

# **ACF-Metals**

## **The Arizona Carbon Foil Co., Inc.**

### **PRODUCT INFORMATION**

Effective date: January 31, 2018

#### **Who we are:**

#### **WE MAKE CARBON FOILS OF ANY THICKNESS WHATSOEVER!**

For forty-eight years (since 1970), ACF-Metals has been dedicated to providing accurately characterized ultra-thin foils and coatings of carbon in thicknesses less than one micron (1000 nm, 0.00004") up to a few microns thick, at competitive prices, to customers around the world. Carbon foils are provided in any thickness whatsoever. We provide carbon foils to be used as targets, strippers, and extractor foils in accelerators, have seen our carbon foils with thickness less than 10 nm sent with confidence to Halley's comet (and other comets) and to planet Saturn and its moons, and have seen our carbon-film attenuators used in thousands of reliable fiber-optic-communication hookups. Current activities involve research to make foils having greater tolerance to ion bombardment and the extreme temperatures reached in high-current stripping applications. 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 for production purposes, specialized foils for research, or only a single unique foil or coating for a completely innovative application.

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## **1 INTRODUCTION:**

The carbon foils made by ACF-Metals are used in nuclear, space, optical, chemical, medical and microscopy research. This document focuses on the properties and uses of those foils, whether made by vacuum evaporation or by methods that result in graphitic foils. Such foils are of special interest because they are the most common stripper foils (charge-changing foils) and target-supporting foils involved in research and manufacture using particle accelerators. ACF-Metals has attempted in this writeup to provide answers to questions by researchers in these areas, but we are also happy to try to answer questions by e-mail that are not covered here.

Please reference this document, if you quote from it, as: John O. Stoner, Jr., ed., ACF-Metals Advertising Literature "Product Information" (2018).

ACF-Metals is always glad to receive reprints of work for which our foils were useful, and to receive suggestions for improving this writeup.

Carbon foils are produced in many forms and by many different methods. A summary of methods was given by Stoner and Miller (2006). [References begin on p. 29 at the end of this document.]

The two most-common allotropes, (basic crystalline structures) of solid carbon are graphite and diamond. Graphite [which includes highly-oriented polycrystalline graphite (HOPG), vitreous carbon and graphene as special cases] has primarily  $sp^2$  atomic coordination. This is the most stable form [cf. Pauling (1988)]. Graphene (see Section 1.1 below), diamond and HOPG foils are available commercially [EMS (2014), Applied Diamond (2017), Sigma-Aldrich (2013), Minteq (2017), Structure Probe (2017)]. Diamond ( $sp^3$  coordination) ranges from gemlike crystals of the highest chemical and crystallographic purity through mixtures having many different compositions containing vacancies, graphite or or other elemental impurities. So-called diamondlike carbon (DLC) has a fraction of  $sp^3$  coordination that depends on method of production, and typically has relatively low long-range order.

The most commonly used carbon foils for use as strippers, charge changers or targets in nuclear accelerators are currently arc-evaporated carbon foils. These are complicated mixtures involving graphitic bonds, diamond-like bonds, interstitials, vacancies and impurities, both intentionally and

unintentionally introduced. Evaporated carbon foils are often nearly amorphous, and consist of randomly intermixed  $sp^2$  and  $sp^3$  domains [Koptelov *et al.* (1989)]. A general review of amorphous carbon structures was given by [Chhowala (2003)]. Early reviews of properties of carbon films and foils were carried out by [McLintock and Orr, (1973), Goma and Oberlin (1980), and [Robertson (1986)]. Structures of several kinds of thin carbon foils were studied by transmission electron microscopy and by electron diffraction by [Dollinger *et al.* (1991)]. Extensive practical experience with thin carbon foils has been obtained by electron microscopists, who often assume that the thinnest evaporated carbon foils are amorphous [cf. Fan and Cowley (1985)]. Foils often are made of a single component; examples are resistively evaporated carbon [Maier-Komor (1972)], arc-evaporated carbon [Dearnaley (1960), Jaggi *et al.*, (2006)] or pulsed-laser-ablated carbon [Maier-Komor *et al.* (2006)], or as a combination of two or more components [cf. Zeisler and Jaggi (2008); Zeisler and Jaggi (2011); Jaggi *et al.* (2014)]. Within each type, specialized varieties including alloys are sometimes used to obtain foils that are particularly long-lived when used in accelerator beams [cf. Sugai *et al.* (2006), Sugai *et al.* (2008a), Sugai *et al.* (2014), and the discussions below]. Composition of arc-evaporated foils is further complicated by the fact that their  $sp^2/sp^3$  fractions differ at the anode (50/50) and at the cathode (60/40) of a dc arc [Koptelov *et al.* (1989)].

The structures of polycrystalline graphite (PCG) foils are also complicated, but have been studied to lesser extent than for evaporated foils.

For many applications, the microscopic structure of a foil is of minor interest. However, microscopic structures do affect the lifetimes of foils subjected to particle bombardment, e.g., placed in the paths of beams of high-energy particles. Foil lifetimes diminish as particle beam fluences increase. Improvement of in-beam lifetimes (survival times) of foils in intense particle beams continues to be of interest, and reports of improvements in foils continually appear, especially in the proceedings of conferences involving uses of accelerators in physics. The Customer can find references to these conferences at the websites for SNEAP (Society of Northeast Accelerator Personnel), WTTC (Workshop on Targets and Target Chemistry), INTDS (International Nuclear Target Development Society), and IPAC (International Particle Accelerator Conference), among others. The improvements typically involve microstructural modifications due to improved manufacturing techniques and/or alloying of the

primarily carbon foil with boron or other elements. [cf. section 2.25 below.]

In practice the most important parameter of a foil, from the

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point of view of using it in an experiment, is the foil's areal density. The areal density is calculated by

Areal density = (mechanical thickness) x (mass density)' which is the same quantity as (mass of foil/area). The usual units for this quantity are micrograms per square centimeter, ( $\mu\text{g}/\text{cm}^2$ ) or milligrams per square centimeter ( $\text{mg}/\text{cm}^2$ ).

The charge states, energy losses, stopping powers, energy straggling, and ranges of ions that traverse foils, depend on the foils' areal densities, and have been studied extensively both experimentally and theoretically for ion-beam analysis. These topics continue to be studied for application in the field of very large, high-energy particle accelerators. A tabulation of charge states of ions traversing carbon foils was published by [Sayer (1977)]. The extensive work by [Ziegler (1977-1980)] on stopping powers and energy losses of fast ions in matter has been reworked into several computer programs to make the data more easily accessible [cf. Pawlak and Greene (2011), Ziegler (2013) and the Sandia Ion Beam Analysis calculation programs and databases, B. Doyle (2015)]. Methods for calculating stopping powers continue to be improved [Javanainen (2012), Schiwietz and Grande (2012)].

[See section 3.02 below for information on charge-state equilibrium vs foil thickness.]

Areal densities are provided for all foils supplied by ACF-Metals. This quantity has several advantages: It is a quantity calculated easily from macroscopic mass and area measurements. It is often proportional to particle energy loss, and it does not require the knowledge of geometrical thickness of a foil, or its mass density (volume density, in g/cc), quantities that may be difficult to obtain.

### **1.1 THINNEST AND ALMOST THINNEST FOILS**

The thinnest foils of carbon appear to be graphene foils, that can be made down to a single atomic layer. Such a layer, a network of hexagonally linked carbon atoms, is the fundamental building block of graphite. Within an individual graphite crystal, each individual graphene layer is expected to have nearest-neighbor spacing (bond length) 0.1415 nm [Pauling (1988); Nanoprobes (2013)] and spacings between successive graphene planes about 0.3354 nm, 1/2 the height of a unit cell. The areal density of a single graphene plane = mass density of graphite x plane spacing. Since the theoretical density of a perfect graphite crystal is 2.265 g/cc and the spacing between planes is 0.3354 nm, the theoretical areal density of a single graphene plane is  $0.07597 \mu\text{g}/\text{cm}^2$ .

It has an areal density of  $3.82 \times 10^{15}$  carbon atoms/sq.cm.

The thermal properties of graphene have been extensively discussed by [Pop *et al.* (2012)]. An introduction to graphene can be found in Wikipedia (2012 *et seq.*). Recent articles [e.g. Chen *et al.*, (2009a), Guo *et al.* (2009), Garaj *et al.* (2010), Sutter *et al.* (2010), Gonçalves *et al.* (2010), Nair *et al.* (2010), Nelson *et al.* (2010), Lee *et al.* (2011), Karnik (2014), Hu *et al.* (2014), Colapinto (2014)] describe many possible applications of these materials. The properties of folded (kirigami) graphene sheets used as mechanical actuators have been studied by [Blees *et al.* (2015)]. Progress in this field is rapid, and methods have progressed to the making of small quantities of much larger graphene foils [Zheng *et al.* (2010), Lock *et al.* (2012)]. Design of graphene windows for transmitting high-power accelerator beams has been discussed by Wang *et al.* (2014), with the aim of utilizing the large thermal conductivity of graphene.

With its related subjects of nanotubes, buckyballs, and similar structures, graphene is currently an active research area; this material has found a few applications in accelerators to date. Some thick graphene foils have been tested for use as accelerator targets [von Reden and Sichel (2003), Marti *et al.* (2010); Hasebe *et al.* (2014), Kupka *et al.* (2014), Pavlovsky *et al.* (2016)]. A few applications for stripper foils based on carbon nanotubes are described by [Hasebe *et al.* (2014), von Reden and Sichel (2003), and von Reden *et al.* (2007)].

ACF-Metals has been advised that its arc-evaporated carbon foils having approximate areal densities of  $2 \mu\text{g}/\text{cm}^2$  sometimes show graphene layers [Noli (2011)].

Several materials are competitive for the title of "Almost Thinnest" foils. Six research areas appear to use these:

(a) Stripper foils for charge-changing, especially in van de Graaff accelerator terminals, where carbon foils having areal density nominally  $2 \mu\text{g}/\text{sq.cm}$  are common, but use of foils down to  $0.5 \mu\text{g}/\text{sq.cm}$  is reported. Diamond foils for this purpose were described by [Liechtenstein *et al.* (1996, 1997, 1998, 2010), M. Ivkova *et al.* (1995), and Ma *et al.* (2011)]. Thicker diamond foils are sometimes used for stripping in large accelerator facilities (see below).

(b) Space physics, especially, time-of-flight studies of space plasmas, where nominally  $0.5 \mu\text{g}/\text{sq.cm}$  carbon start-stop foils are typically used, e.g. on the Cassini mission [Krimigis *et al.* (2004)] to Saturn and its moons, launched in year 1997, and ended in September 2017. Such foils are typically mounted on

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nickel meshes having 13 lines/mm or 330 lines/inch [J. Baldonado (1998)]. Mechanical properties of such mounted foils have been discussed by [Collins, *et al.* (2009)]. More recent references include the description of the STATIC instrument of the MAVEN Mars probe [MAVEN (2014)], and the MMS Science Instruments description [NASA (2014)]. See McComas *et al.* (2004) for an extensive list of space projects using ACF-Metals' carbon foils. See also M. Gruntman (1997), and Ritzau and Baragiola (1998) for properties of mounted foils. Attempts have been made to replace carbon foils with micropore optics for similar purposes [Cadu *et al.* (2012)]; graphene foils may now be superior for these purposes [Ebert *et al.* (2014)].

(c) Stripper foils for Coulomb-explosion experiments, where formvar foils down to about 0.5  $\mu\text{g}/\text{sq.cm}$  (Both *et al.* (1987), and gold foils down to 10 nm thickness are used [S. Chelkowski *et al.* (1999) and references therein].

(d) Electron scattering (e,2e) experiments for measuring electronic momentum densities in foils [cf. Hayes *et al.* (1991), Storer (1995)].

(e) Substrate development and materials science in transmission electron microscopy, in which carbon foils nominally 5 nm thick (1  $\mu\text{g}/\text{sq.cm}$ ) often are used as substrates, ACF-Metals has mounted foils nominally 2 nm thick on transmission electron microscopy (TEM) grids, and a report recently indicates the use of gold foils of thickness only 3nm thick [Y. Kondo *et al.* (1999)]. Kawasaki *et al.* (2009) and Ueda *et al.* (2007) report the use of carbon foils only 8 nm and 10 nm thick, mounted on TEM grids, to support pressure differences in an environmental cell in TEM.

(f) Foils used as targets for laser-ion acceleration, typically parylene or similar materials only a few molecules thick [Aurand *et al.* (2014)]. Diamond-like foils were made by [Ma *et al.* (2011)] for this purpose with thickness down to 3 nm (corresponding to areal density of 0.6  $\mu\text{g}/\text{sq.cm}$ ).

ACF-Metals' carbon foils have been thinned by baking to about 1.4  $\mu\text{g}/\text{sq.cm}$ , and further by oxygen-plasma thinning, down to carbon areal density measured by Rutherford backscattering (RBS) of 0.8  $\mu\text{g}/\text{sq.cm}$  (equivalent thickness 4 nm); these were very fragile and their production was not reproducible. See also Both *et al.* (1987) for information on

thinning C foils.

## **2 EVAPORATED CARBON FOILS**

### **2.0 Introduction:**

There are more than 40 methods for making carbon foils [Stoner and Miller (2006)], but vacuum evaporation is the most popular. A pictorial description of this process is shown by [Stoner and Bashkin (1978)]. ACF-Metals' standard foils are made by pulsed arc evaporation, for thicknesses up to about 8 microns. They are nearly amorphous, but not completely amorphous. Electron microscopy of a foil's cross section shows a laminar structure built up by the individual arc pulses [Stoner and Stoner (1988)]. Proposals have been made to intercalate this structure to improve carbon foils [Hitchcock (2010)]. For foils thicker than about 3 microns, because of the extended time required for production, ACF-Metals usually replaces its standard foils with polycrystalline graphite foils (see Section 4, below). The evaporation of carbon to make foils was done initially more than 60 years ago [Bradley (1954); Cosslett and Cosslett (1957), see also Dearnaley (1960)]. Extensions of this work have been developed by electron microscopists [c.f. Hayat (2000)], by investigators making targets for nuclear physics experiments [Muggleton (1987)], and for the development of spark-gap switches [c.f. Zeng *et al.*, (2012)]. Complex evaporation processes, including processes to introduce alloying elements into such foils, continue to be developed. The latest reports of progress in these areas are found in the proceedings of conferences of the International Nuclear Target Development Society (INTDS), cf. [Steski, Stolarz, and Zeisler, eds. (2011)]. Stolarz (2014) has reviewed the general subject of preparation of nuclear targets.

