

Sputter Deposition of Silver Thin Films on Pet and Laser Annealing for Flexible Strain Sensor

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Abstract

Conductive films in flexible strain sensing are becoming increasingly important in wearable health monitoring, but the creation of high sensitivity coatings on thermo-responsive polymer substrates is largely a challenging issue. This report examines the effects of 1064 nm Nd:YAG pulsed laser annealing (PLA) of silver thin films sputter-deposited on polyethylene terephthalate (PET). SEM, AFM, and four-point probe measurements of characterization showed that laser annealing with 51.3 mJ laser energy results in annealing of continuous films with controlled annealing to form island-like nanostructures with a tunnelling-dominated piezoresistive response. As a result, the optimized thin film delivered a spectacular gauge factor (GF) of about 3713 in tensile elongation and 4.5 in tensile bending, and this indicates that measured laser annealing is a viable plan to achieve the sensitivity of flexible strain sensing components.

1. Introduction

In the last few years, wearable health-monitoring platforms have developed, which today have facilitated a non-invasive and continuous process of physiological measurements of healthcare, fitness, and wellness markets. These devices are constantly being enhanced by the natural attributes of wearability, portability, and built-in intelligence [1]. The presence of sensors that will be very sensitive and at the same time stretchable, which will enable the possibility of the entire range of natural human motion, would determine the best functioning of such platforms [2]. Silver (Ag) is a widely used one of the conductive coating materials due to its high electrical and thermal conductivity not to mention that it has a high optical reflectivity thus it is a preferred material when functional coating of electronic packages is required [3]. Polyethylene terephthalate (PET) is the most frequently used substrate that is flexible in mechanics, optically transparent and resistant to chemicals.

Nevertheless, silver thin films may be damaged in terms of their functionality during the manufacturing process. The coating on flexible material frequently results in microstructure defects and surface roughness which can be attributed to either the oxidation or shadowing effect hence deteriorating performance [4,5]. Secondly, silver is not very adhesive to polymeric substrates, it is therefore not good in flex electronics [6]. Silver thin film also has optical properties that can easily be corrupted when exposed to the atmosphere because of the formation of silver oxides and sulfides [7]. Consequently, the extra treatments are necessary following the deposition to modify the microstructure and the morphology of the surface which would enhance the quality and performance of the whole films.

Conventional thermal annealing processes, in particular Rapid Thermal Annealing (RTA) typically subject all of the substrate to hot conditions. This kind of blanket heating is inconvenient when using flexible substrates such

as PET, which needs a significant amount of power, and has the potential to destroy thermally sensitive components. In these considerations, laser annealing (LA) has been proposed to take its place. In contrast to RTA, LA involves the heating of a localized part of the film with a large-energy beam of laser, therefore, leading to recrystallization and promoting the development of grains, without subjecting the entire substrate to heating [8]. As device geometries continue to shrink, the necessity to not only anneal locally and precisely is more of a requirement, another point in favour of the concept of laser annealing over traditional processors [9].

Although the above are the merits of laser annealing, there remains the problem of establishing the optimal conditions of the laser. The properties such as the wavelength, pulse repetition rate and power density have the decisive influence where improper calibration will result in either inadequate annealing or can result in damage of the film. The present research is in this line where it seeks to generate silver thin films using direct-current magnetron sputtering of PET substrates and an experiment to determine the effect on a spectrum of energy densities of an energy density, Nd:YAG pulsed laser annealing. The study is keen on measuring the change in film morphology, electrical conductivity, and electromechanical behaviour, the objective of which is to determine the annealing conditions that yield high-sensitivity of flexible strain-sensing elements.

2. Methodology

2.1 Material Preparation and Deposition

Polyethylene terephthalate (PET) substrates were cut in the size of 1 cm × 1 cm and 1 cm × 3 cm to fit in the characterization needs. Cleanup and sputtering of substrates were done on a Q300TD DC magnetron sputtering system (Quorum). Pure silver target (99.99%) was used. The chamber was pumped to a base pressure of 5×10^{-5} mbar. A deposition was carried out at 40 mA at a time of one minute to yield a film of 26.9 nm in thickness. The structure of the Ag metal thin film on PET substrate is shown in Fig. 1 and Fig. 2 shows the deposited samples ready to be annealed at different energy levels.

