UNIVERSITI PUTRA MALAYSIA

GOLD NANOPARTICLES SATURABLE ABSORBER FOR ULTRASHORT PULSE GENERATION IN FIBRE LASERS

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Chair : Mohd Adzir bin Mahdi, PhD
Faculty : Engineering

The advantages of ultrashort pulse lasers have triggered a technological tsunami in the laser field, putting pressure on researchers to discover the simplest fabrication method and thus effectively improve the saturable absorber (SA) preparation technique. The optical property of saturable absorption is directly associated to the embedded materials in SAs themselves. Thus, manipulation of the embedded materials may improve the overall performance of the SA. Metallic nanostructures have been known for their optical properties due to the effect of surface plasmon resonance. Among the metallic nanostructures, gold nanoparticles (Au-NPs) are widely investigated due to its saturable and reverse saturable absorption properties. These properties can be tailored to cater for various applications by manipulating its size and shape. There are three important aspects in this research work which include the synthesis of Au-NPs in tetrahydrofuran by pulsed laser ablation, validation of the ablated Au-NPs as a SA in generating mode-locked pulses and investigation on the effect of Au-NP size towards optical pulse profiles. The proposed synthesis of Au-NPs in tetrahydrofuran with stirring condition produced a good size distribution of spherical Au-NPs ranging from 6.0 to 11.5 nm. The size reduction was influenced by the ablation time increment from 7 to 30 minutes. The effect of stirring was also confirmed by comparing the ablated material size without stirring. However, the biggest challenge of using this method was the low yield of ablated Au-NPs. In order to study the effect of Au-NP size, commercially available Au-NPs of varied sizes were purchased; 10, 20, 40 60 and 80 nm. These two batches would be prepared as fillers inside polydimethylsiloxane (PDMS) polymer matrix. To fabricate a mode-locker that can support evanescent wave propagation, a tapered fibre was selected as the preferred waveguide. The embodiment of Au-NPs with PDMS on the tapered fibre was deposited using a spin coating technique. The longitudinal encapsulation of Au-NP/PDMS enables interaction between evanescent wave and matter (surrounding medium). The fabricated Au-NP-based SA was

characterized using a twin balance photo-detection method to obtain its saturation fluence, non-saturable loss and modulation depth. The functionality of the fabricated Au-NP-based SA was proven by incorporating it in a ring cavity erbium-doped fibre laser as a result of optical pulse generation. The same laser cavity was used throughout the research work to minimize uncertainties of loss and dispersion. Based on the experimental findings, both batches of Au-NPs were proven to be able to generate ultrashort pulse with a pulse duration of less than 1 picosecond. This marks the most significant finding of the research work. For the ablated Au-NPs, the average size of 7.8 nm was successfully tested to generate mode-locked pulse at 1554.5 nm with duration in the range of 916 – 994 fs. Even though that the modulation depth of the fabricated SA was only 0.4%, a stable pulse was produced. For the commercially available Au-NPs, mode-locked pulse was attained for all sizes to verify the finding from the previous experiment (ablated Au-NPs). The lasing performance was evaluated by comparing SA characteristics and pulse qualities among the sizes. The optimum pulse performance was realized when the SA was fabricated with 20 and 40 nm Au-NP size. For the former, the time-bandwidth product of 0.34 was demonstrated which was the closest to its bandwidth-limited pulse. For the former, the fabricated SA exhibited 4.0% modulation depth and average pulse duration of 886.7 fs. From the experimental findings, larger Au-NP size of 60 and 80 nm had the tendency to scatter more lights due to its larger cross section. Therefore, the highest transmission loss of 8.56 dB was obtained for 80 nm Au-NP size and the pulse quality deteriorated to 1062.3 fs. The research work has demonstrated the functionality of Au-NPs as a saturable absorption material to generate ultrashort pulses. In addition, the size of nanomaterials has influenced on the characteristics of saturable absorbers that shapes the quality of laser pulse.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENYERAP TEPU NANOPARTIKEL EMAS BAGI MENJANA DENYUT ULTRAPENDEK DALAM GENTIAN LASER

