Study of cake adhesion to membrane during membrane-assisted slurry transfer molding of ceramic matrix composites.

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1. Abstract

Silicon Carbide (SiC) Ceramic Matrix Composites (CMCs) are studied for aircraft engine components to improve the efficiency of engines. Slurry transfer moulding (STM) is a manufacturing process used to fill SiC fibre preforms with a water suspension of SiC particles, a.k.a., a slurry, inside a mould. The slurry is injected into the preform and filtered. The SiC particles accumulate onto the filter, forming a so-called cake. Replacing part of the mould with a pressurized membrane allows the process to adapt to the rigid SiC preform geometric inaccuracies and leads to smooth cake surface creation on the preforms surface. During the removal of the membrane after the injection, the cake adhered to the membrane and fragments were torn off from the part's surface. The objective of this study is to determine the origin of the tears. Four membrane materials were tested. The surface finish membranes were characterized with ISO 4287 and ISO 4288 standards using a microscope (VR–5000, Keyence) and a surface profilometer (SJ-410, Mitutoyo). Preforms used were made by 3D printed of polylactic acid (PLA). The preforms were placed into the mould cavity and covered by the membranes. A pressure of 560 kPa was applied in a membrane cavity to deploy the membrane on the preform. After filling the preform, two removal approaches were tested. First, peeling the membrane from the cake. Second, drying the cake by flowing compressed air under the membrane before peeling. For both approaches, the peeling of the membrane is done with the same calibrated movement. The teared cake surface area stuck on the membrane is quantified by a visual inspection. The results suggest that the roughness of membrane is not the primary cause of tearing. Controlled drying of the cake appears to improve process efficiency.

2. Introduction

Slurry transfer moulding (STM) is an industrial process used to produce ceramic matrix composites (CMC). In this process, a preform is placed in a rigid mould. At least one of the surfaces of the mould cavity is a filter. A slurry, defined as a suspension of ceramic particles in

water, is injected into the mould. When the cavity is full, the supply of slurry is continued, the pressure increases until it stabilizes to a specified pressure. The slurry is filtered and leads the growth of a particle cake, originating from the filter surface, and growing through the preform. The final component shape is defined by the rigid mould surfaces. Then a series of thermal treatments transforms the brittle cake into a solid ceramic matrix [1]. This manufacturing process presents challenges related to the geometrical variability of woven preforms [2,3]. The variability of the preform needs to be corrected after the injection to avoid dimensional inaccuracies. These corrective steps can significantly increase manufacturing costs and may induce damage to the component [4,5].

To accommodate the geometric variability of rigid Silicon Carbide (SiC) preforms, it is necessary to use a mould that can adapt to such differences. A flexible pressurized membrane called the conformation membrane can replace a part of the mould. Figure 1**Erreur ! Source du renvoi introuvable.** shows the chronological steps of the membrane assisted STM process. Figure 1a) shows the first step of the process. The preform is placed in the mould. The conformation membrane is inflated by a conformation fluid, i.e., air, pressurized and injected into a dedicated cavity between the top mould and the membrane (in green). When fully deployed, the membrane fits with the inaccuracies of the preform and prevents the cake from growing above the preform surface. The slurry is injected through holes on the sides of the mould, under the conformation membrane. During the injection, a SiC cake grows inside the preform until it reaches the top of the preform. Figure 1b) shows the formation of a cake at the bottom of the mould, above the filter. The filtrate (i.e., water) flows through the filter and is flushed. Figure 1c) shows the fully formed cake inside the preform, under the membrane.

Replacing part of the mould with a pressurized membrane allows the process to accommodate geometric variability in rigid SiC preforms and results in a smooth SiC cake [6]. However, after the injection, the membrane is removed and fragments of the cake can be torn off from the part's surface.

