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Section A

Improved carbon micro-ribbon targets and stripper foils for the IUCF cooler ring *

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Carbon micro-ribbons have been made which are stronger and a factor of three thinner than any previously made at IUCF. Ribbons of 4.2 $\mu g/cm^2$ thickness and 3.3 μm width were mounted to span 22-mm lengths. Also, open-edged (32-mm) stripper foils of 4-$\mu g/cm^2$ carbon have been prepared with significantly reduced curling at the open edge. For both the foils and ribbons, the progress is the result of efforts to produce uniform, densely distributed microscopically-sized features on the surface of glass substrates.

Along with the grinding technique currently used to texture the substrates, a heat treatment method which improves the electrical conductivity of carbon micro-ribbons, and a method to make wire-grill evaporation masks with more closely regulated spacing of the wires is described. Also, initial experience depositing ultra-thin layers of several desired Cooler target materials onto the micro-ribbons is reported briefly. Carbon micro-ribbons are thought to have considerable potential as backings for Cooler targets.

1. Introduction

At the 15th World Conference of the INTDS in 1990, we recounted the progress made at IUCF toward the fabrication of carbon micro-ribbons (CMRs) and stripper foils since work began on them in 1986 [1]. The thinnest micro-ribbons to be successfully floated in water from a substrate, mounted across a 32-mm wide opening, and moved rapidly through an IUCF Cooler beam, were 7-$\mu g/cm^2$ thick and 11.6 μm wide. They were made by vacuum evaporation–condensation onto smooth glass substrates, using a betaine–sucrose parting agent [2], or onto etched stainless steel with the use of a detergent parting agent (Tergitol 8: 40% solution of Tergitol, Sigma Chemical Co., St. Louis, MO).

Although ribbons produced on etched stainless steel (ESS) have a less corrugated surface than ribbons made on betaine–sucrose (and thus are thinner to the Cooler beam), they are brittle in comparison. When rotated or moved back-and-forth through the Cooler beam (at 35–40 Hz), these ribbons often broke at either of the two ends attached to a frame. The failures led to the development of hybrid ribbons from ESS polished first with Tergitol 8 and then coated at the ends with crystallized betaine–sucrose. The hybrids were a successful approach resulting in the thinnest usable ribbons of the time to be rapidly rotated through the Cooler beam [1].

In work to achieve thinner carbon micro-ribbons since 1990, the limitations presented by ESS substrates began to appear. The most severe of these seem to be the irregular size and distribution of the crystal grains and the width of the grain boundaries (0.5–1.0 μm after the metal is etched) relative to the width of the ribbons achieved. Eleven-micrometer wide and 7 $\mu g/cm^2$ thick ribbons which replicated these underlying surface features could be picked up and mounted with a success rate of 10% or less.

For some months thereafter, the path to thinner ribbons was thought to involve the use of a substrate often used in the past: stainless-steel ferrotype plate (Photo Materials Inc., Chicago, IL), with a polished layer of detergent as the parting agent. Encouraging float-off and pick-up results were achieved when the wire grill mask which was used to define the width of the ribbons was aligned with the direction of the as-received polish marks on the ferrotype plate [1]. Further production trials, however, indicated that good results were not reproduced with regularity. The most probable reason seems to be that the side walls of small sharp-featured surface scratches seen on the metal were partially shadowed during the carbon deposition. Oriented across the width of a ribbon, the scratches surely compromise the ribbon's tensile strength. It was also noted that the ferrotype surface was much smoother than ESS, hence one would expect less elas-

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ticity in the ribbons which replicate it. These limitations became more severe as attempts were made to obtain ribbons with fewer atoms per unit length.

