



Article **Processing of Single-Walled Carbon Nanotubes** with Femtosecond Laser Pulses

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Abstract: There are continued efforts to process and join single wall carbon nanotubes (SWCNTs) in order to exploit their exceptional functional properties for real-world applications. In this work, we report experimental observations of femtosecond laser irradiation on SWCNTs, in order to process and join them through an efficient and cost-effective technique. The nanotubes were deagglomerated in ethanol by an ultrasonicator and thin slurries of SWCNTs were spread evenly on glass substrates. A laser micromachining workstation for laboratory FemtoLAB (workshop of photonics) has been employed to irradiate the different SWCNTs film samples. The effect of laser parameters, such as pulse wavelength, laser power, etc., were systematically tuned to see the possibility of joining the SWCNTs ropes. Several experiments have been performed to optimize the parameters on different samples of SWCNTs. In general, the nanotubes were mostly damaged by the infrared (1st harmonics femtosecond laser) irradiation on the focal plane. However, the less damaging effect was observed for second harmonics (green wavelength) irradiation. The results suggest some joining of nanotubes along the sides of the focus plane, as well as on the center at the brink of nanotubes. The joining is considered to be established within the region of the high field intensity of the exposed femtosecond laser beam.

Keywords: femtosecond laser; SWCNTs; interconnect; joining

1. Introduction

In recent years, the femtosecond laser appeared as a powerful technique to investigate laser matter interactions for micromachining, surface modification, laser surgery, nanostructure generation, structural evolutions, etc. [1,2]. Particularly, femtosecond laser assisted nano-joining has received great attention due to its critical role in the technological development of microelectronics, MEMS, and nano-devices among various applications [3,4]. Several studies were performed on the joining of different materials with a laser beam in the form of bulk, as well as powder, at the nano and micron scales [2,5–14]. For instance, Zhou et al. joined Al and Fe nanoparticles by exposing them to multiple femtosecond laser pulses [15]. The authors suggested that bonding is attributed to intermixing within a region of high field intensity among particles [15]. Similarly, Zolotovskaya and coworkers reported a scalable technique by demonstrating fast and robust joining of clear glass to glass surface, which contained randomly distributed and embedded spherical silver nanoparticles through the irradiation

of a nanosecond pulsed laser [16]. They reported a joint strength of 12.5 MPa by applying a laser fluence of only ~0.13 J/cm² and a scanning speed of 10 mm/s [16].

Among carbon allotropes, single wall carbon nanotubes (SWCNTs) are one of the amazing high aspect ratio nanostructures that possess remarkable electrical, mechanical, thermal, and optical properties with extreme flexibility. Therefore, they have a great potential for nanoscale device applications [17]. An individual nanotube acts as a genuine quantum wire and demonstrates a perfect one-dimensional (1D) nature of conducting modes. Furthermore, the electron motion without backscattering in metallic nanotubes results in perfect conduction, even in the presence of scatters [18]. Therefore, SWCNTs are an excellent candidate for power and signal interconnection due to their ability to conduct large current densities, as the conventional metallic interconnects have severe limitations due to feature size shrinkage [19,20]. Despite their immense significance, there are substantial challenges in synthesizing techniques for their broad range applications. For instance, it is difficult to fabricate the electrically homogenous SWCNTs by controlling the chirality, diameter, and length in a scalable and high-speed manner [21]. Furthermore, SWCNTs are usually grown in the form of a mixture of semiconducting and metallic nanotubes that hinders the broadened application of nanotubes in electronics [22]. Regardless of these challenges, there is an interest to fully exploit their exceptional properties for real-world applications by joining them in networks or bundles while keeping intact their exceptional properties [23]. Based on their amazing electronic transport properties, the microbundle array of carbon nanotubes are showing a promising application in field emission devices [24]. Consequently, it is highly desirable to establish true joints between SWCNTs, having the same order of magnitude of mechanical/electronic properties that is retained by an individual single wall nanotube [23].