### **2.1 Product description:**

These are ultra-thin self-supported carbon foils having natural isotopic composition: 98.9%  $^{12}\text{C}$ , also known as carbon-12, plus 1.1%  $^{13}\text{C}$ , also known as carbon-13, made by arc evaporation from a vacuum arc onto glass microscope slides or other substrates that have been precoated with a parting agent (Figures 1,2,3). They are variously known as carbon targets, charge-changing foils, SEM foils, TEM foils, stripper foils, extractor foils and carbon slides. All forms of carbon have atomic number  $Z = 6$ . The detailed atomic structures of carbon foils have been studied for many years, but are not yet completely understood. Foils are rough and somewhat porous; the difficulties involved in modeling such structures are described briefly by [Gelb 2009].

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Fig. 1. Evaporated carbon foils (approximately 22  $\mu\text{g}/\text{sq.cm}$ ) on glass substrates 25 mm x 75 mm:



Fig. 2. Evaporated carbon foil (approx. 80  $\mu\text{g}/\text{sq.cm}$ , 25 mm x 68 mm) removed from its substrate. The tendency to curl indicates residual stresses in the foil (see section 2.5 below):

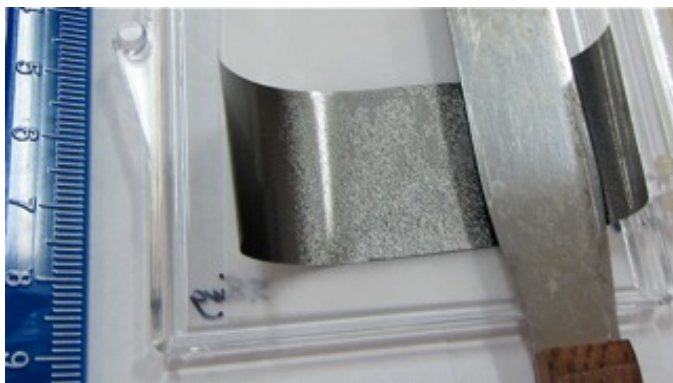
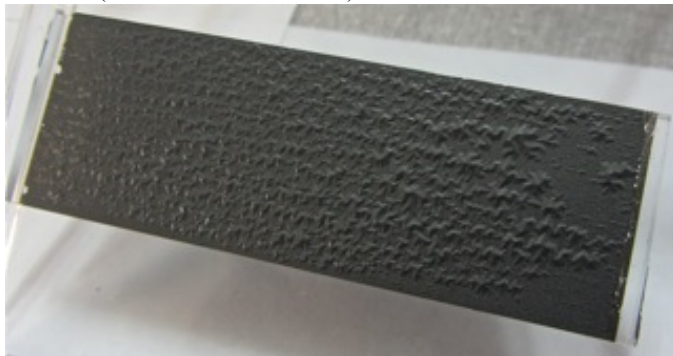


Fig. 3. Evaporated carbon foil (approx. 100  $\mu\text{g}/\text{sq.cm}$ ) on glass substrate 25 mm x 75 mm, showing compression-wrinkle pattern due to internal stresses (see section 2.5 below). This particular foil's substrate is glued permanently into its transparent plastic shipping box. This foil can be removed from its substrate by slowly immersing the substrate and box in water (see section 2.22 below).



## 2.2 Specifications:

Customer specifies the areal density (also known as surface density, specific density, or mass per unit area) of the desired foils. Alternatively the Customer may specify the desired equivalent physical thickness, also sometimes denoted "thinness". To provide specified values of physical thickness, ACF-Metals assumes that the mass density of foils is 2.0 g/cc. 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 68 mm if free-standing. Note that because the smoothness of edges cannot be guaranteed, free-standing foils are warranted to have no major edge defects only within dimensions 24 mm x 68 mm. Prices for all items are listed in our price list, available on request. The catalog number ACF-xxxx specifies the areal density xxxx in units of micrograms per square centimeter (variously written as  $\mu\text{g}/\text{cm}^2$  or  $\mu\text{g}/\text{sq.cm}$  or  $\mu\text{g}/\text{cm}^2$  or  $\mu\text{g}/\text{cm}^2$ ).

Foils in the areal density range 0-180  $\mu\text{g}/\text{cm}^2$ , corresponding to a maximum mechanical thickness of about 0.9 micrometer (0.9 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  $\mu\text{g}/\text{cm}^2$  to equivalent thickness in nanometers, assuming a bulk density of 2.00  $\text{g}/\text{cm}^3$ , multiply areal density by 5. To change areal density in  $\mu\text{g}/\text{cm}^2$  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 48 mm x 68 mm if free-standing. Because the smoothness of edges cannot be guaranteed, free-standing foils are warranted to have no major edge defects only within dimensions 48 mm x 68 mm. The catalog number XCF-xxxx specifies the areal density xxxx in micrograms per square centimeter ( $\mu\text{g}/\text{cm}^2$  or  $\mu\text{g}/\text{sq.cm}$ ).

Free-standing foils without frames are extraordinarily fragile if they have areal densities at or below 100 micrograms per square centimeter. Larger foils are more fragile than smaller foils, because a foil breaks easily if one attempts to bend it in two perpendicular directions simultaneously. In practice, a typical maximum feasible size for an unsupported 100  $\mu\text{g}/\text{sq.cm}$  carbon foil is about 30 mm x 120 mm.

Some other popular sizes and areal densities are described and their prices given in our price list. Still other sizes and thicknesses, foils on special substrates, and foils masked to Customer's specifications, are available by quotation.

Foils on substrates are provided with a parting agent that

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permits removal by floating. Many foils can be cut into pieces while still on their substrates, then floated onto a water surface, or can be cut on the water surface. Each foil piece may be picked up onto a frame having an aperture over which the foil is free-standing (self-supporting), so that it is supported only by its perimeter. Instructions and suggestions for floating and mounting are included with every order for foils, and are described below.

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

All of ACF-Metals' carbon foils are manufactured in the U.S.A.. They are RoHS-compliant (see < <http://www.rohsguide.com/> ) and contain no toxic materials of "very high concern" nor "conflict minerals" (currently Ta, Sn, W, Au). They are categorized under several different industrial codes: In the North American Industry Classification System (NAICS) under #335991, carbon and graphite products; under SIC code 3497, coatings; and under FSC code 9535, metal foil and leaf. The current harmonized tariff code (Schedule B) for carbon foils is 8311.90.0000 (Chapter 83) with ECCN code EAR99.

### **2.3 Reinforcement with collodion:**

**Caution: Collodion is highly flammable, and coating with it should be done only in small quantities, in a well-ventilated workspace, far from sparks, flames, and other sources of ignition.**

Without substrates, foils having areal densities below 5  $\mu\text{g}/\text{cm}^2$  are so frail that they are often difficult to mount over apertures larger than a few mm in diameter. Depending upon the application, foils may be coated on one side with a thin layer of collodion (cellulose nitrate) for additional strength during the mounting process; this layer typically has areal density of 20  $\mu\text{g}/\text{cm}^2$  +/- 10  $\mu\text{g}/\text{cm}^2$ . Areal density of the collodion coating is determined by the appropriate dilution of the material with a solvent [Stoner (1995)]. Some Customers do this coating for themselves. ACF-Metals does this coating upon request, at extra cost. Other users use solutions of Zapon<sup>TM</sup> [cf. Gallant (1972)] or formvar for similar purposes. Collodion has been used for strengthening diamond-like-carbon foils down to 0.5  $\mu\text{g}/\text{cm}^2$  [Liechtenstein (2010)]. Collodion may be applied to a foil by dipping it (on its substrate) into a suitably chosen solution of flexible collodion in amyl acetate, removing the foil and standing it on end on a paper towel to dry. Alternatively, the collodion solution may be flowed across the foil, or sprayed with an atomizer onto the

foil, and allowed to drain as above.

Customer is advised NOT to coat foils in this way if they have begun to separate from their substrates, as the collodion solution may penetrate under the foil and cement it permanently to the slide. Foils that have been collodion-coated by Customer are not warranted by ACF-Metals. Thicker foils that have begun to separate from their substrates, and free-standing foils, may be coated by floating them onto water baths and spraying a collodion solution onto them while they are floating. Such foils may be picked up onto mica sheets for storage and later re-floating.

Collodion may be removed after mounting the foil, either by gently flowing methanol over the foil [Gallant (1972)], or by an oxygen-plasma etch [Stoner (2002)], or by ultraviolet photoetching [Mitrofanov and Tokarchuk (1989)] or by the action of an energetic particle beam (as in the terminal of a tandem van de Graaff accelerator). The latter method was described in detail by [Liechtenstein *et al.* (2006)]. For

instance, a 50 nA, 1400 kV  $^4\text{He}^+$  beam focused to 2 mm x 2 mm will remove such a coating in a few seconds [McIntyre (2002)]. Both collodion and carbon layers are damaged by exposure to atomic oxygen, for instance in low-Earth orbit. [Verker *et al.* (2011)]. Collodion-reinforcement permits the mounting of carbon foils having nominal areal density of 2  $\mu\text{g}/\text{cm}^2$  or less over apertures of 10 mm diameter, or mounting of 10  $\mu\text{g}/\text{cm}^2$  foils with one free edge. Without the use of collodion, such mounting is very difficult.

Some users claim that foils treated in this way, when used as targets for particle beams, have extended lifetimes compared to uncoated foils.

ACF-Metals provides carbon foils with collodion coatings only on one side. Prices for these are included in our price list, or by quotation.

Foils that have been collodion-coated while floating, then picked up on glass or mica sheets, have been floated without difficulty after three years' storage [Tucker (2014)]. There is some evidence that coating a foil with collodion while it is on its substrate reduces its shelf life to about 3 years [Guy (2003)]. Foils that had been collodion-coated, on their substrates, then stored in laboratory atmosphere for eight years, floated poorly, but could be rejuvenated by recoating them [Jacobs (2011)]. We tentatively conclude that foils that are collodion-coated should not be stored on substrates for extended times unless you are willing to recoat them later.

### **2.4 Measurements, tolerances and uniformities:**

Every carbon foil provided by ACF-Metals has had its areal density measured, and is labeled individually. The

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measurements are done optically, near the centers, for foils still on their substrates, and by weighing for free-standing foils. The optical measurement is described in two publications: [Stoner (1969), Stoner (1985)]. Optical calibration has been done by several authors and is known to vary with production method [Cosslett and Cosslett (1957)]; ACF-Metals' method has remained constant since year 1970, and is occasionally reverified. The accuracy for the optical measurements is believed to be  $\pm 10\% \pm 0.5 \mu\text{g}/\text{cm}^2$ . This specification applies only to the carbon content and does not include non-carbon impurities (see below). Weighing measurements for total areal densities of foils above  $200 \mu\text{g}/\text{cm}^2$  have typical uncertainties below  $\pm 5\%$ .

Foils having areal densities below  $100 \mu\text{g}/\text{cm}^2$  are ordinarily chosen from stock to Customer's specifications within  $\pm 10\% \pm 0.5 \mu\text{g}/\text{cm}^2$ . Thicker foils are ordinarily provided to Customer's specifications within  $\pm 15\%$  or better. Foils at tighter tolerances are provided by quotation. Because tighter tolerances require manufacturing of larger quantities of foils, surcharges may be added to the prices of such foils. When a customer orders foils, ACF-Metals replies with a proforma invoice that describes the products ordered. These products conform to the characteristics described in this writeup, unless customer has provided additional specifications that are also included in the invoice.

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

Microscopic nonuniformity of thickness in thin foils can result from small corrugations due to uneven surface of the parting agent [cf. Dollinger and Maier-Komor (1989), Maier-Komor *et al.* (2006)]. Wrinkling of thick foils (see Fig. 3 above) due to stresses in production also causes nonuniformities in thickness. Such nonuniformities are not easily specified, but can be identified in some cases by energy straggling in transmitted ion beams.

### **2.5 Stresses, annealing, and packaging options:**

Stresses usually appear in freshly deposited carbon foils, causing them to release somewhat from their substrates, and curl when removed from their substrates (Fig's. 3,4).

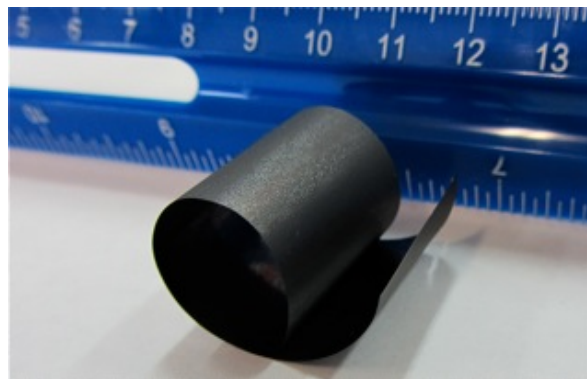


Fig. 4: Evaporated carbon foil, approximately 1.5 microns' thickness ( $300 \mu\text{g}/\text{sq.cm}$ ), removed from substrate without annealing. The radius of curvature in this example is approximately 1 cm:

It has been pointed out by several authors that a foil curls because its stresses vary (presumably monotonically) as a function of depth through the foil. The tubes produced by the curling of foils to relieve such stresses can have some practical applications [cf. Huang *et al.* (2009), Cendula *et al.* (2009)], although for use in microscopy and targetry, the curling is usually detrimental. The stresses are greatly reduced by baking (annealing) the foils. Typical baking times are a few hours in air at temperature about  $250^\circ\text{C}$ .

For foils below  $40 \mu\text{g}/\text{cm}^2$ , curling is usually slight and heat treatment is usually unnecessary. However, such foils can be baked by ACF-Metals if Customer so specifies. In the range  $50\text{--}100 \mu\text{g}/\text{cm}^2$ , effects of 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.

Carbon foils nominally  $25 \text{ mm} \times 70 \text{ mm}$  in the areal density range  $0\text{--}79 \mu\text{g}/\text{cm}^2$  are packaged and shipped in opaque rigid blue plastic boxes, up to 25 foils per box. Alternatively, these foils can be packaged in individual plastic tubes at additional cost. Foils  $50 \text{ mm} \times 70 \text{ mm}$  in the areal density range  $0\text{--}79 \mu\text{g}/\text{cm}^2$  are packaged and shipped in plastic tubes, one or two foils per tube.

Evaporated carbon foils having areal densities in the approximate range  $40\text{--}180 \mu\text{g}/\text{cm}^2$  are annealed (baked) by ACF-Metals before shipment to reduce their stresses. These foils develop wrinkles, bubbles, and tend to be somewhat loose on their substrates (see Fig. 3 above). Foils that have loosened cannot easily be cut on their substrates without risk

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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; this involves opening the box and submerging both the substrate and open box in water.

Especially for foils having areal densities **near  $100 \mu\text{g}/\text{cm}^2$** , several kinds of packaging are used:

Option A (recommended and used by ACF-Metals unless otherwise specified by Customer):

Foils are baked (annealed) at ACF-Metals' plant. Each foil's glass substrate is fastened permanently with double-sided tape into one half of a transparent plastic box containing the foil.

With the box closed, the foil is fully protected from damage in storage and shipping. When the box is opened, the carbon foil is exposed, but the glass substrate cannot be removed from the box. The foil is removed from the glass by slowly immersing only the half of the plastic box that contains the foil into a water bath; the foil floats on top of the water and can then be cut with sharp scissors and mounted on frames, forks or other supports. ACF-Metals does not recommend that such a foil be cut while it is on its glass substrate.