Speculating that an ideally textured substrate surface for CMRs and open-edge stripper foils was likely to be uniformly lumpy on a sub-to-few-micron scale, a search was begun for such existing surfaces. Unpolished silicon semiconductor disks emerged as a promising substrate candidate when viewed at a magnification of 1000 with an optical microscope. This surface was acid etched (20–25 s in 8 parts of 70.5% HNO_3, 1 part 48% HF) to smooth the angular breakout pits, polished with a release agent (Tergitol 8), and CMRs were vacuum deposited onto it. In several trials the CMRs did not release from the silicon in water. The reason was evident at a magnification of 1000 with a scanning electron microscope (SEM). The surface features were quite deep and angular, causing the CMRs to lock into the surface and the edges to be defined poorly. Although the search for a suitable existing surface may yet be fruitful, other work was begun to find ways of abrading substrate surfaces as uniformly as possible on the aforementioned scale.

2. Abrasion texturing of substrate surfaces for micro-ribbons

2.1. Summary of development trials

Ferrotype-plate substrate surfaces were the first ones we attempted to improve by various grinding techniques. While the results from machine-aided methods were poor, longer micro-ribbons were obtained more frequently when small pieces (15 mm × 60 mm × 0.45 mm thick) of ferrotype plate were hand ground with a water slurry of 5 μm particles of Al_2O_3. However, surface textures with microscopically small features could not be produced without also producing numerous random scratches in the surface.

The initial attempts at texturing 60 mm × 20 mm × 1 mm thick pieces of glass microscope slides were done with the use of a circular-motion four-speed grinder–polisher with a 20 cm diameter platen. No matter what the rotational speed, polishing cloth, size of abrasive particle, lubricant, or time of grinding used, the observable surface features (at 1000 ×) were clearly inferior to those won with hand grinding. The grinder–polisher produced curved scratches and/or a polished semblance of scattered wood-grain-like damage sites on the glass.

The next set of trials on glass had a common feature: a plastic guide was used to limit the orientation of most extended-area surface damage to the major axis of the glass. By sliding pieces of glass (15 mm × 60 mm × 1 mm thick) back and forth along the guide, they were abraded either on wet bound-particle paper (sandpaper) or on a water slurry of unbound grinding particles. The sandpapers tested were: 600, 1000, 1200, and 2000 grit (3M WETORDRY Sanding Paper, 411Q and 414Q). The grinding was done as the paper was continuously rinsed with de-ionized water. Using 1200 grit (5.6 ± 0.5 μm) sandpaper, about 10% more micro-ribbons were successfully mounted, when compared with those condensed on ferrotype plate. Water slurries of alumina polishing and grinding powders (Buehler, Lake Bluff, IL; 5, 12.5, and 18–22 μm particle size) used with the same motion, produced glass

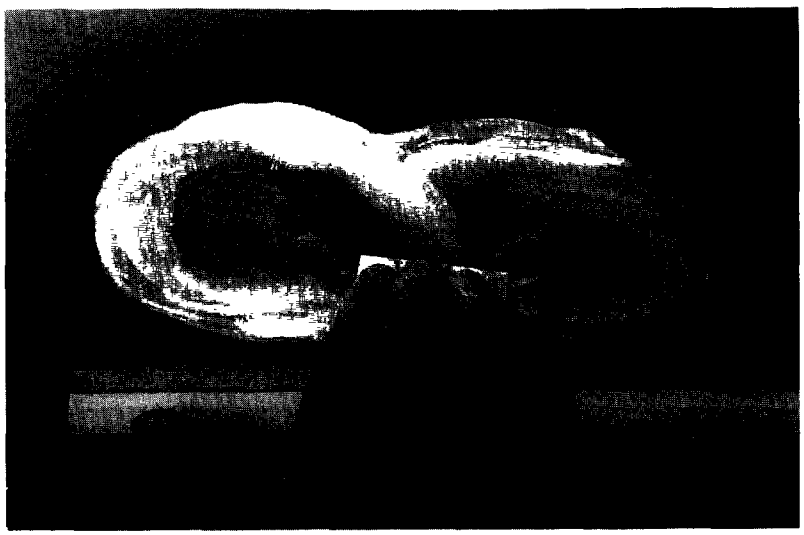

Fig. 1. Texturing glass with 5 μm Al_2O_3 and water on plastic plate.

