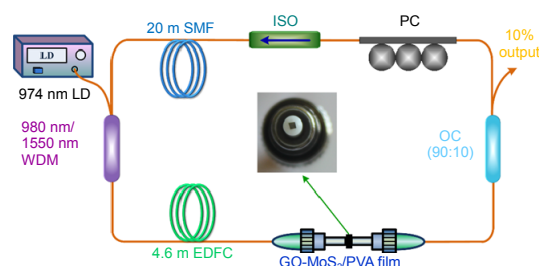


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复合二维材料 GO-MoS₂ 锁模掺铒光纤激光器

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摘要: 为了提升 MoS₂ 可饱和吸收体在脉冲激光器中的稳定性和工作性能, 本论文采用氧化石墨烯(GO)作为胶体表面活性剂, 通过 LPE 的方法剥离出少层 MoS₂, 并进一步开展了少层 GO-MoS₂ 用于掺铒光纤激光器(EDFL)锁模的实验研究。在实验中获得了中心波长为 1558 nm, 重复频率为 7.86 MHz, 脉宽为 1.9 ps 的稳定锁模脉冲激光。当泵浦功率为 60.5 mW 时, 输出功率为 0.48 mW, 脉冲峰值功率为 32.1 W。研究证明, 采用这种方法制备的新型复合二维材料有利于保持少层 MoS₂ 的稳定性, 并且能提高 MoS₂ 可饱和吸收体的损伤阈值, 以获取更大脉冲能量的超快激光。

关键词: 被动锁模; 光纤激光器; 复合二维材料; GO-MoS₂

中图分类号: O436.3

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Composite two-dimensional material GO-MoS₂-based passively mode-locked Erbium-doped fiber laser

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Abstract: In this paper, for improving the performance and stability of MoS₂ saturable absorber, graphene oxide (GO) as colloidal surfactant is used to exfoliate MoS₂ bulk material for obtaining few-layer GO-MoS₂ nano-flakes. Further research on few-layer GO-MoS₂ saturable absorber to mode-lock erbium-doped fiber laser (EDFL) is then conducted. In the experiment, a stable mode-locked pulsed laser is achieved with a center wavelength of 1558 nm, a repetition rate of 7.86 MHz and a pulse width of 1.9 ps. When the pump power reaches 60.5 mW, the output power is 0.48 mW and the pulse peak power is calculated to be 32.1 W. This work shows that the new composite 2D material prepared by this method is beneficial to maintain the stability of few-layer MoS₂ and increase the damage threshold of the MoS₂ saturable absorber for passive mode-locking.

Keywords: passive mode-locking; fiber laser; composite two-dimensional material; GO-MoS₂

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1 引言

锁模光纤激光器因其在材料加工^[1]、光纤通信^[2]、医疗、军事和科学研究^[3-5]等领域的巨大潜力而备受关注,并得到快速发展。这主要归功于它独特而突出的优势,如峰值功率高、脉冲宽度窄、结构紧凑、成本低、光束质量良好和维护方便等^[6]。

目前,锁模光纤激光器的工作原理主要分为主动锁模和被动锁模这两种。主动锁模需要在腔内内置幅度调制器(声光/电光调制器)^[7-8],同时保证周期性的调制信号重复频率为谐振腔基模频率的整数倍。该方法能够实现高且可控的重复率(百 GHz),获得的脉冲激光可调谐性好、稳定度高,是目前光通信系统脉冲光源的选择之一;但激光器的结构往往较为复杂,成本高昂,并且缺乏特殊波段的调制器。近年来,使用可饱和吸收体(saturable absorber, SA)作为强度调制器的被动锁模光纤激光器由于其较为简单的结构和非常丰富的锁模现象^[9]而成为研究的热点,引起了世界各地科研工作者的注意。

