A Dedicated Target Fabrication Laboratory for Low Density Materials, Polymers and Novel Materials

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Introduction

The Central Laser Facility has recently opened a fully refurbished chemistry laboratory within the Target Fabrication Group that is dedicated to the production of low density materials, polymer thin films and other novel chemistry based target components to deliver to the high power laser programs that are run by STFC. There has been, for a number of years, an increasing demand for low density materials as target components with foams being the most requested of the type. A dedicated laboratory for the production of such targets will enable target quality to be increased and also allow the development of new and more complex materials. This article presents the current capabilities of the laboratory and also presents initial results of production and characterisation of low density polymeric foams and thin polymer films.

Laboratory Development

A dedicated cleanroom that is class 10,000 has been equipped with a range of equipment to carry out a number of processes for target manufacture. These include UV lamps for activating photosensitive initiators in processes, critical point dryers for solvent removal from porous materials without collapsing the structure, spin coaters for the deposition spin of polymers and resists, dip coating systems for polyvinyl formal thin film deposition and electroplating baths for the deposition of thick metallic layers. Each of the processes will be integrated with the existing target fabrication capability to complement the current activities and will be further developed to allow novel targets and therefore new science to be investigated.

1a. Polymer Foam Production

Polymer foams are produced using a well-known procedure of UV polymerisation of a monomer [1]; in this case trimethylol propane triacrylate (TMPTA) was used with the solvent polyoxyethylene-4-lauryl ether (Brij 30) and the initiator, Benzil. Using the specified reagents it is possible to produce foams that have a density as low as 5mg/cc but there are limitations to the ability to manufacture free standing foams of the lowest densities as they do not have the physical strength to hold together. The structure holds well from 100mg/cc to 15mg/cc, however, below this density the gel-foam can collapse under its own weight if making a relatively large sample. Large samples are difficult to classify for the material but it has been found that samples made in syringes with dimensions of greater than a few millimeters are prone to collapse. In such cases a support tube or washers is needed to contain the foam during the manufacture process. Low density foams in precision washers have been manufactured for experiments on the Vulcan laser [2] and thin film foam targets have been manufactured for ion acceleration experiments [3].

Other factors that affect the success of foam production include the curing time. A mercury arc UV lamp is used to cure the solutions before setting into methanol. It appears the lower the mg/cc the more time should be dedicated to curing. This effect could be due to the uncertainty of successful curing due to the sample remaining colourless but the samples, even when left standing to continue to cure, take longer to cure completely.

1b. Foam Characterisation

Images of 100mg/cc foams have been taken using an SEM. In order to reduced charging effects in the SEM the foam samples are coated with a few nanometres of gold. Some examples are shown in figures 1 and 2.

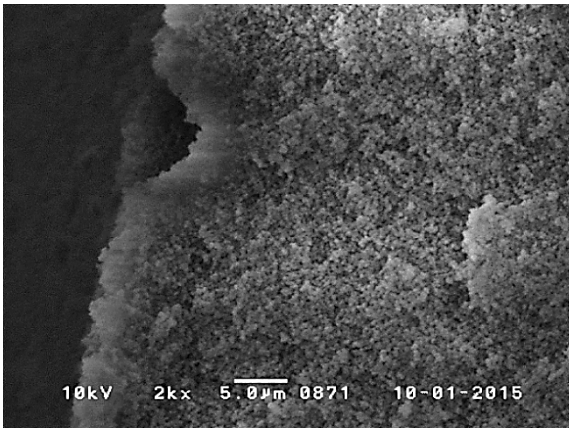

Fig. 1: 100mg/cc foam, Au coated, x2000 mag. Image used to characterise pore size.

