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Low-cost synthesis of high-quality graphene in do-it-yourself CVD reactor

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ABSTRACT

This paper reports design of a very low-cost (less than 2000 USD) chemical vapour deposition (CVD) reactor for graphene synthesis, based on readily available off-the-shelf components. The reactor is intended for in-house graphene synthesis in metamaterial research. The reactor was manufactured and tested, and synthesized graphene samples were characterized by Raman spectroscopy. Measurement results revealed high quality of synthesized single and multi-layer graphene samples.

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1. Introduction

Graphene [1] is material composed entirely of carbon atoms arranged in the two-dimensional hexagonal lattice structure. Due to its one-atom thickness (approximately 3 Angstrom) it is the thinnest material available. In addition, the strong bonds between carbon atoms result in thermodynamic stability of graphene sheets [2] even when suspended in air. Graphene has exceptional mechanical and thermal properties. The Young's modulus is 1 TPa, the tensile strength reaches a value of 100 GPa, while the thermal conductivity is 5000 W/mK [3]. From an electrical engineering point of view, a graphene is actually a zero-gap semiconductor material that supports ballistic transport [4] accompanied with very high charge mobility of 200,000 cm²/V [5] and the sheet resistance of 125 Ω⁻¹ [6]. Particularly interesting is the fact that graphene can be viewed as a plasmonic material with the plasma frequency in the THz band [7]. In principle, plasma frequency can be tuned electrically, by changing electrochemical potential [8]. This interesting property was used in recent proposals of various THz and optical metamaterial-inspired structures such as absorbers [9], antennas [10], single-atom thick waveguides [11], reflectors [12], and lenses [13]. In addition, there have been several attempts to use non-linear electromagnetic properties of the graphene, in order to construct high-speed THz modulators [14]. Of course, all of these interesting engineering applications presume that there is a reliable and affordable graphene synthesis technology.

Production methods range from low-tech to high-tech methods, each one offering pros and cons depending on the final application and available budget. The oldest one is simple scotch-tape mechanical exfoliation of pristine graphite [15]. Graphite itself is a bulk

material composed of many graphene layers stacked together. In the process of mechanical exfoliation, a Scotch-tape is pressed with its adhesive side onto a graphite block. The pressure is applied uniformly by fingers over all contact surfaces assuring a good mechanical contact. When the tape is removed, some layers of graphite block are peeled off by tape's adhesive agent and this material remains fixed on tape surface. Peeled material comprises a very high-quality single-layer graphene (SLG) flakes together with flakes of multi-layer graphene (MLG). The identification of peeled layers and thereof thickness determination (Figure 1(a)) is usually done by applying atomic force microscopy (AFM). Although very simple, the Scotch-tape mechanical exfoliation is not popular, due to its poor repeatability.

Better repeatability can be achieved by chemical reduction of exfoliated graphite oxide (GO) [16]. In this method, a graphite block is chemically delaminated by applying a hot solution of strong oxidative agents such as sulphuric acid. During this process, delaminated graphene sheets are oxidized with OH, COOH, epoxy and other chemical functional groups that make GO soluble in water. In addition, a small volume of graphite block can produce an excessively large volume of GO suspension. The surface that should be coated with graphene is sprayed with GO suspension and left over to dry. Drying process squeezes GO sheets by the capillary force of evaporating water. However, the chemical bonds between different sheets are not formed yet and repeated contact with water would wash away already formed GO layer. Finally, the formation of chemical bonds between the sheets and the removal of functional groups is achieved by a chemical reduction in hydrazine or ascorbic acid solution. The result

