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Arc spectra of different solid reduced graphene oxide samples under microwave irradiation

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ABSTRACT

It is often accompanied by an intense arc when solid-state graphene samples are irradiated by microwaves (MWs); however, arc spectra have not been studied. In this study, the arc spectra of reduced graphene oxide (RGO) powder, three-dimensional (3D) porous RGO, and RGO paper under MW irradiation were measured; these spectra exhibited diverse features. For the same reaction conditions and the same initial sample, the arc spectra should be the same if the reaction process and mechanisms are the same. Further analyses showed that the obtained RGO paper with an evident 2D band, a smaller D band, lower sheet resistance, and lower oxygen content was of higher quality than the RGO powder and 3D porous RGO obtained after MW irradiation. Based on these results, the diverse spectra may suggest different reaction processes and mechanisms. Overall, these findings provide novel insights into the preparation of graphene via MW reduction.

1. Introduction

Due to its convenient and fast heat source, microwave (MW) irradiation heats materials rapidly and efficiently. Carbon materials such as carbon nanotubes, activated carbon, carbon black, and graphene-based materials are highly effective MW absorbents and can be easily heated by MW irradiation [1–4]. This property allows these materials to be transformed by MW heating, giving rise to new carbons with tailored properties for use as MW receptors for indirect heating of other materials, or to act as a catalyst and MW receptor in different heterogeneous reactions [2]. To date, MW has been used to prepare graphene by reducing graphene oxide (GO), accelerating the reaction process, and improving the quality of reduced graphene oxide (RGO) [5–8]. Research on the MW heating of graphene-based materials can be classified into two categories. The first category is liquid phase exfoliation and preparation of graphene-based materials [9–21]. For example, Chen et al. prepared graphene by the rapid and mild thermal reduction of graphene oxide induced by microwaves [9], while Lin et al. carried out microwave-assisted rapid exfoliation of graphite into graphene using ammonium bicarbonate as the intercalation agent [10]. The second category is the preparation and improvement of the quality of solid graphene-based materials [3,5–7,22–30]. For instance, Park et al. prepared high-quality graphene nanosheets via solid-state microwave irradiation synthesis under a hydrogen-containing atmosphere [3], and Voiry et al. synthesized high-quality intrinsic graphene using microwave irradiation [28], which was also mimicked by Prezhdo et al. using reactive molecular dynamics [31].

Previous reports [1,2,32] showed that MW irradiation of carbon materials is often accompanied by an intense arc, heat release, and outgassing, which also occur during the MW radiation of graphene-based solid-state materials (for example GO and RGO) [3,5–7,28,29]. For example, Zhu et al. reported sparking during the MW preparation of exfoliated graphite oxide (MEGO) [5]. Park et al. observed a white light during the preparation of graphene by introducing small amounts of graphene nanosheets (GNS) in GO [3]. Voiry et al. synthesized high-quality intrinsic graphene using MW irradiation and found that the synthesis was accompanied by violent arcs [28]. An intense arc was also observed in our previous study, and the arc spectrum also was recorded [29]. However, arc spectra have not been studied. Moreover, most studies attributed the preparation of graphene by MW reduction to the thermal effect of MW radiation only [3,5]. Other studies have demonstrated not only the thermal effect of MW radiation, but also the rearrangement of the carbon atoms during the process of graphene preparation by MW reduction [7,28,29].

In this work, the arc spectra of RGO powder, 3D porous RGO, RGO paper were recorded and studied under exposure to MW irradiation

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under atmospheric conditions. It was found that RGO powder, 3D porous RGO, and RGO paper show diverse spectral features under MW irradiation, which may signify that different reaction processes occurred in these samples. Further research also suggested that graphene obtained under MW irradiation from RGO paper was of higher quality as indicated by the Raman spectra with an obvious 2D band and smaller intensity ratio of the D band to the G band (I_D/I_G) than those obtained from powder and 3D porous RGO with a broad and strong D band and vanishing 2D band, as was also identified by X-ray photoelectron spectroscopy (XPS) and sheet resistance measurements. These findings are helpful for obtaining a better understanding of the preparation of graphene by MW reduction.

