Experimental report

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Proposal: 1-04-122 **Council:** 4/2017

Title: New properties of carbon for innovative applications

Research area: Materials

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Samples: amorphous carbon powder

Instrument	Requested days	Allocated days	From	To
D7	11	9	16/04/2018	25/04/2018
IN11	10	0		

Abstract:

Magnetic properties of non-sp3 carbon-based systems have prompted intriguing discussions on the possibility of magnetism in carbon in the recent past and it opens plenty of possible applications from spintronic to catalysis or magnetic MEMS. There is an emerging consensus on the existence of magnetism in sp and sp2 carbon materials, which is supported largely by theoretical studies, and there are only a very few experimental observations. Magnetic measurements suggest the existence of magnetically correlated electron spins within nano-sized regions. We propose polarised neutron scattering to study to observe the diffuse magnetic signal coming from the magnetic short ranged correlations and the spin dynamic in the system.

Lithographic patterning of polymers followed by their carbonization via pyrolysis process is an emerging process for the fabrication of micro and nano carbon devices. Pyrolytic carbon can be obtained from a variety of polymers, and each polymer has a characteristic pyrolysis mechanism. Pyrolysis for miniaturized structures is typically performed at 900 ^{o}C, and in most cases, the decomposed polymeric material features the highest concentration of radicals around 600 ^{o}C. These radicals give rise to a paramagnetic behavior, are well-separated from each other, and are expected to be uniformaly distributed. In a previous study, we prepared this paramagnetic form of pyrolytic carbon and analyzed it using electron paramagnetic resonance (EPR), SQUID magnetometry, and various microstructural characterization techniques.

The aim of the experiment on D7 was to understand the nature of the magnetic correlations in the system. For the experiment we prepared a quite big quantity of the magnetic carbon sample by pyrolysis of polyimide at T = 600 ^{o}C under previously optimized conditions (14 g). The optimized conditions for obtaining the lowest possible H content and the highest magnetism were used for sample preparation. Commercially procured glassy carbon particles (size 10-20 μm) were used as the reference material. Commercial glassy carbons are also prepared using pyrolysis of polymers, but at much higher temperatures (typically >2000 ^{o}C), which ensures there are no dangling bonds. This material was used as a reference since it exhibits a very high purity (99.99 % according to the vendor's specifications), and is sp^2 hybridized, which is structurally closer to the test sample compared to other carbon forms such as diamond-like carbon. Various previous studies have shown that high-temperature glassy carbons do not show any magnetism. The reference and magnetic sample were measured on D7 at a wavelength of 4.84 Å. Calibrations were performed, including empty can, vanadium, quartz (rod) and Cadmium measurements. This allowed subtraction of background and for calibrations of the detectors and analyzers.

In our proposal we estimated to need 15 days on D7 to have a statistic good enough to distinguish the magnetic signal from the magnetic carbon respect to the signal from the reference (1 day for set up and calibration, 7 days x 2 for the reference and magnetic carbon sample). The days allocated were 9 and this number was still reduced to 7 because an unexpected shutdown of the reactor and a technical problem of a cold valve on the instrument.

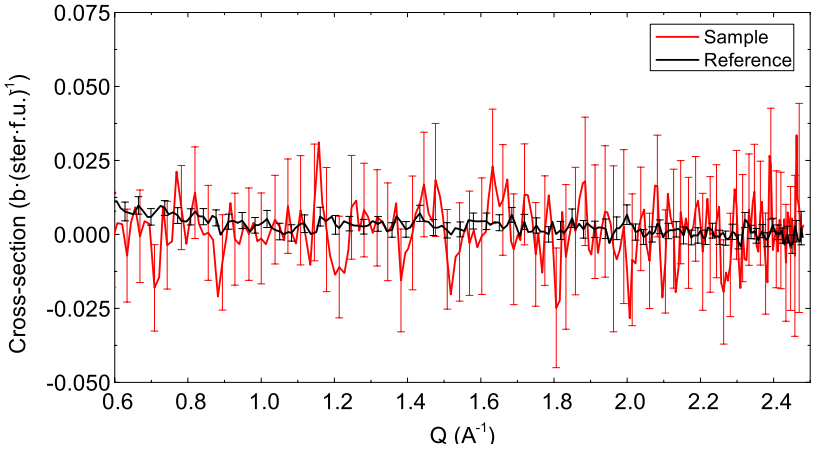

FIG.1 Magnetic signal from the magnetic carbon (in red) and the reference (in black)

It is evident from the results (fig.1) that the allocated beam time was not sufficient to obtain significant results.

Moreover, the reference showed some nuclear peaks due to the material structure (particles with isotropic distribution of graphene sheets) as we can see from Fig. 2. The presence of this strong peak prevents a good correction of the data in the separation of the different components to the signal (magnetic, nuclear+incoherent, spin incoherent) in the low Q region in Fig. 2.

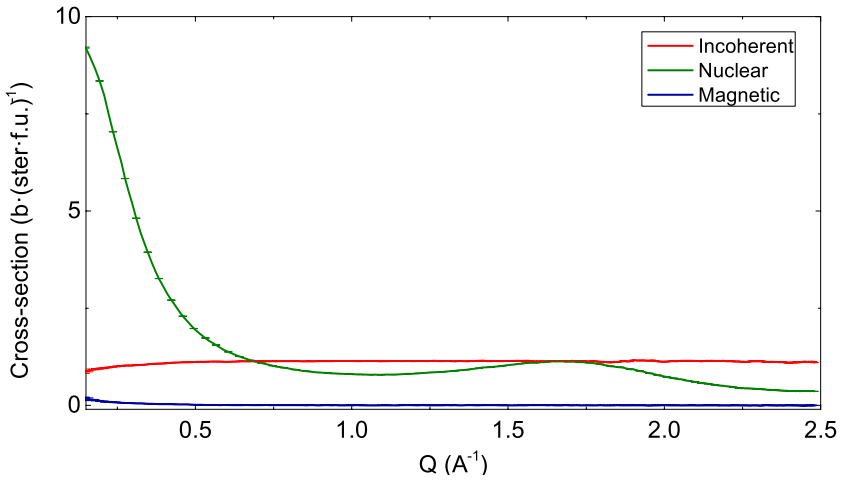

FIG.2 Incoherent, nuclear and magnetic signals from the reference sample.

No publishable conclusions could be drawn from the conducted experiments. D7 is the only instrument that can provide us useful and reliable data on this material and we believe that if we are given the time that is requested (15 days), a measurable signal can be obtained. In addition, in the next experiment (if time will be allocated), we will select and fully characterize a reference material also from structural point of view.