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RESEARCH ARTICLE

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Influence of *Skydrol* immersion at elevated temperatures on the thermo-mechanical properties of a high-T_g anhydride epoxy resin toughened with a hydroxy-terminated polyester

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Abstract

Currently, the application of composites in aerospace parts exposed to higher temperatures and in aggressive media is still severely limited. To replace metal alloys, alternative resins systems with suitable long-term heat resistance are needed. In this study, the effect of the aviation hydraulic fluid *Skydrol* on the thermal and mechanical properties of a high-T_g, anhydride-cured epoxy resin in the unmodified and toughened state at elevated temperature is investigated. An aliphatic polyester diol was selected as an intrinsic toughener and its impact on the thermal, mechanical, and aging properties was determined. Experimental characterization of the aging effects is carried out with dynamic-mechanical characterization, infrared spectroscopy, and electron dispersion x-ray spectroscopy. In addition, the fracture toughness and the fatigue crack propagation behavior are determined. Initially, the toughened system shows an improved fracture toughness. Since oxidation is blocked by the *Skydrol* fluid only thermal degradation takes place as determined by the decrease in glass transition temperature T_g and network density. The thermal degradation leads to a tougher behavior, which is observed in both systems in static and dynamic mode with toughness decreasing with aging time again.

KEYWORDS

ageing, resins, thermosets

1 | INTRODUCTION

At a time when more and more lightweight materials are needed to meet environmental regulations and reducing kerosene consumption in the aviation industry, great efforts have been made to use polymer composites. Here, a weight reduction of only 1 kg in an Airbus A340 would reduce fuel consumption by three tons based on a 20 year service life.[1] In addition to the already well-established use in the fuselage, wings, tail unit and doors, the focus has shifted more and more to the replacement of

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Jan David Hübsch and Martin Demleitner are equally contributing authors.

aluminum or titanium alloys in high-temperature applications, for example, in air duct pipes near to the aircraft turbines or the hydraulic systems. Here, resin systems with high-T_g and temperature stability are needed as temperatures can reach up to 290°C.[2] Although bismaleimide and cyanate ester resins have been on the market for a long time, aiming for high-temperature applications, they have certain drawbacks in terms of costs, mechanical properties and health hazards.[3,4] Here, low-cost epoxy resins which generally have excellent durability and strength, could be an alternative but often lack sufficient chemical and thermal stability. Furthermore, the highly cross-linked network structure often results in inherent brittle behavior prone to fracture, requiring the addition of a toughener. Various types such as core-shell rubber, thermoplastics, silica nanoparticles or intrinsic chain extenders are commonly used to provide additional energy absorption mechanism and increase fracture toughness. With an assumed service life of at least 20 years for aircraft, the material has to withstand high-temperature and aggressive fluids without significant impact on the thermo-mechanical properties. The Airbus A320 short-haul aircraft for example, has three continuously operating hydraulic systems that require a total of 240 L of hydraulic fluid for flight controls, brakes, landing gear, and so forth.[5] Among the most chemically aggressive here is the fire-resistant hydraulic fluid based on various types of phosphate ester and marketed by Eastman Chemical Company under the brand name "*Skydrol*". A study of the reliability of fiber-reinforced composites (FRP) components exposed to aggressive fluids is crucial since severe damage to critical components and human health can occur in the event of leakage. To date, there have been few publications investigating the impact on epoxy resins after immersion in *Skydrol*.

Varley et al. determined the effect of *Skydrol* on the glass transition temperature of a toughened anhydride-cured epoxy system for 120 days at room temperature (RT).[6] They found no effect on T_g which was constant at 160–180°C depending on the toughener content. No uptake of *Skydrol* was observed at an immersion temperature well below T_g. Mairtin et al. stored PA12/carbon fiber composites in *Skydrol* for 500 h at RT and, apart from a slight increase, found no significant effect on tensile and compressive properties.[7] Sala et al. performed static tensile, compression and dynamic compressive Woehler tests on epoxy/aramid fiber composites aged in *Skydrol* at 60°C for 30 days.[8] The tensile modulus decreased to 89.6% and the compressive modulus to 82.6% of the initial values. The tensile strength decreased to 78.4% and the compressive strength to 60.4%. The strong effect on compressive properties could be explained by the sensitivity of the matrix in this test. Markatos et al. mixed *Skydrol* with water (50:50) to investigate the effect of the formed phosphoric acid on the epoxy-based composite HexPly®M32/T700 for 4 weeks at 70°C.[9] After that, a weight increase of 1.5% and a decrease in fracture energy G_{IC} of 27% were observed. In open loop circuits hydraulic fluids in aircrafts must withstand an operating temperature of 60–90°C under normal conditions. In the worst-case, this temperature can exceed 180°C for a short period of time due to the limited heat exchanger capacity.[10] For *Skydrol* a maximum long-term operating temperature of 120°C is specified, beyond which significant degradation of the hydraulic fluid occurs. In contrast to most publications where the maximum aging temperature is 70°C, the authors are not aware of any investigations carried out at higher temperatures approaching 120°C. Considering that material properties can change dramatically at elevated temperatures due to potential nonlinear behavior, this article aims to fill this gap by presenting a detailed study of the effect of *Skydrol* on a high-T_g epoxy-anhydride resin system in toughened and unmodified state at 120 °C on thermal and fracture properties. A polyester diol was chosen as toughness modifier because of its low impact on modulus and strength combined with good processability for fiber-reinforced composites. This determines the suitability of a material for many structural applications that may come into contact with Skydrol or for usage in hydraulic systems, which also need to have mechanical and thermal performance at the same time.

