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STRUCTURAL STUDIES OF CARBON SOOT FILMS

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ABSTRACT

The results of structural (SEM, TEM, ED and XRD) studies of carbon soot films synthesized by graphite are welding technique have depicted that these films are comprising of an assembly of micro-crystalline clusters of different sizes along with amorphous carbon dust. It is worthwhile to mention over here that the sizes and their concentration of micro-crystalline clusters are influenced by the film growth conditions. SEM, TEM, ED and XRD studies have shown that micro-crystalline clusters are having hexagonal symmetry.

Keywords: Structural Studies, Micro-Crystalline Clusters and Carbon Soot Films.

INTRODUCTION

Carbon is the sixth most abundant element in the universe. It is found in many different compounds and plays a dominant role in our life. In addition to the earlier known two crystalline allotropic forms namely diamond and graphite of pure carbon, a new third form has been discovered [1,2]. This third form, known as fullerene based on closed cages of carbon atoms. This is most symmetric and is a perfect sphere of 60 carbon atoms. They discussed the possible structure that might explain the special stability of this molecule (C_{60}) and speculated a closed spherical cage made of 20 hexagons and 12 pentagons. They even named the new molecule buckminsterfullerene in the honour of the structural designer Buck Minister Fuller. The development of experimental methods [1, 2] for synthesizing bulk quantities of stable nano-clusters of carbon viz. C_{60} and C_{70} has opened many avenues of research. The C_{60} became more attractive after the observation of superconductivity in doped C_{60} [3-16]. It has been reported that it is possible to optimize the yield of particular clusters [1, 2, 9, 17] by controlling the inert gas pressure in the evaporation chamber of standard graphite are welding synthesis technique. It could be interesting to synthesize new classes of microscopically heterogeneous materials of a variety of carbon clusters by controlled assembly using the new synthesis method and modifying their properties. It has been seen that optical spectra of nanoclusters can be tuned in wavelength by simply varying the cluster size [10-17]. Therefore it was thought that the energy band structure of the carbon soot films could also be modified by the assemblage of a variety of clusters [17-27]. We have characterized the as gown carbon soot films, synthesized by the standard graphite arc welding technique, using X-ray diffraction (XRD), scanning electron microscopy

(SEM) and electron diffraction (ED). The results are presented and discussed here in the present study.

SYNTESIS OF FILMS

Carbon soot films were synthesized using standard graphite are welding technique. For this purpose spectroscopic pure graphite electrodes were used for deposition of carbon soot film under an inert atmosphere of helium gas [17]. The details of preparation are given in Table 1. Usually one arc discharge lasted for several seconds and several such discharges were used to deposit carbon soot film of 1 μm thicknesses.

Table 1: Synthesis conditions of carbon soot films.

Film Code	Helium gas pressure (Torr)	Number of discharges
F2	100	4
F4	150	7

CHARECTERIZATION OF FILMS

As grown carbon soot films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and electron diffraction (ED) in order to study structural properties. SEM studies were carried out using JEOL JSM – 840 scanning electron microscope. ED studies were done using JEOL JEM – 2000 FX transmission electron microscope. XRD studies were done using Rich and Seifert ISO Debyeflex 2002 diffractometer with CuKα radiation.

RESULTS AND DISCUSSION

The SEM micrographs taken from various parts of the same film (F4) were almost identical, indicating a macroscopic homogeneity (Figure 1). On a micron scale the film consisted of polygonal granules, having rather complex growth of several crystallites resulting from different pressures in successive discharge for growth of films. Almost all the granules were orientated with their flat surfaces nearly parallel to the surface of the substrate. The morphology of grains seems to be spherical at higher magnification.

It was observed from the electron diffraction (ED) studies of the films that the individual granules were crystalline and clearly belong to hexagonal symmetry (Figure 2). However in most of the patterns presence of multiple spots corresponding to both the same and different lattice parameters indicated twinning as well as coexistence of several different crystalline phases with similar crystal structures (Figure 3).

ED studies have also shown that most of the microcrystals were oriented with their c-axes perpendicular to the surface of the substrate. The diffraction patterns taken from integranular material were diffused halos indicating its amorphous structure. Similar results were obtained on several samples from different batches. Prior to taking the ED data the samples were scanned in TEM in order to locate the proper crystallites of size ≈ 1 μm.

