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Synthesis of uniform monolayer graphene on re-solidified copper from waste chicken fat by low pressure chemical vapor deposition

Mohamad Saufi Rosmi[a,b,1,*], Sachin M. Shinde[a,1], Nor Dalila Abd Rahman[c], Amutha Thangaraja[a], Subash Sharma[a], Kamal P. Sharma[a], Yazid Yaakob[a,d], Ritesh Kumar Vishwakarma[a], Suriani Abu Bakar[c], Golap Kalita[a,*], Hajime Ohtani[e], Masaki Tanemura[a]

[a] *Department of Frontier Materials, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466-8555, Japan*
[b] *Department of Chemistry, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, Malaysia*
[c] *Department of Physics, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, Malaysia*
[d] *Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia*
[e] *Department of Materials Science and Engineering, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466-8555, Japan*

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ABSTRACT

A technology for converting waste materials to high quality large-area monolayer graphene film can be significant and thereby obtaining high value-added product. Here, we revealed the transformation of waste chicken fat into uniform monolayer graphene film on re-solidified Cu by a low pressure chemical vapor deposition (LPCVD) technique. The evolve gas analyzer-gas chromatography–mass spectrometry (EGA-GC–MS) analysis of chicken fat oil showed that the free fatty acid in chicken oil decomposed into a short hydrocarbon chains which makes it favorable to use as a carbon precursor for graphene synthesis. Growth of uniform monolayer graphene film on the re-solidified Cu was confirmed by Raman mapping, where 2D to G peak intensity ratio (I_{2D}/I_G) is 3.0 at most of the area. Thus, the use of waste from poultry industry as a carbon source instead of commonly used hydrocarbon gas sources for graphene synthesis can be an approach for green nanotechnology.

1. Introduction

Chemical vapor deposition (CVD) technique has been significantly explored owning to the possibility of synthesizing high quality graphene film in large-area [1–3]. Recent studies also demonstrate control synthesis of bi-layer stacks and large single crystal domains on the metal catalytic substrate [4–10]. The synthesis of graphene film has been achieved by low and atmospheric pressure CVD techniques using methane as the primary carbon source [2,11–14]. Meanwhile, liquid and solid carbon sources, such as ethanol, benzene, camphor, poly (methyl methacrylate) (PMMA) and sucrose have been investigated for graphene synthesis as an alternative to gaseous sources, taking into account the advantages of simpler and controllable CVD process [15–19]. As in line with green nanotechnology concept, solid waste carbon precursor such as camphor, waste plastics, coffee ground, insects, and food waste are gaining more attention in graphene synthesis recently [19–22]. These materials are highly available and can be directly placed in the CVD chamber without any external carbon source supply system. However, considering the possibility of high quality graphene synthesis by a low pressure CVD (LPCVD) technique, the choice of solid or liquid carbon precursor is very much critical as the vapor pressure of precursor significantly influence the carbon flux.

Thus, we explored the feasibility of using waste from poultry processing industries, namely waste chicken fat (WCF) to synthesize high quality graphene in a LPCVD process. WCF is a free fatty acid (FFA) rich resources, thereby making them as a good option for alternative and promising carbon source. Recent reports show that WCF is composed of oleic ($C_{18}H_{34}O_2$), palmitic ($C_{16}H_{32}O_2$), and linoleic ($C_{18}H_{32}O_2$) acids [23–25]. High purity

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[*] Corresponding author at: Department of Frontier Materials, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466-8555, Japan.
E-mail addresses: saufirosmi@gmail.com (M.S. Rosmi), kalita.golap@nitech.ac.jp (G. Kalita).
[1] These authors contributed equally.

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and controllable vapor pressure of WCF widens its usage either in atmospheric pressure CVD (APCVD) or LPCVD. Furthermore, the high consumption of chicken nowadays indicates the high availability of waste discarded by the chicken processing industry as compared to other poultry. With poor current management practices of food waste like incineration, which resulted in secondary pollution throughout greenhouse gas emissions, the utilization of WCF into high value added product would not only reduce the amount of solid waste, but it is also contribute to conserve natural resources, and save energy and hence fuel [26,27]. In this regard, we demonstrate for the first time, the direct utilization of WCF without any special treatment to synthesis high quality graphene by the LPCVD method. The high quality and excellent transparency of homogeneous monolayer graphene were confirmed by Raman, AFM, SEM and UV–vis analysis, respectively. Less wrinkles formation in the as-synthesized monolayer graphene on a re-solidified smother Cu surface was achieved, enabling fabrication of transparent electrode with low sheet resistance.

