ACF-METALS PRODUCT DESCRIPTIONS AND TECHNICAL INFORMATION

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Who we are:

For more than thirty-five years, ACF-Metals has been dedicated to providing accurately characterized ultra-thin foils and coatings of metal and carbon in thicknesses less than one micron thick (1000 nm, 0.00004") up to a few microns thick, at competitive prices, to customers around the world. Carbon foils are now provided in any thickness whatsoever. Many other chemical elements are represented in our product offerings. We have made mounted metal foils only 50 nm thick in diameters up to 910 mm, have seen our foils less than 10 nm thick sent with confidence to Halley's comet (and other comets) and to planet Saturn, and have seen our carbon-film attenuators used in thousands of reliable fiber-optic-communication hookups. Sophisticated measuring systems are used to quantify the properties of these foils. ACF-Metals continues to carry out and publish research on the best ways to produce and mount such foils; the results of such research are regularly made available to our customers.

ACF-Metals is your best source when you need many standard foils or coatings for production purposes, specialized foils for research, or only a single unique foil or coating for a completely innovative application.

For more information, please contact us:

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CONTENTS :

Section	Subject	Page	
-	INTRODUCTION	4	
1.	EVAPORATED CARBON FOILS	4	
1.1	Product description	4	
1.2	Specifications		
1.3	Reinforcement		
1.4	Measurements, tolerances and uniformities		
1.5	Stresses, annealing and packaging		
1.6	Shipping		
1.7	Storage		
1.8	Pinholes, gas leakage and mechanical strength	6	
1.9	Mounted carbon foils	6	
1.10	Optical properties	6	
1.11	Mass density and electrical resistivity	6	
1.12	Impurities (due to starting material)	7	
1.13	Impurities (due to parting agent)	7	
1.14	Impurities (others' work)	7	
1.15	Cutting, baking and floating foils - Introduction	7	
1.15.0	Avoiding foil breakage	7	
1.15.1	Thin foils	8	
1.15.2	Intermediate-thickness foils	8	
1.15.3	Thick foils	8	
1.16	Floating foils off of substrates	9	
1.17	Pickup of foils onto frames	10	
1.18	Adhesives and clamping	10	
2.	CARBON STRIPPER FOILS MOUNTED ON FORKS	11	
2.1	Product description	11	
2.2	Foil types and fork styles	11	
3.	POLYCRYSTALLINE GRAPHITE (PCG) FOILS	11	
3.1	Product description	11	
3.2	Porosity and microscopic nonuniformity	12	
3.3	Handling	12	
3.4	Baking and electrical resistivity	12	
4.	VTF FOILS	12	
4.1	Product description	12	
5.	ISOTOPIC CARBON FOILS	13	
5.1	Product description	13	
5.2	Strengths, stability and floating	13	
5.3	Pyrolytic foils	13	
6.	ELECTRON MICROSCOPY FOILS AND GRIDS	13	
6.1	Product description	13	
6.2	EM-Ultra-Smooth foils	14	

CONTENTS (CONTINUED):

Section	Subject	Page	
63	Carbon- and metal-covered grids	14	
7	MOUNTED FOILS [•] FILTERS AND TARGETS		
7.1	Product description		
7.2	Transmission calculations		
7.3	Standard backings and sizes		
7.4	Standard foil materials		
7.5	Washers used as frames	15	
7.6	Adhesives	15	
7.7	Pinholes	15	
7.8	Stretched (mirror) foils	15	
7.9	Parameters to consider for a quotation	16	
7.10	Standard tolerances and specifications	16	
7.11	Handling mounted foils	17	
7.12	Typical frame designs	17	
8.	METAL FOILS ON REMOVABLE SUBSTRATES		
8.1	Product description		
8.2	Disclaimer		
8.3	Standard products	17	
8.4	Standard materials	18	
8.5	Techniques for floating and mounting metal foils	18	
9.	OPTICAL ATTENUATING FILTERS	19	
9.1	Product description	19	
9.2	Calculation of attenuations	19	
10.	VACUUM SERVICES	19	
	PRODUCT SUMMARY	21	

INTRODUCTION:

Carbon foils are produced in many forms and by many different methods. Information on carbon foils is widely scattered in the physics, chemistry, medical and engineering literature. The foils made by ACF-Metals are used in nuclear, optical, chemical and microscopic research, and the emphasis in this writeup is on foils used for those applications. A summary of methods used for making such foils is given by J. Stoner and S.Miller in Nucl. Instrum. Meth. Phys. Res. <u>A561</u> (2006) 24-37. ACF-Metals has attempted in this writeup to provide answers to the most common questions by researchers, but we are happy to try to answer questions that are not covered here.

<u>1. EVAPORATED CARBON FOILS</u>

1.1 Product description:

EVAPORATED CARBON FOILS. These are ultra-thin carbon foils having natural isotopic composition, made by evaporation from a vacuum arc onto glass microscope slides or other substrates. They are also known as SEM foils, TEM foils, stripper foils, and carbon slides. The foils are amorphous or nanocrystalline, with the atomic binding primarily sp^2 , i.e., graphitic.





1.2 Specifications:

Customer specifies the areal density (surface density, or mass per unit area) of the desired foils. Alternatively the customer may specify the desired physical thickness(es).

Conventional foil sizes (type ACF) are approximately 25 mm x 70 mm, on 25 mm x 75 mm glass substrates, or approximately 24 mm x 69 mm if free-standing. Prices for these are listed in Price Sheet #1. The catalog number ACF-xxx specifies the areal density xxx in micrograms

per square centimeter (variously written as $\mu g/cm^2$ or $\mu g/sq.cm$ or $\mu g/cm^2$ or $\mu g/cm^2$). Foils in the areal density range 0-180 $\mu g/cm^2$, corresponding to a maximum mechanical thickness of about 1 micrometer (1 micron) are provided on removable substrates; thicker foils are ordinarily provided free-standing, between paper sheets in individual transparent boxes.

To change areal density in $\mu g/cm^2$ to equivalent thickness

in nanometers, assuming a bulk density of 2.00 g/cm³, multiply areal density by 5. To change areal density in μ g/cm² to equivalent thickness in microns (micrometers), multiply areal density by 0.005.

Double-sized foils (type XCF) are approximately 50 mm x 70 mm on 50 mm x 75 mm glass microscope-slide substrates, or approximately 49 mm x 69 mm if free-standing. The catalog number XCF-xxx specifies the areal density xxx in micrograms per square centimeter (μ g/cm² or μ g/sq.cm). Prices for these are listed in Price Sheet #2.

Some other popular sizes and thicknesses are described and their prices given in Price Sheet #3. Still other sizes and thicknesses, foils on special substrates, foils masked to Customer's specifications, and foils overcoated with metals, are available by quotation. Isotopic carbon foils $\binom{12}{C}$ and $\binom{13}{C}$ are also available; see section 5 below, and Price Sheet #6.

Foils on substrates are provided as needed with a parting agent that permits removal by floating. Foils can be cut into pieces while still on their substrates, then floated onto a water surface. Each piece may be picked up onto a frame having an aperture over which the foil is free-

standing, supported only by its perimeter. Instructions and suggestions for floating are included with every order for foils, and are described below (sections 1.16 and 1.17).

Carbon foils can be provided as coatings on substrates, without parting agent, by quotation. Such foils are typically not removable from their substrates.

