Compact CVD system with embedded scanning tunnel microscope for graphene formation analysis

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Abstract

Compact CVD setup with embedded STM was built to provide STM measurements at the same 1μm × 1μm region on the sample surface before and after CVD synthesis. Proposed system can be used for different types of materials deposition, including graphene. CVD parameters for graphene formation were found. The structure peculiarities of graphene film were studied and it was discovered that graphene forms nanobubbles structure on highly polycrystalline Ni surface. The phenomenon of graphene nanobubbles recombination and merging under STM scanning with increased difference of potentials between sample and STM tip was also discovered. This phenomenon combined with build “CVD in STM” reactor leads to ability of fast and effective way to distinguish 2D from 3D structure of deposed material even without taking the sample out of the reactor.

Experimental

The CVD setup with embedded scanning tunnel microscope (STM) was designed using principles described earlier [1, 2]. The exceptional feature of this setup is its ability to provide STM measurements at the same point on a substrate before and after heating (up to 1200°C) cycle. The principal scheme of the setup and sample holding system are represented in Fig.1. The heater is made of 0.6 mm Si wafer located on steel needles. Tips of these needles get into laser produced 10 μm holes in the wafer to avoid spontaneous shifts of tips along the surface during heating. Steel tips are attached to the quarts plate. This construction is chosen to minimize heat flow from the heater to the body of the STM. Standard Si wafer was acted as a heater which was resistively heated by DC current supplied by Mo electrodes. Such configuration allows reaching desired constant heater temperature up to 1100°C by applying adjusted DC current. 50 μm thick Ni foil lies on the Si wafer and can be heated by thermal conductivity up to 1000°C. Tunnel voltage is applied to the Ni sample by thin 10 μm thick wire.

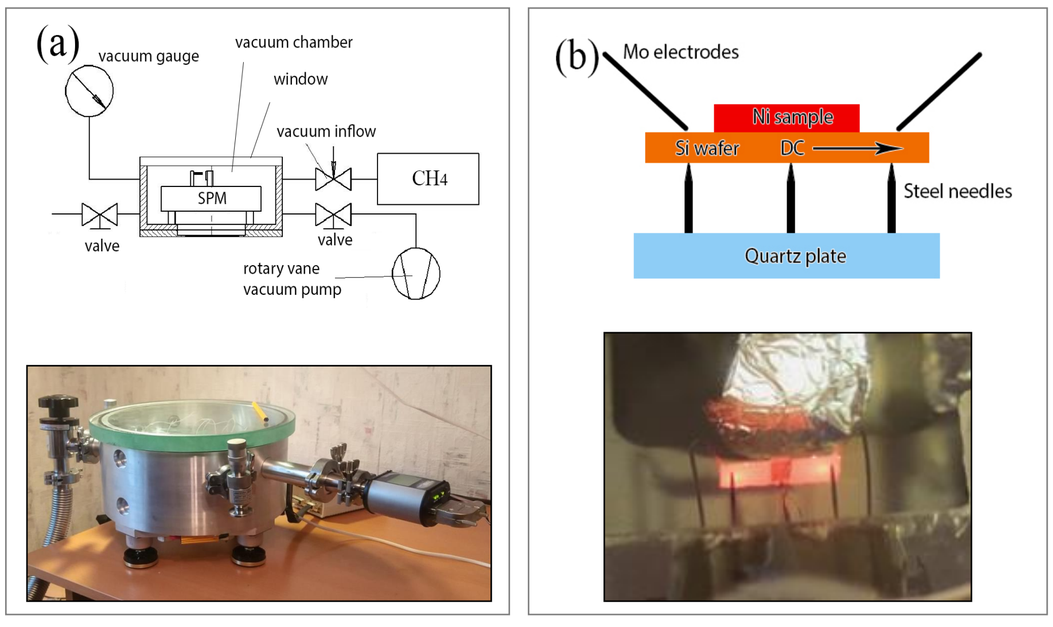

Fig.1. *(a) – scheme and photo image of CVD system with embedded STM; (b) – scheme and photo image of the sample holder with heating system*

