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Section II. Carbon stripper foils

Preparation of carbon micro-ribbon targets and open-edged stripper foils for the IUCF cooler ring *

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The use of 5–15 μg/cm² films of vacuum-evaporated carbon as targets and stripper foils in the IUCF cooler ring requires compliance with the characteristics of the ring. One way to circumvent the upper limit in target thickness of 10^{15} nuclei/cm² (less than 100 ng/cm²) for stationary targets is to move a somewhat thicker ribbon quickly through the beam. Our current methods for fabricating, handling, and mounting carbon micro-ribbons are described.

Stripping-injection of 90 MeV H_2^+ beam from the cyclotron into the cooler is accomplished using 5–15 μg/cm² stationary carbon foils. The requirement that these foils be mounted on a "C"-shaped frame with an open side of 31 mm, proved surprisingly easy to fulfill A technique will be described for producing single-layer stripper foils which have a relatively straight edge that is unsupported across one side.

Introduction

Targetry for the IUCF cooler ring is proceeding along several paths and is being conducted by a cooler target group. Supersonic gas jets, gas storage cells, charged-microparticle streams, skimmer strips, and micro-ribbons have all been used as targets in our ring. The list of available targets grows slowly longer, although each cooler target typically requires substantially more effort than those for use in a single-pass accelerator.

Foremost among the target-limiting factors is the requirement that the target must not depopulate the beam of stored, cooled ions too quickly. The amount of time for 60% of the beam to be depleted must usually be well above 20 s to balance the overhead time of the ring (to fill, strip, cool, and ramp).

1.1. Carbon micro-ribbons

Interest in the possibility of providing carbon strip targets (ribbons), prepared by vacuum evaporation-condensation, began when our attempts to thin commercially available carbon fibers were only marginally successful. These attempts focused on thinning a 10 mm long central section of 30 mm long, 6.5 or 10 μm diameter fibers with the use of an argon/oxygen glow discharge. The approach was abandoned when thickness variations along the length of the fibers were discovered to be the likely cause of corresponding nonuniformities as large as 0.7 μm in the thinned portions.

In initial attempts to produce carbon micro-ribbons, a variety of soaps, salts, an anionic wetting agent, hexadecylamine, betaine/sucrose, and straight sucrose were tested as release agents on smooth glass and metal surfaces. We were unable to make thin ribbons successfully on smooth-surfaced substrates with any release agent other than betaine/sucrose. However, limitations to the use of betaine for micro-ribbons emerged: (i) the crystallized surface could not be made smooth enough to prevent carbon from being deposited under the wires of the grill, linking the ribbons with thinner carbon in some locations, and (ii) the microscopically lumpy surface of betaine (rounded crystals which vary in size and rise from a base layer which varies in thickness) produced ribbons with more atoms/cm of length than expected. Nevertheless, once mounted, the ribbons from betaine have proved to be remarkably durable. They can be rotated at 35–40 Hz in vacuum for several days (fig. 1) and they survive slow pumpdowns and vents well.

Somewhat less forgiving, but thinner, ribbons have been made on etched stainless steel without the use of betaine. The parting agent for these ribbons was Tergitol 8, a commercially-obtained detergent. In use, the ribbons have undergone the jump-rope rotation successfully, but have broken at the frame-connection points when the target frame was repositioned without first reducing the electric field producing the motion. The technique is quite promising, particularly if the ribbons are not rotated through the cooler beam but stretched across frames which move back and forth through it.

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Fig 1. Strobe-illuminated carbon micro-ribbon 8 μg/cm² thick and 30 μm wide, in vacuum and rotating in an electric field at 35–40 Hz The distance between the wires is 32 mm.

This method avoids the use of an electric field and gave superior results in the most recent cooler run.

Hybrid ribbons have been made on etched stainless steel by using betaine on the ends of the steel and a detergent in the center (target) section. These ribbons release from the steel easily and present no additional handling problems. Because they have more durable ends and a thinner middle, they may soon be the preferred ribbon.

The first IUCF micro-ribbons were produced and mounted in 1986 [1]. They were 20 μg/cm, 35 μm wide and 30 mm long. As of this report, our thinnest usable ribbons are 7 μg/cm², 11.6 μm wide and 55 mm long. Refinements have occurred at every stage of the technique and the attainment of significantly thinner mounted ribbons is probable.

