Matter and Radiation at Extremes

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ABSTRACT

The Technological Laboratory of LMU Munich supplies various types of solid-state target for laser plasma experiments at the Centre for Advanced Laser Applications in Garching. Our main focus here is on the production of free-standing, thin foil targets, such as diamond-like-carbon foils, carbon nanotube foams (CNFs), plastic, and gold foils. The presented methods comprise cathodic arc deposition for DLC targets, chemical vapor deposition for CNFs, a droplet and spin-coating process for plastic foil production, as well as physical vapor deposition that has been optimized to provide ultrathin gold foils and tailored sacrifice layers. This paper reviews our current capabilities, which are a result of a close collaboration between target production processes and experiment, using high-power chirped pulse amplification laser systems over the past eight years.

I. INTRODUCTION

Laser physics research at the LMU Munich, especially involving the laser proton/ion acceleration activities based on high-power laser systems at the Centre for Advanced Laser Applications (CALA),[1] needs specific targets in order to investigate and optimize laser-driven, plasma-based particle sources. To meet experimental demand, the Technological Laboratory has established various target production methods over the last few years. One of the key capabilities that has enabled groundbreaking experiments, for example, in radiation pressure acceleration,[2] is the production of ultra-thin, free-standing diamond-like-carbon (DLC) foils. These foils are optically transparent and possess far superior stability, endurance, and smaller minimum thicknesses (3–5 nm) than standard graphite foils. The DLC-Factory, which uses cathodic arc deposition, is now an integral part of target fabrication and seeded complementary activities such as carbon nanotube foam (CNF) production. Such foams are macroscale assemblies of separate carbon nanotubes (CNTs), which are allotropes of carbon with a cylindrical nanostructure. CNFs enable the production of targets with average densities of just a few percent solid density. Due to the nanoscopic structure that is on a much smaller scale than typical laser wavelengths, CNFs can be considered homogeneous at the scale of the laser wavelength and focal spot diameter. When the foams are directly synthesized, they have superior electrical and mechanical properties compared with other productions methods (e.g., the solution-based filtration process[3]). For the direct synthesis of CNF films, a floating catalyst chemical vapor deposition (FCCVD) technique has been established in our lab.

Increasingly available, higher repetition rate, chirped pulse amplification (CPA) laser systems are in need of a great number of targets. To this end, we have investigated droplet and spin coating for the production of plastic foils with thicknesses ranging from a micrometer to a few nanometers.

In addition to low-Z materials, we have also expanded our work to include the use of heavier elements, in particular, gold foils. These foils are produced via physical vapor deposition (PVD) in a dedicated vacuum chamber and can reach thicknesses as low as 5 nm, representing a test case for laser acceleration of high-Z ions.

II. DLC-FACTORY

DLC is a metastable form of amorphous carbon containing a significant fraction of sp^3 bonds (up to 85%[4]). DLC exhibits a high mechanical hardness and is chemically inert; optically transparent DLC films have widespread applications as protective coatings in areas such as optical windows, magnetic storage disks, and micro-electromechanical devices (MEMs).[5] In principle, such films can be produced using various methods, but common to all is that carbon ions with kinetic energies of several tens of electron volt (typically 70–100 eV) impact a substrate. One such method is filtered cathodic vacuum arc deposition (FCVA),[6] which has been used for years in the protective coating of hard carbon films.[6] Filtered cathodic vacuum arc deposition uses a high-current, low-voltage electric arc to vaporize a cathode material, producing an energetic plasma with a high ion density and kinetic energies of several tens of electron volt. Along with the ions, neutral and macro particles are thermally emitted but are filtered out very efficiently using a magnetic duct, bent by 90°. Inside this duct, the neutral and macro particles cannot follow the magnetic field. They hit the duct wall and are thus separated from the carbon ions. The deposition rate of DLC is controlled both by the ion current and pulse rate of the arc. The FCVA method is particularly suitable for the synthesis of thin, self-supporting DLC foils, due to its dramatic ability to reduce both the number of macro particles and the growth rate of film thickness. The latter is essential, as in contrast to protective coatings, internal stress imposes a severe limit during DLC target production. With FCVA, hydrogen-free and highly transparent films with diamond-like properties may be produced.

