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WS₂ 纳米薄膜厚度依赖的三阶非线性吸收特性研究

靳延平¹, 卢春辉¹, 赵奇一², 徐新龙¹

(1 西北大学 物理学院 光子学与光子技术研究所, 西安 710069)

(2 西安邮电大学 理学院, 西安 710121)

摘要: 利用液相剥离法制备了 WS₂ 纳米片, 结合真空抽滤技术, 控制上清液体积制备了不同厚度的 WS₂ 薄膜。在此基础上, 使用 800 nm 飞秒激光的 Z 扫描技术表征了 WS₂ 纳米薄膜的三阶非线性吸收特性。研究发现, 制备的不同厚度的 WS₂ 都表现出可饱和吸收特性, 可饱和吸收主要是由于单光子吸收所引起的泡利阻塞效应引起; 随着厚度的增加, 饱和强度基本不变, 调制深度会有所提高, 但是三阶非线性极化率虚部的绝对值和品质因子会降低。这主要是因为较厚的薄膜缺陷更多, 更容易捕获光生载流子, 从而三阶非线性吸收和厚度有一定的依赖关系。可饱和吸收可以用于调 Q 激光器以及锁模激光器, 为 WS₂ 薄膜在超快开关和超快激光的应用提供实验支持。

关键词: 二硫化钨; 三阶非线性; 可饱和吸收; 厚度依赖; Z 扫描

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0 引言

与传统的块体半导体材料不同, 二维过渡金属硫族化合物作为一种新型的半导体材料, 表现出优异的光学和电学特性, 例如超快载流子动力学, 长的载流子寿命, 高的电子迁移率^[1]。因其优异的光电特性, 二维过渡金属硫族化合物已经成功应用在光电器件领域, 例如光电探测器^[2], 发光二极管^[3], 场效应管^[4], 太赫兹宽带发射器件等^[5-6]。作为典型的过渡金属硫族化合物, 单层 WS₂ 为直接带隙半导体(带隙~2 eV), 块体则为间接带隙半导体(带隙~1.1 eV)^[7]。这种带隙随层数可调的特性使得 WS₂ 在强光作用下表现出很多新颖的非线性吸收特性^[8], 例如饱和吸收, 反饱和吸收和双光子吸收等, 为设计光限幅器件和锁模器件等光子学器件提供了实验和理论方面的支撑。

2015 年 DONG Ningning 等^[9]利用 532 nm 纳秒激光研究了入射光强对 WS₂ 纳米片分散液的非线性吸收的影响, 结果显示 WS₂ 纳米片在较低的泵浦光强下表现出饱和吸收特性, 然而随着泵浦能量的增加转变为反饱和吸收。该转变主要与热诱导的溶剂气泡引起的非线性光散射有关。同样在 2015 年 ZHOU Kaige 等^[10]通过控制离心速度实现了不同尺寸的纳米片的有效分离, 在 532 nm 皮秒激光激发下, 小尺寸 WS₂ 纳米片表现为反饱和吸收现象, 然而大尺寸的纳米片表现为饱和吸收现象, 这种尺寸依赖的非线性吸收特性主要源自边缘缺陷增强了激发态的吸收。2020 年, LU Chunhui 等为了研究尺寸对缺陷的影响, 利用 800 nm 飞秒激光照射 WS₂ 纳米片分散液, 观察到随着纳米片尺寸的减小, 非线性吸收特性出现从饱和吸收到反饱和吸收的转变, 证实了小尺寸表面更容易发生氧化, 从而增强双光子吸收^[11]。然而, 目前关于 WS₂ 非线性吸收的研究大部分是在溶液环境下实现测量的, 很容易引起热诱导的非线性散射并阻碍了非线性固体光子器件的应用。因此, 从实际应用的角度来看, 研究大面积纳米薄膜材料的非线性吸收特性变得尤为重要。

本文利用液相剥离技术制备了大尺寸的 WS₂ 纳米片分散液, 通过控制抽滤体积实现了大面积不同厚度的 WS₂ 薄膜的制备。借助原子力显微镜表征平台, 测得制备的 WS₂ 纳米片约为 3~4 nm, 薄膜厚度最小可达