In general, few studies were conducted on the joining of low dimensional carbon allotropes. For example, in a novel experiment, stable crosslinks between neighboring carbon nanotubes within bundles were formed by moderate electron beam irradiation and reported improved intertube bridging [25]. Moreover, multiwall carbon nanotubes joining were reported by continuous fiber laser irradiation on silicon and silica substrate [26]. The nanoscale welding of MWCNTs for three-second irradiation was attributed to the breaking of C–C bonds and the formation of new graphene layers [26]. Recently, Li and coworkers formed a cross-stacked CNT architecture and reported joining through sp³ covalent bonding under high pressure and temperature through laser heating [14].

Danilov and coworkers ablated dense SWCNT based bulk materials at two wavelengths, with single and multi-shot laser exposure. They reported less damage with shallow material removal, and high damage demonstrated by micro craters by single and multi-shot laser irradiation, respectively [27]. On the other hand, Arutyunyan et al. [28] studied the selective removal of a specific category of SWCNT thin film by resonant ablation through femtosecond pulses. Most recently, the transformation of SWCNT structures into other allotropic carbon structures with effective coalescence through femtosecond laser irradiation was reported by Ha et al. [2].

Despite the enormous potential, the joining of SWCNTs has been a less focused area, and no reports were published that focused on the joining of SWCNT networks in the form of thin film by processing via femtosecond irradiation, to the best of our knowledge. There are several benefits of using the femtosecond laser technique. The irradiation of the femtosecond laser generally has an ultrafast impact that results in non-thermal melting of solids, and thereby is promising for the joining of nanostructures. For instance, the short pulse duration delivers localized high peak power through the laser spot, thus minimizing the surrounding heat diffusion and thermal damages [29,30]. Furthermore, the ultrashort time scale of the femtosecond laser thus non-thermally induces the changes in the structure of SWCNTs, and thereby transforms it into a different allotropic form [31,32]. Thus, the unique properties of the femtosecond laser could be beneficial for joining the amazing one-dimensional material of SWCNTs through femtosecond laser irradiation. Therefore, in this work, a simple and effective strategy has been employed to join the SWCNTs by femtosecond laser irradiation for potential apparitions as highly

conducting interconnects. Detailed scanning electron microscopy (SEM) was performed to analyze the ablated nanotubes, and the effect of laser processing parameters on joining/burning of SWCNTs.

2. Materials and Methods

2.1. Sample Preparation

Single wall carbon nanotubes (SWCNTs) were procured from Emfutur (Vila-real, Spain) in the form of powder. Typically, SWCNTs are measuring a few nanometers in diameter and several microns in length. A small amount, approximately ranging from 0.1 to 0.5 g of agglomerated powder of SWCNTs, was dispersed in 50 mL ethanol. The solution was sonicated by the ultrasonicator (Q500, USA) to deagglomerate and disperse the SWCNTs in ethanol for around half an hour. After sonication, the uniformly dispersed SWCNTs in ethanol mixture were dropped out on glass substrates and made a thin film of SWCNTs. Several samples were prepared with different thicknesses in the range of one to few microns. The schematic for the steps involved in the preparation of SWCNTs film is shown in Figure 1. The thin films of SWCNTs were irradiated by femtosecond laser pulses by adopting different strategies to optimize the various machine parameters.



Figure 1. Schematic illustrating the process for preparation of single wall carbon nanotube (SWCNT) thin film.

2.2. Laser Irradiation

A laser micromachining workstation from FemtoLAB (Workshop of Photonics) having 6 W PHAROS laser and a harmonic generator (Light Conversion, Lithuania) was employed to irradiate the samples according to the desired settings. The system is equipped with a high accuracy linear positioning stage, high performance galvanometer scanners, and versatile micromachining software SCA (Workshop of Photonics, Lithuania). The samples were irradiated with two wavelengths, an infrared (IR) 1028 nm (1st harmonic), and a green 514 nm (2nd harmonic) with a time duration close to 200 fs and a frequency of 100 kHz.

