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**DOI**

[10.1016/j.polymertesting.2017.05.038](https://doi.org/10.1016/j.polymertesting.2017.05.038)

**Publication date**

2017

**Document Version**

Final published version

**Published in**

Polymer Testing

**Citation (APA)**

Zhu, G., Wang, W., Wang, R., Xiao, Y., Yang, Y., Dong, A., Wang, D., Shu, D., Wang, J., Sun, B., & Boom, R. (2017). Thermal degradation behaviour of resins in aluminium composite under isothermal condition. *Polymer Testing*, 61, 448-454. <https://doi.org/10.1016/j.polymertesting.2017.05.038>

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## Material Behaviour

## Thermal degradation behaviour of resins in aluminium composite under isothermal condition



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## ARTICLE INFO

## Article history:

Received 2 January 2017

Received in revised form

26 April 2017

Accepted 24 May 2017

Available online 26 May 2017

## Keywords:

GLARE

Thermal degradation

TGA

Isothermal kinetics

Delamination

## ABSTRACT

As a member of the aluminium composite, GLARE (GLASS fibre/epoxy REinforced aluminium laminates) was used in the upper fuselage of Airbus A380 because of its superior mechanical properties over monolithic aluminium alloys. Thermal processing is a potential method for materials recycling and reuse from GLARE scrap with the aim of environmental protection and economic benefits. Thermal delamination is a crucial pre-treatment step for GLARE recycling. Differential scanning calorimetry (DSC) and Thermogravimetric analysis (TGA) tests have been used to identify the decomposition temperature range of epoxy resins under non-isothermal condition in our previous work [1]. To obtain an appropriate solution for GLARE thermal delamination, the thermal degradation behaviour of epoxy resins in GLARE under isothermal conditions were investigated and isothermal decomposition kinetic models were built up based on DSC and thermogravimetric analysis TGA. The thermal delamination process of GLARE is determined based on thermal analysis results and experimental optimization.

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## 1. Introduction

GLARE (GLASS fibre/epoxy REinforced aluminium laminates) is a member of the Fibre Metal Laminates family, and it has been selected as upper fuselage skin in the superjumbo Airbus A380 because of the excellent damage tolerance, fatigue resistance, flame penetration resistance and 10% weight saving compared to monolithic 2024 Al alloy [2–4]. About 500 m<sup>2</sup> GLARE is employed in each Airbus A380 [5]. With the increased plane orders of Airbus A380, a constant flow of End-of-Life (EOL) GLARE scrap will be generated after retiring of planes within forty years. Moreover, many tons new GLARE scrap have been accumulated during the Airbus A380 manufacturing. Although GLARE is mainly applied in the aerospace industry and the yield of GLARE is limited compared with that of

carbon fibre reinforced polymers (CFRPs), recycling of both the industrial new scrap and EOL old scrap already becomes a relevant issue and should be seriously considered.

Traditionally, in the disposal of EOL fibre-reinforced polymers, much of the waste polymers currently produced is ultimately sent to landfill [6,7], which is a poor solution for management of resources, environmental impact, and economic opportunity [8]. Turning GLARE scraps into valuable resource is important for the sustainable use of the materials in some applications which depends on the compositions and properties of recycled materials. Different methods for fibre reinforced polymers recycling have been developed (though most of them are not yet commercialized), including mechanical recycling, thermal recycling and chemical recycling [7,9,10]. Mechanical recycling of GLARE usually leads to small pieces of aluminium alloy sheet and short glass fibres. Tempelman [11] had developed a low temperature cryogenic liberation process to separate the aluminium sheets and the glass fibres/epoxy layers from GLARE, based upon the large difference of thermal expansion coefficients between glass fibres ( $0.8 \times 10^{-5}/K$ ) and aluminium ( $2.4 \times 10^{-5}/K$ ). But GLARE scrap needs to be cut into

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pieces of 10 mm sheet which causes serious properties degradation of glass fibres and a reduction of yield of recycled 2024 Al alloy. Chemical recycling is based on treatment in a reactive medium, such as nitric acid [12], catalytic solutions [13], alcohol [14], and some subcritical or supercritical fluids [15] etc. Comparing to general fibre reinforced polymers, the challenge in GLARE recycling is the selection of a medium reactive solution which should be inert to the aluminium alloy in GLARE. Even though a suitable medium reactive solution can be found, the efficiency of chemical recycling could be limited because of the good hygroscopicity resistance of GLARE.