2 | EXPERIMENTAL

2.1 | Materials and sample preparation

The epoxy resin used in this work was a cycloaliphatic epoxy Celloxide 2021P (Daicel Corporation, Japan) and nadic methyl anhydride (Dixie Chemical, USA) as a hardener. Resin and hardener were mixed in a weight ratio of 100:123 corresponding to a stoichiometric ratio of epoxy to anhydride groups of 100:85. This ratio is considered optimal in literature to obtain the best thermal and mechanical properties.[11,12] The accelerators used were 1-methylimidazole (0.5 wt%) and a latent imidazole accelerator (Technicure LC80, A&C Catalysts, USA). The toughener used in this work was a thermoplastic reactive adipate-based polyester diol (Placcel 220 EB, Daicel Corporation, Japan) with a content of 25% on the epoxy resin with a OH value of 56.8 and a molecular weight of 2000 g/mol. All components were thoroughly mixed with a dissolver and degassed before casting in steel molds. All chemicals were used as received. The cure cycle was 30 min at 100°C, 2 h at 130°C and 1 h at 180°C. To ensure complete cure, unless otherwise stated, an additional tempering step at 220°C for 3 h was carried out. The compositions of the two formulations are listed in Table 1.

TABLE 1 Composition of unmodified and toughened system

Component	Unmodified system	Toughened system
Celloxide 2021P	100	100
Nadic methyl anhydride	123	123
1-methyl Imidazole	0.5	0.5
Latent accelerator	1.5	1.5
Placcel 220 EB	0	25

2.2 | Aging methods

The samples were weighed before immersion, put in *Skydrol* 500-B4 filled metal cans and sealed tightly afterwards. The cans were stored at 120°C in a convection oven for 1000 h. A fine metal mesh was used to separate the samples and to ensure continuous contact with *Skydrol* over the entire sample surface. After 500 h and 1000 h the samples were removed, cleansed from *Skydrol* excess and weighed to check possible weight loss or weight gain.

2.3 | Material characterization

2.3.1 | Dynamic mechanical analysis

Dynamic mechanical properties were measured according to DIN EN ISO 6721-1 using a Rheometer RDA III (Rheometric Scientific) with a heat ramp of 3 K/min, frequency of 1 Hz and strain of 0.1% with sample size of $50 \times 10 \times 2\ \text{mm}^3$. T_g was taken as the peak of the loss factor peak.

2.3.2 | Differential scanning calorimetry

Differential scanning calorimetry was used to investigate the influence of the annealing step, determine the exothermic energy and ensure complete cure of the samples, they were scanned at 10 K/min in a Mettler Toledo DSC1.

2.3.3 | Fourier-transform infrared spectroscopy

FT-IR spectra were acquired using a ATR-FT-IR Nicolet Nexus 470 spectrometer in the range of 400 to 4000 cm^{-1}. The spectra were obtained using 32 scans at a resolution of 1.25 cm^{-1}.

2.3.4 | Scanning electron microscopy analysis

Scanning electron microscope (Zeiss Leo 1530) was used to analyze the fracture surfaces of the virgin and aged resin formulations. The fracture surfaces were coated with a thin layer of platinum prior to SEM observation. The EDX detector Oxford 6901 was used to identify oxygen and phosphorous content.

