

Nikos and Felix recipes for making stacks

Andres' paper on the stamping setup: arXiv version (<https://arxiv.org/abs/1311.4829>) or 2D materials (<http://iopscience.iop.org/article/10.1088/2053-1583/1/1/011002/meta>)

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How to exfoliate 2D materials (21/02/18)

Every 2D crystal has different mechanical properties and hence requires specific tapes and methods. The preparation of the chips is universal: Sonication in Acetone, IPA (5 min each is enough), sonication in Nitric acid (5min), followed by a 2 min O₂ plasma step (at Tepla, O₂ flow:200 sccm, Power:600 W)

▪ Graphene

Graphene is easy to thin down. Scotch tape Magic or Nitto tape works. For a large yield on the SiO₂ substrates Nitto at 50°C or Magic at 100°C for 5 min works very well. Let them cool down for 5 min and then peel-off. With Magic tape the residual is a lot but you can find many clean flakes. Annealing at 400°C in Ar/H₂ atmosphere removes them completely.

▪ Hexagonal boron nitride

Boron nitride can be exfoliated the same way as graphene. Here you need to use 90 nm thick SiO₂ for visibility and with Magic tape can get the thinnest flakes possible. An alternative way is to place the chips on Nitto fully covered with flakes and leave them for more than a week. There are many thin flakes (10-30 nm) and negligible contamination.

▪ TMDCs

TMDCs are the most difficult materials to exfoliate in large area. On SiO₂ is impossible with Nitto as they break in very small pieces. Magic tape is thicker and doesn't bend that easily and is better for these materials. So Magic at 100°C like for Gr or hBN works well. The yield of ultrathin flakes is low though but the area is large enough to make good devices. For high yield exfoliating with PDMS provides many and large are monolayers. Use Nitto to thin down, then to PDMS and finally use the stamping technique for deterministic transfer or just exfoliate on SiO₂ with PDMS. PDMS might leave Silicate contaminants though.

▪ Mica and talc

These two are hydrophilic and they are very easy to exfoliate. With PDMS the flakes are enormous. Alternatively, Nitto or Magic at 100°C works

▪ FeSeTe, black phosphorus, TiS₃.

These materials have been exfoliated with PDMS and the planes are more strongly coupled and therefore ultrathin flakes are not easy. There are not so many efforts to exfoliate them so far.

- Orpiment, getchellite and brucite

With PDMS they thin well. We found monolayer of orpiment. Direct exfoliation on the substrate hasn't been tried so far.

Recipe for making hBN/G/hBN stacks using PPC (21/02/18)

- first recipe: Hot pick-up with PPC (doi:10.1038/ncomms11894)

1. Exfoliate the hBN and Graphene on SiO₂ as explained above.
2. Place the top hBN flake under the Navitar system and put the glass slide with the PDMS/PPC on top
3. Touch with the PPC the substrate and increase the temperature. The PPC will start expanding and covering the flake. Warm up, above 55°C. Then cool down below 40°C and lift-up. the flake along with others should be on the PPC. Tip: If you didn't lift the flake repeat the process but this time heat to 80-90 °C and then cool down.
4. Place the graphene below now. If the hBN is very thin then you won't be able to see it. For this case when you lift up on the previous step, draw where the hBN is and don't move its position after lifting. Ok you go down and before you touch the h-BN to the graphene you raise the temperature at 110°C. The hot air and the deflected slide will make it difficult to align them. Then touch and go down slowly. Once the interface passes the GR/hBN you should let it expand a bit more. Now the interface of Gr/hBN should be without bubbles and blisters. Go to 120°C and then retract. The hBN should stay on top of the graphene.
5. Anneal the GR/hBN stack at 170°C for 15 min.
6. place PPC again on top. Cool down and once you are below 70-80 °C you can touch and let the meniscus expand. Cool down below 40°C and then pick-up. The Gr/hBN stack is on the PPC.
7. Put the bottom h-BN under the Navitar and align the stack.
8. Go down, though warm-up above 50 °C and then cool down to pick-up as in step 3. Tip: for no bubbles again maybe repeat step 4, followed by step 6.
9. Place the whole stack on the desired substrate.

How to make PPC (21/02/2018)

ask Felix. Kidding. Put 15% of PPC in anisole at 50°C while mixing with a fish magnet until it dissolves. That's it.

Recipe for making hBN/G/hBN stacks using PC (04/04/18)

- notes from email exchange Felix-Nikos

1. PC is in the fridge.
2. You use the 2 glass-slide technique. Then place it on a bubble or slide.
3. Keep temp at 110.
4. When in touch go to 130-140 and cool down to 110 again. Exactly like the ppc but higher working

temp.

5. As you cool down to 110-100 the pc retracts and pick everything!

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