



Article Effects of Precursors Ratio and Curing Treatment on the Icephobicity of Polydimethylsiloxane

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Abstract: Elastomers are intriguing materials for many applications, one of these being icephobic coatings. Elastic modulus and work of adhesion are the key parameters coming into play in ice detachment mechanisms, and can be related to hardness and wettability. Polydimethylsiloxane (PDMS) is widely used for anti-ice applications; however, not many works deal with the correlation between cross-linking grade, curing treatments, and icephobicity. This study focuses on PDMS (Sylgard184[®]) coatings, encompassing four different pre-polymer to cross-linking agent (A:B) ratios ranging from 5:1 to 30:1, and nine curing treatments. The results indicate that increasing the A:B ratio enhances hydrophobicity, softness, and icephobicity, assessed through shear stress measurements. Curing treatments primarily affect hardness and icephobicity, with longer heat treatments resulting in higher hardness and ice adhesion. All samples exhibit promising performances in lowering shear stress values, up to seven times in respect to the uncoated reference for 30:1 ratio. Additionally, a durability assessment is conducted on samples exposed to stress tests in the climatic chamber. A slight deterioration in hydrophobicity across all samples is observed and, notably, a significant hardness increase, around 13%, is experienced for the 5:1 ratio only. The samples also demonstrate an overall robust icephobicity after stress tests, and, for the 30:1 ratio, an average shear stress value four times lower than the reference is maintained. In this work, we highlight the importance of the fine-tuning of the precursors ratio and thermal treatments on the PDMS properties and durability.

Keywords: elastomer; coating; Sylgard184[®]; icephobicity; wettability; hardness; durability

1. Introduction

Surface icing causes severe concerns in many fields, from aerospace to electricity transmission, and many solutions are under development to face it. These solutions include icephobic coatings, capable of hindering ice adhesion by exploiting different properties and mechanisms. Elastomeric coatings are proven to reduce ice adhesion on account of their viscoelastic properties, and, in general, the lower the elastic modulus, the higher the icephobic performances [1].

Polydimethylsiloxane (PDMS) is an elastomeric material, finding applications in many different fields from biomedical to microelectronics [2–4], as it is a biocompatible, physically stable, and chemically inert compound. For icephobic applications, the relevance of PDMS is due its elastic properties and to its low surface energy, about 20 mJ/m² [5], owing to the presence of methyl groups in the silicon chain. The elastic modulus of PDMS, as well as surface energy and hydrophobicity, is related to the cross-linking density, which can be controlled by tailoring the chemical composition [6,7] and kinetics of formation [8].



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Based on this, the aim of the work is to seek for the best experimental parameters to obtain both icephobic and durable PDMS materials.

Sylgard 184[®] is used to prepare PDMS coatings and bulk materials. Four different prepolymer:cross-linker weight ratios (5:1, 10:1, 20:1, and 30:1) and up to nine different curing conditions (ranging from 25 °C to 200 °C and from 35 min to 48 h treatments) are studied. Wettability, hardness, and icephobicity are systematically measured to evidence differences among formulations and to estimate the apparent work of adhesion of ice. Aging at room temperature and stress test cycles in environmental chambers are performed to evaluate overtime variations and to assess the durability. Such an integrated approach allows us to identify the most promising PDMS-based materials among those studied.

2. Materials and Methods

2.1. Materials and Sample Preparation

Aluminum alloy (6082) flat samples ($50 \times 50 \times 2$ mm) were used as substrates. Samples were sandblasted and cleaned with acetone before their use.

Sylgard184[®] (Syl184) was purchased from DowSil[™]. The elastomeric kit is constituted by a prepolymer (A) and a cross-linker (B). According to MSDS, prepolymer is mainly composed of vynil-siloxanes, and cross-linker agents are a mixture of different components containing hydrogen siloxane functional groups. The samples were prepared by mixing the prepolymer and cross-linker in the desired weight ratio (5:1; 10:1; 20:1, and 30:1). After stirring for 30 min, PDMS was degassed for at least 1 h in vacuum and then cast on the substrate to obtain, after the curing process, a film of about 850 µm of thickness. For each sample, a bulk disk of about 10 mm of thickness and 30 mm of diameter was also prepared and used for hardness and FTIR characterizations. All films and bulk disks underwent thermal treatment (tt) in a convection oven at 25, 100, or 200 °C for different times, and then were cooled at room temperature. The tested thermal treatments are reported in Table 1. Hereinafter, the different samples will be referred to as A:B-ttx, in order to discriminate between the different A:B ratios and thermal treatments.