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第一作者: 靳延平(1979-), 女, 工程师, 硕士, 主要研究方向为非线性光学, 超材料设计。Email: yanpinghappy@nwu.edu.cn

通讯作者: 徐新龙(1976-), 男, 教授, 博士, 主要研究方向为超快光学、非线性光学、太赫兹物理。Email: xlxuphy@nwu.edu.cn

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50 nm。利用自主搭建的开口Z扫描系统研究了WS₂薄膜厚度依赖的三阶非线性吸收特性。

1 实验

1.1 WS₂薄膜制备

实验采用液相剥离的方法制备WS₂纳米片,结合真空抽滤技术将获得的少层纳米片均匀堆叠在一起获得WS₂薄膜^[12-13]。通过控制上清液的体积获得不同厚度的WS₂薄膜。合成过程为:称取300 mg的WS₂粉末分散在300 mL的50%异丙醇溶液中,超声破碎仪(Qsonica Q700)在75 W下工作1 h,产生的超声波诱使液体产生气泡,气泡破裂后会产生密集的张力破坏了二维材料层与层之间的范德瓦尔斯力,从而实现块体WS₂的剥离,最终得到不同层数的纳米片分散液。为了避免长时间破碎产生的热量在WS₂纳米片表面发生氧化,在恒温水浴中完成该过程。为了获得单层或少层纳米片,使用高速离心机(Centurion Scientific K241),设置的离心速度为7 000 r/min,成功实现了较厚的纳米片的分离,将获取的上清液静置两天后使用。控制上清液体积(30, 40, 50, 60, 70 mL),进行真空抽滤得到几十至百纳米厚度的薄膜,最后在室温下将薄膜晾干。利用丙酮溶解掉滤膜,将WS₂薄膜样品转移到目标基底载玻片上。

1.2 Z扫描实验装置

实验中利用自主搭建的开口Z扫描技术提取WS₂的非线性吸收参数^[14]。利用钛宝石激光器(Spectra-Physics Spitfire, 800 nm, 35 fs, 1 000 Hz)作为入射光源。聚焦透镜的焦距为175 mm,会聚成束腰半径为35 μm的高斯光束,通过衰减片调节入射光泵浦能量,利用步进电动平移台实现样品在焦点附近移动,利用硅探测器(Thorlabs-PDA100A(-EC))接收透射的光信号。