1.3 Reinforcement:

Foils having areal densities below 5 μ g/cm² are often difficult to mount over apertures larger than a few mm in diameter. Depending upon the application, such foils may be coated with a thin layer of collodion (cellulose nitrate) for additional strength during the mounting process; this layer typically has areal density of 20 μ g/cm²+/- 10 μ g/cm². ACF-Metals does this coating upon request, at extra cost, for foils having carbon areal density in the range range 0-20 μ g/cm². Other users use solutions of ZaponTM or formvar for similar reasons. Collodion may then be removed after mounting the foil, either by gently flowing methanol over the foil [J.L. Gallant, Nucl. Instrum. Meth. 102 (1972) 477-483], or by an oxygen-plasma etch [see J. Stoner, Nucl. Instrum. Meth. Phys. Res. A480 (2002) 171-177], or by the action of an energetic particle beam (as in the terminal of a tandem van de Graaff accelerator). For instance, a 50 nA, 1400 kV 4 He ${}^{+}$ beam focused to 2 mm x 2 mm will remove such a coating in a few seconds (L.C.McIntyre, private communication 2002). Collodion-reinforcement permits the use of carbon foils having nominal areal density of 2 μ g/cm² over apertures of 10 mm diameter, for which mounting without the use of collodion reinforcement is very difficult. Note that collodion should not ordinarily be used on foils that have been annealed, because the coating may seep under the loosened foil, making the foil difficult to remove from its substrate. There is some evidence that a collodion coating on a foil reduces its shelf life to about 3 years (C. Guy, private communication 2003), so collodion-coated foils should not be stored for more than a year or two.

Reinforcement for free-standing evaporated carbon foils can also be provided for foils having areal densities of 90 μ g/cm² and greater. Some users claim that foils treated in this way have extended lifetimes when used in particle beams.

<u>1.4 Measurements, tolerances and uniformities:</u> Every carbon foil provided by ACF-Metals has had its areal density measured, and is labeled individually. The measurements are done optically, near the centers, for foils still on their substrates, and by weighing for freestanding foils. The optical measurement is described in two publications: J. Stoner, J. Appl. Phys.**40** (1969) 707, and Nucl. Instrum. Meth. **A236** (1985) 569-571. Accuracy for the optical measurements is believed to be +/- 10% +/- 0.5 μ g/cm². Weighing measurements have typical uncertainties below +/-5%.

Foils having areal densities below 100 μ g/cm² are ordinarily provided to customer's specifications within +/-10% +/- 0.5 μ g/cm². Thicker foils are ordinarily provided to customer's specifications within +/- 15% or better. Surcharges are added to the prices of foils if tighter specifications are required: For specification to +/-10%, multiply price by 1.50. For specification to +/-5%, multiply price by 3.00. Tighter specifications can be provided at increased prices.

Nonuniformity of areal density across a foil can be measured optically for sufficiently thin foils, and is found always to be better than +/- 10% and typically better than +/- 5% of the central areal density. Foils that can be measured optically can be selected for uniformity of +/-5% at twice the listed price. Nonuniformity of thickness cannot be measured nondestructively for foils thick enough to be opaque, but since these foils are made in the same way as thinner foils, it is believed that their uniformities are similar.

1.5 Stresses, annealing, and packaging:

Stresses usually appear in freshly deposited carbon foils, causing them to curl somewhat when removed from their substrates.

For foils below 50 μ g/cm², curling is usually slight and treatment is usually unnecessary. In the range 50-100

 μ g/cm² stresses become worse with increasing thickness, and the user must determine for his/her application whether treatment by annealing is necessary. Annealing can be carried out by the customer or by ACF-Metals. A typical procedure is described below. Carbon foils nominally 25 mm x 70 mm in the areal density range 0-

100 μ g/cm² are packaged and shipped in opaque rigid plastic boxes, up to 25 foils per box. Alternatively, these foils can be packaged in individual plastic tubes or plastic boxes at additional cost. Foils 50 mm x 70 mm in the

areal density range 0-100 μ g/cm² are packaged and shipped in transparent plastic vials, two foils per vial.

Evaporated carbon foils having areal densities in the

approximate range 80-180 μ g/cm² are annealed (baked) by ACF-Metals before shipment to reduce their stresses. These foils tend to be somewhat loose on their substrates. As a result, they cannot be cut on their substrates without risk of damage, so it is best to cut them to size while they are floating on a water surface (see below). They are often packaged with their substrates glued into immersible non-removable transparent plastic boxes for safety in shipment. Such a foil is removed by the customer by floating as usual, which involves submerging both the substrate and box in water.

Foils having areal densities greater than about 180

 μ g/cm² are annealed and then are completely removed from their substrates by ACF-Metals, and are packaged individually between paper sheets in transparent plastic boxes.

1.6 Shipping:

Foils are guaranteed to reach the Customer without breakage. In cases for which foils appear to be particularly fragile (which can include both mounted foils and annealed foils on glass slides) or for large quantities of foils of any type, ACF-Metals ships the foils in special packaging consisting of a steel box surrounded by soft padding, within a cardboard carton. A charge is made for

this special packaging.

Domestic shipment is made via United Parcel Service (UPS) Second Day Air, or other mode or common carrier specified by the Customer; however U.S. Postal Service is not used.

1.7 Storage:

Foils on substrates are often observed to develop slight crinkling due to humidification of the interface between foil and substrate; this causes a "matte" appearance, is normal and does not ordinarily cause any problems. At room temperature, uncoated foils on slides have no known limit on shelf life. Some customers claim that older foils float more reliably than recently-produced foils, but no systematic studies have been done by ACF-Metals. ACF-Metals stores foils on their substrates in ordinary laboratory atmosphere in Tucson, Arizona, U.S.A. Foils mounted on frames are fragile and are easily broken by shock, vibration, air currents, contact with liquid, or other insult. The shelf life of undisturbed mounted foils appears to be limited only by the stability of any adhesive used. Carbon foils on substrates, freestanding in boxes, and on frames have been stored more than ten years without apparent deterioration.

Occasionally, a customer reports that foils fail to float off. If this happens to you, please contact ACF-Metals at once. In some cases this problem can be attributed to overheating of foils during shipping or storage.

<u>1.8 Pinholes, gas leakage, and mechanical strength:</u> Even with the greatest of care in production, carbon foils have some pinholes and other defects. Very thin foils may stick at tiny adhesions, and pull holes in themselves when removed from their substrates; thus it is difficult to obtain an entire foil without such holes, for foils thinner than about 5 μ g/cm². Evaporated carbon foils are somewhat porous and do not present a perfectly gas-tight barrier. They have, however, been used successfully as windows for low-pressure gas-cell targets [R. Golser *et al.*, Nucl. Instrum. Meth. Phys. Res. **A282** (1989) 185-187; D.C. Weisser, Australian National University, private communication (2005)]. For insight into the difficulties of making a gas-tight foil, see R. Mutikainen, Thin Solid Films **239** (1994) 259-267.

The breaking strength of arc-evaporated carbon foils that are used to support a pressure difference is similar to that of graphite [see T. Call, *et al.*, Nucl. Instrum. Meth. **167** (1979) 33-34].

1.9 Mounted carbon foils:

Carbon (and metal) foils can be provided on washers (section 6.5 below), stripper forks (section 2 below), TEM grids (section 5.3 below), and customer's supports and other mountings (section 6 below), ready for use (





1.10 Optical properties:

Carbon films are used in the extreme ultraviolet, as spectral filters and as supports for metal films used as spectral filters. In addition, they are used in the infrared to attenuate fiber-optic signals. A brief summary of optical properties of evaporated carbon films has been tabulated by W.A. Alterovitz *et al.*, in <u>Handbook of Optical Constants of Solids II</u>, ed. by E.D. Palik, Academic Press, Boston (1991), pps 837 ff.; further extensive information is scattered widely in the scientific literature.

1.11 Mass density and electrical resistivity:

The mass density (mass/volume) of ACF-Metals' standard arc-evaporated carbon foils has been checked by buoyancy, yielding (at $20 \ \mu g/cm^2$): 2.01 +/- 0.02 g/cm³, whereas the bulk density of thick evaporated carbon layers is 1.83 +/- 0.03 g/cm³. Many other investigators have measured densities of carbon foils; results depend upon the method of production. For instance, density of foils at 0.5 μ g/cm² is 1.8g/cm³ [Ritzau & Baragiola, Phys. Rev. **B58**, 2529-2538 (1998)]. Further information concerning the densities of carbon foils can be found in J. Stoner, Nucl. Instrum. Meth. **A303**, 94-98 (1991).

