Polymer-metal interactions and their effect on tool-ply friction of C/PEKK in melt

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Keywords: Thermoplastic Matrix, Tool-Ply Friction, Chemical Bonding, Polymer Degradation

Abstract. Excessive friction or adhesion between tool and surface ply can lead to processing defects during thermoplastic composite manufacturing. A previous study identified a notable increase in friction or adhesion between a mild steel tool surface and C/PEKK with prolonged dwell time at elevated temperature, a phenomenon that could not be explained by our current understanding. This work presents additional friction and differential scanning calorimetry experiments to demonstrate that the increased adhesion results from polymer-metal interactions that lead to polymer degradation and metal-polymer bonding. These findings underline the importance of tool material selection in both processing and characterization of tool-ply friction.

Introduction

High friction or adhesion between tool surface and composite material can lead to fiber waviness defects during the manufacture of thermoplastic composite parts [1–3]. Many research groups have characterized tool-ply friction under processing conditions in an effort to understand and prevent such defect formation. The characterization experiments involve the controlled sliding of a molten thermoplastic composite ply against a representative tool surface [4]. The measured shear stress at the interface typically displays a peak, followed by a steady-state plateau, where the peak and steady-state stress levels depend on temperature, applied normal pressure and sliding rate [5–7].

The mechanisms underlying the observed shear stress response are schematically illustrated in Fig. 1, and include friction, physical and/or chemical bonding, and viscoelastic deformation of the molten polymer. The peak shear stress could be caused by an adhesive bond or static friction between the fibers and the tool, which needs to be overcome before sliding can commence [8, 9]. Additionally, a thin polymer interlayer exists between the fibers and the metal surface, acting as a lubricating film. Here, the evolution of the shear stress is governed by the deformation of the matrix interlayer. The peak stress can then be caused by the nonlinear viscoelasticity of the matrix material [6, 7], a change in the interlayer thickness distribution [5], or by the development of slip at the tool-polymer [5] and/or the fiber-polymer interface [10, 11].

Although the current understanding of these phenomena is well-developed, our previous research [12] brought to light a curious phenomenon: the peak friction between mild steel and Solvay unidirectional C/PEKK significantly increased with dwell time at elevated temperature; an effect that was not observed for ply-ply friction. This unexpected finding challenged our understanding of the governing mechanisms and prompted additional experiments to identify the underlying causes. In this paper, we report results indicating that the increased peak friction is caused by strong polymer-steel bonding and polymer cross-linking, with the latter accelerated by
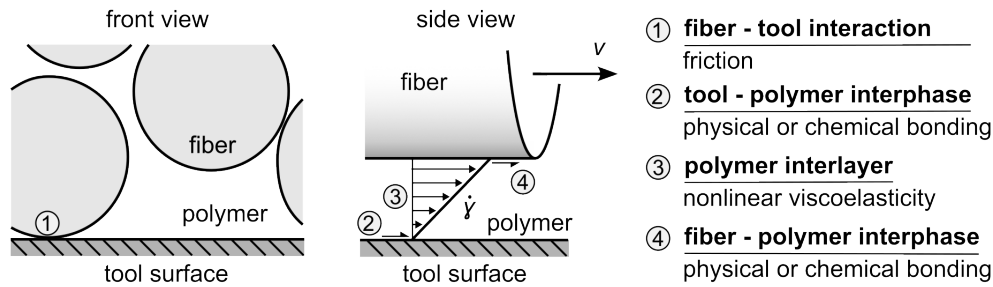
Figure 1. Schematic illustration of the tool-ply interphase, highlighting the mechanisms that influence the friction response.

contact with the mild steel. These findings have implications for tool material choices in manufacturing and friction characterization.

This paper is structured as follows. First, the materials and methods used are described, followed by a brief presentation of the results. In the discussion, we will interpret our findings within the context of the described mechanisms, while the final section presents the conclusions.