1.2. Carbon stripper foils with an unsupported edge

At first, the task of producing 5–15 μg/cm² carbon foils mounted with a 31 mm long unsupported edge seemed to be one of finding a new method of mounting commercially available foils. We were unable to do so. The key to the problem was to produce carbon foils with more mechanical durability. Vacuum-evaporating carbon onto a betaine monohydrate/sucrose layer as suggested by Maier-Komor [2] resulted in foils with the necessary attribute: low brittleness imparted by the surface roughness of the parting agent [3]. With these foils, the unsupported edge can be obtained using a new surgical blade (Feather Industries Ltd., no. 11). Resistance to curling of the foil at the open edge is obtained by applying the betaine with a wiping motion to be described. Open-edged cooler stripper foils of carbon produced by this method have been used for several years at IUCF.

Recently, the foils have also been produced by vacuum-evaporating carbon onto etched stainless steel, polished with Tergitol 8 (40% solution of Tergitol, Sigma Chemical Co., St. Louis, MO). Carbon evaporations onto etched surfaces of unspecified material(s) were reported by Stoner and Bashkin [4] in 1979. The release agent was Creme-Cote (James Varley and Sons, St. Louis, MO). That work resulted in stripper foils of 15 mm diameter (unsupported area) which exhibited extended lifetimes (2 ×) in ^{32}S beams.

The principal advantage to the use of etched surfaces (in lieu of betaine) is that by controlled etching, the surface roughness is inherently adjustable. Therefore, the increase in energy straggling (experienced with foils which replicate a rough surface) [5–7] may be minimized by texturing the surface only as required. Fortunately, in beam stripping, the incident and reaction angles are almost normal to the foil; so, from ref. [5], energy straggling traceable to the use of betaine (or less lumpy etched surfaces) is thought not to be highly significant. Both the betaine and the etched-steel methods will be described. It is uncertain which will prove to be more useful when the injected beam current is tripled by a new ion source in 1991.

2. Procedure for carbon micro-ribbons

2.1. Wire grill evaporation-condensation mask

Vacuum evaporation-condensation masks consisting of 38 μm diameter parallel wires were reported in 1960 by Weimer et al. [8]. Weimer delineated several characteristics of their use in 1964 [9] and gave further information about them in ref. [10] These masks were produced by winding the wire on a split screw which had nearly 19 threads/mm.

The wire grill masks made at IUCF were wound with the assistance of a computer-controlled wire-winding table. The table, routinely in use for making position-sensitive particle detectors, is capable of laying enough wire per winding operation for up to 15 frames, as shown in fig. 2. Although the table can space the wires with an accuracy of only ±10 μm, the resulting gradient in wire spacing is a benefit for ribbon development.

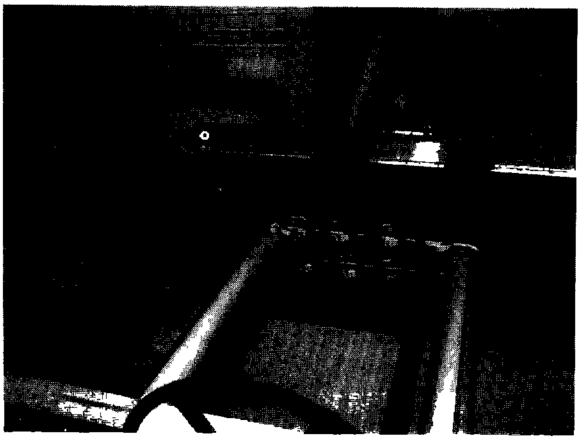

Fig. 2. Preparation of eight wire grills. Photograph showing aluminum ring frames, tungsten wire and a computer-controlled wire-winding table.

Fig 3 Evaporator setup showing substrate in contact with a wire-grill mask, concentric rings on the Al mask frame and a graphite disk in the hearth of the e-gun

A nominal gap of 17 ± 10 μm between each 38 μm diameter wire is presently used for the wire grills. Wire with 20 μm diameter is available which theoretically would produce micro-ribbons with more sharply defined edges and more uniform thickness. However, the closer spacing required with the thinner wire is currently beyond the capabilities of the table. Gold-coated tungsten wire (California Fine Wire Co., Grover City, CA), in use at IUCF for position-sensitive detectors, is currently used for the wire grills. This wire is wound with 100 g of tension force.