The thermal behaviour of resins has been studied in previous works [16–19], and most of resins can be completely decomposed under appropriate temperature [20,21]. Thus, thermal recycling could be a practical solution for the recycling of aerospace Al alloy and glass fibre materials from GLARE scrap. GLARE thermal delamination depends to a large extent on the thermal decomposition behaviour of epoxy resins in GLARE. Thermal analysis of epoxy resins under non-isothermal and isothermal conditions can provide useful information for GLARE thermal recycling. The thermal decomposition behaviour of epoxy resins at elevated temperature (non-isothermal condition) can provide detailed information for GLARE thermal delamination, e.g. initial decomposition temperature, end decomposition temperature, and relationship between decomposition degree and temperature under different heating rates. DSC and TGA tests have been used to identify the decomposition temperature range of epoxy resins under non-isothermal condition in our previous work [1].

Thermal decomposition behaviour of epoxy resins under given fixed temperatures (isothermal condition) is also important to understand the relationships between decomposition degree, holding time and decomposition rate at different decomposition temperatures, which benefits the determination of the delamination process parameters. Thus, in this paper, the decomposition

behaviour and kinetics of epoxy resins in GLARE under isothermal conditions were studied by using thermogravimetric analysis. The decomposition behaviour under both nitrogen and air atmospheres were discussed. The employed temperatures for isothermal thermal analysis were decided according to the decomposition temperature range of epoxy resins obtained by the non-isothermal thermal analysis. The isothermal kinetic models were established based on the rate of mass loss of epoxy resins during thermal decomposition. At last, based on the thermal analysis results of epoxy resins decomposition under isothermal conditions, GLARE scrap delamination experiments at several temperatures were performed to optimize the delamination process.

## 2. Materials and experiments

### 2.1. Materials

The GLARE 3-8/7-(0.3–0.4) new scrap from the Airbus A380 window was used in this research. The GLARE new scrap consists of 8 layers 2024-T3 aluminium alloy sheets with modified epoxy phenolic primer BR 127 on both surfaces of each sheet, bonded together with 7 layers of modified epoxy film adhesive FM 94/S2-glass fibre prepreg system [2,4,22]. The detailed structure of the GLARE scrap is shown in Fig. 1. The total thickness of GLARE is 4.6 mm, and the thickness of prepregs between two aluminium alloy sheets was 0.3 mm.

For isothermal analysis, the initial mass for each cuboid shaped GLARE for test was taken as 60 mg. The test shows that the weight percent of epoxy resins (BR 127 and FM94) in GLARE is 10 wt%, which means that the sample contains 6 mg epoxy resins.

For GLARE delamination under isothermal conditions in a box electric resistance furnace, the size of prepared GLARE scrap was  $50 \times 35 \times 4.6$  mm (Length  $\times$  Width  $\times$  Thickness) and the weight was 20 g.

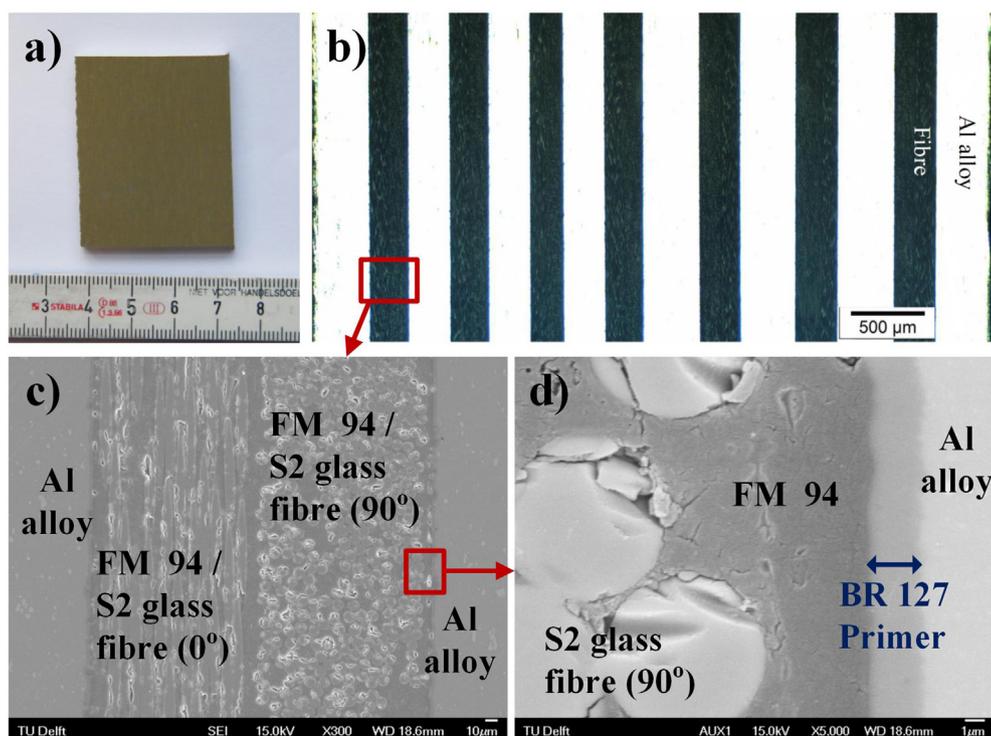


Fig. 1. GLARE scrap from Airbus A380 manufacturing a) front view, b) layered structure, c) and d) SEM patterns, the magnification is 300 $\times$  and 5000 $\times$ , respectively.