2. Experimental details

Graphene films were grown on 20 μm thickness Cu substrate (Nilaco Corp., purity 99.9%) using LPCVD method. The Cu substrate was annealed in an enclosed tube at 1100 °C for 60 min with flow of 100 sccm (standard cubic centimeter per minute) of H_2 gas. Next, Cu substrate was loaded into the quartz tube of length 90 cm and diameter of 5 cm. The chicken oil, which was used as a carbon precursor was extracted from chicken fat and skin by a dry rendering process (Fig. 1a and b). In this process, the fat and skin were heated to 200 °C to separate liquid oil from solid [24]. Magnetic boat containing chicken oil was placed near the opening of the inlet. Fig. 1c shows the schematic diagram of LPCVD used in the graphene growth. The system was pumped down to a vacuum pressure of 2 Pa for 10 min, and then heated to 1080 °C within 120 min with 100 sccm of pure H_2 gas. Then, the mixture of Ar and H_2 gas with a molar ratio of 98:2 sccm were introduced into the LPCVD system and the magnetic boat containing chicken oil was brought into the zone and placed at 5 cm away from the zone for the graphene growth at 1080 °C under pressure of 150 Pa for 60 min. After the completion of the synthesis, the furnace was allowed to cool down to room temperature.

In order to transfer graphene, PMMA was spin coated at 3000 rpm for 1 min followed by baking at 180 °C for 1 min. Next, the transfer was carried out by electrochemical delamination to avoid contaminations. 0.25 M sodium hydroxide (NaOH) was used as an electrolyte at 10 V and 400 mA. The PMMA/graphene/Cu was dipped into a NaOH aqueous solution and used as a cathode of an electrolysis cell with a constant current supply. At the cathode, a water reduction reaction took place to produce H_2 bubbles. The PMMA/graphene layer was observed to be detached from the Cu substrate after a few seconds as a result of the formation of a huge number of H_2 bubbles at the interface between the graphene and Cu substrate. After cleaning with pure water, the floating PMMA/graphene layer was transferred to the target substrate. Finally, the PMMA was removed by treatment of hot acetone (80 °C).

The characterization of WCF, bare and re-solidified Cu, as-synthesized and transferred graphene samples involved evolve gas analyzer-gas chromatography-mass spectrometry (EGA-GC–MS) by Shimadzu GCMS-QP2010, electron back scattered diffraction (EBSD) by JEOL JSM-7001FF field emission SEM equipped with an EBSD detector, atomic force microscopy (AFM) by SPM-5200 scanning probe microscopy, optical microscopy (VHX-500 digital microscope), Raman spectroscopy (NRS 3300 laser Raman spectrometer with a laser excitation wavelength of 532.08 nm), TEM (JEOL JEM 2100, operated at 200 kV), UV–vis spectroscopy (ASCO V–670 K spectrophotometer), and sheet resistance measurement by four probe (T–70 V/RG-7C of Napson Corporation).

3. Results and discussion

In the present work, the growth of uniform monolayer graphene from WCF on Cu substrate by LPCVD is demonstrated. WCF is an abundant waste material that was produced every day in the world and the utilization of this waste material as a carbon precursor for graphene synthesis stand unique. It is sure to have a great impact in recycling process and waste management. To investigate the suitability of WCF in the synthesis of graphene, EGA with GC–MS analysis were done to determine its chemical composition. The chicken oil is examined by EGA-GC–MS to investigate the content of carbon evolve during the thermal decompositions. Fig. 2a shows the EGA of evolve products at different temperatures. EGA spectrum shows the decomposition of carbon source occurred mainly around 400 and 550 °C with retention time of 17.5 and 25.0 min, respectively. Fig. 2b and c show average pyrolysis GC–MS spectra, as complying to further analyze the major product evolve during the decomposition process. At a decomposition temperature of 400 °C, linear carbon chain fragmented with 3, 4 and 5 carbon atoms are observed in abundance, whereas at 550 °C, the accumulated triglycerides which accidentally moved to MS detector was found. Considering the EGA-GC–MS analysis, the WCF was heated around 400 °C in the CVD synthesis process. This will produce abundant of low molecular weight carbon molecules for favorable graphene growth. Previously, Suriani et al. have demonstrated carbon nanotubes (CNTs) synthesis using WCF precursor by heating at 470 °C [25]. In contrast to previous work, we heated the precursor at a lower temperature taking account of the gas chromatography analysis, which enable us to extract smaller carbon chains. The low molecular weight aliphatic carbon can easily dehydrogenated on Cu catalyst substrate, which can be significant to achieve homogeneous uniform monolayer graphene growth.