2. Materials and methods

2.1. Materials

GO powder (Model No., GO1211) and 3D porous GO were purchased from Angxing New Carbon Material Changzhou Co., Ltd. (China). Filter paper with a diameter of 9 cm was purchased from Hangzhou special paper Co., Ltd., (China) and ultrapure water was obtained using an ultrapure water machine (ULUPURE).

2.2. Preparation of RGO powder, 3D porous RGO and RGO paper

In this work, GO paper was prepared according to the method described in reference [29]. Specifically, a 5 mg/mL GO solution and a 0.8% calcium chloride ($CaCl_2$) solution were used to prepare the GO paper. Subsequently, GO powder, 3D porous GO and GO paper were annealed in an oven (DHG-9140A) at 185 °C and kept at 185 °C for 6 h; RGO powder, 3D porous RGO, and RGO paper were obtained. It should be noted that GO paper prepared using this method with an initial heat treatment of more than 170 °C can only react rapidly under MW irradiation.

2.3. Characterization

The Raman spectrum was obtained using a high-resolution laser confocal Raman spectrometer (LABRAM HR EVO). The arc spectra were collected using an Ocean Optics spectrometer (QE65 Pro, Ocean Optics). X-ray photoelectron spectroscopy spectra were recorded using a Thermofisher K-alpha spectrometer (ESCALAB250Xi). The sheet resistance measurements were carried out using a four-probe method using a four-probe resistivity tester (HPS2661).

3. Results and discussion

Fig. 1(A) shows a schematic representation of the experimental procedure. First, RGO powder, 3D porous RGO and RGO paper were respectively placed in a glass vial and then placed into a household MW oven (Media, M3-L253C). Second, the detector of the optical fibre spectrometer was fixed in front of the MW oven panel to collect and record the spectra. After MW irradiation for 1–2 s, accompanied by heat release and outgassing, RGO samples were further reduced, and an arc was observed. Finally, the process of MW irradiation was continued for ~3 s in order to collect and record the arc spectra. The entire experiment was completed in 5 s.

Fig. 1(B) shows the arc spectra of RGO powder, 3D porous RGO and RGO paper under MW irradiation that displayed diverse spectral feature. Fig. S1 showed the digital images of RGO powder, 3D porous RGO and RGO paper used in our experiments. Unlike the arc spectra of RGO powder and 3D porous RGO, RGO paper showed an excellent polychromic source feature (emitted main wavelengths at 421.95, 553.10, 588.69, 765.82 (768.84) nm and a wide spectrum between 588.69 nm and 643.32 nm) when exposed to MW irradiation. Furthermore, the arc spectra of the GO powder, 3D porous GO and GO paper were obtained through triggered MW reduction [29] and are shown in Fig. S2. It is observed that these spectra were consistent with the results shown in Fig. 1(B). This result suggested that the thermal treatment prior to MW irradiation had almost no effect on the arc spectra. Considering the same reaction conditions and the same initial sample (GO), the arc spectra should be the same if the reaction process and mechanisms are the same. Hence, we believe that the diverse spectral features may indicate different reaction process and mechanisms. Moreover, according to Fig. 1(B), when exposed to MW irradiation, RGO powder and 3D porous RGO had almost the same spectral feature with the excitation wavelengths of 588.69, 765.82 (768.84) nm, as was also identified by the results presented Fig. S2. Therefore, we deduced that the reaction process and mechanisms of RGO powder and the 3D porous RGO were the same. Fig. S3 showed the spectrum of the 3D porous RGO prepared by hydrothermal reduction under MW irradiation for which the excitation wavelengths were also found at 588.69 nm and 765.82 (768.84) nm, and the change in the intensity was related to the preparation of hydrothermal reduction of 3D porous RGO.

Considering the use of calcium chloride in the preparation of RGO paper, further experiments were conducted to confirm whether the arc spectra were caused by residual calcium chloride; the results are shown in Fig. 2. According to the results in Fig. 2, the arc spectra of the RGO paper without the treatment of calcium chloride also showed similar polychromic source features, indicating that the spectra of the RGO paper may be not caused by residual calcium chloride. However, the results presented in Fig. S5 also indicated that the treatment of calcium chloride may inhibit the excitation spectra of 588 nm and 765.82 (768.84) nm, particularly at 765.82 (768.84) nm, and may amplify the excitation spectra of other wavelengths.