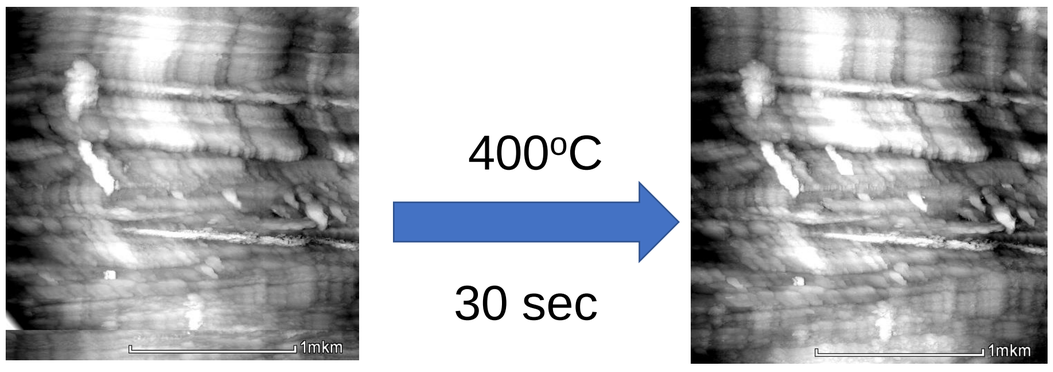

Fig.2. *STM images of Ni surface before and after heating up to 400°C in vacuum for 30 seconds.*

References

[1] A. B. Loginov, R. R. Ismagilov. Designing a scanning probe microscope for in situ study of carbon materials growth processes during chemical vapor deposition. *Journal of Nanophotonics*, 11(2017)032509.

[2] A.N. Obraztsov, A.A. Zolotukhin, A.O. Ustinov, A.P. Volkov, Yu.P. Svirko. Chemical vapor deposition of carbon films: in-situ plasma diagnostics. *Carbon* 41 (2003) 836.

Results and discussion

50 μm-thick Ni foil was heated up to 1000°C in 10mbar methane atmosphere for 15 seconds and then cooled down to room temperature with the rate of 100°C/s. Under such conditions the graphene growth was observed. Typical 3D STM images of sample surface before and after heating are shown in Fig.3. It was found that in some cases graphene forms nanobubbles, which might be easily forced to reorganize or even merge during STM scanning by varying potentials difference between the sample and the STM probe (Fig. 4, 5).

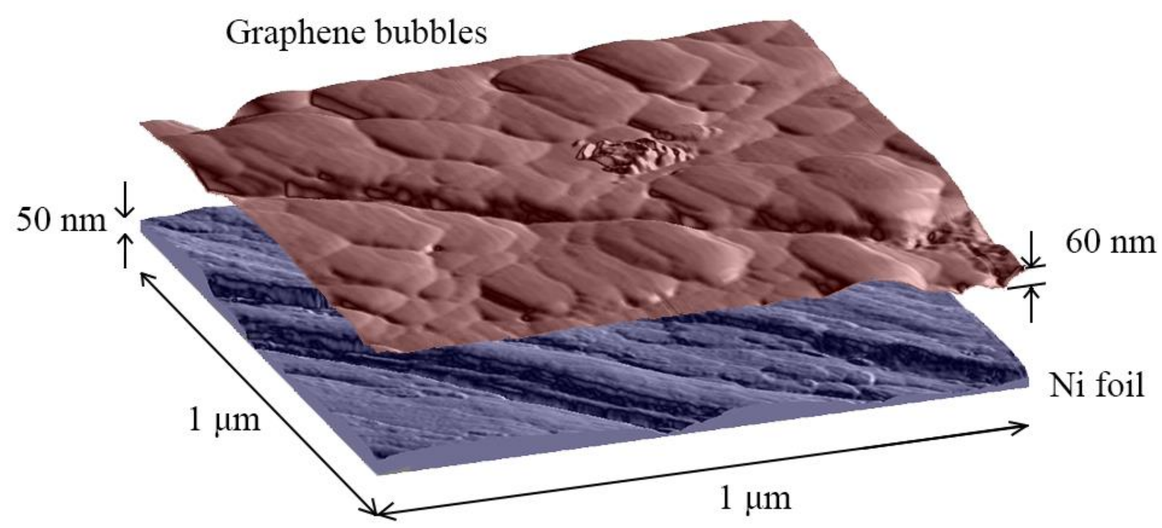

Fig. 3. *3D STM image of the Ni substrate surface before (blue) and after (red) CVD process. Image captures area with 50 nm-deep scratch and shows typical 60 nm nanobubble size, grown on top of relatively flat (R_a=10 nm) Ni substrate*