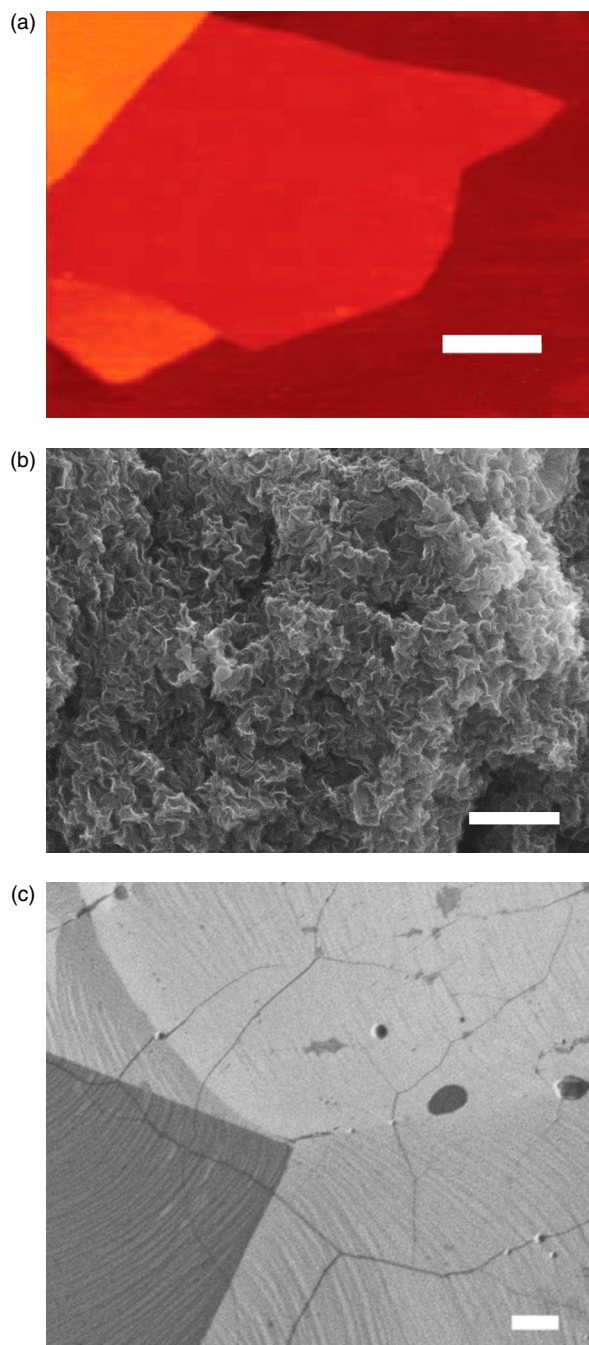


Figure 1. (a) AFM image ([15], used with permission from American Association for the Advancement of Science, licence number 4294310476933) of single-layer graphene: SiO₂ surface (right bottom), 0.8 nm height (central area), 1.2 nm height (bottom left), 2.5 nm height (top left); (b) SEM image of reduced graphene oxide [20]; (c) focused ion beam image of CVD single-layer graphene produced in this work. Dark lines are graphene wrinkles formed in cooling process after synthesis. In all three images, the length of scale bar is 1 μm .

of this process is a compact graphene layer with the high surface area (Figure 1(b)). The method of chemical reduction has good repeatability, it is medium-cost and enables the production of large graphene sheets. However, there are numerous defects and vacancies in material produced by this method. This important drawback prevents the use of chemically reduced GO in demanding applications such as high-speed electronics due to

the poor mobility of charge carriers. Thus, the method of chemical reduction is used for low-end products applications such as conductive electrodes for touch-screen [17] and graphene-based composite materials [18,19].