The Raman spectrum of RGO powder, 3D porous RGO, and RGO paper before and after MW irradiation were measured, as shown in Fig. 3. Compared with the results of Fig. 3, the Raman spectrum of obtained RGO paper showed smaller D band and obvious 2D band, which were different from those of obtained RGO powder and 3D porous RGO with a broad and strong D band and vanishing 2D band after MW irradiation. The smaller D band (I_D/I_G = 0.16) implied a lower defect level after removing the partly oxygen functional groups. And the obvious 2D band indicated MW irradiation could restore the structural integrity of graphene by reordering of the basal plane [21,28]. Table 1 showed the results of the intensity ratio of 2D band to G band (I_{2D}/I_G). According to Table 1, obtained RGO paper showed the larger I_{2D}/I_G (0.654 ± 0.054) than RGO powder (0.132 ± 0.025) and 3D porous RGO (0.275 ± 0.046), and the results indicated that the graphene structures destroyed by oxidation process in RGO paper were better restored when exposed to MW irradiation.

Further experiments were conducted to assess the quality of graphene samples obtained after MW irradiation. The domain sizes of the graphene samples obtained after MW irradiation were calculated according to Ref. [28], and the results are shown in Fig. 4. According to the results presented in Fig. 4 and Table 1, RGO paper obtained after MW irradiation exhibited larger graphene domain size (106.49 ± 29.54) than RGO powder (20.02 ± 1.30) and 3D porous RGO (24.03 ± 4.71), indicating its higher quality.

We also measured the change in the sheet resistance of RGO powder, 3D porous RGO and RGO paper before and after MW irradiation, as shown in Table 2. An examination of the results showed that the obtained RGO paper, RGO powder and 3D porous RGO have lower sheet resistance after MW irradiation than before MW irradiation, indicating their better electrical properties. Furthermore, the results presented in Table 2 also indicated that the electrical properties of the obtained RGO paper with the sheet resistance of 16.87 Ω/□ were superior to those of the RGO powder (1.47 kΩ/□) and 3D porous RGO (56.31 Ω/□) obtained after MW irradiation. The results shown in Fig. S6 also showed the obvious improvement in the electrical properties of RGO paper after MW irradiation.

XPS was further used to determine the quality of the 3D porous RGO

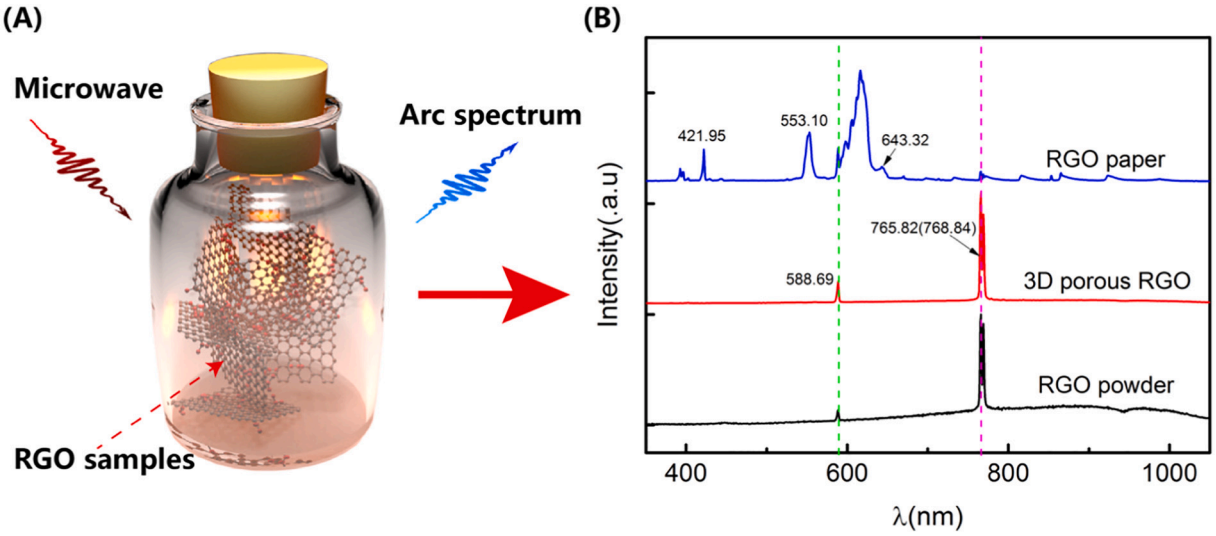

Fig. 1. (A) Schematic diagram of different MW-irradiated RGO samples; (B) Arc spectra of RGO powder, 3D porous RGO and RGO paper under MW irradiation.

