

CORRUGATED THIN DIAMOND FOILS FOR SNS H⁻ INJECTION STRIPPING

R. W. Shaw, V. A. Davis, R. N. Potter, L. L. Wilson, ORNL, Oak Ridge, TN 37831, U.S.A.
C. S. Feigerle, M. E. Peretich, Chemistry, Univ. of Tennessee, Knoxville, TN 37996, U.S.A.
C. J. Liaw, Collider/Accelerator Dept., BNL, Upton, NY 11973, U.S.A.

Abstract

We have prepared and tested corrugated, thin diamond foils for use in stripping the SNS H⁻ Linac beam. Diamond has shown promise for providing ca. 10X increased lifetime over traditional carbon foils. The preferred foil geometry is 10 to 12 mm by 20 mm at 350 microgram/cm², mechanically supported on preferably one, but no more than two, edges. The foils are prepared by chemical vapor deposition (CVD) on a patterned silicon substrate, followed by chemical removal of the silicon. This yields a foil with trapezoidal corrugations to enhance mechanical strength and foil flatness. Both micro- and nano-crystalline diamond foils have been grown. Microwave plasma CVD methods that incorporate high argon gas content were used to produce the latter. Sixteen foils of a variety of characteristics have been tested using the BNL 750 keV RFQ H⁻ beam at a current scaled to simulate the energy deposition in the SNS foil. Long foil lifetimes, up to more than 130 hours, have been demonstrated. Characterization of the foils after beam testing indicates creation of sp² defects within the ion beam spot. Current efforts are centered on development of corrugation patterns that will enhance flatness of single-edge supported foils.

INTRODUCTION

A thin carbon stripping foil is an integral component of the Spallation Neutron Source (SNS) at the injection point to the accumulator ring. The operational lifetime of these foils is a concern, especially considering the design beam current (26 mA) and energy (1.0 GeV) for this facility. A foil changer with ten stations has been fabricated to permit foil replacement without facility shutdown, but foil lifetimes over 200 hours will still be required to maintain operation between regularly scheduled service interruptions. Liaw and coworkers reported extended lifetimes relative to traditional carbon foils in tests of picture frame supported diamond foils at PAC2001 [1]. However, severe curling of these films occurred when the support was reduced to one or two edges. At PAC2003, we reported the use of foil corrugations for strengthening diamond foils and for retaining flatness of foils supported on only two edges [2]. Results of simulated beam lifetime tests were also noted. Since then, a more extensive testing of the lifetimes of these corrugated films has been undertaken and is reported in this paper. Progress on fabricating single-edge supported films is also presented.

FOIL DESIGN AND PREPARATION

The SNS design calls for a foil that is 11 to 20 mm (preferred) high by 10 to 12 mm wide. At most, two edges of the foil can be supported mechanically; however, the preferred geometry is single-edge support, so that the painted ion beam can be rastered on and off the foil without striking the support structure. We now routinely prepare corrugated, polycrystalline diamond foils at 350 microgram/cm² (1 micrometer thickness) that are flat in the two-edge support format. Diamond films were grown on <100> silicon substrates that were first patterned using photolithography to produce a corrugated growth surface. The corrugations are trapezoidal with a parallel line density of 25, 50, 100, or 200 lines per inch. The trench depth was varied between about 5 and 20 micrometers. The trench walls are not vertical, but are at a 55° inclination, due to the anisotropic chemical etch rate of the silicon lattice. After patterning, the substrate was scratched ultrasonically using a diamond abrasive slurry to produce diamond nucleation sites at a density sufficiently high that the nuclei grew together before the desired one micrometer film thickness was reached.

Diamond films were grown on the patterned substrates in a 2.45 GHz microwave plasma-enhanced chemical vapor deposition (CVD) growth chamber, typically using a 2% methane in hydrogen gas mixture. At a 1300 W reactor power and a 50 Torr pressure, a one micrometer diamond film was formed within about 110 minutes. The films were characterized using Raman spectroscopy and Scanning Electron Microscopy (SEM), typically indicating the films as microcrystalline diamond, containing crystallites of a few tenths of one micrometer diameter, and conforming to the pattern of the silicon wafer face. We have also grown nanocrystalline films by adding argon gas to the inlet mixture in concentrations as high as 95%. Limited beam test data on nanocrystalline diamond samples indicated that these may be more robust stripping foils [2]. Modeling of nanocrystalline diamond suggests that strong adhesion at high energy grain boundaries may make this material even stronger than single crystal diamond [3].

Foils were freed from their silicon substrates by chemical etching in a mixture of concentrated hydrofluoric, nitric, and acetic acids (1:1:1). Before inserting the samples in the etchant, the substrate region which was to be retained as support was protected using Teflon™ tape on one face (the diamond film on the reverse face protected that side). The unetched portion of

the silicon wafer served as a mounting bracket; for two-edge supported foils, the silicon mount is in the form of an L-shaped bracket. Here, the corrugations were arranged diagonally on the foil to pin the free corner. A photograph of this foil format was shown in [2].