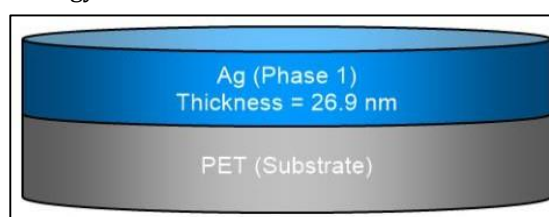


Fig. 1 Structure of Ag metal layer on PET substrate

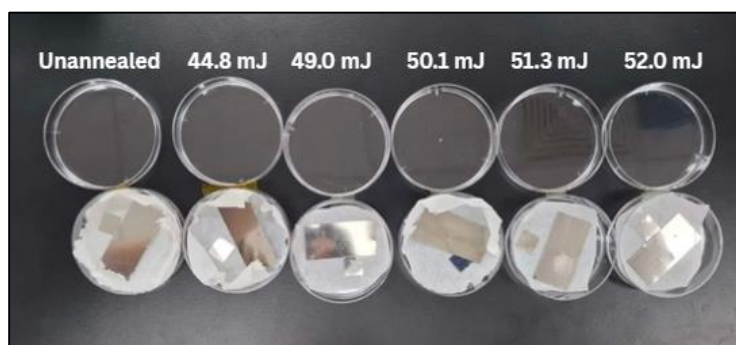


Fig. 2 Samples annealed at different energy levels

2.2 Laser Annealing Treatment

The as-deposited silver thin films have been annealed in air through the use of a Q switched ND YAG laser (Litron Lasers, NANO L290 2D) at a wavelength of 1064 nm. A converging lens was used to obtain a laser pulse of 10 ns, 1 Hz, and 5 mm spot size. Fig. 3 shows the schematic diagram of the setup for laser annealing process. To examine the effect of laser fluence on the film properties, the samples were treated using pulse energy of 44.8 mJ, 49.0 mJ, 50.1 mJ, 51.3 mJ and 52.0 mJ. One of the as-deposited samples was left unannealed to act as a control reference.

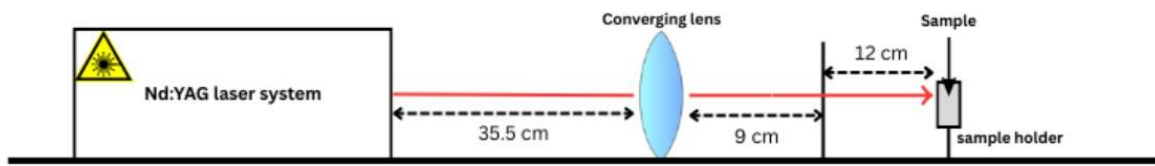


Fig. 3 Schematic diagram of Laser Annealing Setup

2.3 Characterization Techniques

Scanning electron microscopy (SEM) was used to analyse the surface morphology of the films at an accelerating voltage of 20 kV. Topography and surface roughness (root-mean-square, RMS) were measured using atomic force microscopy (AFM) in non-contact mode in a scan size of $5\ \mu\text{m} \times 5\ \mu\text{m}$. Ellipsometry was used to check film thickness. Electrical resistivity was tested using a four-point probe (4PP) system with a distance between points of 1 mm in between and a constant current of 10 mA. The UV-Vis spectroscopy was used to measure the optical properties, which are absorbance and transmittance, in the wavelength range of 300 nm to 800 nm.

2.4 Strain Testing

In order to determine the strain-sensing performance, tensile elongation and bending tests were conducted on the thin films. The samples were the size of $1\ \text{cm} \times 3\ \text{cm}$ and were held in a clamp. Application of a static load at 0 g until 90 g with increment of 10g each step was reached. The change in electrical resistance was expressed in terms of change of relative resistance, $\Delta R/R_0$ [10]. The constitutive relations were used to determine theoretically the tensile strain ($\epsilon_{\text{tensile}}$) and the bending strain ($\epsilon_{\text{bending}}$) of the applied force (F) and the elasticity of PET substrate [11], based on the constitutive equations:

$$\epsilon_{\text{tensile}} = \frac{F}{E \times w \times t} \quad \text{and} \quad \epsilon_{\text{bending}} = \frac{6 \times F \times L}{E \times W \times t^2}$$

where $F=mg$ is the force required, $E=4.7\ \text{GPa}$ is the Young's modulus of PET, w is the width of the substrate, $t=0.25\ \text{mm}$ is the thickness and $L=15\ \text{mm}$ is the distance from the clamps. The gauge factor (GF) was used to measure the sensitivity of the films as the ratio of mechanical strain to change in relative resistance [12]. The strain test was conducted on the silver thin films using the experimental setup as shown in Fig.4 that illustrates the setup for elongation and bending test respectively.

