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Mechanics of Soft PSAs (Pressure Sensitive Adhesives)

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Abstract: The adhesive performances of a PSA (Pressure Sensitive Adhesive) are attributed to their viscoelastic properties. In this paper we analyze the viscoelastic behavior of different PSAs having substantially similar adhesive performance. Linear and non linear analyses were performed using small amplitude oscillatory shear tests and tensile stress-strain tests, respectively. It is shown that linear viscoelastic tests are useful to qualitatively characterize the adhesive performances. However, deeper knowledge can be achieved by non linear viscoelastic tests. The true stress - true strain curves are modeled by using a theory accounting for the interpenetration of micro-network and the linear polymer. It is shown that the same substantial in-service properties can be achieved with adhesives showing different fingerprints in terms of viscoelastic spectra.

Keywords: Pressure sensitive adhesives, tack, shear resistance, peel strength.

INTRODUCTION

Pressure Sensitive Adhesives PSAs are viscoelastic-elastomeric materials, combining simultaneously a liquid-like character to form good molecular contact upon application of light pressure in a short contact time, and solid-like character to resist to an applied stress once the bond has been formed [1-7].

The engineering performances of the adhesives under concern are determined by three non-standard measurements, that quantitatively ascertain their tackness, shear holding power and the peel resistance.

The adhesive tackness is measured by registering the force per unit area required to pull apart the adhesive and the backing previously subjected to low compressive forces in a small time interval. The shear resistance characterizes the behavior of the PSAs under the influence of long lasting stresses [8-13]. The peel strength is a measure of the debonding process and depends from the test geometry, temperature, adhesive thickness and nature of the backing [14-19].

Generally the properties of PSAs are related to their linear viscoelastic properties [20-22] but in reality the adhesive performances involve large strains which cannot be predicted easily by small stress/strain responses, i.e. creep or stress relaxation or by small amplitude oscillation tests [23-26]. The chemical nature [23, 24] and the thickness of the adhesive influence the adhesive performances as well as the surface conditions of the adherent including roughness and surface tension. In fact the surfaces of tape and backing and the bulk properties of adhesives effects are coupled and it would be more accurate to consider adhesive/substrate pairs than adhesives and substrates separately. Nonetheless, given the nature of the backing and the tape, this paper deals with the characterization of the bulk viscoelastic properties of two different adhesives, the work being addressed to the correlation between small strain oscillatory experiments and the stress-strain responses and eventually their influence on “in-service” properties.

Adhesion and tack of polymers are not fundamental material properties like the elastic modulus or the viscosity. They strongly depend on the test methods and the conditions of measurement. The adhesion performance is characterized by the adhesive fracture energy that is determined under well-defined conditions of bond formation and bond separation. Zosel [2, 11] developed an instrument that measures fracture energy and studied the deformation behavior during bonding and debonding. It was shown that the adhesion and tack are connected with the glass transition range of the polymer. The adhesive fracture energy exhibits a maximum in the temperature range above the glass transition region. In this temperature range, the mechanical behavior is determined by intermolecular interactions, so-called entanglements that form a network of temporary cross-links. High tack values require a good deformability of the polymer, i.e. a sufficiently low modulus, and that means the material must have an entanglement network with long chain molecules between two entanglements.

For instance, the formation and growth of fibrils during debonding seems to be crucial to the peel strength (the index of the resistance to bond separation) and the tackiness of polymers used as pressure-sensitive adhesives. Zosel [13, 19, 26] studied the influence of a number of parameters such as contact force and time, surface roughness, temperature, and rate of separation on fibrillation, to establish relations between the nucleation and growth of fibrils and the mechanical behavior as well as the molecular structure of the

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adhesives. Measurements of a large number of model polymers lead to the conclusion that the average mass between entanglements, Me, is an important factor governing fibrilation. Polymers with a high Me above about 1×10^4 g/mol show fibrilation whilst materials with an entanglement length below this limit debond by an apparently homogeneous deformation with lower-energy dissipation. However, Pressure Sensitive Adhesives contain some gel fractions (sometimes being the predominant amount of the mixture), dispersed in micro-domains somewhat entangled to the un-crosslinked phase.