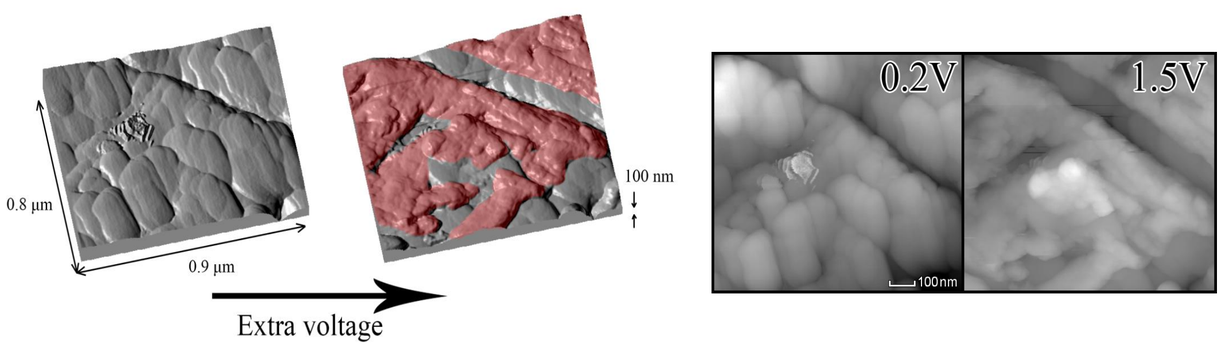

Fig.4. *3D and 2D STM images of difference in graphene nanobubbles morphology under increased STM tip voltage. Changes in morphology are colored red on 3D image.*

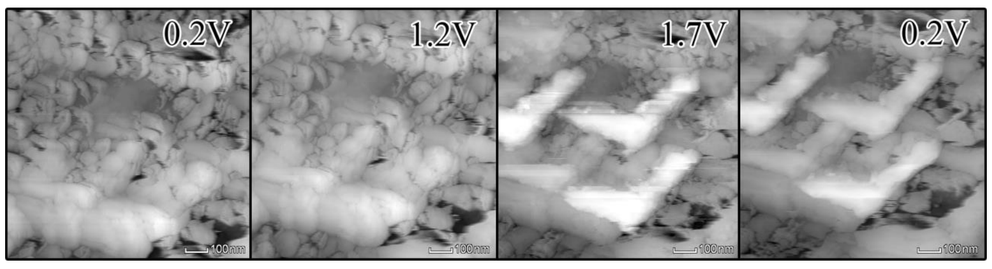

Fig.5. *STM images of graphene nanobubbles morphology with different scanning voltage. Corner-like bubbles appeared under 1.7V STM tip voltage.*

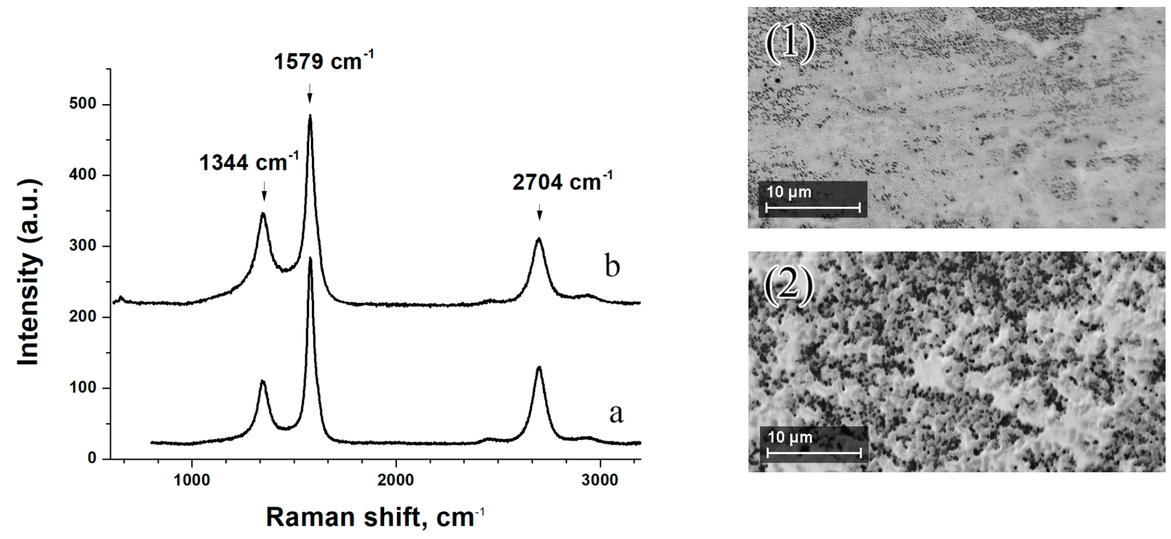

Fig.6. *Raman spectrum and SEM images of obtained films*

Acknowledgements

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