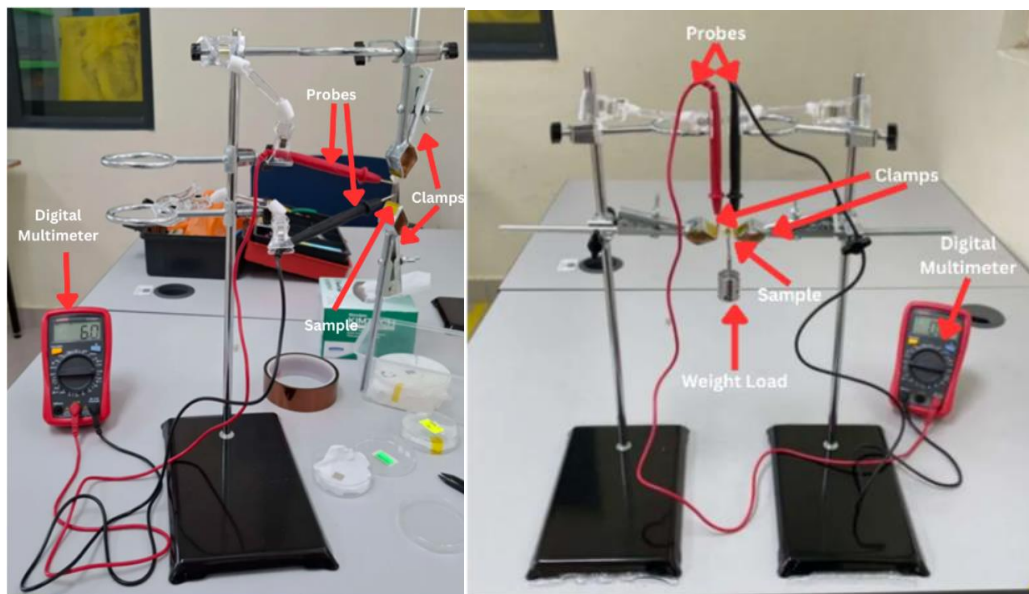


Fig. 4 Photo of setup for strain testing: (a) Elongation and (b) Bending

3. Results and Discussion

3.1 Morphological Analysis

Fig. 3 shows surface morphology of the silver thin films deposited on PET of the as-deposited sample and samples of 44.8 mJ, 50.1 mJ and 52.0 mJ analysing at an accelerating voltage of 20 kV. It can be seen that at different laser annealing energy different structural continuity of the film can be changed. In the annealed samples with lower energy of 44.8 mJ and 50.1 mJ, the continuous film structure is also maintained and both samples evidence the identical results as compared to the unannealed sample. Hence, both energies are not adequate to start the rupture of the film. The surface energy minimization-driven transition between a continuous film and atomically spaced islands is a threshold-sensitive process, which suggests that when the annealing fluence falls below the critical value, the driving force of the thermal process is insufficient to overturn substrate adhesion and rupture the film [13]. Conversely, sample annealed with 52.0 mJ laser energy exhibits high levels of void formation and film rupture that is a sign of structural failure because of excessive heat absorption. All these observations show that moderate laser annealing maintains continuity of the film and improves surface uniformity. However, at high levels of energy, it can ruin the film and this is why the laser parameters should be optimized.

