

Improvements in Fabrication of Elastic Scattering Foils Used to Measure Neutron Yield by the Magnetic Recoil Spectrometer

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Received October 20, 2015 Accepted for Publication December 3, 2015 http://dx.doi.org/10.13182/FST15-235

Abstract — The magnetic recoil spectrometer uses a deuterated polyethylene polymer (CD_2) foil to measure neutron yield in inertial confinement fusion experiments. Higher neutron yields in recent experiments have resulted in primary signal saturation in the detector CR-39 foils, necessitating the fabrication of thinner CD_2 foils than established methods could provide. A novel method of fabricating deuterated polymer foils is described. The resulting foils are thinner, smoother, and more uniform in thickness than the foils produced by previous methods. These new foils have successfully been deployed at the National Ignition Facility, enabling higher neutron yield measurements than previous foils, with no primary signal saturation.

Keywords — Deuterated polymer, magnetic recoil spectrometer, glow discharge polymer.

Note — Some figures may be in color only in the electronic version.

I. INTRODUCTION

The magnetic recoil spectrometer (MRS) is a neutron spectrometer based on the coupling of neutron-to-deuteron elastic scattering and magnetic dispersion of the recoil deuterons. An MRS has three primary components: a CD₂ foil, a focusing/bending magnet, and an array of CR-39 detectors. The operational principle is the following: a small fraction of the neutrons from an inertial confinement fusion implosion hit a CD₂ foil, producing scattered recoil deuterons. Via an aperture, the MRS selects a small fraction of these recoil deuterons and the focusing/bending magnet uses the deuteron momentum to direct them toward various CR-39 detector foils where their impacts are recorded for counting at a later time. The CD₂ foil, the first component of this system, is the topic of this discussion.

Casey et al.² and Paguio et al.³ discuss and describe the early fabrication efforts to make the CD₂ foils as well as the most current methodology used by General

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Atomics (GA). CD_2 foils, such as the one shown in Fig. 1, have been successfully used to date as an independent measurement of neutron yields (Yn) and aerial densities (ρR) (Refs. 4, 5, and 6), ion temperature,⁷ and peak shifts⁸ at both the OMEGA and National Ignition Facility (NIF) facilities.

As the Yn of NIF implosions exceeds 2.5×10^{15} and approaches 10^{16} , the existing high-resolution elastic scattering foils (50 µm thickness) produce primary signal saturation, as shown in Fig. 2a. This type of foil also has a limited lifetime in the target chamber environment. It was possible for these foils to be reused multiple times in the spectrometer, but foil irregularities developed after several uses (Fig. 2b) and resulted in an increased uncertainty in the ion temperature (T_i) measurements reported by the MRS (Ref. 2). In August 2013, the Lawrence Livermore National Laboratory (LLNL) and Massachusetts Institute of Technology (MIT) team responsible for the NIF MRS instrument sought new CD₂ elastic scattering foils to extend the MRS instrument's measurement to the higher Yn regime and address lifetime issues.

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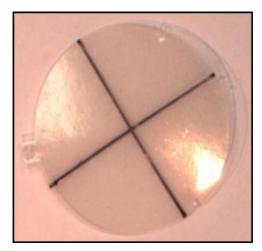
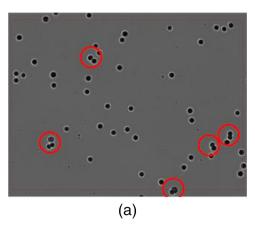


Fig. 1. Low-resolution NIF MRS CD₂ elastic scattering foil



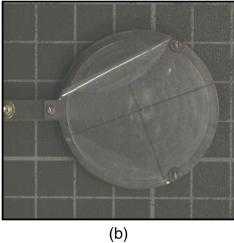


Fig. 2. (a) MRS primary signal tracks for shot N130927 in CR-39 foil, illustrating the track-overlap problem (some saturation). (b) 50-μm-thick CD₂ foil warp and fold after use leading to higher uncertainty in reported measurements.