The metal substrate use for synthesis of graphene also plays important role in monolayer graphene formation [28]. In this experiment, the polycrystalline Cu substrate was used as a catalyst. Fig. 3a–c shows the structural characterization of bare Cu substrate. The surface of the bare Cu substrate appears to be very rough as observed in the optical image (Fig. 3a). AFM analysis in Fig. 3b shows that the bare Cu has a high surface roughness with root mean square (RMS) value of 300 nm. EBSD measurement was performed to investigate the plane of Cu substrate. EBSD result (Fig. 3c) shows that bare Cu substrate consists of different orientations of grain on the surface; mostly covered by Cu (100) and Cu (110) planes. The grain on the surface is associated with the processing steps as well as the difference in the purity of Cu. In this experiment, the Cu substrate was re-solidified at 1100 °C for 60 min under the flow of H_2 (100 sccm) to expand the grain size, remove the native oxide layer and smoothen the surface. The evolution of randomly curve grain boundary is shown in Fig. 3d–f. Optical microscopy image shows that the re-solidified Cu substrate has large grain size (more than 400 μm) with a smooth surface. AFM results in Fig. 3e proved the smoothness of Cu surface as the RMS value is very low (~30 nm). Again, EBSD result shows that the re-solidified Cu substrate consists only Cu (111) plane with minimum amount of Cu (100) plane (Fig. 3f). The EBSD result is supported by XRD pattern which showed the presence of Cu (111) (as shown in inset of Fig. 3f). In LPCVD, previous studies by Robert MJ and Ivan V et al. show that graphene growth is a substrate mediated process, and the shapes of the resulting graphene domains are determined by the symmetry of the underlying facet [28,29]. Cu (111) and Cu (100) play a crucial role in the coalescence of graphene domains and completion of the self-limiting process to produce monolayer

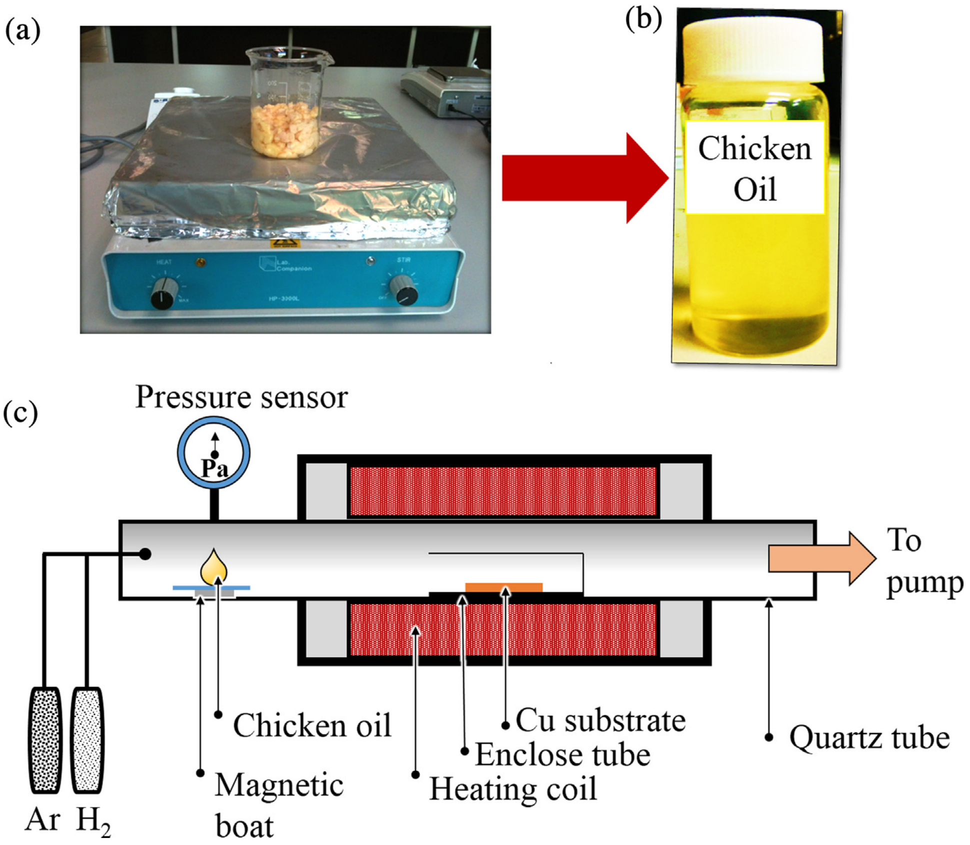

Fig. 1. Photographic image of (a) waste chicken fat and (b) waste chicken oil. (c) Schematic diagram of LPCVD.

