

## SPALLATION NEUTRON SOURCE (SNS) DIAMOND STRIPPER FOIL DEVELOPMENT

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### Abstract

Diamond stripping foils are under development for the SNS. Freestanding, flat 300 to 500  $\mu\text{g}/\text{cm}^2$  foils as large as 17 x 25 mm<sup>2</sup> have been prepared. These nano-textured polycrystalline foils are grown by microwave plasma-assisted chemical vapor deposition in a corrugated format to maintain their flatness. They are mechanically supported on a single edge by a residual portion of their silicon growth substrate; fine foil supporting wires are not required for diamond foils. Six foils were mounted on the SNS foil changer in early 2006 and have performed well in commissioning experiments at reduced operating power. A diamond foil was used during a recent experiment where 15  $\mu\text{C}$  of protons, approximately 64% of the design value, were stored in the ring. A few diamond foils have been tested at LANSCE/PSR, where one foil was in service for a period of five months (820 C of integrated injected charge) before it was replaced. Diamond foils have also been tested in Japan at KEK (640 keV H<sup>-</sup>) where their lifetimes slightly surpassed those of evaporated carbon foils, but fell short of those for Sugai's new hybrid boron carbon (HBC) foils.[1]

### BACKGROUND

Diamond foils are under development for the Spallation Neutron Source (SNS). The use of diamond as a high beam current replacement for evaporated carbon foils was initially studied by workers at Brookhaven National Laboratory (BNL) [2] who irradiated a fully-supported chemical vapor deposited (CVD) foil with a 750 keV pulsed H<sup>-</sup> beam. A useful lifetime of 400 hours was observed for a beam current designed to mimic the SNS foil thermal load. However, CVD diamond films can contain substantial stress, and freestanding foils released from their growth substrates curl badly. We have developed corrugated foil techniques to maintain flatness and have grown and tested diamond foils as large as 17 x 25 mm<sup>2</sup> and supported on only one edge.[3,4] Both micro- and nano-textured polycrystalline foils have been prepared. Generally the latter are flatter. Crystallite particles down to about 50 nm are present in the nano-textured foils and may serve to spread foil stress. These foils were grown in a microwave-powered growth chamber at 2.45 GHz using a starting material gas mixture of 1-2% methane in hydrogen (or the same mixture, diluted in 90% argon for nano-textured material).[5] In the approximately 2 mA BNL beam, reproducible foil lifetimes exceeding about 130 hours

were observed. Our earlier results, including preparation methods and tests at BNL have been described.[3,4]

We have tested diamond foils at the Proton Storage Ring (PSR) at Los Alamos National Laboratory, as well as KEK in Japan. Foils have been loaded at the SNS and used for low power experiments. These test results are described here.

### FOIL FABRICATION NOTES

A few minor changes in the foil preparation procedure have been implemented since our PAC2005 report.[4] Following Akhvediani[6], a *mixed slurry* of diamond particles in an ultrasonic bath was used for the pre-growth substrate surface roughening to create diamond nucleation sites. A 1:1 mixture by weight of diamond 600 grit abrasive particles (30-35  $\mu\text{m}$  diameter) along with particles < 0.25  $\mu\text{m}$  diameter in methanol suspension was used. Stirring was found to improve foil particle size uniformity. This recipe produces high nucleation density; however, the limited size of the microwave plasma ball leads to a film thickness that decreases radially from the reactor centerline. The film thickness deviation is about 10-20% over the full substrate length. The foil thicknesses cited here are averaged values for the entire substrate. In addition, substrate mounting hole milling is now performed *after* silicon patterning to minimize wafer etch pits that can often lead to foil pinholes.

### FOIL LIFETIME TESTS AT KEK

In anticipation of the J-PARC neutron source in Japan, foil experiments have been conducted at KEK. Thin (ranging from 330 to 380  $\mu\text{g}/\text{cm}^2$ ) and thick (430 to 500  $\mu\text{g}/\text{cm}^2$ ) foils were investigated. The KEK Cockcroft-Walton accelerator was used for H<sup>-</sup> (640 keV, 100 and 180  $\mu\text{A}$  dc) irradiations. Some of the foils were supported using SiC fibers. The 3x5 mm<sup>2</sup> beam spot size was smaller than for foil tests elsewhere. Foil pyrometry and *in operando* imaging were available for these experiments. The foils were irradiated until large deformations or cracks were evident. Foil photos for a 500  $\mu\text{g}/\text{cm}^2$  nanocrystalline diamond sample irradiated at two locations (upper and lower) at 100  $\mu\text{A}$  and 180  $\mu\text{A}$  for 29 and 4 hours, respectively, are shown in the left and right panels of Figure 1.

