

Fabricating Micron-Sized Devices for Measuring Magnetic Properties

CNF Project Number: 598-96

Principal Investigator(s): Daniel C. Ralph

User(s): Maciej Olszewski

Affiliation(s): Laboratory of Atomic and Solid State Physics (LASSP), Cornell University
Primary Source(s) of Research Funding: Defense Advanced Research Projects Agency (DARPA),
National Science Foundation (NSF)

Contact: dcr14@cornell.edu, mwo34@cornell.edu

Website: <https://ralphgroup.lassp.cornell.edu/>

Primary CNF Tools Used: Zeiss Supra SEM, Naby Nanometer Pattern Generator System,
SC4500 Odd-Hour / Even-Hour Evaporator, Oxford 81 Etcher, AFM – Veeco Icon

Abstract:

We present various methods for improving the removal of residual polymethyl methacrylate (PMMA) resist in electron beam lithography (EBL) processes. We explore various methods of PMMA removal, including furnace annealing, plasma cleaning, solvent stripping, and atomic force microscope (AFM) tip cleaning. We compare the various removal procedures by measuring the average roughness of the region using AFM. We report that the best method for removing polymer residue is a combination of solvent stripping and AFM tip cleaning.

Summary of Research:

Van der Waals material heterostructures fabricated from exfoliated crystals have atomically smooth layers, forming pristine interfaces between various materials [1]. This allows for studying new and exotic states of matter as well as improving the understanding of physics in the quantum mechanics regime. One drawback of this technique is the different shapes and sizes of the exfoliated flakes, which compose the heterostructures. Especially in the case of geometrically sensitive measurements, such as Hall conductance, we require a very precise device geometry.

This issue can be resolved by a combination of lithography, etching and metal deposition, however these techniques usually involve various polymers which can contaminate the pristine nature of the interfaces, reduce the electrical mobility in the materials, and introduce noise in the measurements. To try and maintain a pristine nature of the interfaces we explore a various combination of methods that can help clean the residual resist from the materials. We explore various methods

presented in literature, including furnace annealing [2], plasma cleaning [3], various solvent stripping [4,5], and AFM tip cleaning [6].

Initially, we tried using various forms of vacuum furnace annealing and plasma cleaning to remove the remainder polymer residue. These tests however an increase of black spots on the devices which has been reported to be residual amorphous carbon atoms [2,3]. We then turned to trying various wet solvent stripping methods using various combinations of acetone, dichloromethane, glacial acetic acid, and Allresist AR700-61.

For each process we ran the same e-beam lithography process on the Naby Nanometer Pattern Generator System (NPGS) using a polymethyl methacrylate (PMMA) bilayer resist, after developing we either etched the devices using the Oxford 81 Etcher or deposited metal using the SC4500 Odd-Hour Evaporator. After the etching and/or metal deposition the devices were soaked in various solvents for varying amounts of

Process Type	RMS Roughness (pm)	Mean Roughness (pm)
Allresist	367.05	303.23
DCM and Acetone	632.43	426.76
Toluene	3047.25	2237.50
Acetone	649.40	524.50
Acetic Acid	1150.23	900.01

Figure 1: Table of the surface roughness of the device region after stripping in various resists. The values are obtained by measuring the roughness across many devices.