Thermal Treatment	Temperature	Time
tt1	25 °C	48 h
tt2	100 °C	35 m
tt3	100 °C	1 h
tt4	100 °C	3 h
tt5	100 °C	6 h
tt6	100 °C	9 h
tt7	100 °C	12 h
tt8	100 °C	24 h
tt9	200 °C	18 m

Table 1. Thermal treatment for curing PDMS. tt1 and tt9 were applied only for samples deriving from a 10:1 ratio.

2.2. Characterization

The FT-IR characterizations were conducted with a FT-IR Alpha 1 (Bruker, Billerica, MA, USA) spectrometer with ATR apparatus (diamond crystal was used as the internal reflection element).

Hardness was measured with a Shore A type durometer, according to the ASTM D2240 standard test method [13] specific for softer elastomer, defining a value within 0–100 H_A (Shore A hardness units). Five measures were recorded on each sample.

The static water contact angles (WCAs) were measured with a DSA 30 Drop Shape Analyzer (Kruss, Hamburg, Germany) with the sessile drop method using 5 μ L volume of water at 20 °C on at least 5 points for each sample. Dynamic contact angles, hysteresis (H), and sliding angle (SA) measurements were performed at 20 °C with the tilting plate method using a 25 μ L volume of water. Five replicates were performed on each sample.

Ice adhesion was evaluated using a shear stress analysis performed with a homemade apparatus described elsewhere [14]. Shortly, ice was formed on the surface of the samples in a $10 \times 10 \times 10$ mm mold filled with deionized water cooled at -15 °C for 2 h. The sample surface was kept at a constant temperature by means of a Peltier cell, and the experiment was carried out in less than 30 s. A mechanical probe, moved with a constant speed of 0.3 mm/s, detached the ice from the surface by applying a tangent force. Thanks to a load cell connected to the probe, the peak force F needed to detach the ice from the sample was registered. The ice adhesion strength (τ) in shear values can be calculated as follows: $\tau = F/A$, where A is the surface of the specimen in contact with the ice. The shear stresses were measured by performing 4 tests on 4 different specimens for each sample. An untreated aluminum alloy surface was also measured for comparison purposes.

2.3. Methods

All of the prepared samples were characterized after the curing process ("cured samples"). To assess the over-time evolution of materials, the samples were then stored in a dry box, and characterizations were repeated after 7, 14, 30, 45, and 60 days ("aged samples"). Stress tests in climatic chambers (described in Table S1) were also conducted on selected aged samples (ST), and, after that, wettability, hardness, and icephobicity were measured. Briefly, they consist of a series of four simulated environmental stresses. Thermal cycles between -30 and 80 °C, and UV irradiation cycles with and without water spraying, were sequentially applied to samples.

3. Results and Discussion

3.1. Cured Samples Characterization

The chemical reaction between the A and B components of Syl184 consists of an addition of Si-H to C=C bonds to give Si-C bonds, as shown below for a generic hydrosilylation reaction, and is accelerated by heat.

$$R_3Si-H + H_2C=CHSiR_3' \rightarrow R_3Si-CH_2-CH_2SiR_3'$$

In this work, tt1 and tt2 are the standard treatments, as reported in the Technical Data Sheet. The heat treatments from tt3 to tt8 were conducted to study the effect deriving from a prolonged heating, while tt9 was performed to assess the effect of a high temperature applied for a short time.

The different A:B ratios were screened with the aim of studying the effect deriving from different degrees of cross-linking, using lower (5:1) or higher (20:1, 30:1) ratios with respect to the recommended formulation (10:1), in order to impart significative differences in mechanical properties. The elastomers were cast on sandblasted Al alloy flat samples to guarantee an optimal adhesion.