For H<sup>-</sup> irradiations at about 110  $\mu\text{A}$  dc, thin microcrystalline foils (2) survived 11 and 14 hr, thin nanocrystalline foils (2) lasted 13 hours each, and thick nanocrystalline foils (2) tolerated 23 and 29 hours. The

foil lifetime increased slightly with foil mass. Irradiations at 180  $\mu\text{A}$  dc, generally resulted in lifetimes reduced by at least fivefold. The lower current resulted in foil temperatures around 1950 K, while the higher current yielded about 2100-2300 K. Measured temperatures are believed to be accurate to about 160 K, mostly limited by the uncharacterized sample emissivity. The KEK apparatus will be modified soon for pulsed beam experiments.

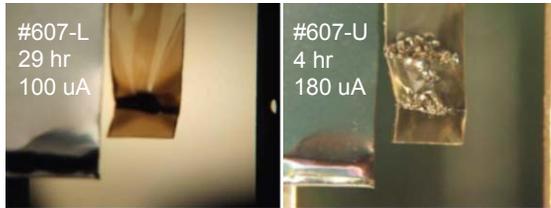


Figure 1: Nanocrystalline diamond foils irradiated at 100  $\mu\text{A}$  (left) and 180  $\mu\text{A}$  dc  $\text{H}^+$  at KEK. The foil is positioned to the right of a metal shield.

### DIAMOND FOIL EXPERIENCE AT PSR

In neutron production mode, the PSR characteristics are 20 Hz repetition rate and  $3 \times 10^{13}$  protons per pulse at 800 MeV for up to 100 kW power. Although the average power is low, the 50 to 70 foil hits per circulating proton are roughly tenfold higher than expected at SNS and

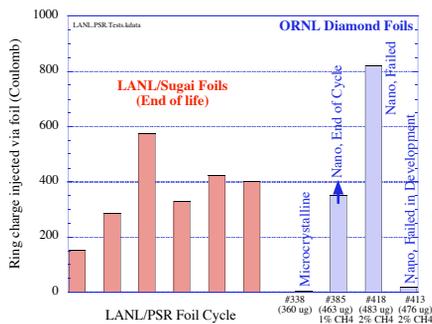


Figure 2: PSR foil longevity experience for LANL/Sugai (red) and SNS diamond (blue) foils.

dominate the foil thermal load. The foils used currently have a nominal  $450 \mu\text{g}/\text{cm}^2$  thickness and are actually stacked individual foils (up to 4) prepared using the modified controlled AC/DC arc discharge method originally described by Sugai.[7] The foil stack is supported by an array of stretched  $4 \mu\text{m}$  carbon fibers. For their first use, foils are annealed by careful ramp-up of the beam current while changing the foil location in a raster pattern to slowly heat the film. The foil results for recent LANL/Sugai foils as well as for diamond foils are summarized in the plot of Figure 2. The six bars shown for LANL/Sugai foils correspond to end-of-life, accumulated injected charge values; the decision to replace a foil was based upon unacceptable losses due to

foil scattering. The average injected charge value for the six foils is 362 C.

Diamond foils were mounted for both of the last two PSR cycles. The initial diamond foil examined was microcrystalline-textured with a  $360 \mu\text{g}/\text{cm}^2$  thickness. It did not survive more than 3 to 4 C of injected charge in a Development Time portion of the PSR cycle. Because of the limited number of foil positions and the demanding production schedule, no additional microcrystalline foils have been loaded at PSR. A  $463 \mu\text{g}/\text{cm}^2$  nanocrystalline foil was tested until the end of a run cycle (352 C of injected charge) and then removed for examination; unfortunately it was broken in handling and could not be returned to the PSR for further use. A nanocrystalline foil grown using 2%  $\text{CH}_4$  in the reactant gases was irradiated until it had seriously deteriorated (820 C of injected charge). Finally, an additional nanocrystalline foil was positioned in the beam, but was damaged quickly during development time, presumably at high beam current. Two thicker ( $500 \mu\text{g}/\text{cm}^2$ ), nanocrystalline foils were loaded in April 2007 and further PSR results should be forthcoming.

In general, two operational differences have been noted for diamond foils relative to the LANL/Sugai foils. First, the first turn losses are higher for diamond foils of comparable thickness. First turn losses are due to the production of neutral hydrogen atoms that then field-strip down stream. Diamond foils must be approximately 10 to 20% thicker to decrease the first turn loss rate to that of LANL/Sugai foils. In both cases, the loss rate decreases with irradiation, possibly due to foil wrinkling, but more slowly for the diamond foils. Second, the foil current for the diamond foils is lower than that for the LANL/Sugai foils by about 2- to 5-fold. The foil current is due to the production of secondary electrons at the foil, and it is not understood why the secondary electron yield is smaller for the diamond foils.

