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Thermal analysis of carbon fibre reinforced polymer decomposition

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Abstract

The increasing number of carbon fibers reinforced polymer (CFRP) waste disposed of in landfills is creating environmental concerns due to the potential release of toxic by-products and the need for recycling. This research work investigates the influence of atmosphere (single and combination of nitrogen and oxygen) and heating rate (5 and 10 °C min⁻¹) on the thermal decomposition of CFRP to recover the reclaimed-cf. The samples were heated up to 420 °C in a nitrogen atmosphere followed by heating in the oxygen atmosphere from 420 °C until the final heating temperature at different heating rates. The thermal decomposition behavior of the CFRP waste was compared by thermogravimetric analysis (TGA). Morphological, chemical and structural analysis of reclaimed-CF was performed using SEM, FT-IR and Raman spectroscopy respectively. A nitrogen atmosphere was significant at the early temperature (<420 °C) to decompose smaller molecules of epoxy resin components, while oxygen atmosphere is needed to achieve a complete separation of reclaimed-CF from their matrix. Thermal decomposition at lower heating rate (5 °C min⁻¹) was found efficiently to eliminate the complex epoxy resin and retain the structure of reclaimed-cf. The particular thermal decomposition technique that leads to a lower final heating temperature (340 °C) is present to recover valuable reclaimed-CF from complex CFRP industrial waste.

1. Introduction

Carbon fiber reinforced polymer (CFRP) composites have been attractive materials for many civil and mechanical applications especially aero composites industry to minimize the weight of components and maximize mechanical resistance. However, CFRP composites have been mostly disposed of in landfills after their end of service life, creating environmental concerns and potential release of toxic by-products. The CFRP type of waste, including out-of-date prepregs, manufacturing cut-offs, production tools and end-of-life (EoL) components (Pimenta and Pinho 2011, Oliveux et al 2015). A large amount of CFRP waste is expected as Sheng Yin et al (2013) reported by the year 2030, 6000 to 8000 commercial planes are probable to reach their end-of-life. At the moment, the possibility of recovering and reusing the reclaimed-CF as reinforced materials in composites are receiving widespread attention due to the direct applications in several industrial fields such as automotive.

Hence, a significant interest in recycling CFRP research has appeared in the last decade for obtaining reclaimed-CF in the desire to keep CF waste out of our landfills and to fill a potential gap between CF supply and demand. Wet chemical degradation and thermal treatments were among the popular methods that showed the ability to produce reclaimed-CF with properties near those of virgin CF at a lower cost. For example, ELG Carbon Fibre, which runs a 1500-metric tonne capacity plant in the UK express cost, the security of supply and environmental sustainability as three main drivers for the use of reclaimed-cf ELG’s reclaimed-CF are said to retain at least 90% mechanical properties to the virgin CF with 40% less price. Therefore, similar weight-saving...
benefits at substantially reduced part cost, making reclaimed-CF attractive for automotive light-weighting applications.

In order to design suitable and efficient recycling methodology, the CFRP complex formulation which generally constitutes an epoxy resin, reinforcement and thermoplastic polymer must be understood well. It was demonstrated that the thermal decomposition of this complex composite is different from one system to another as it depends on the chemical nature of their components (Sergeri and Edward 2004, Afaghi Khatibi et al 2016, Tranchard et al 2017). (Yavuz et al 2014) reported that the prepreg compositions are (1) phosphorus compound (2) an epoxy resin which has at least three epoxy groups and (3) epoxy resin curing agent. Thermolysis has been attracted to recycling methodology due to the lower-cost process incurred, particularly when dealing with highly valuable products such as CF. Earlier, the thermolysis process is expected to show significant improvement in recovering the properties of CF based on the research by Pickering (2004). The reclaimed-CF was reported to show a lower strength degradation of typically 20% with preservation of the original stiffness (Pickering 2004). Fabrication of reclaimed-CF via thermolysis has been studied well (Cunliffe and Williams 2003, Oliveux et al 2015). However, the disadvantageous mainly due to degradation of the reclaimed-CF surfaces when they are contaminated by char produced during the thermal decomposition process. This decomposition requires a post-treatment in a furnace at a higher temperature ( >450 °C) to burn it which also lead to higher operating cost. Although the investigation on the recycling of CFRP waste has been few explored in the various research field, research on the mechanism decomposition is still in its early stages. Thus, further investigation of the efficient and suitable heating profile is essential to recover the valuable reclaimed-CF.