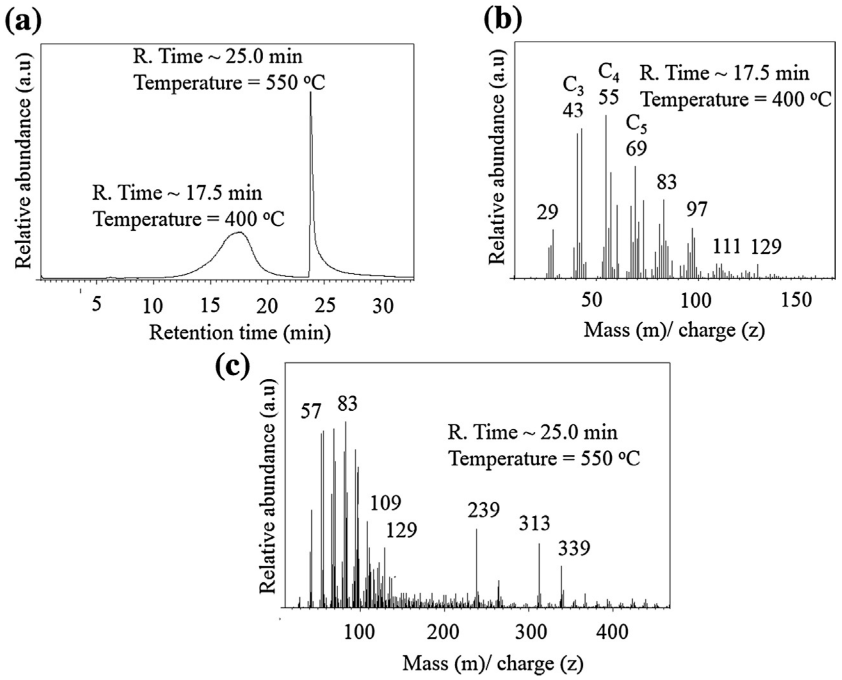

Fig. 2. Evolve gas analyzer-gas chromatography-mass spectrometry analysis. (a) Two main decompositions around 400 and 550 °C with retention time 17.5 and 25 min, respectively. Average mass spectra at two decomposition temperature of 400 and 550 °C show (b) fragmented linear carbon chain and (c) accumulated triglycerides.

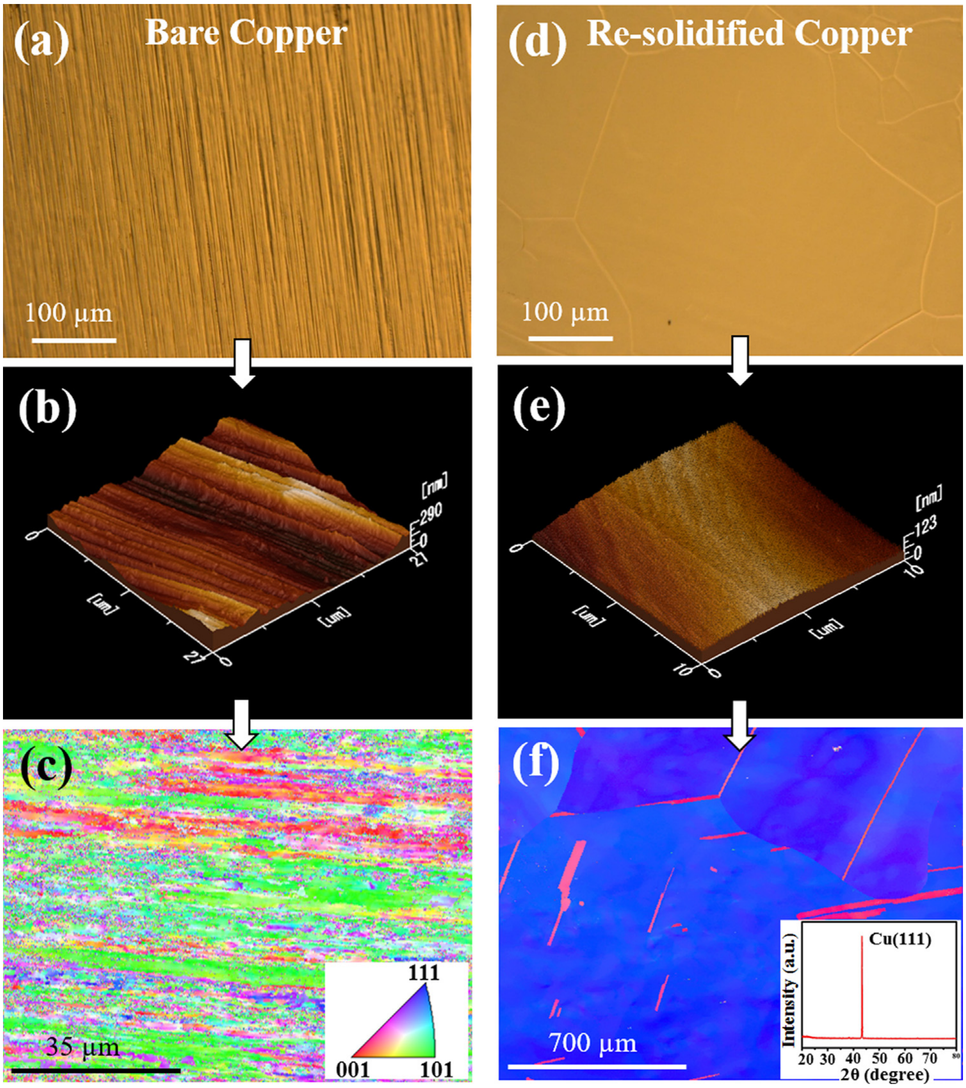

Fig. 3. (a) Optical microscopic image (b) AFM image and (c) EBSD analysis of bare Cu. (d) Optical microscopic image (e) AFM image and (f) EBSD analysis of re-solidified Cu.