Advantages of Option A: Very low probability of damage to the foil. Ease of inspection. Foil is guaranteed against damage in shipping.

Disadvantages of Option A: Inconvenience of having to immerse the box in water to remove the foil.

Option N:

Foils are baked (annealed) at ACF-Metals' plant. Each foil, still on its substrate, is placed either in a transparent plastic tube, or between paper sheets on a foam pad in a transparent plastic box.

Advantages: Convenience of use. Customer familiarity.

Disadvantages: Foil is almost always slightly damaged by contact with tube or paper sheet, and is sometimes badly damaged during shipping or removal. No guarantee against damage in shipping.

Option R:

Foils are baked (annealed) at ACF-Metals' plant. Each foil is then removed from its substrate and placed between paper sheets on a foam pad in a transparent plastic box.

Advantages: No floating required by Customer.

Disadvantages: Extra cost. Difficulty of handling such thin foils by tweezers. No guarantee against damage to foil in shipping.

Option X:

Foils are not annealed. Foils are stored and shipped on their substrates in standard microscope-slide boxes or tubes.

Advantages: Maximum tightness of foil on its substrate.

Disadvantages: Foils may curl, sink and/or break up during storage, during shipping or when floated off, unless Customer bakes (anneals) foils beforehand. Releasing and breakage may occur during storage and shipping because of stresses in the foil. No guarantee against damage to foil in shipping or storage.

Foils having areal densities greater than about  $180 \mu\text{g}/\text{cm}^2$  are ordinarily annealed and then completely removed from their substrates by ACF-Metals. The foils are then free-standing (self-supporting); they are packaged individually between paper sheets in transparent padded plastic boxes.

## **2.6 Shipping:**

Foils are guaranteed to arrive at the Customer without breakage, and to float off satisfactorily [see sections 2.17-2.23 below]. When foils appear to be particularly fragile (which can include either mounted foils or annealed foils on glass slides) or for large quantities of foils of any type, ACF-Metals ships the foils in special acoustic packaging consisting of a steel box surrounded by soft padding, within a cardboard carton. There is an extra charge of U.S.\$50.00 for this special packaging. Shipment is made via air, via United Parcel Service (UPS) or Federal Express (FedEx), or other commercial carrier specified by the Customer. U.S. Postal Service is not used. Shipping of merchandise is ordinarily prepaid by ACF-Metals, with duties and taxes (V.A.T. etc.) collect. For the latter purpose, international customers must provide the necessary tax identification numbers.

If foils arrive broken or with other damage, they will be replaced at no cost to customer. Please make all such claims in writing, preferably by e-mail, within 60 days of receipt.

## **2.6.5 Customs charges**

In every case, Customer is responsible for customs charges. In the unfortunate cases where customer refuses the shipment by refusing to pay the customs charges, the shipment is destroyed and the customer receives no refund.

## **2.7 Surface appearance, storage and warranties:**

Foils on substrates are often observed to develop a matte appearance and/or small wrinkles due to incipient release of the foils from their substrates. This is caused by humidification of the interface between foil and substrate, with resulting release of compressive stresses in the foil. This phenomenon is normal. ACF-Metals does not warrant against the development of this appearance, nor against small bubbles in the foil if it releases locally, so long as the foil floats off of its substrate without breaking. Except by quotation, there is no warranty on pinholes, neither number nor size, nor on surface appearance of foils, which can vary widely depending upon thickness and storage conditions. If customer has any requirement concerning flatness and/or pinholes, that requirement must be specified in his/her purchase order. At room temperature, uncoated carbon foils on glass slides

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have no known limit on shelf life. The only exception to this statement concerns foils having areal densities below about 2.5  $\mu\text{g}/\text{sq. cm}$ , for which there is some opinion that foils older than a few years float unreliably. Some Customers claim however 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, sometimes for years, in Tucson, Arizona, U.S.A..

Unsupported foils that are mounted on frames are fragile and are easily broken by shock, electrostatic attraction, coughs, sneezes, vibrations, air currents, contact with liquid, or other insult. Otherwise, the shelf life of undisturbed mounted carbon foils appears to be limited primarily by the stability of any adhesive used. There is only fragmentary evidence that mounted carbon foils may become more fragile with time, with some tendency to curl; however, if foils must be mounted and then stored for long times (years) it would be sensible to anneal them first. Carbon foils on their original substrates, carbon foils that have been floated and picked up on freshly cleaved mica sheets, carbon foils that are free-standing and stored in transparent boxes, and unsupported carbon foils on frames or cyclotron forks have been stored more than ten years in laboratory atmosphere without apparent deterioration.

Warranty on foils' floating extends one year past date of receipt by Customer. Occasionally, a Customer reports either that foils fail to float off, or that they break up or curl tightly to the extent that they are not usable. If this happens to you, please contact ACF-Metals, preferably within 60 days after receipt, for replacement. In some cases this problem can be attributed to overheating of foils during shipping or storage.

### **2.8 Pinholes:**

Even with the greatest of care in production, carbon foils have some pinholes and other defects. For the purpose of identifying and/or avoiding them, pinholes may be more easily seen with the use of brighter backlighting, higher magnification, and better dark adaptation by the observer. Very thin foils may stick at tiny adhesions, and pull holes in themselves when removed from their substrates; thus for areal densities below about 5  $\mu\text{g}/\text{cm}^2$  it is difficult to obtain an entire foil without such holes, and it is also difficult to find tiny holes when you suspect their presence.

If pinholes must be minimized to minimize unwanted transmitted light or to minimize the presence of unstripped ions in transmitted particle beams, the best method appears to be to superimpose two foils, each that has half the total desired thickness or areal density, so that the holes in one foil are blocked by the other foil [cf. Barstow *et al.* (1987); Mitrofanov and Tokarchuk (1989)]. In such cases, care must

be taken to avoid trapping of air between the two foils.

Evaporated carbon foils have been used successfully as windows for low-pressure gas-cell targets [Golser *et al.* (1989); Weisser (2005); Maier-Komor *et al.* (2006)]. Arc-evaporated carbon foils are not gas-tight; they show some porosity, evident from their transmission of gases when used as barriers between regions of different pressure, even when pinholes are blocked by use of multiple foils [Lagomarsino *et al.* (1991)]. When used as an overcoat to protect reactive metals from oxidation, arc-evaporated layers are not as effective as sputtered carbon layers. Attempts to make denser arc-evaporated carbon foils with self-ion-assisted deposition were not particularly effective [Stoner and Stoner (1987)]. For insight into the difficulties of making a gas-tight free-standing foil, see [Mutikainen (1994)].

No particular pinhole level is warranted by ACF-Metals except by quotation. If you find a foil that appears to have unreasonably many pinholes, please photograph it and e-mail the photo to ACF-Metals so that the foil can be considered for replacement.

Pinholes are observed to form spontaneously in hybrid-type boron-carbon foils when they are operated at temperatures higher than about 1800 K [Sugai *et al.* (2010)].

### **2.9 Mechanical strength, breaking strain and Young's modulus:**

It occasionally is desirable to separate regions of different pressures with ultra-thin foils. For such purposes [Huizenga, *et al.* (1981)] pointed out the easily derived relationship that the burst pressure  $P$  of a film having thickness  $= t$  over a hole diameter  $= D$ , with tensile strength  $= S$ , is approximately  $P = (4t/D)S$ .

Using this relationship, evaporated carbon foils usually behave as if their breaking strength (breaking stress or yield stress) is approximately the same as that of commercial bulk graphite, a typical range of about 10 MPa to 15 MPa [see Ultra Carbon (1977) and Call *et al.* (1979)], or of reactor graphite, 19 to 21 MPa [Li Hongwei *et al.* (2007)]. Similar results were obtained by Golser *et al.* (1989), and by Lagomarsino *et al.* (1991). Note however that Kawasaki *et al.* (2009), and Ueda *et al.* (2008) found that their carbon foils had breaking stresses of the order of 500 MPa, far stronger than predicted from properties of graphite, suggesting the possibility that they inadvertently used diamondlike foils.

Foils thicker than 0.5  $\mu\text{m}$  (100  $\mu\text{g}/\text{cm}^2$ ) are typically wrinkled and therefore non-planar. Random wrinkling develops during the evaporation process and does not disappear during annealing (see Fig. 3 above). This serves to make thick foils

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more resistant to bending, and hence to stabilize them in use as stripper foils [cf. figures in Sugai *et al.* (2006); Jolivet *et al.*, (2008)]. Similar effects have been described in graphene foils [Blees *et al.* (2015)]. Diamond foils used as stripper foils are artificially corrugated to accomplish the same purpose [Spickermann *et al.* (2008a); Plum *et al.* (2008)].

Evaporated carbon foils apparently have no range of plastic strain at room temperature, and are similar to glass in that respect. If forced to bend or stretch beyond their elastic limit, carbon foils typically break at local stress concentrations due to wrinkling. Wrinkling does not increase their macroscopic deformability, as it does with more-plastic materials [cf. Maruyama *et al.* (2011)]. Plastic strain is clearly possible at elevated temperatures; curling and other evidences of strain disappear when foils are baked for several hours in air to 288 °C, or higher temperatures for shorter times [Miller and Stoner (2008)]. The breaking strain can be estimated at room temperature by bending foils around mandrels of successively smaller diameters, and is found from minimum bending diameters to be about 0.0011. This method is sensitive to the presence of microcracks.

If we assume that the breaking stress lies in the range 10-21 MPa (see above) then a range for Young's modulus  $Y$  for evaporated carbon foils can be estimated as the ratio of breaking stress/breaking strain, yielding the range  $Y = 9$  to 19 GPa. For comparison, this range overlaps the range of Young's moduli for bulk graphite, various grades, with and across grain, which covers the range 6.2 to 14 GPa [Ultra Carbon (1977)].

## 2.10 Mounted foils:

Self-supported carbon foils can be provided on rings, washers, stripper forks, TEM grids, Customer's supports and other mountings, ready for use (see Figs. 5a, 5b). More information on mounted foils is given in section 3, below.

Fig. 5a. Evaporated carbon foil, approximately 5 µg/sq.cm, on 70 line-per-inch mesh on mounting ring.

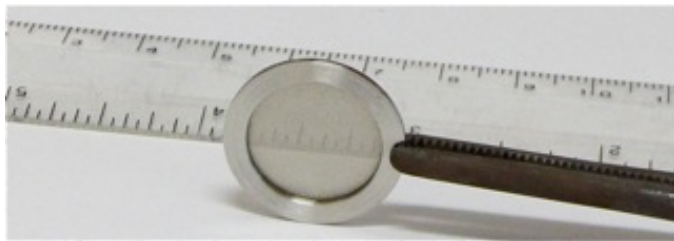
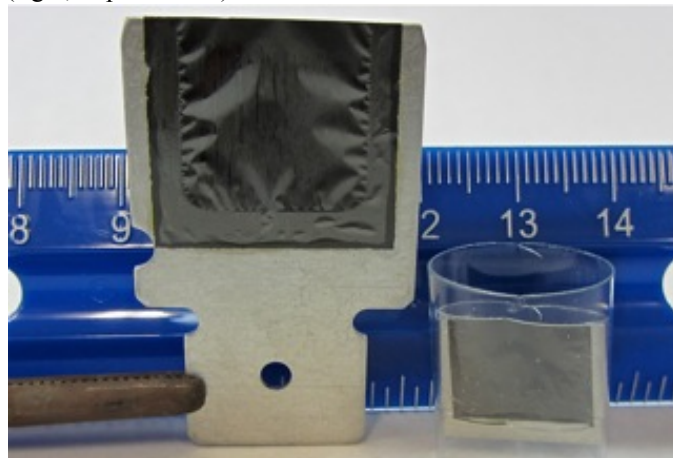


Fig. 5a. Evaporated carbon foils mounted with free edges, approx. 75 µg/sq.cm on cyclotron forks F30 (left) and F12

(right, in plastic cell).



## 2.11 Optical properties and electron transmission:

Carbon films are used in the extreme ultraviolet as spectral filters [cf. Hunter (1973), Stoner & Bashkin (1978)], and as supports for metal films used as spectral filters. In addition, they are used in the infrared to attenuate fiber-optic signals.

The transmittance of a carbon foil is defined as  $T = I/I_0$  where

$I$  is the intensity of light transmitted and  $I_0$  the intensity of light normally incident on the foil. The optical density OD of a foil is then

$$OD = \log_{10}(I_0 / I) = \log_{10}(1/T).$$

Accurate theoretical estimation of the optical transmittance of a carbon foil at a wavelength of interest requires knowledge of the complex refractive index ( $n + ik$ ), also known as the index of refraction, of the foil. Here  $n$  is the real part of the refractive index, and  $k$  is the imaginary part of the refractive index (variously also called the absorption constant or the absorption coefficient or the extinction coefficient). These quantities for several forms of carbon were presented by [Edoh (1983)] from the far infrared to the far ultraviolet. More-limited tabulations of  $n$  and  $k$  appear in many places in the literature. Unfortunately,  $n$  and  $k$  for evaporated carbon layers depend sensitively on the preparation method [Cosslett and Cosslett (1957)], so there are wide variations in the published values of these quantities. Furthermore, measurement of transmittances of carbon foils to find their optical constants are complicated by pinholes [Barstow *et al.* (1987)]. The general connections between optical constants, the (complex) dielectric constant, and transmittance are given by [Born and Wolf (1975)]. In cases for which  $T \ll 1$ , the transmittance of a thin foil at normal incidence can be estimated to first approximation for wavelength  $= L$  and foil thickness  $d$ , as

$$T = 4(n^2 + k^2) / [(1 + n)^2 + k^2]^2 [\exp(-4\pi k d / L)].$$

[see Born & Wolf (1975), section 13.4.1.; see also Maier-

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Komor *et al.* (1991)]

When the transmittance may be approximated as a simple exponential,  $T = [\exp(-4\pi k d/L)] = [\exp(-\mu d)]$ , where  $\mu = 4\pi k/L$  is variously known as the absorption constant, the attenuation coefficient, or the linear absorption coefficient.

Transmittances of carbon foils at photon energies in the range 40-1000 eV can be found using the program by Gullikson (2015). A brief summary of optical properties of evaporated carbon films was tabulated by Alterovitz (1991); see also Stoner (1985)]. Transmission measurements on carbon foils at selected wavelengths were presented by [Maier-Komor *et al.* (1991)] and confirmed by electron-transmission measurements..

Reflectance data for graphite were compiled by [Taft and Philipp (1965) and [Philipp (1977)], and summarized by [Edoh (1983) and Palik (1991)]. Reflectance data were used by [Windt *et al.* (1988)] to derive experimental values of  $n$  and  $k$  for carbon films in the far ultraviolet.

With baking in air at 260 °C for several hours, carbon foils' optical properties change slightly, with absorption coefficient increasing by the order of 10% (see discussion of baking and annealing, section 2.17 below).