In the DLC-Factory,[4,7] the core devices are the DLC deposition chamber and the substrate preparation chamber, located in a clean room environment. Employing the FCVA method, DLC foils are grown on mono-crystalline silicon wafer substrates, the latter's suitability resting in their high surface quality and the relatively high electrical conductance of silicon that is required for the FCVA method.

In order to be able to detach the DLC layer from the silicon wafer surface (and to transfer it to the DLC target holder), a parting agent is used—one that must be water-soluble, enabling the detachment of the DLC layer in the subsequent floating process. We identified NaCl as a most suitable agent for providing this sacrifice layer. Other well known (organic compound) parting agents, such as betaine and Teepol, are more prone to destruction by the highly reactive carbon ions. The NaCl layer is thermally evaporated at high vacuum, at a pressure of approximately 5×10^{-7} mbar, in the substrate preparation chamber prior to DLC deposition. The layer thickness is adjusted to around 300 nm. According to microscopic measurements, the surface roughness of the NaCl layer lies between 100 and 200 nm, which determines the eventual roughness of the DLC surface. NaCl is hygroscopic, i.e., it absorbs moisture from the air; therefore, the prepared substrates with the NaCl layer are stored in a dry argon atmosphere until DLC deposition.

Before deposition, the production chamber (Fig. 1) is evacuated by a cryopump to achieve a high vacuum (around 10^{-8} mbar). Deposition itself takes place in an argon atmosphere at about 10^{-4} mbar (necessary for high-voltage arc creation). The high voltage arc has a pulse duration and frequency of 5–10 ms and 10 Hz, respectively. To control ion energy, a bias of −20 V is applied to the Si substrate. In order to ensure the best possible uniformity in thickness of the DLC foils, the chamber is equipped with a substrate holder, which can rotate with a constant speed during deposition. This technique can mitigate large-scale thickness variations and currently delivers DLC foil thicknesses from 3 to 60 nm.

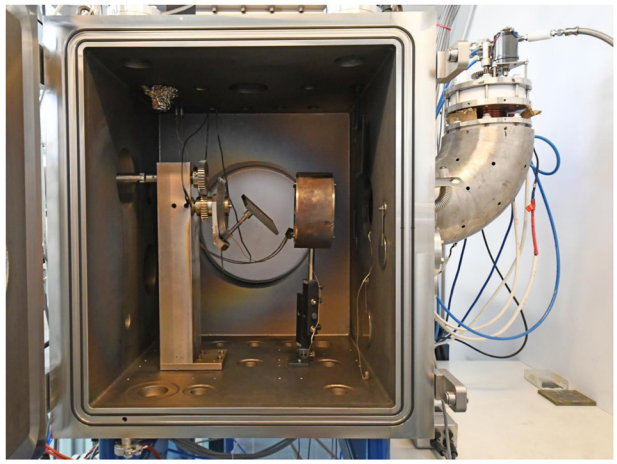


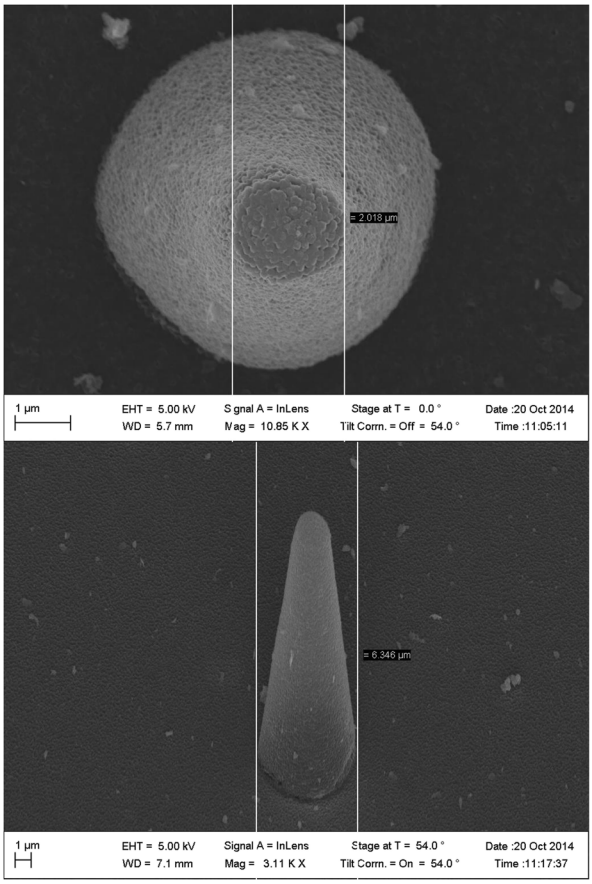
FIG. 1. Vacuum chamber for DLC foil production.