The 1st harmonic (IR) experiments were performed with an average laser power ranging from 0.05 to 80 mW. An aspheric lens with a focal length of 15 mm. For this wavelength, the plane of

laser irradiation ranged from the actual focus plane of the top surface of the SWCNT ropes up to 200 μ m away from the SWCNTs. The 2nd harmonic tests were performed with two types of lenses, a 15 mm aspheric lens and a Zeiss A-Plan 10X dry objective, while for the first harmonic experiments only a 15 mm lens was used. The laser power, which ranged from 0.68 to 40 mW, was used in these experiments. The transitional speed of the stage was varied from 0.03 mm/s up to 0.1 mm/s along the line of laser irradiation for both laser wavelengths used. Experiments were conducted by identifying the plane of focus of the samples with the aid of a machine vision setup included in the femtosecond laser system. Then, according to the experimental settings, a single pass was performed with laser irradiation for each setting for the sample. The SWCNTs processed by femtosecond laser irradiation were analyzed by scanning electron microscopy.

Further analysis of the experimental settings was performed to find the optical properties of the samples for joining. For the current experiments, the spot size (D) of the laser beam was calculated using the following equation, as described in the literature [33]:

$$D = 1.22 \frac{\lambda}{N.A}$$

where λ is the laser wavelength and *N*.*A* is the numerical aperture values of the objective lens. Thereafter, the average fluence was estimated by employing the following equation [33]:

$$F_{ave} = \frac{E * F}{D * v}$$

where *E* is the energy of single pulse, *F* is the frequency of laser pulses, i.e., repetition rate, and *v* is the laser scan speed.

3. Results and Discussion

The as received SWCNTs were characterized by the transmission and scanning electron microscopy before femtosecond laser irradiation. The representative high-resolution transmission electron microscope (TEM) image of SWCNT ropes and the typical scanning electron microscope (SEM) image of SWCNT networks are shown in Figure 2a,b, respectively. The TEM image is showing several SWCNTs in the form of ropes. The zoomed image of SWCNT ropes is shown in the inset of Figure 2a. The average diameter of a single wall carbon nanotube is ~1.67 nm, as estimated from the TEM. It is a well-established fact that a metal catalyst is indispensable for the synthesis of SWCNTs through the chemical vapor deposition process. Therefore, particles inside the SWCNT ropes are metal catalyst particles. The most widely used transition metal catalysts for SWCNTs growth are Fe, Co, Ni, etc. and are reported in several studies [34,35]. It is obvious from the representative image of the SEM that the ropes of the SWCNTs have a complex network pattern after dispersing. A large number of samples of SWCNTs were irradiated by the femtosecond laser to investigate the effect of laser parameters such as pulse wavelength, fluences, and laser power etc. to join the SWCNT ropes into bundles.

Initially, the first set of experiments were performed with the first harmonic (infrared wavelength) for various power settings, and the results varied according to the laser power and plane of irradiation. The experiment was performed in a way by varying the laser scan rate so that the networks of SWCNTs scribed by femtosecond laser for a fraction of a second. Since the femtosecond laser gush energy over a temporal scale was shorter than the phonon-electron relaxation time [36], it is unlikely that they become oxidized substantially during this shorter period of exposure time. However, the slight oxidization, if occurred, is favorable in the sticking of SWCNTs through oxidation debris in the form of bundles in accordance with the literature [37]. The majority of experiments showed that this infrared wavelength ablates the SWCNT bundles at the line of irradiation and largely burned the nanotube bundle network. Typical images for the 1st harmonic experiments with the effect of reduction in fluence are shown in Figure 3. Figure 3a,c show the full and partial destruction of SWCNTs at the laser irradiated area, respectively. The burned nanotubes are clearly shown in Figure 3b in the zoomed picture. Keeping

first harmonics, if we reduce the power further, then less damaging effects are observed as having partial ablation as shown in Figure 3c,d. The quantity of burned nanotubes is substantially lower, with a significant reduction in power with resulting fluences of 169.77 J/cm² to 21.22 J/cm². However, few joining could be suggested at the interface of intact and damaged nanotubes as indicated by the arrows in Figure 3a,c. There is an interesting feature that most of the suggested joining are found at the two sides of the plane of irradiation, as shown by the arrows, and this pattern is observed for most of the experiments. Several experiments that were performed with changing the plane of irradiation and power variations for the 1st harmonics showed similar outcomes. Overall, low power settings for the 1st harmonic experiments offer somehow better results, with less damaging of the SWCNTs predominantly at the edges of the irradiation line.