Usually, large samples of high-quality graphene are synthesized using the chemical vapour deposition (CVD) method [21–23]. This is very well developed but rather an expensive method that applies high temperatures and low pressure. Starting materials are molecules in a gas phase that can be decomposed at a surface of the heated substrate. Products of decomposition can be either in solid or in the gas phase. Gas products can diffuse out of a reaction zone where they are removed by vacuum pump while the solid products remain on the substrate surface. With longer processing time the solid products accumulate as more and more molecules are decomposed. The surface covered in solid products grows gradually and finally forms a new material. In graphene synthesis source of carbon atoms can be hydrocarbon gas or alcohol vapour, typical synthesis temperature is 1000°C and typical pressure is in Torr range. The substrate (also called a catalyst) is usually a foil made of transition metals such as Cu, Ir, Ru and Ni. Different kinds of graphene can be grown, depending on the substrate used in the process. Specifically, the SLG and BLG (bi-layer graphene have two layers) is grown on copper (Cu) foils. On the other hand, the FLG (few layer graphene have three to nine layers) is grown on nickel (Ni) foils. Different kinds of graphene can be grown, depending on the substrate used in the process. Specifically, the SLG–BLG–FLG is grown on copper (Cu) foils while the FLG–MLG is grown on nickel (Ni) foils. The most important advantage of the CVD method is the high quality of synthesized SLG (Figure 1(c)).

The pros and cons of graphene production methods discussed above are summarized in Table 1. A quick examination of the table leads to the conclusion that the CVD method offers the high quality of graphene samples, which is needed for high-tech applications. However, it is clear that the main drawback of the CVD method is its high cost. The cost of CVD-produced graphene is approximately 1–2 USD cm⁻² [24] and its retail price is around 10 USD cm⁻² [25]. Furthermore, the CVD reactor per se is of rather complex construction, which makes it moderately expensive device. For instance, a price of laboratory desktop reactor can easily reach tens of thousands USD [26], which is not affordable for many research groups. Of course, one may opt to use the graphene sheets purchased directly from manufacturers [27,28], that are delivered via public mail service. It is interesting that not many groups use purchased graphene. The main problem is that a graphene sample can easily absorb water vapour and other impurities while exposed to air ambient (during the time between synthesis and the

Table 1. Comparison of graphene production methods.

	Mechanical exfoliation (Scotch-tape method)	Chemical exfoliation/GO chemical reduction	Chemical vapour deposition
Quality of synthesized graphene	Very high	Poor	High
Maximum electron mobility (cm ² /Vs)	200,000 [5]	10 [29]	350,000 [30]
Feasibility of SLG production	Yes, with difficulties	No	Yes
Scalability	Not scalable	Scalable	Scalable
Maximum sample size	Approximately 100 μm	Arbitrary	7.5 m ² [31]
Production price (per 1 cm ²)	–	< 0.1 USD	< 1 USD
Production method complexity	Low	Medium	Very high

use in the experiment). The aim is the minimization of uncertainty and repeatability in the experiments. This resulted in a widespread tendency of in-house synthesis, just before the actual experiment, for which the graphene sample is required.

In this paper, we report a low-cost yet very effective CVD reactor built from common households and readily available off-the-shelf components. We show that samples produced in this reactor have a low concentration of defects that result in high-quality products adequate for use in demanding laboratory devices.

2. Do-it-yourself CVD reactor

Commercial CVD reactors are highly priced due to their complex design that involves high temperature materials, power electronics, precisely machined parts, sophisticated electromechanical valves, gas cabinet with valves and feed lines, vacuum equipment, software/hardware for process automation and individual customer approach in assembly and start-up. The backbone of a typical CVD system is a reactor tube in which processing is done. Depending on reactor application, the tube diameter varies from 10 mm (intended for producing small laboratory samples) to more than 200 mm (intended for processing industrial wafers). Electric heater (in a form of heating wire) is placed in close contact with reactor tube to provide heating. Thermal insulation is often used to prevent the heat loss. Both tube ends are capped with leak-proofed flanges that enable easy opening/closing, samples loading/unloading, vacuuming/venting and gas insertion.

Our reactor (Figure 2(a,b)) directly applies the basic theoretical principles of VA-CVD (vacuum assisted-CVD) explained above. It uses methanol vapour as a carbon source [32], with working temperature up to 1000 °C. The reactor has been assembled from commercial low-cost off-the-shelf components and refurbished household products.

