great interest from a purely scientific and technological point of view [1]. The existence of superconductivity in the compounds  $C_8M$  ( $M = K, Rb, Cs$ ) at low temperatures [2], and more recently the possibility of superconductivity at higher temperatures in  $C_8K$  exposed to the fullerene  $C_{60}$  [3] renews interest in finding even higher temperatures for superconductivity in GICs. From a technological point of view, GICs have attracted attention because: (i) they can be used as battery electrodes, (ii) for their catalytic and selectivity properties, and (iii) because of their absorption of some gases [2, 4].

GICs have been obtained using a variety of methods, mainly involving reaction with the vapour phase of the intercalates, and by chemical and electrochemical methods. Other methods involve the deposition of, for example, alkali atoms onto monolayers of graphite obtained by the thermal decomposition of some hydrocarbon in its vapour phase [5].

In the present work we report on an alternative approach to the synthesis of graphite intercalation compounds by chemical vapour deposition methods. In particular, we start with acrylic acid as the source of carbon, and a solution of KOH in methanol as the source of potassium. The vapours of the two compounds are decomposed at high temperatures on fused silica substrates to form thin films of graphite intercalated with potassium and oxygen.

The experimental procedure is as follows. The chamber is a typical hot-wall reactor, consisting of a quartz tube (2 cm diameter and 100 cm long) located in the interior of a horizontal furnace. The acrylic acid ( $CH_2=CHCOOH$ ) was evaporated from a tungsten crucible placed in the quartz tube at the beginning of the hot zone where the temperature was around 300 °C. The melting point of the acrylic acid is around 140 °C, and readily evaporates above this temperature. To introduce the potassium into the reactor, a 1 N solution of KOH in methanol was prepared. A helium gas flow of 10 sccm was introduced into the reactor through a glass flask containing the KOH solution (the flask was maintained at 90 °C). In this manner, vapour flow through the reactor produces the deposition of thin films on the substrates located in the hot zone at  $\sim 800$  °C. The thickness of the films can be controlled by varying the deposition time and/or the temperature of the acrylic acid source. In our case, a film thickness of around 1000 Å can be obtained in approximately 30 minutes.

XPS analysis of two samples, obtained in different runs, grown on fused silica substrates, indicates the existence of carbon, potassium and oxygen. The atomic average ratio of C/K is around 7.3; it is difficult to quantify the oxygen content because the oxygen signal from the substrate overlaps with that from the film. In any case, we expect that the material obtained contains oxygen due to the fact that the precursors contain oxygen.



**Figure 1.** X-ray diffractograms of two potassium–oxygen graphite intercalation films using Cu  $K\alpha$  radiation. I and II represent the peaks corresponding to stages I and II of intercalation.

In figure 1 we present the x-ray diffractograms of the two samples. We cannot index these spectra as a pure phase, but instead, we index them as a combination of two intercalation stages, namely, stages I and II of a potassium oxide ( $KO_x$ ) intercalated into the graphitic layers. In figure 1 we use arrows to indicate the position of the corresponding peaks labelled I(00 $l$ ) and II(00 $l$ ) for stages I and II, respectively. The positions in the scattering angle indicated by arrows were calculated based on the first peak (highest peak) for each stage. The  $c$  axis repeat distance  $I_c$  is 8.54 Å and 12.15 Å for stages I and II respectively, in close agreement with the reported results by Mordkovich *et al* [6] for potassium–oxygen graphite intercalation compounds.

Our XPS analysis indicates that the ratio C/K is around 7.3; comparing this result with the ideal C/K ratio (4 and 8 for stages I and II, respectively [6]) leads to the conclusion that in our samples the majority phase corresponds to stage II. This is confirmed by the x-ray spectra, where the first peak associated with stage II is somewhat higher than that of stage I.

In conclusion, we have presented an alternative approach to the synthesis of potassium–oxygen graphite intercalation compounds in the form of thin films. The obtained films consist of a combination of stages I and II of a potassium oxide intercalated into graphitic layers. We believe that having GICs in the form of thin films may be useful for application of this material in thin film devices.

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## References

- [1] Fischer J E and Thompson T E 1978 *Phys. Today* July
- [2] Zabel H and Solin S A (ed) 1992 *Graphite Intercalation Compounds II (Springer Series in Materials Science 18)* (Berlin: Springer)
- [3] Fuhrer M S, Hou J G, Xiang X D and Zettl A 1994 *Solid State Commun.* **90** 357
- [4] See for example:  
1995 Graphite intercalation compounds: host effects (M Inagaki, Guest Editor) *Synth. Met.* **73**
- [5] Nagashima A, Tejima N and Oshima C 1994 *Phys. Rev. B* **50** 17487
- [6] Mordkovich V Z, Ohki Y, Yoshimura S, Hino S, Yamashita T and Enoki T 1994 *Synth. Met.* **68** 79