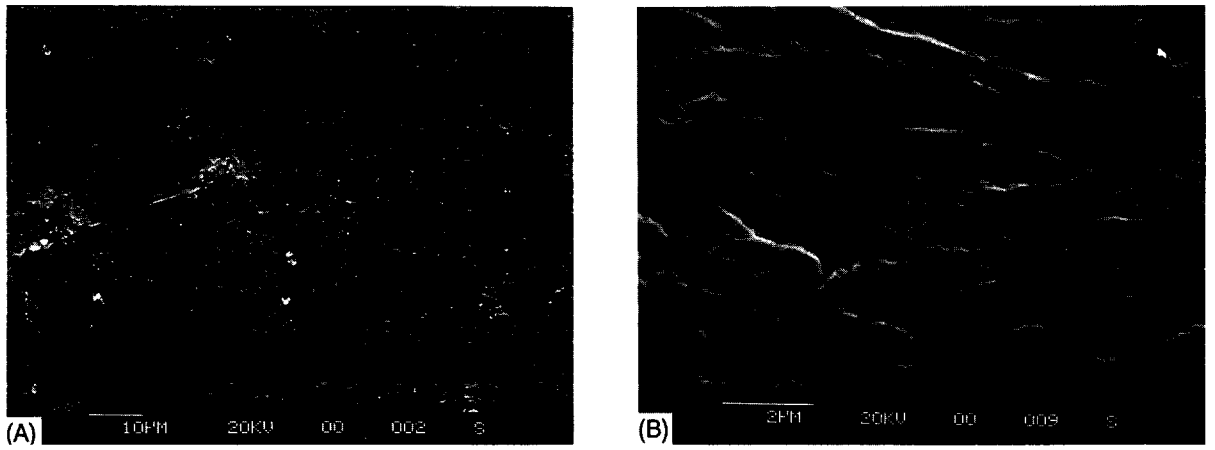

Fig. 2. SEM photomicrographs of a glass substrate textured with 5 μm Al_2O_3 particles: (A) with two carbon micro-ribbons (narrow, dark strips) at a magnification of 1000; (B) softly eroded features of the same surface at a magnification of 10000.

surfaces which were better still. However, the surface scratches formed with either variation of this technique were clearly responsible for the large number of ribbons which broke in lengths too short to be of use ($\leq$ 20 mm). Much better results were obtained by hand grinding the glass on a flat surface without a guide.

Three types of material were evaluated as a flat backing surface for grinding with unbound particles: glass, steel, and plastic. Plastics produced the most closely distributed homogenous effects with glass substrates. Predictably, perhaps, grinding glass on a glass surface resulted in numerous occurrences of break-out areas consisting of 2–10 μm sized pits with sharp angular features. Steel produced fewer of these, but the likelihood of contaminating the surface with steel advised discontinued use of this material. Plastic grinding surfaces produced a high density of microscopically sized features and few surface pits.

2.2. Current procedure for glass

Most of the 6–15 μm wide micro-ribbons (and some as narrow as 3–4 μm) in 4–6 μg/cm^2 deposits of

Fig. 3. Two 32 mm wide stainless-steel target frames each with a carbon micro-ribbon mounted horizontally at the midpoint. The frames (ribbons not visible), shown above the oven, are lowered and centered for a heat treatment at 1300 K between the two inner foil strips.

carbon have been floated and lifted successfully when glass substrates were abraded in the following way.

A microscope slide (Fisher Scientific, Fisher*finest* Premium, no. 12-544-1, 25 mm × 75 mm × 1 mm) is scored and broken to dimensions of 13–15 mm × 60 mm, then cleaned with de-ionized water. A slurry of levigated 5-μm alumina polishing compound (Buehler no. 40-6435-080) with de-ionized water is prepared on a clean flat surface of methyl methacrylate (Lucite or Plexiglass).

The slide piece is ground by hand, using light-to-moderate pressure, as the glass is moved in a figure-8 pattern over an area of about 6 mm × 20 mm (fig. 1). Initially, a thin slurry of the grinding particles produces fewer random scratches in the glass than a thicker, more viscous slurry. As the surface is progressively damaged, the angular breakouts formed early in the process are eroded into rounded features. If grinding the surface for 3–5 min reveals that it was not flat initially, one is advised to start with a new piece. An adequately abraded surface, viewed with an unaided eye, has a uniform frosted appearance. When viewed at high magnification with an SEM (fig. 2A,B), a rather soft-featured landscape is apparent, which is likely to have facilitated the leap to thinner CMRs.