Materials and methods

Materials. All experiments were conducted on APC C/PEKK unidirectional tape material from Solvay. This tape has a fiber volume fraction of 59% and a thickness of approximately 0.15 mm, while it exhibits a glass transition temperature of 159 °C and a melting temperature of 337 °C. A few experiments featured a 50 μm thick PEKK film, which according to the supplier has properties comparable to the matrix used in the APC tape.

The tool surface was represented by a non-alloy cold-rolled steel, known as DC01, with a chemical composition as provided in Table 1. The steel was supplied by Georg Martin GmbH as a foil with a thickness of 50 μm. Prior to the experimental work, the foil surface underwent cleaning using isopropanol. In certain experiments, we applied a release agent (Marbocote 227CEE) to the metal foils following the supplier's recommendation, leaving it to cure for 24 hours at room temperature. We also examined the friction between the composite tape material and polyimide films, which are commonly used in industry for facilitating part release. The polyimide film in question has a thickness of 25 μm, is known as Upilex 25S, and was supplied by UBE GmbH.

Table 1. Chemical composition (max weight %) of DC01 steel.

C	Mn	P	S
0.12	0.6	0.045	0.045

Tool-Ply Friction Measurements. The friction between the molten UD tape material and the metal foil or polyimide film was characterized using a well-established friction tester [4]. A tool-ply friction specimen comprises a UD tape, having a width of 50 mm, sandwiched between two metal foils or polyimide films with a width of 55 mm. The overlap between tape and metal foil or polyimide film was 65 mm. We considered the following contact pairs:

- APC vs. mild steel with and without release coating
- APC vs. polyimide film
- APC vs. mild steel with additional PEKK film at the interface

Fig. 2 schematically illustrates the working principle of the friction tester [13]. The tool-ply specimen is placed between two heated press platens, each measuring 50×50 mm², which apply a normal pressure. The ends of the metal foils are clamped, while the UD tape is pulled through and forced to slide against the metal foils. During the measurement, we logged the displacement and the required pull force. The sliding action was terminated after a displacement of approximately 10 mm. The pull force F was used to calculate the shear stress at the tool-ply interface as:
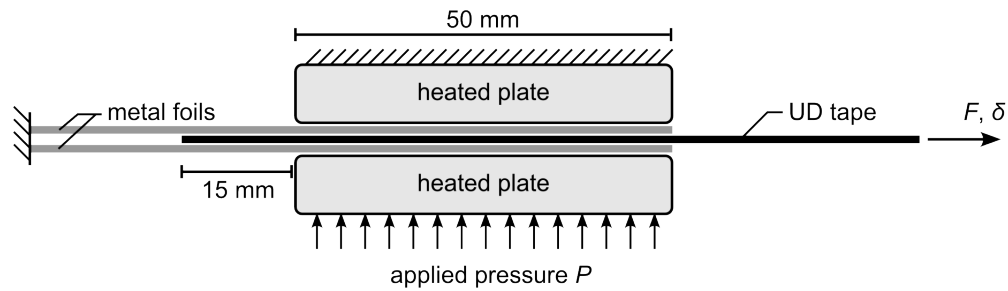
Figure 2. Schematic illustration of the friction test.

$$\tau = \frac{F}{2A},\tag{1}$$

with A as the contact area.

In all friction experiments, we applied a normal pressure of 15 kPa, a sliding rate of 25 mm/min and a platen temperature of 385 °C. The dwell time between closing of the pressure platens and initiating sliding motion was varied between 2.5, 5, and 10 minutes.

Differential Scanning Calorimetry. We conducted differential scanning calorimetry (DSC) experiments, using a TA Instruments Discovery 250, to assess potential chemical changes in the tape materials resulting from the friction experiments. To this end, small pieces of tape material, with a total mass of 8 to 10 mg, were extracted from the APC friction specimens after testing. These DSC samples were then subjected to the following thermal cycle in a nitrogen atmosphere:
1. The samples were heated to 385 °C a rate of 20 °C/min;
2. Following the heating phase, an isothermal segment was maintained for 5 minutes;
3. Finally, the samples were cooled back to room temperature at a rate of 20 °C/min.