Therefore, this work was focused on the thermal decomposition approached for the recycling process of CFRP waste. The main challenge lies in the process of thermolysis which involves a narrow window of exchanging the atmosphere from nitrogen to oxygen, and heating rate. The decomposition stages to recover the CF will be analyzed via morphological, chemical and structural analysis to further examine the condition of reclaimed-CF during the elimination of complex resin matrix from CFRP waste.

2. Methods

2.1. Materials
A roll of carbon fibre reinforced polymer (CFRP) prepreg material waste with unidirectional orientation supplied by industrial waste (CTRM) was used for this research. The plain prepreg made of epoxy resin reinforced with carbon fibers (59%-60% carbon fibers [by volume] and 40% complex epoxy resin).

2.2. Thermogravimetric analysis (TGA)
TGA was carried out on TA instruments TGA 1 brand Mettler Toledo. The balance purge flow was set to 15 ml min⁻¹ and the sample purge flow (nitrogen and oxygen) to 50 ml min⁻¹. Thin square samples were prepared in an alumina crucible and another empty alumina crucible was used as a reference underwent a heating profile from 250 °C to 420 °C in nitrogen with a heating rate of 5 and 10 °C min⁻¹, followed by holding temperature at 420 °C in nitrogen for 30 min and heating from 420 to 800 °C in oxygen with heating rate of 5 and 2 °C min⁻¹.

2.3. Morphological, chemical and structural analysis
Morphology behavior of CFRP and reclaimed-CF were analyzed using scanning electron microscopy (SEM). Elimination of CFRP waste surface functional group was examined using FT-IR spectroscopy (JASCO FT-IR, 6100), recorded in a range of 400 to 4000 cm⁻¹ with a resolution factor of 4 cm⁻¹ via ATR method. Structural Analysis of CFRP and reclaimed-CF were examined via Raman spectroscopy with UniRAM-3500, Micro Raman Mapping, occupied with laser source 532 nm & 785 nm.

3. Results

3.1. Effect of atmosphere in thermal decomposition
Figure 1 shows the effect of different purge gases in the thermal decomposition of CFRP using TGA. In order to examine properly the thermal decomposition of samples, pure gases like nitrogen and oxygen are used. Both thermal decomposition atmosphere results in weight loss during heat-up until 1000 °C. However, the delegation of the decomposition reaction is different for both atmospheres. In an inert nitrogen atmosphere, a single main decomposition step was observed at ~240 to 500 °C as highlighted in the first area (figure 1(A)). Further heating results in the almost constant of CFRP decomposition behavior, with only small changes in their weight loss %, until the final residual weight at 1000 °C is 72.7%. While, in the oxygen atmosphere, apparently, two main decomposition steps were observed (figures 1(a) and (b)), where the first decomposition at ~240 to 500 °C
followed by the second decomposition at 700 to 750 °C. The final residual weight for an oxygen atmosphere is lower than the nitrogen atmosphere at 68.5%.

3.2. Effect of heating rate on thermal decomposition

Figure 2 illustrates the effect of heating rate (5 and 10 °C min⁻¹) on the thermal decomposition behavior of CFRP waste. The thermal decomposition of both samples began slowly until about 300 °C and continue with the same pattern until 500 °C. However, at a higher temperature (>500 °C), different trend reveals when the TG curve for CFRP of 10 °C min⁻¹ shifted to higher temperatures by about 50 °C as compared to 5 °C min⁻¹. The decomposition of samples at 5 °C min⁻¹ and 10 °C min⁻¹ was completed at 620.58 °C and 671.62 °C, respectively and end with the same final residual mass of 0.5 wt%.

