

## Regular Articles

## Soliton mode-locked thulium-doped fiber laser with cobalt oxide saturable absorber

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## ABSTRACT

In this work, we propose and demonstrate a mode-locked thulium doped fiber laser (TDFL) with an integrated cobalt oxide ( $\text{Co}_3\text{O}_4$ ) saturable absorber (SA). The SA is fabricated by suspending  $\text{Co}_3\text{O}_4$  nanosheets in a polymer film before being secured between two fiber ferrules. The laser operates in the anomalous dispersion regime, confirmed by the Kelly sidebands observed in the obtained optical spectrum. Mode-locking operation is obtained at a low threshold pump power of 77.32 mW, with further optimizations made using a polarization controller. The laser generates pulses with a repetition rate of 11.36 MHz and a pulse width of 1.39 ps. The generated pulses are highly stable, with a high signal-to-noise ratio of 46.00 dB and minimum power fluctuations indicating a long-term stability. This work demonstrates a simple and a low-cost laser that would have potential applications for operation near the 2.0- $\mu\text{m}$  region, especially for medical applications.

## 1. Introduction

Passively pulsed fiber lasers are highly desired as laser sources due to their potential use in a variety of applications ranging from material processing to remote sensing, microscopy and medicine. The attractiveness of these sources arises from their robust yet compact form factor, which in turn results low fabrication and operating costs [1–5]. To achieve this, fiber lasers are designed to be either Q-switched or mode-locked, with the latter generating shorter, ultrafast pulses with overall low output energies and the former generating longer and slower pulses but with higher energies [6].

Traditionally, Q-switching and mode-locking in fiber lasers was achieved by actively modulating the losses within the laser cavity active using acousto-optic or electro-optic modulators [7–11]. However, the bulky nature of these modulators, as well as their relatively high cost quickly made this option unsuitable for most real-world applications. As such, research efforts were focused towards the development of passively pulsed devices, which generated a similar output but in a much more compact form factor and at a lower overall cost. This was typically achieved primarily by saturable absorbers (SAs), such as semiconductor saturable absorber mirrors (SESAMs) [12,13] and more recently 2-dimensional (2D) and 3-dimensional (3D) materials such as carbon nanotubes [14–16] and graphene [17–19]. Additionally, recent developments have now seen new 2D and 3D nanomaterials, such as

metals [20,21], topological insulators (TIs) [22–24], transition metal dichalcogenides (TMDs) [25–27] and even exotic materials such as black phosphorus [28–30] being used to obtain passive mode-locked outputs. The fabrication and the nonlinear optical properties of these low-dimensional materials have been widely investigated and reported [31–33], with particular attention being paid to 2D nanomaterials for advanced opto-electronic applications [34–36] due to their unique properties such as low saturation intensity, broadband absorption properties as well as fast recovery and response times [37–39].

Of late, transition metal oxides (TMOs) have now become the focus of significant research interest for use as SAs due to their unique optical characteristics, namely their large nonlinear optical response [40]. As a result of their 2D nature, the bandgaps of TMOs can be tuned without altering their physical properties, giving them a wide-operational bandwidth [41]. TMOs such as nickel oxide (NiO), zinc oxide (ZnO), iron (II, III) oxide ( $\text{Fe}_3\text{O}_4$ ), and titanium oxide ( $\text{TiO}_2$ ) have all been successfully demonstrated as SAs for the generation of pulses fiber lasers [42–45]. The performance of these lasers is comparable to the ones built using graphene or other 2D material based SAs, thus indicating the potential of TMOs as SAs. In this regard, cobalt oxide ( $\text{Co}_3\text{O}_4$ ) is an attractive material for use as an SA as it has a large nonlinear to linear absorption ratio [40], as well as spectral absorption that stretches to the near-infrared (NIR) region [46]. Recently, the use of  $\text{Co}_3\text{O}_4$  as an SA has been reported with the generation of Q-switched pulses at the 1.5- $\mu\text{m}$