Using a heat of fusion of 130 J/g for the matrix, we determined the degree of crystallinity from the crystallization peak measured in the cooling traces. The degradation of PAEKs involves chain scission at the ether or carbonyl linkage, followed by branching or cross-linking [14, 15]. These latter two processes reduce chain mobility, increasing the polymer's viscosity and inhibiting its ability to crystallize [15]. In this work, we utilized the degree of crystallinity as an indicator for polymer degradation.

Differential scanning calorimetry experiments were also conducted on the as-received PEKK film and APC tape material. Four sample pans were prepared, each containing a different combination: one with small pieces of PEKK film, another with a mix of PEKK film and metal foil, a third with pieces of the APC tape, and a fourth with a mix of the APC tape and metal foil pieces. The goal of these experiments was to investigate the influence of the metal foil on the degradation of the matrix material. The samples were first subjected to a 10-minute dwell at elevated temperature under nitrogen atmosphere in the DSC equipment, followed by the same analysis step as outlined above.

Scanning Electron Microscopy. Scanning electron microscopy (SEM) images were captured to examine the bonding between the APC tape and the metal foil after a 10-minute dwell. The metal foil was peeled by hand from the APC tape, requiring substantial force. Subsequently, SEM images were obtained using a Jeol 7200F scanning electron microscope. Additionally, the tape surface was analyzed using Energy Dispersive X-ray (EDX) spectroscopy, using detectors from Oxford Instruments, operating at an acceleration voltage of 10 kV.

Results

Tool-Ply Friction. Fig. 3 illustrates the interfacial shear stress evolution as a function of displacement for all contact pairs considered. The curves for APC in contact with the mild steel foils, shown in Fig. 3a-b, display an initial peak at the start of the test, followed by a subsequent

decrease to a plateau value. Notably, the peak shear stress increases with longer dwell times, reaching levels exceeding the maximum measurable capacity of 200 kPa for metal foils without release agent. Importantly, these results align with our earlier findings [12]. In contrast, the friction response for APC against the polyimide release film, shown in Fig. 3c, does not exhibit an initial peak, and the overall stress evolution also does not show any significant dependency on dwell time.

Fig. 3d shows the shear stress evolution for APC against steel with the PEKK film inserted at the tool-ply interface. Compared to the corresponding case without film, shown in Fig. 3a, both the peak and steady-state stress levels have reduced significantly. Further, although the peak stress increases with dwell time, the increase is not as dramatic as observed in the case without PEKK film, while the steady-state shear stress now also shows an increase with dwell time.

