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Adhesion mechanisms at the interface between two polytetrafluoroethylene (PTFE) films

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Résumé

Cette étude vise à déterminer la fenêtre de paramètres optimum (pression, temps, température) pour réaliser le soudage entre deux surfaces de PTFE et comprendre les mécanismes d'adhésion entrant en jeu. Les principaux mécanismes responsables de l'adhésion à l'interface polymère/polymère sont présentés puis leur caractérisation expérimentale est exposée.

Abstract

This study aims to determine the optimum parameters (pressure, time, temperature) to achieve welding between two PTFE surfaces and to understand the adhesion mechanisms involved. The main mechanisms responsible for adhesion of polymer-to-polymer interface are presented and then their experimental characterization is exposed.

Keywords: polytetrafluoroethylene (PTFE), films, adhesion, interface

1 Introduction

Polytetrafluoroethylene (PTFE) is a thermoplastic semi-crystalline polymer, which possesses exceptional chemical and physical properties including excellent resistance to corrosion, excellent temperature stability, high electrical insulation properties and extremely low friction coefficient. Consequently, this material is used for numerous applications. This work focuses on PTFE films and coated fabrics. Because of its high molecular weight and its high viscosity in the melted state, PTFE thin films are obtained by successive deposition of PTFE dispersions on a matrix which is then peeled out, see Fig.1a [1-2]. From these thin films, composite materials are manufactured by welding together a fabric sandwiched between two films, see image on Fig.1b. Therefore, for optimum performance, it is crucial to control PTFE-fabrics adhesion, resulting from the manufacturing process of these composite materials.

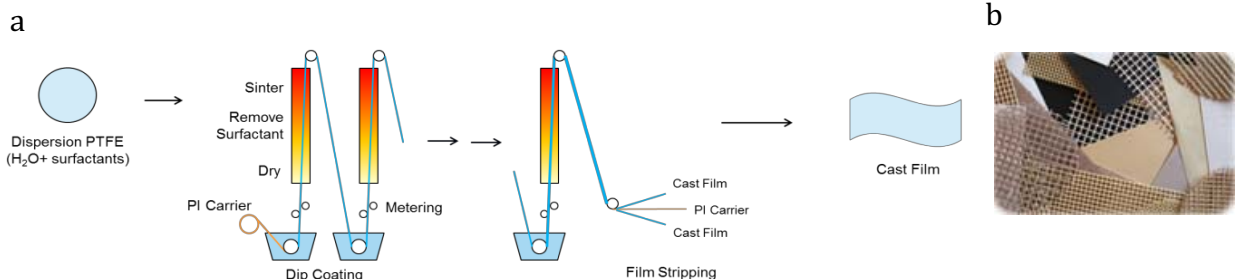


Figure 1. a) PTFE cast films manufacturing process, b) PTFE composite coated fabrics

This work focuses on a model system: two PTFE films without fabrics. The aim is to understand the adhesion mechanisms, determine the mechanical properties and deduce an optimization of films welding.

Here, the main mechanisms responsible for adhesion according to the literature are presented and then focus is on their characterization on our thin PTFE films.

2 Adhesion mechanisms literature review

For all welding processes, the healing of the polymer/polymer interface was described by Wool et al. [3] who identified three main sequential stages, namely: (i) surface rearrangement and surface approach; (ii) wetting; (iii) diffusion. In stage (i), the interface has no mechanical properties as the two distinct faces still exist. In stage (ii), the completion of the wetting stage marks the achievement of intimate contact between the two surfaces. Potential barriers associated with inhomogeneities or porosities at the interface have disappeared, and molecular chains are free to move across the interface in a process of inter-diffusion also called auto-adhesion (stage (iii)), which leads ultimately to the collapse of the interface.

2.1 Intimate contact

The wetting step is indistinguishable from the surface rearrangement one. As it seems that the time scale of wetting phenomenon is one order of magnitude less than surface rearrangement and molecular diffusion, most of researchers have gathered stages (i) and (ii) into one stage generally called intimate contact [4]. Micro-asperities and micro-valleys as well as more macroscopic surface waves lead to a non-perfect contact between the two parts to assemble. But, as commonly admitted in thermoplastic welding, intimate contact is the first step in forming a bond between two thermoplastic surfaces that have been brought together under both heat and pressure [5]. Surface topography can be evaluated by profilometry, but the prediction of topography evolution when the material is subjected to welding pressure appears overwhelmingly difficult.

