NANOINDENTATION OF HIGHLY ORIENTED PYROLYTIC GRAPHITE*

Wei-Hua Xu [T], Yu Wang and Tong-Yi Zhang**
Department of Mechanical Engineering
Hong Kong University of Science and Technology
Clear Water Bay, Kowloon, Hong Kong, China
[T] Present work address: Institute of Chemistry and Graduate School, Chinese Academy of Science,
Beijing 100080, P. R. China
** Corresponding author, Tel. (852) 2358-7192, Fax (852) 2358-1543, E-mail: mezhangt@ust.hk

ABSTRACT

The mechanical response of a material with the spatial layered structure is of considerable interest in many disciplines such as tribology, geology, optoelectronics, biomechanics, fracture mechanics, and nanotechnology. Nanoindentation tests were performed with a Berkovich diamond tip (R≈80nm) on freshly cleaved ZYA (Mosaic Spread: 0.4±0.1degree) Highly Oriented Pyrolytic Graphite (HOPG) (NT-MDT) surfaces. The used Hysitron Nanoindenter system has a load resolution of 0.1μN and a vertical displacement resolution of 0.1nm. The nanoindentation tests were conducted at room temperature and ambient conditions with a loading-unloading rate of 50μN/s. The spacing between adjacent impressions was large enough to ensure that each impression can be treated as an isolated one. In order to check the fracture morphology, the maximum applied loading force increases from 950μN to 9650μN with a step of 300μN.

Scanning Electron Microscope (SEM) and Lateral Force Microscope (LFM) (Park Scientific M5) were used to check the morphology of the impressions corresponding to the loading curves and the numbers of steps around the impressions caused by different indentation loads. The topography of typical indentation impressions and corresponding height profiles were also measured by contact-mode Atomic Force Microscope (AFM).

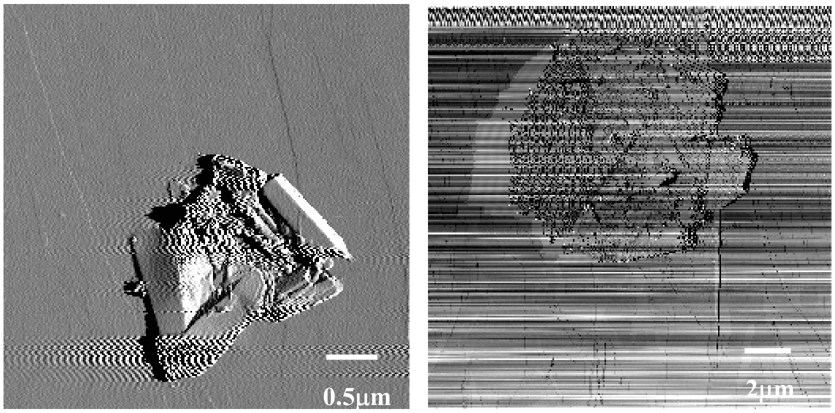


Fig.1 LFM images of the impressions,

(a) for the indentation load of 2150μN and (b) for the indentation load of 9050μN

The Lateral Force Microscopy (LFM) pictures, shown in Fig. 1, indicate that the density of the step numbers near the impression under a high indentation load of 9050µN is obviously larger than that under a low indentation load of 2150µN. Fig. 2(a) is the AFM image of the impression produced by an indentation load of 3050µN, while Fig. 2(b) illustrates the corresponding height profiles. In the rectangle frame of Fig.3 (a) there is a roll-up structure. In order to prove that this structure was formed by the nanoindentation test, we examined the height profile across the length direction from the position near the impression to the position a little bit far away from the impression, as indicated in Fig.3 (b), and the heights are listed in Table I. The height of the roll-up structure decreases with the increasing of the distance from the impression. Such kind of the structure is rolled up by the delaminated graphite layers, which might be caused by the stress field induced by the indentation load.