2 结果与讨论

使用原子力显微镜(Atomic Force Microscopy, AFM)测量制备的WS₂纳米片的厚度,如图1(a)所示,结果

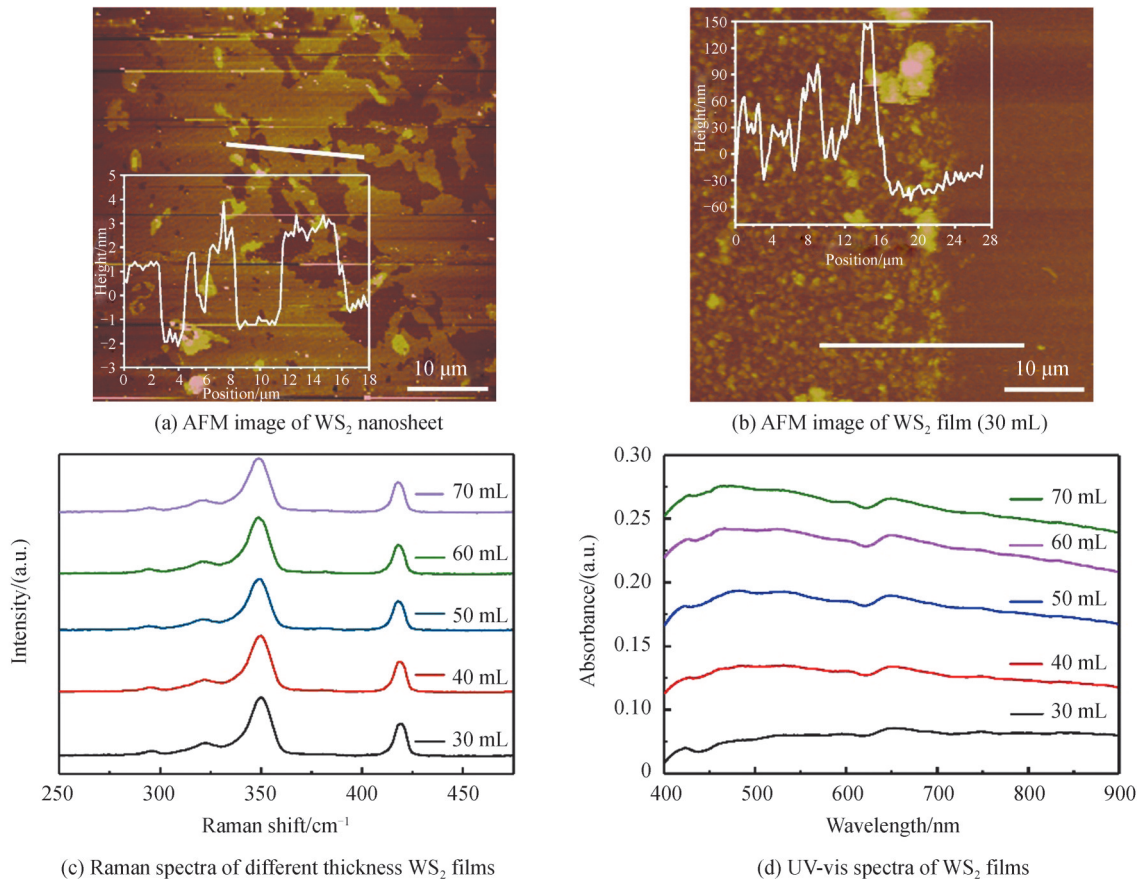


图1 制备的WS₂样品的表征
Fig. 1 Characterization of prepared WS₂ samples

显示纳米片厚度为3~4 nm约4~5层(单层~0.75 nm),单一纳米片的横向尺寸在2~4 μm范围内。如图1(b)所示,测量了抽滤30 mL的薄膜的厚度约为50 nm,抽滤体积为70 mL的薄膜厚度为130 nm,这种纳米片的堆叠更容易在晶界处产生缺陷。测量了不同厚度的WS₂纳米薄膜的拉曼光谱(激发波长为532 nm),如图1(c)所示。不同厚度的WS₂纳米薄膜中均观察到E_{2g}¹(348.76 cm⁻¹)和A_{1g}(~418.84 cm⁻¹)的晶格振动模式,证实了目标薄膜样品被成功制备。使用紫外-可见吸收光谱表征不同厚度的WS₂薄膜的线性吸收特性(400~900 nm),随着厚度增加出现明显的乌尔巴赫(Urbach)带尾,这与局域化的缺陷态增加有关,如图1(d)所示。

使用800 nm飞秒激光作为入射光源,以载玻片基底作为空白对照,研究发现在泵浦能量为700 nJ下并未观察到基底非线性吸收效应,可以排除基底对样品非线性吸收的干扰。图2(a)~(e)为不同厚度(30,40,50,60,70 mL)的WS₂薄膜随泵浦能量的Z扫描实验结果。研究发现随着入射光的能量增强其透射率也不断增加,即为典型的饱和吸收现象。这种饱和吸收是由于激发光子能量(1.55 eV)高于材料带隙引起的单光