Wound wire, 10–15 mm wide (182–273 wires), is glued to aluminum ring frames which have dimensions of 89 mm o.d., 64 mm i.d. and 15 mm thickness (fig. 3). The contact surface of the ring frames is machined with concentric grooves to aid the glue (five-minute epoxy) in anchoring the wires. Positioning the rings is done carefully to ensure that all of the wires stay in one plane during gluing.

When required, more consistently precise wire spacing is achieved by bringing a threaded wire guide into contact with wire which has been wound on the table. A pad of open-cell foam, placed under the wire guide (fig. 4), helps to seat the wire in the threads of the guide. The guide is moved to a new position after an aluminum ring frame is glued to the wire between the screws.

The threads of the wire-guide screws were cut on a machine-shop lathe using the smallest setting of tool feed available on it: 58.4 μm. Burr-free threads on 25 mm diameter rod were easiest to achieve on free-machining brass and more difficult on aluminum or bronze. The cutter was a standard lathe thread-cutting tool, with the following exceptions: (i) the included angle of the cutter bit was 45° and (ii) hand honing and microscopic examination (500 : 1) of the cutter were used to sharpen the tip to a radius of 3 μm. The threads were cut in a single pass to a depth of 50–60 μm.

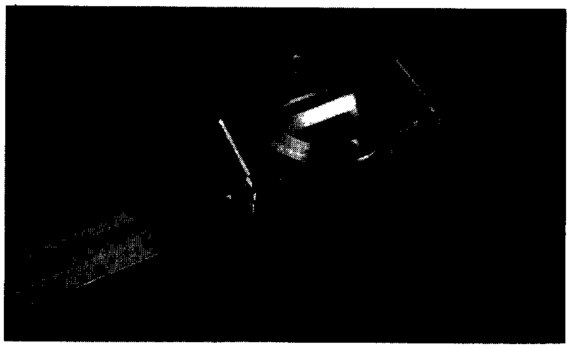

Fig. 4 Wire guide above an open-cell foam pad; wire grill mask positioned between the threaded screws

2.2. Micro-ribbon release agents and substrates

2.2.1. Betaine on glass

The betaine/sucrose is prepared in the same ratio as reported by Maier-Komor [2]: 7/1. The aqueous solution we use is not saturated: 7/1/28.8, i.e. 365 mg of betaine, 52 mg of sucrose, and 1.5 ml of water. This formulation was found easy to use and produced crystallized layers which appear uniform to the eye. The shelf life of the solution is extended greatly by refrigeration.

For betaine micro-ribbons on glass, a 25 mm × 75 mm microscope slide is scored and broken to a length of 60 mm. The slide piece is cleaned with deionized water and wiped dry with a Kimwipe before 15 μl of the betaine solution is placed on the better of the two glass surfaces. The solution is wiped over the surface using a single finger within a tight-fitting latex examination glove. Light pressure and a continuous, small circular motion is used until the solution crystallizes. In most applications, this is done under a heat lamp. When properly prepared, a uniform surface is produced, which is milky in appearance.

If the crystals are scraped from the glass in an area which will lie to the side of the wire mask during the evaporation, the carbon deposited there may be used to obtain a film-thickness measurement by the usual optical transmittance methods. An area free of betaine/sucrose is required, because carbon films deposited on it are much more translucent than those deposited on other commonly used release agents. Although the quartz-crystal monitor (R.D. Mathis TM-100) normally provides a reliable thickness measurement, the optical transmittance of the carbon film (without release agent as described) is used for an independent corroborative measurement.

2.2.2. Detergent on etched stainless steel

Type-304 (18-8) stainless-steel sheet with a bright (2B), scratch-free finish is the material used thus far for etching. It has worked well enough that others have not yet been explored. The sheet (0.5 mm thick) is sheared to a size appropriate for the wire grill mask: ~18 mm × 60 mm. Using a reference surface, the piece is checked carefully for flatness. Cleaning the surface with Alconox, acetone, and water is the only surface preparation necessary before etching.