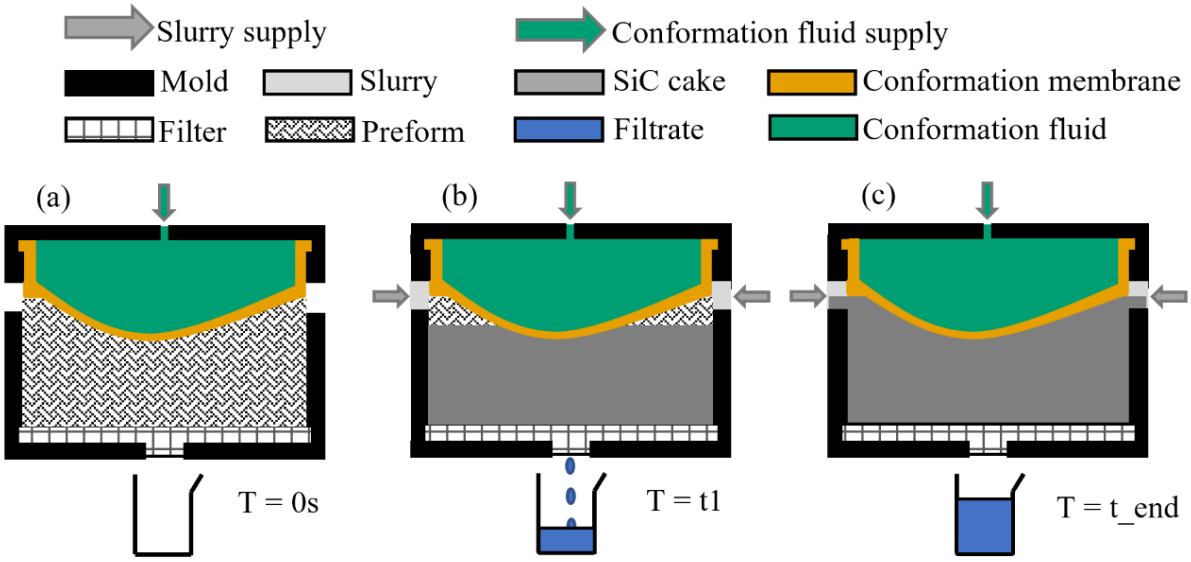

Figure 1. Membrane assisted STM process: (a) The conformation membrane is inflated, (b) The slurry is injected, SiC cake is building into the preform, (c) the final piece made of SiC cake and preform is fully formed

Tearing in the membrane-assisted STM process can be influenced by a number of interconnected parameters, such as membrane mechanical properties, injection process conditions, and interactions between the wet particle cake and the preform. More specifically, tearing at the surface of the cake during membrane removal can be explained by a competition between the cohesive strength of the cake and the interfacial adhesion between the cake and the membrane. When the local membrane to cake adhesion exceeds the cohesive strength of the cake, interfacial fracture occurs. This interfacial fracture process is governed by surface energy and contact mechanics [7]. In the case of a wet cake, liquid bridges between particles generate capillary forces, which increase interparticle cohesion within the cake [8]. At the interface with the flexible membrane, local forces may develop due to pressure variations and confinement effects, which can be interpreted as an apparent suction-induced effect [9]. Surface roughness of either the membrane or the substrate affects the real area of contact and leads to a non-uniform distribution of interfacial forces during detachment [9,10]. The detachment of the membrane from the substrate depends on the mode of interfacial separation (e.g., peeling or tensile loading), which can facilitate or hinder interfacial debonding [11]. The objective of this study is to identify the main factors responsible for cake tearing during removal of the conformation membrane. A comparison of tearing behaviour was conducted for membranes with different surface roughness. In addition, the effect of drying the cake prior to removal of the conformation membrane was investigated.

3. Materials and Methods

3.1. Materials

The slurry was a water suspension of SiC particles. The preform had a surface of approximately 58 × 57 mm^2 and 3 mm thickness. The preforms were made by PLA filament 3D printing. Two different preform were used. Preform1 was composed of 40 cells (16 half-cells and 24 full cells) with a rectilinear infill pattern. The nominal porosity was 88%. Preform2 was composed of 312 cells (48 half cells and 264 full cells). The nominal porosity was 91%. Table 1 lists the sample membranes used in this study. The first column shows the ID composed by the material abbreviation, followed by the thickness, the Shore A hardness (ShA), and one letter for the type, i.e., C or S. The membrane materials were polyurethane (SikaBiresinÆ PX840, Sika Group), thermoplastic polyurethane (Natural CoexFlex™ 60A TPU, Coex 3D), a High Temperature Vulcanizing silicone (HTV silicone, Diatex) and Silicone (LiquaFast ICE ® Castaldo). The thicknesses ranged from 1.4 mm to 3.0 mm. The ShA ranged from 40 to 60.