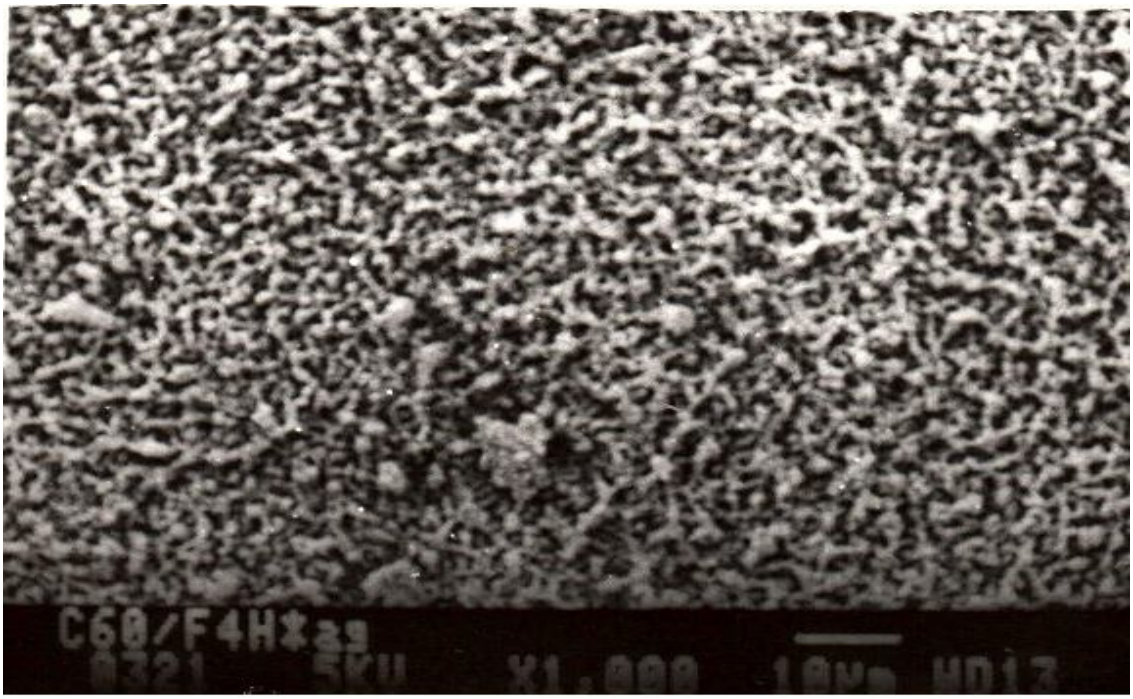

Figure 1 : Scanning electron micrographs of F4 film at lower magnification. Magnification, scale and operating voltage are shown on the micrographs.

