X-Ray Diffraction Properties of Highly Oriented Pyrolytic Graphite

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ABSTRACT

The x-ray diffraction properties of highly oriented pyrolytic graphite (HOPG) were studied for x-ray energies ranging from 4 to 60 keV. In particular, the secondary extinction thickness was determined by recording the peak and integrated reflectivity as a function of depth below the surface. The results showed that for the high quality material investigated a thickness of 200 to 300 μm was sufficient to get 80% of the maximum reflectivity that is obtained for a very thick plate. Primary extinction was important for low energy and still persisted at higher energies. Inhomogeneities of the mosaic structure were observed, too, that make this material not a truly ideal mosaic monochromator crystal. However, quite high peak reflectivities between 35% and 58% were measured at FWHM of 0.25 to 0.45 degrees. A 200 μm thick plate was then prepared and glued on a bending device to manufacture a monochromator or analyser with variable curvature that works from flat down to a minimum bending radius of 10 cm. The successful tests of this device confirmed that HOPG plates much thinner than those commonly used as x-ray monochromators and analysers still have high efficiency and can be curved to achieve dynamical focusing.

Keywords: x-ray diffraction, pyrolytic graphite, synchrotron radiation, x-ray optics.

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1. INTRODUCTION

Highly oriented pyrolytic graphite (HOPG) is a layered synthetic material that shows excellent performance both as a neutron monochromator, analyser and filter1-5 and as a high flux x-ray monochromator and analyser,6 in particular for synchrotron radiation.7-16 It was also proposed as a concentrating reflector for x-ray astronomy.17,18 It consists of crystallites that are relatively well aligned with respect to their c-axis whereas their a-axes are randomly oriented. The microstructure of this one dimensional single crystal is complex. Whereas early neutron diffraction studies reported that HOPG was an "ideal mosaic crystal"1-3, it was found later that both inhomogeneities of the mosaic structure and primary extinction were present that disagreed with the earlier results. More detailed gamma-ray and neutron diffractometry4,5 indicated that high quality HOPG was composed of domains up to 1 mm large that contained a number of smaller mosaic blocks with thicknesses exceeding several micrometers. Model calculations based on a disturbed surface layer and assuming primary extinction gave excellent agreement with the experimental neutron reflectivities.

More recently, x-ray topography19,20 confirmed the latter observations to a large extent. Direct imaging of the domains showed that their orientation distribution around the c-axis was not random, but that correlations extending over distances up to several millimeters were present whose origin, however, could not be explained. The large domains were aligned to no better than 0.5° and had dimensions of up to 0.5 mm perpendicular to the c-axis and between 10-80 μm parallel to the c-axis. Inside each domain, the mosaic blocks were aligned to better than 0.5° and, for the highest quality sample that had the largest slab size, their dimensions were too small to be resolved by topography, probably of the order of microns. Such a complex defect structure is far from the classical mosaic crystal model and makes a reliable prediction of the performance of HOPG as x-ray monochromator and analyser difficult. Although good agreement of experimental data10 with theory based on secondary extinction only9 was found for x-ray energies of 8 and 17 keV, more detailed experimental studies over a wider range of energies are needed. A particularly interesting parameter to be determined is the depth needed to reflect most, say, 80 or 90% of the incident photons of a given energy.

The aim of the present work was to investigate the x-ray diffraction properties of HOPG for energies ranging from a few keV to several tens of keV and to assess its performance for an optimized thickness. The results allowed us to manufacture a device with variable curvature that permits dynamical focusing of x-rays. Thus a full theoretical understanding and a detailed modeling of the diffraction phenomena in terms of more sophisticated extinction theories was not in the centre of interest here but will be the subject of a forthcoming publication. However, in the next section we give a simple theoretical background that is required for a qualitative understanding and a quantitative interpretation of the diffraction properties observed.