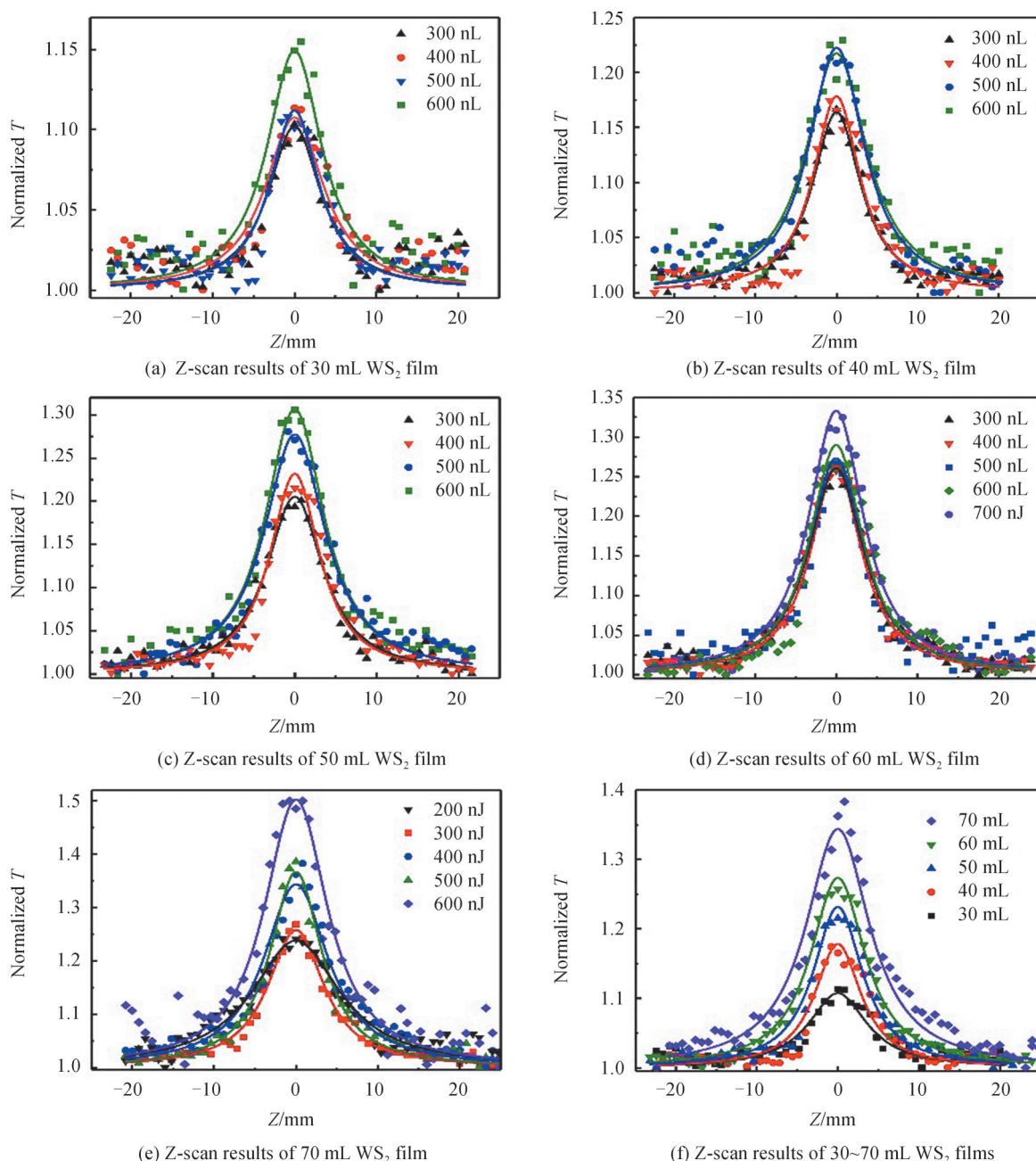


图2 不同抽滤体积下的Z扫描结果

Fig. 2 Z-scan results of WS₂ films with different filtration volumes

子吸收饱和所引起的。为了进一步研究不同厚度薄膜的非线性吸收特性,选取同一入射泵浦能量(400 nJ)下的Z扫描结果,研究发现随着厚度增加,焦点处($Z=0$)透射率变大。为了比较不同厚度的饱和吸收是否有变化,采用式(1)~(2)进行拟合计算^[15]。在此考虑低能量激发机制,样品的吸收系数可表示为

$$\alpha(I) = \alpha_0 + \alpha_{NL}(I) \quad (1)$$

通过归一化透射率曲线计算可得到非线性吸收系数

$$T_{\text{norm}} = \sum_{m=0}^{\infty} \frac{\left[-\alpha_{NL} I_0 L_{\text{eff}} / (1 + z^2/z_0^2) \right]^m}{(m+1)^{3/2}} \quad (m = 1, 2, 3) \quad (2)$$

