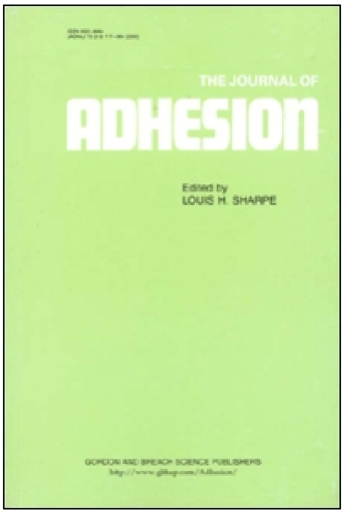

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Determination of Quasiequilibrium Work of Adhesion of Elastic Coatings†

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INTRODUCTION

Present methods for the determination of adhesion bonding of elastic polymeric materials entail certain experimental difficulties. In particular, the necessity of strict centering of the test specimen, and the difficulty associated with application of a homogeneously distributed stress over the whole cross-sectional area (homogeneous detachment or shear), or the excessive expenditure of work resulting from polymer deformation (peel).[1, 2]

We are suggesting a method to determine the quasi-equilibrium work of adhesion during the peeling process for elastic polymeric coatings, the value of which, as was demonstrated experimentally, does not depend on the coating thickness, deformation or rate of peeling.

EXPERIMENTAL

The method is based on the use of roller adhesion meter.[3] The basic construction features a roller from which the coating is peeled at a constant angle. The coating is cast onto the roller from solution or polymer melt. The apparatus provides plots of the work of detachment as a function of rate and allows microscopic examination of the processes occurring at the detachment boundary.

The test roller of the adhesion meter is actually a cylinder, prepared from various materials, and is mounted on roller bearings. The surface of the roller is polished until a highly reflecting surface is achieved and further cleaned with organic solvent.

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The construction of the adhesion meter is schematically shown in Figure 1. The coating under investigation 1 is cast on the test roller 2, which is mounted on roll bearings 3. These secure free rotation of the roller necessary for maintaining a constant position of the surface and angle of detachment (90°) with respect to the delaminating force. The test roller is hung on dynamometer 4. Before conducting measurements, a strip is cut across the hardened

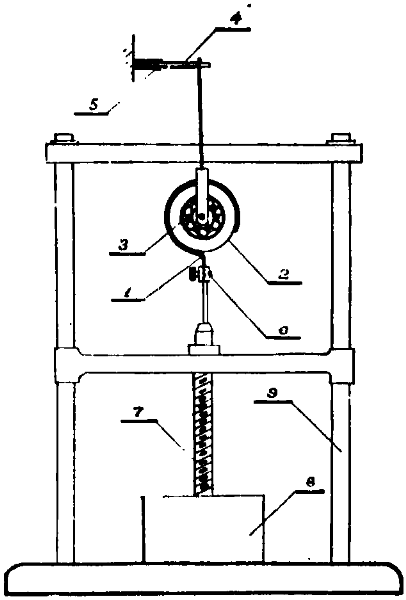

FIGURE 1 Adhesion meter. 1, Polymer film; 2, test roller; 3, bearings; 4, steel plate, dynamometer; 5, sensor; 6, clamp; 7, worm-pair; 8, gear box; 9, guide.

coating having a known width. One end of the strip is then directed to clamp 6 which can be displaced at varying speeds with the help of worm-pair 7, gear box 8 and guide 9.

The dynamometer part of the instrument allows the continuous recording of the peeling force.

RESULTS AND DISCUSSION

Figure 2 shows a test run at known peeling rate. At first a continuous increase of the force *AB* is observed, then it remains almost constant—*BC*. This force corresponds to the peel stress at a given rate of detachment (P_v). The work of adhesion of the coating is determined from the average stress values at detachment relative to unit width of the film coating.

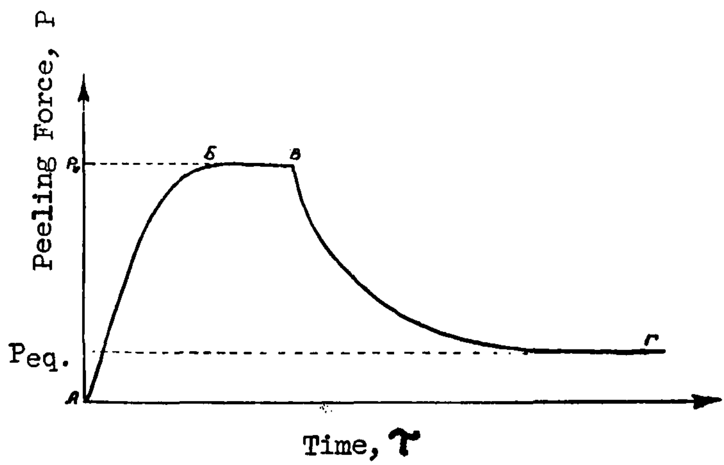

FIGURE 2 The change of peeling force as a function of time at a given peeling rate.

Table I shows the results of work of adhesion determined at different peeling rates for polyurethane films based on a copolymer of tetrahydrofuran with 25% polyoxypropylene (molecular weight 1200) and adduct of toluenediisocyanate with trimethylpropane. The density of the spatial network of the film coatings investigated could vary, depending on the ratio of components used for the synthesis.