The dc electrical resistivity of freshly arc-evaporated carbon was measured to be in the range 1-2 ohm-cm and approximately independent of areal density for foils in the range 2-100 μ g/cm² [see J. Stoner and S. Bashkin, Proc. 5th Int'l. Conf. INTDS, Oct. 1976]. It should be noted, however, that the resistivity has been found by other investigators to depend on thickness, to rise steeply as foils become thin enough for surface scattering to be significant, and to diminish greatly under irradiation by fast ions.

<u>1.12 Impurities (due to starting material):</u>

The manufacturer provides spectrographic analyses for the spectrographic carbon used as the raw material for evaporated carbon foils. This material has an impurity level of 5 ppm ash maximum. Typically the analyses indicate metallic impurities at the level of 0.5 ppm by weight for Fe and Mg, 0.2 ppm by weight for Al and Si, and not detectable for B, Ca, Cr, Cu, Pb, Mn, Mo, Ni, Ag, Sn, Ti, W, V, Zn and Zr, searching for the "ultimate rays" using a d.c. arc cathode-layer technique, using an arc at 15 amperes for 30 seconds.

1.13 Impurities (due to parting agent):

Parting agents used by ACF-Metals are proprietary, applied by hand, then polished with a clean cloth until only a thin, almost invisible layer remains. Different parting agents are used depending on the type and thickness of foil. The following nonvolatile ingredients are typical: Lycine, lycine hydrochloride, sodium carbonate, sodium dodecylbenzene sulfonate, sodium lauryl sulfate, sodium hydroxide, tetrasodium pyrophosphate, sucrose.

Sodium is the principal metallic impurity seen in our carbon foils, at the level of 1000 ppm for a foil having carbon surface density of 20 μ g/cm². Thicker foils show a smaller fraction, and thinner foils show a larger fraction for this component, up to a few atomic percent; we presume that the sodium originates in the parting agent and probably is found only on the back (substrate) surface of the foil. Oxygen is present at typically 1 atomic percent, and hydrogen is present at typically 3-5 atomic percent of the foil. PIXE and RBS measurements show no impurities heavier than sodium, with detection limits at typically 100 ppm or better.

Foils can be baked in vacuum to reduce their impurities. Some of our users report baking foils to higher than 3000K. Foils tend to graphitize at elevated temperatures, typically 1300-2400 K, changing their properties, and graphitization may damage foils. Graphitization of foils has been described by D. Fishback, in <u>Chemistry and</u> <u>Physics of Carbon</u>, P.L. Walker, Jr., ed., vol. 7 (1971) 1-105 ; see also G. Dollinger & P. Maier-Komor, Nucl. Instrum. Meth. Phys. Res. **A282** (1989) 223-235.

Foils with fewer impurities can be provided on mica, without parting agent (see Electron Microscopy foils, section 6 below). However, carbon foils come in contact with water as they are removed from their substrates, so hydrogen and oxygen may be present from this source. The minimum scattering areal density for evaporated carbon foils from any source is ordinarily about 1.5 μ g/cm² [see Ritzau & Baragiola, *op.cit.*]. However,

mounted carbon foils can be made thinner than this limit starting with conventional foils and thinning them using an oxygen plasma etch, down to about $0.8 \text{ }\mu\text{g/cm}^2$.

1.14 Impurities (others' work):

Extensive study of the impurities in carbon foils used as specimen substrates was done during the systematic development of PIXE (proton induced x-ray excitation) analysis in the 1970's. The impurities arise principally from the starting material for evaporation, from the crucible material used (if any) and from the water used to float the foils. The latter source of impurities can be reduced by using certain acidic solutions for this purpose [see Weathers *et al.*, Nucl. Instrum. Meth. Phys. Res. **A303** (1991) 69-78]. Some other references on impurities in carbon foils are as follows: Balzer and Bonani, Nucl. Instrum. Meth. **167**, (1979) 129-133. Raith *et al.*, Nucl. Instrum. Meth. **142** (1977) 39-44. Campbell, Nucl. Instrum. Meth. **142** (1977) 263-273.

Campbell, Nucl. Instrum. Meth. 142 (1977) 263-273.
Herman *et al.*, Nucl. Instrum. Meth. 109 (1973) 429-437.
Kaji <u>et al</u>, Nucl. Instrum. Meth. 142 (1977) 21-26.
Sofield <u>et al</u>, Nucl. Instrum. Meth. 203 (1982) 509-514.
Johansson *et al.*, Nucl. Instrum. Meth. 84 (1970) 141-143.

J. Stoner, J. Appl. Phys. **40** (1969) 707-709. Both, <u>et al.</u>, Rev. Sci. Instrum. **58** (1987) 424-427. Johansson and Johansson, Nucl. Instrum. Meth. **B3** (1984) 154-157.

1.15 <u>Cutting, baking and floating foils - Introduction:</u> THESE TECHNIQUES DEPEND ON THE AREAL DENSITY OF THE FOIL. PLEASE USE A TECHNIQUE APPROPRIATE TO YOUR FOIL! ___

<u>1.15.0 Avoiding foil breakage caused by mistakes in handling of foils:</u>

Methods of avoiding breakage have been summarized recently: See C. Jolivet *et al.*, Workshop on Targets and Target Chemistry WTTC-11, University of Cambridge, August 28-31, 2006; C. Jolivet & J. Stoner, Proc. 23rd International Conference of the International Nuclear Target Development Society, Tsukuba, Japan, Oct. 16-20, 2006. Some of the most common causes of foils' breaking prematurely are: Poor cutting, causing cracks at foil's edges, that later propagate through the foil. Incautious floating: e.g., irregular rate, or too fast. Careless pickup: poor support of frame, strong air currents, dirty or rough frame, foil damaged in pickup, mounting tightly so that differing thermal expansion coefficients cause foils to bulge or split, failure to allow for foil's shrinkage or bulging during use. Careless use:

incautious pumpdown or venting to atmosphere, sudden application of strong beam, allowing beam to hit frame, sharp beam focus causing foil overheating or concentrated local nuclear damage.

<u>1.15.1</u> THIN FOILS (areal density $< 100 \ \mu g/cm^2$): Write in your notes the number on the tag on the back of the slide; this is the areal density of the carbon layer in micrograms per square centimeter.

Experience is the best guide to estimating the largest aperture that you can cover with a carbon foil. One rough guideline is: 1 mm diameter for each microgram per square centimeter. Experts can often do somewhat better.

Carbon foils can be mounted with one or more free (unsupported) edges, or with holes within the mounted area, using specialized techniques not described here. Examples of such foils are described in section 2 on stripper foils, below.

If the foil is tight on its substrate, scrape around the edges of the substrate with a razor blade to separate the main part of the foil from any residual carbon that drapes over the edge. Use a straightedge (but do not touch the foil with it) and a razor blade or other sharp tool to cut the foil into pieces of the desired sizes (see Fig. 4a below). If you cut off about one millimeter's width of the foil all around its perimeter, the remainder of the foil often floats more uniformly. Gently blow off any loose fragments. Some people like to exhale onto the foil to humidify it just before floating it. Particularly for foils on mica, this appears to make releasing much more reliable (D. Bear, private communication, 1994).

If the foil has started to release from its substrate, do **not** cut it on its slide; the foil may splinter where it is cut. Use instead the method described in section 1.15.2 below.