3. Annealing of carbon micro-ribbons

Inconsistent Cooler-beam lifetimes with carbon micro-ribbon targets were eventually understood to be the result of large variations in the electrical conductance of the graphite powder cyanoacrylate-ester (GPCE) adhesive mix [1] used to bond the ribbon ends to metal target frames [3]. In the interim period, the most likely cause of the inconsistent lifetimes was attributed to charge accumulation on the ribbons due to the high value of the measured electrical resistivity of individual ribbons: 0.18–0.29 Ω cm [4]. This range of values, a factor of 10^3 higher than the listed value for synthetic graphite, was sufficiently large to prompt experiments which sought to lower it by heat treating individually mounted ribbons. Decreasing the resistance by a factor of 10^3 proved easy to accomplish.

For the heat treatment setup (fig. 3), a single ribbon was glued at the ends to span the 32 mm opening of each stainless-steel rectangular target frame (0.5 mm thick with outside dimensions of 36 mm × 50 mm). The GPCE adhesive mix was found adequate to adhere the ribbons to the frames during high temperature heat treatment in high vacuum. The oven was assembled in a 45 cm diameter vacuum evaporator. It consisted of two strips of tantalum foil (25 mm wide × 135 mm long × 50 μm thick) clamped between two water-cooled electrodes mounted through the baseplate. The distance separating the Ta-strip heating elements was 21 mm. A U-shaped cover of 1 mm thick Ta was fitted to each electrode post to shield them from excessive heat. Flat metal clamps for the ends of the Ta strips were also made of 1 mm thick Ta.

After centering two horizontally oriented ribbons (one per frame) in the space between the Ta strips and providing heat shielding (three layers of Ta sheet, each 50 μm thick and 1 cm apart) around the assembly, the bell jar was evacuated to 7×10^{-5} Pa (5×10^{-7} Torr). A pressure of less than 3×10^{-4} Pa was maintained

Fig. 4. Setup for winding wire-grill masks: (A) a small wire-winding table and a computer-controlled wire-positioning and tensioning bed; (B) close-up showing two Al ring frames (back to back) with wire and adhesive, spring-loaded axle, optical relay and trip rod to order wire position increments, and a rubber coupling to an ac motor.

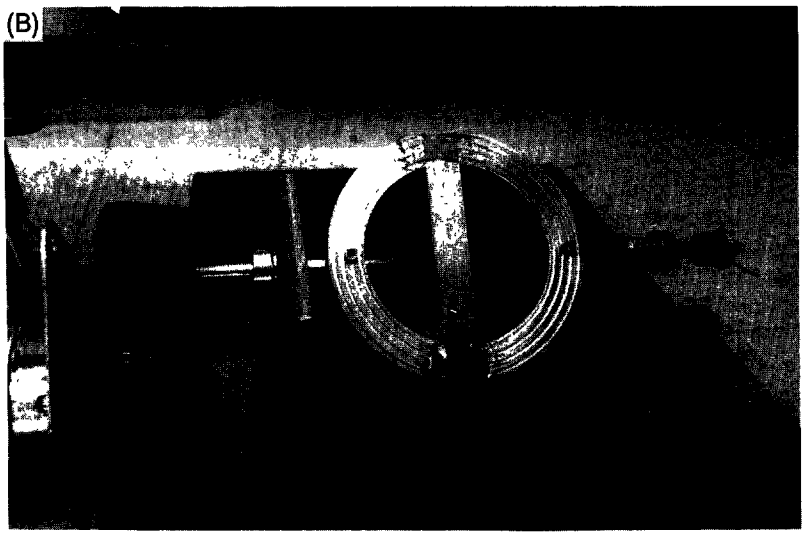

Fig. 4. (continued).

during the time needed to reach about 1300 K. That temperature, measured with an optical pyrometer, was the highest obtainable with the 10 V tap on the 2 kVA power supply used. It was sustained for 9–10 min. A period of approximately 5 min was used to lower the temperature of the Ta strips gradually to below incandescence. Then the power supply was turned off and 30 min were allowed for the stainless-steel target frames to cool before the chamber was vented to room air.