传统的可饱和吸收体包括金属掺杂晶体^[10]、半导体可饱和吸收镜(semiconductor saturable absorption mirrors, SESAMs)^[11]和碳纳米管^[12]等。前两者造价昂贵,难以实现全光纤结构,碳纳米管制作较为简单,也易于光学集成,但只能在相对较窄的波段内运行。直到2004年,曼彻斯特大学 Novoselov 等人^[13]利用机械剥离石墨的方法成功制造了少层及单层石墨烯,自从石墨烯被发现以来,以其为代表的二维材料作为 SA 实现光纤激光器的研究层出不穷,除石墨烯^[14-15]外,还包括拓扑绝缘体(topological insulators, TIs)^[16-17]、过渡金属硫化物(transition metal dichalcogenides, TMDs)^[18-19]和黑磷^[20]等。其中,以 MoS₂ 为代表的过渡金属硫化物是新近开发的二维纳米材料。单层的 MoS₂ 由三层原子层组成,两层硫原子夹着一层钼原子,呈“三明治”结构,不仅具有良好的热稳定性和化学稳定性,还具有超宽带(从微波至中红外)的可饱和吸收特性^[21-22],并且制备简单,价格低廉,可有效降低激光器成本^[23]。

自从2013年 Blau 等人^[24]采用800 nm波段的开孔 Z 扫描技术研究了二维 MoS₂ 纳米片的超快饱和吸收特性,这种材料就开始作为可饱和吸收体用于超短脉冲光纤激光器中。2014年,本课题组^[18]将 MoS₂ 可饱和吸收体分别用于掺铋光纤激光器、掺铟光纤激光器和掺铈光纤激光器,获得了1 μm、1.5 μm 和 2 μm 三

个重要波段的大能量调 Q 脉冲激光,从而证明了少层 MoS₂ 的宽带可饱和吸收特性。Li 等人^[25]使用化学气相沉积法制备了少层 MoS₂,并将其作为可饱和吸收体制作全光纤激光器,产生了稳定的锁模孤子脉冲。同年,Zhang 等人^[26]通过将基于 PVA 的 MoS₂ 可饱和吸收体插入掺铟光纤激光器,获得了中心波长位于 1569.5 nm 的锁模脉冲激光,脉冲宽度约为 710 fs,重复频率为 12.09 MHz。此后的实验中,他们通过使 MoS₂ 沉积在微型光纤上制作成 SA,将锁模脉冲的重复频率提高到了 2.5 GHz^[27]。迄今为止,大多数相关的实验研究所采用的可饱和吸收体均是采用 DMF(N,N-Dimethyl formamide)或者 NMP(N-Methyl pyrrolidone)等有机溶剂剥离出少层 MoS₂,但是该方法制备的 MoS₂ 通常很难具有高的载流子迁移率并且呈现出一定的光学/电学不稳定性^[28],从而限制了 MoS₂-SA 的恢复时间和锁模稳定性。而石墨烯/氧化石墨烯(graphene oxide, GO)则具有极高的载流子迁移率、超快光学响应等特点^[29]。

为了提升 MoS₂ 的性能,一种方案是采用石墨烯/氧化石墨烯(GO)作为胶体表面活性剂,通过 LPE(liquid phase epitaxy)的方法剥离出少层 MoS₂。由于 GO 是一种两性材料,利于保持少层 MoS₂ 的稳定性,GO-MoS₂ 的具体制备方法可参见文献^[30]。另外,由于采用 GO 剥离的 MoS₂ 具有较为统一的厚度,并且溶液中不需要有机大分子溶剂,因此有利于提高 MoS₂-SA 的损伤阈值,以获取更大脉冲能量的超快激光。基于此,本文采用 6000 r/m 制备的少层 GO-MoS₂ 作为 SA,并进一步开展了这种材料用于掺铟光纤激光器(erbium-doped fiber laser, EDFL)锁模的实验研究。

2 GO-MoS₂ 材料表征

如图 1 所示为 GO-MoS₂ 的特性表征图。其中图 1(a)、图 1(b)分别为 GO 和 GO-MoS₂ 的 AFM 图,图 1(c)、图 1(d)分别为对应的 GO 和 GO-MoS₂ 的高度测量图。其中 GO 厚度大约为 1.2 nm,长度约为 220 nm,从图 1(d)中可以看出测量的 GO-MoS₂ 厚度约为 2 nm,考虑到单层 MoS₂ 的厚度为 0.7 nm,可以推断出这里异质结的结构为 GO+单层 MoS₂。XRD 图(1(e))表明,和体块 MoS₂ 相比,GO-MoS₂ 具有一个极高的[002]向,并且[103][105]等特征峰明显消失。图 1(f)为 GO-MoS₂ 的拉曼光谱,其中 A_{1g} 峰有明显的红移。综合 AFM 高度图、XRD 和拉曼光谱图可知,MoS₂ 已被成功剥离至 1~3 层。