## 2.2. Experiments

### 2.2.1. Thermal analysis under isothermal condition

Mass changes of GLARE under isothermal condition in dry nitrogen or air atmosphere were obtained by using TGA device (SETSYS Evolution-1750, SETARAM). The TG device was heated from the ambient temperature to the given temperature at a heating rate of 90 °C min<sup>-1</sup>. The sample was put in an Al<sub>2</sub>O<sub>3</sub> crucible which was connected to a fine Al<sub>2</sub>O<sub>3</sub> rod, which was slowly inserted into the furnace. Total holding time for isothermal treatment is 3 h, and then cooled down to room temperature in furnace without heating power. The gas condition of the furnace during the whole treatment process were set up as 100 ml min<sup>-1</sup> N<sub>2</sub> for inert condition or 80 ml min<sup>-1</sup> N<sub>2</sub> and 20 ml min<sup>-1</sup> O<sub>2</sub> for simulating a flow of air where N<sub>2</sub> and O<sub>2</sub> was mixed firstly before the experiments.

The results of the thermal analysis under non-isothermal conditions in our previous work [1] indicated that there are four steps during the epoxy resins decomposition in GLARE. The first step is attributed to the decomposition of epoxy resins BR127 coated on the Al alloy sheets, and the later three steps are attributed to the decomposition of modified epoxy film adhesive FM94. In order to better understand the decomposition behaviour, the analysis of isothermal kinetics was studied under different temperatures, which are selected according to the decomposition behaviour of GLARE when the heating rate was 1 °C min<sup>-1</sup> (see Table 1) [1]. The temperatures between the initial decomposition temperature of each step and the temperature at maximum decomposition ratio of the corresponding last step were chosen as the given temperatures for isothermal analysis, which was 230 °C, 310 °C, 350 °C and 450 °C respectively. The highest temperature for isothermal analysis was chosen as 450 °C considering the start melting temperature of 2024 Al is 502 °C [23].

### 2.2.2. Isothermal kinetic models

Two general categories of kinetic models were used in the isothermal analysis of resin decomposition: *n*th-order and autocatalytic.

The equations of *n*th-order reaction are described as follows [24–26]:

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \quad (1)$$

$$k(T) = A \exp\left(\frac{-E_a}{RT}\right) \quad (2)$$

$$f(\alpha) = (1 - \alpha)^n \quad (3)$$

where  $\alpha$  is the decomposition degree,  $t$  is decomposition time (s),  $d\alpha/dt$  is the decomposition rate,  $T$  is decomposition temperature (K),  $k(T)$  is the rate constant (s<sup>-1</sup>),  $f(\alpha)$  is the reaction model,  $A$  is the pre-exponential factor,  $E_a$  is the activation energy (J mol<sup>-1</sup>),  $R$  is the universal gas constant, and  $n$  is reaction order.

**Table 1**

Initial decomposition temperatures ( $T_i$ /°C) and temperature at maximum decomposition ratio ( $T_m$ /°C) for each step under non-isothermal condition in nitrogen and air, both the heating rates are 1 °C min<sup>-1</sup> [1].

| Atmosphere | Step 1 |       | Step 2 |       | Step 3 |       | Step 4 |       |
|------------|--------|-------|--------|-------|--------|-------|--------|-------|
|            | $T_i$  | $T_m$ | $T_i$  | $T_m$ | $T_i$  | $T_m$ | $T_i$  | $T_m$ |
| Nitrogen   | 188    | 227   | 255    | 293   | 311    | 341   | 354    | 500   |
| Air        | 188    | 227   | 255    | 298   | 318    | 326   | 356    | 423   |

According to equations (1)–(3), the final equation of *n*th-order kinetic model can be written as:

$$\frac{d\alpha}{dt} = A \exp\left(\frac{-E_a}{RT}\right)(1 - \alpha)^n \quad (4)$$

Systems obeying a *n*th-order kinetics model will obviously reach the maximum reaction rate at the beginning of the decomposition ( $t = 0$ ).