The etch solution is aqua regia, freshly prepared and diluted with an equal volume of water. The steel is fully

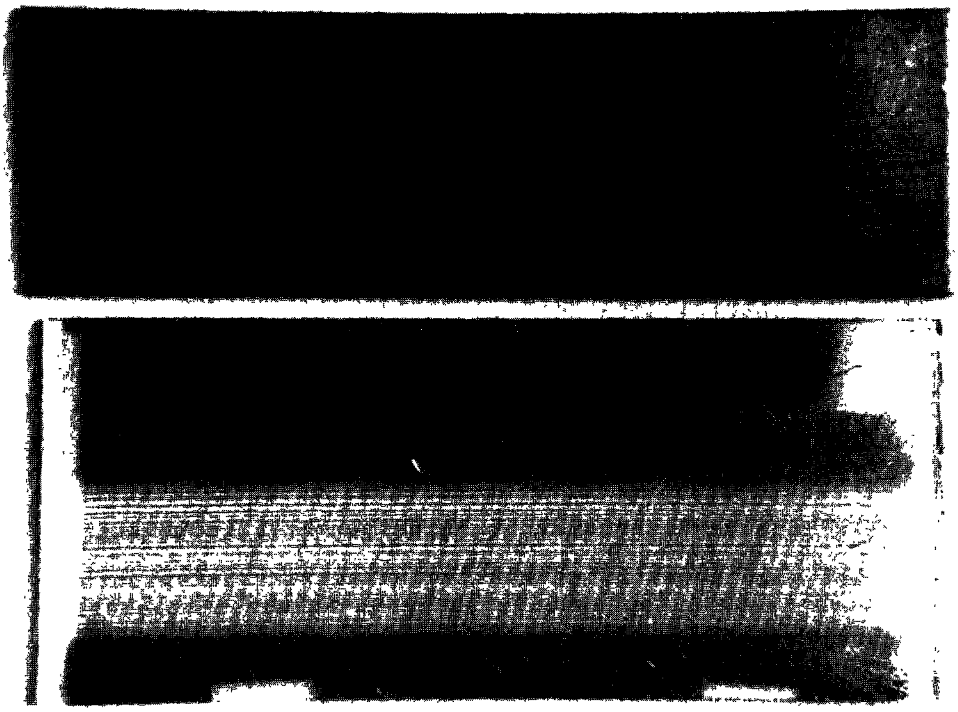

Fig. 5 Top: carbon micro-ribbon deposit: betaine/sucrose on glass. Bottom: carbon hybrid micro-ribbon deposit. Tergitol 8 and betaine/sucrose on etched stainless steel.

immersed for 15 s (typical), followed by a quench in deionized water. At a magnification of 1000 : 1, the most notable change in the steel is that the width of the grain boundaries increase by a factor of 2–3 beyond the original 0.5–1.0 μm.

Thus prepared, the steel substrate is polished with Tergitol 8 (see section 1.2) by pipeting 10 μl onto the surface and wiping it with a Kimwipe until no visible trace remains. One must exercise care not to bend the steel during this process. Tergitol 8 was selected because we wished to evaluate its properties. For this application, it seems to be more than satisfactory.

2.2.3. Detergent with betaine on etched stainless steel

For the hybrid ribbons, the steel substrate is prepared and polished with detergent as in section 2.2.2. Afterward, 3–5 μl of the betaine solution is applied to the steel 5 mm from an end. The solution is crystallized on both ends over the areas about 1 cm from the ends, wiping it beneath a heat lamp as described in section 2.2.1.

2.3. Evaporation

A graphite disk 1.4 mm thick and 9.5 mm in diameter (from Poco Graphite Inc., AXF-5Q rod) is heated in