Oleh

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Kelebihan laser denyut ultrapendek telah mencetus tsunami teknologi dalam bidang laser, dimana kini terdapat tekanan di kalangan penyelidik untuk mencari teknik fabrikasi termudah sebagai ganti yang lebih baik bagi penyerap tepu (SA) yang sedia ada. Ciri optik penyerap tepu adalah berkait secara terus dengan bahan terenap dalam penyerap tepu. Oleh yang demikian, manipulasi bahan terenap ini mungkin boleh memperbaiki prestasi keseluruhan SA tersebut. Nanostruktur metalik terkenal dengan ciri-ciri optiknya disebabkan oleh resonan plasmon permukaan. Antara nanostruktur metalik ini, Au-NPs dikaji secra meluas disebabkan oleh ciri-ciri penyerapan tepu dan penyerapan tepu songsangnya.Ciri-ciri ini boleh di ubah untuk memenuhi pelbagai aplikasi dengan memanipulasi saiz dan bentuk. Terdapat tiga aspek penting dalam kerja penyelidikan ini, yang mana sintesis Au-NPs di dalam tetrahydrofuran menggunakan ablasi laser denyut , untuk mengesahkan kebolehan Au-NPs terablasi sebagai SA dalam menghasilkan denyut mod-terkunci dan menentukan kesan saiz Au-NPs terhadap profil denyut. Sintesis Au-NPs yang di cadangkan di dalam tetrahydrofuran dengan kondisi dikacau telah menghasilkan Au-NPs berbentuk sfera dengan taburan saiz yang baik antara 6.0 ke 11.5nm. Pengurangan saiz sepadan dengan tempoh ablasi dari 7 ke 30 minit. Kesan pengacauan juga disahkan dengan membandingkan saiz bahan terablasi tanpa pengacauan. Walaubagaimanapun, cabaran terbesar menggunakan kaedah ini adalah kuantiti Au-NPs terablasi yang rendah .Untuk mengkaji kesan saiz Au-NPs, bahan yang tersedia secara komersial telah dibeli: 10, 20, 40, 60 dan 80nm. Dua kumpulan ini akan disediakan sebagai pengisi di dalam polimer metrik polydimethylsiloxane (PDMS). Bagi menghasilkan penyerap tepu yang boleh menampung penyebaran gelombang evanescent, gentian tirus lebih di ingini sebagai panduan gelombang. Penyatuan Au-NPs dengan PDMS di atas gentian tirus adalah melalui teknik lapisan putaran. Penyalutan Au-NPs/PDMS secara membujur mombolehkan

