OPERATION WITH CARBON STRIPPING FOILS AT ISIS

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Abstract

The ISIS facility at the Rutherford Appleton Laboratory is a pulsed neutron and muon source for physical and life science research. Up to 3×10^{13} protons per pulse are accelerated to 800 MeV in the 50 Hz rapid cycling synchrotron that serves two spallation neutron targets.

Charge exchange injection of 70 MeV H^- ions into the synchrotron takes place over 130 turns. For over 30 years ISIS has used 40×120 mm aluminium oxide stripping foils, produced in-house [1]. Recently, foil preparation and installation processes have been simplified with the use of commercially available 40×60 mm carbon stripping foils.

This paper summarises operational experiences with diamond-like-carbon (DLC) and graphene foils. Radiological analysis, atomic force microscope (AFM) imaging of foils and off-line irradiation with a 1.5 keV electron gun are also discussed.

OPERATION WITH CARBON FOILS

Initial experimentation from July 2015 to May 2016 demonstrated the use of a 40×60 mm, 100 μg/cm^2 DLC foil, clamped at the top and side, and supported by four strands of carbon fibre to mitigate deformation [2].

Financial year 2016/17 was the first year of ISIS operation in which only carbon-based foils were used. Nine foils were used to inject 758 mAh of H^- beam during the 191 user days, split over five cycles (2016/01-05). Each cycle begins with one or two weeks of accelerator start-up and ends with three development days. Depending on the cycle length, one or two mid-cycle maintenance days are scheduled.

Cycle 2016/01

Cycle 2016/01 was the first ISIS cycle to use only DLC foils. The first foil was installed for the accelerator start-up period and inspected on the mid-cycle maintenance day. Despite appearing to be in good condition on the maintenance day, rising injection losses necessitated a foil change four days later after 140 mAh of beam, Fig. 1. The replacement DLC foil deformed quickly over the last ten days of the user cycle, during which it saw 46 mAh.

Cycle 2016/02

Cycle 2016/02 was scheduled to run only to the second target station, delivering 40 μA at 10 Hz. With the reduced beam intensity and repetition rate, a fresh foil survived the whole cycle. This foil saw 1 mAh at up to 50 Hz during cycle 2016/01 accelerator development time, 32 mAh at 10 Hz in cycle 2016/02 and 17 mAh at up to 50 Hz in the start-up period for cycle 2016/03, 50 mAh of beam in total, Fig. 2. As expected, less deformation was observed during 10 Hz operation than 50 Hz, comparing Figs. 1-2. The peak foil temperatures, calculated using an existing ANSYS model [3], are 383 K at 10 Hz and 492 K at 50 Hz.

Figure 1: DLC foils used in cycle 2016/01 after 140 mAh (left) and 46 mAh (right) of beam.

Figure 2: DLC foil used in cycle 2016/02 after (left to right) 1 mAh, 18 mAh and 32 mAh of beam.

Cycle 2016/03

Cycle 2016/03 was 45 days long, with two planned maintenance days. A new foil was installed for the cycle start and removed on the first maintenance day after 61 mAh of beam. This foil was stored for radiological analysis, which is discussed in the following section. The replacement foil was inspected on the second maintenance day after 46 mAh, Fig. 3. Deformation was clearly seen but the foil remained well constrained by the supporting carbon fibres and was left in for the remainder of the cycle.

At the end of the cycle, after 92 mAh, the foil was removed and replaced with a graphene foil for experimentation in the accelerator development period.

Graphene was chosen as a stripping foil material for its high thermal conductivity and high tensile strength, with the aim of avoiding the deformation seen with amorphous carbon foils. A 200 μg/cm^2 graphene foil, the minimum available thickness, was purchased from Applied Nanotech, Inc. [4] and was mounted without supporting fibres. The graphene foil survived 5 mAh of beam during the 50 Hz accelerator development time, Fig. 4. No injection tuning was required and performance was much more successful than with unconstrained DLC foils [2].

Cycle 2016/04

The graphene foil was left in place for use in the 2016/04 start-up and cycle. It was inspected again on the mid-cycle maintenance day having seen 84 mAh of beam and removed at the end of the cycle after 153 mAh, the maximum irradiation of a carbon-based foil on ISIS so far. The graphene foil deformed during irradiation, but more slowly than DLC foils, and the deformation appeared to stabilise, Fig. 4.

During accelerator development time at the end of the cycle, a 'full height', 40×120 mm, 100 μg/cm^2 DLC foil was installed. This was clamped on three edges, similar to the previously used aluminium oxide foils [1], and had no supporting fibres.

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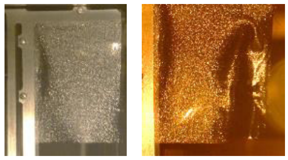

Figure 3: DLC foil used in cycle 2016/03, before irradiation (left) and after 46 mAh of beam (right).