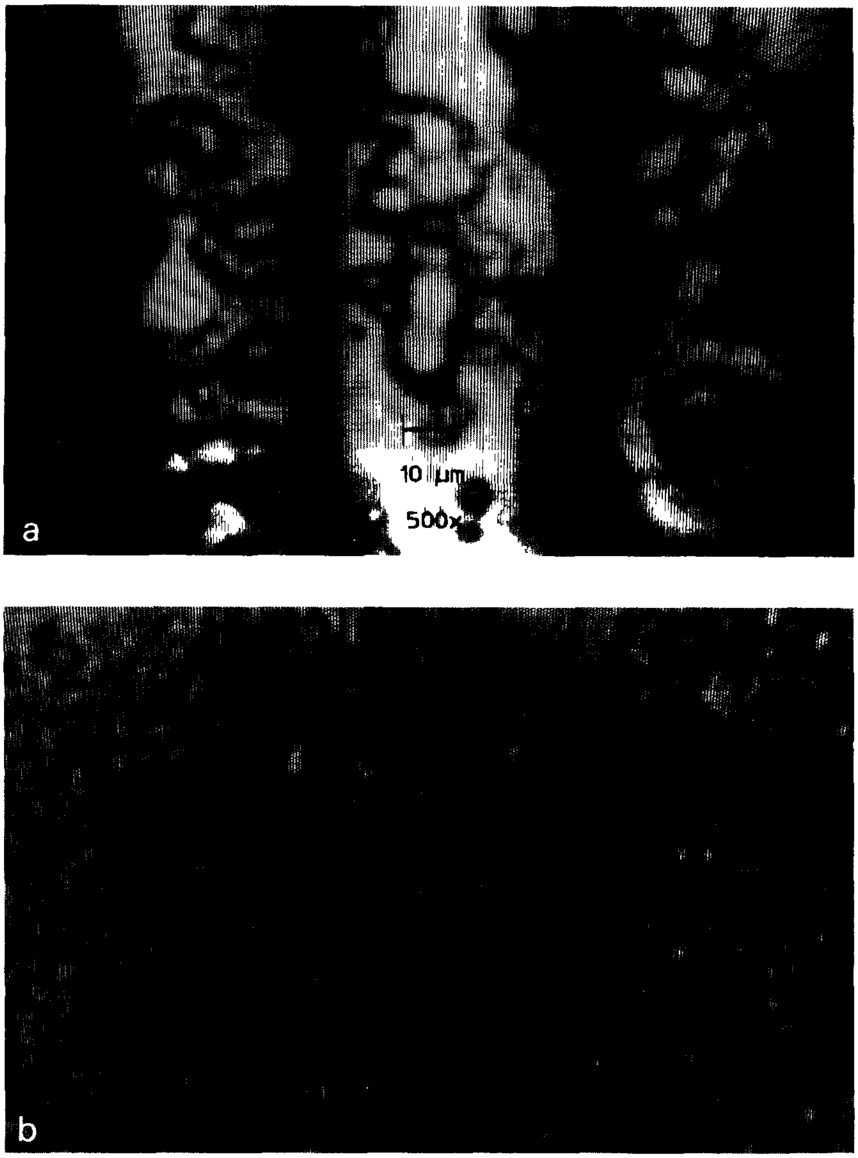

Fig. 6. Photomicrographs. (a) ribbons on betaine/sucrose, (b) ribbons on etched stainless steel

the hearth of a bent-beam electron gun to deposit carbon through the wire grill mask onto the release agent and substrate (steel or glass, fig. 5). The substrate is positioned in direct contact with the wire grill, i.e., supported by it. This is necessary to achieve sharply-defined deposits [9], especially with a roughly textured release agent.

The e-gun has a permanent magnet and is similar in design to one currently marketed by Thermionics Laboratory, Inc. (fig. 3). It is powered by a 0–10 kV dc, 500 mA supply with a 0–6 V ac, 20 A filament transformer. The beam position is adjusted by varying the high voltage setting from 3–4.5 kV.

To shield the release agent on the substrate from the radiated heat of the e-gun filament, the wire mask and substrate are positioned approximately 10° from vertical toward the rear of the e-gun hearth. The head of the water-cooled quartz-crystal thickness monitor is positioned to the side of the mask frame, as close as possible. A thin aluminium sheet is suspended 5 mm below the mask frame to shield all but the area to be coated. No cooling or heat-sinking of the substrate is attempted. The evaporation source to substrate distance is 16–18 cm.

A large stainless-steel shutter is used as a heat barrier to protect the release agent from partial decomposition during the three minutes required to outgas the graphite disk thoroughly and bring it to a temperature of ~ 2150 K. The shutter is then rotated away for 60 ± 10 s. During this minute ($\pm$), the temperature of the graphite disk is increased until an evaporation rate of 0.5 $\mu g\ cm^{-2}\ s^{-1}$ is obtained and the desired amount of carbon is deposited. At least 25 minutes at high vacuum is allowed for cooling before the evaporation chamber is vented to lab air. Figs. 6a and b are photomicrographs of ribbons on betaine and etched stainless steel.

2.4. *Float-off, handling, and mounting*

These three operations are performed under a laminar-flow panel (Liberty Industries model 9FPD) which stands 70 cm above the surface of a lab bench (fig. 7). It is equipped with a variable-speed motor, a pre-filter, and a HEPA 0.3 μm filter. The gentle downward flow of dust-free air, produced by the panel, also facilitates the storage of unmounted individual micro-ribbons for several days. Handling and mounting the ribbons without the panel would be difficult at best.