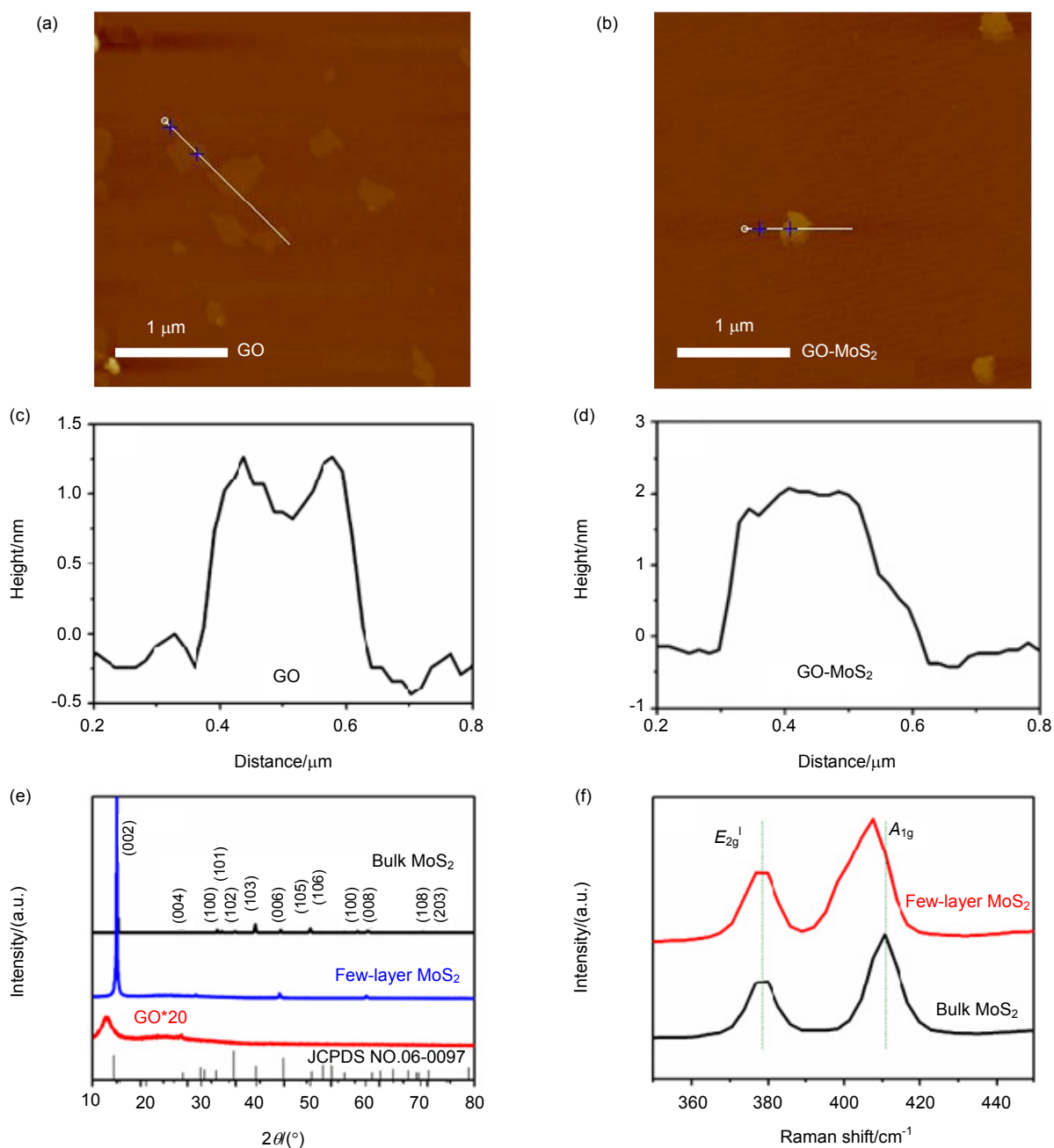


图 1 (a) GO 的 AFM 图; (b) GO-MoS₂ 的 AFM 图; (c) GO 高度图; (d) GO-MoS₂ 高度图; (e) GO-MoS₂ XRD 图; (f) GO-MoS₂ 拉曼图

