

THERMAL ANALYSIS OF A UNIAXIAL CARBON FABRIC REINFORCED SILICONE RESIN

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Abstract

This study, which focuses on carbon fibre (CF) reinforced silicone matrix composites (CFRS-C), an alternative to conventional polymer matrix composites, aims to understand their thermal properties better. Silicone elastomer's high elastic deformation capability is the primary factor in the choice of silicone matrix. The aforementioned materials are particularly appealing in the design of deployable systems used in the aircraft sector due to their folding capability.

Better adhesion of silicone elastomer and plain weave carbon fibre fabric was succeeded by modifying the surface of the fabrics inside 20 wt.% nitric acid solution. Thermal Gravimetry (TG) and Differential Scanning Calorimetry (DSC) were used to examine the materials' thermal properties in this study's scope. Thermal tests showed that composite materials exhibited thermal resistance up to 368 °C. The silicone components utilized in the study can form carbon fibre-reinforced composites that exhibit thermal stability up to the specified temperature. The use of carbon fibre enhanced the thermal stability of silicone elastomers. Additionally, a change in the number of layers impacts the composite's thermal stability.

Key words: silicone elastomer, carbon fibre, thermal gravimetry, differential scanning calorimetry, thermal behaviour, surface modification

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Introduction. Thermosetting resins such as epoxy, polyester, and vinyl ester, mostly preferred today, exhibit a rigid behaviour after curing. Since the carbon fibres embedded in the rigid matrix do not have mobility when the composite material is bent and twisted to fold, excessive stress concentration occurs on and around the carbon fibres, and the material gets damaged before it reaches the folding degree [1,2]. This phenomenon led to the decision to mix the fibres with silicone elastomers, a more flexible substance. Fibres embedded in the silicone elastomer can show a high degree of elastic deformation ability with the application of low stress; even if they are folded with a high curvature diameter, this ability of the silicone will allow them to micro-buckle undamaged [3–5]. The usage of silicone as a matrix material has been employed in a variety of investigations. In a study by DATASHVILI et al. [6] at Munich Technical University, a composite reflector antenna design and scaled version were produced using silicon reinforced with carbon fibre material with a triaxial woven $[60^\circ/0^\circ/-60^\circ]$ structure. VOCKE III et al. [7] utilized silicon matrix composites in a study to change the one-dimensional wing length of an aircraft. It is possible to create composite materials with significant deformation capacity without deformation by adapting elastomer materials with high deformation capabilities as the matrix material.

As the designed materials are subjected to higher degrees of temperature, their thermal behaviour needs to be investigated. VAIMAKIS-TSOGLAS et al. [8] demonstrated that the addition of 5 wt.% TiO_2 in silicone elastomers enhanced the thermal degradation temperature of silicone elastomer films by 20°C . According to the findings of OGLIANI et al. [9], silicone elastomers thermally deteriorate in a hierarchical pattern depending on the degree of PDMS chain mobility. Strongly cross-linked networks have worse thermal stability than networks with lower cross-linking density. In another study, Al_2O_3 fibres with 2 nm diameter and 100 nm length were utilized by YAO et al. [10] to enhance silicone's thermal stability. They improved thermal degradation from 330°C to 370°C by 30 wt.% fibre reinforcement. KHOSLA et al. [11] blended 50–250 μm length CF into polydimethylsiloxane with various weight percentages, and the thermal stability of their composite improved according to their TGA analysis results.

Our aim in this study was to see how the thermal behaviour of our CF fabric reinforced composite materials, which became foldable thanks to silicone matrix, changed. Because although there are studies with nanoparticle reinforcement in the literature, not many studies have been found on the thermal behaviour of silicon elastomers reinforced with long fibres.

Materials and method. Two brands of silicone resin with trade names TSE3488T and RTV 830 were employed as matrix materials. Tenax-E HTA 40 3k CF fabric was used to reinforce silicone elastomers. This fabric is plain weave, 200 gr/m^2 , the diameter of fibres is $7\text{ }\mu\text{m}$, and its density is 1.76 g/cm^3 . The main chains in the structure of silicone polymers contain siloxane (Si-O) bonds. Other organic groups like polydimethylsiloxanes attach to the main chain, creating a 3D