To float the micro-ribbons, the plane of the substrate is inclined at $\sim 30°$ to the surface of the deionized water. The substrate must also be oriented so that the ribbons are progressively floated together as they are lowered into the water. The betaine ribbons stay together, when floated, due to the links between them (section 1.1). Each must be separated carefully from the neighboring ribbons when pulled from the water. The ribbons having detergent as the release agent are not bound together except by the surface tension of the water. When bunched, the ends of these latter ribbons can be separated to select one for mounting; they separate readily when the tip of a micro-pipette containing a small amount of ethyl alcohol is brought near.

Fig. 7. Laminar flow panel with wooden pick-up sticks held in the exterior folds of the filter. Each holds an unmounted micro-ribbon.

A thin wire (20 μm diameter, 15 mm long) taped to an end of a wooden applicator stick (2 mm diameter, 145 mm long) is used to pull individual ribbons from the water. To accomplish this, 2–3 mm of the wire are submerged near one end of a ribbon. This end is then approached with the wire at $\sim 90°$ to the surface of the water. For betaine ribbons (or ribbon ends) no adhesive is used on the wire. For the others, 2 mm of the wire end are dipped into a thin solution of contact adhesive. After the wire contacts the ribbon, the wire is slowly advanced above and along the length of the ribbon as the wire is withdrawn from the water. As the end of the wire (with the ribbon attached) clears the water, the axis of the wire is nearly vertically aligned. The motion to separate and lift the ribbon is, of course, an upward one which maintains the lifted portion vertically over the separation point in the water or ahead of it. When the trailing end is free, the ribbon is stored (until it is selected for mounting) by pushing the opposite end of the pick-up stick into the exterior folds of the laminar-flow panel filter. With this arrangement, the ribbon is fully extended downward.

A measurement of the ribbon width is obtained at this time by microscopic examination of a short piece. A second pick-up stick is prepared by lightly coating the attached wire with contact adhesive. The piece is acquired by passing the ribbon to the second pick-up stick, making contact with the ribbon at a point ~ 2 mm below the end of the wire on the first pick-up stick. A piece of ribbon sufficiently long will usually remain on the end of the first wire; if not, the procedure is repeated. Once acquired, the piece is transferred to adhesive tape for measurement.

The ribbons have been mounted on a variety of forks and rectangular frames. Although the required width of the frames is 32 mm, ribbon lengths up to 55 mm are used to span the distance when the ribbons are to be rotated through the cooler beam, using a jump-rope motion. A blend of cyanoacrylate instant adhesive (Loctite Super Bonder 495) and powdered graphite is used to secure the ends of the ribbons to frames. It is prepared (immediately before use) by mixing graphite into a small quantity of the adhesive until the liquid mix becomes noticeably more viscous. The graphite serves to polymerize the cyanoacrylate within a few minutes and also to make the blend conductive. Thus prepared, small quantities are transferred with a needle probe to a frame. Immediately thereafter, under the laminar-flow panel, a ribbon on a pick-up stick is brought into contact with the adhesive. To mount a ribbon with a specific amount of slack, the plane of the ladder is inclined at ~ 30° from vertical so that the ribbon will contact the bottom point of adhesion first. The free end of the ribbon is then "flown" into contact with the adhesive there. The desired slack in the ribbon is established by allowing the ribbon to bow downward through the frame (under the influence of the airflow of the panel) before the upper end of the ribbon is glued. An overcoat of the adhesive blend is applied to ensure good mechanical and electrical contact with the frame.

3. Procedure for carbon cooler stripper foils

3.1. Stripper foil substrates and parting agents

3.1.1. Betaine on glass

The glass substrate size is usually 95 mm × 65 mm × 0.5 mm. This is adequate to produce four cooler stripper foils per evaporation from each of two such substrates used. It is also the largest size practical for use with our evaporation geometry and method of applying the release agent.

Because the stripper foils must be resistant to curling at the free edge, the release agent is applied in a slightly different way than is used for the micro-ribbons. For stripper foils, the glass substrate is wiped with a single finger within a loose-fitting pvc glove of 0.1 mm thickness. The desired thickness of crystallized betaine/sucrose (~ 200 μg/cm^2) is essentially the same, but a non-circular, back-and-forth motion is used beneath the heat lamp until the solution crystallizes. The macrostructure which results contains wipe marks of closely-spaced, thin parallel lines. When replicated in the evaporated carbon foil, the wipe marks produce resistance to curling in the unsupported edge which will be perpendicular to them.