As seen in Table I, the value of quasi-equilibrium work represents 10–40% of the work of adhesion determined at the limiting rate of detachment. If we examine the data obtained at different speeds of detachment, it is seen that the work of adhesion depends on the rate of peeling and increases with

TABLE I

Work of adhesion for polyurethane films as a function of network density and peeling rates

Substrate	Ratio NCO/OH = 2:1				Ratio NCO/OH = 4:1				Ratio NCO/OH = 2:1 with 50% TMP (trimethyl propane) additive			
	Work of adhesion × 10^{-5} erg/cm²			Quasi-equilibrium work of adhesion × 10^{-5} erg/cm²	Work of adhesion × 10^{-5} erg/cm²			Quasi-equilibrium work of adhesion × 10^{-5} erg/cm²	Work of adhesion × 10^{-5} erg/cm²			Quasi-equilibrium work of adhesion × 10^{-5} erg/cm²
	1[a]	2	3		1	2	3		1	2	3	
Steel	4.70	3.70	2.90	0.47	6.90	6.00	4.80	1.97	9.00	7.30	6.00	2.25
Brass	4.50	3.75	2.65	0.34	6.50	4.90	4.50	1.50	12.50	11.50	7.80	—
Duralumin	3.30	2.65	2.00	0.34	3.15	3.00	2.90	0.75	3.50	3.30	3.20	0.67
Glass	2.90	2.90	2.00	0.27	2.60	2.45	2.20	0.30	2.80	2.60	2.40	0.59

[a] 1, 2, 3—Work of adhesion corresponding to peeling rates of 0.3, 0.1 and 0.25 cm/sec, respectively.

increase of film (coating) thickness (Figure 3, curve 1). This effect is usually observed when the peel method is used.[4] The increase in the work of adhesion takes place until a given film thickness is achieved, and in every case, this increase is a function of the composition of the boundary layer and polymer properties: elasticity, mechanical rigidity, fluidity, etc. This effect is related to the stresses created during bending and the deformation of the film along its thickness at the point of detachment from the substrate. The work expanded during film deformation will increase with the increase of rate of deformation, thus increasing the value of the work of adhesion.[5]

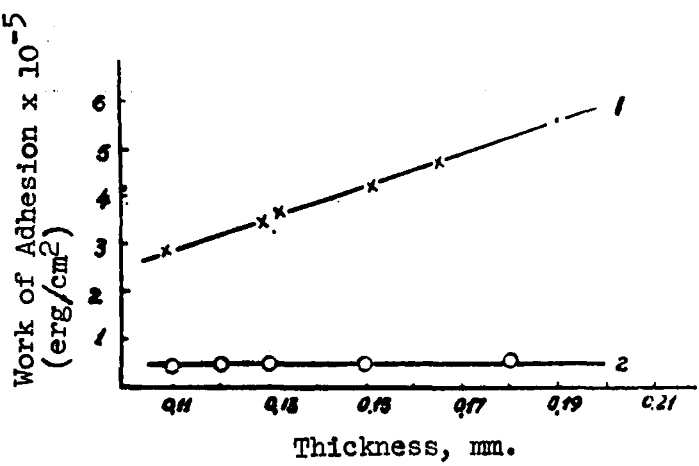

FIGURE 3 Dependence of the work of adhesion on film thickness for steel substrate (NCO/OH = 2 : 1). 1, Peel rate 0.025 cm/sec; 2, quasi-equilibrium work of adhesion.

Thus, in order to obtain comparative values for the work of adhesion, it is necessary to study films of equal thickness and at equal peeling rate. However, methods for obtaining films of equal thickness entail many experimental difficulties. Moreover, in case of evaluating the adhesion of different films, or even films of a similar nature but varying in network density, the use of films of equal thickness does not always provide comparative results, since the mechanical properties of these films are different and the work expended during the deformation process at the point of detachment will be different.

We suggest a different evaluation method which eliminates the aforementioned shortcomings (Table I).

If, following the determination of the peeling force at given peeling rate, the clamp displacement is stopped, then as a result of deformation relaxation, the stress created in the film will detach it from the test roller. This occurs because a deformed film tends to return to its initial size. The peeling process will continue until the stresses in the film are equalized by the interaction forces between the test sample (film) and substrate surface (adhesion forces) CD (Figure 2), then the peeling process ends (Point D, Figure 2). We designate the value of residual stress per unit film width as the quasi-equilibrium work of adhesion. This characterization can be considered as the work of adhesion at zero peeling rate.

In contrast to the usual values of the work of adhesion at different peeling rates, the value of quasi-equilibrium work of adhesion, determined by us using the above described method, for polyurethane films based on oligo-ethers does not depend on film thickness and initial rate of detachment as seen in Figure 3 and Table II.

TABLE II

Change in the work of adhesion as a function of peeling rate

Substrate	Peeling rate cm/sec	Work of adhesion $\times 10^{-5}$ erg/cm^2	Quasi-equilibrium work of adhesion $\times 10^{-5}$ erg/cm^2
Steel	0.3	6.84	0.65
	0.1	4.68	0.60
	0.025	3.73	0.54
Brass	0.3	5.98	0.97
	0.1	5.10	0.98
	0.025	3.04	0.98

Thus, the value of quasi-equilibrium work of adhesion after eliminating effects dependent on film thickness, its deformation, rate of peeling and the related electrostatic phenomena characterizes more correctly the value of molecular interaction at the surface boundary. Consequently, the determination of the value of the quasi-equilibrium work of adhesion represents a more accurate characterization of the interaction of polymeric films with solid surfaces than the normally determined values for work of peeling in case of elastic films (coatings).

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