2.3.5 | Fracture toughness and fatigue crack propagation

Fracture toughness testing was carried out by using the compact tension (CT) method according to ASTM E-394-81 using a Zwick universal tester. Prior to testing, specimens were 'pre-cracked' by inserting a razor blade into the machined notch. The specimens were then placed into a jig and tested at a cross-head speed of 1 mm/min and a pre-force of 2 N. After fracture, the exact crack length was measured from the fracture surfaces and the fracture toughness at crack initiation, and the critical stress intensity factor, K_{IC} was calculated by using the following equation:

$$K_{\mathrm{IC}} = \frac{F_m}{t\sqrt{w}} \frac{a}{w}, \tag{1}$$

where F_m is the maximum load at failure, t is the specimen thickness and (a/w) a geometry factor according to ISO 13586. Critical energy release rate G_{IC} was determined by using Equation 2:

$$G_{\mathrm{IC}} = \frac{K_{\mathrm{IC}}}{E_{\mathrm{SH}}} \left(1 - \nu^2\right), \tag{2}$$

where E_{SH} is the modulus of elasticity determined after the method of Saxena and Hudak using an extensometer mounted on the specimen front, and ν is the Poisson ratio set to 0.35 for the epoxy resin. Fatigue crack propagation (FCP) tests were performed at 23 °C and 50% relative humidity, with a computer-controlled servo-hydraulic test machine (Hydropuls MHF, Schenck) based on ISO 15850/ASTM E647. Compact-tension (CT) specimens with 41.2 mm length and thickness of greater than 3.5 mm were used for the FCP testing. A sharp crack is initiated by loading the mounted specimen with a low sinusoidal load. At least three specimens were tested for reproducibility. A detailed overview about the method and calculations can be found in the publication of Kothmann et al.[13]

3 | RESULTS AND DISCUSSION

3.1 | Thermal properties

The heat of reaction was determined by dynamic heating to 300 °C and a value of 258 J/g was obtained for the unmodified and 218 J/g for the toughened system. This 15% decrease in curing enthalpy is in good agreement with the inert toughener content of 10% in the overall formulation. Without the temper step only small residual energies of about 10 J/g (which represents around 4% of total energy) were found as can be seen in Figure 1. However, applying the temper step of 3 h at 220°C led to complete cure of the material and a significant increase in the glass transition temperature from 220 °C to about 255 °C. This is confirmed in literature where the most significant T_g increase occurs in the later stages of curing. Only a small drop in T_g from 259 to 254 °C is visible after addition of the toughener.

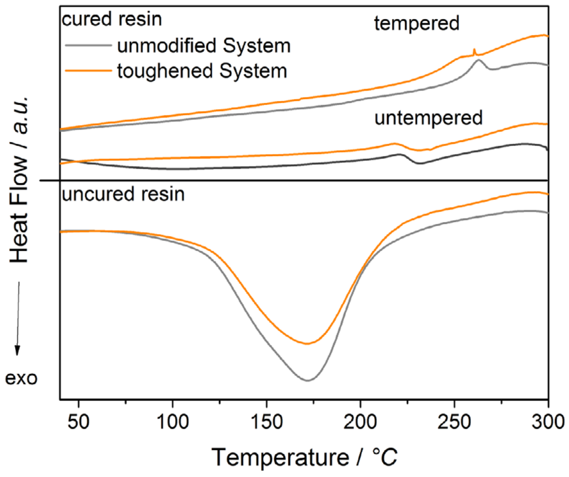

FIGURE 1 DSC curves to detect curing enthalpy and influence of temper step on T_g and degree of cure. [Color figure can be viewed at wileyonlinelibrary.com]

To avoid confusion between post-crosslinking and real aging mechanisms only fully-tempered samples were stored in Skydrol. After 1000 h, no significant weight change could be detected. This is in agreement with the available literature where uptake of hydrophobic materials such as automatic transmission fluid (ATF) into epoxy resins is only slightly observed at immersion temperatures below T_g.[6,14]

3.2 | FT-IR spectroscopy and EDX

FT-IR surface measurements were made (see Figure 2) with the signal normalized to the peak at 790 cm^{-1} associated with the oxirane group of cycloaliphatic epoxy resins to make the intensities comparable, as complete cure was assumed.[15] Since immersion in *Skydrol* effectively blocked oxidation of the material by oxygen, the carbonyl group at 1730 cm^{-1} showed no significant increase in signal intensity. The higher absorption at 2900 cm^{-1} and a shift from a CH_2 to a CH_3 stretching peak indicates chain scission.[16] The ether bond signal at 1100 cm^{-1} remained nearly constant throughout the aging time due to the rather high activation energy of ether bond dissociation with 350 to 389 kJ/mol. In contrast, the C-C bond dissociation energy is between 284–368 kJ/mol in aliphatic structures.[17,18] The shoulder around 3300–3500 cm^{-1} may be assigned to R-OH stretching, based on the initial toughener and further degradation of the matrix and toughener leading to more free hydroxyl groups.