3.1.2. Detergent on etched stainless steel

The method described in section 2.2.2 to etch and polish type-304 stainless steel has also been used for cooler stripper foils. For production evaporations of the foils, the size of the steel substrate is 95 mm × 65 mm × 0.5 mm. A 23 mm × 95 mm × 0.5 mm piece has been used to obtain carbon for two foils by locating it to the side of the wire grill mask during evaporations for micro-ribbons.

3.2. Evaporation

The technique employed to evaporate carbon for the cooler stripper foils differs little from the one used for the micro-ribbons (section 2.3). The evaporation geometry is notably different. For the stripper foils, the two substrates must be positioned further to the rear over the e-gun to avoid direct exposure to the radiated heat of the gun filament. The plane of each substrate is positioned normal to a line from the graphite disk to be evaporated; nevertheless, the rough texture of the surfaces results in a wide range of deposition angles. They are also stationed to the left and right (as close as possible) of the head of the water-cooled quartz-crystal thickness monitor. No cooling or heat-sinking of the substrates is attempted.

3.3. Float-off, mounting, and handling

Before floating on deionized water, the evaporated carbon is cut on the substrate with a razor blade into 18 mm × 40 mm pieces. For the betaine foils, the pieces are cut so that the wipe marks described in section 3.1.1 run perpendicularly to the 40 mm long sides. The large size of the glass substrates allows selection of areas which have the most uniform wipe marks. During floating, the carbon separates quickly and uneventfully from the substrate unless the release agent has been overheated.

Individual carbon pieces are slowly lifted from the water onto 0.5 mm thick, "C"-shaped aluminum frames to which a razor blade has been clipped temporarily (fig. 8a). This mounting operation is usually done with the razor blade positioned at the bottom of the assembly, because the survival rate of the thinner foils (5–10 $\mu g/cm^2$) is higher. Side-positioned blade withdrawals result in overly taut mounted foils. There is little mechanical stress in the foils; therefore, the thinner ones can be mounted on a clean non-smooth surface without an adhesive. If required, a thin coating of contact adhesive or five-minute epoxy is sprayed or wiped over the frame (but not the razor blade) before mounting.

Cutting the foil along the razor blade to produce the free edge is not difficult. After the mounted foil is allowed to dry, cutting is accomplished by drawing a surgical blade (Feather no. 11) along the edge of the razor blade. The motion most used is a repeating short stroke with very light pressure against the razor blade. Afterward, the clips are released and the blade is allowed to fall away (figs. 8b and c). Variation from a straight line in the cut edge of the foil is rarely as much as 0.3 mm.