In order to release the foils from the substrate, a dedicated floating assembly is used. It has a linearly moving substrate holder (metallic clamp), which can drive the substrate with a slow and constant speed in distilled water. The water dissolves the parting agent, and the DLC foil floats on the water's surface. When the foil is completely detached, it can be transferred from the water to the target holder. Typically, the target holder is a thin stainless-steel plate with many small (e.g., 0.5 mm diameter) holes. The transfer is performed by pulling the DLC foil with the holder plate out of the water in such a way that the DLC layer is finally deposited onto the target holder surface, covering all (or most of) the holes. After the final drying, the target plate is ready for the laser experiment in which the laser beam is directed through a specific hole and can interact with the freestanding DLC foil.

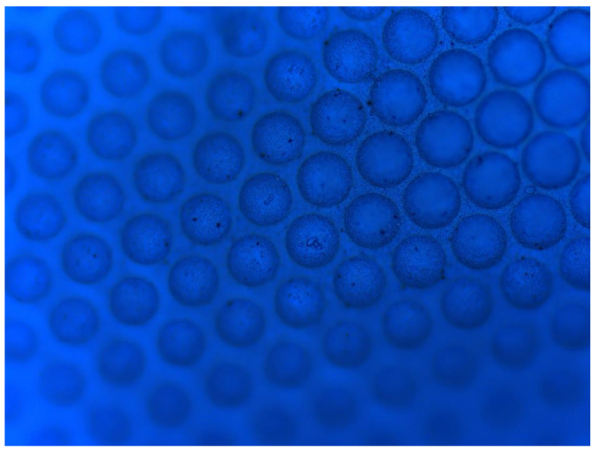
The upper limit for the thickness of DLC foils currently depends on the floating process. Foils with thicknesses higher than 60 nm exhibit such strong internal stress that they break up into small pieces during floating. The lower limit of the foil thickness depends on the drying process, and for 3–5 nm, for example, we observe a total hole-coverage of only a few percent. Triple-point drying could be a viable solution for this problem and a potential next step.

Characterization of the final thickness is carried out on reference silicon wafers, upon which lines are drawn with permanent marker instead of using a parting agent. After DLC deposition, the marker can be removed by a suitable solvent (e.g., isopropanol), leaving an edge whose height can be measured by atomic force microscopy (AFM). Other metrological methods, such as white light interferometry, confocal microscopy, or spectral reflectometry are available in our laboratory and employed, for example, for characterization of plastic foils. These methods, however, are not well suited to on-substrate DLC characterization due to the uncommon optical properties (high refractive index and absorption) of DLC film.

In addition to the synthesis of planar DLC target foils, the latest developments in the DLC-Factory have included the production of structured targets in the form of hemispheres or cones [Figs. 2(a) and 2(b)].



(a)



(b)

FIG. 2. (a) Microscope top and side view of the DLC-cone. (b) Microscope view of the hemispherical target structure.

The detailed production process will be reported in a future publication; a brief outline will be presented here. Non-planar DLC foils rely on the availability of a master model, which can be copied onto the 2.5D structure (3D structures with a small thickness, e.g., wrinkled alumina foil). The master model must be able to withstand the harsh conditions of the DLC-production process. One method used to produce the master model is two-photon polymerization. Different nano-imprint techniques must be employed to transfer the resist-master onto another material (e.g., Ormostamp); however, other masters can be used directly, e.g., glass micro lens arrays, which are normally used in Shack Hartmann wave front sensors. When aiming to build high-aspect ratio microstructures, the master must be rotated around two axes while coating it with the sacrifice and the DLC layers. The floating process in this context is identical to that of the planar DLC-foil case.