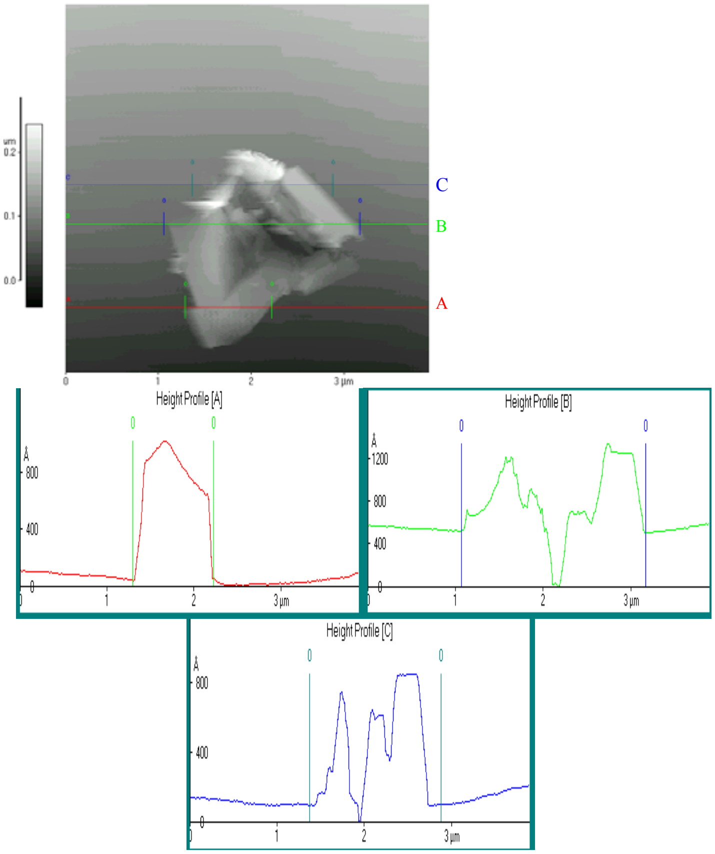


Fig.2. (a) AFM topography(3.91µm x 3.91µm) of the impression caused by the load of 3050µN and (b) the associated height profiles.

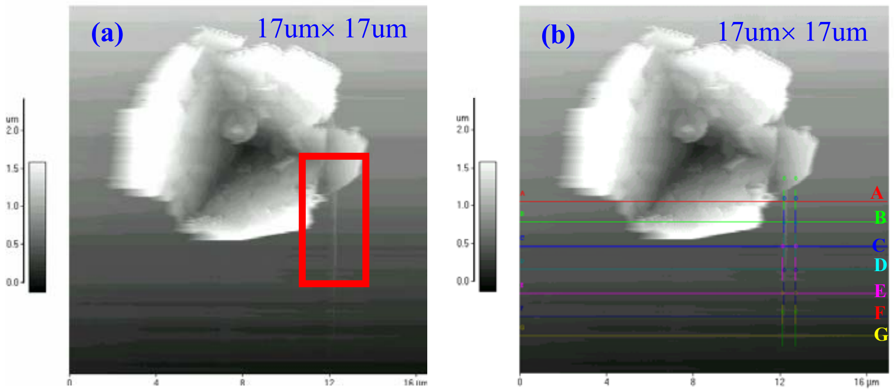
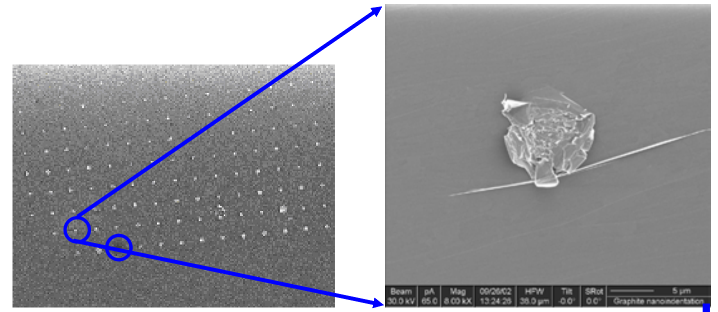


Fig.3. Topography of indentation impression by the load of 8150uN and the associated height profile

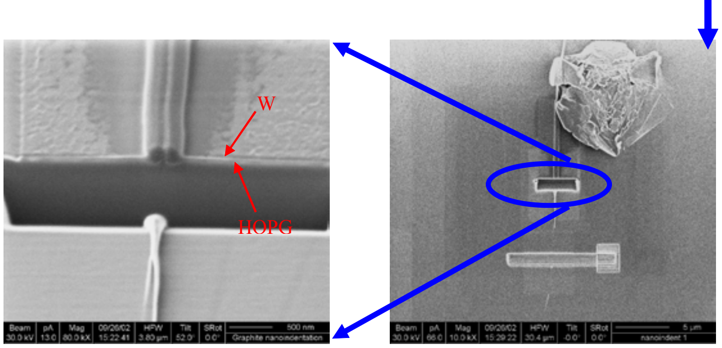
Table I. The height decrease of the roll-up structure near the impression

Line	A	B	C	D	E	F	G
Height(µm)	0.133	0.134	0.103	0.0812	0.0429	0.0215	0.00192



The distance between each impression is 60µm

Use FIB (FEI®) to cut the roll-up structure and view the cross-section



Tilt the sample 45°

Fig.4.The overview impression grid pattern, the SEM impression image under the 8440µN load and the corresponding SEM image of cross-section using FIB to cut the roll-up structure