Despite the complexity of peeling process, where the peel strength is generally dominated by large energy dissipation due to fibrils formation, it is frequently reported that the performances of PSAs can qualitatively be correlated to a selected window of their linear viscoelastic spectrum [17-20]. Instead, the peeling performances of PSAs subtend a rather high strain and strain rate process where the fibrils formation is generally preceded by cavitation phenomena [20-28], i.e. the formation of spherical voids inside the adhesive. The cavitation phenomenon is completely dominated by bulk relaxation modulus of the adhesive and arises due to the plain strain conditions that hold during the peeling just behind the peeling front. In many instances these phenomena dominate also in structural adhesives even if they lay in their glassy state [29-42]. Indeed, the intensity of the three-dimensional stress state during peeling is strongly dependent on the flexural stiffness of the tape, once the surface properties of the backing and the tape (namely, the roughness and the surface tension) and the adhesive thickness are established. The presence of voids allows the fibril formation due to the rapid transformation of the three-dimensional state of stress into a unidirectional tensile stress states to within the adhesives parts confined by voids. However, the process requires deeper micro-mechanical analysis and doesn’t represent the focus of the present work. Furthermore, the sticky behavior and the shear holding power of PSAs can be also gathered empirically selecting frequency intervals to within the master curve obtained by shifting the isothermal dynamic mechanical tests to a reference temperature by means of the frequency/time-temperature superposition principle. Thus, the “in service” properties of PSAs are correlated at least to two independent viscoelastic functions, namely the shear and the tensile relaxation moduli.

The master curves can be used as the finger print of a given adhesive formulation. Therefore, in the industrial practice the formulation changes are triggered to modify the viscoelastic spectrum according to the desired in-service performances [1, 2, 6, 16].

In this paper the linear and non-linear viscoelastic properties of two different adhesives exhibiting equivalent performances are analysed by means of standard small amplitude oscillatory tests (SAOT) and stress-strain curves. The PSAs under study can be considered as rubbers with entanglements [41-44] where a sort of polymer network with entanglements coupling is realized. Therefore the elasticity of polymer networks, i.e. the stress-strain response will be analysed in the framework of a theory explicitly accounting for both cross-links and entanglements. Both the rheological and adhesive properties were expected to be very dependent on the initial latex particle size distribution and this aspect is also outlined in this paper. This work represents also a background study to verify the effect of rigid nanoparticles on the overall behavior of latexes with entanglements [31-36].

MATERIALS

The samples under study belong to two different classes of water-borne PSAs commercially available. In both cases the adhesives have been obtained *via* semi continuous emulsion polymerization, producing latex. Sample A is based on 2ethyl-hexyl-acrylate latex 75%, vinyl acetate 22.5%, acrylic acid 2,5%. The dynamic light scattering(DLS) technique revealed a broad distribution of particles dimensions with a characteristic particle diameter centered at about 1 µm. The total micro networks content was obtained extracting thesol phase. The measured gel fraction was roughly 0.70.

The molecular weight, Mw, of the extracted soluble polymer phase was 1200000 Daltons and the polydispersity index, PDI=1.4 (as determined by Gel Permeation Chromatography). The Peel Strength of Sample A is PS=300 N while the Shear Holding Power, SHP=24hours.

Sample B is based on Buthyl-acrylate 88,5%, Methyl-Methacrylate 10%, and acrylic acid 2,5%. Again DLS measurements revealed a broad distribution of micronetworks dimensions with a mean diameter of 0.350 µm. The gel fraction was 0.70. The molecular weight of the extracted soluble polymer phase was 2000000 Daltons and the polydispersity index, PDI=1.3. The Peel Strength is PA=390 N and the Shear Holding Power is SHP>24h.

The calorimetric glass transition temperatures, of Samples A and B are -36.5°C and -38°C, respectively, as measured by DSC (Differential Scanning Calorimetry) at a heating rate of 10°C/min from -100 to +50 °C.

