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Ethanol catalytic optical driven deposition for 1D and 2D materials with ultra-low power threshold of 0 dBm

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ABSTRACT

We have demonstrated a generalized optical driven deposition method, ethanol catalytic deposition (ECD) method, which is widely applicable to the deposition of a broad range of one-dimensional (1D) and two-dimensional (2D) materials with common deposition parameters. Using ECD method, deposition of 1D material carbon nanotubes and 2D materials MoS_2, $MoSe_2$, WS_2 and WSe_2 on tapered fiber has been demonstrated with the threshold power as low as 0 dBm. To our knowledge, this is the lowest threshold power ever reported in optical driven deposition, noting that the conventional optical driven deposition has a threshold typically near 15 dBm. It means ECD method can significantly reduce the power requirement and simplify the setup of the optical driven deposition as well as its wide applicability to different materials, which benefits the research on optical nonlinearity and ultrafast photonics of 1D and 2D materials.

1. Introduction

1D and 2D materials have received wide attention since the discovery of graphene and carbon nanotubes (CNTs). Transition metal dichalcogenides (TMDs) belonging to a type of 2D materials have similar optical properties such as optical nonlinearity, tunable bandgap and wideband absorption spectrum [1–12]. Among various applications from these properties of 1D and 2D materials, e.g., saturable absorption, optical power limiting and wavelength conversion [13–26], many applications require a long interactive length between the optical field and the materials. One general solution is to deposit the materials on a tapered fiber. When light with proper optical power is injected into a tapered fiber, the leaked optical field from the waist of the tapered fiber can apply an optical force to the small material flakes in the dispersions and attract them to the tapered fiber. This method is known as optical driven deposition [27,28]. Optical driven deposition allows a controllable deposition of the materials by monitoring the optical loss during the deposition process. However from the previous reports, it can be found that optical driven deposition may have very different power requirement for different materials, that is, one needs to choose different power levels to deposit different materials. This makes the deposition process very dependent on experience and repetitive test. Moreover, nearly all the current optical driven deposition works reported a relatively high injected optical power, typically near 15 dBm or even higher, to start the deposition. This high power requirement exceeds the output power level directly from a normal laser diode and usually needs an additional optical amplifier. Therefore it will be very beneficial if a generalized optical driven method can be found, which is widely applicable to many kinds of 1D and 2D materials with common deposition parameters and allows a very low power threshold affordable by a normal laser diode.

In this work, we have further developed our recently demonstrated ethanol catalytic deposition (ECD) method [28] to meet the above requirements and achieved the deposition of a wide range of 1D and 2D materials with ultra-low power threshold of 0 dBm. ECD method utilizes ethanol as solvent for the material dispersions. The high volatility of ethanol can significantly accelerate the Brownian motion of the materials in the dispersions and make the deposition much easier and controllable. Here we have successfully deposited CNTs, MoS_2, $MoSe_2$, WS_2 and WSe_2 onto tapered fibers with ECD method when five materials had same concentration of dispersions and same tapered fiber diameter