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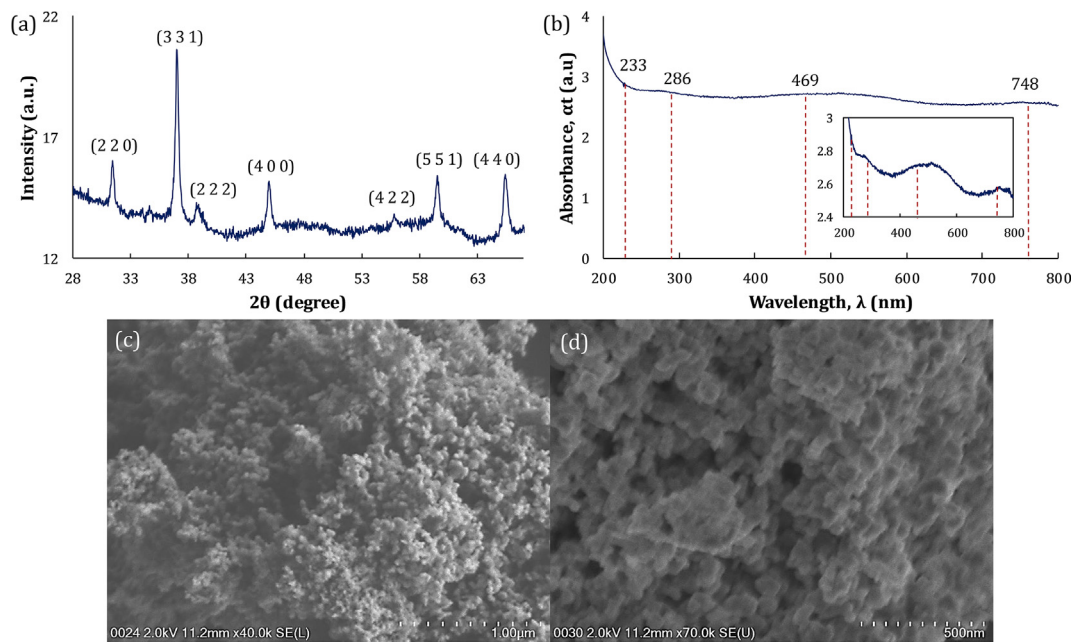


Fig. 1. (a) XRD pattern of  $\text{Co}_3\text{O}_4$  nanosheets, (b) UV-vis spectra of the  $\text{Co}_3\text{O}_4$ -PEO in solution form with a close-up view of the absorption peaks (inset), and FESEM images of the  $\text{Co}_3\text{O}_4$  nanosheets taken at (c) low magnification and (d) at high magnification.

wavelength region [47]; however there are no reports yet on  $\text{Co}_3\text{O}_4$  being used to generate mode-locked pulses in fiber lasers operating at the longer 2.0- $\mu\text{m}$  wavelength region.

In this report, a mode-locked thulium doped fiber laser (TDFL) using a  $\text{Co}_3\text{O}_4$  based SA is proposed and demonstrated for operation in the 2.0- $\mu\text{m}$  region. Lasers operating in the 2.0- $\mu\text{m}$  wavelength region are 'eye-safe', making them suitable for numerous industrial and medical applications [48,49]. Furthermore, the strong absorption coefficient of this wavelength region in water and gases makes them favorable for various spectroscopy and sensing applications [50–52]. The TDFL is configured with a thulium doped fiber (TDF) in a ring laser cavity as well as an integrated SA. A polarization controller (PC) is also used to optimize the laser's performance. The proposed setup has the advantage of being easy to implement and does not require the use of costly and difficult-to-handle modulators. This, combined with its operation in the eye-safe region, gives the proposed system high potential for real-world applications.