Foils in the areal density range 60-99 μ g/cm² may need to be baked before use, in order to reduce their internal stresses. Otherwise they may crack, or curl up and sink when floated. [All foils supplied by ACF-Metals after January 1, 2003, have been annealed if their areal densities are greater than 99 μ g/cm².] To bake, use a vacuum oven, or preheat an oven flushed with dry nitrogen to at least 200 degrees Celsius [We use 215-230 degrees Celsius.]. Then lay the slide horizontally, gently, foil-side down, supported by its ends, in the oven for 30 minutes, and let it cool before removing it. [A foil typically loses 12% +/- 6% of its mass in this process. If baking is done in air, the foil may get much thinner or disappear during baking, due to oxidation.] Float it and pick it up in the usual manner (see below).

1.15.2 INTERMEDIATE-THICKNESS FOILS (areal

densities in the range 100-179 μ g/cm²): These foils are usually still on their glass substrates, but they have been annealed, so have loosened somewhat. (Only if Customer so requests, will foils in this areal-density range be supplied without annealing.) If a foil's substrate has been glued permanently into a plastic box for the purposes of storage and shipping, and/or, if the foil has begun to separate from its substrate, the foil should not be cut on its slide. Cutting it may cause it to splinter. Write in your notes the number on the tag on the back of the slide; this is the areal density of the carbon layer in micrograms per square centimeter. Scrape around the edges of the substrate with a razor blade. Float such a foil onto a water surface, using one of the techniques described below, which usually requires immersing the part of the box to which the foil's substrate is glued. After the foil is floating, cut the foil if necessary on the water surface, using clean sharp scissors, keeping the foil horizontal. Avoid bringing the tips of the scissors together within the foil.



<u>1.15.3</u> THICK FOILS (areal densities of 180 μ g/cm² and greater):

These foils are provided already annealed and released from their substrates, so baking is not necessary. Foils are packed individually between paper sheets in plastic boxes. A foil that is individually packaged between paper sheets in a plastic box is ready to be used. [By

special order, foils having areal densities down to 100 μ g/cm² can be so packaged.] A foil's areal density is marked on the outside of its box. The foil has been annealed to reduce its internal stresses so that it typically shows only slight tendency to curl when removed from its box. A curled foil can usually be flattened onto a piece of paper using two lengths of clean, smooth, straight bare copper wire to hold it down. Such a foil can be cut to size by laying it on a clean, dry glass plate and laying a clean straightedge gently on top of the foil. Using a single-edge razor blade, make several gentle cuts along that edge until the foil is cut through. Alternatively, two razor blades may be used, one to hold the foil against the glass plate, the other to do the cutting (Fig. 4b). A single heavy cut or a dull blade may cause the foil to splinter. Pieces can be handled gently with tweezers, and glued or clamped to frames.



1.16 Floating foils off of substrates:

ACF-Metals uses clean (distilled or deionized) water at room temperature. We know other users that use hot water, or add ethanol or other materials to reduce surface tension, but we do not use these materials. Foils will pick up impurities from the water used to float them [see Raith *et al.*, Nucl. Instrum. Meth. **142** (1977) 39-44], and other references in section 1.14 above.

Four methods are popularly used:

(1) The "droplet" method is carried out by laying the foil on the horizontal bottom of a shallow dish, then using a dropper to put tiny drops of water on the edges and/or cut lines on the foils (Fig. 5). After a few minutes, the water will have crept under the foil and more water may be added. When the foil has released completely, water may be siphoned into the dish to lift the foil as needed, or the foil may be transferred to a large dish of water for the picking-up process. This method has the widest applicability of all of the floating methods, but it may cause cracks in thin foils, as it subjects foils to unpredictable tensile stresses.



(2) Use a mechanical arm, forceps, hemostat, or your own steady hand. Hold the foil on its substrate, foil side uppermost, at about 45 degrees to the horizontal. Lower the foil on its substrate slowly into a dish of water.

(3) Alternatively, the slide may be held fixed and the dish of water raised below it using a laboratory jack.

(4) The smoothest method, preferred for particularly fragile or large foils, is to keep both substrate and dish fixed, and siphon water from an auxiliary container into the dish. Method #4 is shown pictorially in J. Stoner and S. Bashkin, Applied Optics **17**, (1978) 321 ff. If the auxiliary container has smaller volume than the dish, it will not overfill the dish if your attention becomes diverted. If the foil's substrate is glued into a box, the foil + box will be immersed in the floating process, and it is preferable to have the water approach the long side of the foil.

Methods #2.3.4 require the foil to flex, but ideally only in one direction. When a suitable rate of release is found, it is desirable to maintain the release rate as constant as possible until the foil has been completely released. In every case, a light-colored background helps to make the foil clearly visible.

When the water level reaches a foil being floated by immersion, it will begin to float on the water surface.

Foils are **guaranteed** to float off of their substrates with one of these techniques (Fig. 6). If the foil refuses to float off, or is pulled below the water surface, either you are doing it too quickly, or the foil is defective. When the foil has floated entirely off of its slide, remove the slide carefully or drop it onto the bottom of the dish, so it won't be in your way for the pickup process. Remove unwanted fragments of the foil from the water surface by picking them up with the corner of a paper towel, or by sucking them to flow into a small upright empty beaker that you first immerse gently in the water almost to its rim. This will prevent such fragments from sticking to the back of the foil when you pick it up.



Sometimes foil pieces tend to stick together while floating. They can be separated conveniently by touching a boundary between foil pieces with a needle or dropper tip moistened in methanol (W.Lozowski, private communication, 1990), or ethanol or isopropanol.

1.17 Pickup of foils onto frames:

Frames on which foils are to be picked up should be planar, stiff, thin, clean, hydrophilic and without burrs. It is convenient to have at least one straight edge on a frame, over which to drape one corner or edge of the foil. The best thickness for frames is perhaps 0.2 mm. Thinner frames tend to flex unpredictably. Thicker frames can be used but the fillet of water that develops at the perimeter of the hole puts extra stress on foils. We use copper, graphite, aluminum, stainless steel or brass frames. Metals are either lightly sanded under water, or acid-etched, to produce a fresh hydrophilic surface, and then kept under water until they are used. Hold the frame with tweezers or forceps under water at an angle of about 60-90 degrees to the water's surface, and lift the frame below a piece of floating foil so that a corner or edge of the foil drapes over an edge of the frame. Continue



lifting until the foil drapes itself over the hole to be covered (Fig. 7). Covering a large aperture usually requires the aperture to be perpendicular to the water surface. If no frame edge is accessible, a floating piece of foil can be trapped between the frame and a side of the dish, and picked up without draping it over an edge of the frame. Without touching the foil itself, gently blot off any excess water from the frame. When foils are dry, they are ready to use. Although they are fragile, and easily broken by vibration or strong air currents, thin carbon foils can be stored for years in a protected environment.

The above floating and pickup methods are modified and extended for metal foils (see section 8.5 below).

1.18 Adhesives and clamping:

Carbon foils that have areal densities greater than about $20 \ \mu g/cm^2$ often need to be adhered to their frames with an adhesive. We have used epoxies, cyanoacrylate cement, Duco cement, sodium silicate solution, Sauereisen cements, 15% flexible collodion in amyl acetate, and graphite paint. Each adhesive has its own area of utility [See C. Jolivet & J. Stoner, Proc. 23rd World Conf. Intl. Nucl. Target Devel. Soc., Tsukuba, Japan, Oct. 16-20, 2006.]. Frames can also be coated with a thin layer of Apiezon L or Dow Corning high vacuum grease prior to picking up the foil; the grease allows the foil's strains to relax as it is bombarded [J. Greene, private communication, 2003; see also J. Stoner & S. Miller, op. cit. (2006).] [Grease will not prevent the foil from curling.] Let us know what works for you. ACF-Metals is always glad to receive reprints of work for which our foils were useful.

In circumstances for which no adhesive can be used, a frame may consist of a base part on which the foil is laid, and a clamp part, screwed or otherwise fastened to the base part, that prevents the foil from falling off.