graphene, whereas Cu (110) is critical for the nucleation and expansion of secondary graphene layers [30]. Hence, the annealing treatment of Cu substrate to produced Cu (111) in this work favored formation of the high quality monolayer graphene in LPCVD.

To obtain continuous monolayer graphene, the key factor is to control the carbon flux [31]. Zhijuan Z et al. have reported that in the growth condition with low carbon flux, the nucleation and formation of graphene domains are independent of precursor structures [31]. To achieve the low carbon flux environment, a tube with one end closed was used and the re-solidified Cu substrate was placed in it. The usage of enclose tube is also to prevent the unnecessary interaction between the Cu substrate and Cu residue that sublimated and deposited at quartz tube during previous experiment. Furthermore, the decomposition temperature of WCF was kept around 400 °C to attain slow evaporation as very fast evaporation produces inhomogeneous and thicker graphene layers. The low molecular weight carbon molecules from decomposed WCF are catalyzed into active carbon species by Cu and chemically adsorbed on its surface. It is well known that the activation energy for surface diffusion of a carbon atom on Cu is low (~0.06 eV) at high temperature. Thus, carbon atoms is freely diffuse and aggregate on the active sites of the Cu surface to attain a critical supersaturation state and start to nucleate in this supersaturated carbon field [32]. The diffused carbon atoms on the Cu surface are caught by the edge of the nuclei and create a circular shape because of initial “capillarity limitation”, which lessens the interfacial free energy and decays the development instabilities. Consequently, a circular shape gets to be unstable and the growth process was dominated by morphological instabilities. After most of the graphene islands have coalesced to form a large sheet, the absence of diffused active carbon atoms terminates the formation of the secondary multi-layer, resulting in the self-limiting growth of a continuous sheet of monolayer graphene [30,32–34]. As well, the gas mixture ($Ar:H_2$) significantly influence the growth process, where H_2 act as a catalyst to activate carbon for graphene nucleation and growth. H_2 can be active in the competition of CHx adsorption/C-etching on Cu surface, as well as playing an important role in the transition of sp^3 to sp^2 carbon [35]. However, increasing the H_2 content etching effect became more dominant. We observed that using chicken fat oil as precursor a molar ratio of 98:2 for Ar and H_2 was most suitable to achieve uniform monolayer graphene in the CVD process.