## 2. Fabrication and characterization of the SA

The  $\text{Co}_3\text{O}_4$  nanosheets used in this work are obtained by the reactions of a cobalt (II) acetate tetrahydrate  $[\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}]$  precursor, sodium hydroxide (NaOH), and polyethylene oxide (PEO). All reagents are obtained from Sigma-Aldrich while an additional reagent, ammonia ( $\text{NH}_3 \cdot \text{H}_2\text{O}$ ) with a purity of 25%, is obtained from R&M Chemicals. All chemicals are of analytical grade and used without any further treatment.

The  $\text{Co}_3\text{O}_4$  nanosheets used for the fabrication of the SA thin-film are synthesized using the facile hydrothermal method. In this method, the cobalt oxide precursor with a molar concentration of 2 mM is first prepared by dissolving a sufficient amount of  $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  in deionized (DI) water by ultrasonication. Approximately 13 mL of NaOH with a concentration of 3 mM is then added to the aqueous solution, after which the solution is stirred continuously for another 30 min at room temperature. The obtained solution is then mixed with 2 mL of  $\text{NH}_3 \cdot \text{H}_2\text{O}$  using the drop-cast method and stirred at room temperature for another 1 h. The suspended solution is then transferred into a Teflon-lined stainless-steel autoclave and heated in a hot oven at 150 °C for 16 h. After this, the mixture is left to cool at room temperature

before being centrifuged at 4000 rpm for 20 min to obtain the  $\text{Co}_3\text{O}_4$  nanosheets structure, which appear as a black powder precipitate. The  $\text{Co}_3\text{O}_4$  nanosheet precipitate is then washed extensively with DI water and absolute ethanol. This is repeated three times before the final precipitate is dried in an oven at 60 °C for 24 h, giving the desired form of a fine black powder.

While the nanosheets are the optically active material of the SA, their fine and brittle nature does not allow them to be used in their current form within the fiber cavity. As such, the  $\text{Co}_3\text{O}_4$  nanosheets are instead embedded in a Polyethylene-Oxide (PEO) thin film which serves as a host material. The thin film is formed by dissolving 250 mg of PEO powder in 30 mL of DI water, and stirred continuously at 50 °C for 2 h. Approximately 10 mL of the  $\text{Co}_3\text{O}_4$  black powder with a density of  $5 \text{ mg mL}^{-1}$  is then added into the PEO solution, and the entire mixture stirred for another 2 h. The resulting compound is then transferred onto a petri dish and left to dry in an oven at 60 °C for 24 h, forming a polymer film with  $\text{Co}_3\text{O}_4$  nanosheets embedded in the host material. A small portion of the thin film formed is cut out and placed on the face of a fiber ferrule so that it covers the core region. A small amount of index-matching gel is used to hold the polymer piece in place, and another fiber ferrule is then connected to the first using a standard fiber adaptor. This forms the SA assembly which will be integrated into the cavity of the proposed laser.

The synthesized  $\text{Co}_3\text{O}_4$  nanosheets are characterized by x-ray diffraction (XRD) using an Empyrean PANalytical x-ray diffractometer with copper K $\alpha$  radiation at an excitation wavelength of 1.5418 Å. Diffraction peaks at 31.4°, 36.9°, 38.7°, 44.9°, 55.8°, 59.5°, and 65.4° are obtained from the analysis, corresponding to the (220), (331), (222), (400), (422), (511), and (440) planes of  $\text{Co}_3\text{O}_4$  respectively as seen in Fig. 1(a). All peaks are consistent with the JCPDS card 00-042-1467 [53], thereby confirming the successful synthesis of  $\text{Co}_3\text{O}_4$ . No other significant peaks are detected in the spectrum, implying a high-level purity in the fabricated compound. Furthermore, the narrow and high-intensity diffraction peaks observed in the spectrum indicates a high degree of crystallinity in the synthesized  $\text{Co}_3\text{O}_4$  compound.

In addition to this, ultraviolet-visible (UV-Vis) spectroscopy analysis is also performed on the  $\text{Co}_3\text{O}_4$ -PEO when still in its solution form. Characterization is carried out using a Varian Cary 50 UV-Vis Spectrophotometer from Agilent Technologies over a wavelength range

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