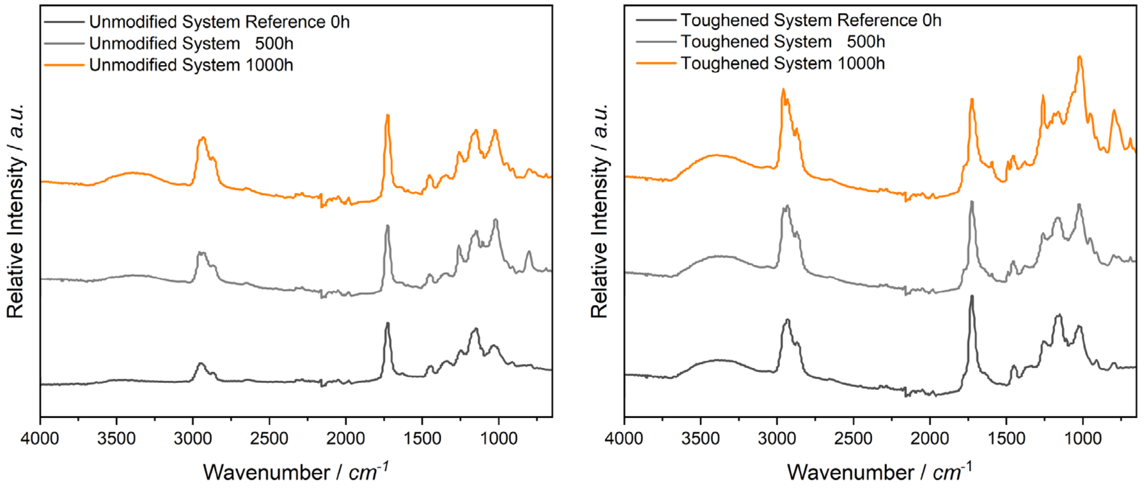

FIGURE 2 FT-IR spectrum of unmodified system and toughened system. [Color figure can be viewed at wileyonlinelibrary.com]

EDX linescan measurements (see Figure 3) with focus on the elements oxygen and phosphorus were made across the width of the CT-specimen to determine possible oxidation and *Skydrol* uptake. It can be seen that no significant oxidation occured as the oxygen counts remained fairly constant and no difference can be seen between the reference and the 1000 h Skydrol aged sample. However, higher phosphorous counts up to 75 μm are detectable. Since *Skydrol* is phosphate ester based, the phosphorous counts can be directly linked to *Skydrol* uptake. This gradient, although small compared to the overall width of 4 mm, can lead to a significant impact on thermomechanical properties, similar to an oxidized layer.[19]

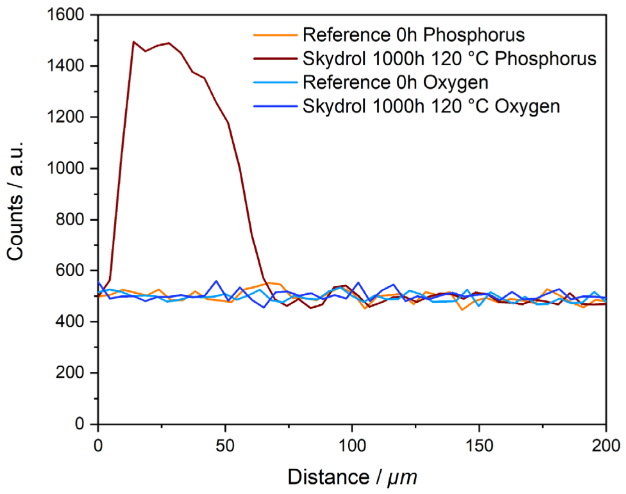

FIGURE 3 EDX counts of oxygen and phosphorus starting from edge of a 1000 h in Skydrol immersed CT-specimen, exemplarily shown for the unmodified system. [Color figure can be viewed at wileyonlinelibrary.com]

3.3 | Dynamic mechanical analysis

DMA measurements and equation (3) were used to calculate the apparent crosslink density of the formulations which was derived from the classical theory of rubber elasticity:

$$\nu = \frac{G'}{3RT} \tag{3}$$

where G′ is the storage modulus in the rubber plateau at temperature T, R is the gas constant and T is the temperature 20 °C above T_g. The use of toughener decreased the crosslink density by about 25% from 1796 to 1364 mol/m^3. Compared to other crosslink density values reported in the literature (e.g., the tri-functional epoxy TGMDA cured with HHPA anhydride with a T_g of 231 °C and a network density of 3886 mol/m^3) the values are quite low.[20] First, this can be explained on the one hand, by the fact that the bi-functional cycloaliphatic epoxy and the rigid structure of the nadic acid methyl anhydride lead to higher T_g values with lower crosslink density compared to other epoxy resins or amine hardeners. In addition, the polyester diol toughener used acts as an intrinsic chain extender which reduces the crosslink density (see Table 2). In the epoxy resin studied, the addition of polyester diols showed promising results improving the fracture toughness without significantly affecting thermal properties such as T_g.[20–22]