Figure 2. Images of single-walled carbon nanotubes before irradiations. (**a**) High resolution TEM image and (**b**) SEM image of SWCNTs.



Figure 3. SEM images of SWCNT samples after IR laser irradiation with fluence reduction effects. (a) 169.77 J/cm² and (c) 21.22 J/cm² with 15 mm lens; (a,c) macroscopic view of area of SWCNTs irradiation, and (b,d) microscopic view of side regions showing connectivity.

To improve the results, the second set of experiments was performed by adopting the green wavelength (GR) 2nd harmonic from the harmonic generator. In this series of experiments, different power reduction levels as low as 541.14 J/cm² were used for GR. The energy per pulse remains in the range of nanojoules. Typical SEM images of irradiated SWCNTs at different power levels are shown in Figure 4. In general, it is observed that less ablating effects are witnessed as compared to IR with GR irradiation on nanotubes. Initially, 848.85 J/cm² fluence was opted with a 15 mm lens. The results shown in Figure 4a,b are almost similar with the first harmonic irradiation but substantially less burning is observed. The joining is believed to occur, as shown by the circles in Figure 4b. The laser parameters were further tuned at green wavelength by changing the power settings as low as fluence 541.14 J/cm² and replacing the lens to 10X. The results are shown in Figure 4c,d. At this lower fluence and lens setting, the ablation has been reduced considerably and joining is suspected at the end (white phase) where the laser interacts with the bundles. The arrows/circles in Figure 4c,d show suspected welded nanotubes represented by the white phase contrast. The best results achieved so far are shown in Figure 4e,f. The selected area in Figure 4e has been zoomed in Figure 4f. The image is showing clearly suspected welded nanotubes with the second harmonic wavelength. Yuan et al. applied a continuous fiber laser on multiwall carbon nanotubes to join them. The authors proposed that the accumulation of absorbed energy as the irradiation time increases leads to the breaking of C–C bonds, and the formation of a robust graphene layer junction that leads to the bonding among MWCNTs. In the current study, with the exposure of the femtosecond laser at very low power and second harmonics, leads to better joining of nanotubes in agreement with the literature [26]. As SWCNTs have a single wall, therefore, they need substantially less energy and time to join and therefore, a more damaging effect was observed during the experimentation on SWCNTs. It is well established that most of the SWCNTs are semiconducting in nature while a small fraction consists of a metallic nature [38]. Furthermore, the metallic nanotubes have a higher thermal conductivity as compared to semiconducting. Moreover, the glass substrate on which nanotubes are dispersed has lower thermal conductivity values. Therefore, it is anticipated that due to the accumulation of absorbed energy that a localized heating has occurred, and results in the form of joining at the end where the ropes/bundles are exposed to the laser irradiation. The SWCNTs are present in the form of a network of ropes/bundles in thin film, while some of the nanotubes are comprised of semiconducting and others are metallic in electrical characteristics. Since each category responds differently with the irradiation of the femtosecond laser, it is difficult to quantify the results of joining due to the complex nature of carbon structures. However, the SEM images suggest that the ropes of SWCNTs within a bundle are joined at the end point where the laser has burned them.

An interesting observation is that both the absorbance and fluence have a substantial impact on the femtosecond laser processing of SWCNTs. For instance, a comparison between the IR and GR irradation effect is conspicuous by analysing Figure 3a,b and Figure 4a,b, where the IR irridation, even with less fluence (169.77 J/cm²), ablated SWCNTs completely on the line of action as compared to GR (848.85 J/cm²), respectively. One of the possible reasons that GR has less destruction of SWCNTs than IR could be the lesser penetration depth of GR as compared to IR in the nanostructured materials [39,40]. Furthermore, optical properties for 15 mm and 10X Zeiss lenses, as well as different fluence values, affect the size of the irradiated zone and welding patterns. This is evident from the SEM images with GR in Figure 4e where the microscope lens (10X Zeiss) showed a more localized effect with less width of the irradiated zone, as compared to the 15 mm lens in Figure 4a with a broadened width of irradiated zone indicated by the red arrows.