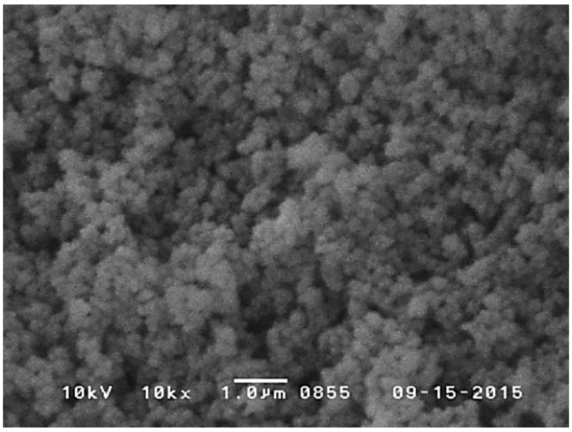

Fig. 2: 100mg/cc foam sample, Au coated, x10000 mag. Image used to characterise pore size and view internal polymer structure.

1c. Future Foam Developments

A development programme has begun to progress low density foam production techniques to increase the yield of these components. Investigation has also begun into alternative monomer, solvent and initiator combinations to enable lower density foams to be produced.

2a. Formvar Production

There is a continual demand for ultra-thin plastic films and one manufacture process is to use dip-coated Formvar (polyvinyl formal). The material is commonly used as a support film for TEM grids for microscopy as it can be made very thin (10nm) and is very strong. Formvar is dissolved in a solvent, 1,2-dichloroethane, for a number of hours. A microscope slide is then dipped into the solution using an automated dip coating machine. The concentration of the solution and the dipping speed determine the coating thickness and vibrations in the dipping process can cause artefacts on the film surface. The automated dip coating machine is designed to have variable speed control and low vibration movement.

2b. Characterisation of Thin Film

Successful film thicknesses down to 15nm have been produced and characterised by floating the film onto a flat plate and subsequently measuring the (step) height using an interferometer, a touch probe and an Atomic Force Microscope (AFM). The three characterisation measurement techniques give the same result within reasonable tolerance. Examples of an Atomic Force Microscope scan and an Interferometer scan on a Formvar thin film are shown in figure 3 and 4 below.

Films have been removed from the substrate and floated off across a range of mounts and have shown that it is possible to manufacture 10nm thick films with a roughness Ra value measured of less than 5 nanometers. Such films resist damage when suspended across an area of at least a few millimeters, making them suitable for laser irradiation.

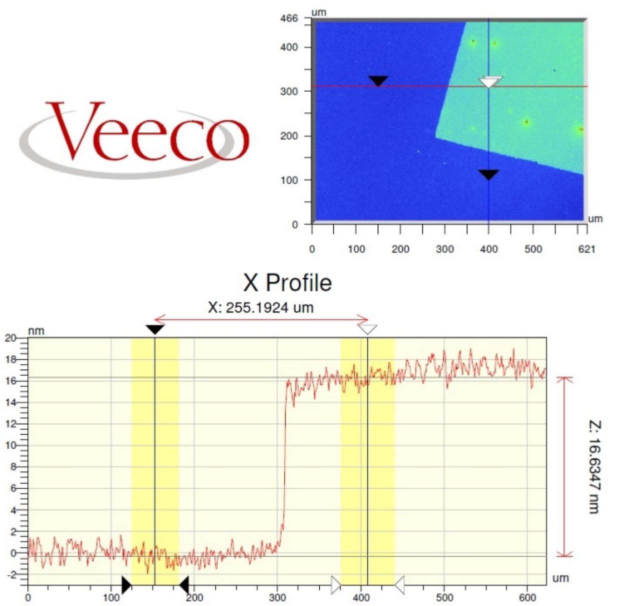

Fig. 3: An interferometer scan of a 16nm thick Formvar film mounted on a glass slide