The reactor tube is based on a fused silica tube (internal diameter of 8 mm, the wall thickness 1 mm and the length of 300 mm), taken from a low-cost domestic infrared heater. Two aluminium flanges were machined with outside dimensions of a standard vacuum flange type KF-16 and centre-drilled with 10 mm drill bit, so reactor tube can tightly fit inside this hole. Flanges are glued to both ends of the reactor tube with PDMS –

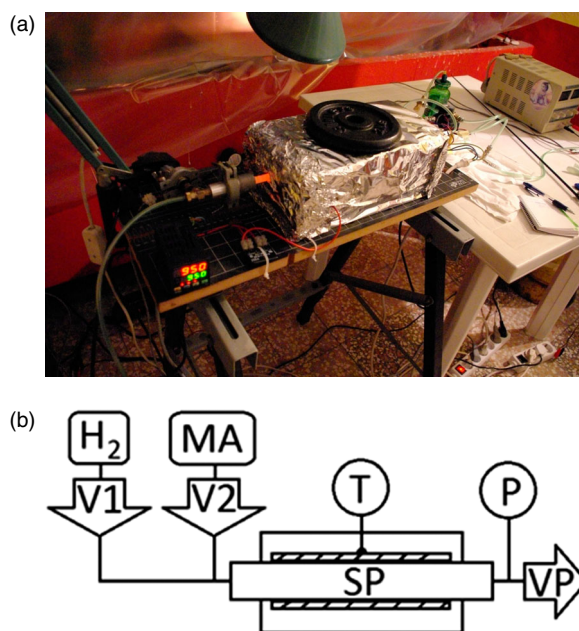


Figure 2. (a) Developed CVD reactor during graphene synthesis; (b) block diagram of reactor: H₂ – hydrogen source, MA – methanol vapour source, V1 – hydrogen needle/electro-pneumatic valve, V2 – methanol needle/electro-pneumatic valve, SP – substrate place, T – thermal controller, P – pressure gauge, VP – vacuum pump. Heater is represented by shaded area.

silicone polymer that is resistant to high temperature. This resulted with leak-proof joints compatible with laboratory vacuum systems. Input flange is naturally cooled with cold gas that flows in the reactor. If the input flange is not cooled with outside fan its temperature will rise but generally, it will be below PDMS's degradation temperature. Output flange is heated with hot gas that flows from the reaction zone to output port. If the output flange is not actively cooled it will rapidly reach PDMS's degradation temperature. Two fans with a diameter of 10 cm were detached from personal computer's power supply and were used as coolers for both flanges.

The reactor tube was wound with a Kanthal wire that acts as an external heater with electric power of 100 W. In order to sustain stable process temperature, the heater was controlled by a cheap thermal controller with K-thermocouple. The heat shield that covers the reactor tube was made of Rockwool, commercial mineral wool that is commonly available as sheets in shops

for civil construction. Rockwool sheets are made of basalt fibres tolerant to high temperatures with melting temperature specified as $> 1000^{\circ}\text{C}$. Unfortunately, maximum usable temperature (in a continuous mode of operation) for this material cannot be found in the datasheets. However, from the experience in this work, we estimate that the uncontrolled temperature increase will destroy a heating wire before the melting temperature of basalt fibres is reached. Thus, the initial testing has proven that the properties of basalt fibres are quite satisfactory for use in the CVD reactor. Measured thermal conductivity (at the working temperature) was in the range $0.2\text{--}0.3\text{ W/mK}$ which would result in a notable heat flux if just single sheet were used. Thus, multiple sheets were stacked together, both below and above the reactor. Typically, it was found satisfactory to use two to three sheets. The outside wall of thermal insulation was overwrapped into domestic

aluminium foil, in order to reduce crumbling of isolation material and to additionally reduce thermal loss to the environment.

