

AFM Characterization of Exfoliated Two-dimensional Material

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1 Introduction

This project is to find the characterization of exfoliated 2D material via AFM imaging. However, it is hard to summarize the characterization of these exfoliated material due to the complicate structure exist on the surface of material and also vary with the type of material. One way to solve this issue is to develop a standard definition of characterization. Here in my work, I focus on terraces which is a common characterization shared by all these material. More precisely, the characterization of terraces are determined by their area, and the statistical distribution of these terraces' area can be a good way to illustrate their characterization. In this report, three kinds of 2D material had been measured, graphite, Fe_3GeTe_2 and MoS_2 .

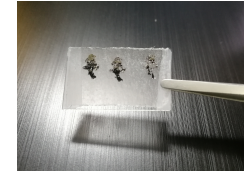
2 The detailed implementations

Firstly, the application of same treatment to all the test sample is required, we exfoliated the bulk material by tape, and then we paste the tape on the glass slide which has double-sided tape on one of its sides, remove the tape, so the second-exfoliated material will hence remain on the glass slide.

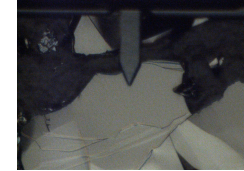
Secondly, we place the sample under the AFM, use the optical microscope to find smooth surface, then randomly choose a point on the surface, approach the cantilever, apply the measurement, repeat it for many times in order to collect enough data.

Thirdly, after processing the images from AFM, we only look for images that can observe terrace on it, and evaluate the area of the terrace or the largest terrace if multiple terraces show up in a same picture.

Finally, we present the data via several histograms, each kind of material will have a histogram correspondingly.

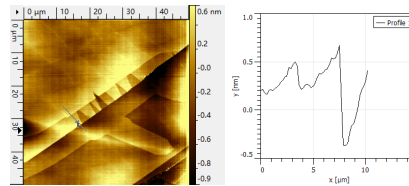


(a)



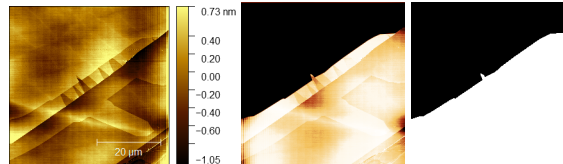
(b)

Figure 1: (a) A exfoliation applied graphite sample. (b) The optical image of a smooth surface via AFM optical microscope



(a)

(b)



(c)

(d)

(e)

Figure 2: Image processing: (a) An original picture from AFM scanning, the material is graphite. (b) The profiles of site 1 in sub fig(a). (c) The picture after processing, which had its background removed. (d) Artificially find and mark the largest terrace in this picture. (e) Use MATLAB to extract the marked area and evaluate its area.

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3 Conclusion and discussion

3.1 Result

I've collected data of 50 samples graphite, 50 samples of MoS_2 , and 7 samples of Fe_3GeTe_2 . The number Fe_3GeTe_2 samples is too small, because this kind of material is not easy to exfoliate, so I only have little of the exfoliated Fe_3GeTe_2 available. And it is either too difficult to find a flat, smooth surface on this kind of exfoliated Fe_3GeTe_2 or the surface are too small. I doubt that maybe it is the quality of this bulk Fe_3GeTe_2 are not satisfying.

The statistical results are shown in Fig[4], from which we can see that all of these histograms appear to have the Gaussian-like outline, despite at some ranges there are bins do not fit pretty well. From the diagrams below we can see that graphite has the largest most possible area (can be found on the exfoliated bulk surface): $1079.1\mu\text{m}^2$, and the most possible area (MPA) of MoS_2 is $928.3767\mu\text{m}^2$, a little smaller than Fe_3GeTe_2 's MPA, which is $946.2078\mu\text{m}^2$. However considering the samples number of Fe_3GeTe_2 , it requires more data to determine the actual MPA of Fe_3GeTe_2 .