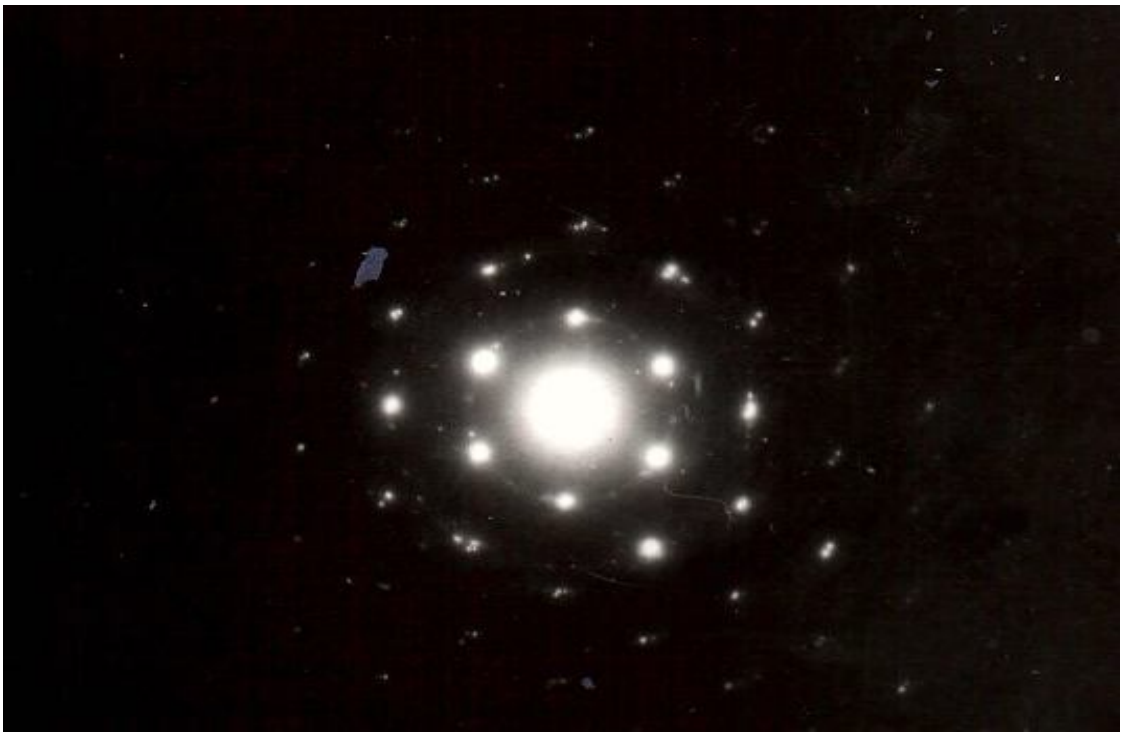

Figure 2: Selected area ED pattern taken from the batch of F4 film on crystallite of size ~ 0.2μm. The pattern clearly depicts the hexagonal symmetry of the crystallite. Multiplicity of spots indicates that the crystallite is a composite [17, 21].

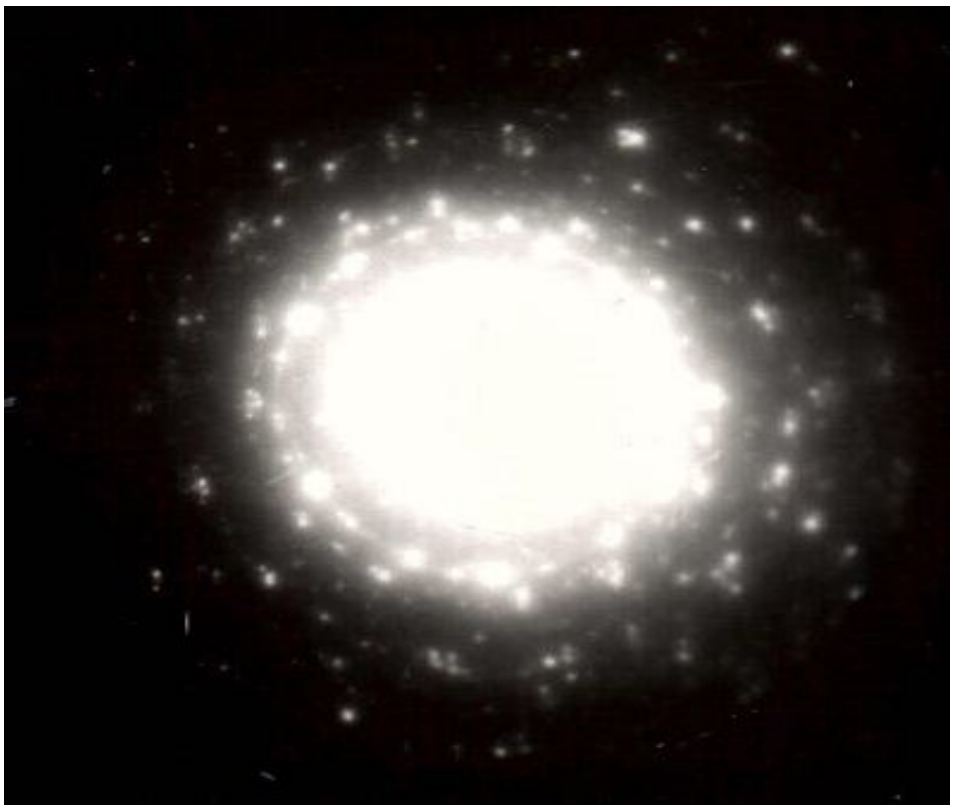

Figure 3: Selected area ED pattern taken from the batch of F4 film on crystallite of size ~ 0.2μm. The pattern clearly indicates the coexistence of several hexagonal crystalline phases having slightly different lattice parameters. Multiple twinning of the crystallites is also evident [17, 21].

It is noted from the XRD studies that only one clear diffraction broad diffraction peak was observed from each film within the range of accessible rotations of the film and this particular orientation occurred when the incident x ray beam was directed parallel to the surface of the substrate i.e. (001) planes of the crystallites. The observed broad diffraction peak with a fine structure obviously is due to the superposition of several underlying peaks [17, 21]. Taking into account the observed hexagonal symmetry the fact that all the reflections signals obtained lie within a narrow range (2Θ = 9.2 to 10.3^0) overlapping with the (100) peak of crystalline C_{60} ($2\Theta = 10.02^o$) [1] and these signals are coming from a set of distinctly different carbon clusters of similar sizes. The crystal lattice parameters calculated using the relation (a) $\{1\backslash d^2 = 4/3(h^2 + hk+k^2)/a^2 +1^2/c^2\}$ from the ED spectra are given in Table 2 [17, 21]. The phases with strongest reflections are referred as major. The relative concentration of the constituent clusters in the carbon soot films from several different batches estimated from XRD data are given in Table 2.