In addition, the gas flow was controlled by a set of electro-pneumatic and needle valves commercially available for aquarium venting. Methanol vapour source consists of 30 ml glass jar with a metal cap on top that can be periodically removed to refill the jar. Cap is drilled and fitted with a hose adapter. To prevent accidental air leaks inside the jar, the contact surface of the cap is greased with silicone grease prior to closing the jar. Before starting the batch it is essential to evacuate jar to the base pressure of methanol vapour to decrease the amount of residual air left inside the jar. For minimizing residual air quantity, one can use multiple rinsing with hydrogen gas followed by evacuation.

A dual stage rotary vane vacuum pump was used for reducing working pressure and to remove the gases

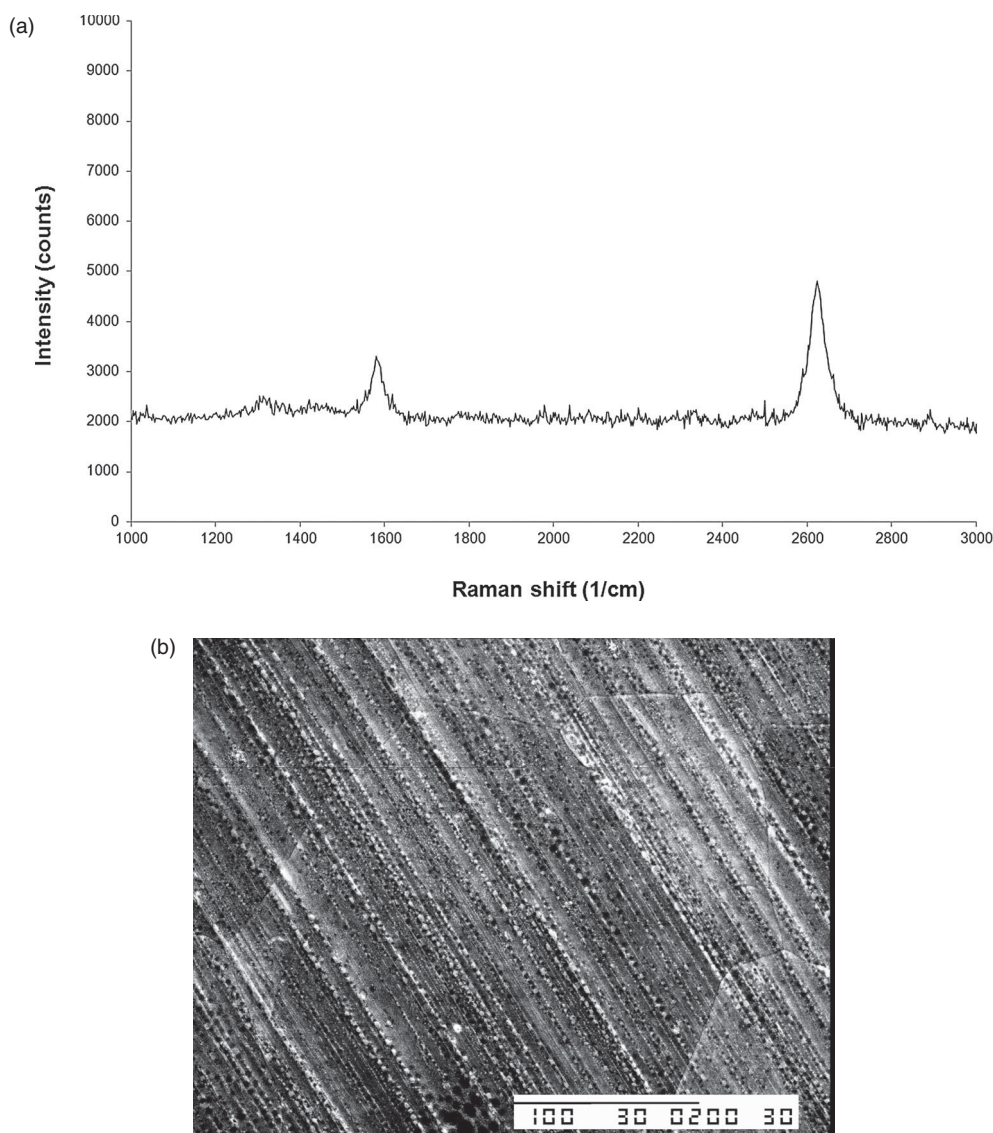


Figure 3. (a) Measured Raman spectrum of synthesized graphene on copper substrate shows high-quality single-layer graphene with negligible defects; (b) SEM (Scanning Electron Microscopy) image of SLG on copper surface shows large graphene domain size of $> 0.1\text{ mm}$. Cold rolling of copper foil produces grooves that are also visible in this image as parallel lines. Size of the scale bar is 0.1 mm .

from the reaction zone. Gaseous by-products were vented into the atmosphere through a bubbler made of a glass jar filled with tap water. Waste gases are carried away from the vacuum pump to bubbler by polyethylene hose that is at least 3 m long. Surface of bubbler must be vented with large fan driven by a brushless motor in order to prevent hydrogen forming an explosive mixture with air. Frequency of formed bubbles is directly proportional to the gas flow rate inside the reactor tube so this simple method can be used to monitor processing gases flow rate. Pirani vacuum sensor was used as a pressure gauge.