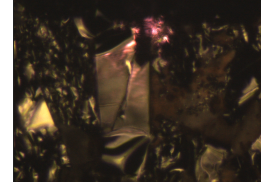


Figure 3: The surface of exfoliated Fe_3GeTe_2 , it is hardly to find a satisfying spot to apply measurement

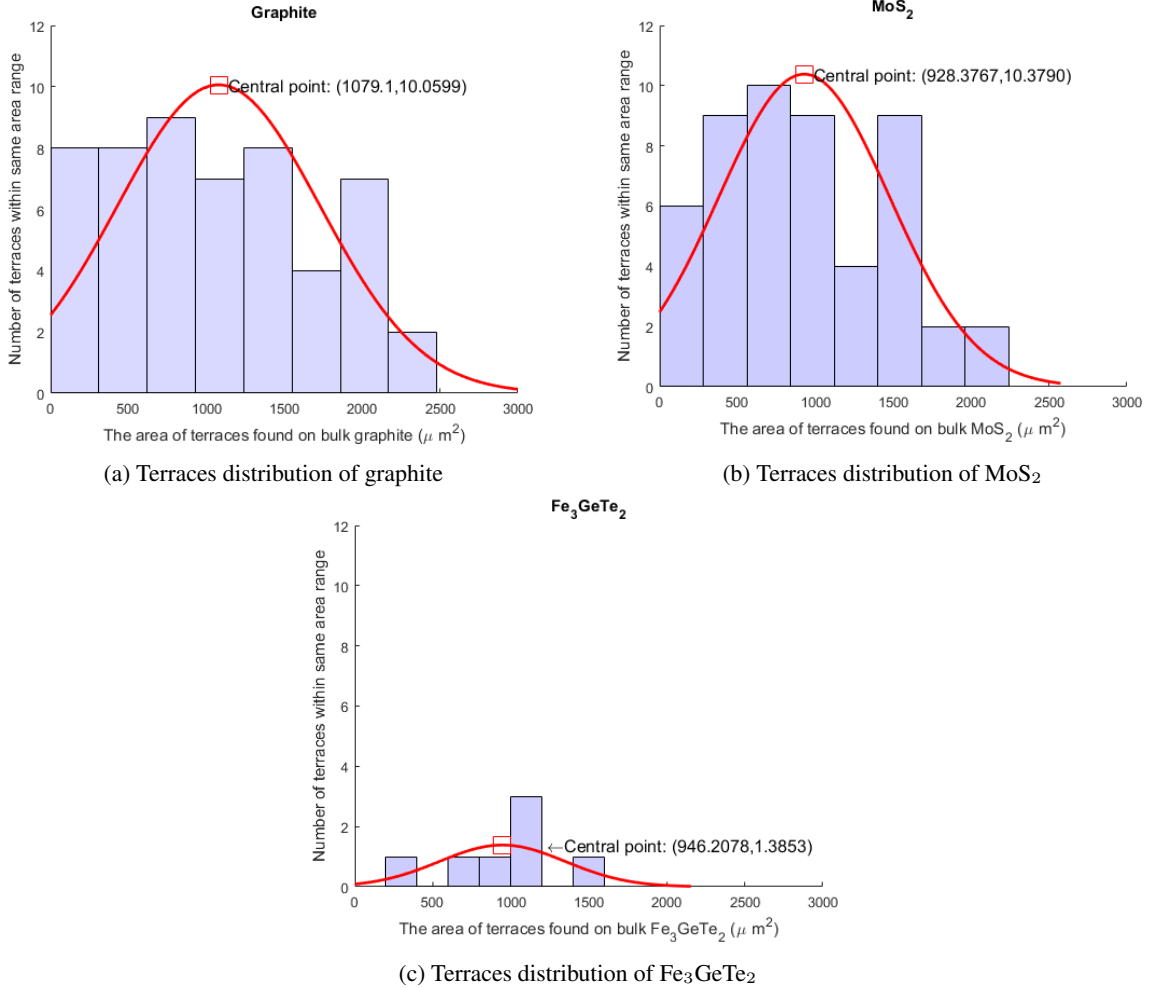


Figure 4: Terraces distribution of three kinds of two-dimensional material

3.2 Conclusion

The distribution of area of exfoliated material in some way reveal that whether the material is easy to exfoliate or not. However, there are another group of data I want to show here, it is the ratio of total measurements to valid measurements. Not all the measurements are valid. For instance, at some images, there is no any terrace can be found, and it appears to have some little bump on the surface. No evidence can support that kinds of images are actually a whole continuous terrace that is too large to find its boundary, we cannot say these are full-size $50 \times 50 \mu m^2$ terraces so the only way is to kick these measurements out of the statistic. What is interesting is that the ratio of total measurement to valid measurement satisfies our common sense of which kind of material is easy to be exfoliated an which kind of material is hard to exfoliated. The table below presents the ratio of these three different kinds of material respectively. Apparently, the ratio from graphite and MoS_2 are at the same level, while the ratio from Fe_3GeTe_2 is way off the former two.