Fig. 1 (a) The AFM image of GO; (b) The AFM image of GO-MoS₂; (c) The height image of GO; (d) The height image of GO-MoS₂; (e) The XRD image of GO-MoS₂; (f) The Raman image of GO-MoS₂

为了制备光纤兼容的 SA 并进一步比较 GO-MoS₂ 与 GO 和 MoS₂ 的可饱和吸收特性, 实验中分别将 GO-MoS₂、相应量的 GO 和 NMP-MoS₂ 掺入 PVA 中, 三种混合液均保证没有沉淀, 分别取等量的溶液在培养皿中并放入烤箱, 在 60 °C 条件下烘烤约 3 h 直至成薄膜。然后分别撕取同等大小的 PVA-GO、PVA-MoS₂ 和 PVA-GO-MoS₂ 薄膜置于两个光纤连接头之间, 构成

光纤型 SA。

采用平衡双探头探测技术^[18]分别测试了三种 SA 的可饱和吸收特性, 如图 2 所示, PVA-GO 和 PVA-MoS₂ 的透过率分别为~0.915 和~0.90, 两者的综合透过率与 PVA-GO-MoS₂(0.753)相比拟, 在这里可以直接比较三者的可饱和吸收特性。从图中明显可以看出, PVA-GO 和 PVA-MoS₂ 的透过率随着入射功率的变化并未有明

显变化,因此认为此二者不具有明显的可饱和吸收特性。相反地,PVA-GO-MoS₂则表现出较为显著的非线性吸收特性,测得的调制深度为~1.51%,可饱和吸收功率为92 MW/cm²,均为二维材料SA的典型值。这充分表明采用GO剥离的MoS₂比NMP等有机溶剂制备的MoS₂性能更为良好。

3 实验装置

为了进一步验证GO-MoS₂的可饱和吸收特性并且获取1.5 μm的超快激光,首先将上述制备的SA置于EDFL中实现锁模。激光器腔结构如图3所示,974 nm泵浦光通过980 nm/1550 nm的波分复用器(wavelength division multiplexer, WDM)注入到腔内泵浦一段4.6 m的C波段EDF(Nufern, EDFC-980-HP)以提供增益,其色散系数为53.6×10⁻³ ps²/m。将制备的

GO-MoS₂-SA作为锁模器件插入腔中,图3中的小图即为光纤端面上的PVA-GO-MoS₂薄膜。使用一个10:90的耦合器(optical coupler, OC)提供10%的激光功率输出,偏振不相关的光隔离器(PI-ISO)使得腔内激光逆时针运转,偏振控制器(polarization controller, PC)用来调节腔内激光的偏振态,而20 m的单模光纤(single-mode fiber, SMF)(-22×10⁻³ ps²/m)用于补偿色散。整个腔长约为26.2 m,腔内总色散为-0.23 ps²,说明激光器运行在反常色散区。采用光谱分析仪(HP 70951B)测量输出锁模激光的光谱,时域特性则由一个10 GHz InGaAs探测器(Nortel PP-10G-FAC)结合200 MHz带宽示波器(Tektronix TDS2024)测量,频谱和自相关迹分别采用一个频谱仪(Gwinstek GSP-930, 9 kHz~3 GHz)和自相关仪(FR-103XL, Femtochrome Research Inc)测量。

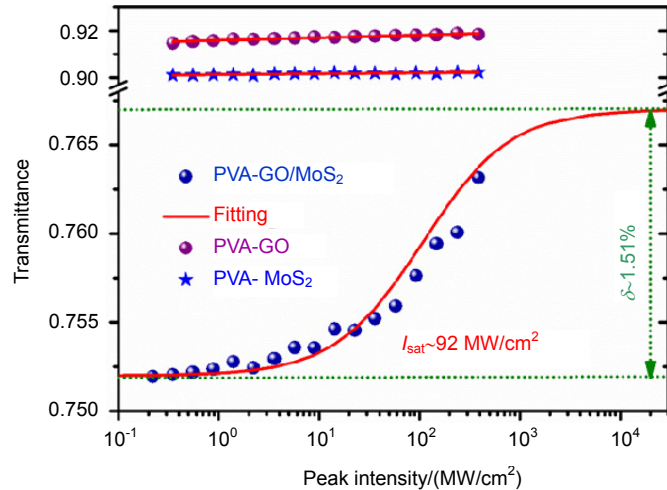


图2 PVA-GO、PVA-MoS₂和PVA-GO-MoS₂的可饱和吸收特性
Fig. 2 The saturable absorption characteristics of PVA-GO, PVA-MoS₂ and PVA-GO-MoS₂