In this research, the decomposition degree  $\alpha$  is defined as [27,28]:

$$\alpha = \frac{m_i - m}{m_i - m_e} \quad (5)$$

where  $m_i$ ,  $m$ , and  $m_e$  are respectively the mass of epoxy resins before, during in and after decomposition process.

Under an isothermal condition, the parameters  $A$ ,  $E_a$  and  $n$  can be obtained through two steps linear regression analysis by using equations (6) and (7) which are modified from equations (2) and (4), respectively:

$$\ln\left(\frac{d\alpha}{dt}\right) = n \ln(1 - \alpha) + \ln k \quad (6)$$

$$\ln k = \ln A - \frac{E_a}{RT} \quad (7)$$

For autocatalytic reaction, the function  $f(\alpha)$  has the following form [29,30]:

$$\frac{d\alpha}{dt} = (k_1 + k_2\alpha^m)(1 - \alpha)^n \quad (8)$$

where  $k_1$ ,  $k_2$  are the rate constants (s<sup>-1</sup>),  $k_1 = (d\alpha/dt)$  at  $t = 0$  and  $m$  is also a reaction order. The maximum decomposition rate appears at about 10–40% of decomposition for an autocatalytic reaction [26].

Under an isothermal condition, the constant  $k_1$  can be obtained as the initial decomposition rate when decomposition degree  $\alpha$  is zero. In order to calculate other parameters  $k_2$ ,  $m$  and  $n$ , equation (8) can be rearranged in the following forms:

$$\ln\left(\frac{d\alpha}{dt}\right) = n \ln(1 - \alpha) + \ln(k_1 + k_2\alpha^m) \quad (9)$$

$$\ln\left\{\left[\frac{\left(\frac{d\alpha}{dt}\right)}{(1 - \alpha)^n}\right] - k_1\right\} = m \ln \alpha + \ln k_2 \quad (10)$$

$$\ln\left(\frac{d\alpha}{dt}\right) - \ln(k_1 + k_2\alpha^m) = n \ln(1 - \alpha) \quad (11)$$

Firstly, we can assume that the reaction order  $n$  can be determined according to the slope of a plot of  $\ln(d\alpha/dt)$  versus  $\ln(1 - \alpha)$ , as described in equation (9). Then, the reaction order  $m$  and the constant  $k_2$  can be calculated from the slope and the intercept of a plot of  $\ln\{[(d\alpha/dt)/(1 - \alpha)^n] - k_1\}$  versus  $\ln \alpha$  by using the obtained parameters  $k_1$  and  $n$ , as presented in equation (10). More precise values of parameters can be obtained when an iterative procedure is used. The new refined parameter  $n$  can be determined from equation (11) after using the previously obtained  $k_1$ ,  $k_2$  and  $m$  values. The new  $n$  can be applied to equation (10) to obtain new values of  $k_2$  and  $m$ . The iterative procedure can be repeated until the values of parameters are tending toward stability.

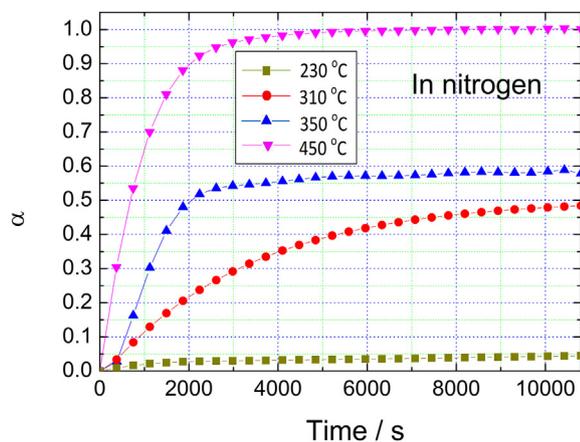


Fig. 2. Conversion versus. time at different isothermal temperatures for epoxy resins decompositions in nitrogen atmosphere.

### 2.2.3. GLARE thermal delamination

The thermal delamination of GLARE scrap was carried out in a Carbolite electric resistance furnace under air atmosphere, and the delamination temperature was decided based on the thermal analysis results of resins decomposition.