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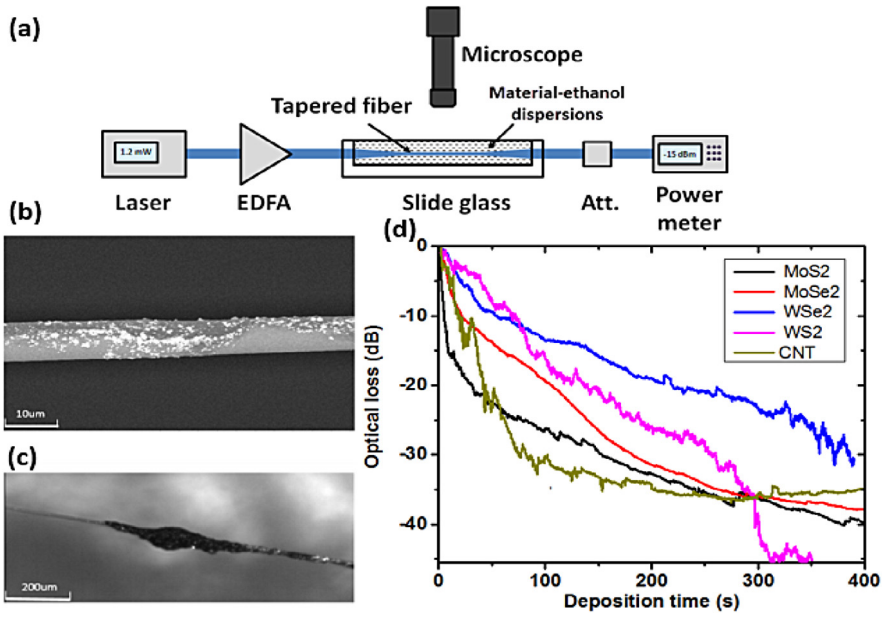

Fig. 1. (a) Experimental setup of ECD method, (b) microscopic image of a $MoSe_2$ deposited tapered fiber, (c) SEM image of a $MoSe_2$ deposited tapered fiber, (d) deposition curves of CNTs, MoS_2, $MoSe_2$, WS_2 and WSe_2.