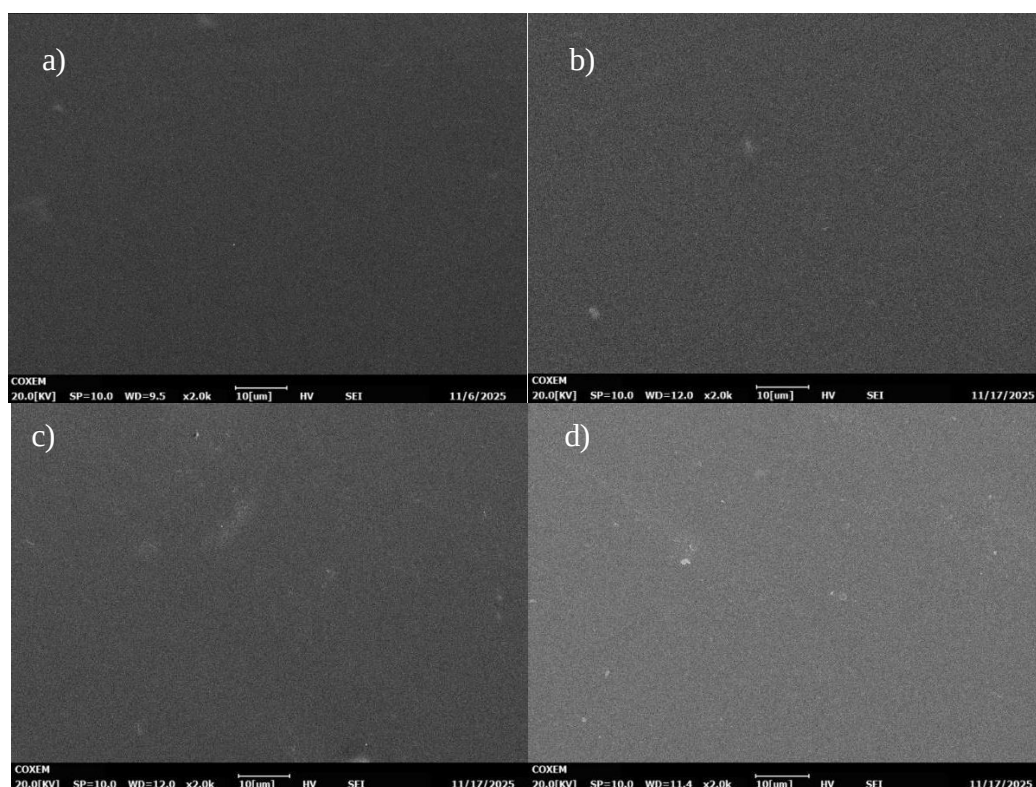


Fig. 5 SEM image of silver thin films a) Unannealed, b) 8V (44.8 mJ), c) 9V (50.1 mJ), and d) 10V (52 mJ)

3.2 Topographical

Fig. 4 shows the topography in three-dimensional AFM images of thin films of Silver deposited on PET of scan size of $5\ \mu\text{m} \times 5\ \mu\text{m}$. As can be seen the changes in the annealing energy of lasers change the surface roughness and grain structure. The roughness of the RMS of materials deposited aids in determining the minimum value of 2.15 nm in the optimum case of RMS roughness versus the optimum laser energy of 51.3 mJ. This flattening effect on the critical fluence demonstrates that the thermal activation was just enough to facilitated the surface diffusion process by which the silver atoms were able to rearrange to form thermodynamically stable and flat nanostructure to reduce surface energy. The best laser annealing conditions are also inclined towards uniform growth of the grain and even distribution of particles that directly translate into lower surface roughness and high quality of the film than the non-uniform agglomeration at non-optimal energies [14]. However, at 52.0 mJ, the roughness increases again to 4.39 nm which is consistent with the film damage observed in SEM. These findings suggest that moderate levels of laser annealing enhance the uniformity of the surface because it facilitates the movement of atoms without major structural disturbance. The rise of the roughness at 52.0 mJ is an indicator that an extreme amount of laser can cause partial film damage, thus proposing that optimization of the conditions under annealing is necessary to maintain constant morphology.