Dynamic-mechanical analysis of the aged specimens was performed as it is considered one of the most powerful techniques revealing early material degradation.[23] Typical aging phenomena such as oxidation and chain scission can be directly seen from the changes in storage modulus and loss factor tan (δ). Chain scission leads to a broader molecular weight distribution and a more heterogeneous network as evidenced by broader tan(δ) peaks.[19] Significant degradation of the network leads to a drop in T_g. The results for immersion in *Skydrol* are shown in Figure 4. Although the storage moduli are comparable at RT, the toughened system shows a continuous decrease early on until T_g. For the aged samples of both systems, a decrease in storage modulus is visible at temperatures below and above the T_g, indicating network degradation. This is more pronounced in the toughened system especially from 150 °C onwards. The T_g for both systems changed only slightly, decreasing by 4 °C for the toughened system, while the unmodified system showed even a slight increase.

TABLE 2 Crosslink density and T_g of unmodified and toughened systems during aging in *Skydrol*

	Unmodified system		**Toughened system**	
	Crosslink density (mol/m^3)	**T_g (°C)**	**Crosslink density (mol/m^3)**	**T_g (°C)**
Reference 0 h	1796	260.2	1364	250.0
500 h	1621	264.4	1065	246.3
1000 h	1493	264.2	961	248.0

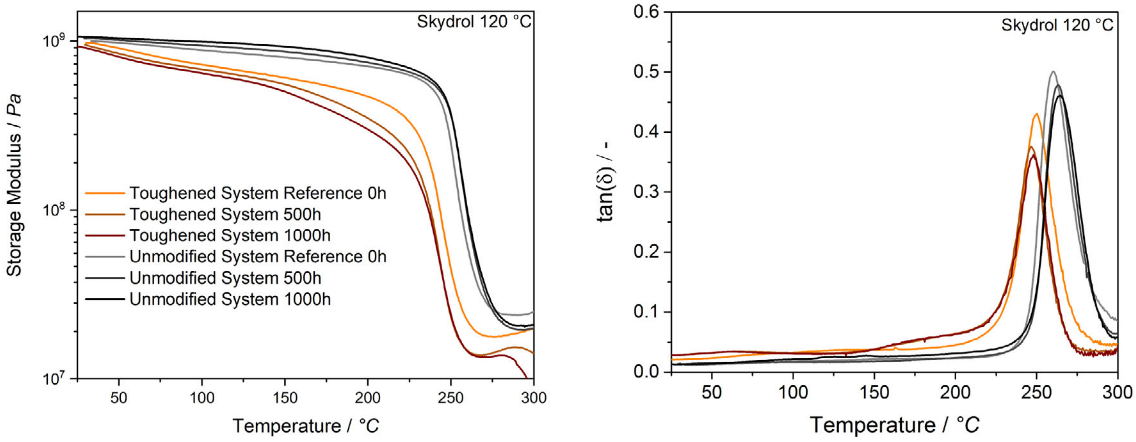

FIGURE 4 Storage modulus (left) and loss factor (right) of unmodified and toughened systems. [Color figure can be viewed at wileyonlinelibrary.com]