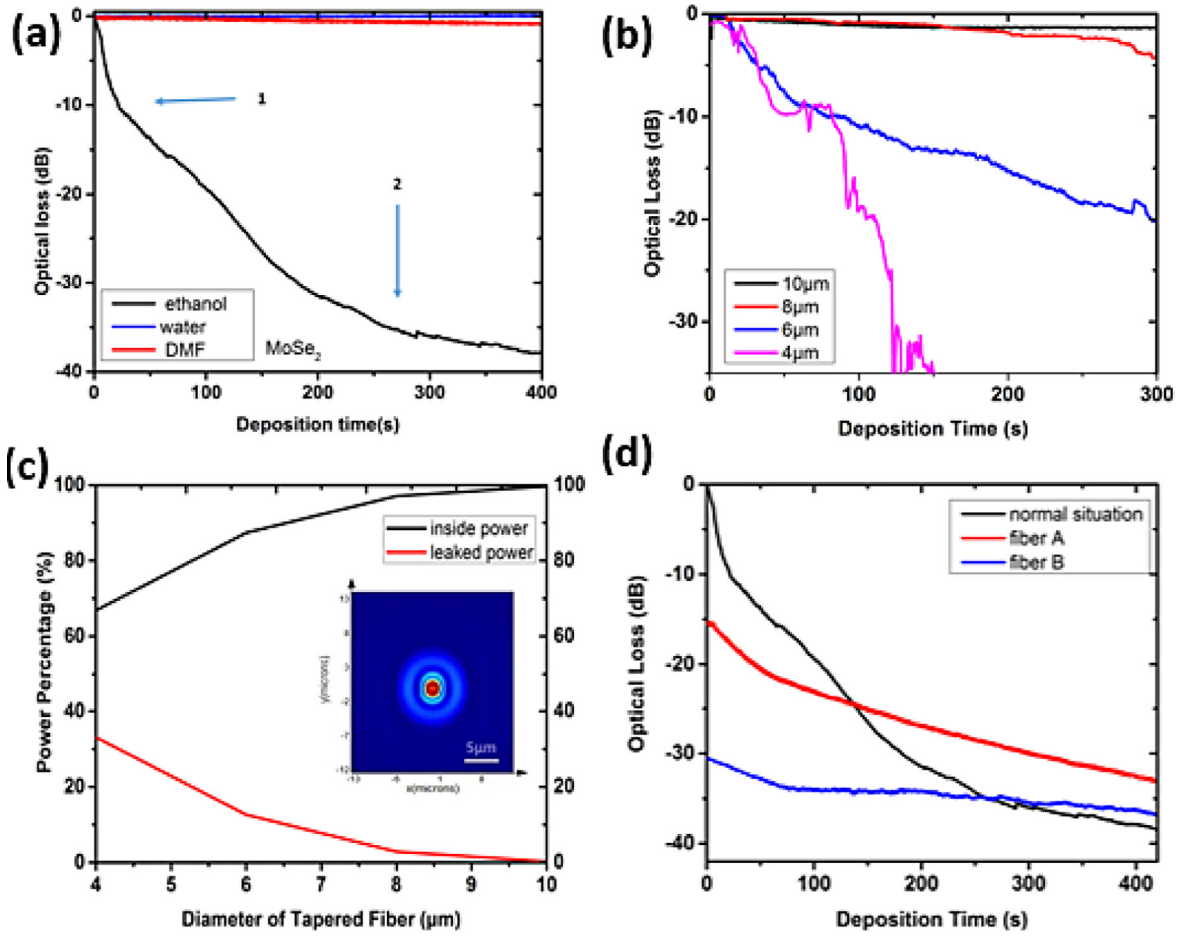

Fig. 2. (a) Deposition process of $MoSe_2$ using three different solvents; (b) deposition process of $MoSe_2$ with 4–10 μm tapered fiber; (c) The percentage of the optical power distribution inside and outside the tapered fiber for different tapered fiber diameters (4, 6, 8 and 10 μm); inset: the optical field distribution in a 4-μm-diameter tapered fiber; (d) deposition process of $MoSe_2$ with new tapered fiber, prior deposited 15 dB-loss tapered fiber and 30 dB-loss tapered fiber.

and injected optical power are used. This demonstration indicates that ECD method is a generalized deposition method suitable for different 1D and 2D materials for a common deposition parameters. Furthermore, the ultra-low power threshold of 0 dBm has been achieved for all these five materials with ECD method. To our knowledge, this is the lowest power threshold ever reported in optical driven deposition. Optimization of

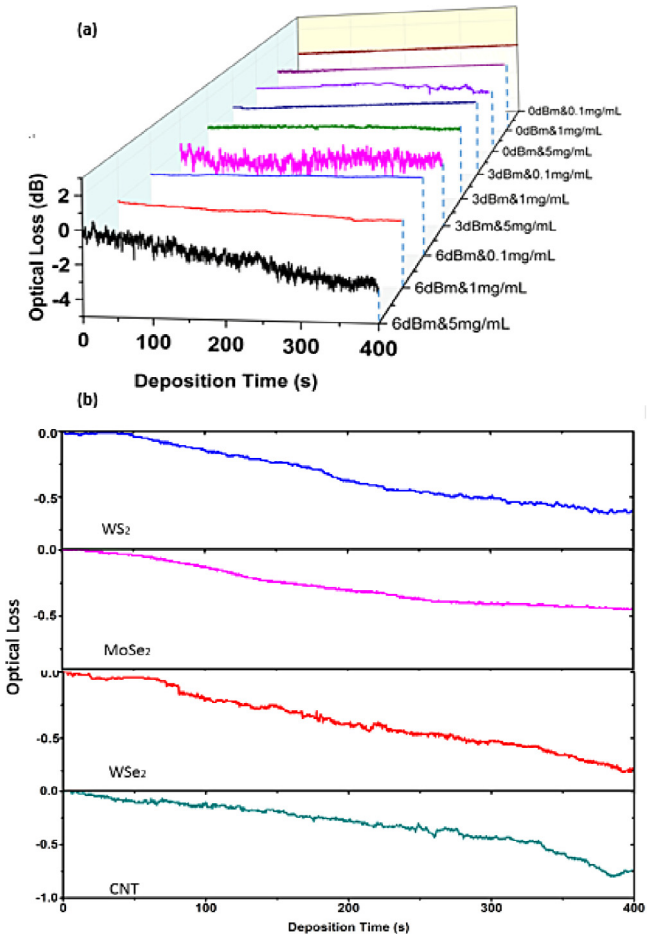

Fig. 3. (a) Deposition process of MoS_2 under different injected power and dispersion density (b) Deposition process of WS_2, $MoSe_2$, WSe_2, and CNT under 0 dBm optical power.