3.3. Morphology Analysis of Decomposed Sample

Figure 3 shows the surface analysis of the reclaimed-CF as compared to CFRP after TGA at different heating rates. Prior to thermal decomposition control shows a smooth flat surface even at higher magnification of SEM (figures 3(a)(i)–(iii)). Both the thermal decomposition process at higher (10 °C min⁻¹) and lower (5 °C min⁻¹) heating rate generally resulted in reclaimed-CF that are completely separated from their epoxy resin (figures 3(b) and (c)). However, further examination at high magnification reveals different surface properties of reclaimed-CF. Higher heating rate produced reclaimed-CF enclosed with many fractured residues (figure 3(b) (ii)) compared to a lower heating rate that produced reclaimed-CF with clean surfaces (figure 3(c) (ii)). Further examination at the highest magnification of SEM shows that higher heating rate degraded reclaimed-CF surface with contaminant residues still scattered along their surfaces (figure 3(b) (iii)). Whereas rough and clean surfaces are observed for reclaimed-CF at a lower heating rate (figure 3(c) (iii)).

Figure 4 shows the cross-sectional analysis of control and reclaimed-CF after thermal decomposition at different heating rates. Commercial CF sizes are mainly in range of 6 to 10 um diameter (Huang 2009).
Figure 4(a) shows control with the diameter of CF in the range of 6 to 8 μm covered with the complex resin matrix. The separated CF after the epoxy resin elimination through thermal decomposition at 10 °C/min (figure 4(b)) shown constancy in size with control, but with degraded end-surfaces and non-unified spherical shape of CF. Reclaimed-CF at a slow heating rate (5 °C min⁻¹) in figure 4(c) were observed to have a more unified spherical shape with smooth end-surface and smaller in size compared to control due to successful elimination of sizing.

### 3.4. Mechanism of CFRP thermal decomposition

The mechanism of CFRP thermal decomposition is illustrated in figure 5. Figure 5(a) represents CFRP decomposition stages demonstrated by SEM micrograph, coupled with TGA (figure 5(b)) during thermal decomposition in an inert atmosphere of nitrogen at the early stage (250 to 420 °C) followed by oxygen atmosphere at high temperature (420 to 540 °C). CFRP is designed to be covered with a complex resin matrix as shown in the schematic diagram (figure 5(C)). The final heating temperature of 540 °C was found effectively produced reclaimed-CF (figure 5(d)) with completely eliminated complex epoxy resin matrix of CFRP waste. Apparently, there are 3 stages of decomposition when the combined atmosphere of nitrogen followed by oxygen is applied during thermal decomposition of CFRP.

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**Figure 3.** Surface analysis of reclaimed-CF as compared to (a) CFRP (control) after thermal decomposition at (b) 10 °C min⁻¹ and (c) 5 °C min⁻¹ heating rate.

**Figure 4.** Cross sectional analysis of reclaimed-CF as compared to (a) CFRP (control) after thermal decomposition at heating rate of (b) 10 °C min⁻¹ and (c) 5 °C min⁻¹.
3.5. Chemical analysis of decomposition stages

FT-IR analysis is a useful tool to characterize the element decomposed when the CFRP degrades. Figure 6 shows the main functional groups degraded during the thermal decomposition process. Different stages of decomposition were selected corresponding to a local maximum of peaks of decomposition at 360 °C (1st stage), 460 °C (2nd stage) and 540 °C (final heating temperature) observed on DTG curves in the previous result. CFRP waste used in the high-end application industry always consist of CF as reinforcement materials impregnated in complex epoxy resins. Apparently, CFRP (control) shows FT-IR spectra of O–H and CH₂ functional groups of epoxy at ~3400 cm⁻¹ and ~2900 cm⁻¹ respectively. Another important functional group observed are C=O (~1600 cm⁻¹), C=C (~1550 cm⁻¹) and C–H (~850 cm⁻¹). Start from the 1st decomposition stage, the
significance elimination of all the important peak of epoxy resin was observed. Followed by 2nd and 3rd decomposition stages (540 °C as final heating temperature), complete elimination of all important functional groups of the epoxy matrix was observed. Finally, only C FT-IR pattern attributed to solid CF was left.

3.6. Chemical analysis of decomposition stages
Raman spectroscopy technique was used to investigate the vibrational and rotational frequency modes of reclaimed-CF during the thermal decomposition of CFRP. Figure 7 illustrates the main features in the Raman spectra of reclaimed-CF called G and D bands, centered at around 1590 and 1380 cm⁻¹, respectively for all stages of CFRP thermal decomposition. The G-band is attributed to the in-plane vibrations of sp²-bonded crystalline carbon and has been observed in single-crystal graphite, while D-band, is assigned to the in-plane vibrations of sp²-bonded carbon with structural imperfections (Ismail and Levent 2015, Deng et al 2019).