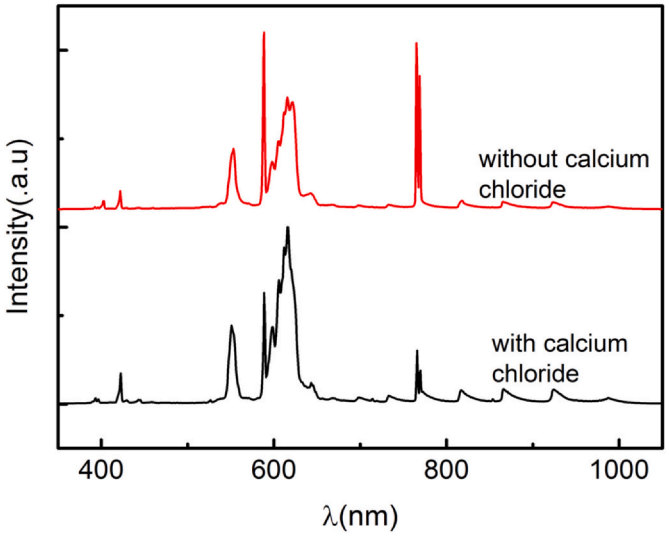

Fig. 2. Spectra of RGO paper with/without the treatment of calcium chloride.

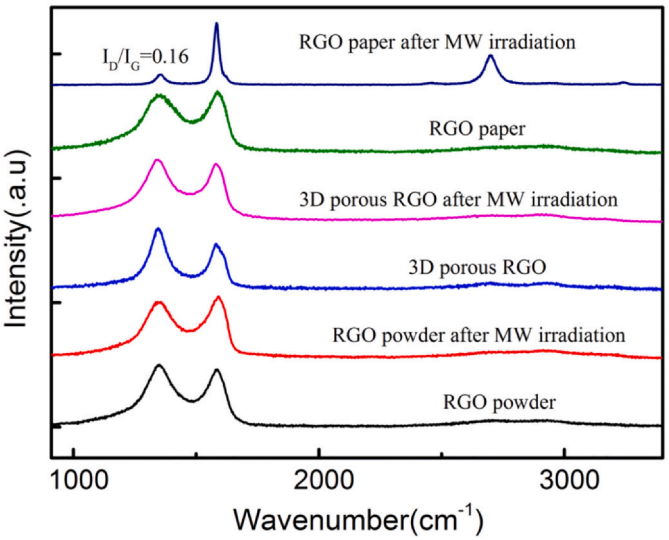

Fig. 3. Raman spectra of different RGO samples before/after MW irradiation.

Table 1
The intensity ratio of 2D band to G band (I_{2D}/I_G).

	RGO powder	3D porous RGO	RGO paper
I_{2D}/I_G	0.132 ± 0.025	0.275 ± 0.046	0.654 ± 0.054

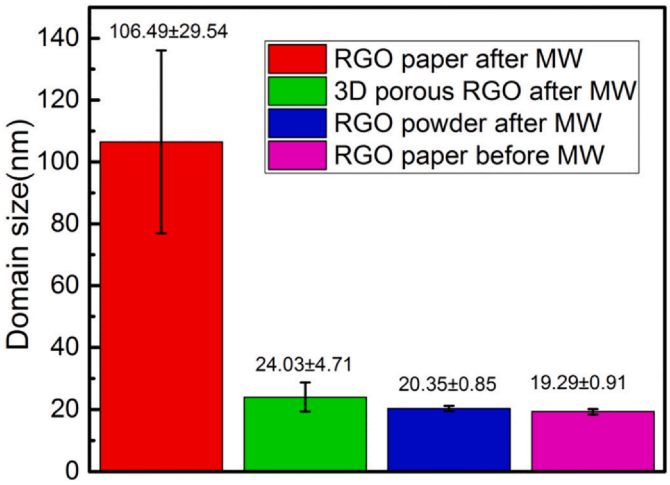

Fig. 4. Domain size of different RGO samples obtained after MW irradiation.