Table 1
Comparison of two methods under same parameters.

6 μm tapered fiber & 1 mg/mL concentration			
	0 dBm	10 dBm	19 dBm
ECD method	~0.0012 dB/s	~0.021 dB/s	~0.087 dB/s
Conventional DMF solvent	~0.0002 dB/s	~0.0032 dB/s	~0.017 dB/s

deposition parameters and analysis on the different deposition process of ECD method are also given. We believe ECD method can provide a generalized and easy deposition for a wide range of 1D and 2D materials which will surely benefit the research related to the light-material interaction of 1D and 2D materials [4,8,29–37].

2. Experiment and results

2.1. Wide applicability of ECD method

One important feature is that ECD method can be applied to the deposition of various materials with similar deposition parameters. To demonstrate this, we have performed the deposition of five materials including 1D material CNTs and 2D TMD materials MoS_2, $MoSe_2$, WS_2 and WSe_2 onto tapered fibers. Material dispersions are prepared by liquid phase exfoliation (LPE). One can refer to [24] for more details of LPE based material preparation. The experimental setup of deposition is similar to the conventional optical driven deposition, shown in Fig. 1(a). The tapered fiber is immersed in the material dispersions. When there is light injects into the fiber, leaked optical field will attract the material nanosheets to attach to the tapered fiber. The output power from the tapered fiber is monitored by an optical power meter. The most important difference is that ECD method utilizes ethanol as solvent of the dispersions. The high volatility of ethanol can significantly boost the Brownian motion of the material nanosheets in the dispersions and thus make the materials easier to be deposited onto the tapered fiber. The slide glass in the setup is customized to have a deep slot in the middle so that the tapered fiber can be completely immersed in the dispersions during the deposition.

As an example, Fig. 1(b) shows a typical microscopic image of the deposited $MoSe_2$ on the tapered fiber and Fig. 1(c) shows the SEM image of the same deposited fiber. The deposited $MoSe_2$ can be clearly observed in both images. Due to the vacuuming process during the SEM measurement, the large volume of the materials are removed and thus only small volume of the materials can be observed in the SEM image. Fig. 1(d) summarizes the deposition process of all five materials –CNTs, MoS_2, $MoSe_2$, WS_2 and WSe_2. The concentration of the material-ethanol dispersions is all set to 1 mg/mL. The tapered fiber diameters are fixed to 8 μm and the injected optical power is fixed to 19 dBm. Here we use a relatively high optical power to further accelerate the deposition process. It can be found that with ECD method the deposition can easily reach >30 dB power loss within only 5 min. It is also observed that the deposition processes are usually fast at the beginning and slow in the end. This is because the leaked optical field is strong at the beginning and gradually became weak after more and more materials wrap around the fiber during the deposition. Also due to the random size of the material nanosheets, the loss induced by each single deposition of material nanosheets vary and therefore the curves of the monitored optical loss are not smooth.

To verify the effectiveness of ECD method, two conventional solvents, water and organic solvent DMF, are compared with the solvent of ethanol used in ECD in a comparative deposition experiment of $MoSe_2$. The deposition parameters are fixed as the previous experiments, i.e., 1 mg/mL dispersion concentration, 8 μm tapered fiber diameter and 19 dBm injected power. The results are shown in Fig. 2(a). In a deposition time of 400 s, the increased optical loss of the tapered fiber using $MoSe_2$-ethonal dispersions is 37 dB while the increased loss of tapered fibers using $MoSe_2$-DMF and $MoSe_2$-water dispersions are only 2.3 dB and 1.2 dB, respectively. Deposition using $MoSe_2$-ethonal dispersions is 16 times and 35 times faster than those using $MoSe_2$-DMF and $MoSe_2$-water dispersions.