Differential Scanning Calorimetry. The extended dwell at high temperature may have induced chemical changes in the polymer. To investigate this hypothesis, tape samples were extracted from the APC friction specimens and subjected to a DSC analysis in a nitrogen atmosphere. The cooling trace was analyzed to obtain the degree of crystallinity. The results are plotted in the left graph in Fig. 4, which shows the calculated degree of crystallinity as a function of dwell time for different tool contacts. These results indicate a reduced ability to crystallize with increasing dwell time for the APC tape specimens in contact with the metal foil. In contrast, this reduced ability to crystallize was not observed for specimens extracted from friction specimens concerning the release-coated steel or the polyimide film.
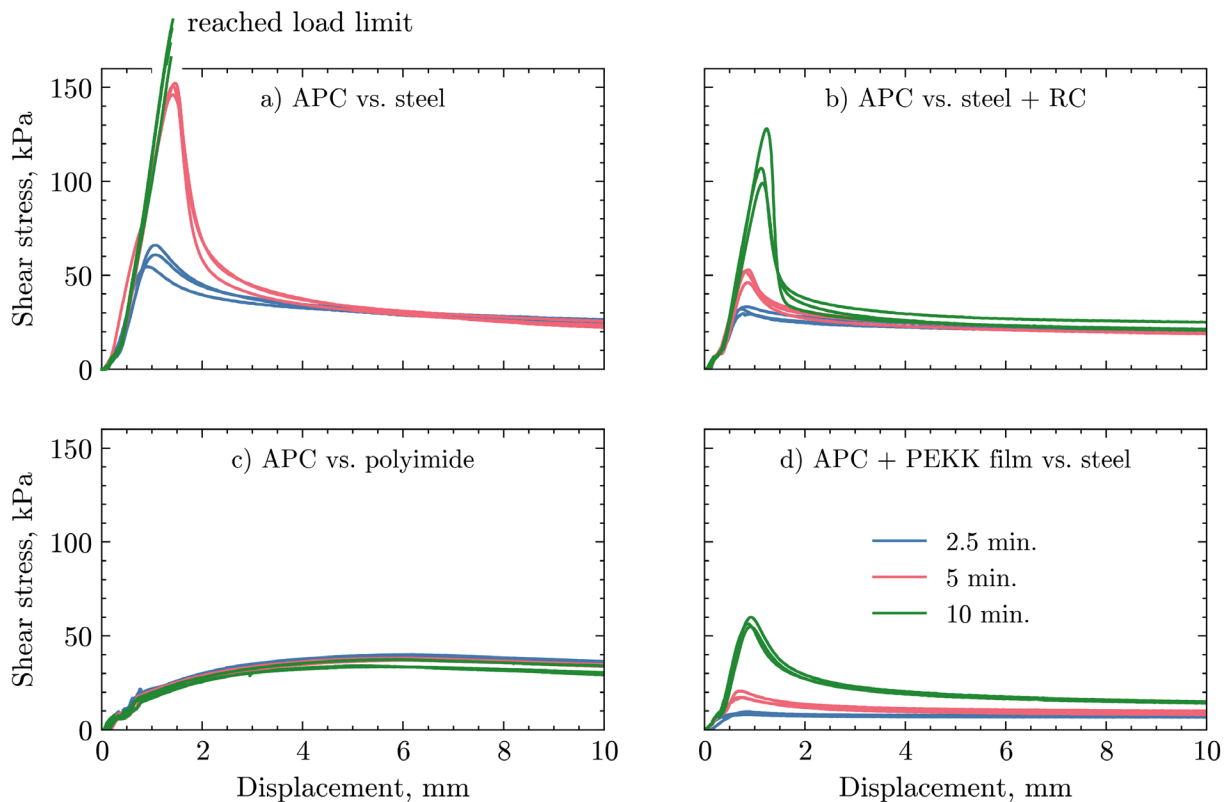