式中, T_{norm} 和 z_0 分别为归一化透射率以及高斯光束的瑞利长度。 I 、 Z 和 α_{NL} 分别为入射光强、样品的传输距离以及非线性吸收率。 $L_{\text{eff}} = (1 - e^{-\alpha_0 L})/\alpha_0$ 为样品有效厚度,其中, L 和 I_0 为样品厚度以及焦点处光强。 α_0 可以根据朗达-比尔定律,通过可见-紫外吸收光谱得到。计算的样品线性吸收率(α_0)分别为 $1.7 \times 10^4 \text{ cm}^{-1}$, $1.87 \times 10^4 \text{ cm}^{-1}$, $2.14 \times 10^4 \text{ cm}^{-1}$, $2.26 \times 10^4 \text{ cm}^{-1}$, $2.20 \times 10^4 \text{ cm}^{-1}$,如图3(a)所示。

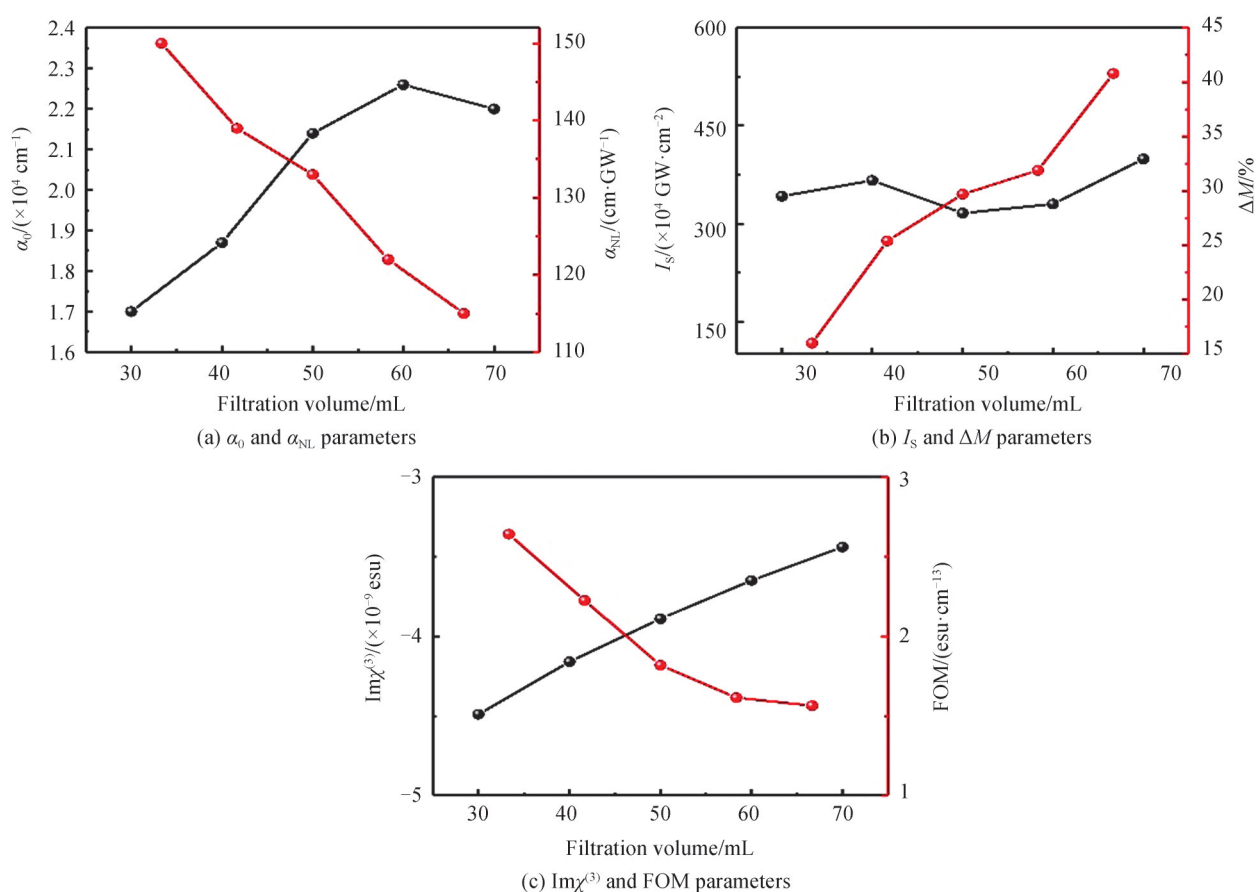


图3 不同抽滤体积下的WS₂非线性参数计算结果

Fig. 3 The calculated nonlinear optical parameters of WS₂ films with different filtration volumes

根据式(1)~(2),通过计算得到30 mL的WS₂的 α_{NL} 约为 -150 cm/GW ,在同等条件下得到40 mL的WS₂ ($\alpha_{NL} \sim -139 \text{ cm/GW}$),50 mL的WS₂ ($\alpha_{NL} \sim -133 \text{ cm/GW}$),60 mL的WS₂ ($\alpha_{NL} \sim -122 \text{ cm/GW}$),70 mL的WS₂ ($\alpha_{NL} \sim -115 \text{ cm/GW}$)。结果表明WS₂具有厚度依赖的非线性吸收,这与传统晶体材料的非线性现象不一致,这可能与样品自吸收效应^[16]以及表面缺陷有关^[17]。本文样品中样品的荧光效率较低,可以排除自吸收效应的影响。从图3(a)中,观察到线性吸收随厚度的增加而增加,这个结果与缺陷态的不断增加有关。