

Synthesizing Cylindrical Specimens of Shape Memory Polymers

Jingkai Guo
(*jguo19@jhu.edu*)

1. Purpose:

The purpose of this protocol is to offer a guideline for synthesizing cylindrical specimens of SMPs, which are suitable for standard uniaxial compression experiments.

2. Materials and Equipment:

tert-Butyl Acrylate, Poly(ethylene glycol) Dimethacrylate, Di(ethylene glycol) Dimethacrylate, 2,2-Dimethoxy-2-Phenylacetophenone

Centrifuge Polypropylene Tubes (50ml), Tube Racks, Balance, Pipette, Pipet Tips, Transfer Pipets with Standard Bulb, Vortex Genie 2 Mixer, Weigh Boats, Spatula, Glass Tubes (Inner diameter 12mm), Rubber Stopper (diameter 12mm), Vacuum Oil, UV Oven, Incubator, Refrigerator, Laser Cutter

3. Steps:

(As an example, the following steps are for the synthesizing of 10 wt% specimens. For 20, 40 wt% specimens the procedure is the same but with different chemical ratios, refer to **Table 1**. The absolute amount of each chemical is subjected to change based on the number of specimens need to be synthesized.)

Step1: Put the polypropylene tube on the balance, and then zero the balance.

Step2: Transfer 1.24ml DEGDMA into the polypropylene tube using a pipette with pipet tip. Measure the mass using the balance.

Step3: Change a new pipet tip. Transfer 2.84ml PEGDMA into the polypropylene tube using a pipette with pipet tip. Measure the mass using the balance. Make sure the mass ratio of DEGDMA: PEGDMA is 3:7.

Step4: Change a new pipet tip. Transfer 45.92ml tBA into the polypropylene tube using a pipette with pipet tip. Measure the mass using the balance. Make sure the mass ratio of DEGDMA: PEGDMA: tBA is 3:7:90.

Step5: Put a weigh boat on the balance. Zero the balance. Add the photoinitiator (2,2-Dimethoxy-2-Phenylacetophenone) to the weigh boat using a spatula until the mass of the photoinitiator is 0.1% of the mass of the polymer solution.

Step6: Add the photoinitiator to the polymer solution. Screw the cap on tightly. Put the tube on the mixer for 1-2 mins. Make sure the photoinitiator is fully dissolved.

Step7: Seal one end of the glass tube using a rubber stopper. Secure the sealing with vacuum oil. Make sure there is no leaking.

Step8: Inject the polymer solution into the glass tube using a transfer pipet with bulb. Seal the other end of the glass tube using a rubber stopper. Secure the sealing with vacuum oil. Make sure there is no leaking.

Step9: Put the sealed glass tube into the UV oven.

Step10: Turn on the UV oven. Expose the glass tube containing polymer solutions to UV light for 20 mins in total. For the first 10 mins, rotate the glass tube every 1 minute to get a uniform exposure to the UV light. For the next 10 mins, do this every 2 or 3 mins.

Step11: Turn off the UV oven. Transfer the glass tube into the incubator. Equilibrate at 70°C for 60-90 mins.

Step12: Cool down the specimen in a refrigerator. Separate the cylindrical polymer specimen from the glass tube. Cut the specimen into several short ones with desired length using a laser cutter.

Mass Ratio (DEGDMA:PEGDMA: tBA)	Volume Ratio (DEGDMA:PEGDMA: tBA)	50ml in total (DEGDMA+PEGDMA+ tBA)
3:7:90 (10 wt%)	2.7726:6.3694:102.8571	1.24ml+2.84ml+45.92ml
6:14:80 (20 wt%)	5.5453:12.7389:91.4286	2.53ml+5.81ml+41.67ml
12:28:60 (40 wt%)	11.0906:25.4778:68.5714	5.27ml+12.12ml+32.61ml

Table1. Mass ratios and volume ratios of three chemicals for 10, 20 and 40 wt%