interaksi antara gelombang evanescent dan jirim (medium persekitaran). Au-NPs SA yang terhasil dicirikan menggunakan kaedah pengesanan photo berkembar seimbang untuk memperoleh ketepuan lancar, kehilangan tidak tepu dan kedalaman modulasi. Kebolehfungsian Au-NPs SA yang terhasil di buktikan dengan menggabungkannya ke dalam rongga cincin laser gentian terdop erbium kesan daripada penghasilan denyut optik. Rongga cincin yang sama digunakan sepanjang kerja penyelidikan bagi mengurangkan ketidaktentuan kehilangan dan penyebaran. Berdasarkan kepada dapatan eksperimen, kedua-dua kumpulan Au-NPs terbukti boleh menghasilkan denyut ultrapendek dengan tempoh denyut kurang daripada 1 piko saat. Ini adalah merupakan penemuan yang paling ketara bagi kerja penyelidikan ini. Bagi Au-NPs yang terablasi, saiz purata sebanyak 7.8 nm berjaya di uji bagi menghasilkan denyut mod-terkunci pada 1554.5 nm dengan tempoh denyut antara 916 – 994 fs. Walaupun kedalaman modulasi bagi SA yang terhasil hanyalah 0.4%, denyut yang stabil dapat dihasilkan. Untuk Au-NPs yang tersedia secara komersial, denyut mod-terkunci diperoleh untuk semua saiz mengesahkan penemuan dapatan kajian sebelumnya. Prestasi laser di nilai dengan membandingkan ciri-ciri SA dan kualiti denyut di antara saiz. Prestasi denyut yang paling optimum direalisasikan apabila SA dihasilkan menggunakan Au-NPs bersaiz 20 dan 40 nm. Bagi yang terdahulu, SA yang terfabrikasi menghasilkan 4.0% kedalaman modulasi dengan purata tempoh denyut adalah 886.7 fs. Dapatan kajian juga mendapati, Au-NPs dengan saiz yang lebih besar iaitu 60 dan 80nm mempunyai kecenderungan utuk menyelerakkan cahaya disebabkan oleh keratan rentasnya yang lebih besar. Oleh yang demikian, kehilangan transmisi tertinggi sebnayak 8.56 dB telah diperoleh untuk Au-NPs dengan saiz 80 nm dan kualiti denyut yang merosot kepada 1062.3 fs. Kerja penyelidikan ini telah mempamerkan kebolehfungsian Au-NPs sebagai bahan penyerap tepu yang mampu menghasilkan denyut ultrapendek. Tambahan lagi, saiz material nano juga mempengaruhi ciri-ciri penyerap tepu yang mencorakkan kualiti denyut laser.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research and the writing of this thesis were done under our supervision;
- supervisory responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2015-2016) are adhered to.

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AOM	Acousto-optic modulator
Ar	Argon
Au	Gold
Au-NP	Gold Nanoparticle
BP	Black Phosphorus
C	Carbon
CH_3CN	Acetonitrile
Cr	Chromium
Cu	Copper
CVD	Chemical vapor deposition
CW	Continuous wave
DMSO	Dimethyl sulfoxide
EDF	Erbium doped fibre
EDFL	Erbium doped fibre laser
EDX	Energy Dispersive X-Ray
Fe	Iron
FESEM	Field-emission scanning electron microscope
FT-IR	Fourier transforms infrared spectroscopy
FWHM	Full width at half maximum
GVD	Group velocity dispersion
He	Helium
HR-TEM	High-resolution transmission electron microscopy

IR	Infra-red
Kr	Krypton
LD	Laser diode
LSPR	Localised surface plasmon resonance
MD	Modulation depth
ML	Mode locking
MoS_2	Layered molybdenum disulphide
N^2	Nitrogen
Ne	Neon
NIR	Near infra- red
NSE	Nonlinear Schrodinger equation
O	Oxygen
OPM	Optical power meter
OSA	Optical spectrum analyser
PC	Polarisation controller
PDMS	Polydimethylsiloxane
PLA	Pulsed laser ablation
PMMA	Polymethylmethacrylate
PVP	Polyvinylpyrrolidone
PZT	Piezo-electric transducer
RF	Radio frequency
SA	Saturable absorber
SAM	Self-amplitude modulation
SESAMs	Semiconductor saturable mirrors

Si	Silicon
SMF	Single mode fibre
SNR	Signal-to-noise ratio
SPM	Self-phase modulation
SPR	Surface plasmon resonance
TBP	Time bandwidth product
TEM	Transmission electron microscopy
THF	Tetrahydrofuran
TMDs	Transition metal dichalcogenides
VOA	Variable optical attenuator
WDM	Wavelength division multiplexer
Xe	Xenon