Table 1. Material, thickness, hardness and type of the different membrane samples used in the study

ID	Material	Thickness (mm)	Hardness (ShA)	Type
PU-2.0-50-C	Polyurethane	2.0	50	Conformation
PU-3.0-60-C	Polyurethane	3.0	60	Conformation
TP-1.4-60-S	Thermoplastic polyurethane	1.4	60	Sample
Si-1.4-40-S	Silicone (HTV)	1.4	40	Sample
Si-1.6-50-S	Silicone (LiquaFast)	1.6	50	Sample
PU-3.0-60-S	Polyurethane	3.0	60	Sample

There were two types of membranes, the conformation (C) and the sample (S) membranes. The conformation membranes are described below. The sample membranes were a rectangle of 120 mm length and 10 mm width. Depending on the experiment, the conformation membranes were covered with a PTFE adhesive layer (6305A13, McMaster-carr). All membranes' surface roughness (Ra, Rz) was characterized in accordance with ISO 4287 and ISO 4288 standards [12,13]. The parameters studied were the arithmetic mean deviation of the evaluated profile (R_a) and the maximum peak-to-valley height of the evaluated profile over the evaluation length (R_z). Due to the optical properties of some materials (transparent or reflective surfaces), two measurement techniques were employed: an optical profilometer (VR–5000, Keyence) for the PU-3.0-60-S membrane, whereas a contact profilometer (SJ-410, 0.75 mN tip, Mitutoyo) was used for the remaining membranes. The cutoff length (λ_c) was set to 0.8 mm. All samples were measured twice, whereas the PTFE layer was measured only once.

3.2. Membrane-Assisted Slurry Cast Set-Up and Standard Injection Pressure

Figure 2 shows a schematic exploded view of the set-up. Figure 2a) shows the slurry injected by the sides of the mould (grey arrows), through the side shims (in blue). The filter was inserted in the bottom mould. The sample membranes (in red), optional depending on the experiment, are placed between the side shim, preform and the conformation membrane (in orange). The top mould was above the conformation membrane and the conformation fluid cavity was between the top mould and the conformation membrane (in green). Figure 2b) shows the top view of the conformation membrane geometry. The rectangular surface of 57 by 58 was the one fitting into the injection cavity and which was pressed against the preform via the conformation fluid i.e., air.