Fig 4 shows the overview impression grid pattern and the SEM impression image under the 8440μN load. The impression spacing is 60μm. The impression within the lower blue circle is a little bit dark due to the sputtering W super thin film to protect the graphite layers during the FIB cutting process. In order to examine the rolled-up structure, the graphite layer was cut by FIB to reveal the cross-section. It is clear that the roll-up structure is indeed hollow. The diameter of this tube is about 300nm. The potential importance of such kind of phenomena induced by nanoindentation is to form nanotubes with different diameters and lengths by controlling the indentation load, the loading rate, and the indenter tip shape. If the formation of nanotubes near the nanoindention impression can be well controlled, we shall be able to fabricate some special kinds of nanotubes.

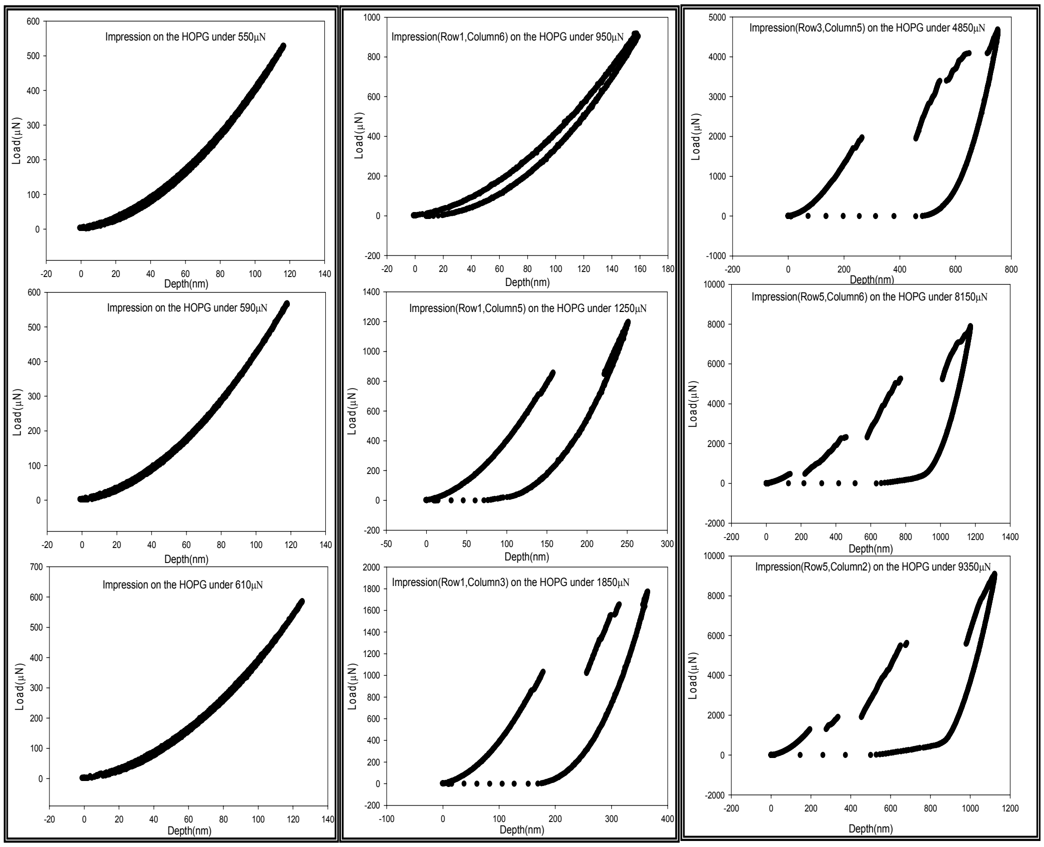


Fig.5.Typical load-unload curve of HOPG with the different applied maximum loads

Fig. 5 shows some typical load-unload force-displacement curves of HOPG with the Berkovich tip. The results indicate that the deformation is purely elastic until the maximum load of about 610μN. When the load is higher than a critical value, the induced stress becomes sufficiently high to break the layers. After the breaking, elastic deformation seems to continue. The mechanical response of graphite stems from the strong interplanar C-C bonds (sp^2) contained in the plane perpendicular to the *c* axis and the weak interplanar van der Waals interactions. With the continuous increasing of the applied maximum indentation load to 1250μN or higher, single pop-in and multiple pop-in phenomena in the force-displacement curves occur during loading. No discontinuities have been observed on the unloading curves. The results show that the critical contact depth for the first pop-in, which corresponds to the fracture of the layers and/or the bonding between the layers, varies in the range of 150~250nm.

