NATURAL GRAPHITE FROM NEOPROTEROZOIC PSAMMITIC GNEISS, INANALO MOUNTAIN, SOUTHERN MADAGASCAR by G. Parthasarathy, A.S. Collins and T.R.K. Chetty. Jour. Geol. Soc. India, v.65(2), August 2006, pp.176-180.

Suresh N. Karkhanis, C/o Analytical Research Laboratory, Department of Polymer and Petroleum Engineering, MIT Engineering College, Kothrud, Pune - 411 038 comments:

At the outset, I must say that the paper made an interesting reading. The XRD pattern as shown in their Fig.2 (p.177) shows a very clean base line. In our experience, this is very difficult to achieve. The rock type encountered by the authors contains carbonates and silicates. The HCl-HF treatment – its is imperative to remove quartz completely, because quartz peak overlaps the (*002*) reflection of graphite (French, 1964; Karkhanis, 1975).

One of the major problems that arises in using HF is that with more basic rocks, some insoluble fluorides of the type $MgAlF_5.H_2O$, $NaAlF_4xH_2O$ and occasionally Fe^{2+}(Al, Fe^{3+}), $F_5.H_2O$ are formed (Karkhanis, 1977a).

Complete removal of mineral material is in practical terms nearly impossible. They appear as synthetic mineral artifacts in XRD patterns. Hence it is not instrumentally feasible to get a smooth XRD baseline. However, this can be prevented to lesser extent by treating the HF residue by strong complexing agent such as 2.5% boric acid or very hot 1N HCl for 24h.

Table below shows all the possible *hkl reflections* (space group P63/mmc for graphite with corresponding *d* values in Angstrom with corresponding relative intensity (RI) (Berry and Thompson, 1962).

Indicies	*d*(Å)*	Relative intensities
002	3.36	10.0
100	2.13	1.0
101	2.033	5.0
102	1.80	0.5
104	1.678	8.0
103	1.544	1.0

* *d* values from Berry and Thompson (1962)

If the Madagascar graphite is sheared as claimed by the authors, I would strongly suggest the authors to look for perturbations due to the extra indices for rhombohedral modification in their samples. As it is they are getting a very clean baseline for their XRD pattern, they should turn this condition into a major advantage. Our experiments have shown that carbon from inorganic precursors – carbonate minerals, can form ordered layers at relatively low temperature of 600°C at 1Kb pressure (Karkhanis, 1977b).

Indicies#	*d*(Å)*	Relative intensities
102/3	2.087	1.0
104/3	1.961	0.5
108/3	1.627	0.5

* *d* values from Berry and Thompson (1962)
\# (Lipson and Stockes, 1942-1943)

G. Parthasarathy, A.S. Collins and **T.R.K. Chetty**, NGRI, Hyderabad – 500 007 reply:

First of all we would like to thank Dr. S.N. Karkhanis (SNK) for his interest in our paper and also in bringing out the list of papers published in abiogenic graphite during 1975-77 to our attention.

SNK states that it is very difficult to achieve a very clean X-ray base lines and also complete removal of fluoride mineral materials is in practical terms impossible. He also presented a table of XRD data on graphite published in earlier literatures (Lipson and Stockes, 1942-43).

We adopted the method described by Wedeking et al. (1983) for the extraction of carbonaceous matter from the rock samples. About 20g of the samples were finely ground to 120 mesh and the powdered samples treated with the acid mixtures of 60% HF and 37% HCl in the ratio 5:2 for 18 hours to dissolve acid soluble minerals. The resultant residue was washed with 1000 ml of double distilled water. The carbonaceous matter residue was then dried at 80°C for three hours. Fluorides were normally not observed in the samples prepared by this method. If fluorides were detected (in sulphide contaminated rocks), the samples were treated with 1:2 M $AlCl_3$ for 24 hours at room temperature. This would remove any traces of fluoride complexes (Wedeking et al. 1983). The method of extraction was not described in detail in the short communication as it was discussed in our earlier publications. We have followed this method in all our earlier studies on carbonaceous materials since 1997. The list of papers is not mentioned here due to space constraints.

In modern powder X-ray diffraction methods it is conventional to present the smoothed XRD pattern, in which the base line would be smooth for the well crystalline powdered sample. A broad peak is usually observed for glassy materials for which the long-range order is absent.

The studied Madagascar graphite sample M03-22 was collected approximately 20 km east of the shear zone (see p.176 in Parthasarathy et al. 2006b) and we have not claimed anywhere that the studied graphite was sheared. We have observed rhombohedral phase of graphite in the Eastern Ghats shear zone samples as well as Achankovil shear zone samples where the sample had been sheared (Chetty and Parthasarathy, 2005; Parthasarathy et al. 2006). We have also observed in our earlier studies on carbonaceous matter from Archaean Dharwar craton, that the well-crystallized graphite forms at temperatures as low as 564°C (Sharma et al. 1998). The XRD data on the Madagascar graphite sample has been found to be in very good agreement with the XRD spacing and intensity presented in the JCPDS cards 23-64 and 41-1487. The tables given by SNS are currently outdated. The standard and update XRD data on graphite is presented in Table 1 for the sake of researchers and readers of the JGSI.

Table 1. Standard Powder X-ray diffraction data on graphite

JCPDS Card 23-64		41-1487		75-1621	
d-spacing in nm	Intensity innm	d-spacing innm	Intensity	d-spacing	Intensity
0.336013	100	0.337675	100	0.339635	100
0.213001	10	0.213890	2	0.213890	3
0.202973	50	0.203883	6	0.203984	14
0.179965	5	0.180759	1	0.180999	3
0.167800	80	0.168145	4	0.169747	5
0.154391	10	0.154800	1	0.155446	4
0.123188	30	0.123411	3	0.123485	3
0.115804	50	0.116034	3	0.116067	5
0.113809	5			0.113180	1
0.112004	20	0.112096	1		
0.105404	5	0.105676	1		

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