The different thermal treatments do not bring to significant variations of the FTIR spectra; however, some differences among the A:B ratios are visible. The spectra of samples cured at tt8 are displayed in Figure 1, where the main signals are as follows: 2964 cm⁻¹ (ν , CH₂-H), 1269 cm⁻¹ (δ , CH₂-H), 1060–1011 cm⁻¹ (ν , Si-O-Si), and 786 cm⁻¹ (ν , Si-CH₃). Samples corresponding to 5:1 and 10:1 ratios evidence a weak signal of 910 cm⁻¹ due to ν Si-H, indicating the presence of an unreacted cross-linker. These signals tend to disappear for high A:B ratios [15].



Figure 1. FTIR–ATR spectra of different A:B ratios in the inset of the signals due to ν (Si–H) at 910 cm⁻¹.

Wettability results are plotted in Figure 2. In all cases, the static WCA falls approximately between 115° and 125°. As also reported in [15,16], WCAs increase when increasing the A:B ratios (30:1 and 20:1); less cross-linked PDMSs exhibit lower surface energies than more cross-linked ones, as reported in [7].



Figure 2. Wettability of the different samples. WCA: water contact angle; H: contact angle hysteresis; SA: sliding angle. An amount of 90° is reported for SA if no sliding is observed.

In general, thermal treatments do not significantly affect the WCAs, as minor differences (lower than 3°) are observed among differently cured samples. However, sample 10:1-tt1 reports considerably higher WCA than its high-temperature-cured analogous samples; this may be indicative of an incomplete cross-linking of the material [7]. Sliding angles and hysteresis were measured to assess water mobility on the surfaces. The samples of 5:1-ttx and 10:1-ttx exhibit dynamic properties, with the former showing the highest droplet mobility on the surface, as reported in the literature [17]. Conversely, samples of 20:1-ttx and 30:1-ttx do not show water mobility, as the drop sticks to the surface even when tilted up to 90°. As reported in [7,18], in the case of a partially cross-linked polymer, the rearrangement of terminal and distal chain segments gives rise to freely protruding ends and loops at the interface. These were found to have a large effect on adhesion hysteresis in case of polymer–polymer adhesion, and can also influence the water mobility. In the case of 20:1-ttx and 30:1-ttx, where the cross-linking of PDMS is reduced, the motion of water is likely hindered by a higher density of free chain segments on the surface.

Hardness measurements with a Shore A durometer are based on the penetration of an indenter into the material. The indentation depth is inversely related to the hardness and can be empirically related to the elastomer Young's modulus. As expected [6], in our study, the mechanical properties are found to be dependent on the A:B ratio and hardness varies coherently among samples prepared with varying prepolymer and cross-linker relative amounts.

In general, the 10:1-ttx samples show the highest hardness. The softest samples are obtained using higher ratios (20:1 and 30:1), whereas the 5:1-ttx samples show hardness values slightly lower than those collected for 10:1-ttx, as depicted in Figure 3a. This is attributable to the presence of unreacted cross-linking segments that act as softeners, as evidenced in the FTIR spectra (Figure 1), and also as reported in [19].



Figure 3. (a) Hardness and (b) shear stress data for elastomers with different A:B-ttx.

PDMS hardness is also affected by thermal treatments. For 5:1, 10:1, and 20:1, the trends from tt2 to tt8 indicate that more prolonged heating results in increasingly harder polymers, suggesting that the progression of the reaction between the prepolymer and cross-linker. For 30:1, a stabilization of hardness values around 11 Ha is observed from tt3 to tt8 (Figure 3a). This points out that no further cross-linking takes place, likely due to the unavailability of the unreacted Si-H groups.

The 25 °C treatment (tt1) gives rise to the softest 10:1 elastomer, while raising the temperature up to 200 °C (tt9) does not lead to a hardness increase when compared to 100 °C. Hardness values measured on these samples are in good agreement with a previous study [20].

According to shear stress measurements (Figure 3b), all of the elastomers evidence icephobic properties, decreasing ice adhesion up to seven times with respect to the bare aluminum alloy (about 350 kPa). PDMS with ratios different from the recommended one (10:1) generally show lower shear stress values, resulting in them being more icephobic; this trend is in good agreement with a previous study conducted on similar samples (in tt3 conditions) with a comparable testing method [17]. In general, longer thermal treatments result in an increase in shear stress values, as also reported in [1]. For 30:1 samples, different

Indeed, an overall good correlation between shear stress and hardness results appears evident from Figure 4.



