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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 44935-2024

**Nanotechnologies - Measurement of the number of layers of
MoS₂ flakes - Raman spectroscopy method**

纳米技术 二硫化钼薄片的层数测量 拉曼光谱法

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for standardization work Part

1. Structure and drafting rules for standardization documents" Drafting. Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents. This document was proposed by the Chinese Academy of Sciences. This document is under the jurisdiction of the National Nanotechnology Standardization Technical Committee (SAC/TC279). This document was drafted by: Institute of Semiconductors, Chinese Academy of Sciences, Hebei University, Taizhou Juna New Energy Co., Ltd., Southeast University, Xiamen Kaina Graphene Technology Co., Ltd., Horiba (China) Trading Co., Ltd., Institute of Physics, Chinese Academy of Sciences, Tianjin University, National Nanotechnology Co., Ltd. Science Center. The main drafters of this document are: Tan Pingheng, Liu Xuelu, Lin Miaoling, Li Xiaoli, Ding Rong, Zhang Qi, Ni Zhenhua, Huang Weiming, Shen Jing, Yang Yang, He Qing, Gao Jie and Shao Yue.

As one of the representatives of two-dimensional layered materials, molybdenum disulfide (hereinafter referred to as "MoS₂" unless otherwise specified) flakes have excellent electrical The performance of nano-photonics, optics, mechanics, and thermals has become the key to the continuation of Moore's Law in the new generation of high-performance nano-photonics devices and the post-silicon semiconductor era. The number of MoS₂ flakes has a significant impact on its optical and electrical properties. For

example, a single layer of MoS₂ is directly The multilayer MoS₂ has a significant luminous efficiency and has good application prospects in optoelectronic fields such as photodetectors and photodiodes. The indirect band gap of MoS₂ decreases with the increase of the number of layers. Compared with single-layer MoS₂, the carrier mobility and current density of multilayer MoS₂ decrease with the increase of the number of layers. The higher the number of layers, the more significant the application advantages in electronic devices such as field effect transistors. Therefore, it is very important to quickly characterize the number of layers of MoS₂ flakes. It has important guiding significance for its production and preparation and the development of related products, and is also the basis for in-depth research on the physical and chemical properties of MoS₂ flakes. The foundation and core of its application development. As a fast, non-destructive and highly sensitive spectral characterization method, Raman spectroscopy has been widely used to measure the number of layers in MoS₂ flakes. For example, in the Raman spectra of single-layer and multi-layer MoS₂ flakes, the peak position difference between the high-frequency Raman vibration modes, the E₁2g mode and the A₁g mode, increases with the The peaks of low-frequency Raman vibration modes, i.e., interlayer breathing mode and shear mode, in MoS₂ flakes with two or more layers increase with the number of MoS₂ layers[3]. The position of MoS₂ has a definite corresponding relationship with the number of layers of MoS₂ flakes [4]. In addition, when MoS₂ flakes are prepared on an oxidized silicon substrate (i.e., the surface is oxidized), When the substrate is a single crystal silicon substrate with a silicon dioxide film formed thereon (hereinafter referred to as "oxidized silicon wafer substrate"), the oxide film below The silicon Raman mode peak height of the silicon wafer substrate also shows a monotonic relationship with the number of MoS₂ flakes [5]. By using the number of features, the number of layers in the MoS₂ flakes can be measured. Since MoS₂ flakes prepared by different methods have great differences in crystallinity and microstructure, the existing characterization methods are not effective. In practical applications, one or more methods should be selected based on the crystallinity and microstructural characteristics of MoS₂ flakes. Suitable characterization methods are used to conduct a comprehensive analysis of the number of layers. The preparation of this document will provide scientific basis for the measurement of the number of MoS₂ flakes prepared by mechanical exfoliation using Raman spectroscopy. reliable basis and standard test methods, and promote the application of Raman spectroscopy in the field of nanotechnology and two-dimensional materials industry. Provide technical guidance for the production and research of two-dimensional materials such as MoS₂ flakes.

Nanotechnology. Layer number measurement of MoS₂ thin sheets Raman spectroscopy

Warning. This document involves the use of lasers, which may cause irreversible damage to the eyes. Wear The corresponding laser protective glasses are strictly prohibited to look directly at the laser to prevent the laser from being reflected by optical components and entering the human eye. Safety training.

1 Scope

GB/T 44935-2024 gives the Raman spectroscopy method for counting the layers of molybdenum disulfide flakes. MoS₂ is the best known of the transition metal dichalcogenides, and its properties depend on the layer count in a way that is not gradual: the monolayer has a direct band gap and photoluminesces, the bilayer does not, and the electrical and optical behaviour changes again at three and four layers. Raman spectroscopy counts the layers without destroying the flake, because the separation between the in-plane and out-of-plane vibrational peaks shifts monotonically with thickness over the first few layers. The standard sets out the principle, the instruments and their calibration, the sample preparation including the transfer of the flake, the measurement steps, the determination of the layer number from the spectral parameters, and the test report, with informative annexes giving a schematic of the typical Raman peak parameters, the transfer operation steps and a worked report example. It takes effect on 1 July 2025.

This paper describes a method for measuring the number of MoS₂ flakes using Raman spectroscopy. This document is applicable to the layer number measurement of intrinsic MoS₂ flakes with 2H stacking and in-plane size greater than or equal to 2µm.

2 Normative references

The contents of the following documents constitute the essential clauses of this document through normative references in this document. For referenced documents without a date, only the version corresponding to that date applies to this document; for referenced documents without a date, the latest version (including all amendments) applies to This document.

GB/T 30544.13-2018 Nanotechnology Terminology Part

3 Terms and definitions

The following terms and definitions as defined in GB/T 33252, GB/T 30544.13-2018 and GB/T 40069-2021 apply to this document.

3.1 Terms related to molybdenum disulfide

3.1.1 two-dimensional material; 2D material It is composed of one or more layers, in which the atoms in each layer are tightly bonded to the adjacent atoms in the layer, and have one dimension (i.e., its A material whose dimensions (thickness) are on the nanometer or smaller scale and whose other two dimensions are usually on larger scales. [Source: GB/T 30544.13-2018, 3.1.1.1]

3.1.2 A two-dimensional material composed of a layer of molybdenum (Mo) atoms and two layers of sulfur (S) atoms stacked together. Note

1. In each layer of MoS₂, the S and Mo atomic layers are arranged in a planar hexagonal array, with 6 S atoms distributed around each Mo atom. Each S atom is surrounded by three Mo atoms, which are bonded by strong covalent bonds. The thickness of each MoS₂ layer is about 0.65nm. Note

2. According to the relative orientation of the upper and lower S atomic layers, 1L-MoS₂ can be divided into two types according to the coordination mode. 2H type with triangular prism coordination and 1T type with octahedral coordination. As shown in Figure 1.

13 Graphene and related two-dimensional materials

GB/T 31225 Ellipsometer method for measuring the thickness of silicon dioxide thin layers on silicon surfaces

GB/T 33252 Nanotechnology laser confocal micro-Raman spectrometer performance test

GB/T 40069-2021 Nanotechnology - Raman spectroscopy for measuring the number of layers of graphene-related two-dimensional materials