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Selective laser treatment and laser patterning of metallic and semiconducting nanotubes in single walled carbon nanotube films

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ABSTRACT

Single wall carbon nanotubes (SWCNT) synthesized using mass production methods such as pulsed arc deposition consist of a mixture of metallic and semiconducting nanotubes. In this work, we report on an approach for the selective removal of either metallic or semiconducting SWCNT by a heat-treatment process with cw-lasers and pulsed lasers with specific wavelengths. The results show that using ultraviolet–visible radiation (with wavelengths between 473 nm and 632 nm) it is possible to remove predominantly metallic nanotubes. In contrast, near infrared lasers with 785 nm and 1064 nm wavelengths can be used to remove predominantly the semiconducting nanotubes. Finally, the fabrication of SWCNT films with an anisotropic distribution of metallic and semiconducting nanotubes is demonstrated using a direct laser interference pattering method.

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1. Introduction

Since the early nineties, single walled carbon nanotubes (SWCNT) have been known for their extraordinary properties [1].

Although technologies like pulsed arc technique were optimized for high quality SWCNT mass production [2,3], many scientific and technical challenges have yet to be overcome. For example, it is still challenging to make use of SWCNT because of van der Waals forces leading to bundling. Exfoliation of SWCNT from bundles is difficult and keeping nanotubes exfoliated in devices is still an issue [4]. Furthermore, the produced SWCNT material contains a mixture of metallic (m-SWCNT) and semiconducting (sc-SWCNT) nanotubes. However, especially for micro and nano electronic devices like logic circuits or field-effect transistors based on SWCNT [5,6] an exfoliation of SWCNT bundle and the separation of m- and sc-SWCNT are required. There are different techniques for separation of a small amount of m- and sc-SWCNT with e.g. density-gradient ultracentrifugation [7] but also gel electrophoresis processes are investigated for up-scaling of the separation process [8,9]. The mostly complex and time-consuming separation of metallic and semiconducting SWCNT is necessary in order to make them processable in electronic applications.

There are also different fabrication techniques available for a preferential synthesis of m- or sc-SWCNT (with purities up to 90%) but the processing of either pure m- or sc-SWCNT is currently just possible for small nanotube amounts [10].

Recently, it has been shown that using different light sources such as lasers or UV-lamps, a preferential removal or degradation of m-SWCNT is feasible [11–15]. The selective laser degradation of sc-SWCNT was observed by Iijima et al., who dispersed SWCNT bundles in hydrogen peroxide (H_2O_2) and treated them with a 488 nm continuous wave (cw) laser [16]. However, any systematic study describing the effect of wavelength, laser intensity and removal conditions has not been realized up to now. In this work, we utilized a laser treatment using both cw- and pulsed systems with wavelengths ranging from UV to NIR to systematically investigate the effect of the laser radiation on the selective removal of mixed m- and sc-SWCNT bundles in air. The SWCNT material used in this work was synthesized by an optimized pulsed arc technique. Raman spectroscopy [17] and optical absorbance spectroscopy (OAS) [18–20] were utilized to characterize the SWCNT bundles before laser modification. Furthermore, thermogravimetric studies were performed to investigate the thermal stability of as-produced SWCNT-material in air. Finally, the fabrication of SWCNT

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films with enriched areas of metallic and semiconducting nanotubes was manufactured using a direct laser interference pattering method with a pulse (10 ns) laser system operating at 532 nm laser wavelength. With this paper we show a promising way for the separation of m- and sc-SWCNT without complex exfoliation and separation steps which are often coupled with a shortening of the SWCNT by sonication [21].

2. Experimental

The SWCNT used in this work were produced with the pulsed arc technique. After the synthesis, the SWCNT were purified from amorphous carbon and from most catalyst particles by H_2O_2 and acid treatment with HCl [22,23]. The SWCNT samples in the experiments were prepared by spraying a thin SWCNT film on an object plate made of quartz glass or silicon. The thickness of the film was in the range of 1 μm.

The optical treatment was performed at six different laser wavelengths (473 nm, 514 nm, 532 nm, 632 nm, 785 nm and 1064 nm) as well as a UV-lamp with 800 W (Xe-Arc-Lamp). The lasers with 473 nm, 514 nm, 632 nm, 785 nm and 1064 nm were continuous wave (cw) lasers. The power densities of the continuous lasers were varied between 0.0085 and 3.5 mW/μm^2. The 532 nm laser was a pulsed laser (Spectra Physics) with a pulse length of ~10 ns. The energy density per pulse was 20 mJ/cm^2. The laser patterning experiments were performed at ambient conditions and evaluated by Raman spectroscopy. The Raman spectra were measured with laser intensities of ~0.0085 μW/μm^2.

Direct laser interference patterning (DLIP) was performed at 532 nm wavelength. In those experiments, the primary laser beam was split into two beams which were guided to the sample, interfering at a specific angle (1.25). Further details can be found elsewhere [24].