When the ribbons were removed from the oven, a dab of silver paint was applied to the bonding areas of the frames and ribbons, to cover the GPCE adhesive. The paint established an ohmic contact and also served to tighten the ribbons which lengthened, perhaps by 5%, on the frames during the heat treatment. Diffusion of the thin carbon targets into the stainless steel frames, as reported in ref. [5], was not a possibility because the micro-ribbons were imbedded in the adhesive mix and not in physical contact with the frames. Aside from the 10^3 increase in the electrical conductivity and the small increase in length of the ribbons, other changes such as an increase in fragility have not been noted. Although heat treatment of the ribbons is currently not required, the method is quick and effective. Silver paint and colloidal silver in water are used currently in preference to the GPCE adhesive mix for bonding ribbons to frames which will not experience high heat.

4. New wire grill evaporation–condensation mask

The goal to produce wire-grill masks wound with more precise wire spacing arose as a consequence of the greater frequency of success obtained for lifting and mounting 1–6 μm wide micro-ribbons. The accuracy of the winding process used thus far was thought to be limited to ±10 μm largely by the table on which the wires were wound before they were glued to aluminum ring frames and cut from the table. An improvement was desirable since the original wire-grill masks made many ribbons in widths no longer of interest.

By using the computer-controlled wire positioning device (used previously with the table) [1] to wind wire directly onto two aluminum wire frames (fig. 4A, B), the accuracy of the wire spacing was improved to approximately ±4.5 μm. The axle of the device is a precision-ground steel rod 6.35 mm in diameter. It is supported by precision bearings. Lateral movement of the axle is restrained by spring loading one end. To minimize vibration-induced winding errors, the supports for the bearings are short, the base is heavy, and the coupling to the motor was made from a large rubber stopper. The rotational velocity was 2 rpm.

Winding 20 μm diameter gold-coated tungsten wire in position increments of 25 μm produced many more of the desired 1–6 μm spacings than were previously possible. The wire was wound with 0.5 N tension force versus a 1 N force previously used to wind 38 μm diameter wire. The smaller diameter of the wire and the closer spacing between the windings hindered anchoring the wire to the ring frames with adhesive. To hold the tension force on the wires, it was necessary to cut grooves in the top and sides of the frame, wind the wire across them, and cover the grooved area and wire liberally with 5-min epoxy (Devcon, Danvers, MA 01923). This adhesive is not the strongest available, but

it may be removed easily when desired by heating it to about 330 K.

5. Vacuum coating of carbon micro-ribbons

Several elements of interest as unpolarized targets in the Cooler ring have been condensed on micro-ribbons in recent proof-of-principle vacuum depositions. There was no indication that the float-off and handling characteristics of the ribbons were affected adversely by coating carbon ribbon deposits on glass with a layer of 0.8–1.5 $\mu g/cm^2$ indicated thickness of Mn, Co, Fe, Pb, or Ru. The source-to-substrate distance was the same as that used for deposition of the ribbons (14.5 cm). Although the single-hearth e-gun of the lab required the vacuum chamber to be cycled to atmospheric pressure before another element could be evaporated (subsequent to a carbon evaporation), two small pieces of Scotch tape which held the glass substrate to the wire-grill mask frame adequately preserved the registration between them.

The accuracy of the quartz crystal monitor for ng/cm^2 coatings on the carbon micro-ribbons is an issue yet to be addressed fully. The R.D. Mathis model TM-100 thickness monitor in the target lab will be calibrated and checked for linearity in the monolayer thickness range of an element by using an ultra-microbalance (100 ng) to determine the thickness of larger area deposits. In ref. [6], Maier-Komor reported the accuracy limitations of several thickness monitors, one of which was the TM-100. From that work, two precautions seem evident to minimize the error in a displayed super-thin thickness on this instrument: (i) the quartz crystal should be new or only very lightly loaded and (ii) a shutter should be used which permits the monitor to stabilize and register a slow evaporation rate before the micro-ribbons are exposed to the evaporant.