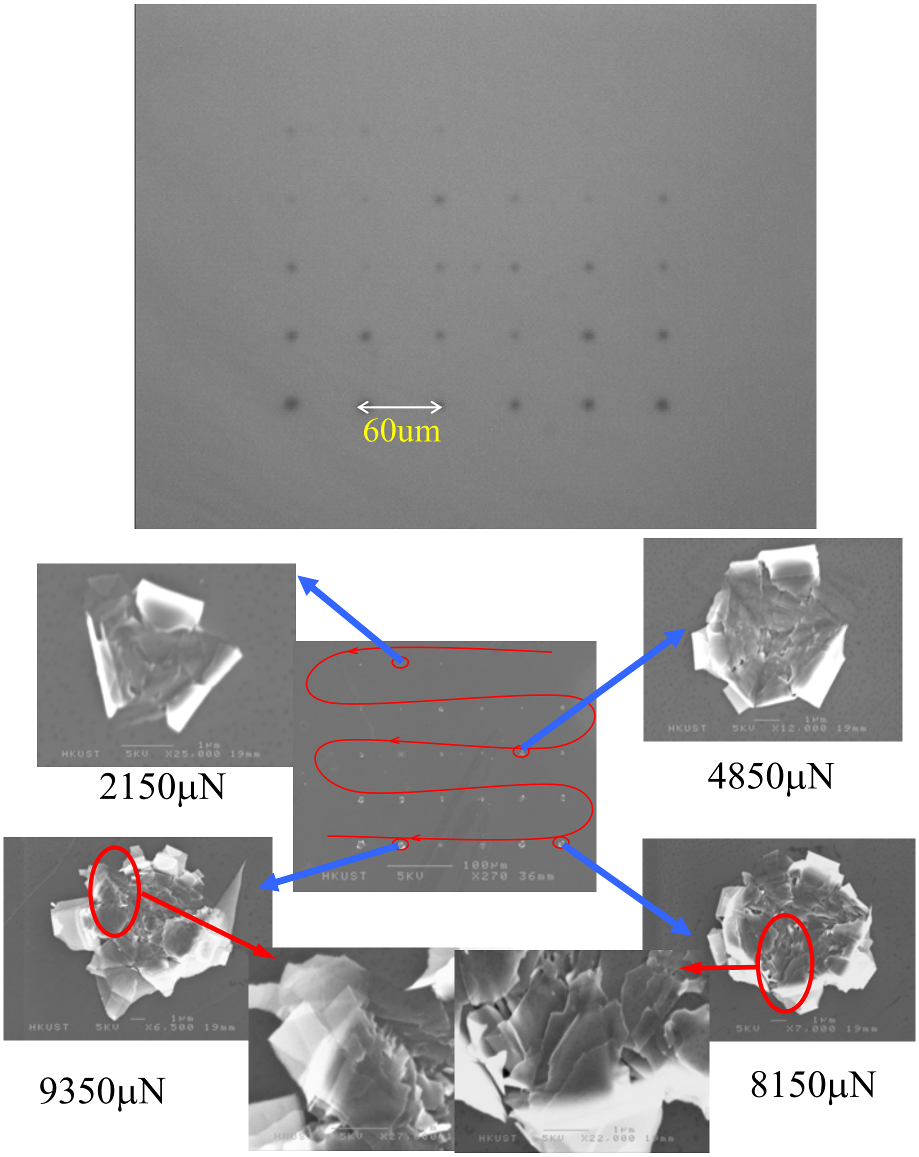


Fig.6. Optical microscope and SEM image of indentation pattern and some typical impressions with the maximum indentation loads of 2150μN, 4850μN, 8150μN and 9350μN.

Fig.6 illustrates SEM indentation pattern corresponding to the above loading-unloading curves. In the image of the rectangular grid indented pattern along the arrow direction, the loading force increases from 950μN to 9650μN with a step of 300μN. It is not easy to find the residual impression with the load smaller than 1250μN. The impressions with the maximum indentation loads of 2150μN, 4850μN, 8150μN, and 9350μN are enlarged also in Fig.6. From the enlarged SEM images, it is observed that sufficiently a high indentation force cause a breakage through a certain number of graphite layers. Actually when this breakthrough occurs, the number of broken graphite layers for a given applied force is not an exact constant, but depends on the sample and the local defect structure of the material. From the load at pop-in, the fracture strength of the layers and/or the bonding strength between the layers may be estimated by the model of elastic field for Hertzian contact including sliding friction for a transversely isotropic material, which will be reported in the near future.

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