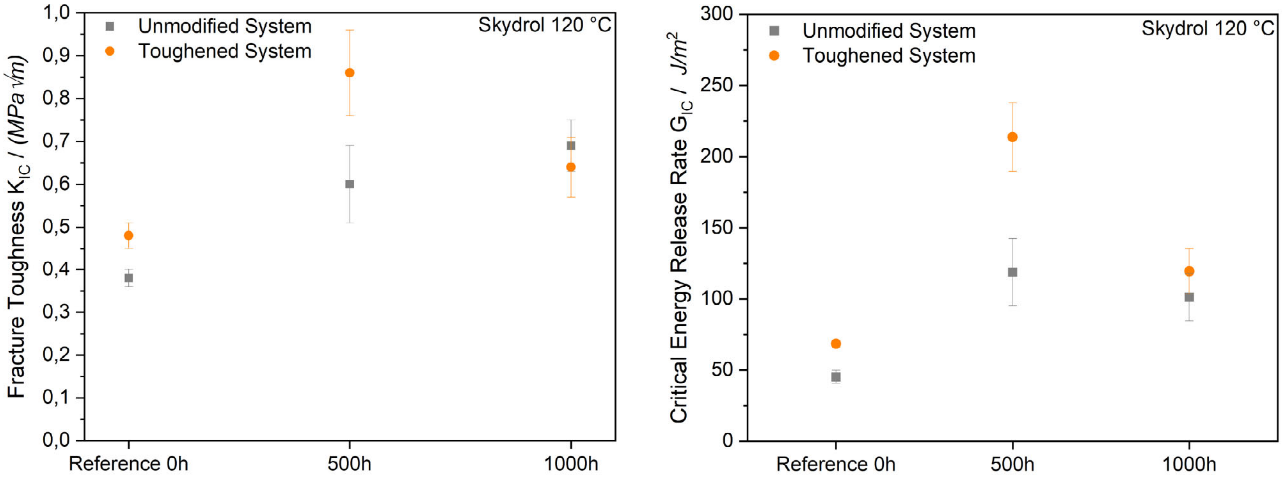

FIGURE 5 Influence of toughener and Skydrol immersion on K_{IC} and G_{IC}. [Color figure can be viewed at wileyonlinelibrary.com]

In general, the toughened system was more affected by immersion in *Skydrol* at 120 °C, as the crosslink density dropped to 70% of the original value after 1000 h. The unmodified system showed better aging resistance at 83% of its initial value. It appears that the aliphatic polyester diol toughener has lower thermal stability resulting in faster and more pronounced network degradation. However both resin systems showed only comparatively minor changes due to degradation in terms of storage modulus or tan(δ) shift.

3.4 | Fracture toughness

The influence of the toughener content and *Skydrol* immersion on fracture toughness is shown in Figure 5. The incorporation of the toughener caused an improvement in toughness of about 26% from 0.38 to 0.48 MPa$^{0.5}$. The introduction of polyester into the epoxy networks resulted in a more ductile fracture with flexible ether linkages and reduction of crosslink density.[24,25] This was confirmed in the literature where Ratna et al. used carboxyl-terminated poly(ethylene glycol) adipate (CTPEGA) in DGEBA resin resulting in a dramatic 100% improvement in toughness at 10% additive, but at the cost of a 20% reduction in modulus. A similar effect was shown by Mafi et al. by varying the crosslink density through the molecular weight of the resin.[26] Critical energy release rate G_{IC} of the neat resin increased over 50%, extending the molecular weight between the crosslinks from 1000 to 4000 g/mol.

For both systems an increase in fracture toughness of up to 50% is observed with 500 h *Skydrol* immersion. After 1000 h a decrease is seen for the toughened and another slight increase for the unmodified system, both converging to a value of about 0.65 MPa$^{0.5}$ which is still above the initial reference values. This can be explained by the reduction of the crosslink density and higher mobility of the polymer chains, which leads to an improved ductility of the resin and thus to a more homogeneous shear yielding allowing better energy dissipation before fracture.

The pronounced effect of the crosslink density of the neat epoxy resin on the fracture toughness is already known from previous experiments, were the change of the molecular weight of a polyether amine from 230 to 400 g/mol (Huntsman Jeffamine D230 or D400 respectively) in combination with TGMDA epoxy resin led to an decrease in crosslink density from 1533 to 1015 mol/kg (reduction of 33%) and subsequently in an increase of the fracture toughness from 0.48 to 2.02 (increase of 320%).[27] An analogous behavior can be seen for the critical energy release rate G_{IC} in the context of measurement accuracy. It can be assumed that the decrease in crosslink density leads to a more ductile behavior for the unmodified system, while the toughened system initially became tougher with immersion but ultimately deteriorated to the point where the crosslink density exceeded a lower limit, which in turn led to decrease in fracture toughness. The SEM analysis shows a smooth surface for the unmodified system with only small amounts of localized plastic shear bands visible (see Figure 6).

In contrast, the toughened system has more shear bands indicating higher ductility and toughness. Since the diol toughener has a low molecular weight of 2000 g/mol and a low content of 10%, no phase separation is visible as reported in the literature.[28] Higher shear yielding is visible for the toughened reference systems after 1000 h *Skydrol* immersion.