较厚的样品缺陷态的增加主要是由于制备的纳米片中的缺陷也会随着层与层的堆叠相应的增加。这种缺陷态的存在会引起更多的非线性散射和能量损失,从而降低非线性吸收^[17]。一定的缺陷的存在会引入缺陷能级,更有利于在红外和近红外的调Q和锁模激光器中的应用。同时饱和光强(I_s)和调制深度(ΔM)也是评价饱和吸收体的重要的性能参数,可以通过式(3)得到。

$$T = 1 - \frac{\Delta M \times I_s}{I_s + I_0 / (1 + \frac{Z^2}{Z_0^2})} / (1 - \Delta M) \quad (3)$$

随着抽滤体积的增加,通过式(3)计算的饱和光强依次为 342 GW/cm², 366 GW/cm², 316 GW/cm², 330 GW/cm², 399 GW/cm², 调制深度分别为 16%, 25.4%, 29.7%, 31.9%, 40.8%, 如图 3(b)。随着厚度的增加,调制深度增大主要来源于材料的吸收增加。较高的调制深度以及相对较低的饱和光强证实 WS₂ 可以作为饱和吸收体应用到光子学器件中^[18]。为了进一步分析样品的三阶非线性极化率虚部(Im χ⁽³⁾),利用式(4)进行计算。

$$\text{Im} \chi^{(3)} (\text{esu}) = \left[\frac{10^{-7} c \lambda n^2}{96 \pi^2} \right] \alpha_{\text{NL}} \quad (4)$$

如图 3(c)所示,得到的抽滤体积为 30 mL 的 WS₂ 的 Im χ⁽³⁾ 约为 -4.49 × 10⁻⁹ esu,在同等条件下得到抽滤体积 40 mL 的 WS₂ (Im χ⁽³⁾ ~ -4.16 × 10⁻⁹ esu), 50 mL 的 WS₂ (Im χ⁽³⁾ ~ -3.89 × 10⁻⁹ esu), 60 mL 的 WS₂ (Im χ⁽³⁾ ~ -3.65 × 10⁻⁹ esu), 70 mL 的 WS₂ (Im χ⁽³⁾ ~ -3.44 × 10⁻⁹ esu)。进一步,薄膜的品质因子(Figure of Merit, FOM),可以从式(5)得到。