## 3. Results and discussion

### 3.1. Isothermal thermal analysis results of epoxy resins

Figs. 2 and 3 show the isothermal decomposition curves of epoxy resins of GLARE at different temperatures in nitrogen and air. The final conversion was enhanced with the isothermal temperature. The decomposition percentage was 4.4% and 7.2% at 230 °C for 3 h when the decomposition atmosphere was nitrogen and air, respectively. According to the conversion curves in nitrogen and air at 230 °C, it is obvious that the oxidising atmosphere can accelerate the decomposition of epoxy BR 127 compared to an inert atmosphere. As we can see in Figs. 2 and 3, at 310 °C, both conversions in nitrogen and air atmospheres were increased with the increasing isothermal holding time through the entire 3 h, and the final conversion was 48.4% and 43.4%, respectively in nitrogen and air, which may be attributed to the more efficient pyrolysis of step 3 in nitrogen compared to that in air. The final conversion at 350 °C after 3 h was increased to 57.8% and 69.4%, respectively in nitrogen and

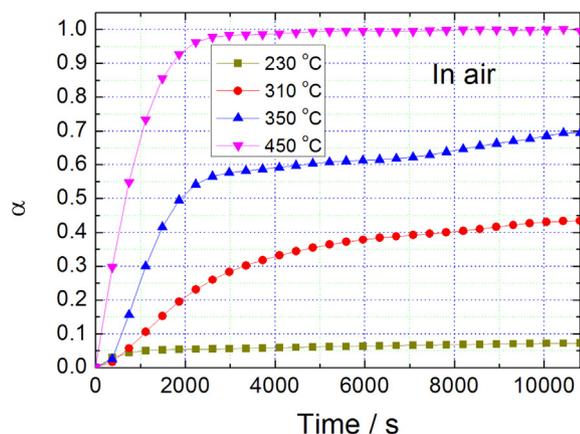


Fig. 3. Conversion versus. time at different isothermal temperatures for epoxy resins decompositions in air atmosphere.

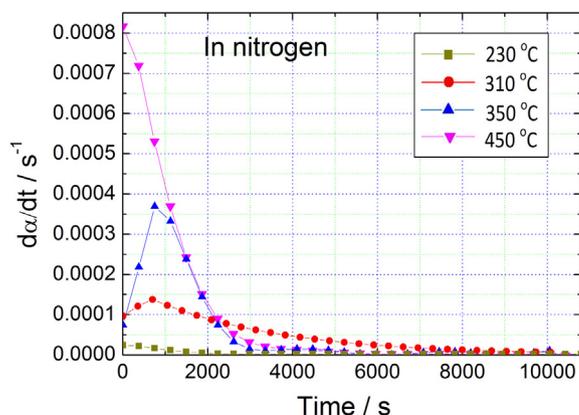


Fig. 4. Conversion rate versus. time at different isothermal temperatures for epoxy resins decompositions in nitrogen atmosphere.

air. All epoxy resins in GLARE can be completely decomposed at 450 °C, the required time was respectively 100 and 70 min in nitrogen and air. In general, an oxidising atmosphere is more preferred for epoxy resins decomposition compared to inert atmosphere.

The curves of the conversion rate as a function of time are shown in Figs. 4 and 5. It can be seen that the peak values of the conversion rate were increased with the increase of isothermal temperature. But, the discrepancy of maximum conversion rates caused by different atmospheres was negligible though the final conversion in air is higher than that in nitrogen.

The conversion rate (as a function of conversion of epoxy resins) at different temperatures in nitrogen and air are shown in Figs. 6 and 7 (symbols) respectively. The decomposition behaviour at 230 °C and 450 °C in nitrogen and air follows the  $n$ th-order mechanism with a maximum conversion rate at the beginning of decomposition. The decomposition behaviour at 310 °C and 350 °C in nitrogen and air proceed through an autocatalytic mechanism, and the maximum decomposition rate appears at about 10–25% of decomposition. The isothermal holding temperatures were selected according to the initial temperatures of four different steps which were defined based on the heat flow under dynamic condition. At 230 °C, the main conversion is attributed to the reaction presented as the step 1 in the dynamic thermal analysis. Similarly, reactions of steps 1–2 are attributed to the major conversion at 310 °C, reactions of steps 1–3 are attributed to most of conversion at 350 °C, and reactions of all steps 1–4 are attributed to the

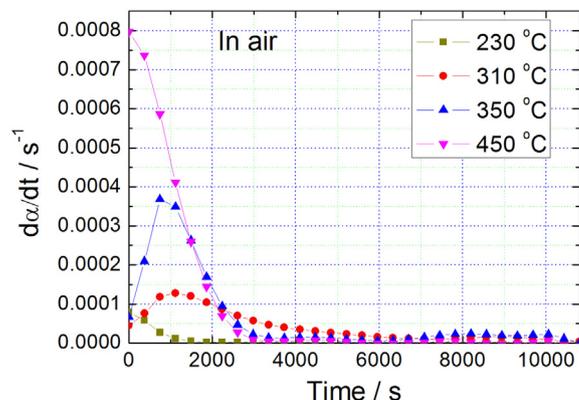


Fig. 5. Conversion rate versus. time at different isothermal temperatures for epoxy resins decompositions in air atmosphere.