2.2 Auto-adhesion

Auto-adhesion is the formation of bonds between two surfaces of an identical polymer in contact at elevated temperature. When two PTFE thin films are put in contact, their welding can occur thanks to different intrinsic phenomena detailed below [5, 6, 7]:

- **Chain inter-diffusion:** Inter-diffusion of macromolecules can occur when two polymer surfaces are brought into intimate contact, see sketch on Fig. 2b. Polymer chains are in a continual movement, called reptation. Inter-diffusion becomes more likely when the temperature is higher than the polymer glass transition temperature. It also depends on the polymer crystallinity, molecular weight, material viscosity, the degree of chain segregation, crosslinking and chain entanglement. So, for this phenomenon, an important parameter to determine may be the diffusion length required to achieve a bond strength equivalent to the one of the bulk materials.
- **Chain entanglement:** Polymer chain entanglement can occur between two polymer surfaces after some inter-diffusion has occurred, see sketch on Fig. 2c. The total density of effective chain entanglements is known to be dependent on the molecular weight distribution, on the contact time and physical properties at the interface. Further, only the relaxed un-entangled segment of the chains can cross the interface. Here, an important parameter is the depth of the mobile surface layer for interfacial entanglements.

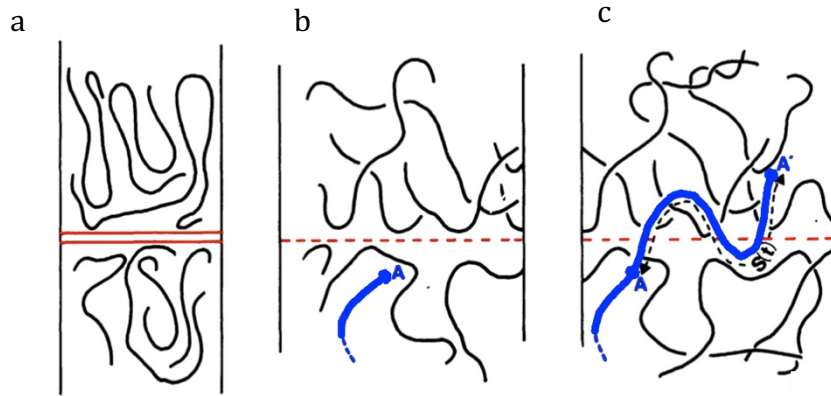


Figure 2. A microscopic picture of polymer-polymer welding. (a) The two blocks are put in contact. (b) Just after contact no chain crosses the boundary. (c) After some time, a chain in blue have reptated by a curvilinear length $s(t)$ and thereby establishes some bridging. Schematic redrawn from [5]

- **Interfacial co-crystallization:** At temperatures close to the melting temperature, crystallization may also occur simultaneously with inter-diffusion of polymer chains at the interface during auto-adhesion of semi-crystalline polymers, which will have great influence on the bonding strength development.

3 Adhesion mechanisms characterization

3.1 Intimate contact

The first necessary condition to obtain intimate contact between two films depends on the material surface roughness. Given the small topographic variation scale, atomic force microscopy is a more accurate tool than the mechanical profilometer. For all sintered films, the average roughness is about 30 nm whereas for films with an unsintered surface state, it is about 70 nm, see Fig.3. Taking into account the low roughness involved and the small film thickness (approximately 30 μm), a high contact pressure should make it possible to obtain a good intimate contact while avoiding porosities trapped at the interface.