To optimize the deposition parameters, different diameters of tapered fiber are also investigated, shown in Fig. 2(b). The same 1 mg/mL concentration of $MoSe_2$-ethanol dispersion is used but the injected optical power is reduced to 10 dBm in the experiment. Otherwise the deposition will become too fast for small fiber diameters and exceed the measurement range of the optical power meter. It can be found the deposition became faster with the decrease of tapered fiber diameter. When the diameter is 4 μm, the scattering loss from the $MoSe_2$ nanosheets is too strong and the deposition process is very difficult to be controlled. On the other hand, the deposition become very slow when the diameter is 10 μm. Thus a proper span of tapered fiber diameter is found to be within 6–8 μm. To further confirm the influence of tapered fiber diameter quantitatively, a simulation on the distribution of optical field in the tapered fiber have been conducted. The simulation is based on a 3D light propagation from a standard single mode fiber to the waist of the tapered fiber through an adiabatic tapering region. The percentage of the optical power distribution inside and outside the tapered fiber for different tapered fiber diameters (4, 6, 8 and 10 μm) is summarized in Fig. 2(c) and a typical distribution of the optical field in a 4-μm-diameter tapered fiber is shown in the inset of Fig. 2(c). It can be observed that the leaked power increased with the decrease of the tapered fiber diameter which enhanced the optical force between the materials and the fiber. When the diameter is 10 μm, the leaked power became very weak and high optical power is required to drive the deposition, which is consistent with the experimental observations. This finding also explain why the deposition is always fast in the beginning