Figure 3. Interfacial shear stress evolution for APC against various tool surfaces, where '+ RC' indicates that a release coating was applied to the steel surface. The colors in the graphs represent different dwell times between closing of the pressure platens and initiating sliding action. All experiments were performed at a temperature of 385 °C, with a normal pressure of 15 kPa and a sliding rate of 25 mm/min.
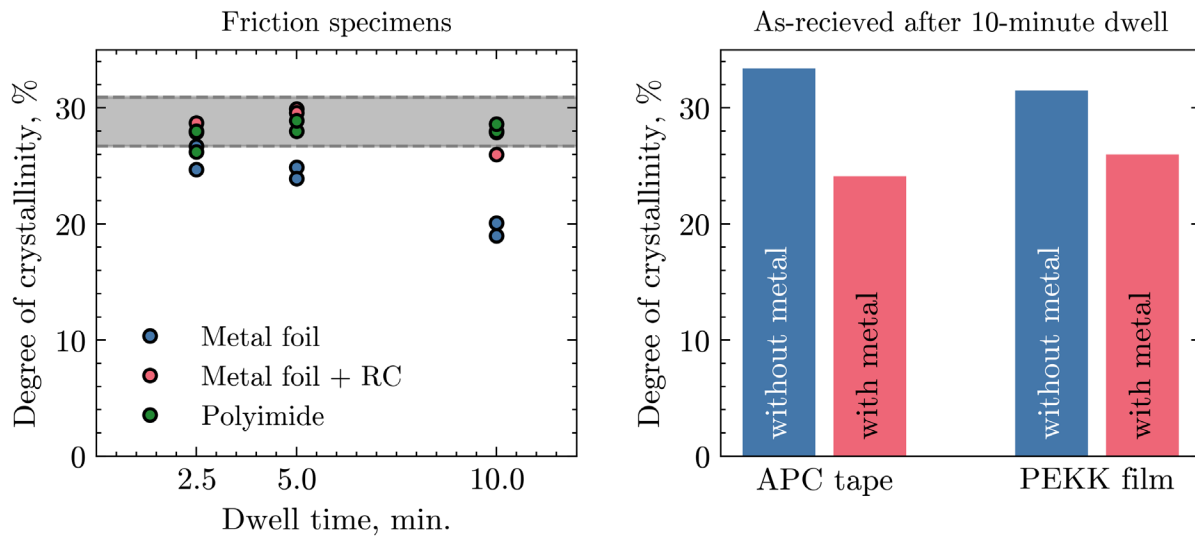
Figure 4. Left: Degree of crystallinity, after cooling at 20 °C/min in a DSC, for APC tape samples extracted from the friction specimens as a function of the dwell time. The color of the markers represent the different tool surfaces that were in contact with the tape. The shaded gray area indicates the maximum and minimum degree crystallinity measured on the as-received, and untreated, APC tape without a dwell. Right: Degree of crystallinity, after cooling at 20 °C/min in a DSC, for as-received APC tape and PEKK film samples with and without metal.

For reference, we also analyzed the as-received APC tape and PEKK film using DSC, both with and without small pieces of steel foil in the same pan. Prior to the analysis, the specimens were subjected to 10-minute dwell. The right graph in Fig. 4 shows the measured degrees of crystallinity. Overall, the results correspond with the data measured on the friction specimens, showing a decrease in the polymer's ability to crystallize when the APC tape or the PEKK film has been in contact with the metal film during a dwell in melt.

Microscopy. Fig. 5 presents two SEM images of a tape surface extracted from an APC-steel friction specimen subjected to a 10-minute dwell. As mentioned, significant force was required to peel the tape in question from the steel foil at room temperature. The images reveal the presence of small iron particles on the tape surface. EDX analysis confirmed that these particles contain a high fraction of iron, indicating material transfer from the steel foil surface to the ply.

SEM images of the corresponding steel foil surface are shown in Fig. 6. The horizontal lines on the metal surface are also present on the as-received foils and are oriented in the foil's rolling direction, which was perpendicular to the sliding direction used in the friction experiment. The SEM images illustrate damage in the form of craters on the foil's surface, which is in agreement with the iron particles found on the tape surface. These observations confirm a strong bond between the mild steel surface and the tape, after a 10-minute dwell at high temperature.