The adhesive film forms in both cases upon water evaporation, when the latex particles stick together *via* inter-diffusion of linear chains permeating the gel phases. The PSAs under study can be considered as rubbers with entanglements, where a sort of polymer network with entanglements coupling is realized.

EXPERIMENTAL

The linear viscoelastic spectra of adhesives under study were obtained using a Rheometric SR 500 dynamic mechanical analyzer (DMA). The isothermal small amplitude-oscillatory tests were performed with a temperature step of 5 °C, from -40 up to 150°C. Disc shaped samples with roughly 2 mm thickness and 25 mm diameter, as measured at room temperature, were used.

The stress-strain experiments have been carried out using the Zwick/Roell Z010 dynamometer equipped with a load cell of 10 N. Rectangular samples of 50x6x0.5 mm^3 were utilized in stress-strain tests conducted at different constant crosshead speeds, from 5 to 500mm /min, at controlled temperature T=25°C. Samples were clamped thanks to their sticky characteristics rather than imposing clamping forces. In both experiments the specimen thickness was reached by stacking several adhesive films previously poured on a silicone paper and dried for 24 hours at 25 °C.

RESULTS AND DISCUSSION

The PSAs under study contain a dispersed crosslinked phase permeated and entangled by the same uncrosslinked polymer that represents the suspending phase. Therefore each micronetwork particle is a rubber with entanglements and this justifies the use of the Ball *et al.* equation [41-44]. The schematic of the adhesives structure is represented in Fig. (**1**). It is well known that many parameters are responsible of the in-service performances of PSAs, including the thermodynamic and the surface finishing properties of baking and the tape and the bulk properties of the adhesive like the chemical formulation of the base polymer, the size distribution of gel particles, the molecular weight and branching of the uncrosslinked polymer and the composition (namely the percent addition of inherently tacky, low molecular weight substances) [1-3, 11-13, 18, 19, 23, 27].

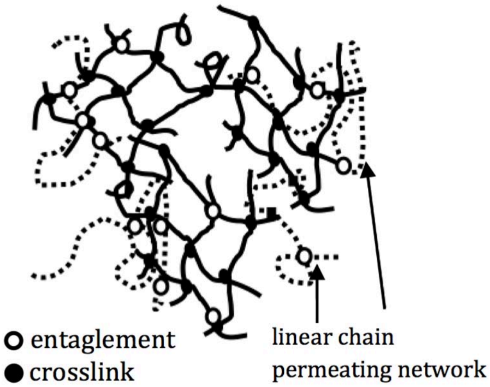

Fig. (1). Schematic view of PSA microstructure.

All the above characteristics regulate the time dependent responses of adhesives, and finally the three main adhesive performances, namely: shear holding power, tackiness and peeling strength. Despite that the "in-service" properties of adhesives are not material properties, they reflect somehow the mechanical responses under small (linear) and large (non linear) deformation.

For instance tack properties are correlated to small strain and low rates of deformation, while during peeling the mechanical stress in play is so high that may induce cavitation with fibril formation where very high strain rates are observed. However, while the linear viscoelastic spectra of homopolymers are quantitatively correlated to their structure (for instance the relaxation time spectrum correlates quantitatively with the molecular weight distribution) in the case of heterogeneous systems like microgels interpenetrated networks the viscoelastic spectra represent only the finger print of given system under study with scarce insights into the system architecture (namely: the amount and microgels size distribution and the entanglements and crosslinks molecular weights). That is due to the multiple subtle interactions among the polymer permeating the gel, the gel fraction itself and the low molecular weight sticky substances [31-40].

Based on these considerations, we focus our attention on both linear and highly non linear viscoelastic responses in order to extract at least qualitatively some correlations with the "in service" performances and the adhesive architecture. Thus, in agreement with the most of the literature around the subject we first check the linear viscoelastic responses in shear in order to select the time/frequency windows where the viscoelastic functions, based on phenomenological criteria, are associated to the "in service" properties. Then non linear responses are checked in tension owing to the fact that, actually, the adhesives under study show strong cohesive failure that manifests with the formation of fibrils after they are triggered by cavitation processes as schematically represented in Fig. (**2**).