Figure 4. SEM images of SWCNTs sample after irradiation with (**a**,**b**) green wavelength (GR) with a fluence of 848.85 J/cm² with 15 mm lens; (**c**,**d**) GR with a fluence of 541.14 J/cm² with 10X Zeiss lens, and (**e**,**f**) GR with a fluence of 2705.71 J/cm² with 10X Zeiss lens.

The SEM images in Figure 4 show that interaction between SWCNTs film and femtosecond laser is predominantly a non-thermal mechanism [41], as described in the two-temperature model [42]. However, at higher fluence it partially ablates the materials accompanied by the joining of nanotubes. It is further highlighted that the SWCNT ropes are tiny clusters of limited atoms as their aspect ratio is high, so there is a possibility of global melting at some points in the film without thermal melting, as reported in the literature and observed in this study [42].

Despite the fact that in nanosecond laser irradiation a similar phenomenon of heating, melting, and evaporation occurred, such a thermal mechanism in the processing of SWCNTs with the femtosecond laser is improbable to exist. This is because the electron's lattice thermal coupling, as well as thermal diffusion to the lattice, takes a substantially longer time than the laser pulse width. Consequently, electrons do not have enough time to transfer energy to lattice [42]. In non-thermal heating, at the

fluence above a surface threshold value [41], the electrons are excited, ejected, and weaken the bonds at surface atoms. This results in surface melting which is beneficial for the joining of nanostructures without damaging the whole entity [41,43].

Figure 5 shows the proposed mechanism for thermal effects of femtosecond and nanosecond laser pulses on SWCNT. For a longer pulse duration, the entire nanotube is heated, as the thermal effects are dominant, and it is equivalent to annealing by the continuous laser wave or conventional thermal sintering [41]. Whereas for femtosecond laser irradiation, only the surface heating will be observed, as laser pulse duration is considerably smaller than the characteristic time for thermal diffusion. Consequently, it is favorable for the joining of nanotubes [44]. As shown in Figure 4d,f, the morphology of the joined nanotubes is almost unchanged and they well kept their original shape. This suggests surface melting, which is somehow attributed to non-thermal effects of the femtosecond laser. The number and quality of nanotube joints are high in femtosecond irradiation of SWCNTs, as clearly shown in Figure 4f, compared to a similar study of continuous fiber laser on multi-walled carbon nanotubes (MWCNTs) [26]. Other authors have also shown similar phenomenon of non-thermal heating in the joining of nanostructures by femtosecond laser irradiation [41,42].



Figure 5. Thermal effects of femtosecond and nanosecond laser pulses on single wall carbon nanotube (SWCNT).

This preliminary study suggests that the femtosecond joining technique for SWCNTs is simple and cost-effective but needs further refinement of experimental parameters, and special sample preparation for specialized applications. The simplicity of this technique overcomes a number of challenges that come across in the conventional joining technique for applications in microelectronic devices and sensors. Further investigations on the characteristics of welded/joined sections are part of our ongoing studies.

4. Conclusions

SWCNTs in the form of thin film were uniformly distributed on glass substrate. A femtosecond laser pulsed with IR and GR irradiated SWCNTs by varying the power, linear speed and lenses. The detailed electron microscopy was performed to see the effect of femtosecond irradiation on burning/ joining. The results showed that in most of the cases for IR irradiation that the nanotubes were predominantly damaged by the femtosecond laser irradiation, even at a low fluence of 21.22 J/cm² with the 15 mm lens. However, the GR irradiation showed somewhat better results for both tested optical lenses compared to the IR wavelength. The best-localized and irradiated resulted patterns were identified for 541.14 J/cm² and 2705.71 J/cm² fluences with less damaging effects, and showed somehow the joining of nanotubes with the 10X Zeiss lens. Due to the complex nature of SWCNTs based on their electrical properties, such as semiconducting and metallic characteristics, further optimization of parameters and characterization are required to understand the mechanism of joined bundles of

nanotubes. This preliminary study shows that the femtosecond laser could play a pivotal role in forming high performance electrical interconnects in nano-systems and MEMS technology.

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