3.5 | Fatigue crack propagation

The fatigue crack growth behavior can be divided into three distinct phases. in the first phase, the initiation of the crack can be described by the threshold value(ΔK_{th}). In the second phase, stable crack growth propagation occurs, commonly referred to as Paris-Erdogan regime, followed by the third phase with instable crack growth described by ΔK_{cf}. The effect of the toughener on FCP can be seen in Figure 7. Despite the high T_g and thus the high network density, improved crack resistance with addition of toughener is visible. The threshold value of crack propagation (ΔK_{th}) and for critical failure (ΔK_{cf})

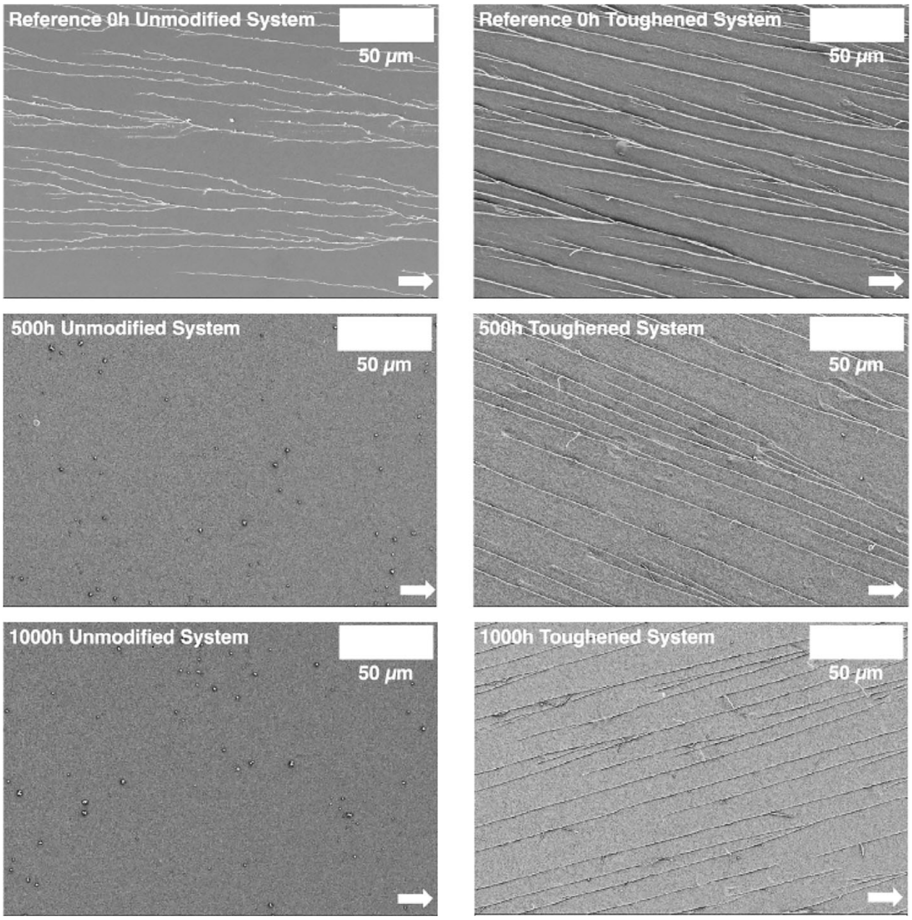

FIGURE 6 SEM images of the fracture surfaces of the toughened and unmodified system before and after immersion in Skydrol. The white arrow shows the direction of crack propagation.

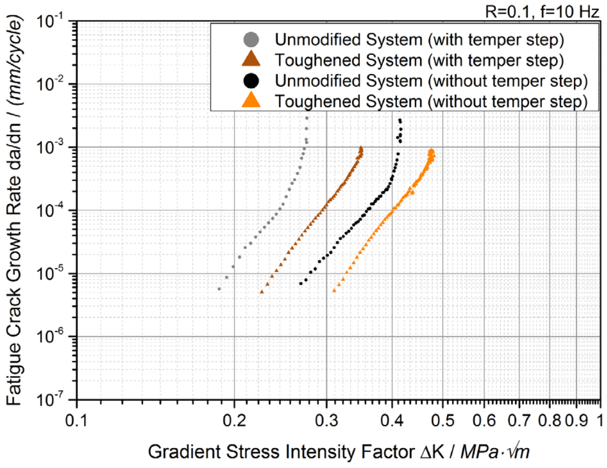

FIGURE 7 Influence of temper step on the fatigue crack propagation behavior. [Color figure can be viewed at wileyonlinelibrary.com]