$$\text{FOM} = |\text{Im} \chi^{(3)} / \alpha_0| \quad (5)$$

如图 3(c)所示,计算得到体积为 30 mL 的 WS₂ 的 FOM 约为 2.64 × 10⁻¹³ esu·cm,在同等条件下得到体积 40 mL 的 WS₂ (FOM ~ 2.22 × 10⁻¹³ esu·cm), 50 mL 的 WS₂ (FOM ~ 1.81 × 10⁻¹³ esu·cm), 60 mL 的 WS₂ (FOM ~ 1.61 × 10⁻¹³ esu·cm), 70 mL 的 WS₂ (FOM ~ 1.56 × 10⁻¹³ esu·cm)。结果表明 30 mL 制备的 WS₂ 样品更适合应用到光子学器件当中。

2.1 机理分析

由于多层 WS₂ 的带隙接近于块体材料,为了进一步理解 WS₂ 的可饱和吸收特性,使用密度泛函理论利用 VASP 软件计算得到了块体 WS₂ 的能带。图 4(a)是 WS₂ 的能带结构,证实 WS₂ 是间接带隙半导体。在价带顶和导带底之间主要有两种跃迁方式,一种是跃迁能量为 1.6 eV 的 K→K 直接跃迁,另一种以及能量为 0.8 eV 的 Γ→Γ-K 间接跃迁^[19]。根据吸收理论,当一个能量为 1.55 eV 的光子入射到 WS₂ 薄膜时,处于价带中的电子将会被激发到导带上,如图 4(b)所示。由于 WS₂ 能带的间接跃迁方式,电子将从价带中吸收光子激发到导带中。激发的电子占据导带中的低能量状态。随着入射能量的增加,导带内的量子态被激发的电子填满^[20]。根据泡利不相容原理,两个电子不能占据相同量子态,造成光子不再被吸收从而使得透射光不断增加,此时 WS₂ 达到饱和。

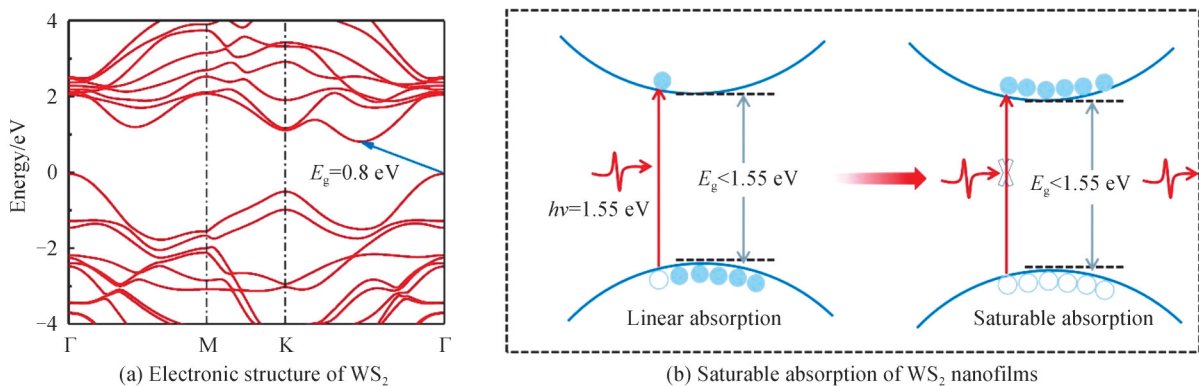


图4 WS₂样品的饱和吸收过程

Fig. 4 The process of saturable absorption of WS₂ nanofilms

3 结论

本文利用液相剥离方法获得了分散均匀的 WS₂ 纳米片,通过控制抽滤体积成功制备了大面积不同厚度的薄膜。Z 扫描结果显示不同厚度的 WS₂ 均表现出可饱和吸收的特性,这主要源自于带隙填充的泡利阻塞

效应。随着厚度的增加,饱和强度基本不变,调制深度会提高,主要源自于样品的吸收随着厚度的增加而增强。三阶非线性极化率的虚部的绝对值和品质因子随着厚度的增加而降低,主要归因于较厚样品的较多的缺陷态使得捕获光生载流子的能力增强。大面积制备的 WS_2 薄膜可作为可饱和吸收体应用于调Q激光器以及锁模激光器,为设计可用于高性能超快开关和超快激光的二维材料提供了参考。

