# NANO PARTICLES IN GROUND GRAPHITE

*R.H. Bragg*, University of California*
*Berkeley, California 94720, USA*
*J.B. Aladekomo, Olafemi Awolowo University*
*Ile-Ife, Nigeria, West Africa*

## Introduction

In studies of the effect of grinding on the structure of graphite, it was found that the crystallite size decreased monotonically and metastable phases characterized by d(002) of 0.338nm, 0.340nm and 0.344nm are produced sequentially, but a limiting diffraction pattern, like Figure 1(a) is observed after 55 hours that is not changed up to 90 hours of grinding.[1] The breadth of the strong peak (d = 0.355nm) indicates a mean crystallite thickness of about 1.07nm. The weak subsidiary peak, calculated to have d = 0.72nm, has been attributed to a new metastable phase, [2] but we show here that it is part of the diffraction pattern of crystallites that are four graphite layers thick. We also show that the strong background is the small angle scattering from single layer "flakes" of graphite about 10 2D unit cells in diameter.

## Analysis

All materials of low atomic number have a surprisingly high Compton background. Removal of this from Figure 1,[3] leaves a pattern that consists of the Bragg diffraction pattern for crystals *N* unit cells in height given by

$$I(x) = C_1[(1+\cos^2 2\theta)/2]\, F^2 \sin^2\pi N x / \sin^2\pi x \quad (1)$$

where $x = 2C\sin\theta/\lambda$, $F = 4f$, f is the atomic scattering factor for Carbon, and N is an integer; and a small angle scattering (SAS) component, expressed as

$$I(SAS) = C_2F^2[[(1+\cos^2 2\theta)/2]/h^n \quad (2)$$

where $C_2$ is a constant, different from $C_1$ and $h = 4\pi \sin\theta/\lambda$.

## Results and Discussion

Figure 1(b) shows the background (Compton) component, Figure 2(a) shows the Bragg component, for N=3. The presence of the weak peak at $\sin\theta/\lambda = 0.07$, in addition to the line breadth of about 8.4 degrees at $\sin\theta/\lambda = 0.14$, are strong indicators that the Bragg component comes from crystallites that are four layers thick. When the intensity changes caused by the polarization factor $(1+\cos^2 2\theta)/2$ and $F^2$ are included, the agreement between observed and calculated is exact.

The component attributed to SAS is a feature universally seen in the diffraction patterns of all unorganized carbons but never explained. Yang and Frindt provided the key to an explanation in a paper.[4]They calculated the diffraction patterns expected for different 10x10 2D layers of graphite that are turbostratically stacked and randomly oriented. Their Figure 2(b) corroborates our explanation of the weak 0.72nm peak, but the implications of their Figure 2(a) for a single layer are far more important. The predicted angular variation of the intensity versus $\sin\theta/\lambda$ below 0.28 agrees with our results exactly. While their results were for flakes 10x10 2D unit cells in area, we have made calculations that indicate that a model of roughly circular flakes of diameter about 2.5nm would also be consistent with our observations.

## Conclusions

Extensive grinding of graphite produces nanocrystalline graphite particles about 1.0nm thick, as well as nano-scale single layer flakes about 2.5nm in diameter. It is suggested that the high background that is always observed in the low angle data of the diffraction patterns of unorganized carbons is due to nano-scale single layer flakes.

## References

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*Currently Distinguished Scientist at MHATT-CAT,
Howard University, Washington, D.C.
*Email: rbragg@mhattcat.howard.edu*

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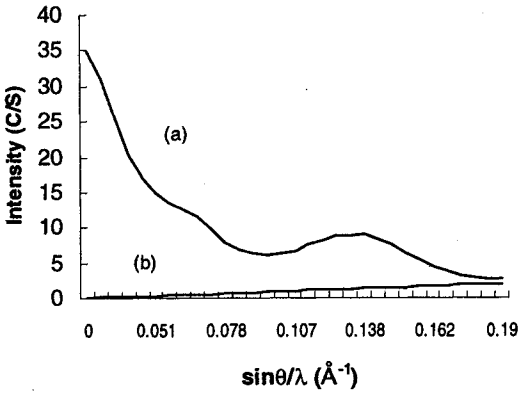


Figure 1. Diffraction pattern for graphite ground 74 hours. (a) Complete pattern. (b) Compton (incoherent) background

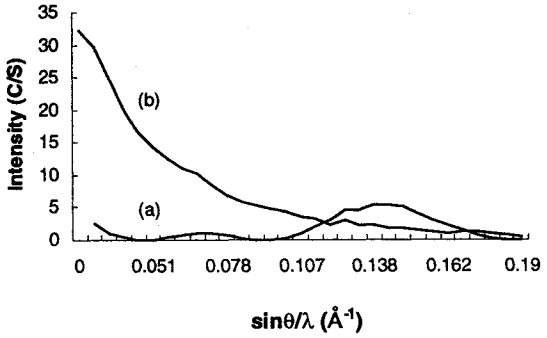


Figure 2. Components of the intensity data for C74 material. (a) Bragg diffraction. (b) Small angle scattering.