	Valid measurement	Total measurement	Ratio (V/T)
Graphite	53	103	51.46%
MoS_2	51	91	56.04%
Fe_3GeTe_2	7	42	16.67%

Table 1: The ratio of total measurements to valid measurements among three kinds of material

Back to the conclusion, from the histograms above a conclusion can be drawn that most the terraces on these exfoliated material surface have the area between $800 - 1200 \mu m^2$. In addition, the terrace area of graphite looks more diverse than MoS_2 , it will let graphite be more possible to have super large terraces than MoS_2 .

3.3 What can be improved:

Automatizing the process of terraces recognition via MATLAB. To realize the automatic recognition, one possible way is to read the Z height image and NCM phase image at the same time, since NCM phase images always contain distinguished boundaries of terrace even these boundaries and terraces are vague in Z height image. Fig [4] is a good example to illustrate how NCM phase image help in recognizing the terraces and their boundaries of a certain area.

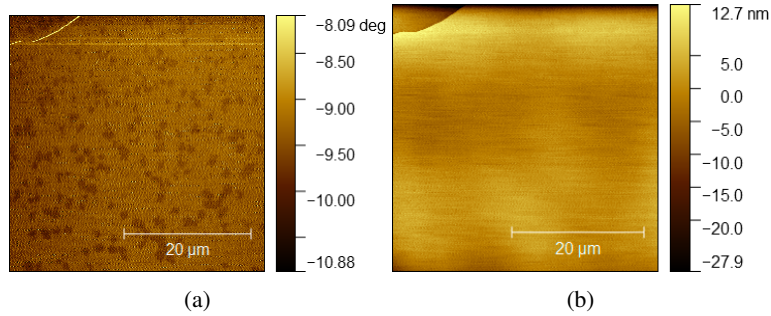


Figure 6: An AFM image of Fe_3GeTe_2 surface: (a) is the NCM phase image of this area. (b) is the Z height image of this area. We can establish the boundary easily by looking on NCM phase image.

However, a few difficulties have to be conquered to achieve such a goal: (1) The ability to process a picture with multiple terraces. While there are many terraces show up in a same picture, even for human, me, it is hard to make the decision which area should be marked to count the area, it could be even harder for the machine to make the decision. (2) The ability to erase the noise due to subtle vibration occasionally occur during scanning. When the AFM is scanning, sometimes it is inevitable to be influenced by some external perturbations, which will create noise signal on the picture, and these noise usually have sharp boundary, like the terraces do. The programme have to be able to determine which is a noise signal and which is a terraces characterization. (3) The ability to distinguish the abnormal structure occur in a picture. On some picture I found some abnormal structure that are not likely to be induced by the noise, and it is also hard to tell what exactly they are. One thing can be sure is that those abnormal structure are not likely to be terraces either.

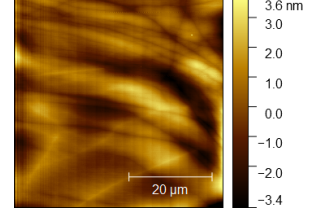


Figure 5: Example of invalid measurement, the material is MoS_2

Improve the quality of sample material. The quality of the material can be crucial to the final result, like I mentioned before, the quality of Fe_3GeTe_2 is not satisfying enough, it's hardly to find a ideal spot to deploy the measurement. A bad quality material may have more disoriented crystal layer, which affect the formation of terraces during exfoliation. For instance, there are another two kinds of material that should have been involved but not, they are Highly oriented pyrolytic graphite (HOPG) and MnBiTe . Both of these two kinds of material have wriggled surface, however, as I known, there is another kind of MnBiTe that has plenty of smooth flat surface because of better manufacture process, but they are not available for me unfortunately.

4 Acknowledgement

I gratefully acknowledge collaboration with Y. Zhang, D. Yue and other fellows of the Zhang Yuanbo Group. Thanks to Zhang Yuanbo Group for providing me such an opportunity to accomplish this report.