III. CNF FABRICATION

CNTs are allotropes of carbon with a cylindrical nanostructure, possessing unusual properties that are highly valuable in many fields. An important application in laser plasma physics, the macroscale assembly of individual CNTs gives CNF, which can be used as a target to provide a near-critical-density thin plasma slab. This is due to the morphology of the foam, shown in Fig. 3. Individual CNT bundles are packed together, forming a foam-like structure. An individual carbon nanotube has a diameter of between 0.4 and 2 nm, a CNT bundle between 2 and 40 nm, and the length of each ranges from 1 μm to 1 cm. Because of the very large length-to-radius ratio of CNTs, the void space of the foam may be as high as 99%, which results in their very low average density, adjustable around a mere few percent of the density of solid carbon. If the foam is fully ionized during the laser-target interaction, this density is near the critical density for typical laser wavelengths, and can therefore be used to efficiently manipulate laser pulses and their interaction with plasmas at ultrahigh intensities.[8–10]

In the LMU Technological Laboratory, CNFs are produced using FCCVD,[3] which is one of the best methods for large-scale synthesis of high-quality carbon nanotubes. The growth mechanism is shown in Fig. 4. Firstly, the catalyst is evaporated upstream of the furnace and transferred to the high-temperature zone by Ar+CH_4 gas flow. As a catalyst, a mixture of 1 part sulfur and 93 parts of ferrocene [$Fe(C_5H_5)_2$] is used. In the high-temperature zone (1100 °C, step 2 in Fig. 4), the catalysts agglomerate as nanoparticles. Such nanoparticles

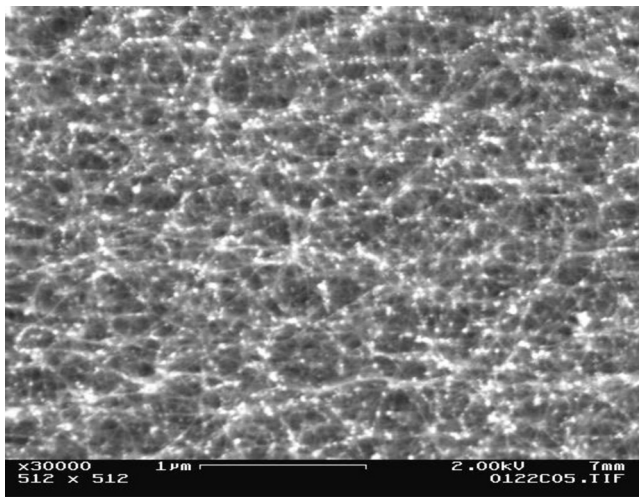


FIG. 3. Morphology of carbon nanotube foam characterized by SEM.

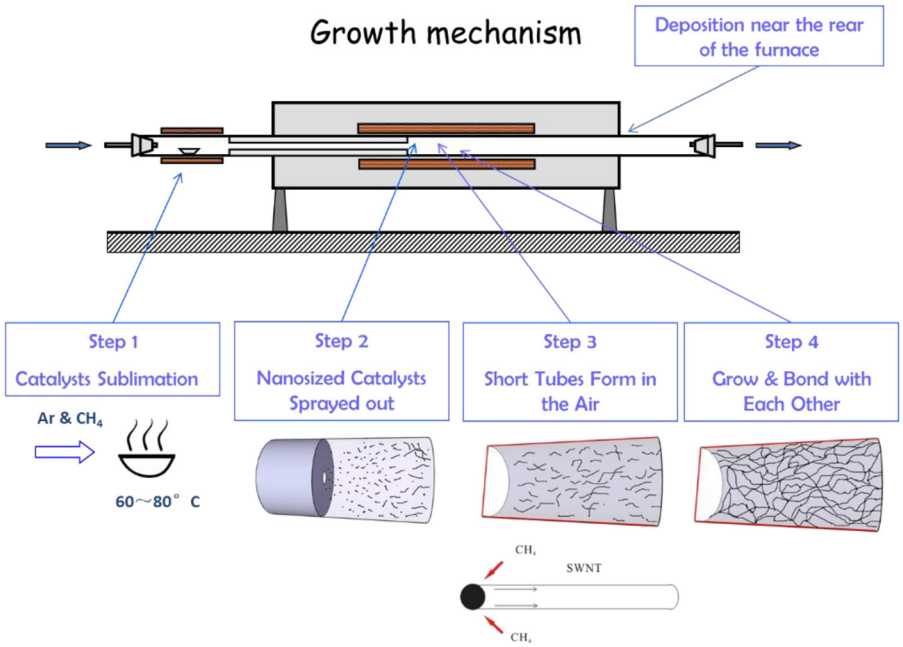


FIG. 4. Growth mechanism of carbon nanotube in CVD system.

are the seeds of carbon nanotubes, and fed by the carbon atoms from the CH_4, short nanotubes grow from the seeds, becoming longer during floating. After a growth time of a few seconds in the gas flow, long nanotubes are dragged out of the high-temperature zone by the gas and deposited on the substrate placed at the rear side of the furnace.