CHAPTER 1

INTRODUCTION

1.1 Overview

Ultrashort pulse lasers can be defined as an optical laser pulse with a pulse duration within the range of picosecond or less. Its capacity to deliver bursts of high intense light onto a localized region translates to efficient energy transfer that offers numerous advantages in the fields of transparent material [1], biology and medicine applications [2], optical metrology [3] and many other. In transparent material processing, scribing and marking without affecting the material surface is possible after high intensity ultrashort pulse is applied, therefore commencing nonlinear absorption process, where localized energy deposition leads to permanent structural changes inside the sample [4]. This type of laser also has played significant and important roles in biology and medicine applications. For example, laser radiation is used in surgery for treatment and diagnosis related to coagulation of retinal excess blood vessel for diabetic patients [5], while pulse laser with 10 ns pulse duration had been employed in tissue ablation, non-invasive treatment for removing stones in kidney, and enabling the tissue fluorescence imaging [2]. Optical metrology is an advance technology where light is used to set the standard that define units of measurement. Optical metrology, on the other hand, is an advance technology where light is used to set the standard for units of measurement. By adopting the ultrafast femtosecond laser, exciting prospects like extensive distance measurement with sub-wavelength resolution over multiple ranges is attainable, like the large scale surface profiling reported in [6].

For the past decade, an increase in the demand for ultrashort pulse lasers operating in visible to mid-infrared spectral range has intensified research activities in this domain. The generation of optical pulses can be achieved via active and passive mode-locking techniques [7][8]. Active mode-locking involves optical modulators which require a driven input signal in a periodic form. These modulators are normally based on optoelectronic devices whereby the pulse duration is limited to picosecond [9]. On the other hand, passive mode-locking is based on a saturable absorber (SA)[10]. It is an essential element that has a specific feature of intensity dependent loss modulation. SA can be classified into two groups; artificial or real SAs. The artificial SAs can be achieved by nonlinear effects such as nonlinear polarization rotation [11]. However, dealing with environmental perturbations can be very challenging as it disrupts the pulse oscillation stability in laser cavities [7][11]. In order to mitigate this drawback, real SA is selected as an alternative to generate shorter pulse durations in the femtosecond region. The first generation of SA was based on semiconductor saturable mirrors (SESAMs) pioneered by U. Keller et al. in 1992 [12]. Her invention created a new scientific discovery path for generating much shorter optical pulses. This versatile device was made possible from current semiconductor technologies. However, the disadvantages of SESAMs that include narrow operational bandwidth due to the semiconductor band-gap structure [13], along with the bulky size and complicated fabrication method using molecular beam epitaxy [14] have prompted for researchers to search for other potential materials.

2D nanomaterials have unique optical properties that offer a vast opportunity in photonic applications. Graphene for example, has an easy and cost effective fabrication method [15], possess gapless linear dispersion of Dirac electrons, ultrafast recovery time, and broad saturable absorption [16]. Other 2D materials such as transition metal dichalcogenides (TMDs), layered molybdenum disulphide (MoS2), and layered black phosphorus (BP) demonstrated better nonlinear optical properties than graphene at particular wavelengths [17]. It has been reported that MoS_2 has stronger saturable absorption than graphene at 800 nm [18] while BP was found to have saturable absorption properties in a wide wavelength range from visible to mid-infrared due to its large bandgap transition [19] .

Metallic nanostructures are known for their optical properties due to the effects of surface plasmon resonance (SPR). SPR is an optical property caused by the coherent oscillations of electron plasma at the surfaces of metallic particles when interacting with light [20]. Nowadays, such particles have been widely applied to various applications such as chemical, biological sensing, microscopic, solar cells and optics[17,18]. Furthermore, because of their field enhancement properties and sensitivity towards the surrounding medium, plasmonic structures play a key role in the development of novel nonlinear optical devices [21-23]. Among metallic nanostructures, gold nanoparticles (Au-NPs) are widely researched owing to its unique electronic, optical and plasmonic properties which have opened up an exciting possibility for ultrafast pulse generation in lasers.

