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PROCEDURE FOR PRODUCING A CYLINDER OF CELLULOSE ACETATE FOAM CONTAINING ONE GLASS MICROSPHERE

J. A. Rinde

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PROCEDURE FOR PRODUCING A CYLINDER OF CELLULOSE ACETATE FOAM CONTAINING ONE GLASS MICROSPHERE

Abstract

This report, which updates UCRL-51708,¹ describes a procedure for producing foam strands containing a single, well-characterized, DT-filled, glass microsphere.

Extrusion Process

APPARATUS

In order to extrude one microsphere in a cylinder of foam and recover it, all steps must be observed with the aid of a low-power stereomicroscope. Without the use of this instrument, the microsphere will be lost; it may even be lost when the microscope is used.

The required apparatus includes the microscope and a base plate that allows the microscope to be turned horizontally; a clear glass Dewar about 25 cm long and 8 cm in diameter; two X, Y, Z micromanipulators, a microvacuum chuck, and a holder for small capillary tubes.

The apparatus is set up as shown in Figs. 1 and 2. In Fig. 1 we show the extrusion apparatus with the glass Dewar, microscope, and X, Y, Z micromanipulator ready for the extrusion. Note that the Dewar has a clear glass plate fused into the side to allow an undistorted view of the extrusion tip. Figure 2 shows the setup used to add the glass microsphere

to the cellulose acetate solution in the glass capillary tube.

CELLULOSE ACETATE SOLUTIONS

The following solutions produce good foam by the extrusion process described below.

Fig. 1. Extrusion apparatus.

1. J. A. Rinde and R. R. Stone, Preparation and Fabrication of Low-Density Microcellular-Foam Laser Targets, Lawrence Livermore Laboratory, Rept. UCRL-51708 (1975).

Fig. 2. Microsphere loading apparatus.

	No. 1	No. 2
Cellulose acetate, E-4655	5 g	3 g
$\text{Mg}(\text{ClO}_4)_2$, anhydrous	10 g	10 g
Water	30 g	25 g
Ethyl $\underline{d}$, $\underline{l}$ -lactate	25 g	42 g
Acetone	30 g	20 g

Solution No. 1 has produced foam of 0.08 to 0.1 g/cc density, and solution No. 2 has produced foam with a calculated density of 0.065 g/cc. Both solutions degrade with time, probably due to the formation of a complex between the cellulose acetate and the $\text{Mg}(\text{ClO}_4)_2$, and should be made up fresh every two weeks.

PROCEDURE

The step-by-step procedure for this extrusion is the following:

1. Make extrusion tips from 1.7-mm-diameter capillary tube by drawing them down and breaking them off at the desired inside diameter. Tips must be clean and flat on the end for extrusion to take place. In general, it is not advisable to reuse an extrusion tip unless it is

cleaned in HNO_3 . The best length for a tip is about 2.5 cm.

2. Fill the glass Dewar with 0°C water, and place in position in front of the microscope. Adjust position of extrusion tip with X, Y, Z micromanipulator and focus microscope on extrusion capillary.
3. For filling, place the extrusion tip in a holder with the extrusion orifice inside the holder. Add cellulose acetate solution to the capillary tube with a hypodermic syringe to fill it about half full. Set aside the capillary tube containing the acetate solution and pick up a 40- to 70- μm -diameter glass microsphere, using the X, Y, Z micromanipulator and the microvacuum chuck. Then place the capillary tube under the microscope again and move the microvacuum chuck inside the end of the tube, so that the glass microsphere almost touches the acetate solution (Fig. 2). Turn off the vacuum and use air pressure to blow the glass microsphere off the end of the microvacuum chuck into the acetate solution. Using the syringe again, add more cellulose acetate to the capillary tube to fill it with solution. At this point, locate the microsphere in the capillary tube and distinguish it from any air bubbles that may be in the solution (see Fig. 3). If the microsphere is located more than 1 cm from the extrusion orifice then place capillary tube on end so that the microsphere

will float up closer to the extrusion tip. It will be necessary to turn the capillary tube around to allow the microsphere to float away from the wall before it will float toward the extrusion orifice. With the microsphere located in proper position and centered in the tube, place the glass capillary tube in the extrusion position and adjust proper focus and field of view of the microscope. You should be able to see both the microsphere in the glass capillary tube and the tip of the tube where the solution will be extruded. Using a rubber bulb, apply pressure to the capillary tube and start extrusion. If everything works well, you will see the glass microsphere move down toward the tip as the extrusion takes place; if the procedure does not work satisfactorily, the microsphere will stick to the wall and

Fig. 3. Glass capillary tube filled with cellulose acetate solution containing a single, 50- μ m-diam, glass microsphere.

Fig. 4. Cellulose acetate gel strand containing a single, 50- μ m-diam, glass microsphere. Extrusion tip is shown at the very top of the photograph.

must be floated off before it will extrude. After you see the microsphere pass through the orifice, reduce the pressure to zero, and allow the foam to soak for 16 h to reduce the magnesium ion concentration. Figure 4 shows a cellulose acetate gel strand just below the extrusion tip.

4. To remove long strands of cellulose acetate gel from a water bath, pick up one end of the strand with a two-prong fork and begin wrapping it over the ends of the fork. Using this method, you can transfer several feet of this soft polymer gel from the water bath to a liquid-pentane bath. This

procedure gives straight strands of foam 6-10 cm long after freeze-drying.

5. To freeze-dry the polymer gel, transfer the gel to a liquid-pentane bath at $-50 \pm 15^{\circ}\text{C}$, located in a shallow Dewar flask. Place this Dewar in a freeze-drying apparatus with a liquid-nitrogen trap and start freeze-drying, leaving the frozen strands covered with liquid pentane. After about 4 h in our apparatus the strands will be completely dry.

6. Using a sharp X-actoknife^o or razor blade, cut the frozen gel (before freeze-drying) to length in a bath of cold, liquid pentane. As an alternative, bond two blades together so that they are 100 μm apart, and make both cuts at the same time. To obtain flat parallel surfaces, the cutting surfaces must be parallel and beveled on the outside only.

^oReference to a company or product name does not imply approval or recommendation of the product by the University of California or the U. S. Energy Research & Development Administration to the exclusion of others that may be suitable.