2. CARBON STRIPPER FOILS MOUNTED ON FORKS

2.1 Product description:

Accelerators often use stripper foils to remove electrons from ions during or after the acceleration process. Carbon is usually the element of choice because of its strength and its low Z (atomic number Z = 6). For these purposes, foils may be mounted on special fork-shaped or L-shaped frames so that the foil has one or more free edges; in this way a beam of negative ions may be steered through the foil without the ions' passing through a massive frame (Fig. 8).



The foil is often rectangular and is typically mounted so that one edge is unsupported. In special cases, foils can be mounted with two, three or four unsupported edges using ceramic or carbon fibers to support the foils. [See Borden *et al.*, Nucl. Instrum. Meth. Phys. Res. <u>A303</u> (1991) 63-68; Sugai et al., Nucl. Instrum. Meth. Phys. Res. <u>A561</u> (2006) 16-23; and papers by Spickermann *et al.*, Plum & Shaw, Jolivet *et al.*, Sugai *et al.* in Proc. 23rd Conf. Intl. Nucl. Target Devel. Soc., Tsukuba, Japan, Oct. 16-20, 2006.] ACF-Metals is pleased to quote on producing foils on other mountings suggested by Customer.

2.2 Foil types and fork styles:

The foil's areal density is determined by the application and is specified by the customer. For negative-particle cyclotrons, typical values range from 50 μ g/cm² to 5000 μ g/cm². The type of carbon used is typically evaporated (type ACF) up to 400 μ g/cm², either polycrystalline graphite (PCG, see following pages) or evaporated carbon up to 1000 μ g/cm², and PCG above 1000 μ g/cm². A low-Z reinforcement coating that adds about 20 μ g/cm² can be provided on these mounted foils at additional cost. A graphite-paint "shorting bar" may be provided for positive electrical connection of foil and fork at additional cost. An adhesive is ordinarily used to connect foil to frame. Depending on the application, this is a graphite paint, or a high-temperature medical-grade epoxy, or a radiation-resistant epoxy [see Jolivet *et al.,op.cit.*(2006)].

Prices and further descriptions of stripper foils on forks are presented in Price Sheet #4.

A few standard types of forks are kept in stock by ACF-Metals. Foils can be provided either on ACF-Metals' type ACF-F30, mod.2, aluminum forks (Mallinckrodttype), or on ACF-Metals' type ACF-10 forks (Syncortype). A sample of either type, and/or a specification sheet showing dimensions for either type are available upon request. ACF-Metals can also quote on mounting foils on specialized frames designed by Customer.

Carbon foils are believed to develop microcracks at edges that are formed by cutting the foils (S. Hitchcock, private communication 2002). ACF-Metals recommends mounting foils having free edges that have been formed by masking during evaporation.

3. POLYCRYSTALLINE GRAPHITE FOILS (PCG FOILS)

3.1 Product description:

Thin polycrystalline graphite (PCG) free-standing foils are made by proprietary methods that yield foils composed of graphite microcrystals, having natural isotopic composition and grain sizes of the order of 1 micrometer, aligned primarily (but not entirely) with their c-axes parallel to the foil's normal. They have several advantages over standard (arc-evaporated) carbon foils: PCG foils can be made with greater areal densities, they are easier to handle, and in areal densities greater than 800

 μ g /cm² they are less expensive than evaporated foils. Such foils have mass densities in the range 0.8-1.2 g/cc, and are porous. They are not expected to be gas-tight. They are typically used as extractor (stripper) foils in accelerators, for which they are as efficient as amorphous carbon foils of the same areal density [D. Varney, private communication, 1995], but for such uses they should be conditioned by gradually turning on the beam [Steve

Binder, private communication, 1996]. They are also used as windows between regions of different vacuums. These foils have been used successfully to transmit synchrotron radiation at power densities up to 500 watts per square centimeter. PCG foils are relatively soft and flexible, and contain volatile impurities (principally water) that are easily removed by baking (see below).



Stock PCG foils are provided in pieces that have nominal dimensions 25 mm x 60 mm, shipped in individual transparent plastic boxes (Fig. 9). Larger pieces (to 300 mm square) and smaller pieces can be provided by quotation. Prices for the most popular forms are given in ACF-Metals' Price Sheet #5. Areal densities are provided within 15% of customer's specification unless otherwise specified. Impurities include approximately 1-3% oxygen; no other impurities are seen at the level of 0.1% by Rutherford backscattering analysis (RBS), or by energy-dispersive x-ray spectroscopy; hydrogen may be present but cannot be detected by either method. The catalog number PCG-xxx specifies the areal density xxx in micrograms per square centimeter.

3.2 Porosity and microscopic nonuniformity:

Because of their porosity on a scale of 1-2 micrometers, PCG foils show a microscopic nonuniform thickness to particle beams. This nonuniformity is approximately +/-40% at areal density 1 mg/cm², diminishing to about +/-20% at 7 mg/cm² [Y. Takabayashi, University of Tokyo, private communication (2000)]. Extrapolation suggests that microscopic nonuniformity of PCG foils would approach +/- 100% near 0.3 mg/cm², consistent with the grain size of this material. Note that in comparing PCG foils with standard carbon foils of the same areal density, the geometrical thickness of a PCG foil will be about twice the thickness of a standard carbon foil.

3.3 Handling:

A PCG foil can be cut easily by placing it between sheets of paper (weighing paper is good) and using **sharp** scissors. As an alternative, a foil can be trimmed between weighing-paper sheets with a **sharp** razor blade and straightedge, with the foil lying on a hard surface. Small pieces can be handled by tweezers; large pieces can be handled with forceps, using a spatula or paper sheet to provide additional support if necessary. If a foil tends to curl, it can be flattened by the same process used to remove water and other volatile impurities: baking at 200 degrees Celsius. The foil is placed between clean sheets of glass spaced apart by perhaps 0.1 mm, and the combination placed in an oven. It is preferable to flush the oven with nitrogen to avoid the risk of oxidizing the foil. Baking for ten minutes is usually sufficient to flatten the foil; we often use a 30-minute cycle if several foils are to be baked at the same time. This treatment renders the foil more brittle than an unbaked foil.

3.4 Baking and electrical resistivity:

A vacuum high-temperature bake can be used to further consolidate the foil, reduce the electrical resistivity, and drive off tightly bonded gases. A mass loss of typically 25% occurs for foils so treated at temperatures in the range 500- 2000 degrees Celsius. Similar experiments on amorphous carbon foils, which tend to graphitize at high temperatures, have yielded similar results, with smaller mass losses.

A few tests of resistivities of PCG foils have been done. Typical values for unbaked foils were 1.6-3.2 ohm-cm; after baking at about 2000 degrees Celsius for a few minutes in vacuum, the resistivity was reduced to about 0.01 ohm-cm, still higher by a factor of 10-100 than the resistivity of bulk graphite. PCG foils have been used as x-ray filters at temperatures up to 1676 degrees Celsius, after vacuum baking at 1000 degrees Celsius (LBL specification #335240).

4. VTF FOILS

<u>4.1 Product description</u>: Thick, free-standing bulkdensity graphite foils in thicknesses of 20 μ m and above can be provided upon special order. These are polycrystalline graphite having a density of



approximately 1.84 grams per cubic centimeter (Fig. 10). The maximum size and minimum order depend upon the thickness. Sizes up to 150 mm x 150 mm are available, depending upon thickness. Prices are given in Price Sheet #6, and by quotation.

5. ISOTOPIC CARBON FOILS:

5.1 Product description: Two types of foils are provided. For areal densities in the range 10-100 μ g/cm², enriched stable isotopes of carbon (¹²C or ¹³C) are electron-beam evaporated onto 25 mm x 75 mm glass slides, producing foils with size approximately 25 mm x 70 mm. Areal-density calibration of foils is via quartzcrystal monitor, which typically yields accuracy of +/-10%. Foils are fully annealed and are sold in minimum lots of 2,3,or 4 slides, depending on areal density (see Price Sheet #6). If a parting agent is necessary, barium chloride or sodium chloride is used. Enrichment of the starting material (prior to evaporation) is 99+% for 13 C, 99.9+% for 12 C; however, the customer should be aware that many processes, including but not limited to absorption of atmospheric carbon dioxide from the atmosphere, can dilute the enrichment of either isotope during or after evaporation.