6. Stripper foils evaporated onto textured glass

Split-free films of 4–7 $\mu g/cm^2$ vapor condensed carbon, 40 mm × 25 mm in area, were floated from glass which had been textured with 12.5 μm particles in the same manner as for the micro-ribbons. Correspondingly, Tergitol was used as the parting agent. The angle of the substrate glass to the water surface for the floating operations was approximately 20°. It was not possible to float large areas of split-free films from glass textured with 5 μm particles.

After the carbon was picked up on C-shaped frames with a razor blade clipped to the two frame ends, so that it closed the open side, the carbon was cut free along the razor blade's sharp, straight edge. Thereafter, the spring clips were removed and the razor blade was allowed to fall away [1]. Unlike the substrates in our earlier report, textured glass produces films which may be mounted tautly on the frames. This is done by positioning the razor blade to the side (rather than at the bottom of the frame) during a slow withdrawal of the carbon film from the float-off bath.

In trials, substrate glass for the stripper foils was also abraded with the bound-particle papers tested for use with the micro-ribbons (section 2.1). The papers were used to produce parallel scratches on the glass, which when oriented perpendicularly to the open edge produced a resistance to curling of the foil along that edge. The method was simply to rinse the paper continuously as the glass surface was rubbed back and forth on the paper along a plastic guide at the edge of the glass. The results were not as satisfactory as those

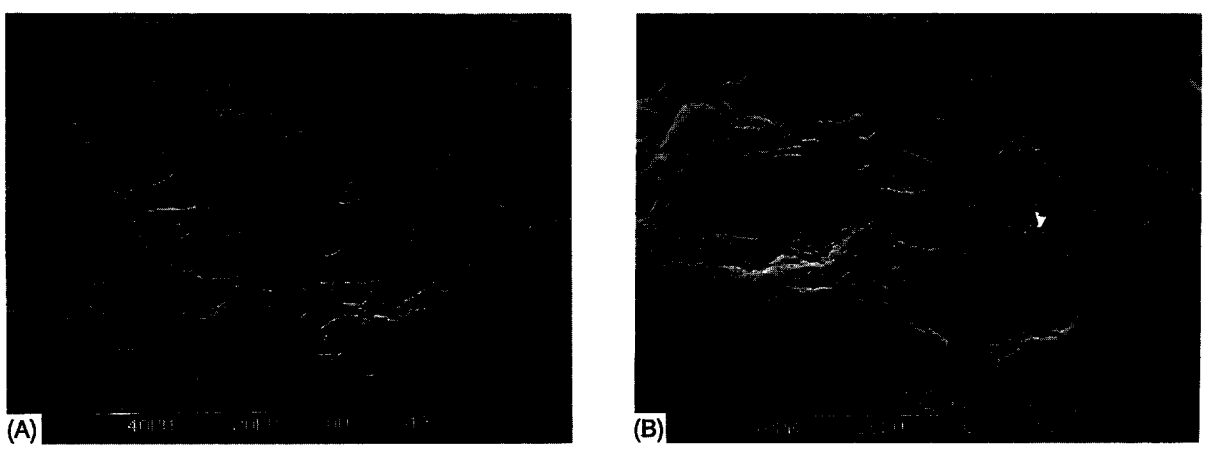

Fig. 5. SEM photomicrographs of a glass substrate (for open-edge stripper foils) textured with 12.5 μm Al_2O_3 particles and 1000 grit paper: (A) at a magnification of 1000; (B) at a magnification of 5000.

achieved with slurries of the 12.5 μm unbound-alumina particles and the figure-eight grinding pattern (fig. 1) used in the above method.