3. Results and discussion

Fig. 1a shows the typical G-peak of the Raman spectra of purified SWCNT measured at a wavelength of 514 nm. The G-peak in this spectrum is a superposition of up to 10 subpeaks from m-SWCNT and sc-SWCNT [17,25–28]. The G-subpeaks from m-SWCNT and sc-SWCNT are split into axial G^+ and tangential G^- modes. Within these modes, the tangential mode of m-SWCNT (G^-_m), and sc-SWCNT (G^-_{sc}), are useful to distinguish between sc- and m-SWCNT (Fig. 1a) [17,25–28]. The highest intensities have the G^-- peaks at ~1540 cm^{-1} and ~1570 cm^{-1} for m- and sc-SWCNT, respectively [17,27]. These peaks were approximated with Lorentzian fits using the G-peak positions shown in Table 1. However, it should be noted that the Raman spectra measured at a laser wavelength of 632 nm have a broad asymmetric G^-_m peak, which is related to the presence of free electrons in the metallic nanotubes [17]. In this case, the G^-_m peak needs to be fitted with a Breit–Wigner–Fano function (BWF) depicting a larger full width at half maximum [17,18,25,28]. The BWF width decreases however for larger tube diameters (wavelengths of 785 and 1064 nm excite large tubes, see Kataura plot in Fig. 1c) and higher photon energies (for example 514 nm and 473 nm). In consequence, the BWF can then also be fitted with the Lorentzian model [17,29].

The produced nanotube material by the pulsed arc technique is comparable to that synthesized by laser techniques and shows a low defect density [2]. The optical absorption of purified SWCNT is shown in Fig. 1b. This spectrum is different from the absorption of graphite because the density of states (DOS) is quite different in one dimension systems like carbon nanotubes [1]. The OAS of SWCNT is characterized by several bands referring to the interband transition energies of van Hove singularities as well as excitons within the nanotubes [18,19,30].

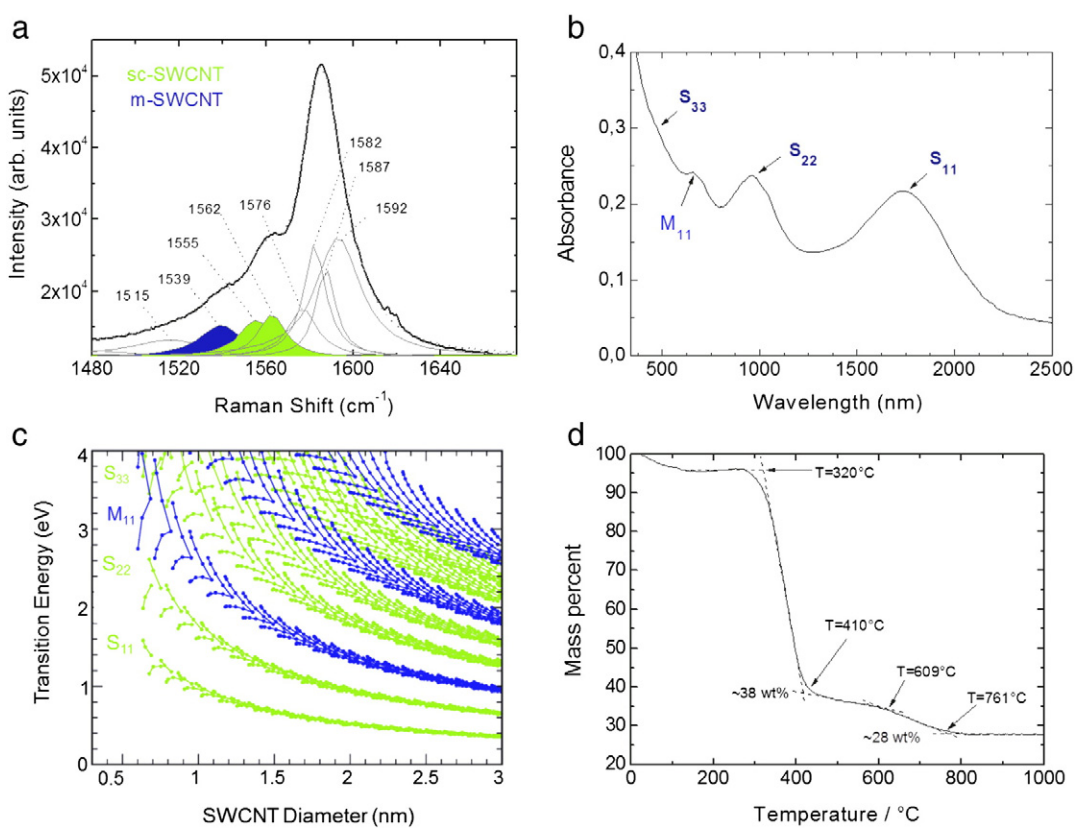

Fig. 1. (a) Typical Raman spectrum of purified SWCNT measured with a laser wavelength of 514 nm. The G-peak is decomposed into his sub peaks, the peaks resulting from metallic and semiconducting nanotubes are indicated as blue and green (in the color version), respectively. (b) Optical absorption spectrum of purified SWCNT measured with UV–vis-NIR spectroscopy. (c) Kataura plot showing the relation between the tube diameter and the excitonic optical transition energies between van Hove singularities in m- and sc-SWCNT [31–33]. (d) Thermogravimetric study of as-produced nanotubes at ambient conditions. .

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