4. Discussion

4.1. Different atmosphere result in different thermal decomposition behaviour
The first decomposition step for both atmospheres, nitrogen and oxygen was attributed to the release of small molecules and decomposition of the organic epoxy matrix (Meyer et al 2009). Decomposition under nitrogen leads to a formation of carbonaceous residue covering the CF due to the carbonization of resin (Tranchard et al 2017). Indeed, it was reported that very small weight loss of CF under nitrogen over the temperature of 300 to 500 °C is assigned to the decomposition of an organic-based sizing compound on the CF (Feih and Mouritz, 2012). It means, only the resin and sizing can be decomposed in a nitrogen atmosphere and left the solid CF as residue. However, high residue (72.70%) even at 1000 °C indicates an unsuccessful separation of CF from their matrix which highlighted in the red circle (figure 1). Under the oxygen atmosphere, the decomposition of the material involves the formation of carbonaceous residue start from the first step of decomposition at 240 to 500 °C (figure 1(a)). Larger weight loss is observed for the oxygen atmosphere at ~350 to 450 °C is accredited to the decomposition of macromolecules by cracking of covalent bonds (Meyer et al 2009). The second decomposition step observed for oxygen atmosphere at 700 to 750 °C is suggested to be correlated with decomposition of CF. Here, the influence of oxidation in an oxygen atmosphere is observed to be higher than the influence of thermal decomposition in a nitrogen atmosphere.

It can be inferred that thermal decomposition using a single atmosphere of nitrogen or oxygen is not effective, since the aim to achieve a complete separation of CF and their matrix is not reached. Oxygen does not influence the decomposition of the resin up to 400 °C, since it burns out everything to carbonaceous residue as reported by (Tranchard et al 2017). However, the oxygen plays a role to complete the decomposition at a higher temperature in the nitrogen-based decomposition of CFRP. Meyer et al (Meyer et al 2009) found similar results of reclaimed-CF after the first step of decomposition at 550 °C in nitrogen followed by the second step at 550 °C in oxidant conditions. The fibres retained more than 95% of their tensile strength without resin residue on the surface. It can be resolved, the combination of the atmosphere is needed for complete separation of solid CF from their matrix as well as maintaining their reinforcement capability.

4.2. Different heating rate result in shifting of thermal decomposition
The first negative derivative of the TG (DTG) curve of CFRP at 5 °C/min and 10 °C/min showed three decomposition phases (figures 2(a)–(c)): the first phase commenced at 300 °C and finished with a shoulder at 420 °C. The second phase occurred between 420 and 520 °C. The third phase takes place between 538 and 620 °C with a maximal decomposition rate of 1.25 wt% °C⁻¹ at a temperature of 600 °C and between 550 and 671 °C with a maximal decomposition rate of 1.0 wt% °C⁻¹ at a temperature of 650 °C for 5 °C min⁻¹ and 10 °C min⁻¹ respectively. This reveals the complete elimination of 40% of epoxy resin and left 60% of solid CF for slower heating (5 °C min⁻¹). Compared with the higher heating rate (10 °C min⁻¹) produced slightly higher
weight% of residue (62.39%), it indicates an incomplete elimination of epoxy resin from recovered CF. Thus, a lower heating rate plays a significant role in producing clean and perfect recovered CF in the thermal decomposition profile.

In the oxygen atmosphere, the possible reason for this temperature shift of higher heating rate (10 °C/min) for about 30 °C compared to the slow heating rate (5 °C/min) is the competitive reactions (Tranchard et al. 2017). A thermal decomposition reaction is said to be autocatalytic if one of the reaction products such as gases evaporated, is also a catalyst for the same reaction. Particularly, decomposition of CFRP is suggested to be catalyzed and promoted by the evolution of NH₃ (Tranchard et al. 2017) and the breakup of chains could be accelerated by the formation of acidic gases during the composite decomposition process. The process of carbon fiber separation from the matrix also could be accelerated by COS and SO₂ gases (Patent No. WO/2014/035393, 2014). The higher heating rate that favors the production of gases and volatiles will shift the overall thermal heat flow to more endothermic (Ka-Leung et al. 2011), as can be seen, occurred at process temperature >500 °C for 10 °C/min compared to 5 °C/min. Thus, it indicates that these gases released from the epoxy decomposition play a catalyzing role and promote the decomposition of the epoxy resin during the pyrolysis in accordance with an autocatalytic reaction.

