Comparative study of graphene TEM sample preparation methods

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Abstract

One of the key points in studying 2D materials e.g. graphene and its derivatives is obtaining clean large-area samples. Moreover, because graphene is a 2D material, its properties can be greatly influenced by its substrate. Transmission Electron Microscopy (TEM) offers one approach for preparing and investigating suspended graphene. Additionally, graphene-based TEM grids benefit from low background noise while at the same time providing firm support to large particles due to its outstanding strength and may be useful as a standalone commercial product [1]. A number of different approaches have been reported for transferring graphene onto TEM grids. The most commonly used method involves fishing graphene with a polymer support (usually poly methyl methacrylate, PMMA) onto a TEM grid and subsequently removing the polymer layer [2]. This produces large area samples but typically results in dirty graphene. Another technique is direct transfer of graphene without any polymer coating [3]. This method results in relatively clean graphene but with a very low yield. We have developed and investigated an alternative method to transfer graphene onto TEM grids, namely polymer evaporation. This is similar to the conventional polymer supported method but involves an additional step whereby PMMA is thermally evaporated under inert/reducing atmosphere. Here a comparative study of direct transfer vs. polymer evaporation is reported. The advantages and disadvantages of each approach are discussed and possible improvements/modifications are outlined. The results suggest that the polymer evaporation method offers higher yield, large area coverage (lateral dimensions as big as ~10μm are attainable) but some polymer residues are detectable. On the other hand, direct transfer has a much lower yield but provides a cleaner surface.

References

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[2] A. Reina, X. Jia, J. Ho, D. Nezich, H. Son, V. Bulovic, M. S. Dresselhaus, and J. Kong, *Nano Lett.*, **9** (2009), 30–35.
[3] W. Regan, N. Alem, B. Alemán, B. Geng, C. Girit, L. Maserati, F. Wang, M. Crommie, and A. Zettl, *Appl. Phys. Lett.*, **96** (2010),113102.

Figures

Fig.1. Schematic representation of polymer evaporation technique

Fig.2. Schematic representation of direct transfer technique

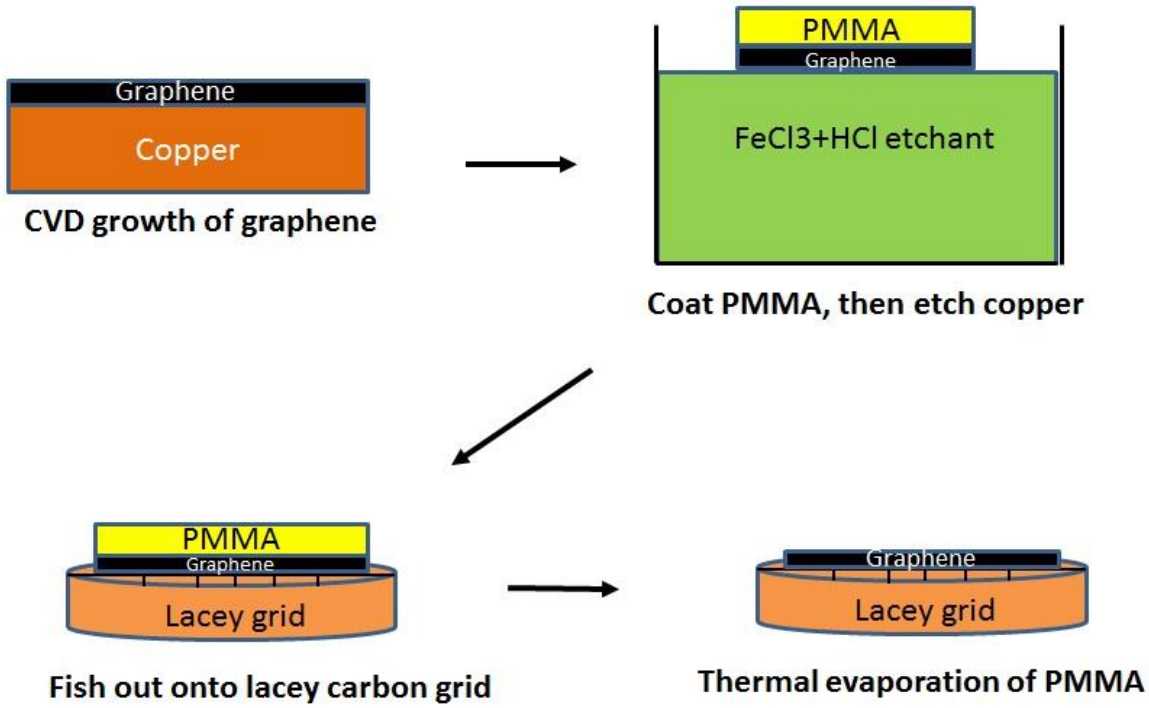
Graphene
Copper
CVD growth of graphene
PMMA
Graphene
FeCl3+HCl etchant
Coat PMMA, then etch copper
PMMA
Graphene
Lacey grid
Fish out onto lacey carbon grid
Graphene
Lacey grid
Thermal evaporation of PMMA