Table 2
The change of sheet resistance before/after MW irradiation.

	RGO powder	3D porous RGO	RGO paper
Before MW	45.46 kΩ/□	247.7 Ω/□	2.11 kΩ/□
After MW	1.47 kΩ/□	56.31 Ω/□	16.87 Ω/□

and RGO paper obtained before and after MW irradiation, as shown in Fig. 5. Fig. 5(A) showed the survey scan of 3D porous RGO and RGO paper before and after MW irradiation. The obtained results showed that small amounts of residual chlorine (0.15%) and calcium (0.65%) were present in the RGO paper. Moreover, the results presented in Fig. 5(A) also showed that RGO samples obtained after MW irradiation were of higher quality, as indicated by the comparison to the XPS results of the RGO samples before MW irradiation. The changes in the C/O ratio also indicated that the obtained RGO paper with a C/O ratio of

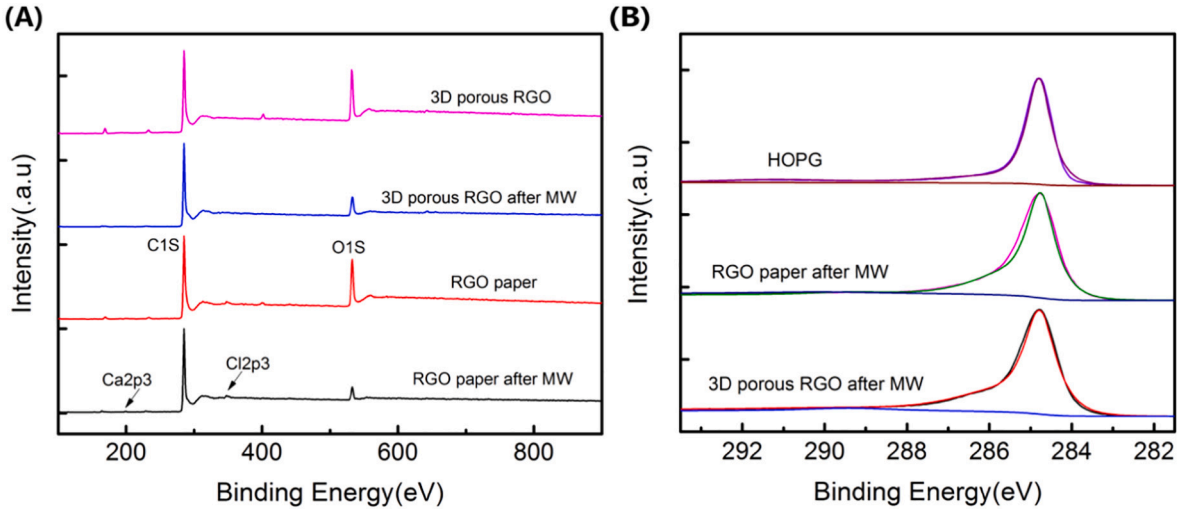

Fig. 5. (A) XPS results of 3D porous RGO and RGO paper before/after MW irradiation; (B) the C1s XPS results of 3D porous RGO, RGO paper obtained after MW irradiation and HOPG.

12.9 was of higher quality than 3D porous RGO with a C/O ratio of 7.9. Fig. 5(B) shows the C1s XPS results of 3D porous RGO, RGO paper obtained after MW irradiation and HOPG. Compared to the C1s XPS results for GO presented in Fig. S7, RGO paper and 3D porous RGO obtained after MW irradiation revealed an effective reduction. Moreover, the results in Fig. 5B also showed that the C1s XPS spectrum for the RGO paper obtained after MW irradiation was closer to that of HOPG than that of 3D porous RGO. Moreover, the X-ray diffraction (XRD) results presented in Fig. S8 also revealed an effective reduction after MW irradiation.