A reasonable concern about the use of corrugated foils is that the effective foil thickness at the wall positions may significantly exceed the design value when the projection relative to the ion beam is considered. Figure 1 shows a sketch of a small portion of one of our foils, for the particular case of 50 lines/inch pitch.

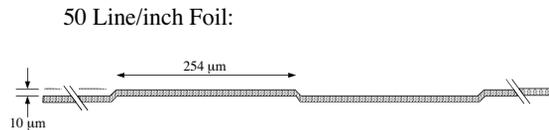


Figure 1. Sketch depicting the corrugation pattern for a foil at 50 lines/inch pitch. The vertical dimension is not to scale.

Note that the wall area is small relative to the total foil area. At 50 lines/inch pitch, the troughs and mesas are approximately 250 micrometers wide, while the walls are approximately 7 micrometers wide for a 10 micrometer trench depth. Thus, the walls represent only $\sim 3\%$ of the full area of the foil. Further, the effective thickness at the wall is only about $2^{1/2}$ times the nominal foil thickness. Thus, the corrugations introduce a limited departure from ideal foil thickness.

FOIL LIFETIME TESTS

The foils were tested in a beam at Brookhaven National Laboratory, using the 750 keV H⁻ Linac (6.7 Hz, 0.5 ms pulse). The beam was approximately circular, with a 3 mm diameter. A 2 mA peak beam current was used for these tests so as to achieve the same energy deposition rate in the foil as for a 32 mA, 1 GeV, 60 Hz SNS beam. The total current was monitored behind the foil, and the lifetime was gauged by the time required for the current to drop to 90% of its initial value. However, tests were halted at about 130 hours, if the 90% current criterion had not been reached.

The test results are summarized in Figure 2. Two foil formats are presented: 12x12 mm window frame foils (supported on all four edges) and the L-bracket (two-edge) format described above. All of these foils survived a shipment to BNL attesting to their robust character when careful handling and packaging were employed. However, many foils arrived broken at Long Island. The nominal foil thickness was 350 micrograms/cm², except for foil #s 3 and 4 that were 650 microgram/cm². Data from a few suspect samples are not shown. The three bars shown at the top of the plot represent foils that were grown in a hot-filament powered reactor (HF) with added argon reactant gas to yield nano-textured foils. The remaining bars all correspond to microwave reactor (MW) grown foils. The data bars are noted as "12x12" for the window frame samples and "L" for the two-edge

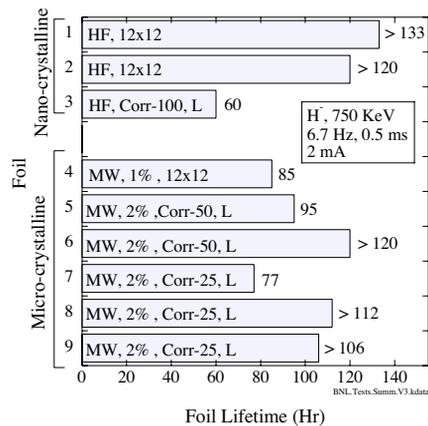


Figure 2. Beam lifetime test results for nano- and micro-textured diamond foils. The lifetime is defined as the time when the downstream current decreased to 90% of its initial value, but all tests were terminated at no longer than about 130 hours.

supported foils. The methane content of the growth mixture for the micro-crystalline foils was 2%, unless otherwise noted. The bottom five bars of the figure correspond to foils that were prepared under essentially identical conditions, except that two were 50 lines/inch and three were 25 lines/inch. For that set of foils a reproducible lifetime of about 100 hours or more was observed.

Some of the tested foils (e.g., an 85-hour tested foil, #4 of Figure 2) were returned to Oak Ridge for post mortem characterization. The beam spot area was wrinkled, torn, and darkened. The samples were characterized using Raman spectroscopy and TEM. The analysis showed that the particle-induced damage only occurred within the beam spot and corresponds to substantial conversion of the lattice to sp² centers. The Raman spectrum of a beam-damaged nano-crystalline foil is similar, except that the lower frequency, disordered Raman band is somewhat more intense.

SINGLE-EDGE SUPPORTED FOILS

We have prepared foils in the single-edge supported format that will be required at the SNS. A photograph of such a foil is shown in Figure 3. The free standing foil (left hand end) is 10 x 16 mm. Two mounting holes are also shown in the silicon frame; these were formed before film growth using an ultrasonic mill. This foil is corrugated (25 lines/inch) with the corrugations running parallel to the long axis of the foil; its nominal thickness is 560 microgram/cm². Even though an end-on view of this foil would show a slight curvature across the width of the foil (about a 1-mm deviation from flat), it appears that preparation of single-edge supported foils is feasible.