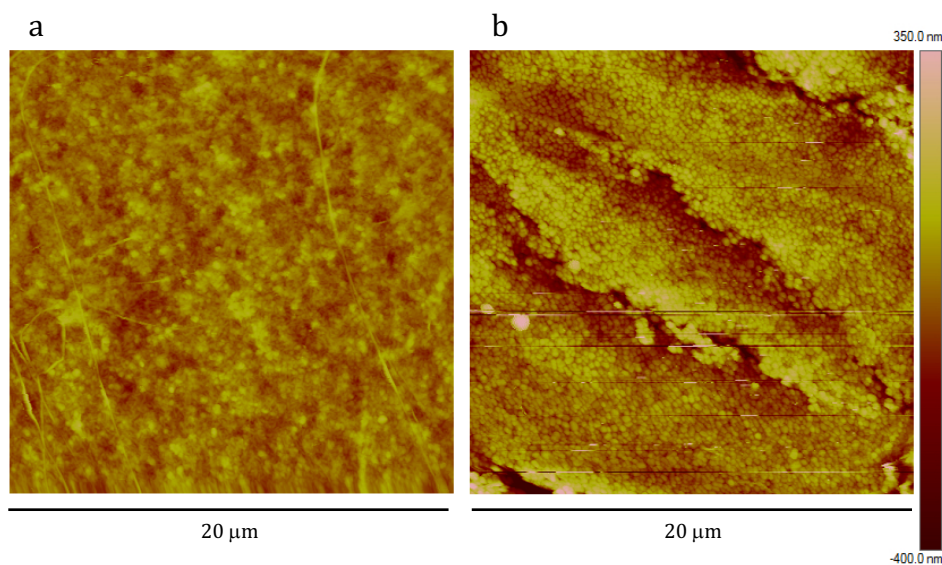


Figure 3. Topography of control films obtained by AFM on 20 μm zones a) sintered film, b) unsintered film

3.2 Inter-diffusion

As mentioned, at temperature higher than the melting temperature, polymer chains can move; this process is called chain inter-diffusion. By performing rheology, the material viscoelastic behavior can be measured with the storage and loss modulus as well as the reptation time.

The experiments performed are with a pseudo-bulk made from a 10-layer stack of 30 μm thick cast films reaching a total thickness of about 300 μm . The 2.5 cm diameter disks are placed in the rheometer (ARES with an oven) equipped with parallel plate geometry and sintered for 1h30 at 350°C with a normal of about 1 N. From this “pseudo-bulk” sample, frequency sweep tests in linear viscoelastic regime are performed at different temperatures above the melting temperature.

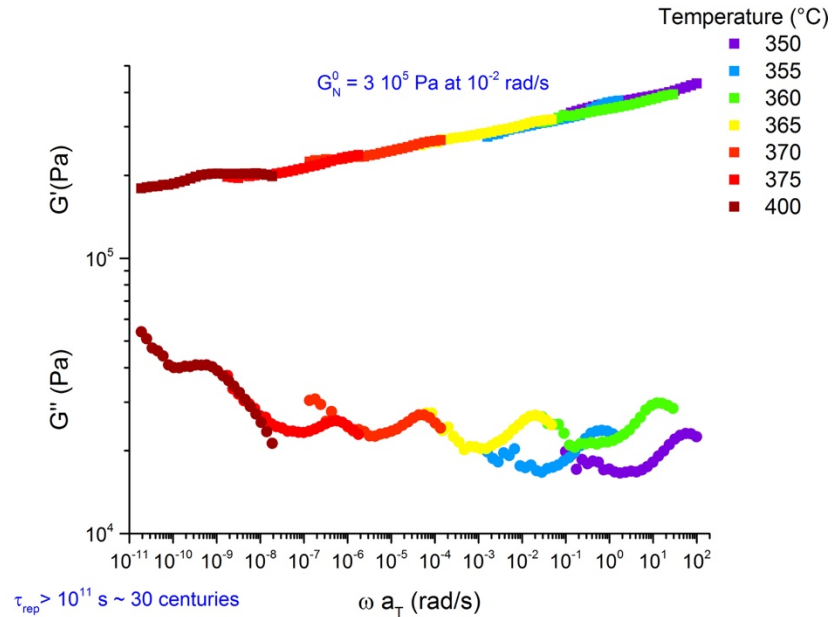


Figure 4. Time-temperature superposition master curve with reference temperature 350 °C of G' , G'' versus ωa_T ; for a “pseudo-bulk” made of 10 layers sintered together

By shifting the recorded curves, thanks to the time-temperature superposition principle, the master curve is obtained at the reference temperature of 350°C, see Fig. 4. The value obtained for the storage modulus is in agreement with previous data from the literature [8]. The crossover of the storage and loss modulus can be used to estimate the reptation time. Here, there is no crossover, the reptation time is larger than 10^{11} s or 30 centuries. Consequently, the chain inter-diffusion mechanism appears presumably irrelevant for adhesion at our time scale and hence co-crystallization becomes the most likely phenomenon responsible for adhesion.

4 Conclusion

After exposing the different adhesion mechanisms between two surfaces of the same polymer, intimate contact and chain inter-diffusion have been characterized on our thin PTFE films. From rheology experiments, inter-diffusion is not the main mechanism at our time scale. Further work is needed to estimate the macromolecular diffusion as a function of the welding parameters following Zhang et al. [9].

Mechanical adhesion test will be implemented in order to obtain a quantitative evaluation of the adhesion energy as function of welding pressure, time and temperature. The observation of the resulting fractured surface by optical microscopy, atomic force microscopy or electron scanning microscopy can be indicative of the presence of interfacial co-crystallization and we hope to obtain hints about interfacial crystallization morphology variation with welding parameters.

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