Discussion

In this section, we will discuss the possible interactions between mild steel and the PEKK polymer in the APC tape. Focusing on metal-catalyzed branching or cross-linking and surface bonding, we propose a mechanistic model of the interphase region to explain our experimental findings.
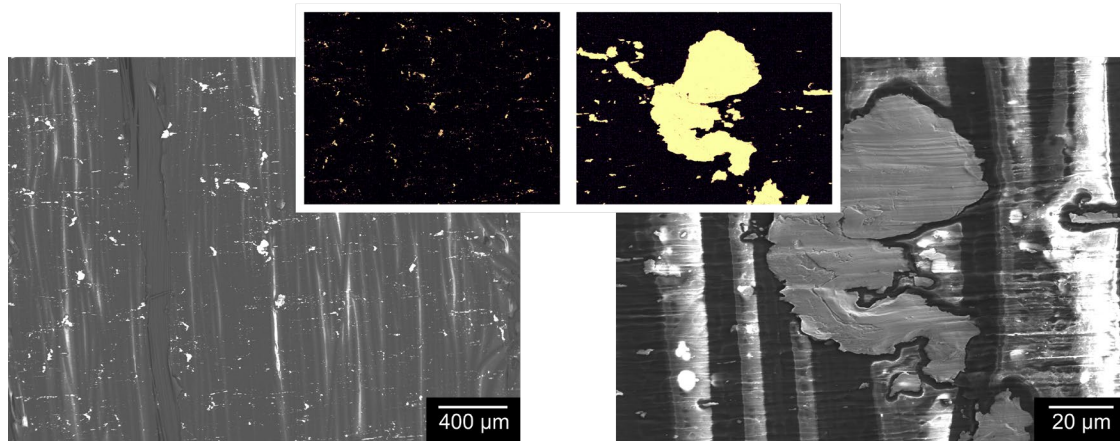
Figure 5. Scanning electron microscopy images showing the presence of iron particles on the fracture surface of the APC tape. The left image shows an overview, while the right a detailed view of an iron particle. Both inset images show the corresponding EDX maps for iron. The tape was extracted from an APC-metal friction specimen that was subjected to a 10-minute dwell.
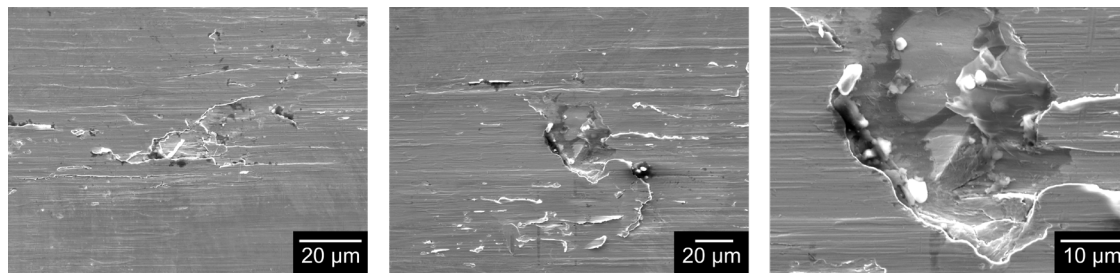

Figure 6. Scanning electron microscopy images showing damage on the metal surface. The metal foil was extracted from an APC-metal friction specimen that was subjected to a 10-minute dwell.

Metal-Catalyzed Chain Branching or Cross-Linking. The DSC results reported in Fig. 4 show a reduction of the polymer's ability to crystallize after prolonged contact with mild steel at elevated temperature. This effect indicates chain branching or cross-linking [15], and is only observed in cases involving contact with the mild steel, suggesting that the steel accelerates or causes polymer degradation.

Literature on the catalytic effect of a metal tool surface on polymer degradation during processing is limited, and we did not find any information specific for poly(aryl-ether-ketones). Some studies on polycarbonates are available though, and provide valuable insight. Sonnenberg et al. [16] studied the interactions between tool steel and a polycarbonate melt. Through X-ray Photo-electron Spectroscopy and Auger Electron Spectroscopy, they demonstrated the diffusion of metal atoms from the steel surface into the polymer melt. Notably, iron ions reacted with the polymer chains, resulting in the formation of iron-polycarbonate complexes that agglomerate through cross-linking and lead to the immobilization of polymer chains. Further investigations by Theile-Rasche et al. [17] correlated the degree of degradation in polycarbonate with the percentage of iron at the alloy surface. Although degradation was most pronounced near the interface, their experiments showed a degraded layer with a thickness 250 to 300 μm after one hour dwell at processing temperature. Notably, these authors also found that the presence of chromium inhibits polymer degradation. Possibly this is caused by a chromium oxide layer that inhibits iron diffusion.