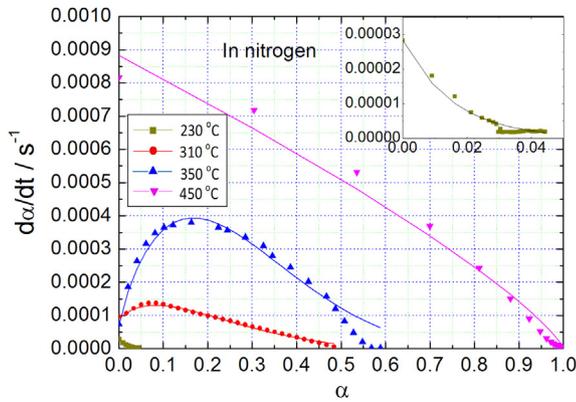


Fig. 6. Comparison of experimental (symbols) and theoretical (lines) conversion rate versus conversion at different isothermal temperatures for epoxy resins decomposition in nitrogen.

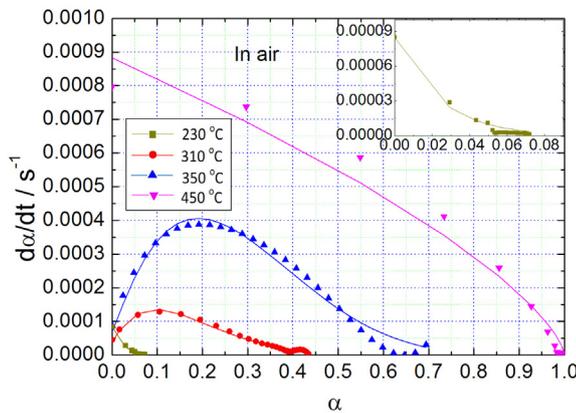


Fig. 7. Comparison of experimental (symbols) and theoretical (lines) conversion rate versus conversion at different isothermal temperatures for epoxy resins decomposition in air.

conversion at 450 °C. Thus the resins decomposition behaviour are much different at different isothermal holding temperature, leading to two different decomposition mechanisms.

The obtained parameters of the kinetic models after regression analysis based on equations (6)–(7) and equations (9)–(11) are listed in Table 2.

For the  $n$ th-order decomposition mechanism (at 230 °C and 450 °C), the activation energies in nitrogen atmosphere were a little lower in comparison to those in air atmosphere at the same holding temperature, but the initial conversion rates  $k$  (maximum conversion rate) in air were higher compared to those in nitrogen, especially at 230 °C. It is consistent with previous non-isothermal

analysis results. As we can see in Table 1, the required start temperature for step 1 and step 4 in nitrogen was a little lower than that in air, indicating that the start-up of decomposition should be much easier in nitrogen compared to that in air, at 230 °C and 450 °C. But the conversion in air was higher than that in nitrogen at 230 °C for 3 h, and the required time for complete decomposition in air was shorter than that in nitrogen at 450 °C, as shown in Figs. 2 and 3. For the autocatalytic decomposition mechanism (at 310 °C and 350 °C), both constants  $k_1$  and  $k_2$  obey the Arrhenius form. The constant  $k_1$  governs the early stage of autocatalytic reaction and the term  $k_2\alpha^m$  represents the effect of the reaction products on the conversion rate [31,32]. Thus, as shown in Table 2, the values of  $k_2\alpha^m$  in air are obviously stronger than those in nitrogen, indicating that the effect of reaction products on conversion rate is more significant within an oxidising atmosphere.

The comparison of the experimental data with the decomposition rates obtained from the kinetic models is presented in Figs. 6–7. It is clear that most of the predicted values agree with the experimental data.