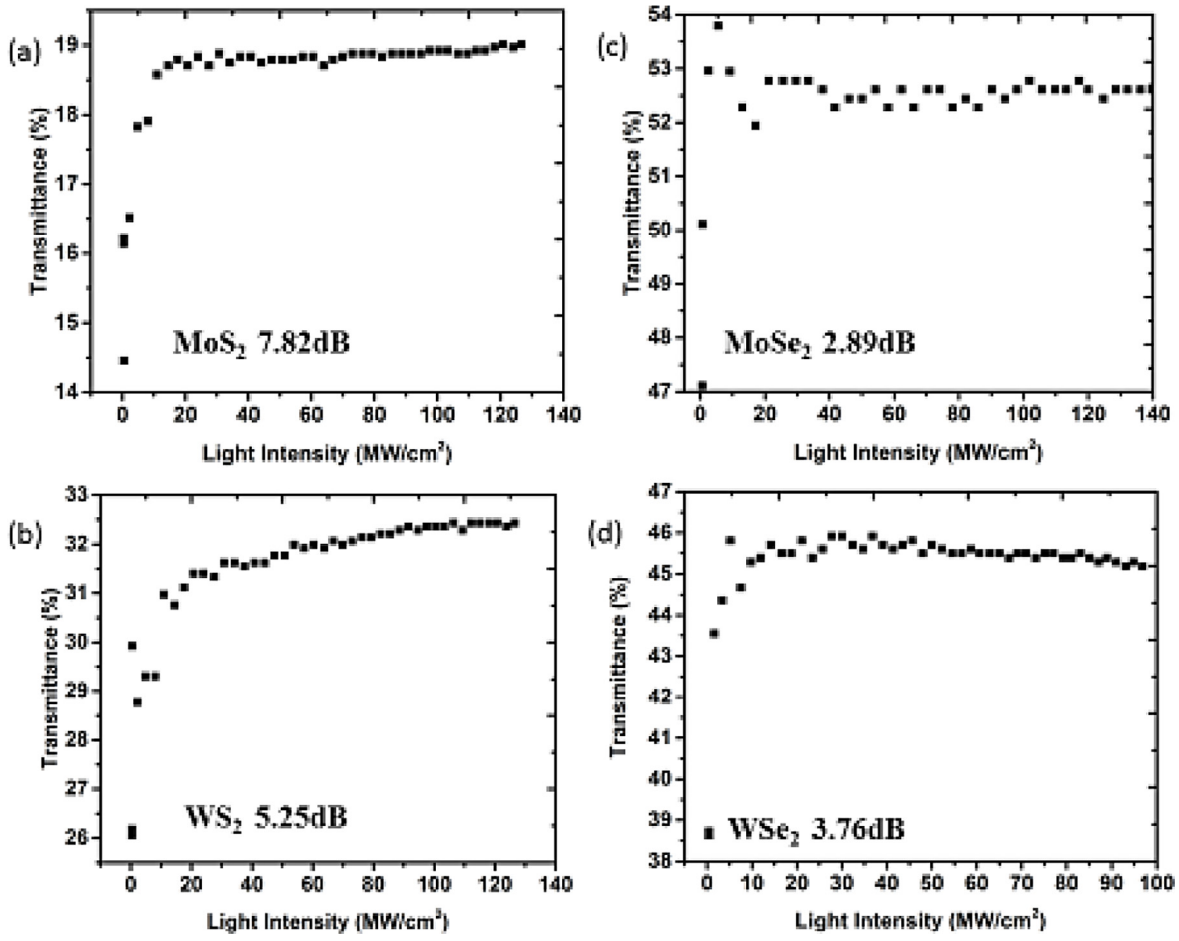

Fig. 4. Saturable absorption of (a) MoS_2, (b) WS_2, (c) $MoSe_2$ and (d) WSe_2 deposited tapered fiber.

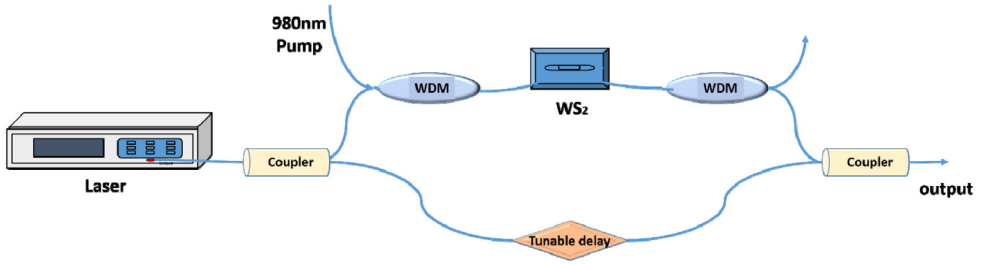

Fig. 5. Experimental setup of the all optical modulator based on WS_2 deposited tapered fiber.

and slow in the end, e.g., point 1 and 2 shown in Fig. 2(a). This is due to the fact that the tapered fiber is gradually wrapped by the materials during the deposition which results in less leaked optical power and therefore weaker interaction between the light and the materials.

Another important concern is how the prior deposition will affect the later deposition. To answer this question, we have used two deposited tapered fiber with 15.32 dB loss (denoted as fiber A) and 31.08 dB loss (denoted as fiber B) respectively to carry out new deposition experiments. The results are shown in Fig. 2(d). It can be observed that the deposition became very slow and difficult when there had already been materials wrapped on the tapered fiber. Higher loss in fiber B means more prior deposited materials and thus even weaker leaked optical field. As a result, the deposition on fiber B is even more difficult than fiber A.

2.2. Ultra-low power threshold of ECD method

Besides the wide applicability and high deposition rate of ECD method, the other important contribution of ECD method is that it allows an ultra-low power threshold to drive a deposition process. To avoid the ambiguity, we define the power threshold as the minimum power to drive a deposition with increased loss no less than 0.5 dB in 400 s with a concentration of 1 mg/mL of the material dispersions. To make a complete investigation on the injected optical power and concentration of material dispersions, three concentrations (0.1 mg/mL, 1 mg/mL and 5 mg/mL) of MoS_2 dispersions are chosen and the tapered fiber diameter is fixed to 8 μm. Fig. 3(a) shows the recorded deposition with three optical power levels (0 dBm, 3 dBm and 6 dBm) combined with three concentrations. As expected, high power/concentration ratio leads to a higher deposition rate than low power/concentration ratio.