Figure 4. Shear stress vs. hardness plot of the samples.

Shear stress is related to the square root of the shear elastic modulus, which is in turn related to Shore A hardness. However, considering the shear stress vs. hardness plot (Figure 4), samples appear to distribute along four distinct trend lines, according to their ratio. Given these trends, it is worth asking if the different A:B ratios lead to differences in ice adhesion, regardless of hardness. In other words, can the differences in shear stress values be related to variations in the work of adhesion of ice and surfaces?

According to Equation (1) [21], the shear stress (τ) at which a rigid stud (such as ice) is detached from a soft film is given by the following:

$$au_{ice} \sim \left(\frac{a}{h}\right) \sqrt{\frac{W_a G}{h}}$$
 (1)

where *a* is the length of the stud, *h* is the film thickness, W_a is the work of film adhesion, and *G* is the shear elastic modulus, which is related to Young's elastic modulus (*E*) by the following:

$$G = \frac{E}{2(1+\nu)} \tag{2}$$

where ν is the Poisson's ratio, given at 0.5 for generic PDMS [22].

E, expressed in MPa, can be estimated from Shore A measurements by several empiric approximations. Here, the following was chosen [23]:

$$E = \frac{(15.75 + 2.15 \times Ha)}{100 - Ha} \tag{3}$$

Considering a constant thickness of 850 μ m for all the samples, the W_a was calculated using Equations (1)–(3), and is reported in Figure 5.



Figure 5. Work of adhesion of different coatings calculated from Equations (1)–(3).

Samples of 30:1-ttx and 20:1-ttx evidenced significantly higher W_a values than 5:1-ttx and 10:1-ttx. This increase is likely due to the higher adhesive and frictional forces on these surfaces, as described in [18]. Evidence of such an effect is the concomitant lack of water mobility found for samples 20:1 and 30:1, for which SA is >90° and H is not measurable. From a macroscopic point of view, 20:1 and 30:1 surfaces result in being rather sticky to the touch, irrespective of the thermal treatment undergone.

3.2. Aged Samples Characterization

The hydrophobic properties of all samples showed no significant changes in WCA, SA, and H angles over 60 days. The only significant variation is observed for $10:1_tt1$, with a slight reduction in WCA; over a period of 7 days, the initial contact angle of 125.2° decreased to 122.5° and further dropped to 117.6° after 60 days.

Hardness variations for 100 °C treatments (tt2–tt8) are plotted in Figure 6; in most cases, the trend indicates a slight increase. According to ASTM D2240 [13], only a variation of ± 2 of hardness value is to be considered significant.



Figure 6. Hardness values during the 60 days of aging for polymers cured at 100 °C.

A dual effect of the A:B ratio and curing treatment on the evolution of hardness was observed. Generally, 20:1-ttx and 30:1 ttx show smaller increases in hardness over time, whereas 5:1-ttx and 10:1-ttx evidence more pronounced variations. Longer curing treatments (tt4–tt8) lead to minor hardness variations, reaching a plateau value within the first 7 days. Conversely, short thermal treatments, especially in the case of 5:1 and 10:1 ratios, exhibit significative changes even after 30 days. Furthermore, for a given A:B ratio, hardness measured at the 60th day remains reasonably similar to the initial one. Importantly, the values are strictly dependent on the duration of the thermal treatment. This confirms that different mechanical properties can be imparted by judicious choice of thermal treatments and A:B ratio.

For the 10:1 ratio, tt1 and tt9 show that, even varying curing temperature, this behavior is maintained, reporting initial values of 32.5 and 54 and, after 60 days, 43.6 and 57.9, respectively (see Figure S2 in the Supplementary Materials).

Shear stress data do not exhibit significant variations over time across all of the tested samples, as only minor increases are recorded (see Figure S1 in the Supplementary Materials). Even if the inherent error of shear stress analysis (15%) does not allow us to consistently correlate an increase in hardness to any slight augment in ice adhesion, a trend is somehow observable for each ratio. By plotting hardness vs. shear stress, the collected data consistently fall on the same straight line observed at the initial time (Figure 7). The grouping according to different ratios is also maintained.



Figure 7. Shear stress vs. hardness plot of the aged samples. Point size represents different timestamps.

