Synthesizing Membranes of Shape Memory Polymers

Jingkai Guo
(jguo19@jhu.edu)

1.Purpose:

The purpose of this protocol is to offer a guideline for synthesizing thin membrane specimens of SMPs, which are suitable for accurate tensile tests with Dynamic Mechanical Analyzer (DMA).

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, Microscope Glass Slides (75mm*25mm), Thin Rubber Sheet (1mm thick), Clips, Knives, Tissue Culture Dishes, UV Oven, Incubator

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: Sandwich two rubber sheets between two glass slides as spacers. Clip the glass slides at two ends. **(Fig.1)**

Step8: Inject the polymer solution into the space between two glass slides using a transfer pipet with bulb.

Step9: Put the clipped glass slides into a tissue culture dish. Do this carefully to avoid leaking of the polymer solution. Put the dish into the UV oven.

Step10: Turn on the oven. Expose the glass slides containing polymer solutions to UV light for 20 mins.

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

Step12: Cool down the specimen. Remove the glass slides to get the polymer membrane. Cut the membrane to desired size if necessary.

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%

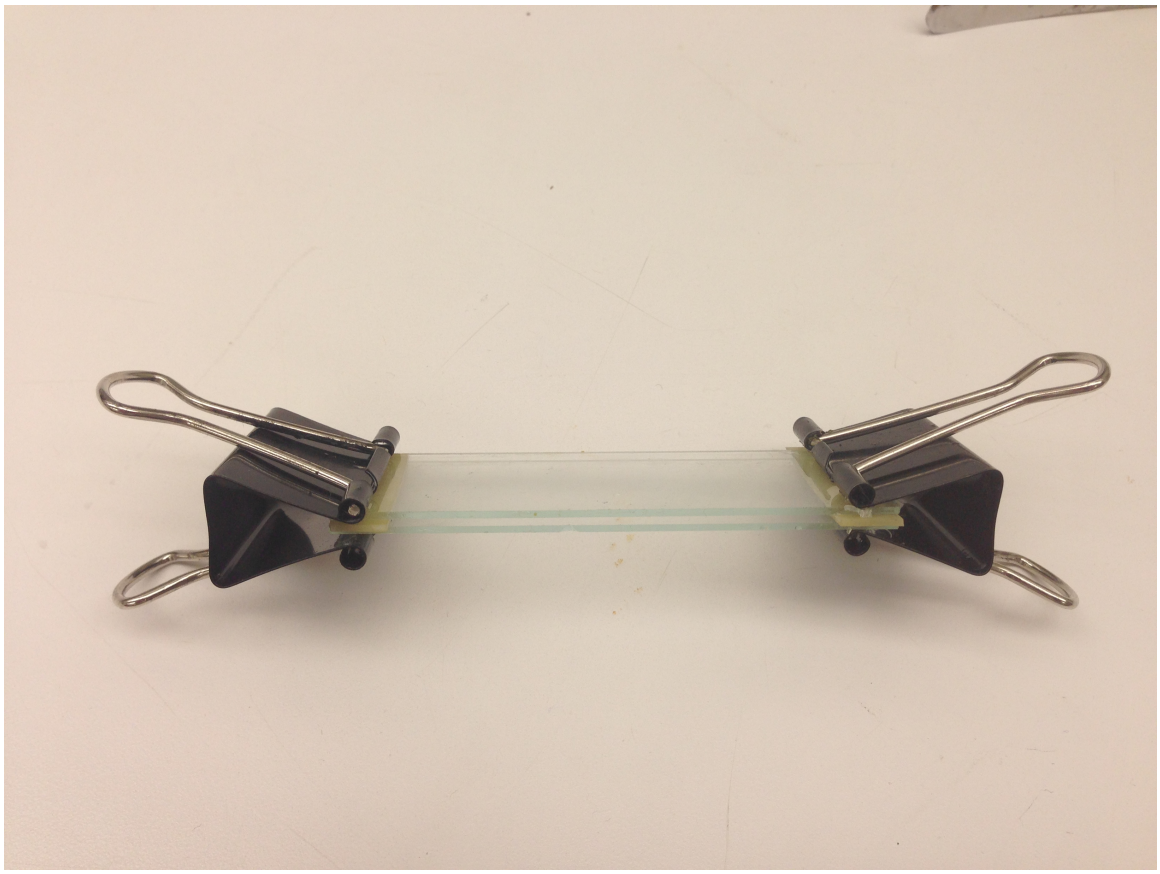

Fig1. Clipped glass slides separated by rubber spacers