The Cu-foil was used as a substrate material for synthesizing SLG while the Ni-foil was used for FLG and MLG. Metal foil (dimensions 50×5 mm, thickness 0.2 mm) was cut with scissors from a larger sheet, washed in isopropyl alcohol, followed by rinsing in deionized water and placed in the middle of the reactor tube. After the vacuum pump was turned on, the pressure was reduced to remove air, water, and solvent vapour from the reaction zone resulting in a base pressure of 2–3 mTorr. After this first step, the solenoid valve for hydrogen gas was turned on. The flow of 100 SCCM raised the working pressure to 1–2 Torr. Five minutes after starting hydrogen flow the heater is turned on. Hydrogen served as a substrate oxidation protecting agent while the reactor reached its working temperature of 1000°C . Typical heat-up time is 5 min. The substrate annealing was continued (at previously set working temperature) for next 30 min to clean metal substrate surface from oxides and to initiate growth of substrate grain size as is recommended in [33]. Annealing process enlarges Cu grain size from a typical range of 10–20 μm to 100–200 μm or more. Using a substrate with larger grain size will produce graphene with fewer boundaries per square area and therefore such material will have higher quality. In the next step, the H_2 flow was stopped and the methanol vapour pressure of 15 Torr was introduced for the next 30 min. Finally, the methanol flow was stopped and the heater was turned off, allowing the reactor to cool down to room temperature with an average rate $50^\circ\text{C min}^{-1}$.

3. Graphene characterization

In the first step, the initial characterization of produced graphene samples was performed. Synthesized graphene on copper was characterized by Raman spectroscopy [34]. The result shows the SLG all over substrate surface with a negligible amount of defects (Figure 3(a)). The 2D peak at 2700 cm^{-1} is higher than the G peak at 1580 cm^{-1} indicating the presence of a single-layer graphene. Furthermore, negligible D peak at 1350 cm^{-1} shows a low concentration of defects [21,35]. As expected, it was found that the substrate annealing in hydrogen significantly lowers the concentration of graphene defects due to the reduction of