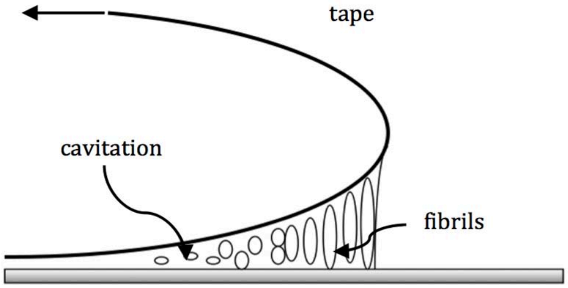

Fig. (2). Schematics of fibril formation during 180° Peel test. The extent of fibrils elongation depends on the tape stiffness that regulates the intensity of three-dimensional stress to within the adhesive.

The linear viscoelastic spectra of samples A and B are reported in Figs. (**3**, **4**), respectively.

The isothermal data, in terms of in phase and out of phase shear moduli, G'and G"were obtained spanning the frequency from 0.1 to 100 rad/s. Linearity was checked at the highest frequency varying the oscillation amplitude to the thickness ratio from 10^{-4} up to 1. The actual amplitude at each temperature was selected to be well within the instrument sensitivity, in the linear range. Then, the time-temperature superposition was adopted to obtain the master curves, both in terms of G' and G'', selecting T=20°C as the reference temperature. In analogy with a procedure commonly adopted for homopolymers, we identify a "plateau modulus" G_N^0 in our systems that is the G' value at the frequency corresponding to the minimum of Tanδ(G"/G')[3, 4]. Accordingly, we estimate G_N^0 = 0.022 MPa for sample A, and G_N^0 = 0.062 MPa for sample B. The above values are somehow representative of the equilibrium "strength" of our networks, accounting for but not limited to the presence of topological constraints at molecular level.

The two samples show completely different viscoelastic response, as denoted both by their different G_N^0 values and from the crossing of G" and G' data of Sample B at low frequency, a case not encountered in Sample A. Instead, the in-service properties as described in the materials section are almost equivalent. However this occurrence is not unexpected in the light of the discussion above. For the sake of a better understanding, the viscoelastic response of the materials under study was checked in tension roughly simulating the state of uniaxial stress to which fibrils are subjected once they are formed. In this respect, let us mention that there exists a wide literature production around the subject of uniaxial response of networks with entanglement [41-44]. The relevant molecular molecular models for crosslinked systems with entanglements are based on the microscopic picture of the entropic elasticity of chains that are responsible of the fact that, on macroscopic length scales, polymer networks deform as elastic solids. Among the other, two substantially equivalent approaches are mostly utilized. The first one describes the dependence of the stress on the elongation ratio λ for uniaxial deformed networks using Rubinstein and Panyukov non-affine slip-tube (NAST) model of elasticity [42-44]. Similarly, in the second approach, referred to as the Ball theory [41], the correlation between tensile stress and the stretching ratio comes out modeling the adhesive as a network permeated by linear chains. It is assumed that the strength of the network benefits simultaneously by both crosslinks and entanglements. However the entanglement effectiveness is correlated to the crosslinked network density. The Ball theory is adopted in this paper, leaving to a further study the comparison of the two approaches. Written in terms of true stress, σ, andstretching ratio, λ, the Ball *et al.* theory assumes the following form:

$$\sigma = \frac{\nu kT}{2}\left[\varphi_{CL}\left(\lambda^2 - \frac{1}{\lambda}\right) + \varphi_{ENT}\left(\frac{\lambda^2(1+\eta)}{\left(1+\eta\lambda^2\right)^2} + \frac{\lambda^2-\lambda}{(\eta+\lambda)^2} - \frac{1}{\left(1+\eta\lambda^2\right)}\right)\right] \quad (1)$$

whereν accounts for the network density as in the classical rubber theory, φ_{CL} and φ_{ENT}=(1-φ_{CL}) are the crosslinks and the entanglements fractions, η is measure of the entanglements mobility to within the gel network. However, it must be emphasized that in the above cited theories the network is the continuous phase, while our system the microgel particles are, in fact, the dispersed phase. This aspect will be highlighted in the next section where the stress/stretching ratio tests will be discussed.