### **2.12 Mass density and thermal properties:**

The mass density (mass/volume) of ACF-Metals' standard arc-evaporated carbon foils has been measured by buoyancy, yielding (at 20  $\mu\text{g}/\text{cm}^2$ ): 2.01 +/- 0.02 g/cc, whereas the bulk density of thick evaporated carbon layers was 1.83 +/- 0.03 g/cc. Many other investigators have measured densities of carbon foils; results depend upon the method of production.

For instance, density of foils at 0.5  $\mu\text{g}/\text{cm}^2$  was found to be 1.8 g/cc [Ritzau & Baragiola (1998)]. and for thicker foils found to be 1.82 g/cc [Kennedy *et al.* (1967)]. Further information concerning the densities of carbon target foils was summarized in [Stoner (1991)].

The researcher must exercise caution when using densities obtained by flotation (Archimedes' method) because of measurement uncertainties introduced by surface roughness and internal voids in foils [see discussion by Rouquerol *et al.* (1994)]. If crevices, pits and holes are present, the interface between water and carbon cannot exactly follow the shape of the carbon, but is affected by wetting angle and sidewall angles [cf. Parry *et al.* (1999)]. This implies that the density measurements by immersion in fluids will at some accuracy level depend on details of the pore and surface structure.

Many examples are known of experimental deviation of density of thin films from density of bulk solids. These experiments are well understood from early simulations of

ballistic aggregation of thin films [see Bartholomeusz (1988). Stoner (1988, 1989, 1990)]. Those simulations also predict a slight decrease of foil's density with distance from the substrate on which deposition is made.

Extensive recent work [see references in Makeev and Srivastava (2009)] provides much more information concerning relationships between atomic coordination number, density, and other physical properties of carbon foils, and allows for the prediction of thermal conductivity as a function of temperature, the theory yielding about 1.5 W/(m-K) at 300 K. Experimental work has been reported by Bullen *et al.* (2000), for films of various compositions.

In attempts to predict the operating temperatures of carbon target foils in intense ion beams, it is necessary to have information on both optical and thermal properties. The specific heat capacity of carbon foils has been reported to be 0.6908 J/(gK) at room temperature, rising at higher temperatures [Chou *et al.* (2008)]. The average emissivity was assumed to be 0.8 by these authors. An effective emissivity of 0.3 to 0.4 was found experimentally for composite foils by Sugai *et al.* (2006); Dollinger and Maier-Komor (1989) used a value of 0.75 for thick foils. Marti *et al.* (2010) found that emissivity = 0.4 fitted their high-temperature data. Emissivity of 0.9 was used by [Tahir *et al.* (2014)]. Assuming that radiation at a single wavelength is incident on and transmitted by a foil at a well-defined angle, the simplest relationship among reflectivity (or reflectance), transmissivity (or transmittance) and absorptivity (assuming emissivity = absorptivity) is

reflectivity + transmissivity + emissivity = 1

which enables measurement of emissivity by measuring the other two quantities. [Kupka *et al.* (2014)] have in this way shown that the emissivity of a carbon foil can vary in the range from 0.25 to 0.80, for temperatures in the range 25 - 450 °C. It is noteworthy that the radiative cooling efficiency for a target foil increases significantly as the power deposited in it by an incident ion beam raises the foil's temperature.

The temperature in a carbon arc is typically 5000-6000 K [Wikipedia (2017)]. The melting point  $T_m$  of carbon at low pressure is about 3823 K. During the growth of evaporated carbon foils, the atoms, ions and clusters of carbon vapor originating in the arc have effective temperatures  $T$  of thousands of K. These are quenched in a small fraction of a millisecond to approximately room temperature (near 300 K) upon collision with the growing film. The structure-zone model [Movchan and Demchishin (1969), Thornton (1986)] of a material with melting point  $T_m$  predicts that in Zone 1, for which the substrate temperature is below 0.3  $T_m$  (about

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1147 K, or 874 °C, for carbon) the surface and volume diffusion rates are expected to be slow compared to the time required to form the film. Such a film then is poorly consolidated, with many vacancies. Only if the substrate temperature is in Zone 2, for which  $0.3 T_m < T < 0.5 T_m$  (in the range about 1147 K to 1912 K, or 874 °C to 1638 °C for carbon), does diffusion in the growing surface fill in enough of the vacancies to allow a well-developed columnar structure to form on a microscopic scale. In Zone 3, for which the substrate temperature has  $T > 0.5 T_m$  (1912 K or 1638 °C for carbon), both surface and volume diffusion rates are sufficiently large to permit the development of macroscopic crystals during the formation of the film.

Electron microscopy of carbon foils evaporated onto room-temperature substrates and intended for use as nuclear targets indicates that such foils are initially amorphous or nanocrystalline, with the atomic binding largely  $sp^2$ , i.e., graphitic, but their structure is observed not to be stable as produced. Instabilities in the structures of freshly evaporated foils anneal out slowly at room temperature, and more rapidly as temperatures approach 800 K [Miller *et al.* (2008)]. An equivalent activation energy for the stresses that cause curling of foils can be estimated from an Arrhenius graph of times required for reduction of curling at various temperatures, and is of the order of 0.34 eV. However, there may be several different annealing processes that reduce curling, that in concert yield this effective phenomenological value.

Carbon foils that are used as accelerator targets, especially as stripper foils, often operate at elevated temperatures. Experimental properties of carbon foils used for stripping intense beams of heavy ions, and alternative methods of stripping such ions, have been discussed recently [Marti *et al.* (2010), Marti (2012), Kupka *et al.* (2014)]; see also section 2.18 below. Some dimensional changes of foils at high temperatures have been described in the paper by [Miller *et al.* (2008)]. The usual equations for the vapor pressure and evaporation rate of carbon as a function of temperature are given in that paper.

The usual formula, attributed to Langmuir, for evaporation rate of a single species is

$$m = P[M/(2\pi RT)]^{1/2}$$

$m$  = vaporization rate,  
 $P$  = equilibrium vapor pressure,  
 $M$  = molecular weight,  
 $T$  = absolute temperature in K.

The vaporization coefficient =  $\alpha$  of any material is defined for practical purposes as

$\alpha$  = (actual vaporization rate)/(evaporation rate calculated via the Langmuir formula from the vapor pressure).

When one assumes that the Langmuir formula is correct for some material, the assumption is equivalent to assuming that  $\alpha = 1$ .

However, carbon does not evaporate as a single species, but as C, C<sub>2</sub>, C<sub>3</sub> and other species. If the evaporation rate of carbon is calculated based on the vapor pressure of monotomic C only, it cannot be correct. The evaporation rate, and therefore foil lifetimes against evaporation, must thus include the vaporization coefficient [see Thorn and Winslow (1957)]. Experimental measurements of the vaporization coefficient for graphite, mentioned by [Zavitsanos (1966)] were as follows;  $1 \times 10^{-3}$  (claimed by Zavitsanos to be an error), 0.095 (as measured by Zavitsanos), 0.15 as measured by Thorn and Winslow, and 0.07 [Burns *et al.* (1964)]. It is apparent that neglect of the vaporization coefficient can cause significant errors in calculation of evaporation rates of foils.

Several other effects should be considered for use of foils at high or low temperatures, or subject to high irradiance: These effects include: (1) If foils are mounted on frames or meshes having coefficient of thermal expansion different from carbon, they will split at extreme temperatures [cf. Keski-Kuha (1989), Jolivet and Stoner (2008)]. (2) Evaporated carbon foils expand in length and width irreversibly by about 5% when heated to 800 K or above, and PCG foils shrink in length and width by about 1%. These effects have been seen by other users and have been described in detail by [Dollinger and Maier-Komor (1989)], and [Miller *et al.* (2008)]. They occur in the absence of a particle beam and therefore are not due to "ion hammering" proposed by [Benyagoub and Klaumunzer (1993)]. (3) Foils interact with residual gases in the vacuum chamber and can be damaged by those gases if the pressures are sufficiently high. A striking example of erosion by residual gas can be found in [Maier-Komor *et al.* (2006)]. (4) Evaporated carbon foils graphitize in different ways at different temperatures. Several investigators have studied these effects; some references to that work are given in [Stoner & Miller (2006)] See also [Marti *et al.* (2012)]. Probably the most extensive work on thermal changes of thin carbon foils was carried out by [Dollinger *et al.* (1991)]. Additionally, diamond foils graphitize when the temperature approaches 1800 K. This effect, which is consistent with the entry into structure zone 3, has been seen by several authors [cf. Sugai *et al.* (2006)]. Sputtered amorphous carbon foils are graphitized by irradiation at sufficiently high levels by soft x-rays [Chalupsky *et al.* (2009)] and it is reasonable to expect similar modification of evaporated carbon foils.

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### **2.13 Electrical resistivity and surface energy:**

The dc electrical resistivity of ACF-Metals' freshly arc-evaporated carbon was measured to be in the range 1-2 ohm-cm, approximately independent of areal density for foils in the range 2-100  $\mu\text{g}/\text{cm}^2$ , and has been used to monitor the thickness of growing foils [Stoner and Bashkin (1976)]. It should be noted, however, that the resistivity has been found by other investigators [cf. Toyoda and Nagashima (1959)] to depend on thickness, production details and exposure to air, to rise steeply as foils become thin enough for surface scattering to be significant, and to diminish greatly under heat treatment and/or irradiation by fast ions.

Freshly evaporated carbon foils are hydrophobic on their exterior surfaces, but hydrophilic on their substrate surfaces. Differences in electrical resistances of foils are seen depending on which face of a foil is contacted. Resistivity measurements on carbon foils must therefore be done with care to account for such effects [Sommer *et al.* (2008)]. In many literature reports it is not clear whether surface resistances have been considered.

For most carbon foils, surface energies are poorly known. A surface energy for DLC (diamondlike) coatings on magnetic hard discs of 53 mJ/m<sup>2</sup> was quoted by Brunner *et al.*, (2009), but electron microscopists have known for many years that the hydrophilicity of carbon evaporated coatings can be changed readily by exposure to different laboratory atmospheres [M. Hayat (2000)], and there is always the suspicion that such exposure, especially to glow discharge, may introduce impurities to the foil surface [Wall *et al.*, (1985)]. This suggests that in practice the surface energy is usually unknown.

### **2.14 Impurities (due to starting material):**

The manufacturer provides spectrographic analyses for the spectrographic carbon used as the raw material for arc-evaporated carbon foils. This material has a maximum impurity level of 5 ppm ash. Typically the analyses indicate metallic impurities at the level of 0.1 ppm by weight for Al, Fe, Ni, Si, V, Mg, 1.3 ppm by weight for B, and not detectable for Ca, Cr, Cu, Pb, Mn, Mo, Ni, Ag, Sn, Ti, W, Zn and Zr, searching for the "ultimate rays" using a d.c. arc

cathode-layer technique at 15 amperes for 30 seconds.

### **2.15 Impurities (due to parting agent):**

Parting agents used by ACF-Metals are proprietary, in some cases applied by hand or brush, then polished with a clean cloth until only a thin, almost invisible layer remains. Different parting agents are used depending on the intended type and thickness of foil. The following nonvolatile

ingredients are typical: Lysine, betaine, lysine hydrochloride, sodium carbonate, sodium dodecylbenzene sulfonate, sodium lauryl sulfate, sodium hydroxide, sodium palmitate, sucrose. Beginning in year 2015, no elements other than H, C, O and Na are present in the parting agents.

Sodium is the principal metallic impurity seen by Rutherford backscattering analysis in ACF-Metals' carbon foils, at the level of 1000 ppm for a foil having carbon surface density of 20  $\mu\text{g}/\text{cm}^2$ . 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 has been found at typically 1 atomic percent, and hydrogen has been found 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 temperatures higher than 3000 K.

Foils with fewer impurities have been provided by arc-evaporating onto freshly cleaved mica substrates, without parting agent. However, carbon foils are in contact with laboratory air when removed from the coater, and in contact with water as they are removed from their substrates, so adsorbed hydrogen and oxygen are expected to be present on both sides of a foil. The minimum scattering areal density for evaporated carbon foils is ordinarily about 1.5  $\mu\text{g}/\text{cm}^2$  [Ritzau & Baragiola (1998); see also Stoner (1969)] Part of this non-carbon component is presumably tightly bonded oxygen and hydrogen atoms. Mounted carbon foils can be made thinner (and much more fragile!) than this limit by starting with conventional foils and thinning them using an oxygen plasma etch, down to about 0.8  $\mu\text{g}/\text{cm}^2$ .

### **2.16 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 and later. 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 [Kocur *et al.* (1983), Weathers *et al.* (1991)]. Other sources of information on impurities in carbon foils include [Balzer and Bonani (1979), Raith *et al.* (1977), Campbell (1977), Herman *et al.* (1973), Kaji *et al.* (1977), Sofield *et al.* (1982), Johansson *et al.* (1970), Stoner (1969), Both *et al.* (1987),

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Johansson and Johansson (1984)].

Note that impurities in foils and their mountings that are exposed to intense particle beams can be significant sources of radioactivity; see section 3.4 below.

## **2.17 Baking, breaking, cutting and floating foils -**

### **Introduction:**

THESE TECHNIQUES DEPEND ON THE AREAL DENSITY OF THE FOIL. PLEASE USE A TECHNIQUE APPROPRIATE TO YOUR FOIL!

The stresses in freshly evaporated carbon foils are observed to relax in times of the order of a few weeks; these processes are greatly accelerated by annealing at 200-260 °C. Arc-evaporated foils expand irreversibly at temperatures greater than 800 K [Dollinger and Maier-Komor (1989); Miller *et al.* (2008)]. (See section 2.12 above.) They graphitize at elevated temperatures, typically 1300-2400 K, further changing their properties. Graphitization of foils has also been described by [Fishback (1971)]. All of these changes may damage mounted foils. On the other hand, controlled graphitization of mounted carbon foils has been proposed [Buijsse *et al.* (2015)] as a method of improving their qualities when they are used as supports for transmission electron microscopy (TEM).

A foil that has been cut to size often has nearly invisible microcracks at its cut edges. These can serve as failure points if the foil is bent or otherwise stressed, either during mounting or in use. A foil typically lasts longer in use (a) if microcracks are reduced by avoiding unnecessary cutting, (b) if stress is minimized by using only a few small areas to which adhesive is applied, and (c) by providing for stress relief of the foil during use. Examples can be seen for foils that are mounted with the use of fibers, or in cages [Jolivet and Stoner (2008), Stoner *et al.* (2008), AAPS (2009), Marti *et al.* (2010), Jaggi *et al.* (2015)].

### **2.18 Avoiding foil breakage:**

Carbon foils break in use due to a variety of mechanisms: random atomic displacements, graphitization, thermal stresses, shock waves, sublimation, among other causes [Schlitt *et al.* (2013)]. Simulations of processes leading to foil breakage by high-current uranium ion beams have been carried out by [Tahir *et al.* (2014)].