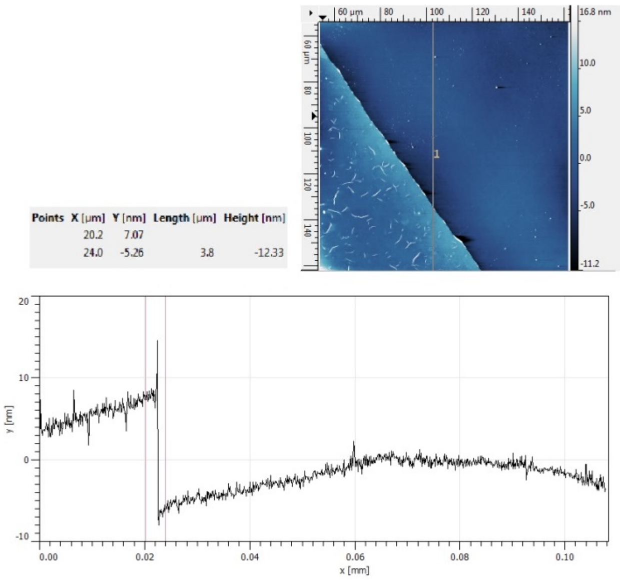

Fig. 4: An AFM scan of a 12nm thick Formvar film mounted on a silicon wafer

2c. Future Developments

Target Fabrication plans to improve the quality of the films by developing the dip coating system to further reduce errors. Development to produce thinner films of the order of 5nm in thickness is also an area of future research and also to investigate different characterisation methods for the films.

3. Spin Coating

A high specification large area spin coating system (figure 5) has been purchased by the department. The system is used to deposit a range of materials onto silicon and glass substrates. With spin coating it is possible to accurately control the thickness of a photoresist or a polymer film by varying the spin speed. The capability to deposit poly(methyl methacrylate) (PMMA) layers of up to 40um has been shown by depositing many layers on top of each other. The uniformity of the deposits was excellent (+/-5%) and shows promise for manufacture of target components with ablator layers or for the manufacture of MEMS devices with thick coatings.

Fig. 5: Large area spin coater for (thin) film production

4. Electroplating

The chemistry laboratory provides a designated area for the electroplating of metals that had been conducted in the past at the Target Fabrication cleanroom. The technique is used for the deposition of thick metal layers (up to 30µm) and allows the coating of complex and/or intricate forms. The deposition of a metallic coating onto an object is achieved by putting a negative charge on the object to be coated and immersing it into a solution which contains a salt of the metal to be deposited. In other words, the object to be plated is made the cathode of an electrolytic cell.

The equipment used is a JB Aqua 5 Heated Bath from Grant, a Thurlby PL310 Power Supply and a fume cupboard with suitable ammonia filters (figure 6).

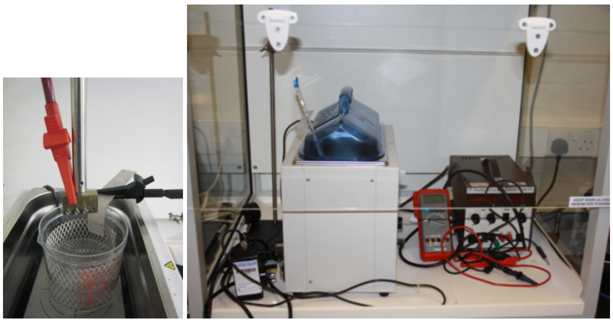

Fig. 6: Electroplating equipment showing close-up of (one type of) plating vessel (left image)

Gold and palladium have been plated using electrolytes based on complex ions of ammonium gold sulphite and palladium diammino-nitride.

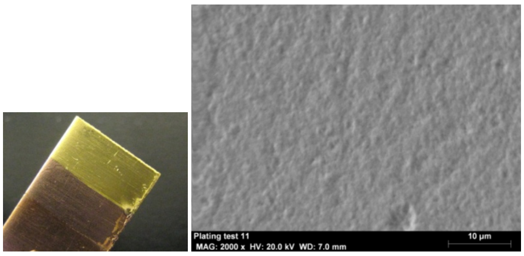

Fig. 7: Optical and SEM images of electroplated gold

Copper plating capabilities are aloso planned to be introduced in the future. The use of pulsed current for the metal plating of high aspect ratio structures is another area of activity.

References

1. J. Vac. Sci. Technol. A, **8**, 968 (1990) *John W. Falconer, Wigen Golnazarians, Michael J. Baker and Douglas W. Sutton*
2. Phys. of Plasmas, **18**, 012704 (2011) *L. Volpe et al*
3. CLF Annual Report 2013-2014, "Automated Production of High Repetition Rate Foam Targets", *F Hall, C Spindloe, D Haddock and W Nazarov*