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Thickness-dependent Third-order Nonlinear Optical Absorption of WS₂ Nanofilms

JIN Yanping¹, LU Chunhui¹, ZHAO Qiyi², XU Xinlong¹

(1 School of Physics, Northwest University, Xi'an 710127, China)

(2 School of Science, Xi'an University of Posts & Telecommunications, Xi'an 710121, China)

Abstract: Two-dimensional (2D) layered materials with excellent properties such as strong spin-valley coupling, high current on/off ratio and carrier mobility, have gained great attentions in optoelectronic applications including photodetectors, field effect transistors and light-emitting diodes. Meanwhile, many Novel Nonlinear Optical (NLO) phenomena such as saturable absorption, reverse saturable absorption and two-photon absorption have also been observed. Based on these nonlinear absorption behaviors, ultrafast photonic applications including Q-switching, mode locking and optical limiting have successfully been designed in 2D materials. The device performance is strongly dependent on their thicknesses due to tunable electronic properties with layer. For example, giant two-photon absorption can be observed in monolayer WS₂ film with 1.9 eV while excellent saturable absorption has been demonstrated in multilayer. However, the related research about NLO nonparametric processes with layer thickness, which is crucial to design high performance photonic devices, was seldom reported due to uncontrollable thickness in large-area. Therefore, seeking for an efficient way to produce large-scale and controllable film is highly desirable. To date, mechanical exfoliation have been developed to acquire few-layer nanosheet through exfoliating bulk material layer by layer. However, these methods are low reproducibility and low yield and even limit their application in photonic devices. Compared with mechanical exfoliation method, liquid phase exfoliation was quickly developed and most 2D materials were successfully exfoliated to few-layer or monolayer nanosheet. The nanosheet dispersion can be used to form wafer-scale film by vacuum filtration technique, which provides an effective way to prepare large-area film. Herein, WS₂ nanosheet dispersion was prepared by liquid phase exfoliation and then WS₂ films with different thicknesses were prepared by a vacuum filtration method with different volumes. The atomic force microscopy confirmed the nanosheet length is between 2 and 4 μm and the thickness of thin film is 50 nm for 30 mL in filtration volumes. Raman spectroscopy was used to characterize the sample composition and the typical Raman peaks confirmed the preparation of WS₂ films. UV-Vis spectra was used to obtain the linear absorption and verified the defects in the grain boundary. The third-order NLO absorption of prepared WS₂ nanofilms was studied by a Z-scan technique based on 800 nm femtosecond laser. It is found that all WS₂ with different thicknesses show the characteristics of saturable absorption, which is mainly due to the Pauli blocking effect caused by single photon absorption. To confirm the absorption process, the electronic structure was calculated by density functional theory and the calculated bandgap of bulk WS₂ is approximately 0.8 eV. The photon energy is higher than the bandgap, which verified the single photon absorption occurs. The nonlinear absorption coefficients are -150 cm/GW, -139 cm/GW, -133 cm/GW, -122 cm/GW, and -115 cm/GW from 30 mL to 70 mL with per 10 mL filtration volumes. Obviously, the absolute value of nonlinear absorption coefficients decreases with the increased thickness. This decrease is mainly due to the reason that thicker films with high density defects capture more photoexcited carriers and generate more nonlinear scattering as well as energy loss, resulting in the dependence of third-order nonlinear optical absorption with thicknesses. The existence of certain defects will introduce defect energy levels, which has a great potential in the application of infrared and near-infrared Q-switched and mode-locked lasers. With the increase of thickness, the saturation intensity is basically unchanged, and the modulation depth increases while the absolute values of the imaginary part of the third-order nonlinear optical susceptibility and figure of merit decrease. Our results provide provide experimental support for the applications of WS₂ thin films in ultrafast switchers and ultrafast lasers.

Key words: Tungsten disulfide (WS₂); Third order optical nonlinearity; Saturable absorption; Thickness dependence; Z-scan

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