To avoid primary signal saturation at high Yn, scattering foils with a lower deuteron content are required. Since a lower deuteron content is not easily achievable through polymer chemistry, thinner and smaller area foils were identified as suitable path forward options as long as the basic geometry and precision metrology of the deposited foil was retained (Fig. 3). Simply fabricating CD₂ foils via the known heat press method was not an option since it had been previously demonstrated to be limited to foil thicknesses greater than 35 μm.

Glow discharge polymer (GDP) deposition of a deuterated cross-linked polymer with a D to C ratio of 1.4 (CD_{1.4}), as measured by combustion analysis, has been performed for many years to fabricate thin wall capsules. Additionally, a method of depositing a large-area GDP cross-linked polymer (CH_{1.4}) foil onto a planar substrate had been recently developed to improve the thickness uniformity of samples fabricated in a single deposition/coating run. By applying these two techniques to the MRS foil fabrication, a CD_{1.4} coating could be fabricated that adhered to the substrate and was thinner, smoother, and more uniform in thickness than the CD₂ foils fabricated via the heat press method. In addition, the CD_{1.4} coating has fewer D atoms for each C atom, resulting in a decreased sensitivity of the foil to incident neutrons, allowing the measurement of larger Yn.

II. METHOD

One side of a 2500-µm-thick tantalum substrate foil (Goodfellow, 804-212-30) was polished to a roughness of less than 100 nm root-mean-square (rms) (Ref. 11). This mirrorlike surface is shown in Fig. 4. The tantalum

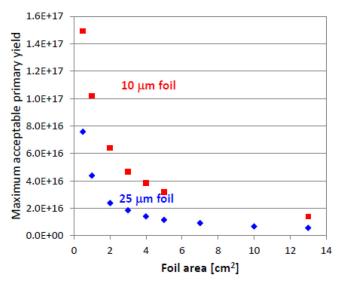


Fig. 3. Modeling results illustrating that upper-yield limit can be increased to $>10^{17}$ using a 10- μ m-thick film of 1 cm² area.



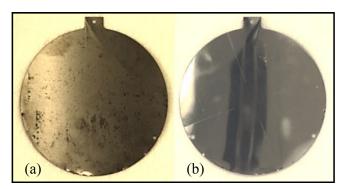


Fig. 4. Tantalum test foils: (a) unpolished side and (b) polished side (with laser-scribed lines).

substrate was subsequently processed by a Trumpf TruMark Station 5000 to texture and mark the polished tantalum surface with the appropriate design, and subsequently to cut the tantalum substrate to the final shape, as shown in Fig. 5.

A coating mask was cut from either a polyimide (PI) film (Kapton, Goodfellow, 907-825-78) or silicon wafer (Silicon Quest International, 230 to 250 μ m thick, type P) to ensure that the deposition would only coat the desired area of the tantalum substrate. Polyimide was used first because it had been used previously to fabricate small GDP dots onto a Si substrate. It was observed that some of the CD_{1.4} was deposited under the PI mask, so a silicon mask was chosen to compare the edge quality of the CD_{1.4} coating. The coating mask apertures were laser machined to diameter, verified with a Nikon MM400 microscope, and centered over the tantalum substrate using the scribed markings to aid alignment. The assembly was supported on a thick silicon wafer and secured.