The CVD (chemical vapor deposition) system at the Technological Laboratory is composed of a four-channel gas delivery system, a Nabertherm[11] high-temperature furnace, a 1.5 m-long quartz tube, and a water-cooling system. The gas delivery system is the most important part of the CVD, capable of simultaneously delivering four different gases to the CVD system. The system can be subdivided into four independent components which are the gas bottle, channel valves, flow meters, and the digital controller.

The collection of CNF is very simple: one can either place a thin-foil target at the rear of the furnace to coat it with CNF, or, peel off the foam from a substrate with tweezers to use it as a free-standing near-critical-density target. Typical growth times of CNF range from a few minutes to a few tens of minutes, depending on the desired thickness. We found that, due to strong van-der-Waals forces, free-standing CNF slabs as thin as about 10 μm could be produced. The thickest foam grown in one run was about 150 μm; however, thicker foams may be obtained by performing multiple runs successively.

The density of CNF is characterized by measuring its mass per unit volume, where the mass is determined using an ultra-microbalance. To match the precision and accuracy of the ultra-microbalance, mass measurements are typically performed on tens-of-micrometer-thick CNF samples covering an area of 1 cm $\times$ 2 cm on a silicon wafer. After tens of minutes' growth, the CNF-coated silicon wafer is taken out from the quartz tube and cooled to room temperature. The mass of the CNF-coated wafer is measured first, after which the bare Si wafer mass is measured after removing the CNF. The mass of the CNF is obtained from the difference of the two measurements. The thickness of the CNF on the Si wafer is measured prior to removal with a calibrated confocal microscope by identifying the front surface of the CNF and surface of the Si wafer. Series measurements are performed on samples with different growth times and different growth parameters such as the catalyst feeding rate, growth temperature, and wafer position. CNF thickness linearly increases with growth time for a given growth parameter, and its density depends on the growth parameters. Currently, the mass density of CNF can be controlled in the range of 2–12 mg/cm^3, but it can be increased by mechanically compressing the foam using a custom-made foam compressor. The compressor can decrease the foam thickness from 100 μm to 10 μm with an accuracy of $\pm$2 μm, thereby increasing the average CNF density by a factor of 10.

IV. PLASTIC FOILS

A. Spin-coating

Spin-coating is a common method for the fabrication of thin films found in a wide range of applications. The principle is simple: a small amount of a specific solution is placed on a fast-rotating planar substrate, which, through centrifugal forces, spreads over a large area. Evaporation of the solvent results in a thin liquid or solid film. Depending on the size of the spin-coater and the substrate, highly uniform films with a diameter of up to 30 cm are achievable.[12] In microelectronics, especially, spin-coating is used for applying thin films on silicon substrates (e.g., for polymeric photoresist layers to transfer circuit designs). Spin-coating has also proven particularly useful in providing nanometer thin foils to access the laser-driven, light-sail, ion-acceleration regime.[13] The combination of volatile solvent and high rotation speed enables us to produce ultrathin foils down to 5 nm.

For spin-coating in target fabrication at the LMU, we use a mixture of formvar[14] with different solvents. Standard microscope glass slides (75 $\times$ 25 mm^2) serve as substrates. Larger area foils can be achieved by using silicon wafers; however, these are a rather

cost-intensive alternative. The substrate is placed on the spin-coater, which can rotate with variable frequencies up to 250 Hz. A well-defined volume of solution is applied centrally to the substrate with a pipette. To prevent splashing of the liquid, a protective cap can be placed on the spin-coater. By rotating the substrate, the solution is distributed homogeneously and any redundant material is removed quickly. The solvent evaporates almost instantaneously and leaves behind the nanometer-thin formvar foil. Therefore, the spin-coater can be switched off immediately and the target processed further. After cutting the foil into the desired shape, it can be removed from the substrate using the floating technique, as described in the context of DLC foil production.