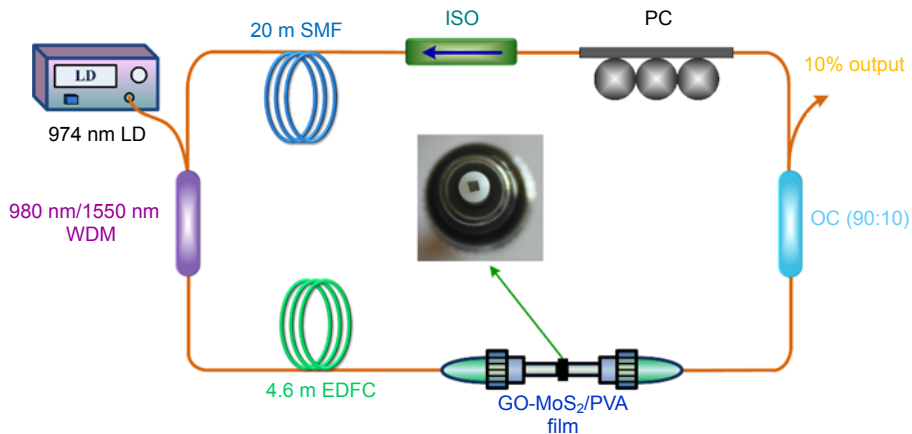


图3 GO-MoS₂锁模EDFL结构图
Fig. 3 The structure of GO-MoS₂-based EDFL

4 数据分析

EDFL的激光阈值为2.3 mW, 当进一步将功率增加至20.5 mW时并适当调节PC, EDFL开始出现稳定锁模。在泵浦功率为57.4 mW时, 我们测量了锁模激光的典型特征。如图4(a)所示为锁模光谱, 中心波长为1558 nm, 由于实验中使用的是C波段EDF, 说明腔内损耗较小, 光谱3 dB带宽为1.9 nm, 具有明显的Kelly边带, 表明是典型的反常色散区锁模孤子, 并且一阶的Kelly边带距离中心频率的波长差仅为4.46 nm, 说明腔内的脉冲具有较高的峰值功率。图4(b)为锁模的时域脉冲序列, 其脉冲周期为~127.2 ns, 相应的重复频率计算为~7.86 MHz, 与腔内往返时间相匹配, 说明其处于基频锁模状态, 另外, 通过观察可以发现, 锁模脉冲的抖动明显小于5%。图4(c)为测量的锁模脉冲自相关迹, 采用高斯曲线进行拟合, 得到脉宽为~1.9 ps, 是典型的二维材料锁模EDFL的脉宽,

采用公式计算得到锁模脉冲的时间带宽积(time-bandwidth product, TBP)为0.446, 略大于孤子锁模极限0.44, 表明锁模脉冲只有很小的啁啾。为了验证锁模运转的稳定性, 我们测量了锁模激光的RF(radio frequency)频谱, 测量过程中RBW(resolution bandwidth)设置为30 Hz, 结果(图4(d))显示该锁模的重复频率为7.8576 MHz, 信噪比(signal-noise ratio, SNR)>55 dB, 证实了锁模的稳定运转, 图4(d)的内置小图为300 MHz范围内的宽带RF频谱, 该频谱并未呈现出明显的幅度调制, 证明该锁模状态为连续波锁模, 腔内的调Q效应得到有效的抑制。另外实验中随着泵浦功率的增大, 输出锁模激光功率也线性增大, 当泵浦功率为60.5 mW时, 输出功率为0.48 mW, 计算得到脉冲峰值功率为32.1 W。