The tantalum substrate, coating mask, and silicon wafer were precisely placed into a GDP coater with the appropriate deuterium precursor gases, and the deposition was performed over 2 days at the $\delta = 12 \text{ mm}$ setting, creating a uniform thickness of the CD_{1.4} coating, utilizing the off-center rotating technique described by Schoff et al.¹⁰ and the strong CD coating parameters described by Nikroo et al.12,13 The resulting CD_{1.4} coating was measured using white-light interferometry to determine its thickness. To determine the uniformity of the CD_{1.4} coating, several locations on the coating were measured: 10 to 12 points each along the x- and y-axes, and 12 points around the circumference, 1 mm in from the edge. Leica confocal microscopy, as described by Carlson et al.,14 was used to verify the white-light interferometry measurement and standard deviation. Noncontact surface optical profiling was used to determine the surface roughness (rms) of the CD_{1.4} coating using a Bruker Contour GT (also known as WYKO). The WYKO scans were taken using the vertical scanning interferometry mode at 10× magnification. Tilt removal and data leveling were used to ensure the high-mode roughness was captured without distortion from low-mode disruption. Stylus profilometry of coatings near the edge of the deposited films was performed using a Dektak 6M instrument from Veeco (now Bruker Instruments).

III. RESULTS

Tantalum substrates with a $CD_{1.4}$ coating of thicknesses covering 10 to 40 μm and diameters ranging from 11 to 23 mm were fabricated using the described technique and delivered for use in the NIF MRS. Table I

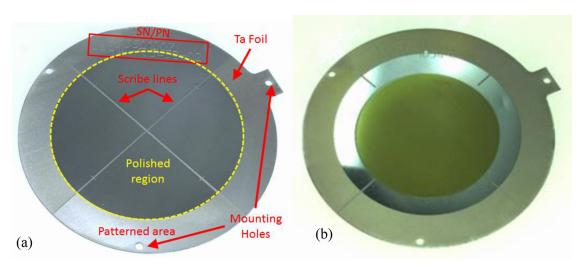


Fig. 5. (a) Polished tantalum substrate with hatching, scribing, and laser cutting completed. (b) 40-mm tantalum substrate with 20-mm CD_{1.4} deposited coating.



CD Foil ID	Mask Type	Diameter (mm)	Average Thickness (µm)	Thickness Variation 1σ (μm)	rms (nm)
13501001	Polyimide Silicon wafer Silicon wafer Silicon wafer Silicon wafer	11.0	10.3	0.1	135
13501002		23.0	45.0	2.0	24
13501003		23.0	36.0	1.0	69
13501004		16.8	23.3	0.2	141
13501005		15.8	19.6	0.3	162

summarizes the diameters and thicknesses of $CD_{1.4}$ foils that have been fabricated. The thickness and diameter of the $CD_{1.4}$ coating can easily be customized to the specifications to extend or tailor the MRS measurement capabilities based on the expected Yn of an experiment.

The foil with serial number 13501001 is shown as an example in Fig. 6a. White-light interferometry was performed to characterize the film thickness and the results are shown in Fig. 6b.

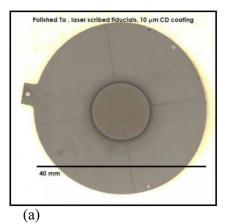
The diameter of a $CD_{1.4}$ coating was measured using a Nikon MM400. The rms roughness for the 40- μ m-thick $CD_{1.4}$ coating on a polished tantalum substrate showed 24 nm rms in a representative 300 μ m² area, whereas the rms for pressed CD_2 foil is \sim 270 nm. The Dektak 6M showed a roll-off area of about 400 μ m (Fig. 7).

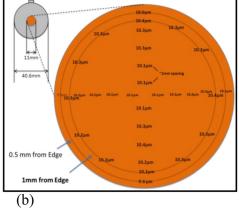
Leica confocal microscopy as described by Carlson et al. ¹⁴ was also used to map the thickness of a $CD_{1.4}$ coating (Fig. 8). The Leica and white-light interferometry were in agreement within 2σ . The advantage of using the Leica system is the automation, as it takes fewer person-hours to get more data than white-light interferometry.

IV. DISCUSSION

The substrate chosen for the CD_{1.4} deposition was tantalum, which was selected for its nuclear stability and limited interactions with incident neutrons. Other potential substrate materials would absorb some of the neutron energy and generate unwanted charged particles, as would occur with stainless steel or aluminum.