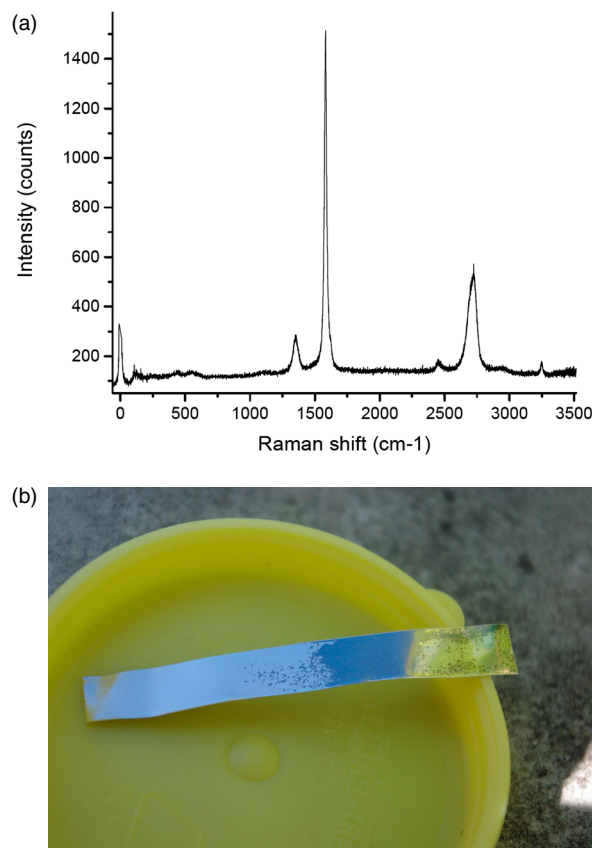


Figure 4. (a) Measured Raman spectrum of grey deposit on nickel substrate shows multi-layer graphene with medium concentration of defects resulting in low-quality of such product; (b) MLG on nickel substrate can be identified with unaided eye as grey deposit almost uniform in colour intensity (middle-right part). Rapid cooling part of nickel substrate had no visible deposits (left part).

substrate surface oxides and the enlargement of grain size (Figure 3(b)).

In addition, the multi-layer graphene was synthesized using a nickel foil base and using the same process parameters (Figure 4(a)). A number of layers can be controlled by methanol vapour exposition time and the substrate cooling rate as a more critical parameter [36]. If the substrate cooling is rapid, the precipitation of carbon from nickel matrix will be slight, if any, resulting in poor graphene deposits on the substrate surface (Figure 4(b)). As a rule of thumb, a recommended value of substrate cooling rate is in range $1\text{--}10^\circ\text{C min}^{-1}$.

The low cooling rate resulted with the Ni-substrate fully covered with compact MLG deposit (Figure 5(a)). To the unaided eye, the deposits of five samples looked uniformly greyish. However, the optical microscopy revealed the variations of light intensity at localized areas, the dimensions of which resemble the substrate grain size. The most probable cause of this effect is the variation of carbon segregation speed from nickel matrix with respect to the different crystal orientation of substrate grains. The measured Raman spectrum of the aforementioned samples revealed the high quality of MLG material (Figure 5(b)).