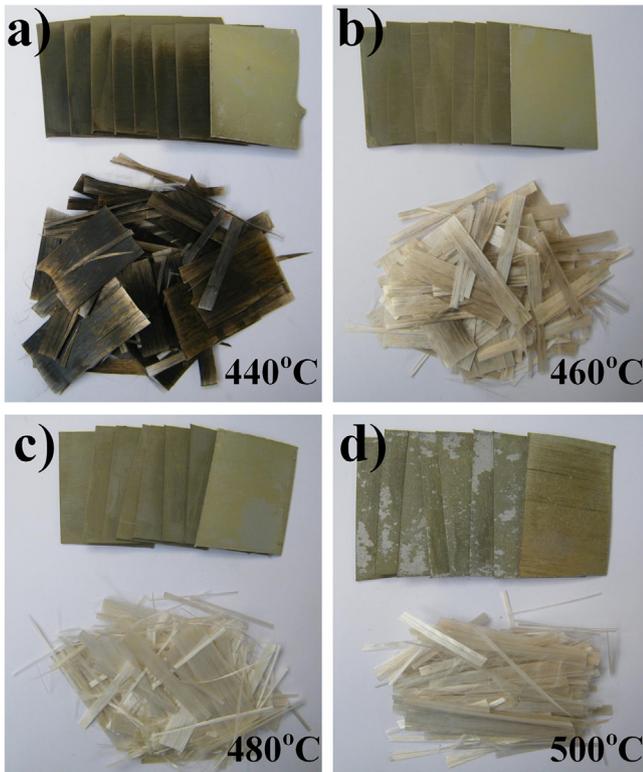
### 3.2. GLARE delamination

The thermal analysis of epoxy resins decomposition under non-isothermal and isothermal conditions provide useful information for the optimization of process parameters of GLARE thermal delamination. It is obvious that an oxidising atmosphere is more preferred for GLARE delamination. Different from thermal analysis where the mass of GLARE sample was only 0.06 g, a longer holding time would be required for large-sized GLARE scrap delamination because of the influence of kinetic factors, such as scrap size and air convection. An appropriate holding temperature is important for GLARE thermal delamination with high efficiency. The holding temperature should be high enough to ensure that epoxy resins can be completely decomposed at this temperature while avoiding the oxidation of 2024 Al alloy. Based on previous isothermal analysis results for epoxy resins decomposition in air (Fig. 3), the suitable holding temperature for GLARE delamination should be selected in between 450 °C and 502 °C (start melting temperature of 2024 Al alloy).

In order to determine the optimum holding temperature for GLARE delamination, four different holding temperatures were employed, 440 °C, 460 °C, 480 °C and 500 °C, respectively. GLARE delamination experiments were conducted in an electric resistance furnace under air atmosphere, the size of GLARE scrap is 50 × 35 × 4.6 mm (Length × Width × Thickness) and the weight was 20 g for delamination, the results are shown in Fig. 8. The aluminium sheets and S2-glass fibre were well separated after thermal delamination even though the holding temperature was just 440 °C. The results also show that some oxidation spots occur on the surface of Al sheets when the holding temperature was 500 °C, as shown in Fig. 8d. A strong exothermic peak can be found

Table 2  
Parameters of kinetic models for epoxy resin decomposition at four temperatures.

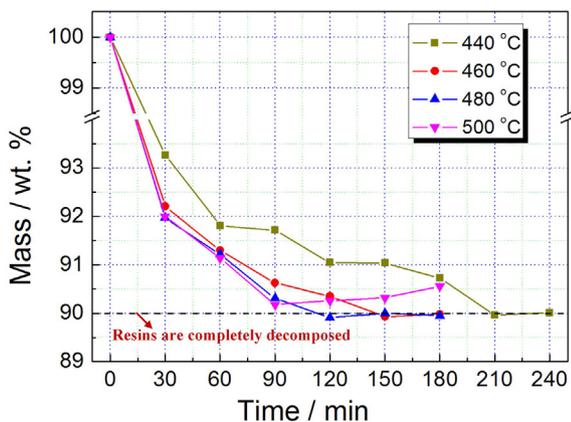
| Atmosphere | Temperature °C | nth-order |                 |   |       | Autocatalytic        |                      |      |      |
|------------|----------------|-----------|-----------------|---|-------|----------------------|----------------------|------|------|
|            |                | $E_a$ /KJ | $A/\times 10^3$ | $k = A \times \exp(-E_a/RT)/\times 10^{-5}$ | $n$   | $k_1/\times 10^{-5}$ | $k_2/\times 10^{-3}$ | $m$  | $n$  |
| Nitrogen   | 230            | 84.08     | 15.24           | 2.83  | 63.15 | /                    | /                    | /    | /    |
|            | 310            | /         | /               | /   | /     | 9                    | 1.13                 | 0.89 | 5.67 |
|            | 350            | /         | /               | /   | /     | 7                    | 3.80                 | 0.89 | 4.11 |
|            | 450            | 112.92    | 126.99          | 88.28                                       | 0.80  | /                    | /                    | /    | /    |
| Air        | 230            | 85.66     | 66.31           | 8.44  | 42.12 | /                    | /                    | /    | /    |
|            | 310            | /         | /               | /   | /     | 4                    | 3.13                 | 1.03 | 8.46 |
|            | 350            | /         | /               | /   | /     | 7                    | 5.83                 | 1.10 | 4.34 |
|            | 450            | 114.18    | 156.67          | 88.31                                       | 0.68  | /                    | /                    | /    | /    |



**Fig. 8.** GLARE thermal delamination at different temperatures after 120 min in air. The holding temperature was a) 440 °C, b) 460 °C, c) 480 °C and d) 500 °C respectively.

in DSC curves of GLARE under air atmosphere [1] which causes the temperature around the Al sheet to be higher than the preset furnace temperature of 500 °C. The real temperature in the furnace should be close to the start melting point of 2024 Al alloy, resulting in plenty of oxidation spots on the Al sheets. Thus, 500 °C is not suitable for GLARE thermal delamination.