Table 2: Lattice parameters of the constituent carbon clusters crystallites of films calculated from the ED pattern under hexagonal symmetry using the procedure given in [17, 21].

Film code	Lattice Parameter of Major Phases ($a \pm 0.05$)A^0		Lattice Parameter of Trace of minor phases ($a \pm 0.05$) A^0		
F2	10.40	9.90	10.95,	10.10,	9.50
F4	10.48	9.95	11.00,	10.15,	9.55

It is worth mentioning here that the presence of various minute impurity clusters (C_{30} to C_{130}) was detected by high – resolution transmission electron microscopy even in purified C_{60} solids [11]. These cage shaped stable molecules can form separate crystalline phases within the C_{80} lattice. But the essential difference between our samples and those reported in [1] is that we have grown the samples having constituent crystalline phases in comparable quantities, while in purified C_{60} solids they are only as impurity in trace quantities.

CONCLUSION

Characterization of carbon soot films using X-ray diffraction, scanning electron microscopy and electron diffraction techniques has shown that the films contain a composition of carbon clusters of different concentrations and sizes which could be affected by varying the film growth conditions. The films comprised of highly textured microcrystals of hexagonal symmetry, immersed in amorphous carbon dust. It has been established that by the graphite arc welding technique one can grow carbon nanoclusters in different compositions simply by varying number and duration of arc discharges and controlling the inert gas pressure in the evaporation chamber of graphite arc welding technique.

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REFERENCES

1. Kratschmer,W. *et al*,(1990). *Nature*, **347**: 254
2. Kratschmer,W. *et al*,(1990). *Chem. Phys. Lett.*, **170**: 167
3. Hebard,A.F. *et al*, (1991).*Nature*, **350**: 600 (1991).
4. Rosseinsky,M.J. *et al*,(1991). *Phys, Rev. Lett*. **66**:2830
5. Holozer,K. *et al*,(1991). *Science,* **252**: 1154
6. Kelty,S.P. *et al*,(1991). *Nature*, **352**: 223
7. Tanigaki,K. *et al*,(1991). *Nature*, **352**: 222

8. Amotz,D.B. et al,(1991). *Chem. Phys. Lett*., **183**: 149

9. Stugerwalk,M.L. and Brus,L.E.(1989). *Ann. Rev. Mater, Sci.,* **19**: 471

10. Wang , Su and Buseck,P.R.(1991). *Chem, Phys. Lett.,* **182**:1

11. Haddon, R.C. *et al*,(1991). *Nature*, **350**: 320

12. Bethune,D.S. *et al*,(1990). *Chem. Phys, Lett.,* **174**: 219

13. Garell, R.L.*et al*, (1991).*J. Am. Chem Soc*. **113**: 6302

14. Raghavachari,K. and Rohifing,C.M.(1991). *J. Phys, Chem*., **95:** 5788

15. Deniell,R. *et al*,(1992). *Solid State Commmun,* **81**: 257.

16. Boekholt,M. and Guntherodt,G.(1991). *Physica* C,**181**: 179

17. Gringoryan,L.S. *et al*,(1991). *Solid State Commun,* **81**:853

18. Grigoryan,L.S. *et al,*(1992). *Journal of Raman Spectroscopy,* **23**: 127-129.

19. Grigoryan, L.S. *et al,*(1992). *Chemical Physics Lett*., **199**: 360-364.

20. Sudhakar,N. *et al*,(1992). *Physics Lett*. A., **170**: 235.

21. Sharma,S.V. *et al*, (1993).*Physics Lett*. A, **177**:153 .

22. Narayan,J. *et al,* (1995).*J. Sci. Technol*., **9**:753.

23. Wei,Q *et al*, (1999).*Composites*: Part B **30:** 675

24. Sharma,S.V.(2003). *School Science*, XLI – **1**: 3

25. Sharma,S.V.(2006). *School Science*, **44** (3): 18.

26. Epelbaum,E. *et al*, (2011).*Phys. Rev. Lett*., **106**:192501

27. Shin,S.J. *et al*, (2012).*Carbon*, **50:** 5340.