To minimize the uncertainty in the Yn measurement, the thickness of the deposited $CD_{1.4}$ foil must be accurately measured and mapped. Goodfellow specifies its tantalum foil thickness at $\pm 10\%$ in the as-rolled condition, so a caliper measurement of the $CD_{1.4}$ foil that included the tantalum substrate could have up to 25- μ m variation in the thickness measurement. This amount of measurement variation was not acceptable. The rough surface of the off-the-shelf foil also could increase the variation in the actual CD foil thickness. Polishing the tantalum substrate provided a uniformly flat surface, reducing the thickness variation point to point in any deposited coating and also facilitated the use of white-light interferometry to measure the coating thickness, enabling 0.3 μ m repeatability.





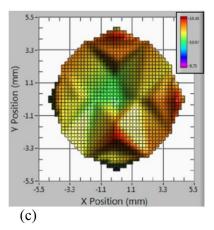


Fig. 6. (a) Image of tantalum substrate, serial number 13501001, with a 10-μm CD_{1.4} coating; (b) diagram showing where the 29 white-light interferometer thickness measurements were made; and (c) interpolated plot estimating surface uniformity.

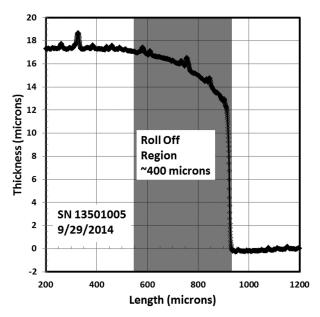


Fig. 7. A stylus profilometry (Dektak surface profiler) trace of a film's edge profile as deposited through a silicon mask. The edge roll-off occurs in approximately 400 µm. Roll-off was determined from the point where the thickness drops off more than the thickness variation of the foil to the level of the tantalum substrate.

The laser scribing and cutting produced a tantalum substrate that met the design specifications for the mounting foil and allowed direct replacement of present CD₂ foils; i.e., there was no re-engineering of the mounting configuration or hardware. The scribe marks enable visible alignment of the foil inside the target chamber for optimal measurement. The hatching around the outside edge of the tantalum substrate reduced reflectivity during alignment.

Figure 9 demonstrates the difference between the two mask materials that were tested. The silicon wafer mask produced a sharper roll-off than the PI mask. This is likely caused by undercutting of the $\mathrm{CD}_{1.4}$ being reduced with the Si mask: the stiff Si tends to be in more intimate contact with the Ta substrate than the flexible PI. The sharper coating edge is more desirable because it helps to reduce the uncertainty in the amount of deuterium present in the coating; therefore, the Si mask was used for subsequent coatings.

Deposition of CD_{1.4} polymer has three primary advantages over the heat press method, as summarized in Table II. First, very thin coatings with a specified area can be deposited directly onto the tantalum substrate, which provides support without the need for holding the foil. The heat press method is presently

CH4_SCD_13501005 Coating Thickness

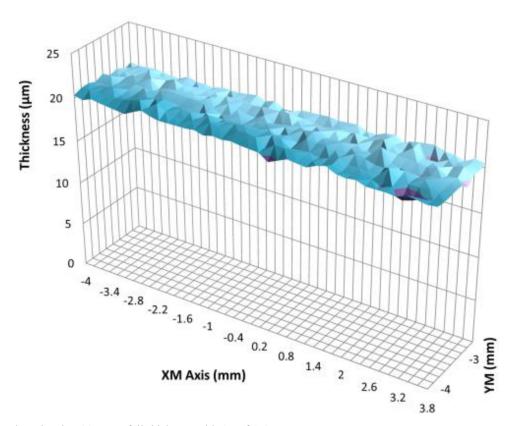


Fig. 8. Leica data showing 20.5- μm foil thickness with 1σ of 0.4 μm .