Methods of avoiding breakage of foils due to mistakes have been described [Jolivet & Stoner (2006 and 2008)]. Some causes of foils' breaking prematurely are: Touching a foil with bare hands, which leaves fingerprints and tears. Careless 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 by frame, non-planar frame, strong air currents, dirty or rough frame, mounting the foil so tightly that differing thermal expansion coefficients cause foils to bulge or split, failure to allow for foil's shrinkage or bulging during use. Careless use, vibration, electrostatic forces, impact, vacuum accidents, incautious pumpdown or venting to atmosphere, sudden application of strong beam, allowing direct or scattered beam to hit frame [cf. Boyd (2011), Cousineau *et al.* (2011), Plum *et al.* (2011)], sharp beam focus causing foil overheating with resulting rapid evaporation and/or concentrated local nuclear damage.

## **2.19 Baking and cutting thin foils (areal density < 100 $\mu\text{g}/\text{cm}^2$ ):**

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.

Occasionally, foils in the areal density range 40-99  $\mu\text{g}/\text{cm}^2$  may need to be baked before use, in order to reduce their internal stresses if they crack and/or curl and sink when floated. [Foils supplied by ACF-Metals after January 1, 2003, are usually annealed if their areal densities are greater than 50  $\mu\text{g}/\text{cm}^2$ .] To bake, preferably use a vacuum oven, or preheat a nitrogen-flushed oven to at least 200 degrees Celsius [We use 260 degrees Celsius; 160 °C is too low.]. Then lay the slide horizontally, gently, foil-side down on a rack, 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 much above 260 °C in air, the foil may get much thinner or disappear due to oxidation.

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 stray 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. 6a 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. Thin foils are permeable; atmospheric water vapor penetrates the foil to reach the parting agent. 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). As an alternative, lay a piece of household aluminum foil on top of the slides in a box, lay a wet piece of paper on top of the aluminum foil, and close the box for an hour or so.

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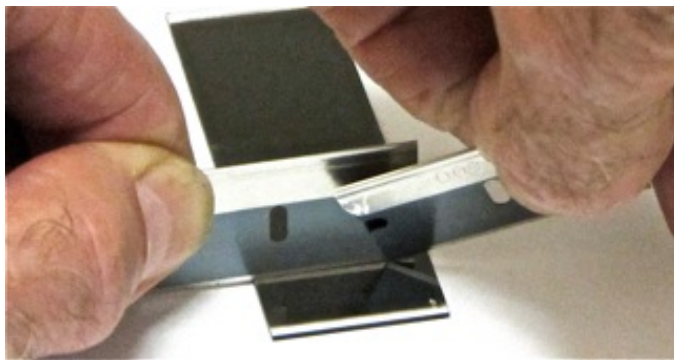


Fig. 6a. Cutting a foil on its glass substrate, using a single razor blade. The straightedge is supported above the foil but not touching the foil, and both straightedge and foil are clamped or taped in place for rigidity.



If the foil has started to release from its substrate, do **not** use a single blade to cut it on its slide; the foil may splinter where it is cut. Either use a double-blade method, in which one blade reduces the spread of damage due to cutting (Fig. 6b below), or use a method described in Sections 2.20 and 2.21 below. Float it and pick it up in the usual manner (see below).

Fig. 6b. Cutting a foil on its glass substrate, using one blade to guide the cut and minimize the spreading of cracks into the foil, and the other blade to cut the foil to size.



## **2.20 Cutting intermediate-thickness foils (areal densities in the range 100-179 $\mu\text{g}/\text{cm}^2$ ):**

These foils are usually still on their glass substrates, but they have been annealed, so may 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, then 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, if necessary cut the foil on the water surface, using clean **sharp** scissors, keeping the foil horizontal. Avoid bringing the tips of the scissors together within the foil.

## **2.21 Cutting thick foils (areal densities of 180 $\mu\text{g}/\text{cm}^2$ and greater):**

These foils are provided already annealed and released from their substrates, so baking and floating are 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. [Foils having areal densities down to 100  $\mu\text{g}/\text{cm}^2$  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 **sharp** razor blade, make several gentle cuts along that edge until the foil is cut through. Or, two razor blades may be used, one to hold the foil against the glass plate, the other to do the cutting (Fig. 6b). A single heavy cut or a dull blade is likely to cause the foil to splinter. Pieces can be handled gently with tweezers, and glued or clamped to frames.

An alternative method of cutting was developed by C. Jolivet: Lay the foil on a piece of weighing paper on a dry glass plate. Lay the edge of a razor blade on the foil and press down, gently at first, then harder as the blade is slowly tilted about its edge as an axis of rotation. The foil will usually break neatly along this edge. If the foil tends to splinter, replace the weighing paper by a drop of water. Float off the foil pieces after cutting is complete.

More-elegant methods of cutting involve the use of electron

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beams [Brumwell (2001); Fischbein and Drndic (2008)]. When cutting must be avoided, foils can be prepared to final dimensions by evaporating them through masks.

## **2.22 Floating foils off of substrates:**

ACF-Metals uses clean (distilled or deionized) water at room temperature (near 25 °C) for all foils except those thinner than about 3 µg/sq.cm. For the thinnest foils it is desirable to use hot water, often 50-60 °C. Some users add ethanol or isopropanol up to 20% by volume (as limited by foils' sinking) [J. Baldonado (1998)], or other materials to reduce surface tension, but we do not ordinarily use these materials. Sometimes the thinnest foils will only release well on water for which the temperature is > 90 °C.

Foils will pick up impurities from the water used to float them [see Raith *et al.* (1977) and other references in section 2.16 above], so care must be taken to minimize impurities in water used for floating. Stray water droplets drying on carbon foils can cause localized stresses and resultant breakage of very thin foils [cf. Chen *et al.* (2009)].

Four methods (#1-4 below) are popularly used. Special methods are used for foils on mica (methods #7):

Method #1: The "droplet" method is carried out by laying the foil (on its substrate) on the horizontal bottom of a shallow dish, then using a dropper to put tiny drops of water on the edges and/or on the cut lines on the foils (Fig. 7, next page). 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 on its slide 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.

Method #2: Use a mechanical arm, forceps, hemostat, or your own steady hand. Hold the foil's substrate, foil side uppermost, at about 45 degrees to the horizontal. Lower the foil on its substrate slowly into a dish of water. See Fig. 8a. When the water level reaches a foil being floated by immersion, the foil will begin to float on the water surface.

Fig. 7. Droplet method for floating a foil off of a glass, mica, or metallic substrate. The foil is first cut into pieces of the desired size and shape, and a medicine dropper or similar tool is used to place small droplets of water on the intersections of the cut lines.

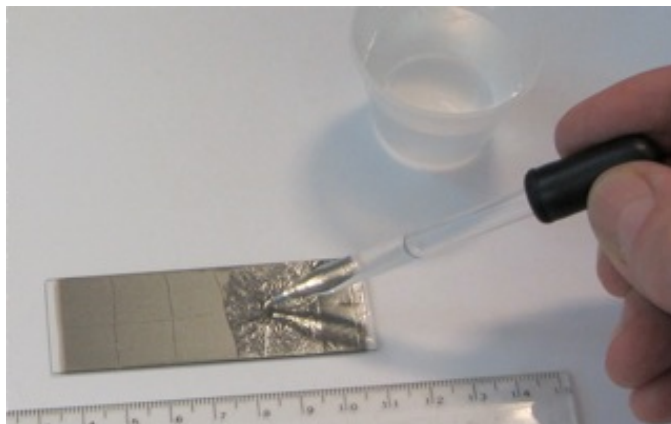
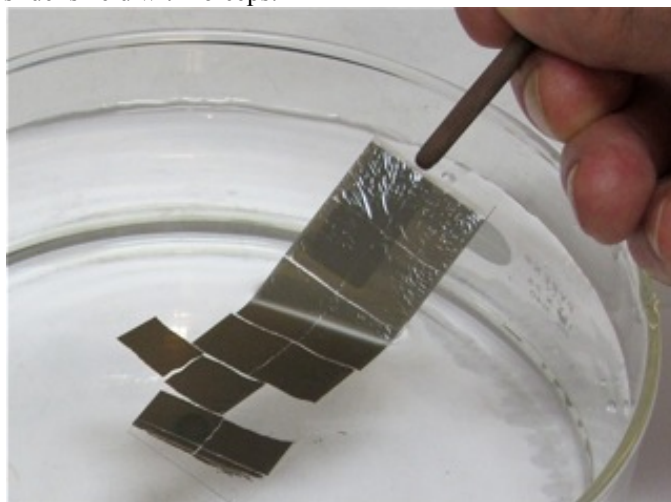


Fig. 8a. Floating a foil by immersing its substrate by hand, into a water bath. The foil in this photo is on a glass slide; the slide is held with forceps:



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

Method #4: The smoothest method for particularly fragile or large foils, is to put the foil (often mounted at a 45° tilt) and the frame on which the foil is to be mounted (supported vertically), in a large tub. A glass aquarium is convenient and inexpensive. The top of the foil and the top of the frame are located below the expected high-water level. Siphon water

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from an auxiliary container into the tub. This method was shown pictorially in [Stoner and Bashkin (1978)], was used for aluminum neutralizer foils 910 mm diameter [Stoner (1989)] and is illustrated below (Figure 8b-8f).

Fig. 8b. Apparatus for floatoff by siphoning. The foil is first cut to shape on its substrate, and is then supported at about 45° tilt in the lower aquarium (lower right side). Two foil frames (barely visible) are held vertically by clamps, near the foil. A small pump, not shown, transfers an initial batch of distilled water from a nearby bottle to the upper aquarium. Flow rate from the upper to the lower aquarium is controlled with a pinch clamp.

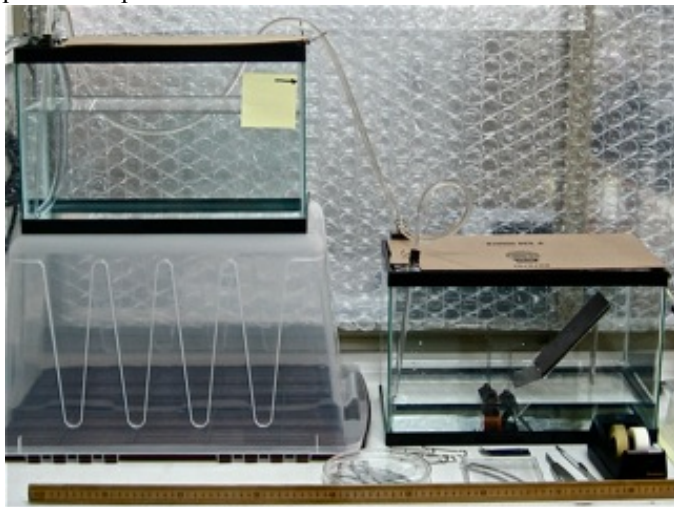


Fig. 8c. A cardboard mask is used to guide a scalpel blade for cutting the desired foil pieces. The foil is then placed on its support in the lower aquarium.



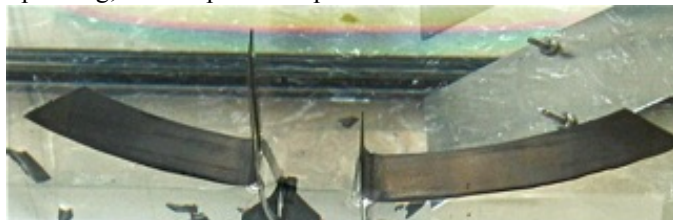
Fig. 8d. Water is added at a controlled slow rate to the lower aquarium until the foil is floating freely. Pieces of foil may be moved on the water surface by gently blowing on them, or by gently nudging them with a clean dissecting needle. Tare pieces of the floating foil are then carefully removed, and the desired pieces are steered over the frames:



If the source container has smaller volume than the recipient tub, it will not overflow the tub if your attention becomes diverted. It will still be necessary to watch the process and prevent the floating foil from drifting to a wall to which it might unexpectedly attach. If the substrate of the foil being gloated is glued into an open 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. After the foil is floating freely, and water covers the frame, the foil can be gently steered above the frame, and the water in the tub siphoned out, lowering the foil onto the frame.

Note that for any of these techniques, if the floating process is interrupted, and the partially released foil is allowed to re-attach itself to its substrate, and/or if the parting agent under the unreleased part of the foil is recrystallized by the high humidity near the water surface and then dried, the foil may not release later.

Fig. 8e: Water is then removed from the lower aquarium by siphoning; the foil pieces drape over the frames:



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Fig. 8f: When dry, the foils on their frames are ready to be used:



Methods #2, #3, and #4 require the foil to flex only in one direction, important for minimizing cracks. 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.

**Methods described below, #7(i) and 7(ii) for floating and mounting carbon foils that have been floated off of their glass substrates, and remounted onto mica sheets for storage.** Each foil on its mica sheet is packaged in a tube or clear plastic box. The mica sheet is already taped using Scotch Magic Mend tape (#810) to the bottom if a box is used.

DO NOT try to peel off the tape. The mica may be delaminated, or may bend, if you try to peel off the tape. The foil may be destroyed as a result.

Do not cut the foil while it is on the mica sheet. The foil will almost certainly be damaged if you try to do so.

**Method #7(i) :** Leave the mica sheet in its box. Open the box; lay it on a horizontal surface. Use the "droplet method" of floating the foil. Use a medicine dropper or a small pipet (pipette), and put a few small drops of distilled water on the mica in contact with one end of the foil. The water will creep under the foil. Add water a drop at a time as it does this. The entire foil will gradually separate from the mica. When separation is complete, add water more rapidly to box until the foil is floating in it. Lower the box into a spacious bath of distilled water and transfer the foil onto the surface of the bath. Remove the box. NOW cut the foil into pieces (if desired) using a pair of SHARP CLEAN scissors. [Wiss® scissors are nice for this.]. Don't lift the foil out of the water, or submerge it, or allow the points of the scissors to come together within the foil, as you cut it. Now the pieces of foil can be picked up on your frames. Use an adhesive if appropriate.

**Method #7(ii):** Open the box and lay it on a horizontal surface. Cut the tape along the two edges of the mica sheet. Lift out the mica sheet WITHOUT BENDING THE MICA. Lay it on a slightly larger piece of glass. Proceed with floatoff as above, or by your other favorite method. Use the glass sheet to prevent surface-tension forces from bending the mica if you dip it into a water bath. Cut the foil into pieces and pick them up as described above.

Foils are **guaranteed** to float off of their substrates with one of these techniques. 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 substrate, remove the substrate 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 up with a pipette or dropper tip, or by allowing them to flow into a small upright empty beaker that you first immerse gently in the water almost to its rim. This will prevent 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, toothpick or dropper tip moistened in methanol [Lozowski (1990)] or ethanol or isopropanol. In fact, it is often not necessary to touch the boundary; alcohol vapor evaporating from the moistened tool and condensing into the water on which the foil pieces are floating is sometimes sufficient to cause them to separate.