值得一提的是, 当进一步增加泵浦功率时, 由于峰值功率过高, 导致孤子分裂, 腔内输出二阶谐波锁模, 这也是反常色散区锁模孤子的典型特征。

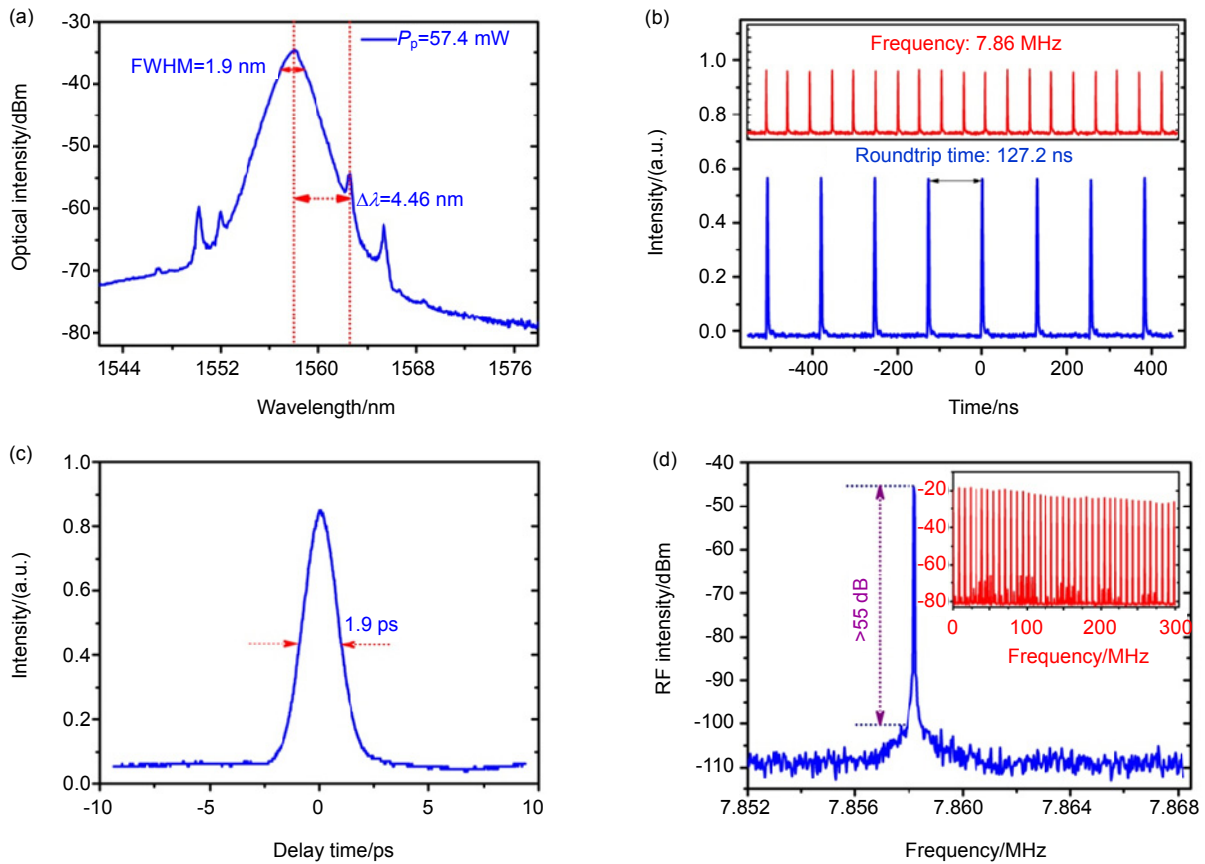


图4 GO-MoS₂锁模EDFL特征。(a) 光谱; (b) 脉冲序列; (c) 自相关迹; (d) 频谱

Fig. 4 The mode-locking characteristics of GO-MoS₂-based EDFL. (a) Optical spectrum; (b) Pulse sequence; (c) Autocorrelation trace; (d) RF spectrum

5 结论

本文开展了少层 GO-MoS₂ 用于掺铒光纤激光器 (EDFL) 锁模的实验研究。在实验中获得了中心波长为 1558 nm 的 EDFL 孤子锁模, 脉宽和脉冲能量分别为 1.9 ps 和 61 pJ, 重复频率为 7.86 MHz。当泵浦功率为 60.5 mW 时, 输出功率为 0.48 mW, 脉冲峰值功率为 32.1 W。研究证明, 采用这种方法制备的新型复合二维材料有利于保持少层 MoS₂ 的稳定性, 并且能提高 MoS₂ 可饱和吸收体的损伤阈值, 以获取更大脉冲能量的超快激光。所制作的锁模激光器具有成本低、重频高等特点, 将有望应用于生物医学领域。