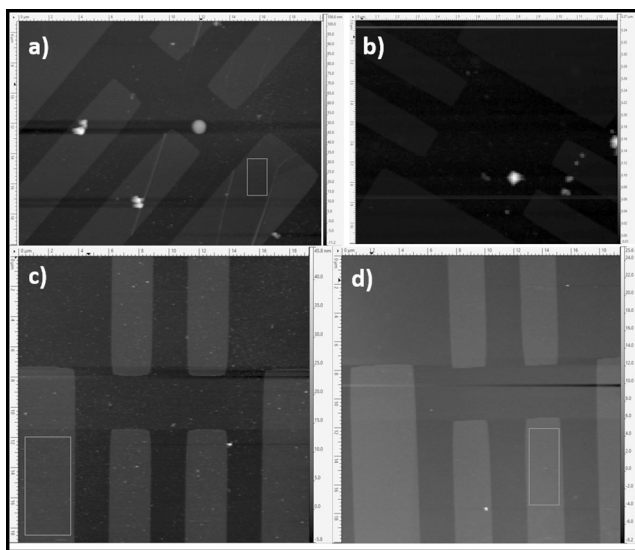


Figure 2: An AFM topography scan of a sample device after a) acetone stripping, b) acetone/dichloromethane stripping, c) acetic acid stripping, d) acetone/Allresist stripping.

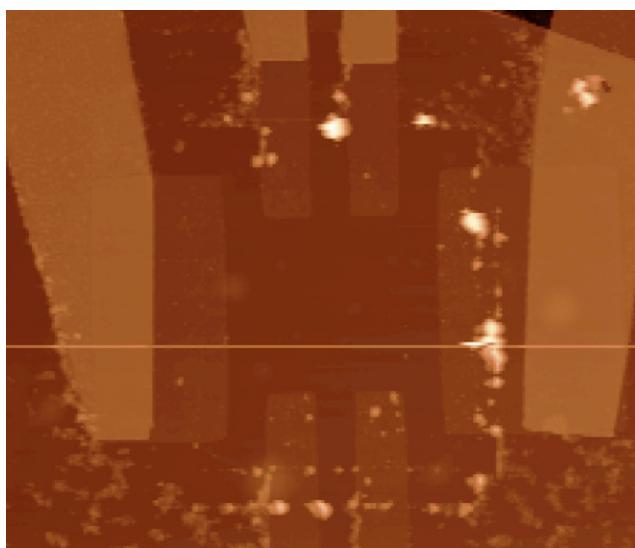


Figure 3: An AFM topography scan of a sample device after the AFM tip cleaning procedure done after acetone stripping.

time. The roughness of the devices was then measured using tapping mode AFM, sample scans for various solvents are shown in Figure 1. Further we analyzed the roughness for various regions and compiled the average roughness value for different solvent methods in Figure 2. We were able to conclude that soaking in Allresist was the best solvent at removing PMMA residue from the device areas.

Finally, we attempted to clean the devices using an AFM tip. This was done by taking an AFM tip in contact mode and dragging it over the surface of the device moving the PMMA residue around. As shown in Figure 3 this causes the PMMA residue to move around however it does not remove the resist but simply relocates it to a different region on the chip. This process proved to be by far the best, often yielding roughness of less than 300 pm.

In summary, we showed that using various solvents for stripping can improve the removal of residual PMMA, however AFM tip cleaning is by far the most efficient method of removing the polymer.

This research allowed us to determine the most optimal method of removing the residue from our devices and improving the interfaces in the heterostructures.

References:

- [1] Castellanos-Gomez, A., Duan, X., Fei, Z. et al. Van der Waals heterostructures. *Nat Rev Methods Primers* 2, 58 (2022).
- [2] Wang, X., et al. Direct Observation of Poly (Methyl Methacrylate) Removal from a Graphene Surface. *Chem Mater* 29, 5 (2017).
- [3] Cunge, G., et al. Dry efficient cleaning of poly-methyl-methacrylate residues from graphene with high density H_2 and H_2-N_2 plasmas. *Journal of Applied Physics* 118, 12 (2015).
- [4] Honghwi P., et al. Optimized poly (methyl methacrylate)-mediated graphene-transfer process for fabrication of high-quality graphene layer. *Nanotechnology* 29, 415303 (2018).
- [5] Tyagi, A., et al. Ultra-clean high-mobility graphene on technologically relevant substrates. *Nanoscale*, 14, 2167 (2022).
- [6] Chen, S., et al. Tip-Based Cleaning and Smoothing Improves Performance in Monolayer MoS_2 Devices. *ACS Omega* 6, 5 (2021).