For applications in which possible impurity of barium, sodium and/or chlorine must be minimized, foils can be produced on cleaved-mica substrates. However, these foils may have cracks or other defects due to the layered structure of the substrate, and it is correspondingly more difficult to obtain pieces with mechanical integrity sufficient to cover large apertures.

5.2 Strengths, stability and floating:

Customer should be aware that carbon foils produced by electron-beam evaporation (which includes isotopic carbon

foils in the range 10-150 μ g/cm²) are not as strong as foils made by arc evaporation, and it is correspondingly more difficult to float and mount them. These foils are fully annealed; as a result, they are usually already partially released from their substrates. Foils have been stored satisfactorily for periods of greater than one year in laboratory atmosphere before being floated off. Floating of these partially-released foils is best done by the droplet-floating technique (see above), starting at one end of the foil after the long edges of the foil have been scraped, and after about 1 mm of the foil has been cut off using the double-blade technique (see above). Cutting of the foil into pieces for mounting is best done with sharp scissors after the foil is floating on a water surface.

5.3 Pyrolytic foils: For areal densities in the

approximate range 1000-1200 μ g/cm², foils are produced by thermally cracking hydrocarbon gases. The production method is described by J. Stoner and S. Miller, *op. cit.*. The same enrichments are available as for thinner foils; however, ACF-Metals provides these foils already mounted on Customer's frames. Currently a maximum foil size of about 15 mm x 15 mm is available.



6. ELECTRON MICROSCOPY FOILS AND GRIDS:

6.1 Product description:

The use of a transmission electron microscope usually requires specimens that are transparent to the electron beam.

Frail specimens are often supported on very thin carbon foils (usually called "films" by the microscopist), in turn mounted on support grids. Carbon is used because of the foils' relatively high strength, inertness and minimal observable structure in the microscope. The thinnest and purest carbon foils are made by electron-beam evaporation onto freshly-cleaved mica plates (Fig. 13). These are ACF-Metals' EM-Ultra-Smooth foils. The foils are then floated off and picked up onto conventional TEM grids, typically 3 mm in diameter and having a mesh structure to support the foil (Fig. 14). For applications that do not require the highest purity and resolution, arc-evaporated carbon (see previous pages) can be used at lower cost; however, particulates from the carbon arc may be present in such foils, and the parting agent adds to the uncertainties in the structure and in the purity of the foils.

6.2 EM -Ultra-Smooth Foils:

These are electron-beam evaporated carbon foils on freshly water-cleaved mica substrates. No contaminations appear due to parting agent, because no parting agent is used. The foil has nominal dimensions approximately 25 mm x 70 mm. Such foils can be provided in nominal thicknesses from 2 nm to 50 nm. The catalog number EMU-xx gives this thickness xx in nanometers (see Price Sheet #7).



6.3 Carbon- and Metal-Covered Grids

These are standard 3-mm-diameter, 200-mesh copper TEM grids with the shiny sides covered with carbon films, or with metal or oxide films specified by Customer. Some materials that have been used in this way are: aluminum, aluminum oxide (alumina), boron, gold, silicon, silicon dioxide (silica), silicon monoxide, silver, titanium and zirconium.



Lot size is 100 grids, coated with a film of carbon (either arc-evaporated or e-beam evaporated), or other material, at Customer-specified thickness, 10 nm or greater. Grids are packaged in conventional grid boxes having recesses to support the grids individually. Prices are by quotation; however for some standard products, prices can be found in Price Sheet #7.

7. MOUNTED FOILS: FILTERS (EUV AND X-RAY) AND TARGETS:

7.1 Product description: These are ultra-thin foils, sometimes isotopic foils, removed from their substrates, and mounted on frames. Frames may be washers, rings, or more-complicated assemblies. Except for the simplest standard assemblies, these products are made to Customer's specifications and are priced by quotation.

Mounted foils are often used as bandpass filters, to transmit certain extreme-ultraviolet (EUV) or soft-x-ray wavelengths while absorbing others, typically in the wavelength range of 1 nm to 100 nm, corresponding approximately to the photon energy range of 1000 eV to 10 eV. Extensive information concerning the x-ray properties of matter can be found at <http://xdb.lbl.gov/> and <http://www-cxro.lbl.gov/optical constants/>. Other applications include: Synchrotron-radiation-beam locators [See C. Jolivet and J. Stoner, AIP Conf. Proceeding 879, "Synchrotron Radiation Instrumentation" (2007) 1077-1080, Daegu, Korea, May 28-June 2, 2007.], selectively transmitting films that protect delicate optics (spectrometers etc.), ion-beam neutralizer foils, ultra thin foils for neutral-particle time-of-flight (TOF) detectors [See D.J. McComas et al., Rev. Sci. Instrum. 75 (2004) 4863-4870.], nuclear targets, collectors for space-debris measurements, and streak-camera photocathodes.

For specialized purposes, some metal foils can be mounted with free (unsupported) edges. If customer has such an application, please inquire for ACF-Metals' views on feasibility.

7.2 Transmission calculations: Transmission curves in the EUV and X-ray regions are calculated by the Customer, typically using tabulations [e.g. B.L. Henke, E.M. Gullikson, and J.C. Davis, Atomic Data and Nuclear Data Tables <u>54</u>, 181-342 (1993)], or using the programs that can be found at

<http://www-cxro.lbl.gov/optical_constants/>. Transmission of the visible and infrared regions is often blocked, at least in part, by the same or by auxiliary layers; the transmission properties of these are often calculated from the data in the volumes: <u>Handbook of</u> <u>Optical Constants of Solids</u>, edited by E.D. Palik, Vol. 1 (1985) and Vol. 2 (1991). 7.3 Standard backings and sizes: Foils can be mounted either without backings (depending on thickness and material) or with mesh or foil backings for additional strength. Standard ring-mountings are stainless-steel washers having inner diameter 11.1 mm or 13.4 mm. Standard stretched foils (see below) are usually mounted on stainless-steel washers having inner diameter of 11.1 mm. Larger mountings, up to 100-mm-diameter or larger in special cases, are done by quotation. Our largest mounted foil to date had an inner diameter of 910 mm. ACF-Metals often mounts foils on Customer's mounting frames; this is done by quotation.

7.4 Standard foil materials: Foils of the materials listed below are mounted in thicknesses typically from 20 nm to 1000 nm, depending upon mechanical strength and mounting method. Typical prices are given in Price Sheet #8. Aluminum Aluminum oxide (alumina) Bismuth (on C) Boron, natural and isotopic Carbon, natural and isotopic Chromium Cobalt Collodion (cellulose nitrate) Copper (on C) Dysprosium (on C) Germanium Gold Indium (on C) Iron Magnesium Manganese Molybdenum Nickel Niobium Silicon Silicon dioxide (silica) Silver Tin Titanium Tungsten (wolfram) Vanadium Yttrium (+Al) Zinc (on C) Zirconium

Other materials, other thicknesses, multilayers, etc., are provided by quotation.

<u>7.5 Washers used as frames:</u> Some standard stainless steel washer washers used as frames for mounted foils are available from ACF-Metals at no additional cost. These

have approximate dimensions as follows:					
Inner diameter	Outer diameter	<u>Thickness</u>			
3.6 mm	15.9 mm	0.8 mm			
4.4	19.0	1.0			
4.9	11.2	1.3			
5.2	17.5	1.3			
7.9	18.6	2.0			
9.5	22.2	2.6			
11.1	25.4	1.9			
11.9	23.4	2.0			
13.5	27.0	2.4			
16.7	33.3	2.4			

0 11

...