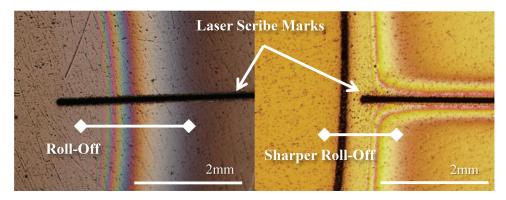


Fig. 9. White-light interferometry of the edges of the coatings, comparing and contrasting the PI and silicon masks. Observation of interferometric fringes from the coating provides a good assessment of edge roll-off and coating that occurred under the mask. The left image shows a gentle roll-off produced by the PI mask, determined by the large size of the interferometric fringes; the right image shows a steeper roll-off produced by the Si mask, illustrated by the shorter length of the interferometric fringes.

TABLE II
Summary of Coated CD Advantages Over Pressed CD

	Pressed CD	Coated CD _{1.4}
Lower limit of thickness	35 μm	≤10 µm
Surface roughness	500 to 1000 nm	40 to 200 nm
Thickness uniformity	5 μm	≤2 µm

limited to producing foils greater than 35 μ m. If CD₂ polymer could be pressed to 10- μ m foil thickness, it would be flimsy at the large areas needed for this application. Additionally, it would continue to suffer from the warping and bending that is currently observed in the 50- μ m-thick foils and each area design would require a new mounting and holding setup.

Second, the $CD_{1.4}$ coating has low-amplitude surface roughness in high modes. The heat press method produced foils with a rms roughness of 500 to 1000 nm, whereas the deposition process produced foils with a rms of 40 to 200 nm.

Third, thin deposited films demonstrate a thickness uniformity similar to or better than foils produced by the heat press method. The heat press method produced films with a standard deviation of thickness of 5 μ m, whereas the CD_{1.4} coatings had standard deviations of 2 μ m or less. The thickness uniformity of an elastic scattering foil is very important to reducing systematic error in a MRS measurement.

V. CONCLUSION

This paper does not focus on the experimental results of interest to the physics community; however, it is important to note that the new foils performed effectively. On July 7, 2014, the MRS super-high-resolution elastic scattering foil was used on NIF shot N140707. The LLNL and MIT MRS teams reported that the foil performed as expected. It did not warp, deform, nor delaminate from the substrate. They reported a preliminary primary Yn of 4.3×10^{15} with $\pm 6.7\%$ uncertainty. 15

In summary, a novel GDP deposition technique was used to fabricate large-area thin CD_{1.4} foils to a uniformity of less than 5% over a 20 mm diameter (Fig. 10 shows an image of the final mounted foil after a shot). Improvements to the thickness uniformity, surface roughness, and thickness uncertainty of the MRS elastic scattering foil were realized. This technique trades off deposition rate for deposition uniformity and has enabled a new class of large and nominally flat foils. Re-engineering the fabrication process for these elastic scattering foils led to an improvement in the systematic error parameters associated with the elastic scattering foil.

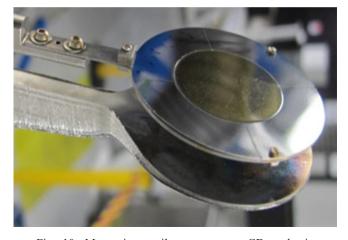


Fig. 10. Magnetic recoil spectrometer CD_{1.4} elastic scattering foil post shot inspection after shot N140707 (picture courtesy of LLNL) (Ref. 16).



Acknowledgments

This work is supported by the U.S. Department of Energy under contracts DE-NA0001808 (GA) and B585716 and B595867 (LLNL).