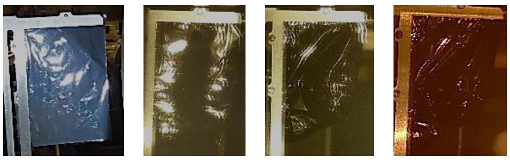

Figure 4: Graphene foil (left to right): before installation, after 5 mAh, 84 mAh, and 153 mAh of beam.

Cycle 2016/05

The full height foil was inspected before and during the start-up for cycle 2016/05 where it saw 13 mAh of beam. This foil failed suddenly five days into the 52 day cycle, after 42 mAh, with a tear along the top edge, Fig. 5.

It was replaced with a 40×60 mm, 100 μg/cm^2 DLC foil with fibres and was inspected on the first maintenance day, after 27 mAh. It was replaced on the second maintenance day after 93 mAh to ensure availability until the end of the cycle. This foil saw 83 mAh until the cycle end, which concluded with a planned week of 10 Hz operation, Fig. 6.

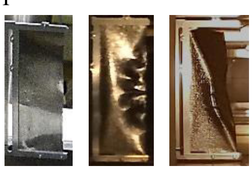

Figure 5: 40×120 mm DLC foil (left to right): before irradiation, after 13 mAh of beam and failure at 42 mAh.

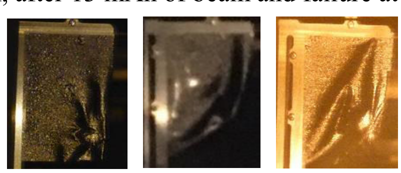

Figure 6: 40×60 mm DLC foils used in cycle 2016/05 after (left to right) 27 mAh, 93 mAh and 83 mAh of beam.

Summary of 2016-17 Operations

Operational experience with carbon-based foils has been very similar to that with aluminium oxide foils, achieving typical injection efficiencies of 98-99% and no detectable change in beam losses. ORBIT [5] models of ISIS injection were used to estimate the emittance blow-up due to beam scattering and foil traversals, with varying foil thicknesses and dimensions, Fig. 7. However, ISIS beam dynamics are dominated by space-charge effects, and the ORBIT models also show that the foil scattering can be compensated by small adjustments of injection painting, in agreement with empirical experience.

Mounted foils are stored in cool and dry conditions, in boxes containing silica beads to control humidity, as exposure to moisture can lead to degradation. However, foils prepared several months in advance have been observed to curl in storage, Fig. 8. Supporting fibres have been added to constrain the curled foils for future use.

The manual foil change procedure is simpler and quicker with carbon foils, potentially reducing staff doses. However, as the foil degrades, injection losses and activation of the foil area increase. Depending on loss levels and cool-down time (typically one hour) the working area dose rates are 250-1000 μSv/hr, with a typical staff dose of 30-40 μSv per foil change.

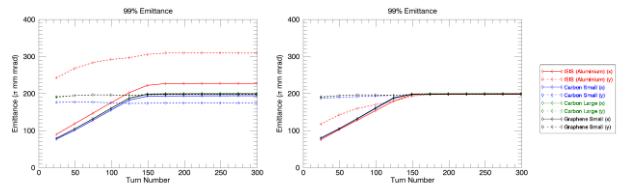

Figure 7: 99% emittance with no space charge or apertures for the ISIS painting scheme (left) and small painting adjustments to achieve the same final emittance (right).

Figure 8: Graphene foils observed to curl in storage.

OFF-LINE ANALYSIS OF CARBON FOILS

Radiological Analysis of Irradiated Foil

The foil used for the first 16 days of cycle 2016/03 was retrieved and stored in the synchrotron hall for 100 days until removal for gamma-ray spectroscopy to identify the residual nuclei. A Canberra BE3825 detector was used at distance of 65 cm from the foil for 15 minutes. The strongest peak, at 477 keV, corresponds to Be-7, Fig. 9.

Combining the irradiation history, including eight foil traversals predicted from ORBIT modelling, and physical detector details the C-12 to Be-7 cross-section was calculated as 10±2 mb. Comparing this to published values of 22-50 mb [6] suggests that the number of foil traversals is overestimated by a factor of at least two.

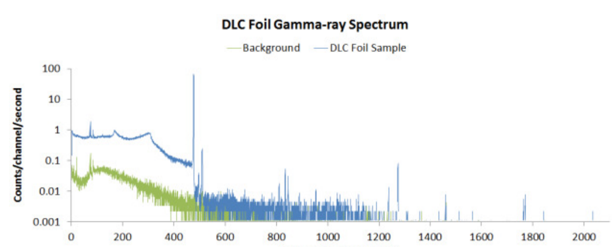

Figure 9: Gamma-ray spectrum, green shows the background and blue the foil sample.