3.3. Stressed Samples Characterization

The samples were investigated to assess their durability in harsh simulated environments. To mitigate the effect of aging time, only samples matured for more than 60 days underwent stress tests.

Wettability characterization sheds light on some changes, as follows: all of the stressed samples revealed a significant decrease in WCA, albeit retaining a hydrophobic behavior (Figure 8).





Figure 8. WCA, H, and SA values measured for the 60-day aged samples and stressed samples (ST in the legend).

By comparing different A:B ratios, the WCAs remain slightly higher for the 20:1-ttx and 30:1-ttx, with respect to the 5:1-ttx and 10:1-ttx ones. Noticeably, water mobility on 10:1-ttx proved to be almost unaffected by the stressing process, whereas 5:1-ttx exhibit a sensibly worse performance in both H and SA values.

Hardness measurements of the samples subjected to stress tests revealed noticeable differences, primarily observed among the different A:B ratios, as reported in Figure 9. Indeed, aged 5:1-ttx samples show a further significant increase in hardness of about 13% across all curing treatments. Stressed samples 30:1-ttx, 20:1-ttx, and 10:1-ttx display smaller hardness variations instead, especially for longer thermal treatments.



Figure 9. Hardness measured on the 60-day aged and stressed samples (ST in the legend).

Despite the slight deterioration caused by stress cycles, the samples still maintain good icephobicity, reducing ice adhesion by more than four times in comparison to the Al alloy reference (350 kPa), as reported in Figure 10. However, standard deviations leap to far higher values, suggesting that the polymer surface is less homogeneous after the stress tests.



Figure 10. Shear stress values measured for the 60-day aged samples and stressed samples (ST in the legend).

The change in the anti-icing properties does not exhibit a clear trend associated with the duration of the heat treatment or the A:B ratio. The 5:1-ttx and 30:1-tt3 samples experience the most significant ice adhesion increase, in line with their variation in hardness and wettability. On average, the 30:1-ttx coatings maintain the lower ice adhesion, especially the 30:1-tt7, which exhibits the best behavior with an ARF of 4.

4. Conclusions

In this work, the role of the precursor ratio and curing treatment for the anti-icing application of PDMS has been clarified, highlighting the importance of a fine-tuned formulation. Starting from commercial Sylgard 184[®], here we have demonstrated how the variation of the A:B ratios and polymerization conditions can have a profound effect on the elastomer hardness and also on the hydrophobicity, anti-icing, and durability of the materials.

Specifically, increasing the A:B ratio up to 30:1 gives rise to less cross-linked PDMS, resulting in more hydrophobic, softer, and more icephobic elastomers. However, ratios of 30:1 and 20:1 led to sticky surfaces with no water mobility. For these materials, even if lower shear stress values were observed, higher calculated W_a were systematically found, irrespective of the thermal treatments.

The main effect of the curing treatment was related to hardness, with longer heating treatments leading to higher hardness. The same correlation was found between shear stress values and thermal treatments, accordingly, with the hardness increase.

The monitoring of the hardness change over a period of 60 days showed its tendency to an increase over time, the extent of which was dependent on the A:B ratio and curing treatment. Generally, higher A:B ratios and prolonged curing treatments (tt4–tt8) show smaller increases in hardness. Among the samples belonging to the same A:B-ttx series, a clear correlation between the final hardness value and the type of thermal treatment applied can be traced.

The samples' durability was assessed with climatic chamber stress tests cycles. Thermal cycling, water spraying, and UV exposure were applied to the samples for 4 weeks.

All of the samples evidenced a deterioration in hydrophobicity, regardless of the A:B ratio and thermal treatment, although maintaining a hydrophobic behavior.

No systematic hardness variations were found, except for the 5:1-ttx samples, where it increased, on average, by ten points. Accordingly, the icephobicity of the stressed samples did not evidence meaningful variations, except for the 5:1-ttx, which showed an increase in

the shear stress values of about 40%. However, good icephobic properties were observed even after stress tests, confirming them to be promising materials for anti-icing applications. According to our experimental data, considering the results for the as-prepared, aged, and stressed samples, the most promising for icephobic application is 30:1-tt7.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/coatings14070901/s1, Table S1: Description of stress tests; Figure S1: Shear stress values within 60 days of aging; Figure S2: Hardness values within 60 days of aging.

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