### **2.23 Pickup of foils onto frames and meshes:**

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.

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Experts can often do somewhat better. Please be aware, however, that even though carbon foils can be mounted with unsupported diameters of greater than 10 cm, the shipping of such foils without breakage depends crucially on the diameter, areal density, mounting method, and packaging method. An approximate size limit for shipping mounted unsupported  $50 \mu\text{g}/\text{cm}^2$  foils is 50 mm unsupported diameter, the size for which 25-50% breakage can be expected. Carbon foils can be mounted with one or more free (unsupported) edges (see Fig. 5a above), or with holes within the mounted area, or with reinforcement of meshes or fibers, using specialized techniques not described here. Examples of such foils are described in the section on stripper foils, below.

Frames on which foils are to be picked up should be planar, stiff, thin, clean, hydrophilic and without burrs. [Mounting foils on exteriors or interiors of cylindrical or dome-shaped surfaces is much more difficult.] 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, titanium, stainless steel or brass frames. Metals are preferably either lightly sanded under water, or acid-etched, to produce a fresh hydrophilic surface, and then kept under water until they are used. [Precoating a frame with a thin layer of epoxy (e.g. Tra-Bond 2151) and allowing the epoxy to cure partially before picking up a foil, provides secure attachment of the foil to the frame for long-term storage.] 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 (Figures 9a, 9b, 9c).

Fig. 9a. Typical pickup sequence for foil onto washer used as a frame. A piece of foil is sequestered, preferably near the edge of the water surface.



Fig. 9b. The frame is pressed against the piece of foil so that the foil drapes over the aperture to be covered, and then is raised slowly. The foil usually adheres to the exposed surface of the frame.



Fig. 9c. When lifted out of the water bath, the foil adheres to the entire perimeter of the washer (frame):



Covering a large aperture usually requires the plane of 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.

Electroformed meshes are sometimes used as supports for ultra-thin foils. Some techniques for mounting foils on meshes are described by Hunter (1973) and Steele (1976). [See also section 1.1b above.] Meshes, originally available from Buckbee Mears Co., are more recently available from Precision Eforming Co. (see references below). Care must be taken to avoid broken, stretched, deformed or other defective regions in meshes, and to pick up foils on the shiny side, not the matte (rough) side of meshes. Foils that are picked up onto meshes sometimes adhere poorly, so that vibration or air currents may damage the foils. Metal meshes (already mounted on frames) may be collodion-coated by dipping them into 15/85 v/v flexible collodion in amyl alcohol, and blowing the liquid out of the holes in the mesh before it dries, using compressed air. If properly done, a thin layer of collodion then remains on the mesh bars. The coating can be visualized by viewing the mesh in specularly reflected light; colored fringes indicate a coating thickness of a few microns. A carbon foil picked up on such a mesh adheres more reliably

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than one picked up on bare metal. Adhesion can be made still more reliable by then placing the assembly in saturated amyl acetate vapor for a few minutes.

Meshes that are already mounted on frames can also be coated with a thin layer of epoxy by the method of Steele (1976), utilizing a vaporizer. As a quicker alternative, a mesh can be painted on both sides with a 50:50 v/v mixture of freshly mixed epoxy [Duro (1992)] in 100% ethanol, using a soft brush. The mesh is then blotted on both sides with Whatman #1 filter paper. It is more difficult to obtain a uniform coating with this method.

When foils are dry, they are ready to use. Although they are fragile, and easily broken by vibration, impact or strong air currents, thin carbon foils can often be stored for years in a protected environment.

#### **2.24 Adhesives and clamping:**

Carbon foils that have areal densities greater than about  $20 \mu\text{g}/\text{cm}^2$  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. Our current favorite epoxies are Henkel/Hysol Tra-Bond 2151 and Master-Bond EP42HT. Each adhesive has its own area of utility [Jolivet & Stoner (2006) and (2008)]. Frames can also be coated with a thin layer of Apiezon L, Krytox LVP 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 [Greene (2003); Stoner & Miller (2006)] [Grease will not indefinitely prevent a thick foil from curling.]. Customer should be aware that the abovementioned vacuum greases and many epoxies creep visibly onto carbon foils, at room temperature, over distances of several mm in times of the order of 40 hours. Vacuum greases typically have vapor pressures of the order of  $10^{-4}$  to  $10^{-5}$  torr at temperatures of  $200^\circ\text{C}$ , so usually cannot be used at elevated temperatures.

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. For an example, see fork type ACF-F25 (Section 3 below).

#### **2.25. Lifetimes of carbon foils in particle beams:**

For more than 35 years, investigators have been trying to maximize the time that foils will survive in particle beams. Long survival times are desirable for several reasons: Data streams are interrupted by foil breakage. Signal-to-noise ratios are increased if larger beam currents can be used. Accelerator downtime is reduced when stripper foil lifetimes

are increased. The exposure of supporting personnel to radiation is reduced through replacement of foils less often. Production of isotopes for commercial purposes becomes more profitable if not interrupted by foil breakage.

Early methods for improving lifetimes of foils utilized slackening of mounted foils to permit limited deformability without breaking. A mounted foil's frame may be compressed radially to slacken a foil mounted on it [Tolfree (1985)]. Blowing on the foil judiciously, immediately after pickup will slacken it. Use of thick support frames, for which a water fillet pulls the foil slightly into the hole in the frame, accomplishes the same objective. Using a parting agent that dries with a microscopically rough surface [Ophel *et al.* 1998]] lengthens in-beam lifetimes, presumably because of the slight slackening. The temperature of a foil may be raised above room temperature to increase its lifetime, and/or its surroundings may be cooled to reduce the thickening that occurs if condensable vapors are deposited and decomposed on the foil's surface. Another method for extending a foil's lifetime is to utilize a large target foil or wheel, rapidly moving the foil to expose new areas to bombardment [cf. Folger *et al.* (1989), Bokemeyer and Folger (1989), Greene *et al.* (2001), Okuno *et al.* (2009), Marti *et al.* (2010), Tahir *et al.* (2012)].

Alternative treatments - laser annealing, electron bombardment, and "flashing" foils using flashbulbs or flash lamps, have been used to anneal, thin and slacken them, extending their survival times in ion beams [Maier-Komor *et al.* (1982), Tolfree (1982), Rowton (1988), Rowton (1994)]. The effects of slackening and flashing, together with changes in target carbon foils due to irradiation by fast beam particles were studied by Dollinger and Maier-Komor (1989) using electron diffraction and electron transmission, in order to elucidate the damage mechanisms, and to propose methods of improving lifetimes of foils. Work using these techniques has been reported by [Okuno *et al.* (2009)]. Additional properties of foils and their stability under irradiation by fast ion beams were considered by [Koptelov *et al.* (1989)].

Direct comparison of individual experimenters' measurements of lifetimes of foils is rarely possible because particle type, particle energy, particle current, beam diameter, foil thickness, and type of carbon used, vary from situation to situation. A few representative examples of typical lifetimes for conventional arc-evaporated foils are as follows:

0.6 MeV  $\text{d}^+$ ,  $2 \mu\text{g}/\text{cm}^2$  (collodion coated), 36  $\mu\text{A}$ , 30-60 minutes [Harper (2011)].

9.0 MeV  $\text{Fe}^+$ ,  $\text{Cu}^+$ ,  $\text{Ni}^+$ ,  $2 \mu\text{g}/\text{cm}^2$ , 0.5-1.0  $\mu\text{A}$ : 30 minutes;

8.5 MeV  $\text{d}^+$ ,  $2 \mu\text{g}/\text{cm}^2$ , 10  $\mu\text{A}$ : several hours;

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18 MeV  $\text{H}^-$ , 50  $\mu\text{A}$ , 75  $\mu\text{g}/\text{cm}^2$ , 2000 to 3000  $\mu\text{A}$ -hours [various authors].

30 MeV  $\text{H}^-$ , 400  $\mu\text{g}/\text{sq.cm}$ , 6-30 mA-hours, 6.2 mm beam spot diameter [Harris (2014)].

2.7 GeV  $^{238}\text{U}^{38+}$  20  $\mu\text{g}/\text{cm}^2$  11 h, 30 mm foil diameter, 6 emA pulses, 1 Hz, 100  $\mu\text{s}$  [Barth *et al.* (2010)].

We note that Korenev *et al.* (2016) claimed extended lifetimes for graphene foils used for stripping  $\text{H}^-$  in an 11 MeV cyclotron. Their results require confirmation.

Experimental studies have shown that foils having diamond-like character, foils made by complex arc and/or laser evaporation methods, multilayer foils, foils having isotropic crystal structures, and foils with controlled amounts of boron added to them, can sometimes have greatly extended lifetimes in accelerator beams. For example, a pyrolytic graphite foil described in [TRIUMF (2010)] survived without observable damage for 17 weeks at 500 MeV, nominally 90  $\mu\text{A}$ , about 5 times as long as a conventional foil. An overview of progress in the topic of long-lived target foils was published by Sugai *et al.* (2000). Improvements are often accompanied by greater complexity and lower efficiency in production, resulting in higher costs and limited availability. Nevertheless, work continues to develop, evaluate and use such foils [c.f. Frazier (2005?), Muto and Monroy (2005), Jaggi *et al.* (2006), Maier-Komor *et al.* (2006), Zeisler and Jaggi (2008), Spickermann *et al.* (2008a), Spickermann *et al.* (2008b), Sugai *et al.* (2006, 2008a, 2008b, with updates by Sugai and Takeda (2010), E. Baron (1979), Sugai *et al.* (2010), Takagi *et al.* (2010), Muirhead and Heighway (2011), Stoner (2012), Okuno *et al.* (2014), Kupka *et al.* (2014)], Jaggi *et al.* (2014), Jaggi *et al.* (2015). Surveys of the 2013 status of the field of charge-state strippers (by M.A. McMahan, section 7.1.7.1) and the field of stripper foils for  $\text{H}^-$  beams (by M.A. Plum, section 7.1.7.2) appear in [Wu Chao *et al.* (2013)]. The latest reported comparisons of different types of foils appear to be those of Bracco *et al.* (2017).

When a high-energy particle traverses a carbon foil, the track (thermal spike) that is produced may momentarily have a high temperature, resulting in localized melting, rapid quenching, and recrystallization. Simulations of some of these processes, the phases that appear, and attribution of some of the resulting structures to processes on the phase diagram for carbon (see Fig. 9d), have been analyzed by Sorkin (2003). Foils made of carbon nanotubes appear to be intolerant of bombardment by the heaviest ions [as are most foils (!); see von Reden *et al.* (2007)].

On the basis of successful work in other fields to improve the radiation tolerance of nanocomposites, it can be expected that further theoretical and experimental work in multicomponent foils will further improve foils' lifetimes [cf. Demkowicz *et al.* (2010)]. For the highest fluences of the heaviest ions, gas and liquid strippers often replace solid foils [Reed and Kondrashev (2009), Silverman (2009), Okuno *et al.* (2009), Marti *et al.* (2010), Kuboki *et al.* (2010), Okuno *et al.* (2011), Imao *et al.* (2012), Imao *et al.* (2014), Scharrer *et al.* (2015), Paul *et al.* (2015), Hasebe *et al.* (2015), Momozaki *et al.* (2015)]. Gas strippers typically operate in steady-state and produce charge states that are lower than for foils, but this can be partially overcome by appropriate design [Okuno *et al.* (2011)] or by pulsing a gas stripper jet [Barth *et al.*, (2017)]. Laser stripping has also been proposed for high-power beams [Galambos (2011)].

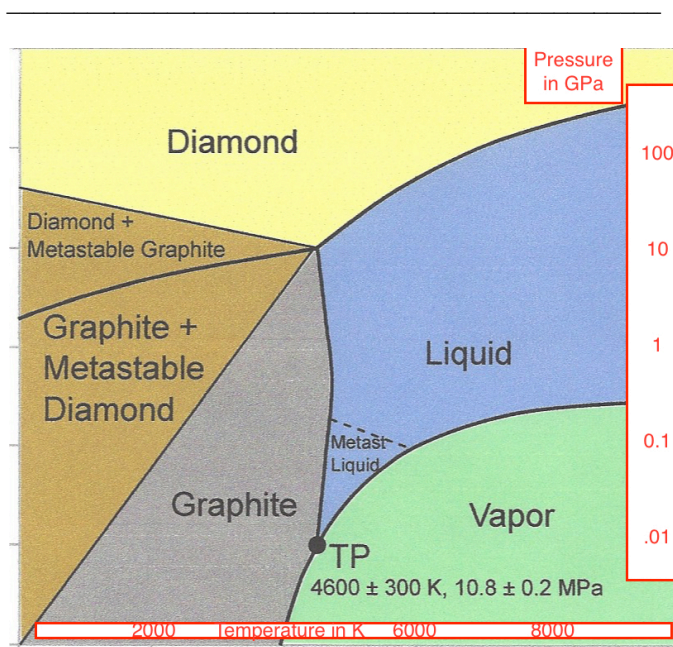


Figure 9d: Theoretical carbon phase diagram from [Wikimedia (2017)]. Note that this reference claims that considerable disagreement exists between theory and experiment.

Analytical and/or phenomenological methods of estimating foil lifetimes of arc-evaporated carbon and cracked-ethylene foils appear in the physics literature from time to time. Analysis of the energy dependence of several parameters affecting foil lifetime was carried out by Dollinger and Maier-Komor (1989). [Nickel (1982)] has pointed out that better predictive accuracy of foils' lifetimes is possible when effects of the temperature of the foil are included.

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Simple formulae were provided by [Baron (1979); Auble and Galbraith (1982); Nickel (1982)] among others. The formula given by Auble and Galbraith for the lifetime =  $T$  of an evaporated carbon foil can be written as

$$(T)(F) = \text{Foil lifetime} \times \text{beam fluence} = [k(E/\text{eV})/(Z^2M)]$$

where  $k$  is  $0.0018 (\mu\text{A-minutes}/\text{mm}^2)$  [Note that this number was mistakenly quoted as 0.018 by McMahan (2013)].

$F$  is the beam fluence [i.e. particle current ( $\mu\text{A}$ )/area]  
 $E$  = kinetic energy of the incident particles,  
 $Z$  = atomic number for the incident particles, and  
 $M$  = mass number for the incident particles.

This formula, which does not consider effects due to the temperature of the foil, provides surprisingly accurate estimates of many in-beam foil lifetimes with the use of heavy ions. Unfortunately, its accuracy is poor when applied to foils' stripping beams of hydrogen ions.