A FLUKA [7] model of the foil, bracket, and fibres for the cycle irradiation and decay time was compared to the measured gamma-ray spectroscopy results, Table 1. The activation due to Be-7 and residual nuclei inventory agree well with measurements. The largest source of uncertainty in the model is the contribution of foil traversals.

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Table 1: Measurement and FLUKA Comparison

	Measurement	FLUKA
Be-7 Components	96.3% peak area	>99% residuals
Be-7 Activity (Bq)	2.14×10^{7}	7.94×10^{7}
Dose Rate (μSv/hr)	250	310

Measurements of DLC and Graphene Foils

Visually the graphene foils appear smooth whereas the DLC foil structure is rough and can include large grains. Measurements of DLC and graphene foil thickness and surface roughness were made using a touch probe, surface profiler and AFM, similar to previous characterisation of the ISIS aluminium oxide foils [1].

A 100 μm^2 area from each foil was imaged with the AFM and the RMS roughness for DLC was double that of graphene, results are shown in Table 2.

An areal density estimate for the graphene foil of 240±36 μg/cm^2 was calculated using the average measured thicknesses and quoted bulk density 1.8±0.2 g/cm^3 [4].

A 1463±48 mm^2 sample of DLC foil weighed 1.880±0.005 mg, giving an areal density of 129±5 μg/cm^2. Combining the average measured thickness with the accepted DLC density (2 g/cm^3) gives an areal density estimate of 115±10 μg/cm^2. The quoted tolerance of DLC foil thickness was ~10% [8].

Table 2: Thickness and Roughness Comparison

	Thickness (μm)		Roughness (nm)
	Touch Probe	Profiler	AFM
	(±0.05 μm)	(±0.0005 μm)	(±0.15 nm)
DLC	0.60	0.5470	29.50
Graphene	1.30	1.3740	16.20

Irradiation with 1.5 keV Electron Gun

A DLC foil was irradiated with a 1.5 keV Kimball Physics (EFG-7) electron gun and deformation recorded through time-lapse photography.

The stopping power of 1.5 keV electrons in carbon is ten times that of 70 MeV protons and the 1.5 keV electrons are fully stopped within 50 nm of foil material. With the maximum achievable 100 μA electron beam current, a 3.6 mm diameter beam spot was used to best replicate the power density of the 15 mm ISIS injection beam spot. Although this experimental setup does not reproduce the full power of ISIS operations, it provides an environment for tests without risk to beam time.

The beam spot size was focussed on a phosphor screen prior to foil irradiation. Deformation of the foil was seen within 0.1 s of irradiation, but over a further 17 hours no significant deterioration was observed, Fig. 10. Time-lapse photography at 0.1 s intervals showed the foil fluttering, most notably when beam was pulsed at 50 Hz.

A second mounted DLC foil was annealed at 400°C for two hours (between a two hour ramp-up and two hour cool-down) after which the grained DLC structure appeared smoother and tension in the foil changed. This annealed foil was irradiated, as described above, for 11 hours and no visible deformation was seen, Fig. 11.

ANSYS simulation of the electron beam test predicts a peak foil temperature of 200°C. The Micromatter DLC production process involves 2-4 hours of annealing at 160-240°C, depending on the initial foil condition [8]. A significant improvement in foil reaction to beam after the higher annealing temperature was observed in this study.

Figure 10: (Left to right) DLC foil mounted in vacuum chamber, 3.6 mm 100 μA electron beam spot focussed on a phosphor screen beneath the foil, observed deformation.

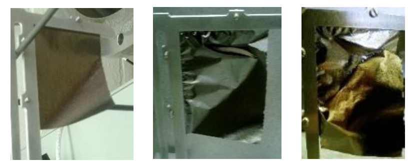

Figure 11: (Left to right) DLC foil prior to annealing, after annealing at 400°C, after 11 hours DC irradiation.

SUMMARY AND FUTURE PLANS

Commercially available carbon-based foils are easier to handle and install than the previously used fragile aluminium oxide foils. Under beam they perform well, with no immediate measurable change in beam losses. However, the foils have been observed to degrade, reducing the usable lifetime.

The only carbon-based foil to withstand an entire user cycle was the graphene foil from cycle 2016/04, seeing 153 mAh of beam. To maximise availability and minimise staff dose the foil should survive a full user cycle. Assuming that a cycle of 45 days runs with 90% availability at 220 μA the foil would see 214 mAh of beam, so further work on extending foil lifetimes is required.

There are plans to continue operational tests with graphene foils, compare 100 μg/cm^2 hybrid-boron-carbon (HBC) and 200 μg/cm^2 DLC foil performance, and further optimise the foil dimensions and thickness. Initial results from high temperature annealing tests are promising and, along with strategies for improving handling and storage, may offer increases in foil lifetime.

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