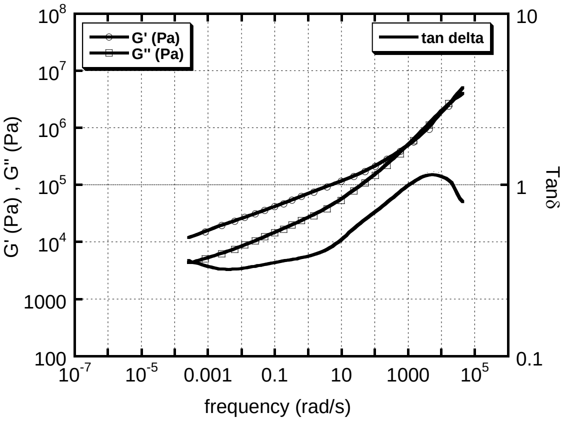

Fig. (3). Master curve of Sample A. Reference temperature T=20°C.

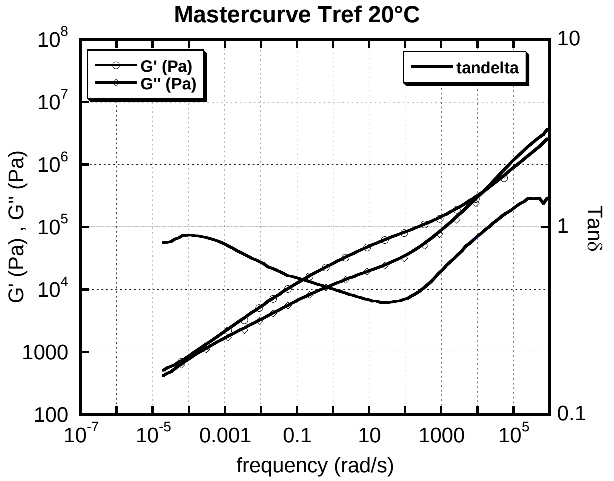

Fig. (4). Master curve of Sample B. Reference temperature T=20°C.

The uniaxial stress-stress behavior reflects the extent of fibrils elongation during peeling, including their stable elongation flow, which is acted by voids nucleation (i.e. the cavitation extent) a condition actually correlated with the bulk relaxation modulus. In Figs. (**5**, **6**) the experimental results in terms of true stress-stretching ratio are reported for two adhesives at different crosshead speed. The solid lines represent the curve fitting based on the Ball theory expressed by equation 1. In peeling tests the strength of adhesives is strictly correlated to its ability to form fibrils upon cavitation occurs. As expected samples A and B show completely different behavior even if the in service performances, and in particular the peel strength for sample A is little lower then for sample B. Instead, higher stresses are required to stretch sample A compared to sample B.

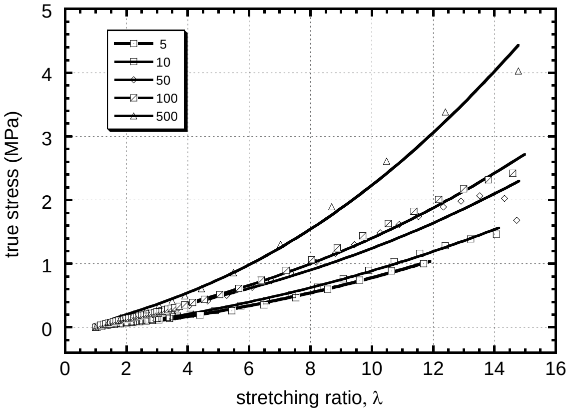

Fig. (5). Sample A: Uniaxial stress-strain tests at different crosshead speed (mm/min) as indicated in the inset. Symbols: experimental data (only part of the acquired data are represented for the sake of clarity). Solid lines: curve fitting according to Ball theory.

The above behavior is not unexpected even in the light of the linear viscoelastic behavior of Samples A and B.