### **3 CARBON STRIPPER FOILS MOUNTED ON FORKS**

#### **3.0 Introduction; PET analyses and carbon stripper foils:**

A medical PET (positron emission tomography) scan requires positron-emitting radioactive nuclei to be injected into the patient; specific types of cameras can then measure the location(s) within the patient where the radioactive decays occur later. Nuclear isotopes having appropriate chemical forms are chosen for applicability to image specific types of cancers and/or lesions. A review by [Schmor (2010)] describes the principal cyclotrons used for producing radioisotopes for biomedical applications, and lists the isotopes commonly produced for these purposes. Many PET and combined PET/CT scans use radiopharmaceuticals that are produced in negative-ion cyclotrons. These machines create beams of negative hydrogen ions ( $\text{H}^-$ ), accelerated to high energy; carbon foils, known as stripper foils or extractor foils, are then used to remove the electrons, so that the beam may be steered out of the cyclotron. Beams of high-energy protons, typically in the energy range 8-30 MeV, are produced in such machines, and are used for making the  $^{11}\text{C}$ ,  $^{13}\text{N}$ ,  $^{15}\text{O}$  or  $^{18}\text{F}$  often used for these purposes.

A summary of popular chemical forms for the most common PET isotopes,  $^{11}\text{C}$ ,  $^{13}\text{N}$ ,  $^{15}\text{O}$  and  $^{18}\text{F}$ , produced in small accelerators, and medical conditions for which each has been used can be found in [Scott and Hockley (2012)]. The most

popular chemical form used currently appears to be ( $^{18}\text{F}$ ) fluorodeoxyglucose (FDG).

It has been found recently that antibodies and other biological entities can be labeled with radioisotopes and used to locate specific cancer cells (by "immuno-PET") that cannot be found with simpler chemical tracking [Mestel (2017)]. The radioactive nuclide chosen for each application must decay by positron emission and have appropriate lifetime and chemical properties. A review of the most popular unconventional nuclides being applied to PET has been provided by [Holland *et al.* (2010)], and numerous applications of immuno-PET have been described by Bailly *et al.* (2017).

The half-lives of isotopes used for PET are usually in the range 2 minutes to 2 hours. Each nuclear decay at a cancer site releases a positron that annihilates with a nearby electron, producing a pair of 511-keV x-ray photons traveling in opposite directions. An array of x-ray cameras external to the patient detects both members of the pair, and the line connecting the two detection events fixes a line through the cancer site. A single analysis combines many such lines to locate the cancer sites. [Because of the increased risk of cancer due to the use of radiopharmaceuticals, magnetic-resonance imaging (MRI) is sometimes used as an alternative to PET and PET/CT when possible; see Kwee (2014), Lee and Levy (2012)].

A brief introduction to this field is provided by J. Alonso (2013). Carbon is usually the element of choice for the stripper foil because of its strength, low toxicity and low  $Z$  (atomic number  $Z = 6$ ). Other materials are useful for specialized purposes [c.f. Gulbekyan *et al.* (2009)].

Cyclotrons' beam currents are typically  $50 \mu\text{A}$  for PET radioisotope production and typical irradiation times are 30 minutes, resulting in production of typically 1.5 Ci activity [56 gigabecquerels (GBq)]. [Scott&Hockley (2012)]. A typical medical radioactive dose provided to a patient for PET has an initial activity of 370 megabecquerels (MBq), or  $3.7 \times 10^8$  decays per second, yielding a total radiation dose of the order of 2 to 9 millisieverts (mSv), or higher for some organs [Huang *et al.* (2009); RSNA (2017)]. This may be compared to the approximately 3 mSv per year average radioactive dose received by each person due to background radiation and cosmic rays.

Worldwide, according to [NuPECC (2014)], more than 30M (30 million) nuclear-based medical procedures are performed per year, largely using radioisotopes. In year 2010, about 1.74 million PET (positron emission tomography) and CT (computed tomographic) medical diagnostic scans were done in the U.S.A., involving more than 2000 hospital and non-hospital sites [Muschlitz (2011), Ward (2011), IMV (2017)].

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A typical cost to the patient is \$5000-\$7000 per scan. We estimate that roughly 4M such procedures are carried out worldwide per year.

WTTC, Workshops on Targets and Target Chemistry, have international meetings on a regular basis to report progress in production methods for medical radioisotopes; their most recent meeting is described at [<http://wttc16.us>]. A comprehensive report on Nuclear Physics for Medicine was published recently [NuPECC (2014)]. Procedures used to make radiopharmaceuticals for PET have been compiled by [Scott and Hockley(2012)]. Two reports published by the International Atomic Energy Agency have summarized the principles and practices of producing radionuclides by cyclotron methods [IAEA (2008)] and the detailed properties of those radionuclides [IAEA (2009)]. In year 2013 there were about 875 operational cyclotrons worldwide, with about 790 of these operating at 20 MV and below, i.e., capable of producing isotopes used for PET (NuPECC, p.121).

For the operation of each negative-ion cyclotron to make PET radioisotopes, at least one foil costing a few dollars is absolutely necessary. Because our customers and their medical patients depend on the availability of such foils, ACF-Metals attempts to maintain a stock of stripper foils of all the usual thicknesses at all times. Foils' sizes range from 4 mm x 12 mm to at least 17 mm x 17 mm, and thicknesses range from 0.25  $\mu\text{m}$  (50  $\mu\text{g}/\text{sq.cm}$ ) to at least 50  $\mu\text{m}$  (10 000  $\mu\text{g}/\text{sq.cm}$ ) depending on beam particle energies.

For the highest efficiency of production of a well-characterized proton beam, it is desirable to use a stripper foil whose thickness is sufficient to strip close to 100% of the incident  $\text{H}^-$  ions to protons. Methods for estimating the dynamic equilibrium charge state of a fast particle that is penetrating a foil can be found in [Sigmund (2014)]. The theory typically involves rate equations for the creation and destruction of the various ionic species involved. The advantages of high stripping efficiency in a thick foil must be balanced against the beam deterioration resulting from scattering in the foil [cf. Goddard *et al.* (2010)]. On the other hand, stripping foils are usually mounted with at least one free (unsupported) edge having length 10 mm to 20 mm, so that the particle beam need not pass through a massive support as the foil is inserted. The foil must be thick enough to be strong enough to be mounted with such a long free edge.

K. Shima *et al.* (1999) have compared experimental and theoretical carbon foils' areal densities needed to completely strip incident  $\text{H}^-$  ions having various incident energies up to 10 MeV, and Goddard *et al.* (2010) have suggested the necessary carbon foil's areal density for complete stripping at incident energy of 160 MeV, with the results:

Particle energy (MeV)	Required areal density ( $\mu\text{g}/\text{sq.cm}$ )
1	1.5
4	4
10	10
160	150

From these limited data, it is difficult to avoid the conclusion that for incident  $\text{H}^-$  particles at energy X(MeV), the optimum foil's areal density, considering only stripping efficiency and minimum thickness, is approximately X( $\mu\text{g}/\text{sq.cm}$ ). In practice, however, one must consider any effect of foil's survival time as it depends on thickness, and the practical difficulties of mounting and handling foils thinner than about 50  $\mu\text{g}/\text{sq.cm}$ , in optimizing the choice of foils.

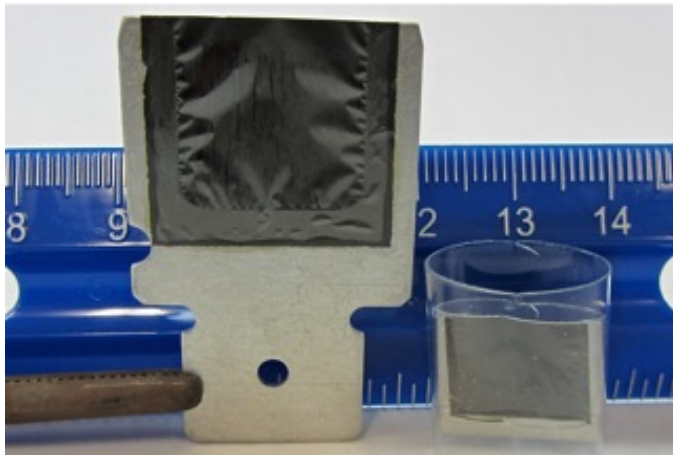
Stripping ions in sufficiently intense particle beams, a stationary foil may break too quickly to be useful. Then rotating targets, or laser, liquid or gas strippers can be used (see section 2.25 above).

### **3.1 Product description:**

A stripper foil is usually rectangular, preferably crack-free, and is typically mounted so that one edge is unsupported, and aligned within +/- 0.5 mm with the "tines" of a support fork (Fig. 10a below). Typical foil thicknesses range from 0.25 micron to 50 microns. Foils are sometimes mounted with two, three or four unsupported edges using ceramic or carbon fibers to support the foils [Borden *et al.* (1991), Sugai *et al.* (2006), Spickermann *et al.* (2008), Jolivet *et al.* (2008)]. If carbon foils are to be mounted on forks intended for use at elevated temperatures, the forks should be manufactured of a material having low coefficient of thermal expansion, e.g., graphite, molybdenum, tungsten, titanium or Pyrex<sup>TM</sup> [Jolivet & Stoner (2008); Minteq (2017)]. ACF-Metals is pleased to quote on producing foils on other mountings suggested by Customer.

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Fig. 10a. Carbon foils, nominally 0.38 microns thick ( $75 \mu\text{g}/\text{sq.cm}$ ) on F-30 mod. 2 cyclotron fork (left side) and on F-12 mod. 2 cyclotron fork (right side, in plastic cell).



An adhesive is ordinarily used to adhere foil to frame. Depending on the application, this is a graphite paint, or a high-temperature medical-grade epoxy, or a radiation-resistant epoxy [Jolivet *et al.* (2008)]. For mounting foils on aluminum frames or forks that do not reach temperatures above about  $200^\circ\text{C}$ , ACF-Metals has found that a high-temperature, radiation-resistant epoxy is much more reliable than the historically popular sodium silicate or cellulose nitrate adhesives. If Customer expects to use temperatures higher than  $200^\circ\text{C}$ , please contact ACF-Metals for suggestions on mounting techniques and adhesives that will avoid premature breakage of the foils. See section 2.12.4 for remarks on thermal properties of foils.

**DO NOT USE** solvent-based adhesives (Duco cement, polymethylmethacrylate glues, and such) to fasten PCG foils to frames.

### **3.2 Stripper foil types:**

The foil's areal density is determined by the application and is specified by the Customer. For the largest accelerators, extensive theoretical and experimental simulations are done to identify the best compromise among stripping efficiency, beam loss due to nuclear scattering, emittance increase of the circulating beam, thermal stability, etc.. [Aiba *et al.* (2009), Goddard *et al.* (2010), Weterings *et al.* (2014), Tahir *et al.* (2014)]. For negative-ion cyclotrons used to manufacture PET radioisotopes, typical values range from  $50 \mu\text{g}/\text{cm}^2$  to  $5000 \mu\text{g}/\text{cm}^2$ . The type of carbon used is typically evaporated

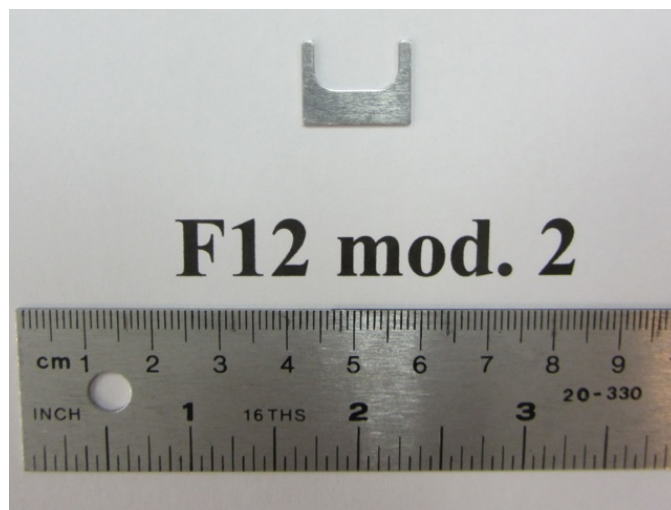
(type ACF) up to  $500 \mu\text{g}/\text{cm}^2$ , either polycrystalline graphite (PCG, see following pages) or evaporated carbon from 400 to  $1000 \mu\text{g}/\text{cm}^2$ , and PCG above  $1000 \mu\text{g}/\text{cm}^2$ . Collodion reinforcement coating to one or both sides of a foil, that (temporarily) adds typically 20 to  $40 \mu\text{g}/\text{cm}^2$  to the total areal density can be provided on evaporated foils at additional cost. Such reinforcement must be used in order to mount foils in the areal density range  $10 \mu\text{g}/\text{cm}^2$  to  $50 \mu\text{g}/\text{cm}^2$  having free edges. Some users claim that collodion-coated foils have longer in-beam lifetimes than uncoated foils. A graphite-paint or silver-paint "shorting bar" may be provided to create an additional electrical connection of foil to frame at additional cost.

### **3.3. Fork styles and photos of forks**

Prices of stripper foils, free-standing, on substrates, or on forks are presented in our price list. A few standard types of forks are kept in stock by ACF-Metals. These are ACF-Metals' types ACF-F30, mod.2, ACF-F12 mod.2., ACF-F19, ACF-11 and ACF-F25a,b., shown on the next two pages, Figs 10b, 10c, 10d, 10e and 10f. A sample and/or a specification sheet with a dimensioned drawing for any type is available upon request. ACF-Metals can also quote on mounting foils on other specialized frames designed by Customer.

ACF-Metals does not mount foils on Customer's forks, frames or substrates that have been exposed to accelerator beams, because of the possibility of long-lived nuclear activation.

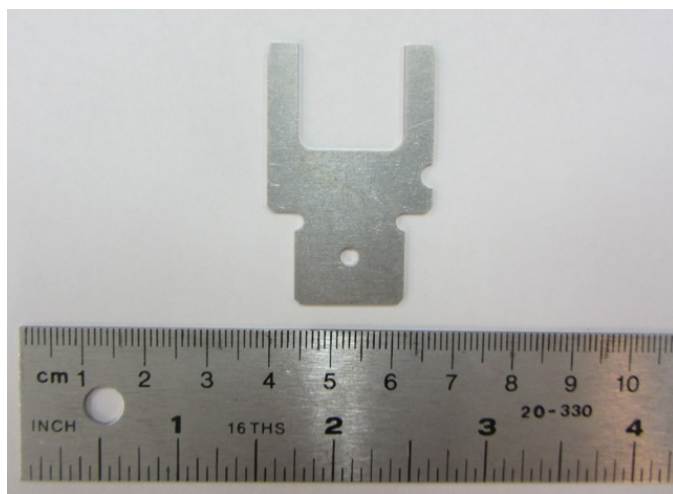
Fig. 10b: Type ACF-F12 mod. 2 fork. All forks shown on these pages are ordinarily constructed of type 6061 aluminum, or of purified graphite, approximately 1 mm thick.