A similar branching or cross-linking mechanism may be active in PEKK when it is in contact with a mild steel surface. The presented friction data supports this idea. Most notably, the extreme peak shear stress observed in the case of APC tape and the mild steel indicates that the polymer

present between the fibers and the steel has cross-linked up to a degree that it acts as a gel or solid instead of a lubricating film. At shorter dwell times, branching and cross-linking may cause an increase in viscosity but not yet to an extent that it leads to complete immobilization of the polymer chains. Similarly, the increase in peak and steady-state shear stress with dwell time in the case of the inserted PEKK film between APC and the uncoated steel, illustrated in Fig. 3d, also point to an increased viscosity of the lubricating polymer film. Here, the film can still act as a lubricant, even after a 10-minute dwell, because of its substantial thickness.

Finally, although the DSC data did not indicate chain branching or cross-linking for the release-coated friction specimens, the increasing peak shear stress with dwell time, shown in 3b, suggests otherwise. Possibly, the release coating acts as a barrier, limiting the diffusion of iron into the polymer and minimizing branching or cross-linking to only a surface layer of the tape, where it can have a significant influence on the measured shear stress but has a negligible effect on the measured DSC response, as the latter is governed by the bulk of the tape.

Surface Bonding. The high shear stresses required to initiate sliding between the APC tape and the uncoated metal, particularly after a 5- or 10-minute dwell, suggests strong bonding between the polymer and the metal. This is supported by the fracture surfaces, presented in Fig. 5 and 6, which illustrate damage to the steel surface and transfer of metal particles to the tape. While the nature of this bonding is unclear, our findings point to a chemical interaction between the polymer and metal surfaces.

Such chemical interactions have been reported in literature, albeit for different material systems. In the late 70s and early 80s, Burkstrand [18–20] for example demonstrated increased polymer-metal adhesive strength that could be related to the formation of metal-oxygen-polymer complexes at the interface for several polymers including polystyrene and PMMA. Recently, Wiesing et al. [21] employed XPS analysis to show the formation of Fe(II)-rich oxides at the interface between tool steel and molten polycarbonate.

The observed high peak stresses also imply strong bonding between fiber and matrix. In ply-ply friction experiments, we found an apparent critical fiber-matrix slip stress of approximately 35 and 55 kPa for C/PEEK and C/LM-PAEK, respectively [10, 11]. The proposed slip mechanism in these cases involves the disentanglement of chains, that are adsorbed on the fiber, from the chains in the bulk. Our inability to induce any sliding action in the APC-steel case, with a dwell of 10 minutes, suggests that polymer disentanglement is inhibited, which is consistent with the branching and cross-linking deduced from the DSC experiments.

Proposed Mechanism. The observed increase in friction between mild steel and APC with prolonged dwell time can be attributed to polymer-metal interactions. Based on our experimental results and existing literature, we propose a mechanistic model, schematically illustrated in Fig. 7. First, iron ions diffuse into the molten PEKK polymer at the interphase between fibers and tool. These ions interact with the polymer chains, leading to metal-polymer complexes through branching or cross-linking, similar as was observed for polycarbonates [16, 17]. As a result, the polymer viscosity increases up to a point where the thin layer between the metal and fibers becomes a gel and loses its lubricating function. Simultaneously, chemical bonding takes place between the polymer and the steel, possibly by the formation of metal-oxygen-polymer complexes at the interface [18–21]. The resulting adhesion between polymer is so strong that excessive stress is required to initiate sliding between tape and metal.
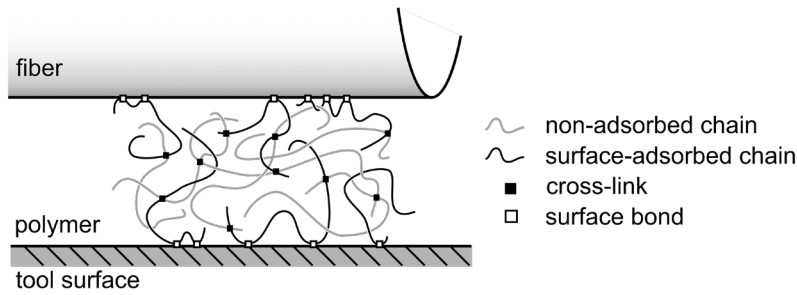
Figure 7. Schematic illustration of the tool-ply interphase, highlighting the mechanisms that govern the observed peak shear stress for APC and steel (inspired by [22]).

