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Observation of the local density of states of MoS2 nanosheets using atomic force microscope-assisted tunneling spectroscopy

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論　文　内　容　要　旨

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学位論文の 題　　目	Observation of the local density of states of MoS_2 nanosheets using atomic force microscope-assisted tunneling spectroscopy (原子間力顕微鏡補助による走査トンネル顕微鏡を用いた MoS_2 ナノシートの局所状態密度観察)		

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- 5.3 Determination of the band onsets and doping type
- 5.4 Precision of the STS measurements
- 5.5 Spatial mapping of LDOS on MoS_2 nanosheets

6 Summary and prospectives

I. Introduction

Recent breakthrough in fabrication of graphene, i.e. mechanical exfoliation method, has paved the way for investigation of other layered materials including molybdenum disulfide (MoS_2). Unlike graphene, MoS_2 has an intrinsic bandgap which is critical in device application. MoS_2 nanosheets have extensively been studied in the recent years using the transport technique. For instance, the transport measurement on a monolayer MoS_2-based transistor has demonstrated high current on/off ratio (~10^8) and a sharp subthreshold slope of 74 mV/dec which reflects low energy dissipation. In the transport techniques, one measures the average electronic properties of the material that is typically a few micrometer. Also, the metallic contacts and the back gate oxide layer can affect the electronic properties. Therefore, to avoid the above mentioned unwanted effects and investigate the local electronic properties, a local probe technique such as scanning tunneling spectroscopy (STS) is favorable. STS can be done using scanning tunneling microscope (STM). However, the typical STM is not suitable for STS studying of exfoliated nanosheets of layered materials. Because the combination of the substrate and the nanosheet forms a not atomically flat surface on which STM becomes slow (due to decay length of tunneling current, which is of the order of a few angstroms). Moreover, STM is limited to conductive surfaces meaning that it cannot easily be applied on back-gated devices, e.g. nanosheets deposited on Si/SiO_2 substrates. In contrast, atomic force microscope (AFM) has a larger decay length (~ nm) than STM. Beside it is applicable on insulating substrates as well. In the first part of this work, a technique of atomic force microscope (AFM)-assisted tunneling spectroscopy under ambient conditions is proposed which is suitable and efficient for studying the local electronic properties of exfoliated layered materials such as MoS_2. Moreover, in this method, the vertical thermal drift (VTD) which induces instability between the probe and the sample in room temperature STS measurements is actively compensated by AFM feedback. The second part of the results focus on application of the above mentioned system to investigate the doping type of the MoS_2 nanosheets.

II. Methodology

In this work, an AFM is modified in order to perform scanning tunneling microscopy (STM) and spectroscopy (STS) as well. AFM is used to make a fast and efficient access to the targeting nanosheet and STS is utilized to probe the local density of states (LDOS) and their spatial evolution. The vertical thermal drift is also actively compensated during STS measurements.

III. Results and discussions part 1

The AFM image taken in the relatively large area of 10 × 10 μm^2, on a freshly cleaved highly ordered pyrolytic graphite (HOPG) which includes a maximum surface corrugation of 11

nm. The image was recorded at the scanning speed of 15 µm/s for a total time of 5.6 min. If STM was used to scan the same area, the scanning speed (total scanning time) should have been reduced (increased) at least three orders of magnitude to not get a tip crash. After scanning the large area, we selected a smaller area of 0.5×0.5 µm^2 and performed STM imaging.

Simultaneous measurements of cantilever deflection and the tunneling current while the STM feedback loop (FBL) switched into AFM-FBL were performed. We found that switching the FBL does not change the deflection and hence the tunneling current remains the same. Moreover, after switching the FBL, the tunneling current was maintained for >70 s, indicating that AFM FBL compensated for the vertical thermal drift. In the case where no FBL is used (in typical STS measurements) the tip might crash to the sample (in room temperature measurement) within a few seconds. Finally, we conducted AFM-assisted single tunneling spectroscopy on HOPG using a lock-in technique. The obtained dI_t/dV_s curve shows the typical 'V-shaped' characteristic of HOPG. Moreover, the observed smooth curve implies a fairly high signal-to-noise- ratio and no tip crash in the long spectroscopy time.