1.2 Problem Statement

According to the literature, Au-NPs are small gold particles with typical sizes ranging from 1nm to 100nm [20]. Optical properties for particles size greater than 10nm govern by the particle dimension known as the extrinsic effect [20]. Whereas, for smaller than 10nm Au-NP, its intrinsic properties become dominant for the electrodynamics effect is independent of particles size dimension and eventually is a source of an additional surface damping [20]. It has two basic nanostructures which are the gold nanorods and gold nanospheres. This material has been investigated and proven to pose saturable and reverse saturable absorption properties [24]. On top of that, other features of Au-NPs, include large surface to volume ratio [21], and the position of SPR can be tailored depending on the size and shape of Au-NPs. As particle size increases, the absorption wavelength of surface plasmon resonance shifts to longer wavelengths and as the increment continues towards the bulk limit, surface plasmon resonance wavelengths move into the infrared portion of the spectrum [21-24], signifying that the tunability of gold nanoparticle saturable absorption waveband is possible across a wide bandwidth [27].

There are many methods applicable to synthesize Au-NPs. The notable four methods are physical, electrochemical, photochemical and liquid reduction, which can be further classified as top down or bottom-up processes [15]. However, these conventional methods have some limitations including the usage of hazardous and toxic chemicals as reduction and capping agents which can be harmful to the environment. Moreover, the undesired components such as unreacted surfactants and other reagents have to be removed to maintain the purity of the colloids. Hence, other approaches, aiming for the safe and easy synthesis of nanoparticles are needed. Pulsed laser ablation (PLA) is a green method in which nanoparticles are removed from the metal plate by laser beam radiation [28]. The method demonstrated by Compagnini et al. in alkanes produced Au-NPs with hydrocarbon chain [29]. This achievement has opened up possibilities for other researchers to study the synthesis of nanometal particles in other organic solvents such as dimethyl sulfoxide (DMSO), tetrahydrofuran (THF) and acetonitrile (CH_3CN)[30]. These organic solvents are commonly used in organic synthesis, however, are usually incompatible with most molecules used in Au-NP functionalization, thus, limiting its potential to be applied with hydrophobic polymer. Among other organic solvents, one study investigated the synthesis of Au-NP using the PLA technique with THF as a medium solvent [30]. Despite reporting good stability, no further investigations were reported on direct synthesis of Au-NPs in THF with regards to size-controlled particles, nor were the ablated nanoparticles ever tested in an SA for femtosecond mode locked laser.

To date, there have only been limited reports of fibre lasers using Au-NPs as a SA for generating Q-switched and mode-locked pulses [24-26]. Based on recent publications, the most common method implementing Au-NPs in SA fabrication is by placing thin nanocomposites film in between fibre ferrule connectors to achieve either q-switching or mode-locking operations[33][34][35][36]. In particular, for passive mode-locking operation, the pulse generated from fibre lasers operates in femtosecond regime has extremely high peak powers. Since the method is prone to the thermal damage due to its extreme intensity, alternative approaches befitted for high thermal damage SA were proposed. One of the alternatives was to manipulate an evanescent field interaction scheme of the propagating light with nanomaterials deposited onto a microfibre [37][38]. However, to ensure a full homogenous coverage when depositing Au-NPs on tapered fibre is challenging task. The only method that has successfully proven to deposit nanoparticles on microfibre is based on optical deposition technique [39]. In this method, the nanoparticles which are graphene are ultrasonicated in chemical solutions and a portion of this mixture was dropped on the microfibre. However, the thickness of the thin film nanocomposites along the tapered region was inhomogeneous and repeatability of the process was not guaranteed. Spin coating has yet to be reported for Au-NP deposition. Nonetheless, it well known in thin nano film fabrication for wafer technology [40]. To achieve this, the tapered fibre plate is placed on the designated chuck, and the chuck will spin in vacuum space for a specific time and speed. This will ensure the same thickness for all the fabricated SAs.