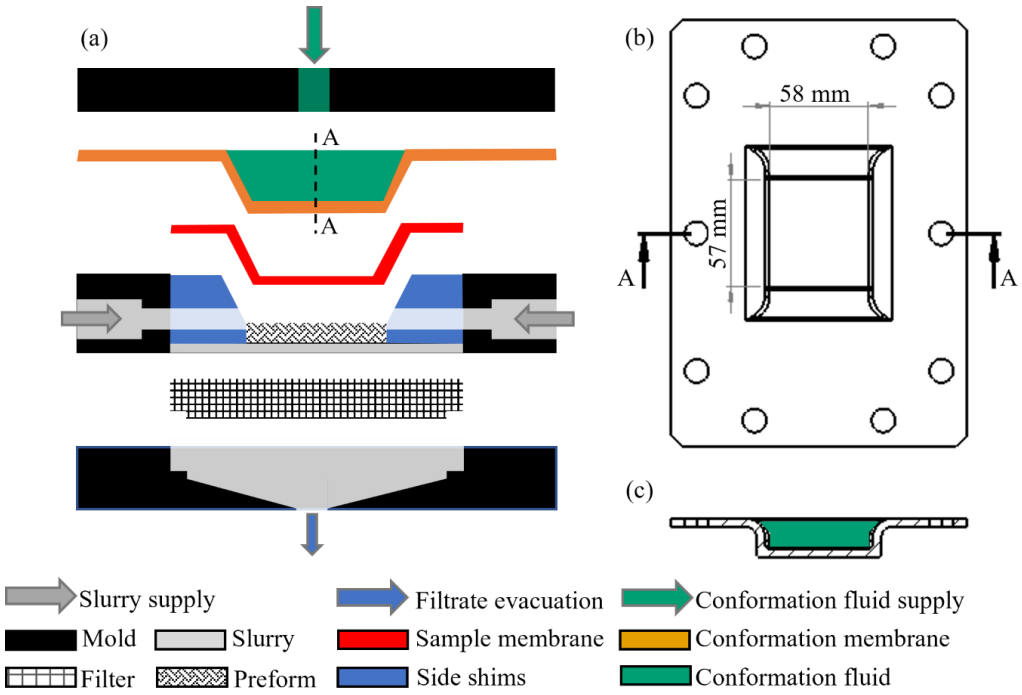

Figure 2. Schematic of the experimental set-up: (a) Exploded view of the mould, with the optional sample membrane used for the roughness study shown in red. (b) View of the conformation membrane from the conformation cavity. (c) Sectional view of the conformation membrane showing its geometry and the conformation cavity.

Before slurry infiltration, the conformation membrane was inflated in the mould with a conformation pressure of 560 kPa. The air pressure in the conformation cavity was controlled using a pressurized vessel fitted with a valve and a pressure gauge. The slurry injection flow rate was 40 mL/min until the injection pressure stabilized at 540 kPa. Then, the flow rate was adjusted using PID control to maintain the injection pressure at 540 kPa. The injection process lasted approximately 30 minutes to ensure the complete formation of the cake within the preform. At the end of the injection, the pressure in the injection cavity decreased until it reached the atmospheric pressure. The conformation membrane was then manually deflated using the previously described pressurized vessel. Finally, the conformation membrane was manually peeled off.

3.3. Tearing Characterization

Erreur ! Source du renvoi introuvable. Table 2 summarizes the experimental campaign, including the membrane types used (C or S), the experimental variations, and the number of repetitions performed. All experiments used a conformation membrane. Some experiments used a conformation membrane covered with a PTFE adhesive layer, indicated by the letters PTFE before the membrane acronym. Reference experiments were performed following the standard injection procedure described in Section 3.2. The surface roughness experiments investigated the influence of surface roughness, as described in Section 3.3.1. The drying at 69 kPa and drying at 210 kPa experiments investigated the influence of the drying step, as described in Section 3.3.2. These experiments allowed the assessment of the effects of membrane surface roughness and cake drying on cake tearing at membrane removal.

The tearing was characterized either by the presence of cake fragments adhering to the membrane surface, or, when this was not sufficiently visible on the membrane, by the areas of missing material on the cake itself. A quantitative grid-based approach was adopted. The surface was divided into a regular grid, corresponding to the physical cells of the PLA preform. Each cell was visually inspected. A score of 0 was assigned to intact cells. A score of 0.5 was assigned to partially torn cells. A score of 1 was assigned to fully torn cells. The Tearing Index (TI) was then defined as the sum of the scores over the inspected grid surface divided by the number of cells.

Table 2. Summary of the experimental campaign, showing the membrane used, the experimental variation investigated (reference procedure, surface roughness, or cake drying), and the number of repetitions performed.

Membrane		Reference	Surface roughness	Drying at 69 kPa	Drying at 210 kPa
C	S				
PU-3.0-60-C	n/a	1	-	-	-
PTFE-PU-3.0-60-C	n/a	-	-	1	1
PU-2.0-50-C	n/a	1	-	-	1
PTFE-PU-2.0-50-C	n/a	-	-	-	1
PTFE-PU-3.0-60-C	Si-1.4-40-S	-	2	-	-
	Si-1.6-50-S	-	2	-	-
	PU-3.0-60-S	-	2	-	-
	TP-1.4-60-S	-	2	-	-