Production tests have shown that the best surface quality is achieved when using pyridine as a solvent (dichloroethane and nitrobenzene have also been tested). In order to obtain a variation in thickness, several solutions of formvar, with mass-concentrations between 1% and 11%, have been tested. Using these concentrations and a standard rotating frequency of 200 Hz, highly stable formvar foils ranging from 15 to 600 nm were produced, with diameters up to 30 mm (Fig. 5). The standard deviation of thickness across the foil on a single substrate yielded 1%–5%, except for that of the 1% mass-concentration, which had a standard deviation of 10%. Thicknesses were measured with a confocal microscope and an atomic force microscope (AFM). Figure 6 shows average formvar foil thickness values and standard deviations for mass concentrations between 3% and 7%. When attached to the target holder, these plastic foils can be stored for at least two weeks without a degradation in quality. Compared with DLC foils, spin-coated formvar targets can be produced much faster with a larger range in thickness.

B. Droplet method

Here, the plastic solution is dripped, using a pipette, on a distilled water surface.[15,16] The solvent evaporates, the plastic polymerizes, and after a few seconds, a foil that floats on the water is created. The advantage of this method is that it omits the floating process and potentially enables more rapid production rates. In our first tests, we used a single pipette to release a drop of formvar-dichloroethane solution on to a water surface. The cross-sectional area over which the foil thickness was satisfactorily uniform was too small, however, and thickness reproducibility was poor. This problem was resolved by use of a multi-channel pipette, in our case one that possessed sixty-four channels. The tips were arranged in a 4×16 array separated by 5 mm.

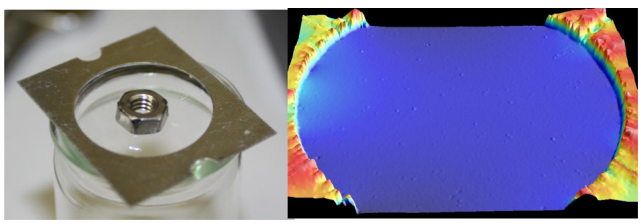


FIG. 5. A formvar foil with thickness 40 nm spanning over a circular aperture of diameter 20 mm is sufficiently stable to hold a ~600 mg object (left). Spin-coated formvar foils feature unique surface quality, as evidenced by the topography/topographic profile of a mounted 15 nm foil (diameter 0.5 mm), measured with a confocal microscope (right).

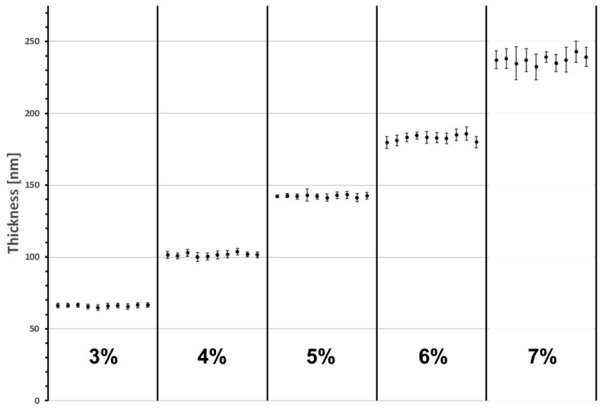


FIG. 6. Average formvar foil thicknesses and standard deviations for mass-concentrations 3%–7%.

Each tip could drip off the solution acting as a single pipette, meaning that many droplets were deposited simultaneously onto a relatively large surface area of the water. Due to the low mass of each drop, the solution did not penetrate the surface, but floated on the water (for a total solvent volume of up to 200 μl). Experiments have shown that the number of tips could be decreased to 4×4 and the distance between them increased to 2–2.5 cm, without significant loss of foil quality, in terms of uniformity in thickness. Tests with polystyrene as plastic and chloroform as solvent have also been performed (all four combinations of two plastic materials and two solvents were investigated). In this set of combinations, formvar and dichloroethane proved optimal. By varying the concentration of the solution between 0.125% and 10%, formvar foil thicknesses from 25 nm to 2000 nm were obtained. The mean standard deviation of the foil thickness, both in terms of reproducibility as well as homogeneity, was found to vary from 6% to 16%, and did not depend on the mean foil thickness. This method is faster than the spin-coating technique, but has some disadvantages. We noticed that formvar develops a large number of defects (bubbles of different shapes and sizes inside the foil), covering up to approximately 20% of the whole surface. In addition, and probably as a result of the defects, very thin free-standing formvar foils were not sufficiently stable to be used in experiments. If these limitations could be resolved, the droplet method could be automated by the use of a pipetting robot and automated pick up.