Earlier research stated that the heating rate can also affect the overall energy consumption, product quality and yield of the thermal decomposition recovering process (Ka-Leung et al. 2011). Based on the fact that pyrolysis (nitrogen atmosphere) is an overall endothermic process but performs exothermically at its early stage, the pyrolysis energy consumption could be reduce in order to reach its full potential by trapping the exothermic heat released in the beginning of the pyrolysis process and using it to fulfill the energy requirement of the endothermic reactions at the end of the process (Kwok-Yuen et al. 2011). This can be reached through applying a lower heating rate at the early stage of the nitrogen atmosphere and trapping more exothermic heat and lead to lower energy consumption for the oxidative reaction at a higher temperature.

4.3. Morphological behaviour of reclaimed-CF
Many fractured residues scattered at the surface of reclaimed-CF after thermal decomposition at the higher heating rate is attributed to incomplete decomposed resin and pyrolytic carbon (Meyer et al. 2009). This result is aligned with TGA analysis of the 3rd decomposition stage of CFRP for 10 °C/min heating rate at temperature 550 °C which produced higher final residues (62.39%) compared to 5 °C/min heating rate at temperature 540 °C which produced lower final residues (60%). Since the epoxy resin content is ~40% of the CFRP, a higher heating rate (10 °C min⁻¹) still has ~2% epoxy resin which is not completely burned and removed from recovered CF surfaces. This implies slower heating resulted in more complete epoxy resin removal than faster heating.

In the case of CFRP, through thermal decomposition in a nitrogen atmosphere, the polymeric matrix is broken down and by-products formed consist of organic materials in gas and/or liquid form followed by complete burning of pyrolytic carbon in an oxygen atmosphere at high heating temperature, leaving the CF reinforcement in solid form. (Meyer et al. 2009) reported earlier that in the oxidative atmosphere, the CF is still covered by matrix residues at 500 °C and completely removed at 600 °C. However, the final temperature, 540 °C applied in this study reveals clean and clear CF at a slow heating rate with a combined nitrogen atmosphere at the early stage and oxygen atmosphere at high temperature. The thermal decomposition approached consists of atmosphere, and heating rate, which are the key parameters that must be carefully adjusted because they lead to lower final heating temperature and reflects a change in the mechanism of CFRP thermal decomposition.

4.4. Mechanism of CFRP thermal decomposition
Compared to CFRP (control), the first stage of decomposition in a nitrogen atmosphere approximately at 300 °C reveal the degradation of epoxy resin that recovered the CF at this stage is still tightly bonded to each other due to incomplete removal of epoxy resin. Next, the second stage of thermal decomposition produced a clearer and looser bundle of CF, but still with sizing and epoxy resin covered on their surfaces. Finally, at approximately 540 °C, clean and smooth CF that separated from each other is observed. Temperature 540 °C in the oxidative atmosphere is recognized as the final heating temperature to sustain solid CF and removed almost 100% of epoxy resin and sizing.

Apparently, there are three significant stages of the decomposition mechanism. As illustrated in figure 5, during the 1st stage of decomposition, the nitrogen atmosphere is an important factor that contributes to the transform of the matrix into smaller molecules. These smaller molecules evaporate from the material and, can be used as an energy source for the process due to their high calorific value (Liu Farnsworth and Tiwari 2017). Here, the advantage is reclaimed-CF will not be degraded in a nitrogen atmosphere. However, some pyrolytic carbon (residue) bonded to the reclaimed-CF surface is still remains. This reclaimed-CF is further heated in the nitrogen atmosphere to remove the epoxy resin and sizing completely. When oxygen is purge with a very slow heating rate
after 2nd decomposition stage, at temperature 420 °C, the complete burning of pyrolytic carbon takes place until the final heating temperature 540 °C. Figure 5 reveals a clean and completely separated CF at the final heating temperature. (Meyer et al 2009) found that in the synthetic air environment, within the temperature range from 580 to 600 °C, nearly all the matrix can be removed. However, based on the results presented, this study suggests that further heating above 540 °C will lead to the third decomposition stage of CF and cause degradation of their surfaces.