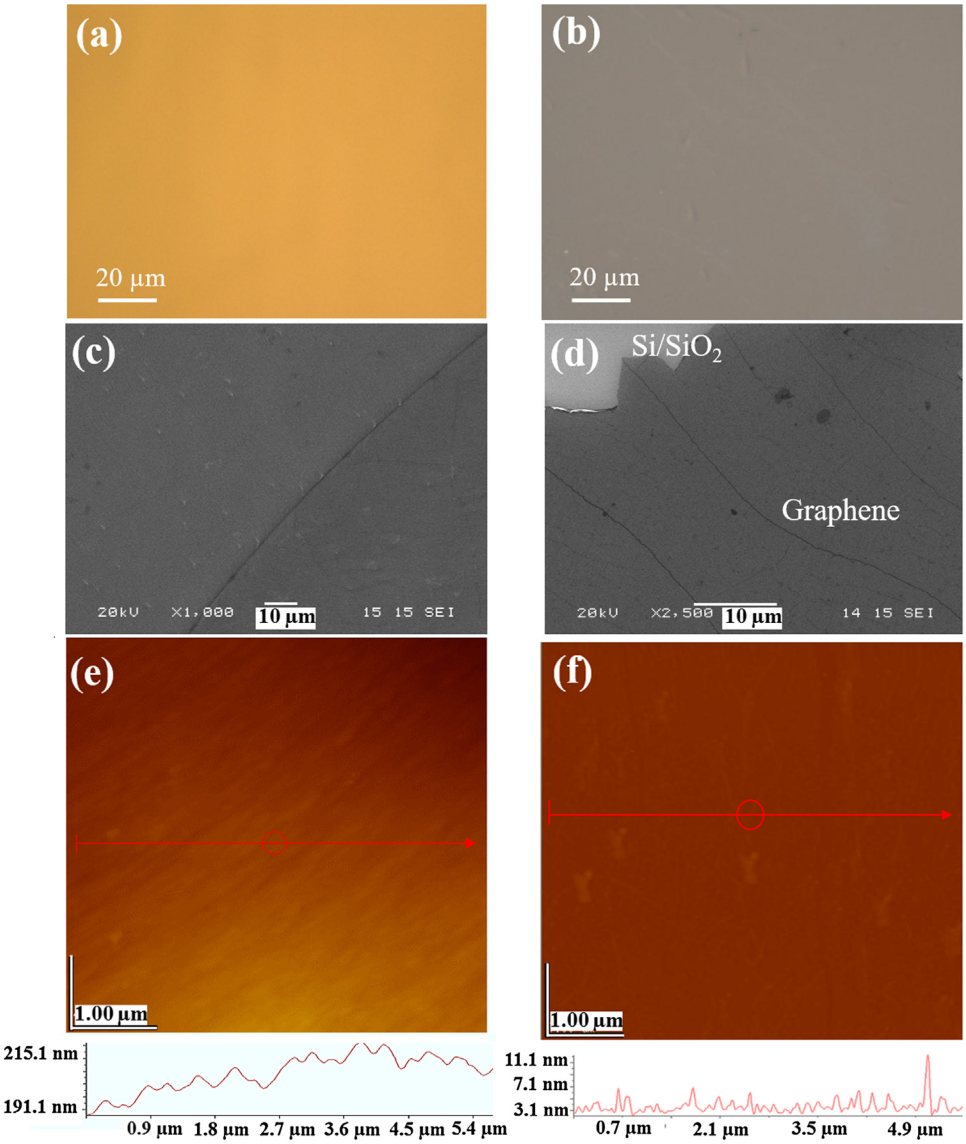

Fig. 4. Optical microscopic images of graphene (a) on Cu substrate and (b) transferred on Si/SiO_2 substrate. SEM images of graphene (c) on Cu substrate and (d) transferred on Si/SiO_2 substrate. AFM studies of graphene (c) on Cu substrate and (d) transferred on Si/SiO_2 substrate.

The structural morphology of the as-synthesized and transferred graphene film was investigated using optical microscopy, SEM and AFM studies. Fig. 4a and b shows the optical microscope images of the as-synthesized and transferred graphene film. The transferred graphene film is continuous with no patches and PMMA residues observed. Further observation on synthesized and transferred graphene was done using SEM analysis. Fig. 4c and d shows the as-synthesized graphene and transferred graphene on Si/SiO_2, respectively. It can be seen that the synthesized graphene was continuous with less wrinkles. The smoothness and less wrinkle properties of the graphene were further investigate using AFM analysis. As can be seen in Fig. 4e, the graphene synthesis on Cu has low RMS value, 29.8 nm, which indicate high smoothness of graphene on Cu substrate. The transferred graphene on Si/SiO_2 also showed high smoothness and less wrinkle formation where the RMS value measured was less than 5 nm (Fig. 4f).

In order to examine the uniformity of the graphene film, a Raman mapping over 90 × 90 μm^2 area was explored. Most of the scanned area has a signature of $I_{2D}/I_G > 3$, $I_D/I_G < 0.1$ and FWHM of ~34 cm^{-1}; which further confirms the uniform coverage of high quality monolayer graphene (Fig. 5a–c). Fig. 5d shows the Raman spectra of the transferred graphene film on the Si/SiO_2 substrate. An intense graphitic G and second order resonance 2D peaks are observed at 1584 and 2695 cm^{-1}, respectively, while the intensity of defect related D peak is relatively low for the synthesized graphene.

TEM images and selected area electron diffraction (SAED) pattern were observed to investigate the crystal structure of a monolayer graphene film derived from WCF. The graphene was transferred to a C-net TEM grid, where most of the area of graphene was determined to be crystalline by its hexagonal diffraction pattern (Fig. 6a) and was continuous as shown in Fig. 6b. A randomly chosen monolayer edge of the graphene is shown in Fig. 6c. The edge of graphene corresponds to monolayer graphene. The graphene film was transferred to a quartz substrate prior to UV–vis analysis. The graphene film exhibit transmittance of ~96%