Fig.1.

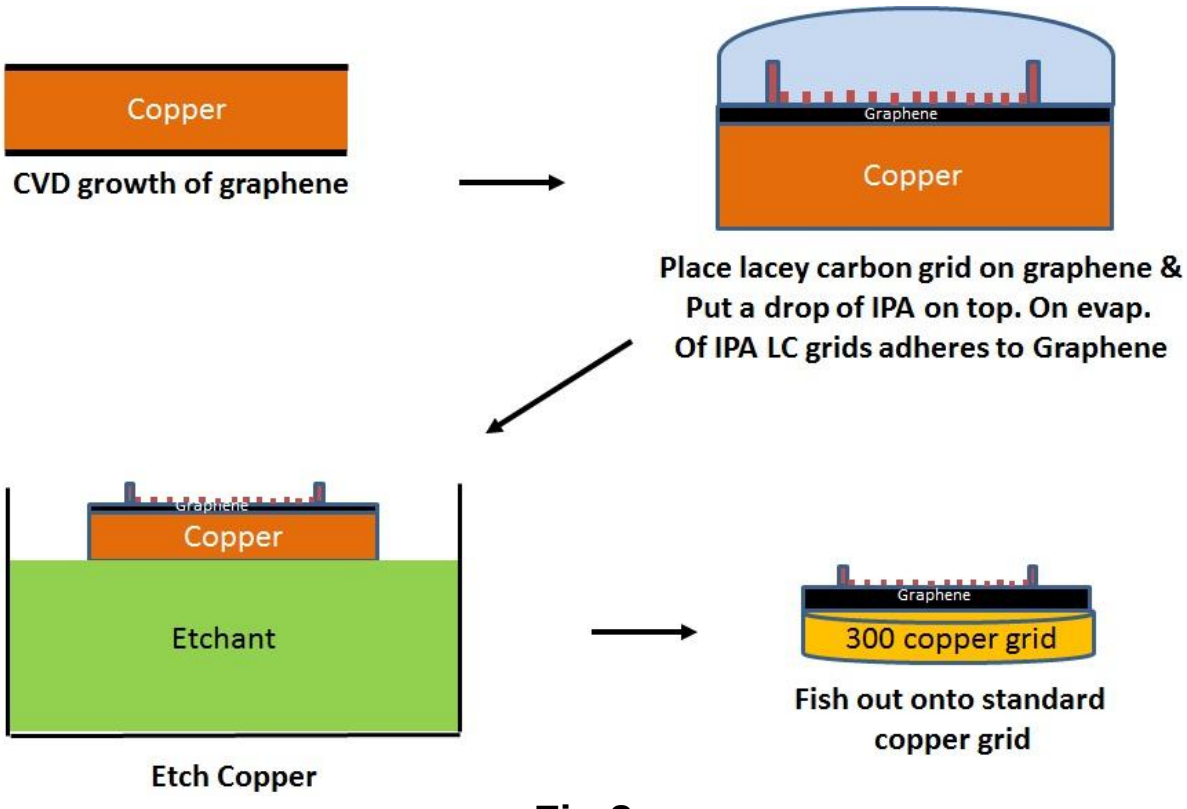
Copper
CVD growth of graphene
Graphene
Copper
Place lacey carbon grid on graphene &
Put a drop of IPA on top. On evap.
Of IPA LC grids adheres to Graphene
Graphene
Copper
Etchant
Etch Copper
Graphene
300 copper grid
Fish out onto standard
copper grid

Fig.2.