To illustrate, let us limit our discussion to the initial (linear) stretching rates, SR (SR=crosshead speed/sample length), used in tensile experiments. It can be easily recognized that SR spans between 0.17 and 0.0017 s^{-1}, as the crosshead speed spans between 5 and 500 mm/min and the initial sample length is 50 mm. Selecting a similar window of shear oscillation rate, in linear viscoelastic spectra of Figs. (**3**, **4**), it appears that the elastic component of Sample A is almost one order of magnitude higher than that of Sample B.

The rheological equation adopted is strictly dependent to supramolecular parameters such as the entanglement molecular weight, extent of crosslink and so on. This can allow us to correlate the molecular parameters of the system to its flow behavior. For instance, the entanglement coupling permits significantly long-range motions of segments of polymer chain and affects especially the behavior of polymer networks. Indeed, the rubbery plateau, that we checked by using the method utilized for homopolymers, appears only when the polymer contains entanglements. To illustrate, we are reporting the strain rate dependence of φ_{CL} and ηthat even if the Ball theory assumes constant parameters as the theory doesn't account for any time-dependent phenomena. We assume that φ_{CL} in our systems is not the real crosslinks fraction but the fraction of links that act as crosslinks. Accordingly, as φ_{CL} increases the sliding parameter suffers restricted motion and should decrease as effectively shown in Fig. (**7**).

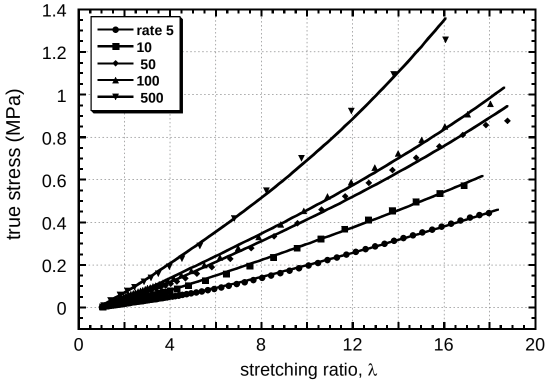

Fig. (6). Sample B: Uniaxial test at different crosshead speed (mm/min) as indicated in the inset. Symbols: experimental data (for the sake of clarity only part of the acquired data are represented). Solid lines: curve fitting according to Ball theory.

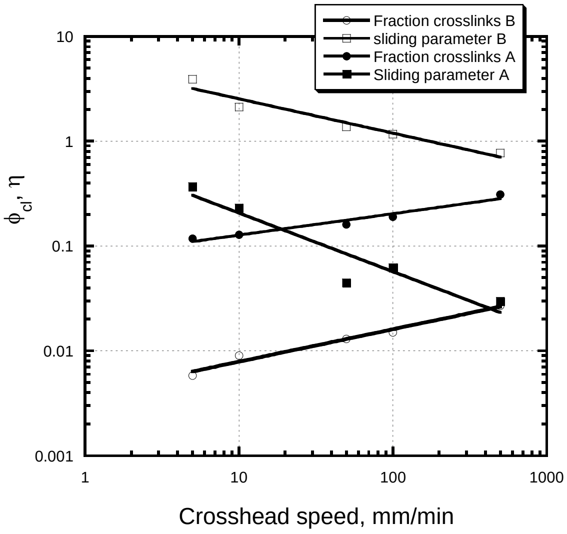

Fig. (7). The effect of stretching rate on the parameters of Ball theory. Symbols (as indicated in the inset) come out from best fitting the stress/stretching ratio data with equation 1. Lines: guide to the eye.

CONCLUSIONS

The PSAs performances depend on both the shear and the tensile relaxation moduli and eventually their relative placement. Chemical modifications should be driven accounting for both the molecular architecture of gel network and the length and the number of linear chains permeating the networks, actually responsible of the disentanglement rates.

Finally, the adhesive properties of two multimodal latexes with different particle size distributions were investigated. Both gave significantly higher adhesion energies and clear evidence of a fibrillar detachment process. This important result suggests that the spatial distribution of gel domains in the dry film and the molecular connectivity between those gel domains also play an important role in controlling its adhesive properties.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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