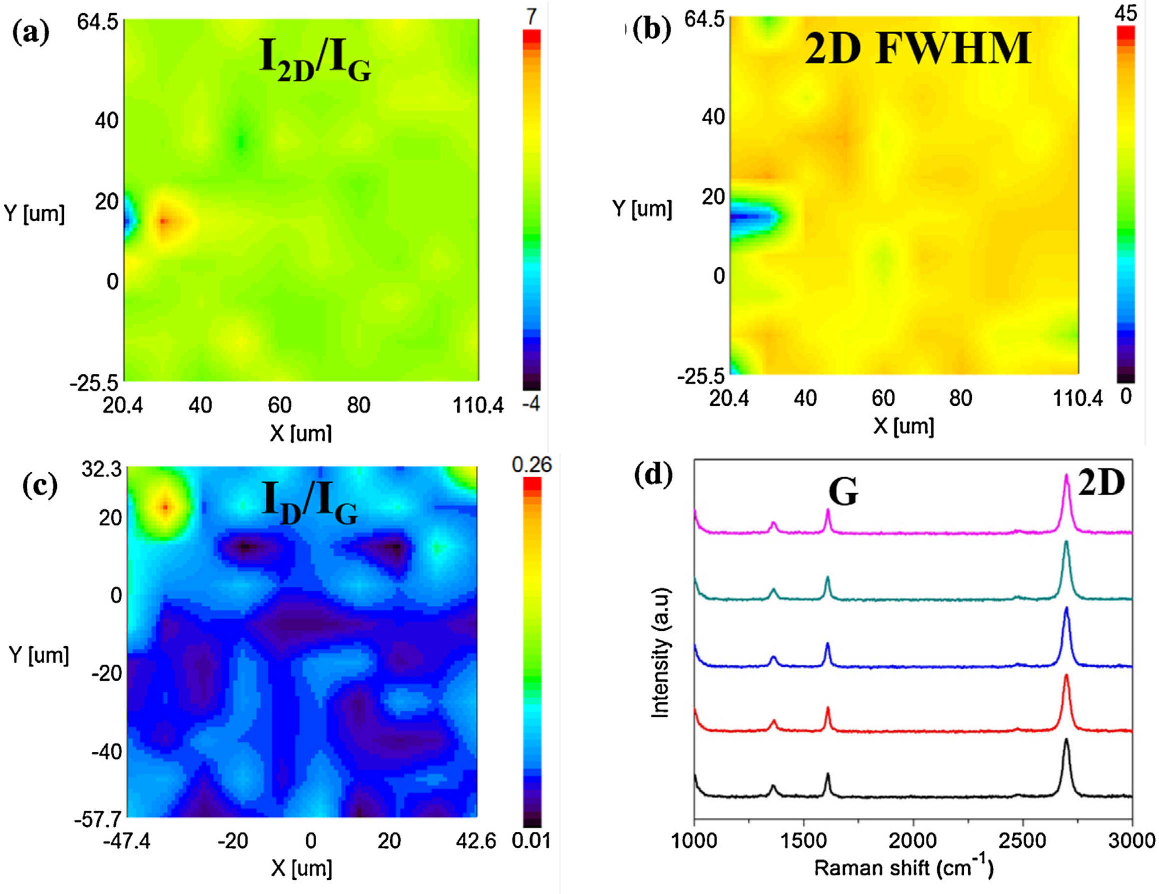

Fig. 5. Raman mapping of graphene transferred on Si/SiO$_2$ substrate (a) I_{2D}/I_G (b) FWHM of 2D and (c) I_D/I_G. (d) Raman spectra of graphene transferred on Si/SiO$_2$ substrate at randomly selected 5 different points.

at 550 nm corresponds to the monolayer nature of graphene as shown in Fig. 6d. In the photograph image (Fig. 6e), the graphene film on quartz slide is optically uniform and transparent. Also, the sheet resistance (Rs) of the graphene film was found to be in the range of ~300 Ω/square measured by the four probe method. This finding shows that the highly conducting graphene film can be synthesized from WCF. This value is comparable with the sheet resistance of monolayer graphene reported by previous research groups. Bae S. et al. have reported the lowest sheet resistance of a monolayer graphene as 125 Ω/square, while Sun Z. et al. and Ruan G. et al. have achieved sheet resistance in the range of 1.2–3.0 kΩ/square [3,15,21].

The growth of monolayer graphene formation on Cu (111) is in line with the result reported by K. T. Park et al. which stated that Cu (111) promotes rate monolayer graphene growth due to smaller lattice mismatch with the graphene domain, resulting in faster adsorption of the carbon species [36]. This finding confirmed that the atomic arrangement of the underlying Cu substrate is responsible for self-limiting growth of monolayer graphene in LPCVD conditions. Besides, the similarity between the graphene produced in LPCVD using WCF, ethanol and methane prove that in the growth condition where carbon flux is low, self-limiting growth of graphene on Cu surface is independent of the precursor structure [37,38]. It should be noted that the facet dependent growth behavior in LPCVD is as opposed to that reported for APCVD process, where high-index Cu facets do not behave as barriers for the continuity of a graphene sheet [39]. It is due to the high pressure of precursor in APCVD which generates more active carbon atoms and reduces the effective diffusion length. Hence, it accelerates the nucleation at the Cu steps of high-index facets, which results in more graphene nuclei to expand into a continuous graphene sheet [38]. In the APCVD process, a control of high flow rate and partial pressure of H$_2$ and carbon precursor are essential as the growth in APCVD is not self-limiting. Thus, the extension of growth time and carbon precursor will result in an increase in the graphene layer number. This is due to the high levels of carbon supersaturation on the Cu surface as a result of excess carbon supply [37].