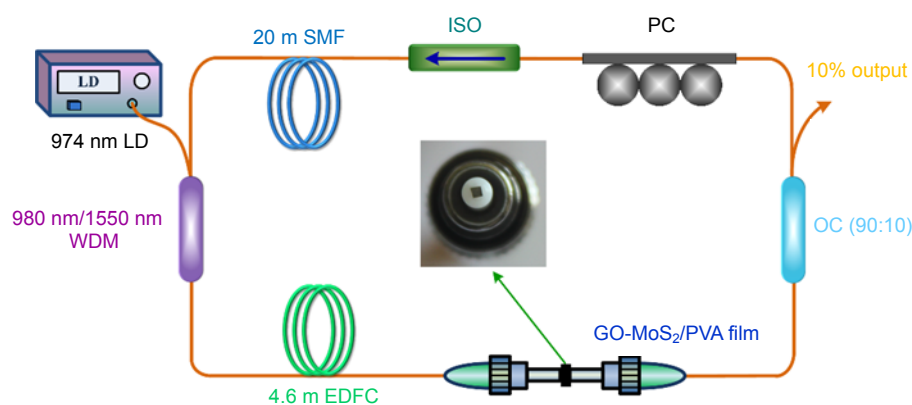
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Composite two-dimensional material GO-MoS₂-based passively mode-locked Erbium-doped fiber laser

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The structure of GO-MoS₂-based EDFL

Overview: In recent years, mode-locked fiber lasers have attracted extensive attention owing to their wide applications, such as material processing, optical communications, medicine, range finding and scientific research. This is because of their unique and outstanding advantages such as high peak power, narrow pulse width, compactness, low cost, high beam quality and ease of maintenance. Compared to active mode-locked fiber lasers which require a built-in amplitude modulator in the cavity, passive mode-locked fiber lasers using saturable absorbers (SA) as an intensity modulator have become a hotspot due to their simpler structure and abundant mode-locked phenomena. Traditional saturable absorbers include metal doped crystals, SESAMs and carbon nanotubes. For fiber lasers utilizing metal doped crystals or SESAMs, it is difficult to achieve an all-fiber structure, and they are also usually costly. The carbon nanotubes are relatively simple to fabricate and also easy to be integrated optically, but they only operate in a relatively narrow band. Until 2004, Novoselovks et al. of the University of Manchester successfully fabricated few layers and single layer of graphene by mechanically stripping graphite. Since graphene was discovered, the two-dimensional materials have been used as SAs in fiber lasers. In addition to graphene, there is an endless stream of research on topological insulators, transition metal dichalcogenides, black phosphorus and so on. Among them, the transition metal dichalcogenides represented by MoS₂ is a newly developed two-dimensional nanomaterial. The monolayer of MoS₂ consists of three atomic layers with a layer of molybdenum atoms sandwiched by two layers of sulfur atoms, which has good thermal and chemical stability. Furthermore, few-layer MoS₂ had been ambiguously verified to exhibit enhanced optical saturable absorption and can possess the stronger light-matter interaction. In this paper, for improving the performance and stability of MoS₂ saturable absorber, graphene oxide (GO) as colloidal surfactant is used to exfoliate MoS₂ bulk material for obtaining few-layer GO-MoS₂ nano-flakes. Further research on few-layer GO-MoS₂ saturable absorber to mode-lock erbium-doped fiber laser (EDFL) is then conducted. In the experiment, a stable mode-locked pulsed laser is achieved with a center wavelength of 1558 nm, a repetition rate of 7.86 MHz and a pulse width of 1.9 ps. When the pump power reaches 60.5 mW, the output power is 0.48 mW and the pulse peak power is calculated to be 32.1 W. This work shows that the new composite 2D material prepared by this method is beneficial to maintain the stability of few-layer MoS₂ and increase the damage threshold of the MoS₂ saturable absorber for passive mode-locking.

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