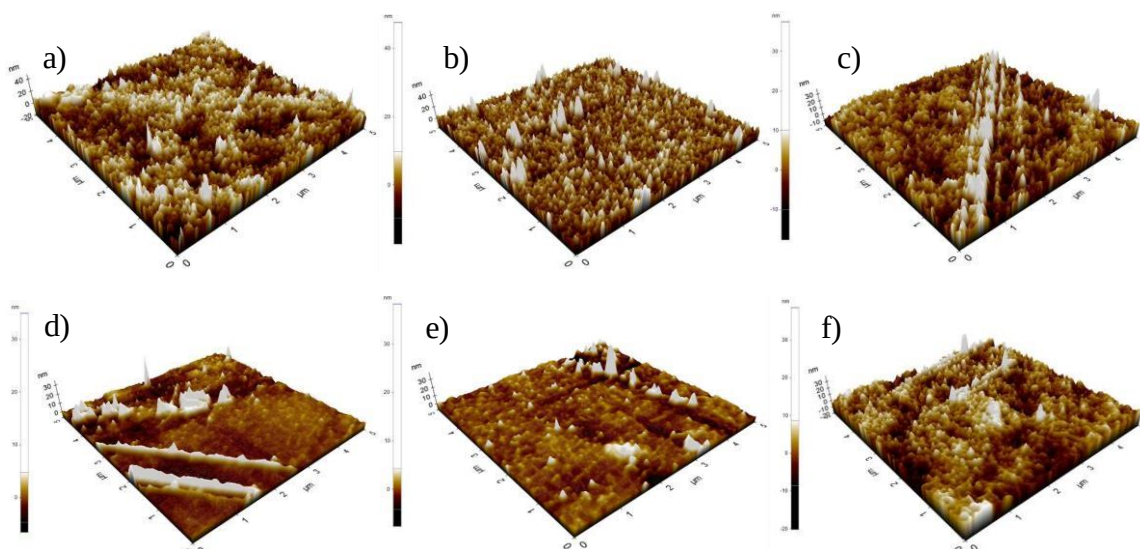


Fig. 6 3D Topography image of silver thin film a) Unannealed b) 44.8 mJ c) 49 mJ d) 50.1 mJ e) 51.3 mJ f) 52 mJ

3.3 Electrical

Fig. 5 shows the average electrical resistivity of the silver thin films on PET through Four-Point Probe method on both the deposited and the post-treatment sample. It is evident that the measured resistivity of laser annealed films are lower than resistivity of the unannealed film where it dropped from $4.46 \times 10^{-2} \Omega \cdot m$ to $1.43 \times 10^{-4} \Omega \cdot m$ at 50.1 mJ. It fluctuates slightly at 51.3 mJ with a resistivity of $1.63 \times 10^{-4} \Omega \cdot m$ and dropped back to the absolute minimum of $1.36 \times 10^{-4} \Omega \cdot m$ at 52.0 mJ. The observed improvement indicates that laser annealing effectively repairs defects in the silver thin films. The rapid and localized heating induced by laser annealing allows precise thermal control, which promotes the growth of more ordered micro-columnar grains. As a result, electron scattering at grain boundaries is reduced, leading to a decrease in the electrical resistance of the thin film [15]. This variation at 51.3 mJ could indicate a shift in conduction path which could be as a result of the development of the discontinuous island structure proposed by AFM data. The enhancement of electrical performance due to laser annealing is because of reduction in defects and enhanced grain connectivity produced by laser annealing. The grooves left by the grains are minimized and the grains are aligned making the channels the charge carriers have to move along to be more continuous making the flow of electrons through the film to be more efficient. The slight rise at 51.3 mJ is associated with the initial formation of isolated islands that is noted in AFM and the conduction is generally high. These findings suggest that the optimization of the laser annealing energy will improve the transfer of electrons without damaging the film structure.