### DIAMOND FOIL EXPERIENCE AT SNS

The SNS is configured with a twelve-station foil changer, with ten positions for foils. The design foil size is  $12 \times 20 \text{ mm}^2 \times 300 \mu\text{g}/\text{cm}^2$ . The foils are mounted at  $30^\circ$  to the beam, and thus the foil physical thickness is  $270 \mu\text{g}/\text{cm}^2$ . At full design power, the SNS is expected to produce a time-averaged 1.4 mA  $\text{H}^+$  beam (60 Hz) at 1.0 GeV, corresponding to 1.4 MW and  $1.5 \times 10^{14}$  protons per pulse. Seven to ten foil hits per proton are expected, and the foil thermal load will be dominated by the circulating beam. On a hits/second basis, the SNS foil load is about three-fold higher than that for the PSR.

Diamond foils were first loaded into the SNS in January 2006. A diamond foil was in use when the first neutrons were created at SNS on April 28, 2006. Only one diamond foil has been damaged beyond usefulness, during a high intensity experiment. During a subsequent foil loading in November 2006, a used but intact foil was removed for examination. It had experienced about 7.8 C of injected charge. A photograph of this foil is shown in

Figure 3. The darkened rectangular area in the upper corner corresponds to the footprint of the circulating beam. Also, a slight curl at the foil corner is evident.

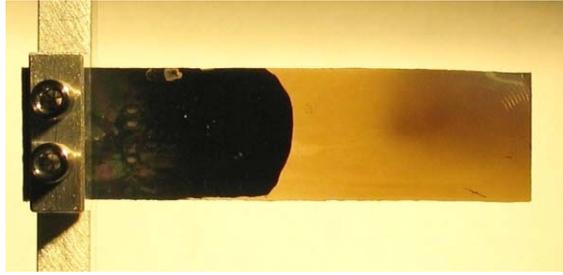


Figure 3: SNS 12x20 mm<sup>2</sup> foil after use. The image has been rotated 90° CCW from the true orientation. Damage is apparent in the figure upper right corner.

Early in the ring commissioning period, it was discovered that a design change to the ring injection chicane magnets had the unintended consequence of high beam loss in the injection dump beam line. Efficient simultaneous transmission of the H<sup>0</sup> and H<sup>-</sup> waste beams was not possible. A short-term solution, put into place in November 2006, was to use a thicker and larger 17 x 25 mm<sup>2</sup> x 425 µg/cm<sup>2</sup> primary stripper foil. This foil can fully intercept the incoming H<sup>-</sup> beam so there is little to no H<sup>-</sup> beam entering the injection dump beam line. This allows the beam line to be optimized for just a single beam, which reduces the beam loss in the injection dump beam line. Modifications to the injection dump beam line are now in progress. The design foil size and thickness has not changed, and return to the nominal foil parameters within the next year is intended.

Currently the SNS operating power is about 60 kW for low current machine tests and neutron production; a 90 kW maximum power has been achieved. The design operating power is expected to be reached in the Summer of 2009. A 15 µC (9.6x10<sup>13</sup> ppp) SNS stored proton record was attained using a nanocrystalline diamond foil. A single diamond foil (nanocrystalline, 17 x 25 mm<sup>2</sup> x 463 µg/cm<sup>2</sup>) was used for almost all of the production run that ended in April 2007; that foil was used to deliver 92 C of charge to the spallation target between January and April 2007.

### SUMMARY

The current generation of accelerators that are under construction or commissioning and that require stripping foils are pushing the limits of traditional carbon foil technology. Diamond foils have been prepared and irradiated at several accelerators around the world to gauge their utility for high power operation. Nanocrystalline foils seem superior. Evaluation experiments using H<sup>-</sup> have been conducted at KEK in Japan, the Proton Storage Ring at LANL, and the SNS in Oak Ridge. Large-area, freestanding polycrystalline diamond foils – up to at least 17 by 25 mm<sup>2</sup> at 300 µg/cm<sup>2</sup> thickness can be fabricated at a few foils per week rate.

These foils do not require supporting fibers and are conveniently mounted using a portion of their silicon growth substrate. Corrugation of the film is the key to maintaining foil flatness.

Experiments using a 640 keV, 180 µA dc H<sup>-</sup> beam at KEK produced foil operating temperatures up to 2200 K, limiting the foil lifetime. At 110 µA, 10 to 30 hour lifetimes were observed. The lifetime results at PSR, a machine with about the same peak but about one-third the average foil thermal load of the SNS, are promising, with neutron production operating mode lifetimes of several months duration. Low-power experiments at the SNS confirm the positive PSR results. Further foil developments will be required for the power upgrade planned for the SNS.

### ACKNOWLEDGEMENTS

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