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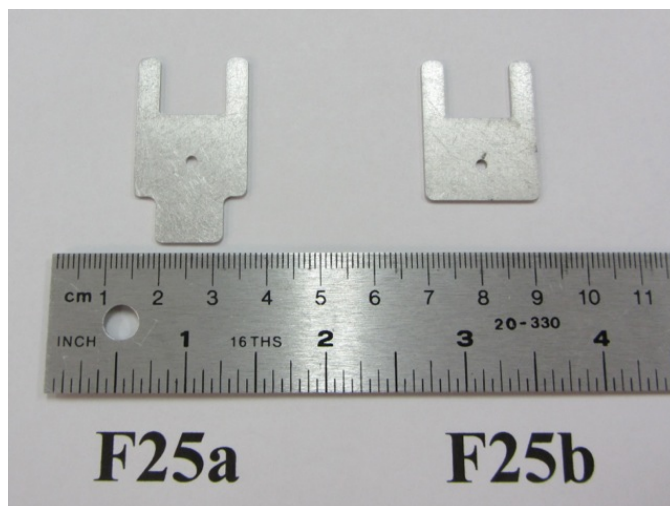
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Fig. 10c. Top view of Type ACF-F30 mod. 2 fork.



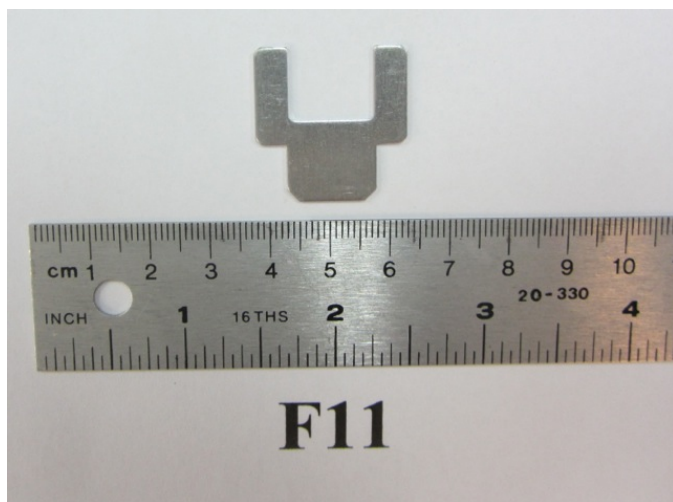
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Fig. 10e: Type F25 fork (two parts):



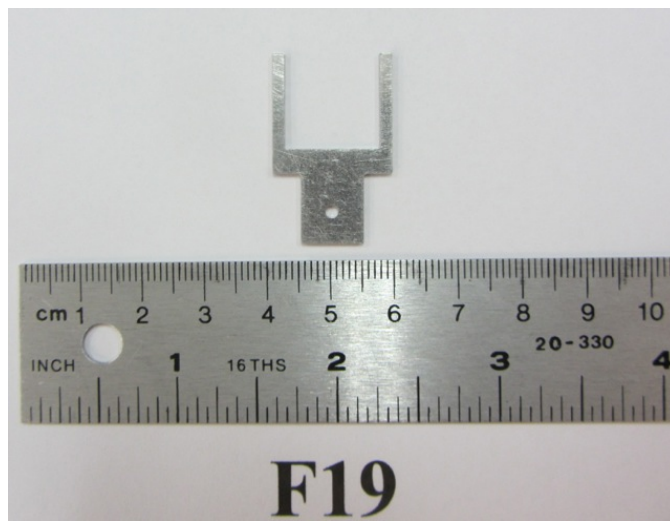
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Fig. 10d: Type ACF-F11 fork.



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Fig. 10e: Type F19 fork.



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### 3.4 Nuclear activation of cyclotrons, foils and forks:

The Coulomb barrier energy for protons incident on an atomic nucleus at rest can be estimated as the electrostatic energy of a proton that is at rest and is just in contact with that nucleus. This estimates the minimum kinetic energy needed to initiate a nuclear reaction by bombarding protons. [It ignores the possibility of excitation at lower energies via tunneling through the Coulomb barrier.] In the laboratory frame, this energy is  $U(Z_1, Z_2)$ , approximately given by

$$U(Z_1, Z_2) \text{ [in units of MeV/\AA]} = [(A_1 + A_2)/(A_1 A_2)] \\ [Z_1 Z_2 / (A_1^{1/3} + A_2^{1/3})] \\ \text{[Posocco (2008)]}.$$

Here

$A_1$  = mass number for projectile,

$Z_1$  = atomic number for projectile,

$A_2$  = mass number for target nucleus,

$Z_2$  = atomic number for target nucleus.

For incident protons,  $A_1 = Z_1 = 1$ , and the Coulomb barrier energies  $U(Z_1, Z_2)$  range from about 1.8 MeV for Li targets, 1.98 MeV for C targets, to about 12.8 MeV for U targets. The proton energies (typically 8-30 MeV) used in medical accelerators to create pharmaceutical isotopes are usually much higher, and are therefore high enough to create unwanted radioactivity by nuclear collisions in stripper foils, cyclotron forks, and other accessories within the vacuum system. Although cyclotron stripper forks are often primarily aluminum ( $^{27}\text{Al}$ ,  $Z_2 = 13$ ,  $A_2 = 27$ ), there are often alloying elements in them, both intentionally for the purpose of desired mechanical properties, and unintentionally because of impurities.

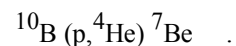
Proton irradiation of carbon foils ( $Z_2 = 6$ ,  $A_2 = 12$  or  $13$ ) creates beryllium-7 nuclei, which may remain in, may recoil out of, or may diffuse out of the foils and contaminate nearby surfaces [TRIUMF (2010)]. Sometimes accelerator operators use foils composed of mixtures of carbon and boron, to obtain longer in-beam foil lifetimes compared with pure carbon foils. Nuclear activation of the boron in such foils causes additional types of radioactivity of the foils and their surroundings, and requires additional care in their replacement by maintenance personnel.

Conventional carbon foils produced by ACF-Metals are almost entirely carbon-12, with 1.1% of carbon-13. [See page 6 above.] Both isotopes are stable. In radiopharmaceutical production, a typical carbon foil might extract 2000 or more microampere-hours of protons at particle energies of 8-15 MeV during its useful lifetime, after which it is replaced by a fresh foil. At these bombarding energies, there are few radioactive nuclei produced by proton-carbon reactions; most

radioactive nuclei produced have half-lives less than 30 minutes, and typically have decayed before a technician can open the shielding to replace the foil. In particular, the production of beryllium-7 by proton activation of carbon-12 has a threshold at 32 MeV [Dickson and Randle (1951)], and so does not ordinarily occur at typical radiopharmaceutical production facilities.

However, beginning about in year 2010, longer-lived stripper foils were developed by incorporating boron in them. With boron in the range 15-25% by weight, foils' lifetimes in beam can be extended significantly. Such foils have been made experimentally by several investigators, and are now available commercially (but not from ACF-Metals!). The boron introduces an extra problem for radiation safety in the laboratory.

Natural boron has two stable isotopes, boron-10 and boron-11. Proton excitation of the boron-11 content of stripper foils produces little long-lived radioactivity. Proton excitation of the boron-10 content produces beryllium-7, a radioactive nucleus with a half-life of 53.2 days. This isotope is well known as a persistent contamination in accelerators, and has been found in stripper foils in high-energy accelerators. The nuclear reaction at typical energies for production of beryllium-7 is



This reaction's cross section has most recently been reported by Wiescher *et al.*, (2017), and peaks near 0.1 barn (i.e.,  $1 \times 10^{-25} \text{ cm}^2$ ) in the proton energy range 1-10 MeV. Because the cross section is known, the radioactivity of a stripper foil due to beryllium-7 produced in it can be estimated. As an example, a current of 50 microamperes of protons through a 75-microgram/cm<sup>2</sup> foil that is 20% by weight of natural boron results in the production of  $5.4 \times 10^6$  beryllium-7 nuclei per second. These radioactive nuclei end up either in the foil, or on the foil's frame, or on the foil's surroundings, where they decay with a half-life of 53.2 days. The resulting radioactivity begins at zero for a fresh foil and rises toward 5.4 MBq (0.18 mCi) if the irradiation continues for several half lives.

Technicians should therefore dispose of used stripper foils as radioactive materials, if they contain boron. Minimizing this problem can be done by using ACF-Metals' carbon foils, which contain no boron. Alternatively, it would be possible to use only isotopically enriched boron-11, no boron-10, in the manufacture of foils for which boron is necessary.

The radioactivity resulting from ion bombardment of other materials can in principle be estimated from tabulations of nuclear data. A central reference for this purpose is the report by the International Atomic Energy Agency [IAEA (2001)],

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and the website <<http://www-nds.iaea.or.at/medical/>> . Perhaps the most extensive compilation of proton-induced reactions is contained in the European Activation File, for which a free DVD is available [EAF (2007)]. A program useful for finding cross sections for interactions of beams with elemental targets has recently been made available by [Doyle (2015)].

Roesler and Silari (2013) listed the contaminant nuclides of principal radiological interest that are produced by the irradiation of carbon, aluminum, iron, stainless steel and copper in accelerators. Practical aspects of decommissioning a cyclotron have been reported by Sunderland *et al.* (2012)], and more recently by Paans and de Jong (2017). The principal induced activities in concrete shielding and in accelerator components were identified and quantified by them as due to (n,gamma) activation of naturally occurring metals, some of which are found as alloying elements or impurities in aluminum alloy forks. In cyclotron components, decays of isotopes produced in Havar foils used as windows, the aluminum 6061 tank and targets, copper components, and magnet steel by various reactions were still observable two weeks after ceasing operation.

Greater challenges for controlling radioactivity are found in the stripper-target areas of very large accelerators, e.g., the rapid cycling synchrotron (RCS) of the Japan proton accelerator research project J-PARC. There the particle energies are in the range 181 MeV to 3 GeV, and sufficient numbers of particles undergo nuclear reactions in the stripping foil to produce secondary reactions in the foil frame and surroundings [Yoshimoto *et al.* (2015)]. The production of  $^7\text{Be}$  (half life 53.28 days) and  $^{22}\text{Na}$  (half life 2.62 years) in the foil and its frame are strong direct and secondary contributors to the residual radioactivity. Furthermore, impurities of Ca, Fe, Ni at typically 100 ppm levels in their foils originated in their composite electrodes used for arc evaporation; those impurities were observed to cause localized crystallization of the target foils when irradiated, with consequent deformation of the foils [Yamazaki *et al.* (2015)].

Induced radioactivity is also produced at high energy electron accelerators. Methods for estimating radiation levels at such facilities have been discussed by Fasso *et al.* (1999).

#### **4. POLYCRYSTALLINE GRAPHITE FOILS (PCG FOILS)**

##### **4.1 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. These are not pyrolytic foils. PCG foils 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  $800 \mu\text{g}/\text{cm}^2$  they are less expensive than evaporated foils. See Fig. 11, next page. 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 [Varney (1995), and have in-beam lifetimes competitive with arc-evaporated foils. For use as stripper foils they should be conditioned by gradually turning on the beam [Binder (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, and have been used to strip uranium beams at energies of 50 MeV per nucleon at RIKEN in Japan [Hasebe *et al.* (2015)].

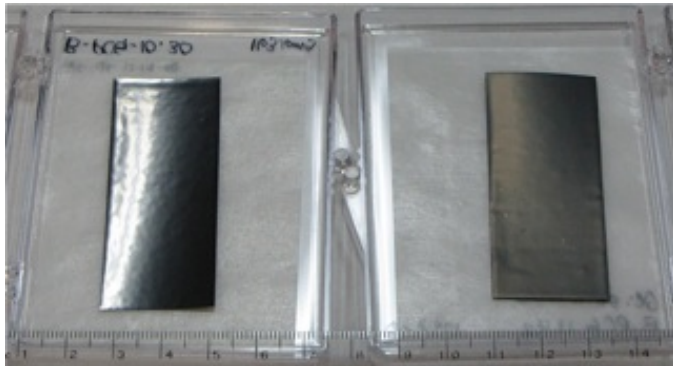
PCG foils as produced are relatively soft and flexible, and contain volatile impurities (presumed to be water) that are easily removed by baking (see below). Baking makes the foils very brittle. Outgassing appears to be complete above 800 K. PCG foils shrink irreversibly in their transverse dimensions by about 1 percent if heated above 1000 K [Miller *et al.* (2008)].

Electron micrographs of PCG foils show them to consist primarily of randomly stacked flat graphite platelets oriented with their c-axes perpendicular to the plane of the foil. The average spacing of the platelets perpendicular to the foil plane is unknown. The packing of the platelets is not particularly dense nor regular. As a result, the effective mass density of PCG foils is only about 1 g/cc.

Stock PCG foils are provided in pieces that have nominal dimensions 25 mm x 60 mm, or larger, shipped in individual padded transparent plastic boxes (Fig. 11, below). Larger pieces (to 300 mm square) and smaller pieces can be provided by quotation. Prices for the most popular forms are given in our price list. Areal densities are provided within 15% of Customer's specification unless otherwise specified. Known 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-xxxx is the areal density xxxx in micrograms per square centimeter.

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Fig. 11. PCG-10000 foils (10 milligrams per square centimeter. Left view: substrate side. Right view: growth side.



#### **4.2 Porosity and microscopic nonuniformity:**

Because of their grain size and porosity on a scale of 1-2 micrometers, PCG foils show a microscopic nonuniform thickness to particle beams. This nonuniformity is approximately  $\pm 40\%$  at areal density  $1 \text{ mg/cm}^2$ , diminishing to about  $\pm 20\%$  at  $7 \text{ mg/cm}^2$ , [[Takabayashi (2000)]. Extrapolation suggests that microscopic nonuniformity of PCG foils would approach  $\pm 100\%$  near  $0.3 \text{ mg/cm}^2$ , 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 is about twice the thickness of a standard carbon foil.

#### **4.3 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  $200^\circ\text{C}$ . It is preferable to flush the oven with nitrogen to avoid the risk of oxidizing the foil during baking. The foil is typically placed between clean sheets of glass spaced apart by perhaps 0.1 mm. 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 much more brittle than an unbaked foil.

DO NOT USE solvent-based adhesives to fasten PCG foils to

frames.

PCG foils have been stored in laboratory atmosphere for many years without any observed deterioration.

#### **4.4 Baking and electrical resistivity:**

Resistivity measurements on carbon foils must be done with care to avoid effects due to surface water layers [Sommer *et al.* (2008)].

A vacuum high-temperature bake can be used to consolidate a PCG foil, reduce its electrical resistivity, and drive off tightly bonded gases. BE AWARE that PCG foils become extraordinarily brittle upon being baked. A mass loss of typically 25% occurs for foils so treated at temperatures in the range  $720\text{--}2270^\circ\text{C}$ . Similar experiments on amorphous carbon foils, which tend to graphitize at high temperatures, have yielded similar results, with smaller mass losses. [See section 2.19 above.]

A few tests of resistivities of PCG foils have been done. Typical values for unbaked foils were 1.6-3.6 ohm-cm ( $\Omega \text{ cm}$ ); after baking at about  $2000^\circ\text{C}$  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  $1676^\circ\text{C}$ , after vacuum baking at  $1000^\circ\text{C}$  (LBL specification #335240).

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