The weight changes of GLARE during thermal delamination were shown in Fig. 9. As mentioned before, the weight ratio of the resins in GLARE is 10 wt%, thus it can be concluded that the resins are completely decomposed when the mass decrease of 10% is achieved during delamination. The required holding time for completely delamination of GLARE was decreased with increasing holding temperature, as shown in Fig. 9. The delamination processes was finished after 120 min when the temperature was



**Fig. 9.** Effect of thermal condition on the weight loss of GLARE scrap.

480 °C, but the required holding time was increased to 210 min at a lower temperature of 440 °C. The weight of GLARE was increased with the increase of holding time after 90 min at 500 °C which was attributed to the oxidation of 2024 Al sheets. According to the results of GLARE delamination, the appropriate delamination process was found to be 480 °C × 120 min.

### 3.3. Reuse of recycled materials

After GLARE thermal delamination, the obtained 2024 Al sheets can be recycled by the conventional refining method using the molten salt as refining agent, the metal yield can reach as high as 95% [33]. Then the recycled 2024 Al alloy is possible to be reused in non-critical aircraft components (such as stiffeners, flaps, and other relatively low-to-moderately stressed components made of sheet, plate, or extrusions), non-aircraft application (train bodies, truck bodies), as well as casting (2xx.0 alloy). In some cases, minor adjustment to the composition of recycled aerospace alloy is necessary to meet the reuse requirements.

The strength of recycled glass fibre will be decreased due to the high decomposition temperature of 480 °C. The room temperature strength of S2 glass fibre was decreased from ~5700 MPa to ~2850 MPa after thermal exposure at 480 °C according to the data reported in the released file of AGY (the supplier of S2 glass fibre for GLARE material) [34]. The 50% strength loss of the recycled S2 glass fibre is consistent with the reported strength degradation of glass fibre after reheating at 500 °C for 1 h [35]. Though the strength of the recycled glass fibre is decreased compared to the virgin glass fibre, however, previous studies have suggested that the recovered glass fibre by pyrolysis may be recycled back into composite plastics as a partial replacement for virgin material in the production of Bulk Moulding Compound or as filler in concrete [36].

## 4. Conclusions

The isothermal thermal analysis is employed to study the decomposition behaviour of GLARE, which provides significant information for the optimization of process parameters for thermal delamination of GLARE. Based on the results of non-isothermal analysis in previous work, four holding temperatures of 230 °C, 310 °C, 350 °C and 450 °C were selected to investigate the isothermal kinetics of resins decomposition in GLARE. The conversion was 4.4%, 48.4%, 57.8% and 100% when samples were kept at 230 °C, 310 °C, 350 °C and 450 °C respectively for 3 h in nitrogen, while the conversion under the same conditions in air was 7.2%, 43.4%, 69.4% and 100% respectively. All epoxy resins in GLARE can be completely decomposed at 450 °C, and the required time was 100 min and 70 min respectively in nitrogen and air. Thus, an oxidising atmosphere is more preferred for resins decomposition which is consistent with the non-isothermal analysis results of our previous work. Two decomposition mechanisms, nth-order for decomposition at 230 °C and 450 °C and autocatalysis for decomposition at 310 °C and 350 °C, were derived from the isothermal analysis. The optimum GLARE thermal delamination process should be at 480 °C for 2 h based on the results of thermal analysis and the experimental optimization.

## Acknowledgement

This work is funded by the Royal Netherlands Academy of Science and Arts (KNAW) under Project No. 10CDP026, the Shanghai Sailing Program No. 14YF1401800, the joint fund of the Ministry of Education of China under Project No. 6141A02022210, the Industry-University-Research Cooperation Annual Plan of Shanghai under Project No. CXY-2016-004 and the National Industrial Basis

Improvement Project under Project No. TC160A310-12-1. The authors would like to thank Dr. Guiming Song and Dr. Z. Zhang (Department of Materials Science and Engineering, TU Delft) for their support on STA tests. The authors are also grateful to Dr. Peter Kortbeek (Fokker Aerostructures BV) for providing the GLARE scrap.

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