

# Polymer blend sample preparation

TN01062 Preparation of polymer samples available from the *Lateral force, Phase imaging, and Force modulation* mode kits

## Introduction

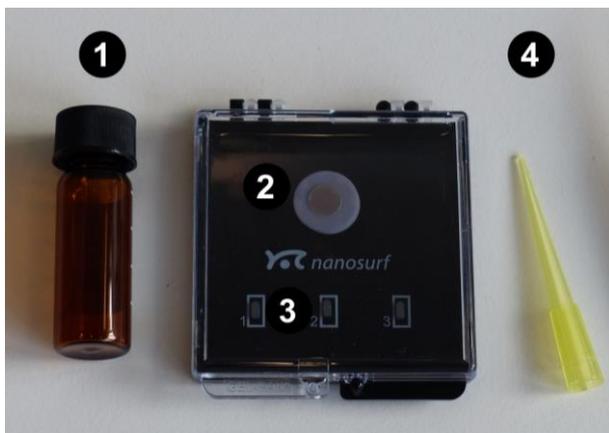
The SBS-PMMA, SBS-PS, and SBR-PMMA samples are blends of polymers dissolved in an organic solvent. After spreading the solution on a flat substrate and upon evaporation of the solvent, the components of the polymer blends separate in to well-defined phases.

To prepare the polymer mixture, individual solutions of each of the polymers are prepared. Thereto, the polymers are dissolved in toluene and then a mixture with equal amounts of each polymer is prepared. The different mode kits each contain a glass vial with a different mixture of two polymers. The **Lateral force mode kit** contains a mixture of **SBS** (poly(styrene-butadiene-styrene)) and **PMMA** (poly(methyl methacrylate)), the **Phase imaging mode kit** contains a mixture of **SBS** and **PS** (polystyrene), and the **Force modulation mode kit** a mixture of **SBR** (styrene-butadiene rubber) **PMMA**.

As samples containing SBS and SBR are not very stable over time, the samples should be prepared prior to the experiment. After preparation, the samples can be used for about four weeks, depending on the storage conditions. The samples will change its properties (e.g. the size and distribution of the different phases) during that time.

## Sample kit content

The following items are part of the imaging mode sample kits.



- ❶ Screw-cap vial with polymer solution ( SBS/PMMA, SBS/PS, or SBR/PMMA dissolved in toluene)
- ❷ Mica sample on a sample support
- ❸ Cantilevers (3 pcs)
- ❹ Pipette tip (5 pcs)

## Sample preparation

### Required materials

In order to prepare the samples, a few items are required:

- Pipette tip (included)
- Polymer solution (included)
- Mica substrate (included)
- Forceps (e.g. the one from the AFM tool set)
- Tissue paper (ideally lint-free)
- Adhesive tape
- Latex or nitrile gloves

Optional items

- Ethanol or isopropanol
- Compressed air or nitrogen
- Spin coater
- Micropipette (e.g. 2-20 µl) with appropriate pipette tips

### Procedure

#### ① Note

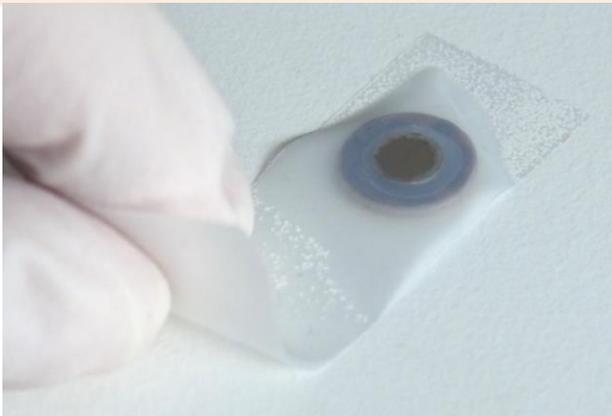
Polymers are dissolved in toluene. Toluene is an organic solvent that is highly flammable and that may be harmful to health. Follow the guide lines given in the MSDS for toluene available from [www..](http://www..)

#### ① Note

In order to prevent contamination of the sample during preparation and to protect yourself from skin contact with the polymers dissolved in toluene, always wear gloves during sample preparation. Just open the screw-cap vial as long as needed.

As for all samples that should be deposited onto a substrate for AFM-based investigations, a clean and flat surface is required. Here, we use a small disc of muscovite mica. The mica disc is glued to a PTFE foil/metal disc assembly to allow better handling. By cleaving off a few layers of mica from the substrate, a clean and flat surface can be created.

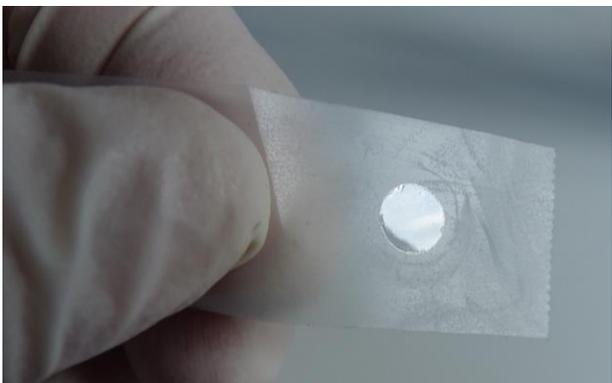
1. Clean the area of the table on which the sample should be prepared on using ethanol or isopropanol, if available.
2. Take the Mica sample out of its box and place it with the mica facing up on the table.
3. Take an approx. 4 cm long piece of adhesive tape and press one end of it with its adhesive side onto the mica. Use your finger to nicely attach the tape to the mica.