The most tear-resistant open-edged stripper foils of 10–15 μg/cm^2 were made on a surface produced by texturing the glass surface first with 12.5 μm particles as described. The glass was then subjected to directionally oriented scratching with 1000 grit paper. Viewed with a magnification of 1000 and an angle of 45° (fig. 5A,B), the resulting surface exhibited roughly parallel scratches on 5–25 μm sized, mostly smooth areas which were located between 3–25 μm sized areas covered with small pits.

For stripper foils of 4–6 μg/cm^2, it was neither necessary nor desirable to scratch the glass after grinding the substrate glass with 12.5 μm particles. These thinner foils had very little or no tendency to curl at the open edge. The scratches only produced weaknesses in 4–6 μg/cm^2 mounted foils which made them more likely to tear back from the open edge along the sides of the C-shaped frames.

7. Results and conclusions

Condensing carbon onto substrates of textured-glass microscope-slide pieces polished with a detergent has produced the best carbon micro-ribbons and stripper foils for the IUCF Cooler in our experience. Once successfully textured, these substrates may be and have been used many times with equally satisfactory results.

Further development work on the methods used for texturing glass substrates will focus on eliminating large-scale imperfections attributable to the grinding–polishing procedures. The use of a vibratory polishing machine (e.g., Buehler model: Vibromet 2) may produce surfaces with fewer imperfections than is currently possible with hand grinding techniques. With mechanical vibratory motion, it will be possible to investigate the effects of polishing glass pieces for long periods of time using particles 0.3–2 μm in size.

A modified saddle-field ion source is being constructed at IUCF. The design details follow that of Muggleton [7]. As evidenced in refs. [7–9], sputter deposition will be an ideal method of coating carbon micro-ribbons with desired enriched target isotopes.

At this writing, an improved carbon micro-ribbon of 4.2 μg/cm^2 and 3.3 μm width has been used twice for test runs to calibrate detectors in a Cooler experiment which requires a carbon target [10]. Unpolarized proton beam energies of 200 and 350 MeV with stored currents of about 1 mA per filling cycle were available during the last run. The beam–target interaction rate was optimized by a target-positioning feedback loop which allows the target to skim the edge of the Cooler beam. The target ladder is shown in fig. 6. An interaction luminosity of 10^{29} cm^{-2} s^{-1} was provided by the feedback loop. Beam lifetimes of approx. 20 s were recorded when 600 μA of stored beam was used before the ring was refilled. Although higher stored beam currents are desired, this combination of factors is thought to be adequate to do the experiment [10]. The micro-ribbon survived both test runs.

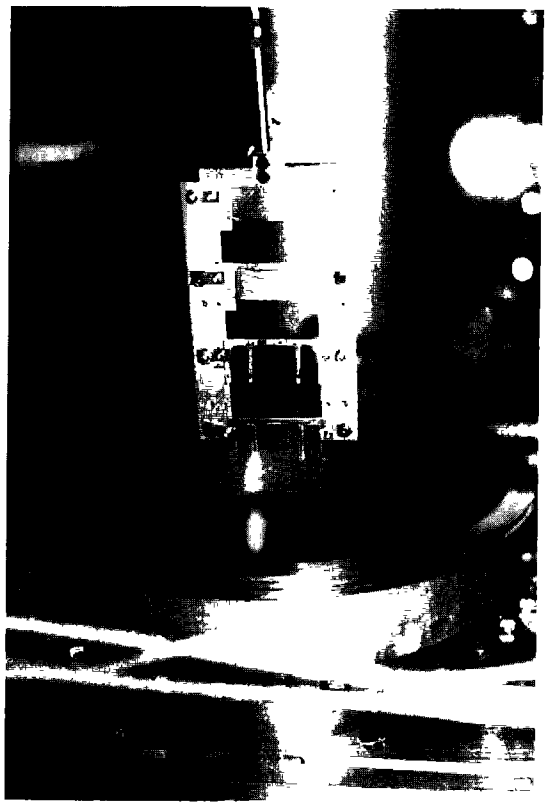

Fig. 6. Target ladder for beam-skimming with two open-edge stripper foils and two carbon micro-ribbons (ribbons not visible on 22 mm spaced horizontal projections at right); α source at left for detector calibration.

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