layout after cross-linking reactions [12]. For the production of composites, the base compound TSE3488T(A) was mixed with 10 wt.% curing agent TSE3488T(C). The mixing ratio for base compound RTV830(A) and curing agent RTV830(B) was the same; 10:1. For better impregnation through the carbon fibre fabric, the viscosity of the matrix resins was decreased by mixing 30 wt.% xylene. The surface of the fibres forming the fabric is coated with epoxy solution during the production of fibres to be more compatible with epoxy, acrylic, polyester, and vinyl ester resins. Since it was observed that the epoxy-coated surface of fibres and the silicone elastomer could not provide good adhesion, the production of composite material was achieved with a better interfacial adhesion after the carbon fibres were heat treated in 20 wt.% nitric acid solution at 110 °C for 60 min. The impregnated silicone was cured at 150 °C under 2.3 bar pressure for 30 min. Structurally, three types of composite materials were produced: single, double, and 4-layer. The detailed production steps of composites were described elsewhere [13]. The DSC and TG analysis of composites were made with NETZSCH-Jupiter STA-449 (Fig. 1). The matrix and fibre components of the composites were also analyzed. DSC and TG analyses were carried out at 1000 °C with a heating rate of 10 °C/min. Single, double, and 4-layer composites with TSE3488T matrix were labelled as T1, T2, and T4, respectively. Similarly, the ones with RTV 830 were named R1 for single, R2 for double, and R4 for 4-layer composites.

Results and discussion. DSC and TG graphs of TSE3488T and RTV 830 matrix materials and carbon fibre reinforcement are superimposed in Fig. 2.

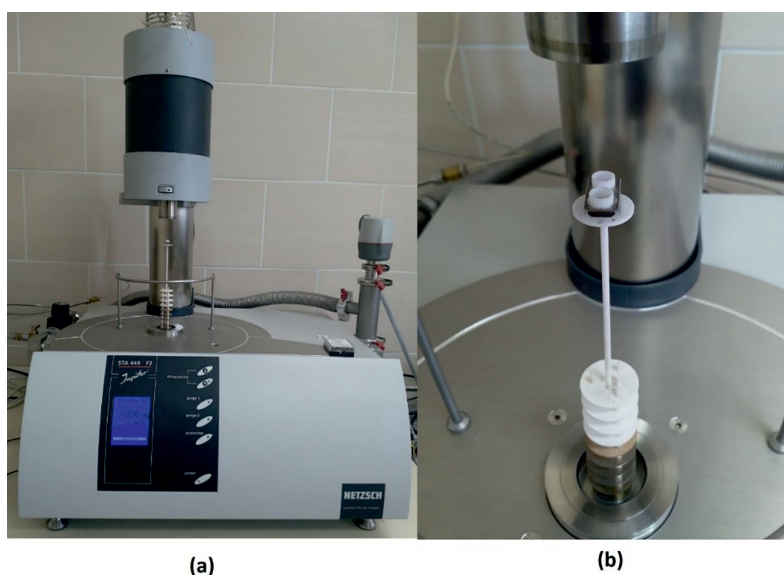


Fig. 1. NETZSCH Jupiter STA-449 DSC-TG Analyzer. (a) general view of the equipment, (b) the crucibles in which the analyzed and the reference sample were placed