Theile-Rasche et al. demonstrated that an increase in chromium content in the steel can strongly inhibit polymer degradation [17]. To validate this finding, we conducted additional friction experiments using stainless steel foils, from grade X5CrNi18-10, featuring an 18% chromium content (see Table 2). Fig. 8 shows the shear stress evolution for the different dwell times, with the response for mild steel included for direct comparison. Some initial slip was observed between the stainless steel foil and the pressure platens, noticeable at the beginning of the curve (< 1 mm). In contrast to mild steel, where the peak shear stress increases quickly with dwell time, the influence of dwell time on shear stress evolution for stainless steel is minimal. This observation aligns with the work of Theile-Rasche et al. [17] and supports the hypothesis that iron ions play a key role in polymer degradation. Overall, these results underline the importance of tool material selection on friction and, hence, defect formation during manufacturing.

Table 2. Chemical composition (max weight %) of X5CrNi18-10 stainless steel.

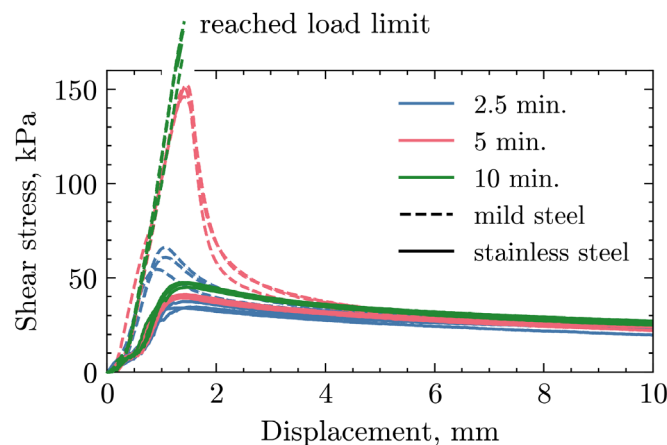| C	Si	Mn	Ni	P	S	Cr	N
0.07	1	2	8-10.5	0.045	0.015	17.5-19.5	0.11

Figure 8. Interfacial shear stress evolution for APC against mild steel (DC01) and stainless steel (X5CrNi18-10). The colors in the graphs represent different dwell times between closing of the pressure platens and initiating sliding action. All experiments were performed at a temperature of 385 °C, with a normal pressure of 15 kPa and a sliding rate of 25 mm/min.

Conclusions

Investigating the impact of dwell time on tool-ply friction is essential, as excessive friction can cause defect formation during the manufacturing of thermoplastic composite parts. In this work,

we investigated the effect of dwell time on the tool-ply friction response in melt for APC tape materials. The tool materials included mild steel, stainless steel and polyimide film. In line with earlier observations, the peak shear stress was found to increase significantly with dwell time in the case of the uncoated mild steel surface; an effect which was not observed for stainless steel or polyimide film.

The observed increase in peak shear stress for the mild steel surface is believed to be caused by metal-polymer interactions. Iron diffuses from the surface into the polymer, where it forms iron-polymer complexes that cause polymer chain branching and cross-linking. As a consequence the viscosity of the polymer increases up to a point where it cannot act anymore as a lubricating film. Simultaneously, these interactions also lead to strong adhesion between the polymer and the steel surface. Combined, these two effects result in strong adhesion between the tape and mild steel. The minimal impact of dwell time on the shear stress evolution in the case of a stainless steel surface, in contrast to mild steel, aligns with literature and emphasizes the role of iron ions in polymer chain branching and cross-linking. Overall, our findings emphasize the importance of tool material selection in manufacturing processes to ensure defect-free thermoplastic composite parts.

Acknowledgements

Data availability
The data belonging to this work is available via the 4TU.ResearchData repository [23].

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