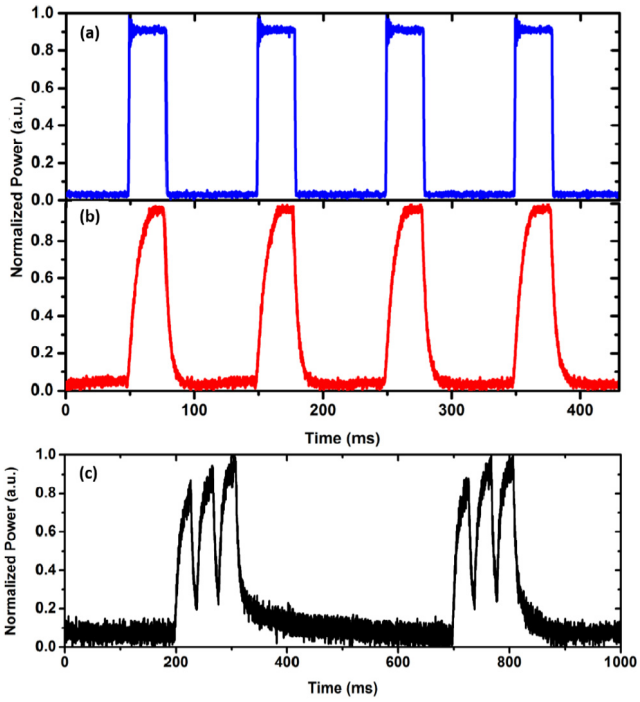

Fig. 6. (a) Pulsed pump light; (b) modulator output; (c) pulse breaking when the modulator is over-driven.

In a deposition time of 400 s, the 6-dBm injected power with 5 mg/mL, 1 mg/mL, and 0.1 mg/mL result in an increased optical loss of 3.2 dB, 1.19 dB and 0.37 dB respectively, while 3-dBm power correspond to 0.96 dB, 0.53 dB and 0.07 dB respectively, and 0-dBm power correspond to 0.63 dB, 0.43 dB and 0.17 dB respectively.

These results indicate that ECD method is able to drive the deposition with an optical power as low as 0 dBm in a wide range of concentration from 1 mg/mL to 5 mg/mL. Based on our definition, the deposition power threshold is 0 dBm at 1 mg/mL concentration. It is also found that there is drastic fluctuation of the monitored optical loss during the deposition with the concentration of 5 mg/mL which is due to the drastic Brownian motion process in high concentration. In such a high concentration, the nanosheets are frequently attached to or collided off from the tapered fiber which make the deposition process very difficult to control and result in an unsmooth deposition curve. Thus an optimal choice of dispersion concentration will be from 0.1 mg/mL to 1 mg/mL. Fig. 3(b) summarizes the deposition curves of the other four materials with 0 dBm injected power and 1 mg/mL concentration. These curves confirm that 0 dBm is a general threshold for these materials and wide applicability of ECD method is also demonstrated. Table 1 shows the comparison of the mean deposition rates (with relative errors less than 20%) of ECD method and conventional deposition with DMF solvent under same parameters. It can be observed that ECD method exhibited a much faster deposition rates under same injected power and thus a much lower threshold than the conventional deposition with DMF solvent. To our knowledge, a power threshold of 0 dBm enabled by ECD method is the lowest power threshold for optical driven deposition ever reported. This power level is directly affordable by a normal laser diode without the need of an additional optical amplifier.