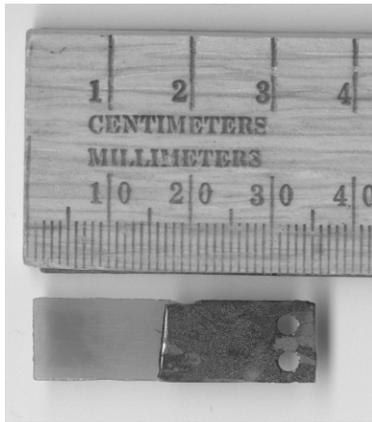


Figure 3. Photograph of a corrugated foil ($560 \mu\text{g}/\text{cm}^2$) that is supported on only one edge. A mm/cm scale is shown for reference.

Most recently we have begun to experiment with more advanced corrugation patterns. These entail more detail than simply a pattern of parallel lines. Several designs were considered, but a nested, U-shaped pattern was accepted. A test lithography mask was fabricated that contains several variations on the nominal design. The varied parameters were the line/inch pitch (25, 50, and 100 line/inch), the curvature at the base of the U (1, 2, and 3 mm radius), and the fraction of the foil surface area that is corrugated (40 and 60%). A total of 15 patterns was included. Five of the patterns are shown in Figure 4 along with a sketch of how the pattern is situated on the SNS substrate format. Note that the termini of the vertical lines were allowed to continue onto the portion of the sample supported by the silicon substrate to minimize fracture at the silicon/foil boundary.

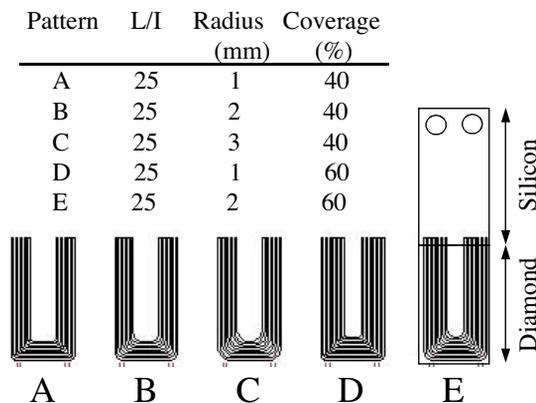


Figure 4. Five corrugation patterns for 2D support. The pattern noted as E is shown with a sketch of its location on an SNS format wafer. The pattern details are collected in the inset table. The figure is not to life scale.

Several foils have been grown and etched using the 2D patterns of Figure 4. As yet, none of the single-edge supported foils has been tested at an ion beam. They are, not unexpectedly, more fragile than L-bracket supported foils. Generally, unsuccessful foils failed in handling after chemical etching, i.e., drying or mounting. Interestingly, our success rate for processing grown films into a final product has been greatest for foils with 100 line/inch pitch and 1 mm radius of curvature, a somewhat counter intuitive result. Further preparation and testing of single-edge supported foils is underway.

SUMMARY

Double-edge and single-edge supported, corrugated diamond foils have been prepared and tested for use in H stripping for the SNS. Corrugation of the stressed, thin foil maintains the flat character necessary for use in beam stripping. Consistent lifetimes at or beyond 100 hours have been demonstrated in a lower energy ion beam arranged to simulate the foil energy deposition rate at the SNS. Further studies are underway to explore additional 2D corrugation patterns, compare nano- and micro-textured foils, test foils in higher energy beams, and to identify means to extend foil lifetimes

ACKNOWLEDGEMENTS

SNS is managed by UT-Battelle, LLC, under contract DE-AC05-00OR22725 for the U.S. Department of Energy. SNS is a collaboration of six US National Laboratories: Argonne National Laboratory (ANL), Brookhaven National Laboratory (BNL), Thomas Jefferson National Accelerator Facility (TJNAF), Los Alamos National Laboratory (LANL), Lawrence Berkeley National Laboratory (LBNL), and Oak Ridge National Laboratory (ORNL). MEP acknowledges a Summer Undergraduate Research Experience (SURE) fellowship, supported by Science Alliance, a UT Center of Excellence. RNP and VAD acknowledge appointments to the U.S. DOE Energy Science Undergraduate Laboratory Internship (SULI) Program at the Oak Ridge National Laboratory.

REFERENCES

- [1] C.J. Liaw, Y.Y. Lee, and J. Tuozzolo, "Lifetime of carbon stripping foils for the spallation neutron source", PAC2001, Chicago, IL, p. 1538.
- [2] R.W. Shaw, A.D. Herr, C.S. Feigerle, R.I. Cutler, C.J. Liaw, and Y.Y. Lee, "Thin diamond films for SNS H-injection stripping", PAC2003, Portland, OR, p. 617.
- [3] P. Koblinski, D. Wolf, F. Cleri, S.R. Phillpot, and H. Gleiter, "On the nature of grain boundaries in nanocrystalline diamond", MRS Bulletin **23**, 36 (1998).