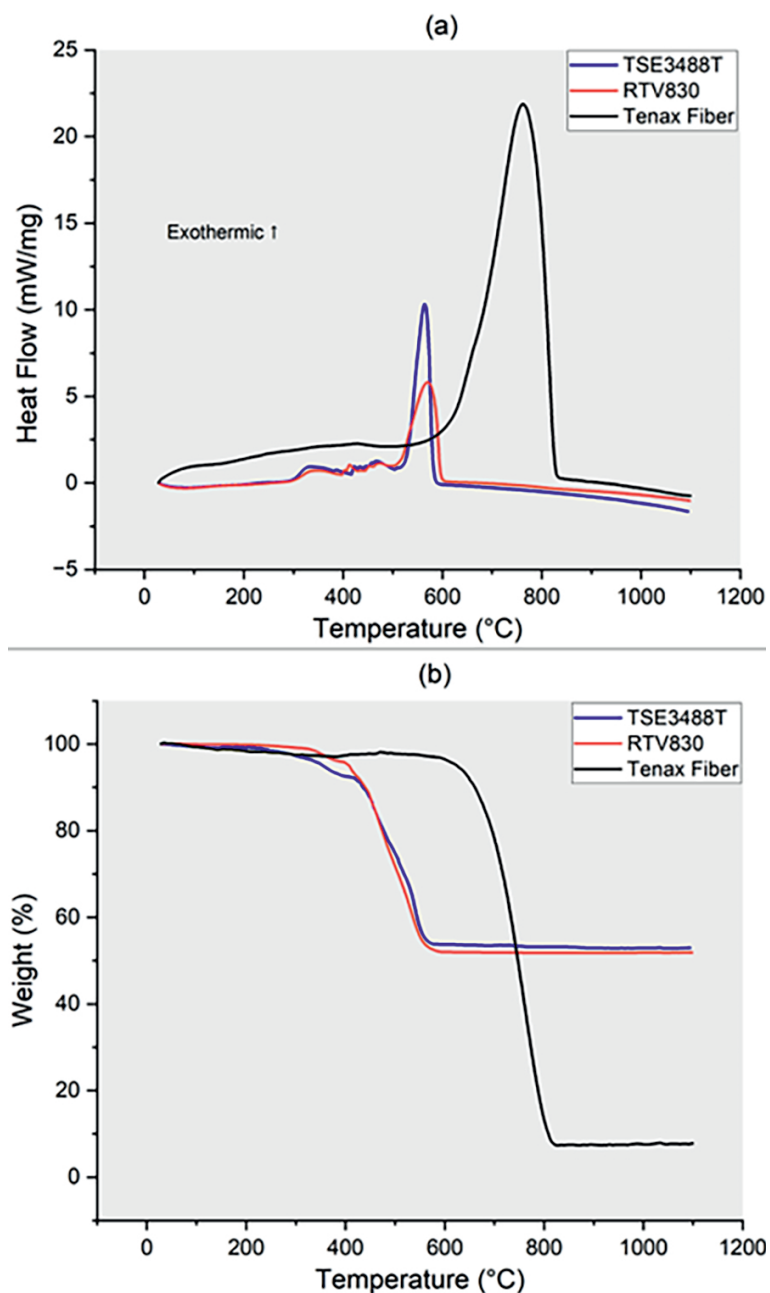


Fig. 2. (a) DSC and (b) TG curves of the components of composite materials

Looking at the TG analysis of carbon fibres in Fig. 2b, a mass loss of about 2% is observed between 350–550 °C. The reason for this mass loss may be the organic components on the surface of the carbon fibres [14]. Afterwards, the DSC curve of the carbon fibre gives an exothermic peak at ≈ 772 °C.

Looking at each DSC and TG analysis graph, this peak indicates that the carbon fibres were burned entirely at this temperature [15]. The DSC and TG curves for these two silicones show that the thermal degradation of TSE3488T and RTV 830 started at approximately 338 °C and stopped at 600 °C and 613 °C, respectively. The approximate temperature at which silicones show the most significant thermal degradation is 580 °C. Another peak detected in the DSC curves of silicones is related to CO₂ and water, which occurs with the separation of the polymer into oligomers [16]. DSC data in Fig. 2b indicate that the crucial weight loss starting temperature for carbon fibre is 625 °C, for TSE3488T is 354 °C, and for RTV 830 is 408 °C.

According to the data shown in Fig. 3, it is evident that reinforced silicones have a greater decomposition temperature, which rises as the number of layers increases. The thermal stability of TSE3488T silicone and composite materials