Fig. 6 shows the arc spectra of metal (iron wire and aluminium foil) under MW irradiation for the excitation wavelengths of 588.69 nm and 765.82 (768.84) nm. It is observed that the spectra were almost the same as those of the RGO powder and 3D porous RGO. These results indicated that RGO powder and 3D RGO exhibit metallic properties when exposed to MW irradiation. Hence, we believe that the reaction process of 3D porous RGO is the same as that of the iron wire and aluminium foil exposed to MW irradiation. It is well-known that the exposure of a metal to MW irradiation is often accompanied by an intense arc. This is because the free electrons on the metal surface gain enough energy under MW irradiation to be excited out of the surface, ionizing the air and causing an intense arc due to the thermal effect of MW irradiation [33,34]. Therefore, the reaction process and mechanism of the arcs observed for RGO powder and 3D porous RGO can be attributed to the thermal effect of MW irradiation.

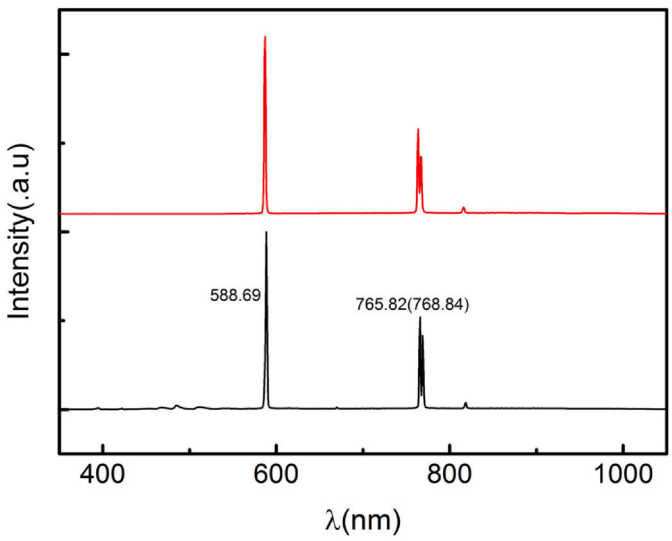

Fig. 6. Arc spectra of iron (Fe) wire and aluminium foil under MW irradiation.

Because RGO paper obtained under the same conditions was of higher quality than the obtained RGO powder and 3D porous RGO, combining the diverse spectral features shown in Fig. 1, we speculated that the reaction process of RGO paper under MW irradiation may be different from that of RGO powder and 3D porous RGO. Fig. S9 showed the photoluminescence spectra of RGO paper, and the results suggested that the arc spectrum of RGO paper under MW irradiation was not the result of photoluminescence. Our previous study showed that a rearrangement of the carbon atoms may occur in the process of triggered MW reduction [29], as has also been reported in other studies [7,28]. Therefore, we believe that except for the bands at 588.69 nm and 765.82 (768.84) nm, the arc spectra of RGO paper under MW irradiation may be the results of the carbon atoms rearrangement, and the reaction process of RGO paper under MW irradiation is the result of the thermal effect of MW irradiation and carbon atom rearrangement.

4. Conclusions

In summary, the arc spectra of RGO powder, 3D porous RGO and RGO paper were studied under MW irradiation, and the results showed diverse spectral features. Under MW irradiation, RGO powder and 3D porous RGO exhibited almost the same spectrum as an iron wire and aluminium foil, which was caused by air ionization and was attributed to the thermal effects of MW irradiation. However, the spectra of RGO paper showed an excellent polychromic source behaviour that may be the result of the thermal effect of MW irradiation and carbon atom rearrangement. Furthermore, the Raman spectra, XPS and sheet resistance results showed that the obtained RGO paper was of higher quality than RGO powder and 3D porous RGO after MW irradiation. This study is helpful for better understanding of the preparation of graphene by MW reduction.

CRediT authorship contribution statement

Jiang Wenshuai: Conceptualization, Methodology, Writing-Original draft preparation, Writing-Reviewing, Funding acquisition; **Li Zhenxin**: Data curation, Design of methodology; **Zhu Yongtao**: Sample preparation; **Xin wei**: Data curation; **Yu Yi**: Writing-Original draft preparation, Writing-Reviewing, Funding acquisition.

All authors read and contributed to the manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.diamond.2020.108060.

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