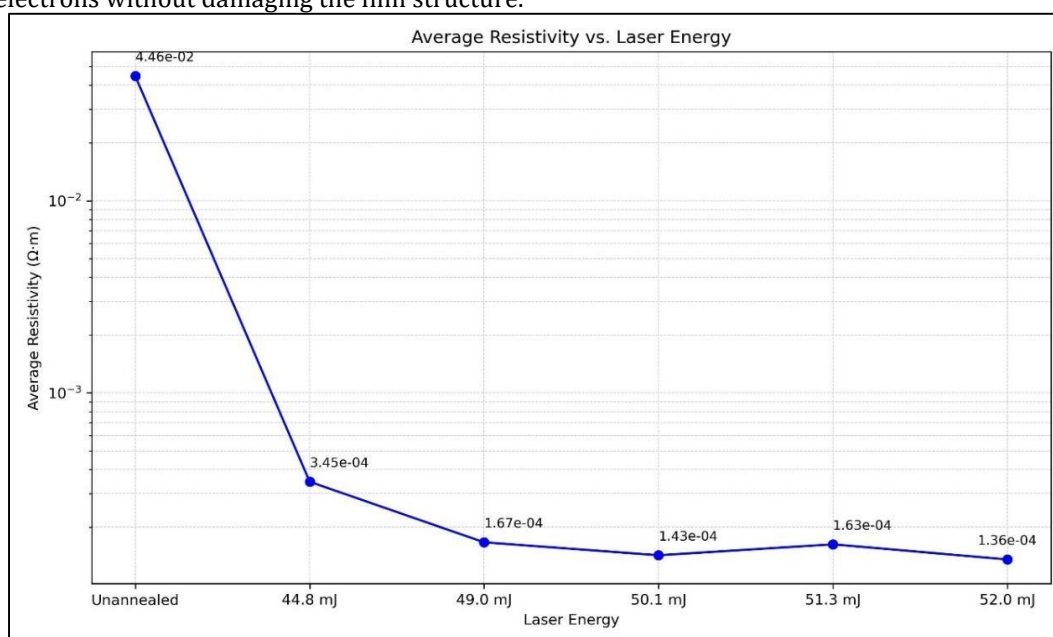


Fig. 7 Average resistivity of thin film before vs after annealing at different laser energy

3.4 Optical

Fig. 6 shows absorbance spectra of thin films of the Silver deposited on the PET at different wavelengths between 200 nm and 800 nm. One can see that the optical resonances in the film are caused by the variation in the laser annealing energy. All the samples have flat profiles, whereas the sample with a treatment of 51.3 mJ exhibits a particular absorbance peak of 350 nm to 450 nm. This finding is optical evidence that the silver film has been converted to discontinuous nanostructures. Noble metal nanoparticles like Silver can be explicitly identified using UV-Vis spectroscopy due to their own Surface Plasmon Resonance wavelength showing the presence of optically active nanostructures despite not necessarily being visible in SEM [16]. Localized Surface Plasmon Resonance peak at laser energy of 51.3 mJ, which is the creation of optically active nanostructures, agrees with the AFM and resistivity data. This affirms that laser annealing with the best fluence will facilitate the creation of functional nanoparticle networks which will improve optical and electrical characteristics. The film is disrupted by higher fluences at 52.0 mJ leading to the downgrade of this optical activity.