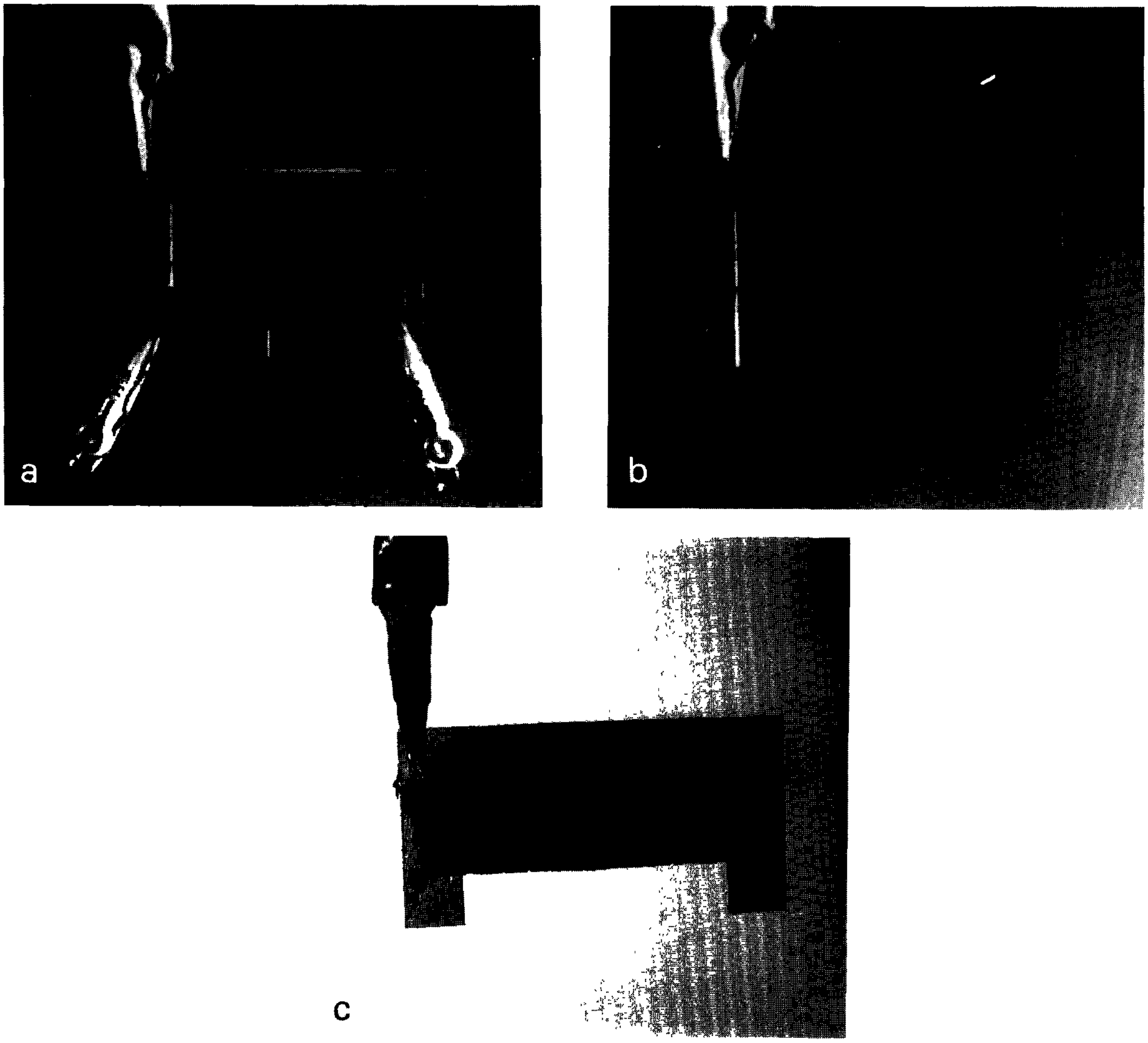

Fig. 8. (a) Al frame supporting a 6 $\mu g/cm^2$ stripper foil (14 mm × 31 mm unsupported area) with a razor blade clipped temporarily to the bottom. (b) After the bottom edge is cut and the clips released. (c) Betaine foil showing wipe marks perpendicular to the open edge

The cooler stripper foils are handled with the usual care given thin carbon foils. In air currents, the common mode of failure for a foil is for it to tear back along the frame at the two ends of the cut edge. However, perfectly still air is not required to install the foils successfully in the beamline.

4. Results and discussion

The ribbons from betaine have survived several trials in which they were heated to incandescence with the lab e-gun and with 20–25 μA of 400 keV H^+ beam. Ribbons of ^{13}C have also been made successfully. However, no ribbon has yet been heated significantly in the cooler beam or in some way shown to be a target of choice.

If it proves possible to use the ribbons as a support for ng/cm^2 coatings of isotopically enriched isotopes, the range of possibilities will be greatly extended for cooler targets. Sufficiently long cooler-beam lifetimes with coated ribbons will require exceptionally thin uncoated ribbons.

Another very promising prospect for solid carbon targets and target supports for cooler rings has recently surfaced. Dr. M. Lake [11] will attempt to grow carbon fibers with diameters of 10–20 nm across 31 mm wide graphite frames for IUCF. Applied Sciences has expertise in growing long carbon fibers from a bed of fluidized catalyst particles within a furnace. These totally invisible fibers are usually simultaneously thickened by CVD to 6–10 μm and then graphitized. Dr. Lake believes his process can also produce fibers of the original thickness across the frames at 200–300 fibers/mm.

No cooler stripper foil has failed in the 90 MeV H^+ beam currents presently available from our ion sources; however, a new source is near the construction stage. Because the new ion source has been designed to increase the available beam current by a factor of 2–3, it may significantly shorten the in-beam lifetime of the foils.

Acknowledgements

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