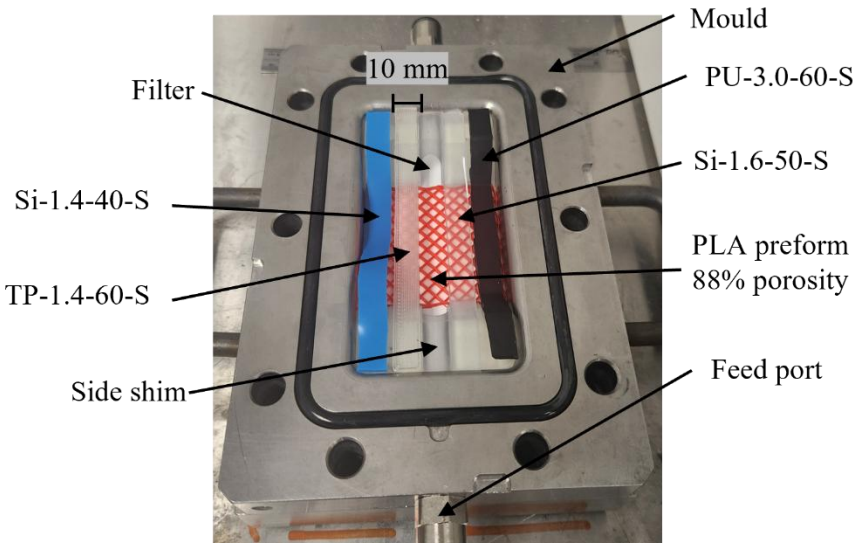

Figure 3. Photo of the sample membrane in the mould before covering it with the conformation membrane. From left to right are respectively the two silicone samples, the FDM made PU and the TPU samples.

3.3.1. Effect of Membrane Surface Roughness on Cake Adherence

Figure 3 shows how the sample membranes with various surface roughness were placed above the red PLA preform. The four samples were tucked in the side shim and covered with the conformation membrane. They were left unconstrained above the preform but laterally positioned and secured using side shims placed against the preform edges. Before slurry infiltration, the conformation membrane was deployed to maintain the sample membrane against the preform and to prevent displacement during injection. It was assumed that, under the initial conformation membrane pressure, all membrane samples were pressed into contact with and constrained against the preform. The conformation membrane of 60 ShA (PTFE-PU-3.0-60-C) was covered with a PTFE adhesive layer and used with preform2. After the cavity pressure was released at the end of the injection, the pressure in the conformation cavity was then slowly released to atmospheric pressure during 23 seconds by manually opening the pressurized vessel described in Section 3.2.

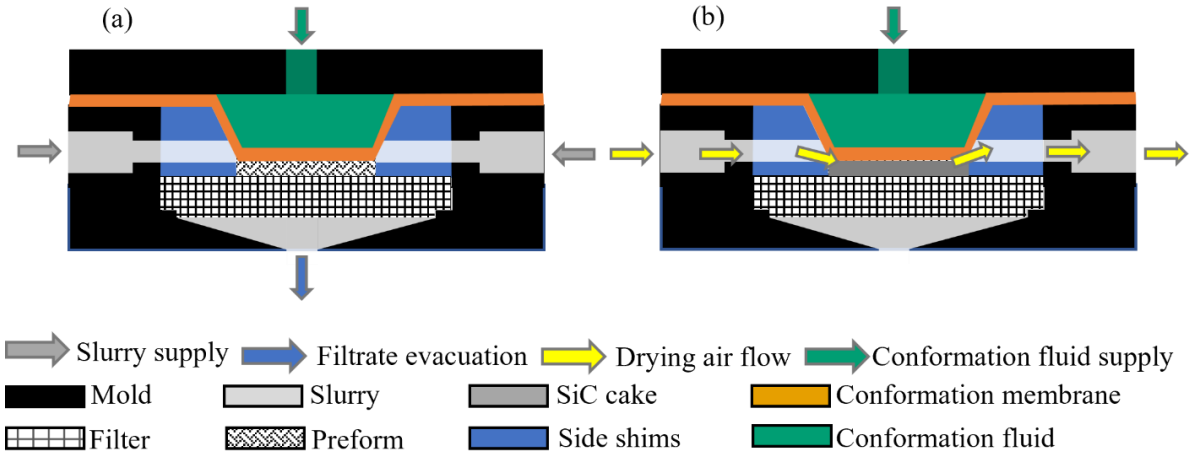

Figure 4. Drying experiment steps: (a) cake formation with the conformation membrane inflated; (b) drying by air injection through the feed port while the conformation membrane remains inflated.