4. Conclusions

We have demonstrated for the first time, the synthesis of high quality monolayer graphene using WCF as a carbon precursor in LPCVD technique. EGA-GC–MS analysis showed that WCF is decomposed into aliphatic carbon chains with low molecular weight. The re-solidification of Cu substrate has successfully achieved by high temperature annealing, which produced smooth surface having big grains of Cu (111) facet. Uniform high quality monolayer graphene was obtained on re-solidified Cu as confirmed by optical microscopy, TEM and Raman spectroscopy analysis. Less wrinkles formation in the synthesized monolayer graphene was achieved as confirmed by SEM and AFM analysis, enabling the

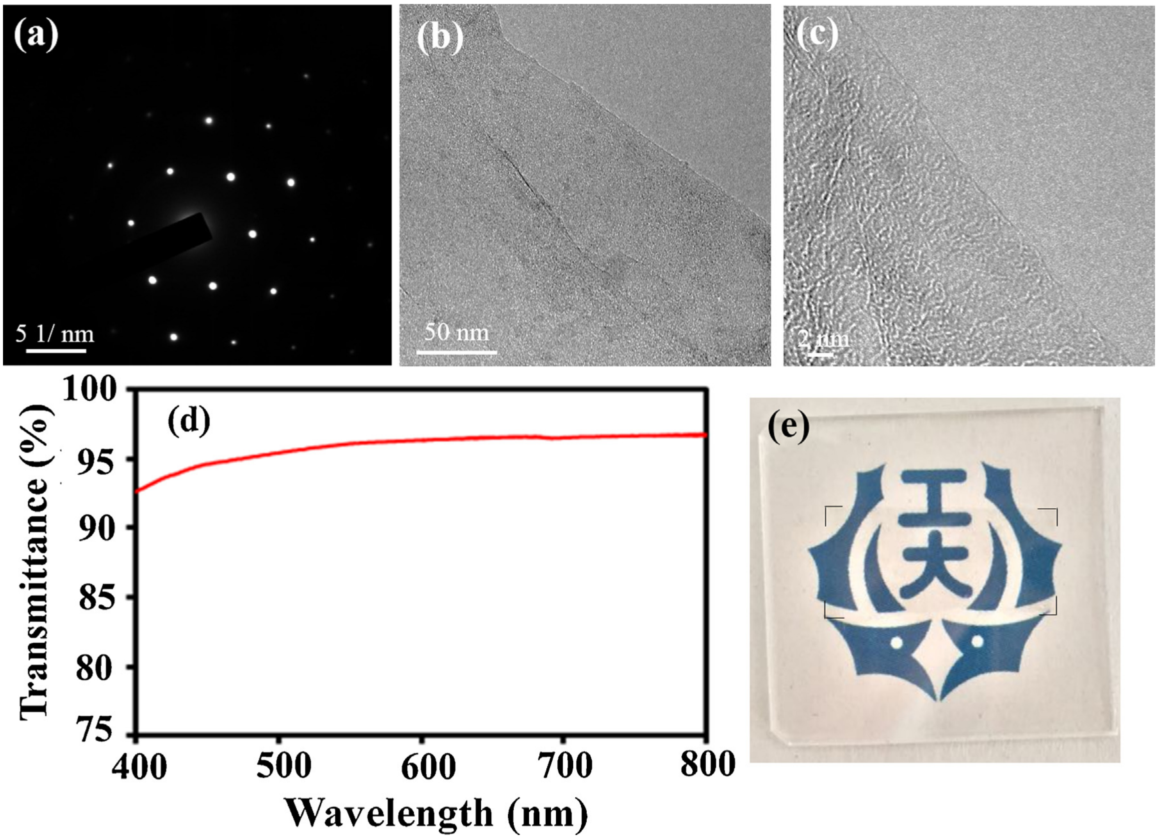

Fig. 6. Diffraction pattern and TEM images of the graphene. (a) SAED pattern, (b) low magnification image of a graphene sheet, and (c) the edge of monolayer graphene. (d) UV–vis spectra of graphene transferred on quartz slide. (e) Photographic image of monolayer graphene film of 1 × 2 cm in size on a 1 mm thick quartz slide.

fabrication of transparent electrode with low sheet resistance. Thus, our findings show that uniform monolayer graphene can be synthesized on re-solidified Cu substrate using WCF as carbon precursor.

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