V. THIN FOIL GOLD TARGETS

Physical vapor deposition (PVD)[17,18] allows for the production of targets within a thickness range of 5–1000 μg/cm^2 for many elements and chemical compounds. The targets can be either self-supporting or evaporated onto a graphite backing with a minimal thickness (for stability) of 5 μg/cm^2. Using this method, Au targets for laser acceleration of heavy ions were produced. The goal was to obtain free-standing gold foils as thin as possible (about 5 nm) and with an acceptable survival rate (10%). Gold targets are of interest because they are chemically inert, and therefore can easily be cleaned to be free of surface contaminants, using, for example, heating. Gold foils hence represent a practicable test case to study the radiation pressure and

light sail acceleration of heavy ions. The targets can be stored for several days without undergoing oxidation or other reactions.

In our standard procedure, Au foils are coated on microscope slides (75 × 25 mm^2). Once again, a water-soluble parting agent between the substrate and the foil enables foil release in a floating process, after deposition. Three types of parting agents have been tested: Teepol soap, betaine, and sodium chloride. In the first step, a slide surface was covered with parting agent, after which a thin gold layer was deposited on the parting agent side of the slide in the high vacuum chamber, using PVD. The main component of the evaporation set-up inside the chamber is a tungsten boat, which contains the gold that is evaporated, and a movable metallic plate serves as shutter for defining the starting and stopping time of evaporation. The substrate is mounted above the shutter in a metal frame, and the evaporation rate and final-layer thickness are monitored by means of an oscillating quartz crystal. After evaporation, the gold layer is removed from the substrate by means of the floating method as described for DLC foils.

Due to the requirements of laser-ion-acceleration experiments, self-supporting, as-thin-as-possible Au foils are in high demand. With a view to meeting such a demand, the standard procedure described above required optimization. As flatness of the substrate surface is critical, it was decided to use silicon wafers instead of microscope slides. The use of sodium chloride as a parting agent with an 8 $\mu g/cm^2$ thickness resulted in minimal surface roughness and allowed us to reduce foil thickness to 5 nm. After transfer to the target holder, we exposed the foils to the light of an infrared lamp to speed up the drying process. For the thinnest foils, however, this last step of the procedure resulted in foil damage. Close to the end of the drying process, the surface tension of the water layer most likely caused a rupture of the free-standing Au foil covering the 200-μm wide holes of the target holder. This rupture can be avoided, however, by transferring the target holder with the gold layer on top to an isopropanol bath. The lower surface tension decreases the probability of foil ruptures, leaving more holes covered with intact gold foils. In general, when using this production method, the following damage effects may occur: displaced gold foil (some holes are not covered), drying defects (described above), and double layers or macro-fracture of the gold foil. For a 200 μm hole diameter and 5 nm foil thickness, a production efficiency of between 30% and 50% was achieved.

VI. SUMMARY AND CONCLUSIONS

We have described the procedures and main results of investigative target production research at the Technological Laboratory of LMU, while focusing on ultrathin foils that are relevant for laser-driven ion acceleration research, employing the high-power laser systems at CALA. Table I summarizes the current capabilities.

Near-term experiments will likely reveal challenges. One obvious current limitation is the manual production process. When narrowed down to detailed specifications of target material and thickness, large numbers of targets can be produced using mass production methods such as provided for by MEMS.[19] Interesting approaches for continuous target delivery include liquid and frozen jets, drapes or droplets (see, for example, Refs. 20–23) as well as liquid crystals.[24] In light of the fast growth of available peak power in laser systems, we envisage advancement in the near future with regard to creating solutions to permit the transferal of our current manual processes to processes of automation and further optimization. These improvements will be needed to simplify and speed up target production to the level demanded by the thousands of targets inherent to each experimental campaign. Conversely, experiments with increased laser power, in particular, at the PW levels found the lasers at CALA and other facilities emerging worldwide,[25] will likely reveal new challenges that require urgent identification and the development of comprehensive mitigation strategies. Immediate availability of large quantities of targets possessing a wide range of materials, densities, and thicknesses will provide a solid base for this highly interesting field of laser-targetry research.

TABLE I. Current capabilities of free-standing foil target production and applied processes.

Material	Thickness range
Diamond like carbon	3–60 nm
Carbon nanotube foam	10–150 μm
Formvar (spin coating)	5–600 nm
Formvar (droplet method)	25–2000 nm
Gold	5–500 nm

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