Other frames can be provided by ACF-Metals or by the customer at additional cost.

<u>7.6 Adhesives:</u> When an adhesive is used to fasten a mesh and/or foil to a frame, ACF-Metals' usual choice is Vac SealTM, a two-component epoxy having relatively low outgassing rate. Radiation-resistant epoxy, heat-resistant epoxy or graphite paint are used in special cases. [See section 1.18 above.] If an adhesive must be used to fasten a foil to a mesh, the adhesive is usually cellulose nitrate. If such an adhesive is used, it covers only the grid bars, and not the squares of unsupported (free-standing) foil between bars.

<u>7.7 Pinholes:</u> Ultra-thin foils usually have pinholes. Depending upon material, thickness and method of mounting, foils can sometimes be provided such that no holes are visible with examination at 40x magnification. Prior determination by the customer of the tolerable level of pinholes is necessary for the pricing of special-order foils (see below).

ACF-Metals does not warrant its foils to be pinhole-free, nor for gas-tightness nor for light-tightness, nor to support pressure differences, except by written quotation. Some of the difficulties involved in producing such foils are presented by R. Mutikainen, Thin Solid Films **239** (1994) 259-267.

<u>7.8 Stretched (mirror) foils:</u> Conventional mounted foils often are slightly limp and/or wavy (Fig. 15), which makes them more tolerant of mechanical stresses applied to the foil and/or frame. For some applications, however, foils must be mirror-smooth and flat (Fig. 16). Such foils are produced in a multistep process, with successive remountings of the foils. The process is described in the papers, J. Stoner, Nucl. Instrum. Meth. Phys. Res. A480 (2002) 44-49 and C. Jolivet and J. Stoner, AIP Conf. Proceeding 879, "Synchrotron Radiation Instrumentation" (2007) 1077-1080, Daegu, Korea, May 28-June 2, 2007. There is an additional

charge for this stretching process. Materials mostcommonly provided in this form are chromium, iron, titanium and aluminum, though some other materials can be used. Stretched foils are not tolerant of deformation of their frames or of contact with other objects; such deformation or



contact easily causes permanent wrinkles or tears to appear in the foil. Stretching cannot be carried out satisfactorily on certain materrials, particularly evaporated carbon, silicon, alumina and silica foils.

Stretched foils are usually mounted on stainless-steel washers having inner diameter of 11.1 mm, outer



diameter of 25.4 mm and thickness of 2.6 mm; experience has shown these to be sufficiently rigid to protect the foils in most applications. Special taperedrim washers have been used to obtain the flattest foils; such frames can be provided by ACF-Metals or by the customer (Fig. 17).



Stretched foils can be optically flat near their centers [See J.. Stoner and H. Supp, Appl. Opt. 8 (1969) 707.]. Irregularities in the flatness of the foil appear near the perimeter due to non-flatness of the rim or in the thickness of the adhesive.

7.9 Parameters to consider for a quotation :

A mounted foil is an assembly having several parts: a. The foil itself, either a single component, or a sandwich of several thinner layers of different materials. b. A supporting mesh (sometimes optional) to strengthen the foil.

c. A frame to keep the foil nearly planar, and to allow handling and fastening the foil into other apparatus.d. Adhesives to hold the assembly together.

ACF-Metals' mounted foils are mounted on frames that the customer specifies, either supplied by the customer or by ACF-Metals. The least-expensive foils use stainless steel washers as frames, and are assembled with epoxy adhesives. ACF-Metals collaborates with customers to provide other designs and construction materials as needed. For mounted foils that must block stray light thoroughly, double-foil filter designs are available (see below), and special techniques are used by ACF-Metals to reduce pinholes in the foil.

Customer should consider the following parameters when buying mounted foils:

a. Clear diameter, or dimensions of aperture to be covered.

- b. Thickness and composition of foil material.
- c. Mountings to be used (Fig's 15-19).

d. Standard specifications if satisfactory (see below), or revisions of those specifications if needed.

e. Limits on transmission of radiation by pinholes

f. Shock-loading or acoustic-loading requirements (e.g., for rocket launch).

g. Specifications of unusual adhesives or other materials.

h. Requirements on thermal cycling and/or particle bombardment and/or other environmental stresses.

7.10 Standard tolerances and specifications:

Thickness accuracy: +/-15% Thickness uniformity across the aperture: +/-10% Open (pinhole) area: <1% (in practice usually <<1%) Mesh support: nickel, 90%-transmitting, 5-7 μ m thick, or other materials if necessary.

7.11 Handling mounted foils:

A mounted foil combines strength with great fragility. Mounted foils are guaranteed to survive shipment to the customer. Some foils on mountings can be designed to withstand the rigors of rocket launch. However, a foil less than 0.2µm thick will usually be <u>destroyed</u> by touching it gently with <u>any object</u>. It can also easily be destroyed by excessive vibration, by requiring it to withstand too great a pressure difference, by putting liquid on it, by extreme heating or radiation damage caused by particles or radiation, or by other stresses, e.g. due to differential thermal expansion, flexure, or electrostatic charging. Many foil materials deteriorate when exposed to water vapor and/or oxygen, and should be stored in vacuum or in a desiccator.

7.12 Typical frame designs:

Typically designs for mountings for metal foils depend upon whether transmission of x -rays/euv is to be maximized, or whether unwanted radiation due to pinholes and other out-of-band radiation is to be reduced as far as possible. Examples of practical designs are shown in Figures 18 and 19.



8. METAL FOILS ON REMOVABLE SUBSTRATES:

8.1 Product description:

Metal foils, and foils of other materials, can be provided

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on metal or glass substrates, with parting agent to permit removal of the foil by the customer, by floating. These are available as standard products and as custom



assemblies. These are provided so that customers can mount their own foils at lower cost, or mount them on assemblies that are not easily shipped.

8.2 DISCLAIMER - PLEASE NOTE: (1) Foils on substrates have a limited shelf life and should be used within 30 days of receipt. (2) These foils are always produced under conditions to permit their being floated, and are tested by ACF-Metals before shipment. ACF-Metals guarantees that these metal foils will float off satisfactorily, if floatoff is carried out by one or more techniques described below, within 30 days of receipt of foils. However, ACF-Metals does not guarantee that any customer can mount them on his/her frames. (3) Personnel at ACF-Metals will be pleased to advise the customer concerning the feasibility of customer's intended application, in cases for which ACF-Metals has experience. Some experience and luck are needed to mount foils successfully. ACF-Metals suggests that the customer practice with carbon foils first; carbon is the easiest material to mount as foils.

8.3 Standard products:

Foils are produced on substrates, (Fig. 20) with parting agent to permit removal of the foil by floating (in water). Substrates are ordinarily 4 each, glass microscope slides, 25 mm x 75 mm, so that the foil size is 4 each, 25 mm x

70 mm. In some cases a metal plate is used as substrate. The foil size is then 1 each, 100 mm x 100 mm. Thickness is typically Customer's choice, 20 nm to 1000 nm; some materials may not be usable over this range, and some materials may have greater ranges.



8.4 Standard materials are as follows:

Aluminum Aluminum oxide (alumina) Bismuth (on C) Boron, natural and isotopic Carbon, natural and isotopic Chromium Cobalt Collodion (cellulose nitrate) Copper (on C) Dysprosium (on C) Germanium Gold Indium (on C) Iron Magnesium Manganese Molybdenum Nickel Niobium Silicon Silicon dioxide (silica) Silver Tin Titanium Tungsten (wolfram) Vanadium Yttrium (+Al)

Zinc (on C)

Zirconium

Prices depend upon the difficulty of making the foil, and typical prices are given in ACF-Metals' Price Sheet #9. Please contact ACF-Metals for quotations on other materials, isotopic foils, other thicknesses, or other sizes. Tolerance on thickness is +/- 15%. Most metal foils are made to order, since shelf life is limited for many materials. Delivery is typically 3-4 weeks, depending upon material and orders' backlog. THESE FOILS SHOULD BE USED WITHIN 30 DAYS OF RECEIPT!