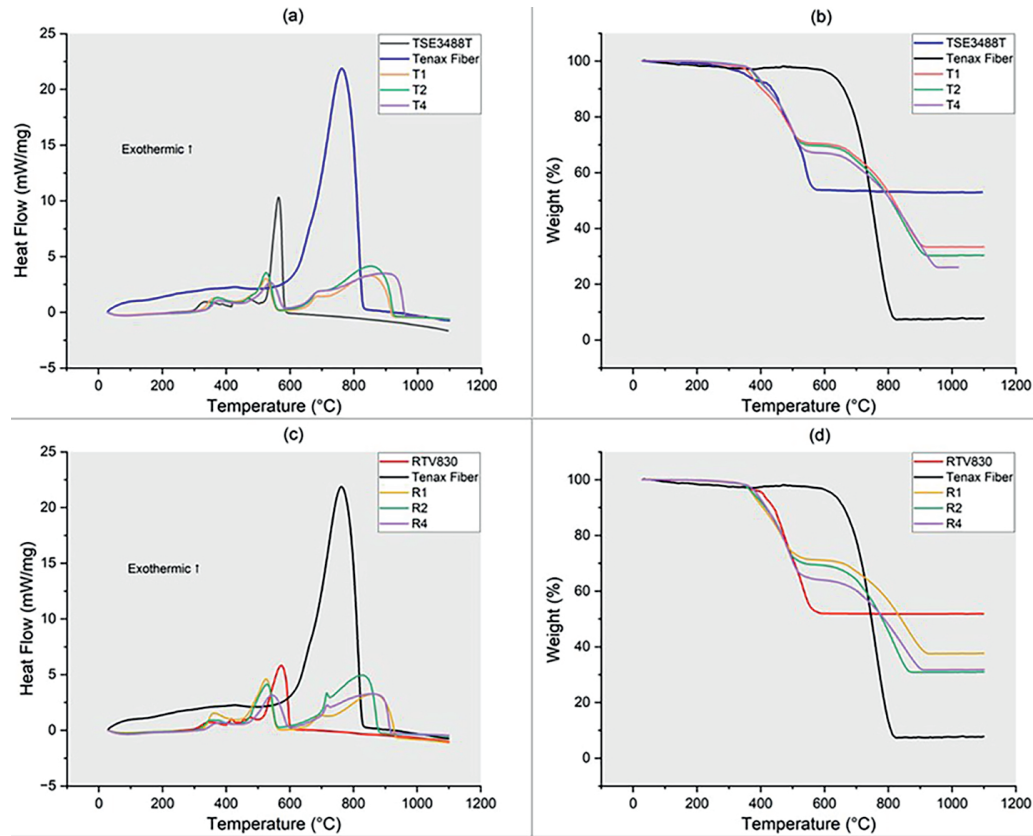


Fig. 3. DSC and TG curves of composite materials. (a) DSC curve of CF reinforced TSE3488T silicone elastomer, (b) TG curve of CF reinforced TSE3488T silicone elastomer, (c) DSC curve of CF reinforced RTV 830 silicone elastomer, (d) TG curve of CF reinforced RTV 830 silicone elastomer. DSC and TG curves of components were superimposed on each graph for better comparison

T a b l e 1

Critical temperatures as a result of DSC and TG analyses of composite materials

Specimen name	Temperatures at which critical mass losses occur (°C)		
	5% loss	10% loss	50% loss
T1	368	403	816
T2	380	417	805
T4	386	421	810
R1	372	405	839
R2	377	415	781
R4	387	417	787

produced with this silicone is high. The thermal behaviour of composites is similar to the thermal behaviour of the matrix materials and is influenced by changes in the number of layers and carbon reinforcement. However, when looking at the curves of all TSE3488T composites, it can be seen that there are extra exothermic peaks around 800 °C. These peaks indicate the temperatures at which the carbon fibre reinforcements started to burn when embedded in a silicone matrix. Figure 3c,d which shows the curves for RTV 830 silicone composites, also presents extra carbon fibre burn peaks.

Table 1 summarizes the thermal behaviour of CF reinforced silicones of composites with various numbers of layers. Specimen T1 had a 5% mass loss at 368 °C. Considering all samples, this is the lowest point where a critical amount of mass loss occurred. Decomposition reactions of composite materials are similar to the matrix material but rise with carbon fibre reinforcing. BUZAROVSKA et al. [17] stated in their study that the rise in decomposition temperature was due to the nanoparticles blocking the heat flow. Plain wave carbon fibre fabric may have created the same effect in our research. And also, SILVA et al. [18] mentioned that in comparison to the unfilled PDMS, the barrier impact of the TiO₂ particles on the diffusion of volatiles may have encouraged the shift of the primary weight loss processes to higher temperatures. Yao et al. [10] claim that the filler-matrix interface may act differently from the matrix during thermal degradation because each filler's layers are firmly and loosely linked, which restricts chain mobility. Restricted chain mobility leads to improved thermal stability [19,20]. The carbon fibres embedded in the silicone before curing may have interacted with the silicone elastomer during the curing process and reduced the molecular chain flexibility after.

Conclusions. In this study, we examined the thermal behaviour of composite materials with a flexible matrix material that we produced in previous studies [13]. The lowest temperature at which the silicone components reach a visible mass loss has been determined as 368 °C. Carbon fibre reinforced composites produced with these silicone components can show thermal stability up to

the mentioned temperature as composites can be under service until the matrix material degrades. Another conclusion of this study is that adding carbon fibre improves silicone elastomers' thermal stability. And the thermal stability of the composite is affected by the change in the number of layers.

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