The conformation membrane was removed, and the sample membranes were manually peeled off using the same calibrated motion. The experiment was repeated twice, with new sample membranes for each test.

3.3.2. Effect of Drying the Wet Cake before Removing the Membrane

Figure 4 show a schematic of the cake drying experiments. Figure 4a) shows the standard injection process during cake formation. Figure 4b) shows the airflow passing through the mould to dry the cake, from one feed port to another. The injection was carried out as described in Section 3.2. Before reducing the conformation pressure, one of the feed ports was disconnected and used as an air inlet. Airflow was injected through the mould (see Figure 4b). The drying time was 360 seconds. An initial test was conducted by injecting air through the mould at a pressure of 69 kPa to assess whether a lower drying intensity would be sufficient to prevent tearing. In a subsequent experiment, the air pressure was increased to 210 kPa. In both cases, after drying, the mould was opened and the conformation membrane was manually peeled off. For this study, both conformation membranes were tested with and without the PTFE covering on a preform1.

4. Results and Discussion

Table 3**Erreur ! Source du renvoi introuvable.** summarizes the Ra and Rz values of the membrane samples for the roughness experiments. The roughness of the conformation membrane is defined by the PTFE layer covering its surface. Si-1.4-40-S exhibited the smoothest surface, with the lowest Ra (0.16 µm) and Rz (1.01 µm). In contrast, Si-1.6-50-S exhibited the highest Ra (5.22 µm) whereas TP-1.4-60-S exhibited the highest Rz (28.47 µm). The selected membranes exhibited a wide range of Ra and Rz values, providing a broad spectrum of surface roughness for the study.

Table 3. Surface roughness of the sample membrane and conformation membrane. The results are reported as the mean and standard deviation of two measurements for each membrane sample. Since only one measurement was performed for the PTFE layer, no standard deviation could be calculated.

ID	Ra (µm)		Rz (µm)	
	Mean	Std. Dev.	Mean	Std. Dev.
TP-1.4-60-S	0.97	0.08	28.47	2.63
Si-1.4-40-S	0.16	0.06	1.01	0.41
Si-1.6-50-S	5.22	0.24	6.97	0.32
PU-3.0-60-S	0.67	0.02	3.83	0.206
PTFE layer	0.38	-	1.47	-

Table 4. Tearing Index (TI) for all experiments as a function of membrane type and experimental conditions (reference procedure, surface roughness, and cake drying). For the roughness results, column C corresponds to tearing occurring on the conformation membrane, and column S corresponds to tearing occurring on the sample membrane. A dash (–) indicates that the experiment was not performed.

Membrane		Reference	Roughness		Drying 69 kPa	Drying 210 kPa
C	S		C	S		
PU-3.0-60-C	n/a	0.58	-	-	-	-
PTFE-PU-3.0-60-C	n/a	-	-	-	0.53	0.00
PU-2.0-50-C	n/a	0.00	-	-	-	0.09
PTFE-PU-2.0-50-C	n/a	-	-	-	-	0.00
PTFE-PU-3.0-60-C	Si-1.4-40-S	-	0.65	0.02	-	-
	Si-1.6-50-S	-		0.01	-	-
	PU-3.0-60-S	-		0.00	-	-
	TP-1.4-60-S	-		0.03	-	-

Measurements of the TI are compiled in **Erreur ! Source du renvoi introuvable.** where the TIs are shown in their respective experiment column for a given set of conformation and sample membrane. In the reference experiment, tearing was observed only on the PU-3.0-60-C membrane (TI of 0.58), whereas the PU-2.0-50-C membrane showed no signs of tearing (TI of 0.00). The roughness study experiment was conducted with the conformation membrane PTFE-PU-3.0-60-C and the sample membranes described in Section 3.1. In these experiments, no significant tearing was observed on the sample membranes (TI between 0.00 and 0.03). However, tearing occurred on the conformation membrane covered with a PTFE layer. The TI, calculated on the exposed surface of the PTFE-PU-3.0-60-C, was 0.65. The drying experiments with a drying pressure of 69 kPa when using the PTFE-PU-3.0-60-C resulted in a TI of 0.53. Finally, all drying experiments at 210 kPa had no significant TI measured (TI between 0.00 and 0.09).