3. Discussion

3.1. Saturable absorption of 2D materials

2D materials deposited tapered fiber have many useful nonlinear applications such as optical saturable absorber. To confirm the saturable absorption under different deposition time, we have deliberately deposited the five materials and then measured the saturable absorption with a standard two-arm transmission measurement. The measured transmission curves are shown in Fig. 4. It can be found that all the five different deposited materials showed saturable absorption. Materials with higher original loss on the tapered fiber has smaller modulation depth because the optical field is attenuated more. Fig. 4(a) shows the MoS_2 deposited tapered fiber with a loss of 7.82 dB. It has a modulation depth of 4.5%, a saturation intensity of 20 MW/cm^2 and a non-saturable loss of 81%. Fig. 4(b) shows the WS_2 deposited tapered fiber with a loss of 5.25 dB. It has a modulation depth of 6.5%, a saturation intensity of 85 MW/cm^2 and a non-saturable loss of 67.5%. Fig. 4(c) shows the $MoSe_2$ deposited tapered fiber with a loss of 2.89 dB. It has a modulation depth of 5.7%, a saturation intensity of 30 MW/cm^2 and a non-saturable loss of 55%. Fig. 4(d) shows the WSe_2 deposited tapered fiber with a loss of 3.76 dB. It has a modulation depth of 3.3%, a saturation intensity of 20 MW/cm^2 and a non-saturable loss of 47.5%. These results indicate the validity of the prepared TMDs deposited tapered fiber as a nonlinear device for photonic application such as mode-locked lasers and Q-switched lasers.

3.2. Thermal-optic modulator based on 2D materials

Photothermal effect is another optical property of 2D materials. To verify this property, we have incorporated a WS_2 deposited tapered fiber into a fiber Mach-Zehnder interferometer (MZI), shown in Fig. 5. The light is injected from a mode-locked laser near 1550 nm and then split by a 70:30 coupler. The 70% power is combined with 980 nm pump power by a wavelength division multiplexer (WDM) and propagated through the WS_2 tapered fiber in one arm. The residual pump light is extracted from a second WDM after the WS_2 tapered fiber. A tunable delay line is inserted in the other arm to control the length. The output 1550-nm light of two arms is combined together by a 50:50 coupler. In our experiment, WS_2 absorb the pump light, generate heat (photothermal effect) and modify the refractive index of the tapered fiber due to its thermo-optic effect. Then the refractive index change of the fiber modify the optical phase of the light propagating through it and finally result in intensity modulation via MZI.

Fig. 6 shows the results of the intensity modulation. We have injected a pulsed electronic signal into the pump driver and generate the 980-nm pulsed pump light, shown in Fig. 6(a). The repetition rate of the pulse train is 10 Hz and pulse duration is 30 ms. The output 1550-nm light with clear rising and falling edge is detected, shown in Fig. 6(b). *The rising time and falling time are ~7 ms and ~4 ms, respectively.* When the peak power of pump light increase and the corresponding optical phase change exceeds 2π, pulse breaking is also observed, shown in Fig. 6(c). The clear intensity modulation means 2D materials may have potential applications in optical switching, routing and signal processing.

4. Conclusion

In this paper, we have applied the ethanol catalytic deposition method to deposit 1D and 2D materials onto tapered fibers and demonstrated the wide applicability of ECD method on different 1D and 2D materials. The deposition efficiency of materials-ethanol dispersions is several tens of times higher than the conventional materials-water or DMF dispersions. Moreover, by optimizing deposition parameters, the threshold power to drive a deposition is found to be as low as 0 dBm for ECD method, which is much lower than the conventional optical driven deposition. Saturable absorption of 1D and 2D materials deposited tapered fiber are also provided. In short, we have demonstrated ECD method which is applicable to the deposition of a wide range of 1D and 2D materials with same deposition parameters and exhibits a threshold power down to 0 dBm directly affordable by a normal laser diode. Our work provides a generalized and easy deposition method and will benefit the research on light-material interaction and ultrafast photonics.

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