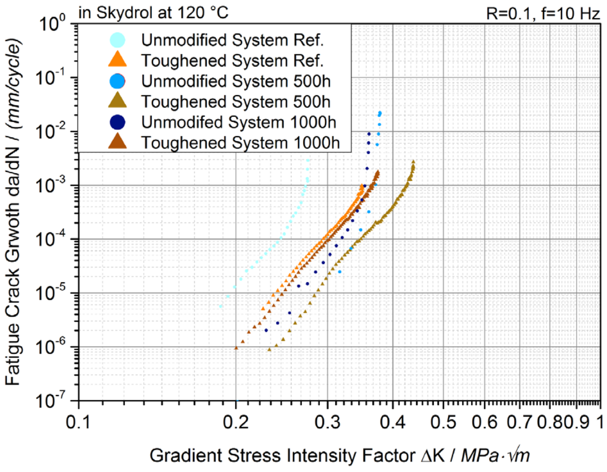

FIGURE 8 Influence of *Skydrol* immersion at 120°C on the fatigue crack propagation. [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 3 Overview of threshold value (ΔK_{th}) and for critical failure (ΔK_{cf}) of fatigue crack propagation measurements

	Unmodified system		Toughened system	
	ΔK_{th}	ΔK_{cf}	ΔK_{th}	ΔK_{cf}
Reference 0 h	0.18	0.27	0.22	0.34
500 h	0.31	0.37	0.23	0.43
1000 h	0.22	0.36	0.20	0.37

both increase by approximately 23%. Comparing the tempered with the untempered sample a decrease of 25% for the threshold is visible. The values for critical failure for the tempered system are in good agreement with static K_{IC} measurements. For the critical failure value, the unmodified system showed a higher sensitivity to the temper treatment with a decrease of 35% compared to 25% observed for the toughened system. Faster crack propagation is visible for the unmodified system especially near the critical failure, indicating lower fracture resistance at higher loads.

Similar to the static fracture toughness measurements, an increase in FCP resistance is visible after storage in *Skydrol* (see Figure 8). After 500 h the toughening effect due to degradation is more pronounced for the unmodified system with an increase in $K_{\max}$ of about 40%, whereas the toughened system only increased around 13%. After 1000 h no significant difference is observed between the toughened and the unmodified system with both reaching a $K_{\max}$ of around 0.37, which is still higher than the initial values. Table 3 gives an overview of the threshold and critical failure values for the reference and aged systems.

Therefore, the addition of polyester diol toughener can be considered beneficial only at modest temperatures and at the beginning of the material's life, since it's faster deterioration with increasing immersion time at higher temperatures leads to an equalization between the different systems. Here, the polyester diol toughener seems to affect the overall performance over aging time. This can be linked to the lower thermal stability of the ester bonds, which at a certain point significantly degrade the network structure. In this context, other toughener for resin systems with better thermal stability, such as polysulfones, might be worth investigating.

4 | CONCLUSION AND OUTLOOK

In this study the influence of *Skydrol* hydraulic fluid on the degradation behavior of a high-T_g anhydride-epoxy resin at elevated temperatures was determined. In addition, the influence of polyester diol toughener on the thermo-mechanical behavior was investigated. The glass transition temperature remains largely unaffected by the incorporation of toughener despite a decrease in crosslink density leading to a higher fracture toughness. By using a temper step, fully cured samples with high T_g can be achieved at the expense of increased brittleness. After immersion in *Skydrol*, only minor effects on the thermo-mechanical properties were observed, as the fluid uptake was not significant, whereas crosslink density decrease was more pronounced for the toughened system. Using EDX microscopy, a diffusion of up to 75 μm of *Skydrol* inside the specimen were observed. The decrease in crosslink density, determined by DMA measurements, due to thermal degradation led to an initially higher fracture toughness as more shear yielding was possible. Here the maximum was reached at 500 h immersion, and a decrease was observed at 1000 h. Similar results were obtained for FCP measurements. The results suggest that the addition of this intrinsic toughener in terms of service life would be futile in a challenging environment due to faster degradation and chain scission of the network. These results and the fact that the investigated material system yields a high T_g of up to 255°, this resin could be suitable for high-temperature structural applications or use in hydraulic systems. However, before the material is considered for use, the issue of oxidation stability needs to be addressed. This will require further studies on long-term thermal and oxidation stability for example, in air. In addition, other potentially high-temperature stable toughener such as polyether sulfone (PES) are worth testing in combination with the developed resin system.

AUTHOR CONTRIBUTIONS

Jan David Hübsch: Investigation (equal); writing – original draft (equal). **Martin Demleitner:** Investigation (equal); writing – original draft (equal). **Simon Bard:** Investigation (supporting); writing – original draft (supporting). **Philipp Berendes:** Funding acquisition (lead); project administration (equal); writing – review and editing (supporting). **Volker Altstädt:** Project administration (lead); supervision (lead); writing – review and editing (lead). **Christian Mittelstedt:** Conceptualization (lead); writing – review and editing (supporting).

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CONFLICT OF INTEREST

There are no conflicts of interest associated with this publication.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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