Foils are not warranted to be light-tight, nor for pickup over specific apertures nor to survive specific handling after removal of substrate unless this is guaranteed explicitly in ACF-Metals' quotation.

8.5 Techniques for floating and mounting metal foils: Different metals and different thicknesses of the same metal behave differently when they are floated off of their substrates. Some float without incident. Others tend to sink and/or curl. Some are strong; others are too frail to mount. The following suggestions may help the customer, who should already be familiar with ACF-Metals' procedures for floating carbon foils (see above) before trying a metal foil.

If a technique for floating does not seem to work, please call for advice **before** all of the foils have been destroyed.

Metal foils are usually floated and mounted using many of the same techniques as for carbon foils (see above). However, there are some differences. Metal foils on parting agents, deposited either on glass or metal substrates, are often hydrophilic on their exterior surfaces and may refuse to float on the water surface, preferring to sink. If you observe this as you begin to float off the foil, STOP AT ONCE! [Stop also if the foil begins to curl badly.] Either problem will probably get worse if you continue. It often happens that a foil that has been immersed in water will either disintegrate, or never release again. Foils that curl into 1-2-mm-diameter tubes cannot be uncurled. Remove the foil from the water, dry the wet parts by blotting, and try a different technique for floating.

A problem with sinking can sometimes be alleviated by very slow floating (preferably by siphoning), or by droplet-floating (see above).

If these methods do not prevent the foil's sinking, there are four techniques still to be tried:

A Metal foils can be coated with a very thin (typically

10 nm or less) paraffin layer to make their outer surfaces hydrophobic. This is best done during manufacture, but if the customer has the necessary equipment, the coating can be done any time before floating the foil.

<u>B</u> Immerse an edge of the foil in water, then withdraw it. As the water begins to creep under that edge, mangle the edge slightly with a needle or a razor blade. Wait a minute or two, and attempt to float the foil slowly, starting at the mangled edge. This method works occasionally.

<u>C</u> The foil can be dip-coated in a water-free mixture of 15% flexible collodion and 85% amyl acetate (by volume), allowed to dry, then floated by any of the above methods. This is a good method also for reinforcing thin foils so that they can be mounted more easily. After the foil has been mounted on frames, the collodion may be removed by either immersing the foil in methanol (carefully!) or by draining methanol across the foil while it is held vertically, using a pipet to introduce methanol on the upper edge of the frame (for this technique see J.L. Gallant, Nucl. Instrum. Meth. <u>102</u> (1972)477), or by dry ashing in an oxygen plasma. [Do not use dry ashing for C, Mn, V, or Cu foils.] A dry-ashing procedure is described in Nucl. Instrum. Meth. Phys. Research <u>A480/</u>1-2 (2002) 171-177.

<u>D.</u> As a last resort, both for sinking problems and curling problems, you can glue your frames to the foil before removing the foil from its substrate. ACF-Metals uses epoxies for this purpose. When the glue has cured, immerse the foil + frames + substrate assembly in distilled water . When the water has penetrated under the foil and the frames, each frame can be gently pried off of the substrate, with the foil still attached to its frame, by gently sliding a single-edged razor blade under the frame. This process is described in Nucl. Instrum. Meth. Phys. Research <u>A480</u>/1-2 (2002 44-49. An experienced person can do this successfully typically about 50% of the time, with e.g. titanium foils 2-cm diameter and 0.5 microns thick. The foil on its frame can then be lifted out of the water and allowed to dry.

9. OPTICAL ATTENUATING FILTERS

9.1 Product description:

Long-distance telephone calls and LAN messages often go through ACF-Metals' carbon-based attenuators, used in fiber-optic communications networks. These attenuators are thin carbon-based filters that provide the desired attenuation at the infrared wavelength being used. Several conventional sizes and shapes are used: circular (style ATS, diameter 0.091", or ATD, diameter 0.125"), or approximately rectangular, 0.050" x 0.140" (style ATF, Fig. 21) or 0.050"x 0.129"(style ATX), all dimensions typically +/-0.002". The accuracy of measurement of the attenuation is typically 0.5dB or better. The filters are composed of an amorphous carbon layer laminated between polyester sheets; the total thickness is 0.00375" +/- 0.0015". The refractive index for the polyester is 1.64. Compared to gelatin filters, these are relatively insensitive to high temperature, humidity, and handling. These filters are 100% visually inspected at 10x for zero pinholes in the central region



and 100% transmission- inspected for correct attenuation at 1300nm test wavelength in free space. Filters are **made to order** with Customer-specified attenuations in the range 1-30dB at Customer-specified wavelength between 400nm and 1550nm. Customer should be aware that attenuator filters have attenuation values typically 1 dB to 1.5 dB higher when placed in a connector, compared to their attenuation in free space, and that these filters are <u>not</u> neutral-density filters.

8.2 Calculation of attenuations:

To evaluate the extent of non-neutrality of these filters, the transmission T of a filter can be calculated approximately according to $T = \exp[-4\pi kd/(wavelength)]$ where k is the absorption coefficient of carbon at the wavelength specified, and d is the thickness of the carbon layer. Absorption coefficients of carbon in the visible

and near infrared spectra can be found in the article: J. Stoner, Nucl. Instrum. Meth. A236, 662-664 (1985).

Optical attenuators can be made with a high degree of neutrality over the wavelength region from 800 nm to 1600 nm. These have the disadvantage that they are somewhat more reflective than carbon-based attenuators. Further information is available upon request.

9. VACUUM SERVICES:

Specialized vacuum services are available for production of coatings or foils to customers' specifications. These include oxygen etching for removal of hydrocarbon coatings, and metal and carbon evaporations onto customer's substrates. Base prices are listed in Price Sheet #10. Please request a quotation for your own special job.



FOILS & COATINGS FOR YOUR SCIENTIFIC APPLICATIONS NATURAL AND ISOTOPIC CARBON FOILS AND COATINGS: o Areal densities from 0.1 to 125 000+ micrograms per square centimeter ($\mu g/cm^2$). Extensive stock of natural carbon foils ready to ship. o Length and width up to 30 cm; standard sizes of 25mm x 69mm and 48mm x 69mm in stock. o Amorphous carbon or polycrystalline graphite depending on thickness. o Composition is well specified. o Accuracy in areal density is typically 10% or better. o Quick delivery anywhere. o Carbon-12 and Carbon-13 available up to >1000 μ g/cm². o Carbon-based optical attenuators to 30 dB. METAL FOILS AND COATINGS: o Many metals available, typically to 1μ m thick: Aluminum, Antimony Bismuth Boron, Boron-10, Boron-11 Carbon (any thickness); Carbon-12, Carbon-13 (to $> 5 \mu m$) Chromium, Cobalt Copper Dysprosium, Gadolinium, etc. Germanium, Gold Indium, Iridium Iron Magnesium, Manganese, Molybdenum Nickel, Niobium Platinum, Rhodium Silicon Silver Tantalum, Tin Titanium, Tungsten Vanadium. Yttrium Zinc, Zirconium and other metals, plus oxides, salts, on glass slides with or without parting agent, or free-standing, or mounted. o Thicknesses from a few nanometers to a few micrometers. TARGETS AND X-RAY FILTERS: o Metal or carbon foils mounted on your frames (or ours). o Mesh, plastic or foil backings if you require them. o TEM grids covered with carbon, metal or oxide films. o Combinations of elements available. o Quick response to requests for quotations. ACF-METALS The Arizona Carbon Foil Co., Inc. 2239 E. Kleindale Road Tucson AZ 85719-2440 Telephone: 520-325-9557 Fax: 520-325-9493 Net: http://www.techexpo.com/WWW/acf-metals

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