In a nitrogen atmosphere, an increase in the matrix weight loss was detected for the first 60 min at 300 °C and became much smaller at 420 °C. Hence, the decomposition reaction is time-dependent at the lower temperature range, which indicates the importance of a nitrogen atmosphere for thermal decomposition at the early heating temperature. Neither high temperatures nor long residue time of more than 60 min is required when pyrolysis is performed in a nitrogen atmosphere (Liu et al 2017). Relatively, the oxidation reaction is much more time-dependent. An increase in weight loss with residue time was observed in an oxygen atmosphere, and therefore the exposure time of the reclaimed-CF in this temperature should be short and minimum enough in order to avoid oxidative reclaimed-CF damage. As an alternative, the very slow heating rate is applied in this study during the oxidative reaction to expose only the minimum oxidative reaction and completely remove the epoxy resin and pyrolytic carbon from reclaimed-cf

4.5. Chemical analysis of CFRP thermal decomposition

Generally, epoxy resins, used in the high-end application industry, show appropriate thermal resistance performances in addition to the required high mechanical properties (Boulanghien et al 2018). However, in a nitrogen atmosphere, the amount of oxygen-containing functional groups on the surface of the reclaimed-CF can be decomposed significantly. Further heating in the oxidative atmosphere complete the burning of all the oxygen-containing functional groups and organic-based sizing that covered the CF surfaces. A final heating temperature of 540 °C appears then to be the efficient final heating temperature for the thermal decomposition process in order to maintain acceptable strength for CF (Pickering 2004). Hence, FT-IR analysis supported the proposed heating profile of CFRP thermal decomposition in combination atmosphere of nitrogen followed by oxygen until final heating temperature of 540 °C at low (5 °C min−1) heating rate to produce reclaimed-CF in this study.

4.6. Raman spectroscopy analysis of CFRP thermal decomposition

Raman spectroscopy as a method of condition assessment for reclaimed-CF revealed that the Raman band related to C–C vibrations in graphite is present in all stages of CFRP thermal decomposition at ~1585 cm−1 (Washer and Blum 2008). Hence, the reclaimed-CF structure is preserved through CFRP decomposition stages. It can be seen that the spectral line shape changes depending upon the decomposition stages as well as the atmosphere. With an increase in heating temperature from control to the final decomposition stage, Raman G bands tend to narrow in peak width because the impregnated reclaimed-CF structure has gradually become visible. As compared to control, the 1st and 2nd decomposition stages occurred in a nitrogen atmosphere, shows reclaimed-CF Raman G band become narrower while D band becomes broader. This might be attributed to significant CFRP matrix decomposition which results in reclaimed-CF graphitic properties become apparent. Interestingly, as the atmosphere change to oxygen, the D and G band intensity rapidly reduces, whereas that for the peak shape still remains. This might be attributed to the complete burning of the resin matrix and amorphous property of reclaimed-CF exposed. The similar observation for the virgin CF reported recently, suggests that this reclaimed-CF mainly consist of the sp2 carbon layers and the residual D band is similar to the behavior of amorphous carbon (Okuda et al 2018). Hence, Raman spectroscopy analysis is significant to further explore the evolution of the reclaimed-CF structure throughout the decomposition stages.

5. Conclusions

The recycling process of CF from CFRP waste via thermal decomposition is proposed to be an effective method. Via this technique, a single atmosphere of nitrogen or oxygen is found ineffective. Oxygen does not influence the decomposition of the resin at an early temperature. A combination of nitrogen atmosphere at the early temperature followed by an oxygen atmosphere at higher temperature is needed to achieve a complete separation of reclaimed-CF from their complex epoxy resin matrix while maintaining their structure to act as reinforcement material. Lower heating rate leads to lower energy consumption, complete elimination of resin matrix and production of a higher quality of reclaimed-cf. Further morphological, chemical and structural analysis done by SEM, FT-IR and Raman spectroscopy supported those findings proven that thermal decomposition with careful examination of exchanging the atmosphere from nitrogen to oxygen at the lower heating rate is promising to recycle CFRP waste from the high-end industry at low final heating temperature.
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