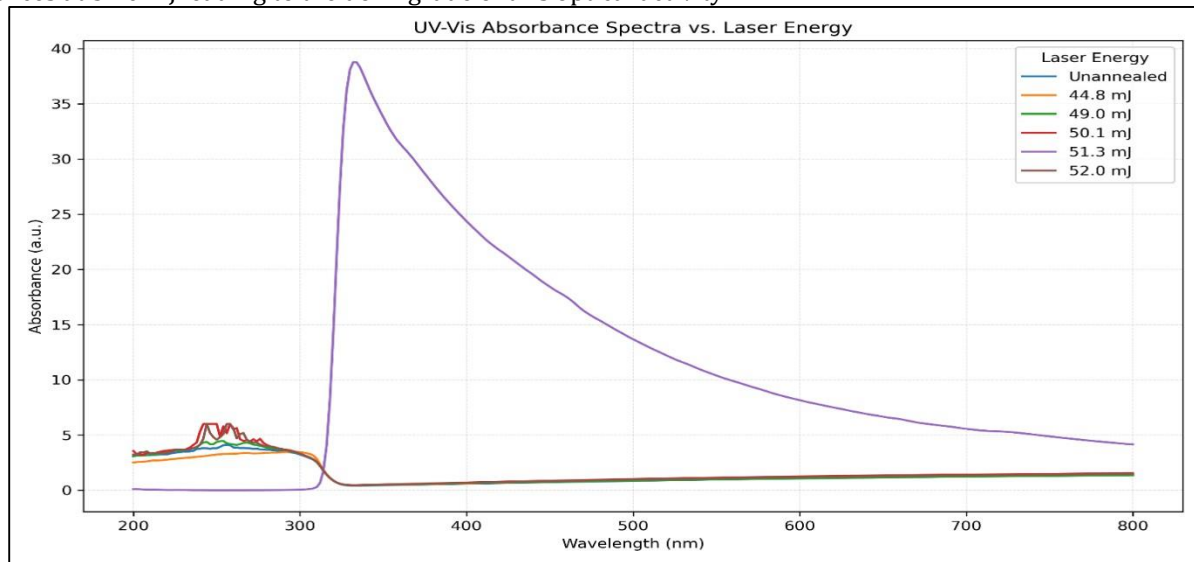


Fig. 8 Absorbance spectra for thin film before and after post-annealing

1.1 Strain

Fig. 7 shows the change in Gauge factor and the change in relative resistance of thin films of Silver deposited on a PET under tensile elongation. It is also clear that differences in the annealing energy in lasers vary exponentially with the sensitivity of the films. The highest response is observed to be by the sample that was treated at 51.3 mJ with the highest giant Gauge Factor of 3,713 with the non-linear resistance change. A tunnelling conduction between the neighboring conductive islands (as opposed to bare metallic stretching) contributes to the resistance to strain change, amounting to about 85% in nano granular film, which confirms the tunneling conduction in the film [17]. Conversely, the sample that was treated with 52.0 mJ has a major decrease in sensitivity to a Gauge Factor of about 1402 following the destruction of this optimized network. These findings indicate that the conductive network is very sensitive to annealing energy and that optimum protein laser annealing sustains structural connectivity to achieve peak strain response but high energy reduces performance.

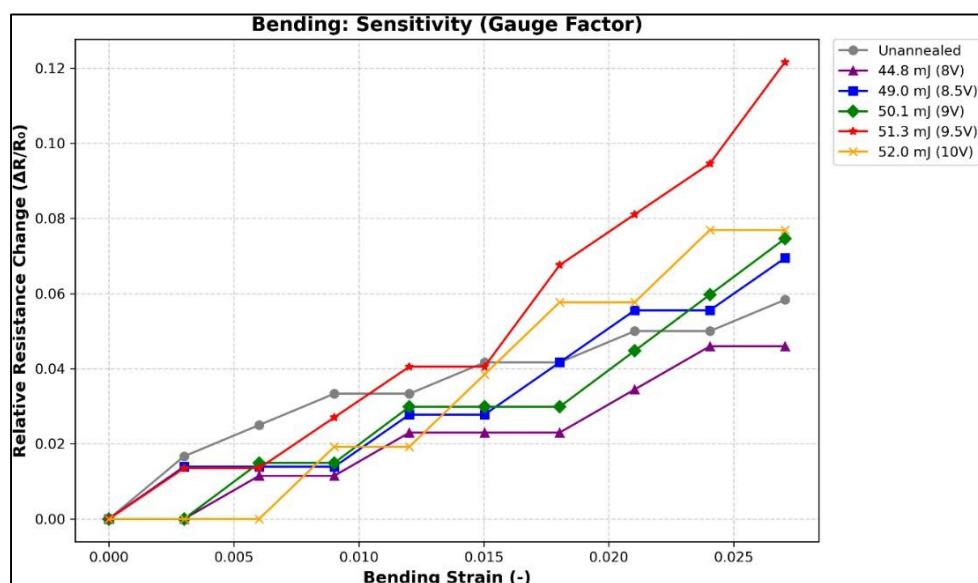


Fig. 7 Graph of Sensitivity of Elongation and Bending

4. Conclusion

This research paper has managed to prove that Nd:YAG pulsed laser annealing is a very efficient technique to optimize silver thin films on PET substrates to use in strain sensing of flexibilities. Although grain sintering at lower laser energies kept the film in a continuous form, the best energy of 51.3 mJ caused controlled annealing of the solid state, converting the film to a uniform network of isolated nanostructures as shown by AFM and UV-Vis analysis. This special morphology trade-off electrical conductivity and mechanical sensitivity on a piezoresistive tunnelling-dominated process that provided a phenomenal Gauge Factor of 3,713 in tensile strain. By contrast, more energetic (52.0 mJ) laser irradiation led to the damage of substrates and rupture of films, which validates the importance of high precision in the control of laser fluence in the creation of high-performance, ultra-sensitive wearable sensors.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design, data collection, analysis and interpretation of results, draft manuscript preparation:** Trisson Cheong Kok Ming, Ahmad Hadi Ali. All authors reviewed the results and approved the final version of the manuscript.

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