4. Use the forceps to hold the metal disc on the table while quickly peeling off the adhesive tape from the mica.



5. A thin layer of the mica should have stuck to the tape. You can see a shiny circle on the adhesive tape. If there is no circle or the circle is not complete (e.g. only half a circle), repeat steps three and four.



6. Make sure that you do not touch the clean mica surface with your fingers. Do not turn the mica sample upside down.

The clean mica surface serves as substrate for the polymer film. The dissolved polymers have to be deposited onto the mica substrate. This can be either done by simple casting a drop of the polymer blend solution on to the mica or by spin coating the polymer.

### **Drop-casting of polymer blend solution**

1. Place the freshly cleaved mica substrate, the vial with the polymer solution, a piece of (lint-free) tissue paper and a yellow pipette tip (or a micropipette if available) in front of you.

2. Open the vial and briefly dip the narrow end of the pipette tip into the solution.

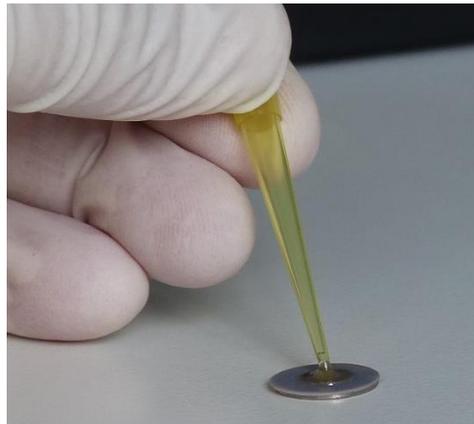
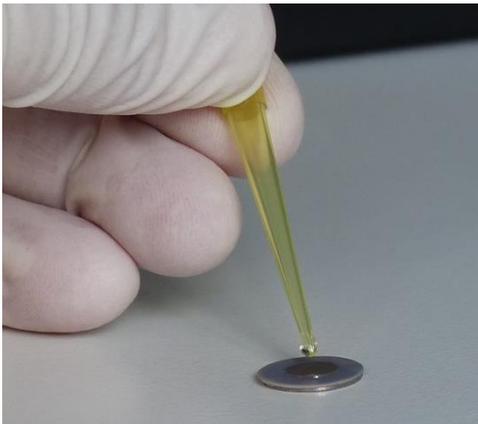


A small amount of the solution will be soaked into the pipette tip.



If you use a micropipette, please aspire 5  $\mu\text{l}$  of polymer solution.  
Close the vial.

3. Touch the mica substrate with the narrow end of the yellow pipette tip. Use a finger to apply gentle pressure to the wide end of the pipette tip, thus pressing out the liquid from the narrow end. If you use a micropipette, release the aspired volume onto the mica disc. Physical contact between the pipette tip and the mica helps to dispense the polymer solution onto the mica surface.



- Pick up the metal disc using the forceps and gently tilt the disc by 45°. Carefully soak off excess solution using the tissue paper by touching the edge of the mica disc with the tissue paper.



#### ① Note

If available, you can use compressed air or nitrogen instead of tissue paper to remove excess polymer solution by blow-drying.

- Leave the sample dry for another minute to make sure all solvent has evaporated.

### Spin-coating with polymer blend solution

Instead of drop casting the polymer film onto the mica surface, the polymer film can also be deposited using a spin coating device. This usually results in flatter and thinner polymer films.

#### ① Note

When using a spin coater, follow the instructions and safety guidelines provided by the spin coater manufacturer.

- Place the metal disc with the mica in the spin coater.
- Place the vial with the polymer solution and a yellow pipette tip (or a micropipette if available), in front of you.
- Open the vial and dip the narrow end of the pipette tip into the solution. A small amount of the solution will be soaked into the pipette tip. If you use a micropipette, aspire 5  $\mu\text{l}$  of polymer solution.
- Remove the pipette tip from the vial and close the vial.
- Touch the mica substrate with the narrow end of the yellow pipette tip. Use a finger to apply gentle pressure to the wide end of the pipette tip, thus pressing out the liquid from the narrow end. If you use a micropipette, release the aspired volume onto the mica disc.  
Physical contact between the pipette tip and the mica helps to dispense the polymer solution onto the mica surface.
- Immediately turn on the spin coater for a few seconds.

#### ① Note

When using a spin coater and a micropipette, you can also turn on the spin coater before depositing the polymer solution onto the mica disc. Thus, the polymer solution can be applied to the rotating disc. Depending on the rotation speed, differently sized structures may form. The time that the solvent is present during the phase separation process of the polymers influences the shape/size of the resulting polymer phases.