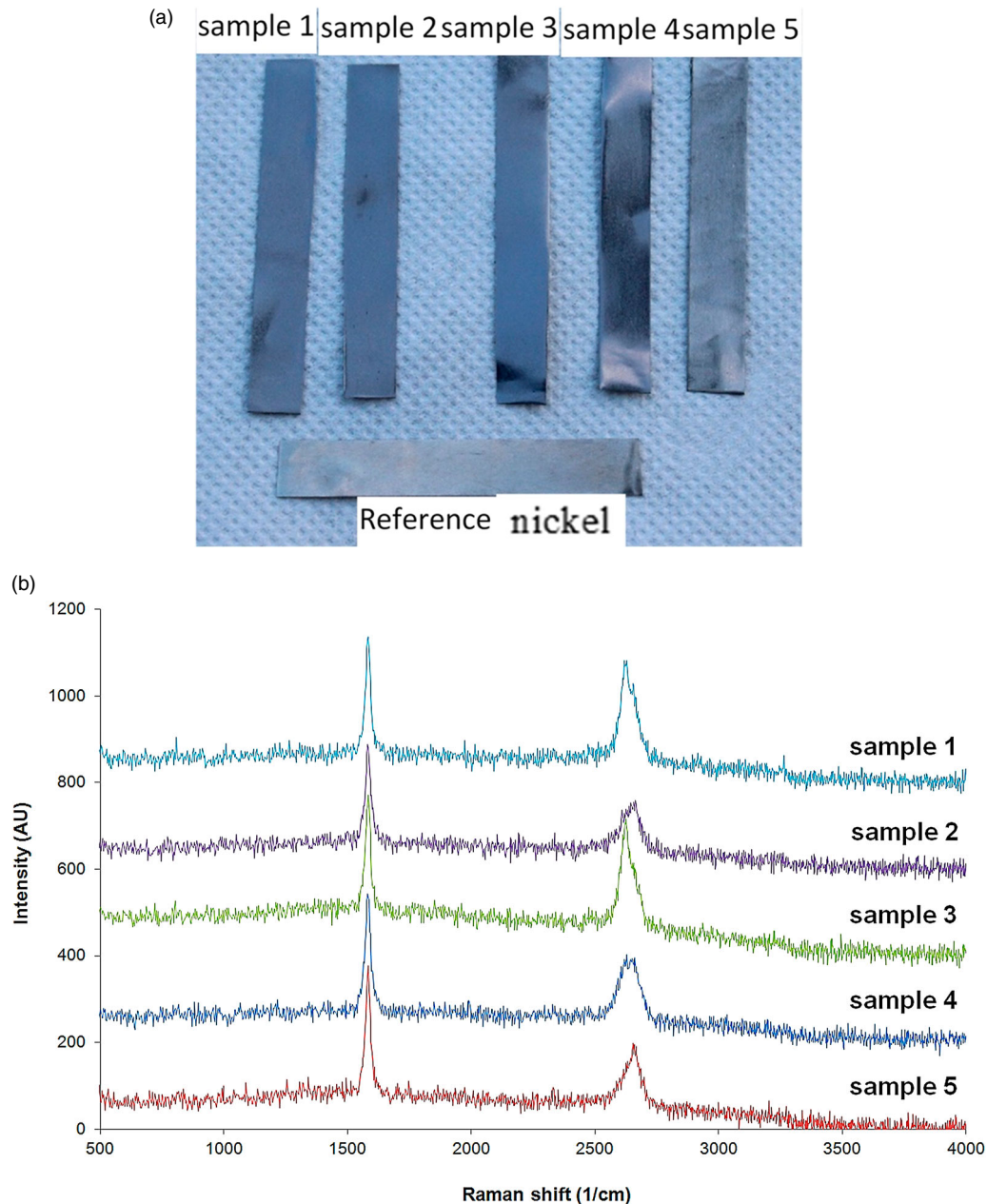


Figure 5. (a) Nickel substrates 1–5 (top left is sample 1, top right is sample 5) covered with the MLG deposits. The samples are of a dark-grey colour, and they can clearly be distinguished from the reference nickel substrate (bottom) (it is of a shiny silver colour, typical for metals); (b) measured Raman spectrum of produced samples. It was found that the spectrum is generally uniform across all substrate surfaces. The deposits have negligible concentration of defects and therefore they are of high quality.

All presented results show that the properties of a synthesized graphene are very similar to those of commercially available graphene sheets [27,37].

4. Conclusion

The low-cost (less than 2000 USD) small CVD reactor for graphene synthesis, based on readily available of-the-shelf components, has been designed, prototyped and tested. Synthesized samples of a single-layer graphene were characterized by Raman spectroscopy, revealing a material of high quality suitable for use in the microwave and optical experiments. However, the multi-layer graphene samples that were produced with high cooling rates had unsatisfactory quality with

most of the surface completely uncovered. Reducing the cooling rate resulted in reproducible uniform deposits of MLG with good quality. Fused silica tube from the low-cost domestic infrared heater was used in this work as CVD reactor tube. The inner diameter of the tube is 8 mm which presents a limitation to maximum sample width. The future research will involve electromagnetic (microwave) and optical characterization of the synthesized graphene samples. It will be done by the measurements of scattering parameters of a graphene sheet placed in the waveguide, and by performing optical ellipsometry. The final goal is the use of synthesized graphene in the construction of a simple microwave metasurface-based absorber.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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