Regarding the surface roughness of the membrane, the results shown in **Erreur ! Source du renvoi introuvable.** suggest that surface roughness alone is not the primary factor governing surface cake tearing during membrane removal. For all sample membranes, regardless of surface roughness, no significant TI was observed. This observation is further supported by the fact that cake tearing occurred on the conformation membrane PTFE-PU-3.0-60-C used during the surface roughness experiments. That PTFE-covered conformation membrane had among the lowest values of Ra (0.38 µm) and Rz (1.47 µm).

Among all experiments without drying, it was observed that the reference experiment PU-2.0-50-C membrane showed no tearing during the reference test without drying (TI of 0.00). A TI of 0 was unexpected since the reference experiment with the comparable membrane PU-3.0-60-C had a TI of 0.58. The PU-2.0-50-C had a lower thickness and lower ShA than the PU-3.0-60-C, suggesting that membrane stiffness may play a role in cake surface tearing. However, the PU-2.0-50-C reference experiment deviated from the standard procedure. The injection time exceeded 30 min. Once the cake had completely filled the preform, further injection led to cake growth within the injection port. As a result, the cake inside the injection cavity may have undergone

partially natural drying or increased consolidation. To confirm this hypothesis, the experiment should be repeated following the standard procedure.

A suction-related effect may explain the tearing of cake at membrane removal. Except for the reference experiment PU-2.0-50-C, all experiments with significant TI were done with the standard injection procedure that resulted in residual water observed at the surface of the cake after membrane removal. A thin water layer may increase the adhesion forces between the membrane and the cake. The TI was 0.58 on the PU-3.0-60-C during the reference experiment and an average of 0.65 on the exposed PTFE-PU-3.0-60-C during the two roughness experiments. It is to be noted that the absence or presence of a PTFE adhesive layer on the surface did not seem to affect the measured TI.

Under a drying pressure of 69 kPa and a drying time of 360 s, tearing was partially reduced, with a TI of 0.53. To further investigate this effect, the drying pressure was increased to 210 kPa while maintaining the same drying time. At 210 kPa, tearing was strongly reduced or did not occur (TI between 0.00 and 0.09). The reduced TIs indicate that drying the cake may be the primary factor to reduce cake tearing during membrane removal. However, an optimal operating window for drying pressure and drying time remains to be identified in future experiments. Moreover, the airflow distribution through the cake within the mould remains unclear. It is possible that airflow preferentially through specific paths, resulting in non-uniform drying of the cake surface, which could explain the TI value of 0.09 observed for the PU-3.0-50-C membrane. The membrane hardness may also influence airflow distribution by promoting preferential flow paths, potentially leading to incomplete or non-uniform drying of the cake.

5. Conclusion and Further Work

The results of this study indicate an improvement in the process when the cake is dried prior to peeling. By properly controlling drying time and pressure, tearing may potentially be prevented. This progress may enable the production of smooth surfaces on complex-shaped aerospace components using a membrane-assisted STM process. Moreover, the results suggest that membrane roughness is not the main or sole factor responsible for tearing. Further experiments are therefore required to fully understand the mechanisms involved. These investigations should begin by exploring a range of drying times and pressures in order to define an optimal operating window for this parameter space. Once such conditions have been established, the process should be validated on representative geometries, such as turbine blades and vanes. Membrane hardness was not extensively investigated in this study. Once an optimal drying condition is defined, it would be relevant to examine the influence of different hardness levels. It can be hypothesized that membrane hardness affects airflow distribution, potentially leading to non-uniform or less effective drying. In addition, another parameter not addressed in this work is the chemical nature of the membrane, which may also play a significant role in the observed behaviour.

6. References

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