References

- J. A. FRENJE et al., "A Neutron Spectrometer for Precise Measurements of DR Neutrons from 10 to 18 MeV at OMEGA and the National Ignition Facility," *Rev. Sci. Instrum.*, 72, 854 (2001); http://dx.doi.org/10.1063/1. 1323243.
- D. T. CASEY et al., "The Magnetic Recoil Spectrometer for Measurements of the Absolute Neutron Spectrum at OMEGA and NIF," Rev. Sci. Instrum., 84, 043506 (2013); http://dx.doi.org/10.1063/1.4796042.
- 3. R. R. PAGUIO et al., "Fabrication of Thin CH and CD Films and Patterned Films Using a Heat Press Technique for the NIF and OMEGA Magnetic Recoil Neutron Spectrometer," *Fusion Sci. Technol.*, **63**, 268 (2013); http://dx.doi.org/10.13182/FST63-2-268.
- J. A. FRENJE et al., "First Measurements of the Absolute Neutron Spectrum Using the Magnetic Recoil Spectrometer at OMEGA (Invited)," *Rev. Sci. Instrum.*, 79, 10E502 (2008); http://dx.doi.org/10.1063/1.2956837.
- 5. T. CASEY et al., "Measuring the Absolute Deuterium-Tritium Neutron Yield Using the Magnetic Recoil Spectrometer at OMEGA and the NIF," *Rev. Sci. Instrum.*, **83**, 10D912 (2012); http://dx.doi.org/10.1063/1.4738657.
- J. A. FRENJE et al., "Probing High Areal-Density Cryogenic Deuterium-Tritium Implosions Using Downscattered Neutron Spectra Measured by the Magnetic Recoil Spectrometer," *Phys. Plasmas*, 17, 056311 (2010); http://dx.doi.org/10.1063/1.3304475.

- M. GATU JOHNSON et al., "Neutron Spectrometry—An Essential Tool for Diagnosing Implosions at the National Ignition Facility (Invited)," *Rev. Sci. Instrum.*, 83, 10D308 (2012); http://dx.doi.org/10.1063/1.4728095.
- 8. M. GATU JOHNSON et al., "Measurements of Collective Fuel Velocities in Deuterium-Tritium Exploding Pusher and Cryogenically Layered Deuterium-Tritium Implosions on the NIF," *Phys. Plasmas*, **20**, 042707 (2013); http://dx.doi.org/10.1063/1.4802810.
- S. A. LETTS et al., "Fabrication of Polymer Shells Using a Depolymerizable Mandrel," Fusion Technol., 28, 1797 (1995); http://dx.doi.org/10.13182/FST28-5-1797.
- 10. M. E. SCHOFF et al., "Fabrication of Large-Area Glow Discharge Polymer–Deposited Foils," *Fusion Sci. Technol.*, **70**, 372 (2016); http://dx.doi.org/10.13182/FST15-243.
- 11. G. C. RANDALL et al., "Developments in Microcoining Rippled Metal Foils," *Fusion Sci. Technol.*, **63**, 274 (2013); http://dx.doi.org/10.13182/FST63-2-274.
- 12. A. NIKROO et al., "Recent Progress in Fabrication of High-Strength Glow Discharge Polymer Shells by Optimization of Coating Parameters," *Fusion Sci. Technol.*, **41**, 214 (2002); http://dx.doi.org/10.13182/FST41-214.
- 13. A. NIKROO et al., "Mechanical Properties of Thin GDP Shells Used as Cryogenic Direct Drive Targets at OMEGA," *Fusion Sci. Technol.*, **45**, 229 (2004); http://dx.doi.org/10.13182/FST45-2-229.
- 14. L. C. CARLSON et al., "Automation of NIF Target Characterization and Laser Ablation of Domes Using the 4pi System," *Fusion Sci. Technol.*, **67**, 762 (2015); http://dx.doi.org/10.13182/FST14-833.
- 15. M. G. JOHNSON, MRS foil improvements, Massachusetts Institute of Technology, Personal Communication (2015).
- 16. R. BIONTA, MRS foil improvements, Lawrence Livermore National Laboratory, Personal Communication (2015).