IV. Results and discussions part 2

The AFM-assisted STS employed to study the doping type of MoS_2 nanosheets. STS measurements reveal both n- and p- type behavior of the nanosheets. The LDOS measurements performed on several nanosheet with different thickness. By comparing the onset of valance and conduction bands [(VB) and (CB)] the doping type of nanosheets were determined. In summary, the thinner nanosheets with the thickness less than ~ 14 nm exhibit n-type while the thicker ones (thicker than ~ 14 nm) show p-type behavior.

V. Conclusion

In summary, an atomic force microscope-assisted tunneling spectroscopy method was developed. Its performance first was demonstrated on the HOPG as a reference sample. Alternating AFM and STM measurements on this sample achieved using a conductive cantilever under ambient conditions. The access to both AFM and STM signals enables us to perform AFM-assisted tunneling spectroscopy. In this method, we navigate the tip to a spot/area of interest on wide and non-atomically at samples by AFM prior to spectroscopy. The active compensation of the vertical thermal drift for stable tunneling spectroscopy measurements was successfully achieved using AFM feedback. AFM-assisted STS is appropriate for the study of local electronic structures on layered material devices. The implementation of AFM-assisted STS was successfully extended to studying of local electronics properties of MoS_2 nanosheets. Point spectroscopy reveals both n- and p-doping behaviors. The thickness dependence of the doping type was observed. The thinner nanosheets exhibit n-type while the thicker ones show p-type behavior.

論文審査の結果の要旨

本論文はAFM（原子力顕微鏡)アシスト型トンネルスペクトロスコピ測定装置の開発と開発した装置を用いた MoS_2（二硫化モリブデン）薄膜の STS（走査トンネルスペクトロスコピ）測定から構成されている。

AFM アシスト型トンネルスペクトロスコピ測定技術は、機械的剥離法で様々な基板の上に付着させた小片の原子層薄膜に対して、室温で高速にナノプローブを近づけ、STS測定を行うために独自に開発したものである。開発した装置では、バンドギャップなどを局所的に測定できるトンネルスペクトロスコピ測定を、ドリフトが大きい室温で安定に実現するために、AFMフィードバックを用いたトンネルスペクトロスコピ測定が実現されている。具体的には、AFM装置を改良してAFM アシスト型トンネルスペクトロスコピ測定を実現するとともに、AFMフィードバックとSTS測定の両立の観点から、硬さが最適なカンチレバーの選択を行っている。AFMを併用することで、小片へのアプローチを通常のSTM装置に比べて桁違いに高速で行うことを可能にし、効率的なSTS測定が行えるようにした点も開発した装置の重要なメリットである。新たに確立した装置の性能は、グラファイト表面を用いてチェックしており、室温でも安定にトンネルスペクトロスコピ測定が実現できることが実証された。この成果は、J. Microscopyに2014年に出版されているが、原子層薄膜以外にもソフトマテリアルなどの室温トンネルスペクトロスコピ測定に広く使用できる有用な技術として、二人のレフェリーからも高い評価を得ている。

さらに、本論文では新たに確立したAFM アシスト型トンネルスペクトロスコピ測定技術を用いて、金薄膜上に機械的剥離法で付着させた様々なMoS_2小片薄膜についてSTS測定を行った。装置の特徴を活かすことで、様々な剥片に効率的にアプローチして、AFM アシスト型トンネルスペクトロスコピ測定を行い、局所的なバンドギャップを求めるとともに、ゼロバイアス、すなわちフェルミ準位の位置から、薄膜がp型的な特性を有しているか、n型的な特性を有しているかを明らかにした。得られた結果は、膜厚の厚いMoS_2がp型的な振る舞いを示すのに対して、膜厚が薄くなるにつれて、n型的な振る舞いになる傾向を示している。さらに、STS測定を二次元的に行うことで、MoS_2のバンドギャップが局所的に変化していることも示した。この変化の原因としては、歪によるバンドギャップの変調や欠陥の影響が考えられるが、本研究で開発した装置を今後活用することで、これらの解明が進むことが期待される。

以上の内容は、自立して研究活動を行うに必要な高度の研究能力と学識を有することを示している。したがって、Amin Vakhshouri君提出の博士論文は，博士（理学）の学位論文として合格と認める。