Au-NPs are fascinating due to its unique nonlinear optical properties, whereby its most highlighted features are large third-order nonlinearity [41] and broad absorption governed by localized SPR. Third-order nonlinearity and SPR properties are governed by the particle size of the material which can be tailored accordingly to cater to the need of the intended application. Most of the research on SA in regards to Au-NPs are done by employing gold nanorods [31] [42]. However, it has been reported that interfaces between polymer and spherical nanoparticles is better compared to gold nanorods due to the curvature of the spherical surface of gold nanoparticles, whereby such differences may affect thin film surface thickness resulting in a moderately compressed nanocomposite polymetric layer with higher filling density of AuNPs [43]. In spite of that, limited studies have been reported on 1.55 μm fibre laser using spherical Au-NPs to generate mode-locked pulses. Thus, this has motivated the proposed project to investigate on the effects of Au-NP size towards the performance of Au-NP-based SAs in ultrashort pulse laser systems.

1.3 Research Objective

a) To synthesize Au-NPs directly in THF by implementing pulsed laser ablation method
b) To fabricate Au-NP-polymer based saturable absorber and demonstrate its functionality in erbium-doped fibre laser
c) To compare and analyse the effects of Au-NP size on ultrashort pulse laser performance

1.4 Research Scope

Figure 1.1 is the research scope of the project. Overall, the work focuses on the process of incorporating Au-NP nanocomposites on a microfibre SA and the performance of the Au-NP SA in a femtosecond laser cavity. Two types of Au-NPs have been investigated; synthesised Au-NPs directly in THF via PLA method and commercialized Au-NP/polymer. At varied sizes, these Au-NPs were integrated with microfibre SA. The fabricated SA will be deployed in an erbium-doped fibre laser (EDFL) for testing its functionality to generate ultrashort pulses.

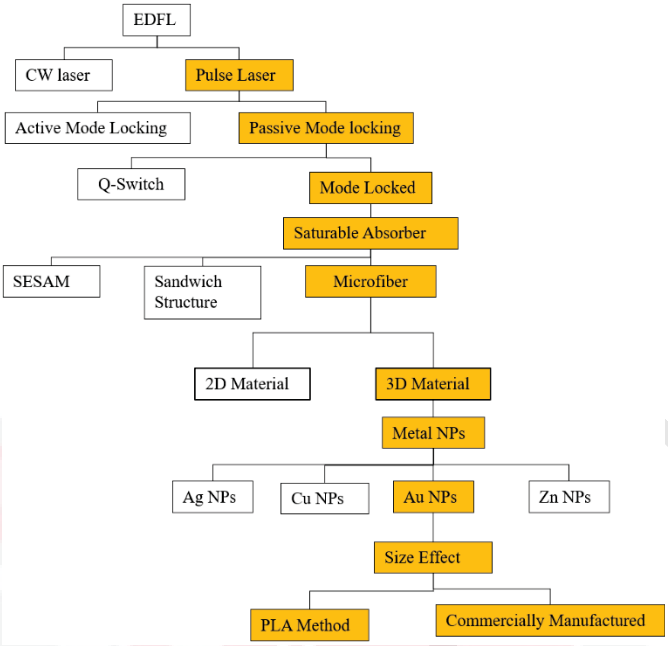

Figure 1.1: The scope of the research

1.5 Thesis Organization

There are 6 chapters in this thesis. Chapter 1 is the introduction of the thesis which explains the application of mode locked pulse lasers in the industry, the trends in passive SA fabrication and the motivation of the study. Objectives are also explained along with the research scope presented in Figure 1.1.

Chapter 2 is the extensive literature review that covers from the recent research done on gold nanoparticle-based SA, theoretical background on the optical saturable absorption that enables gold nanoparticles to be integrated in SA and pulse evolution of the soliton-based mode locked mechanism. Chapter 3 elaborates on the synthesis of gold nanoparticles using the PLA method, for which the formation of the particles in the liquid is explained in detail. Chapter 4 describes the SA fabrication process and mode locking performance by introducing the gold nanoparticle-based SA produced by the PLA synthesis in an EDFL cavity, while Chapter 5 elaborates the effects and comprehensive comparison of different gold nanoparticle size towards the performance of the gold nanoparticle-based SA in femtosecond pulse laser. The last chapter will conclude on the overall observation and findings based on the experimental results as discussed previously. Achievements, problems and future recommendations useful for future research will also be discussed.

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