

Peel Adhesion Apparatus Using the Compound Pendulum and Its Practical Performance

HIROYOSHI OBORI,¹ MITSURU TAKENAGA,^{2,*} and AKINARI KASAI²

¹Department of Research & Development, Teraoka Seisakusho Co., Ltd., 1-4-22 Hiromachi, Shinagawa, Tokyo 142, Japan and ²Department of Applied Physics, Faculty of Science, Science University of Tokyo, 1-3 Kagurazaka, Shinjuku, Tokyo 162, Japan

SYNOPSIS

An apparatus for measuring the peel adhesion behavior is developed on the basis of the circular motion of the compound pendulum. The peel force versus peel rate characteristics at the increasing and decreasing rates of peeling can be measured in a half cycle of rotation of the pendulum. A personal computer plays the important roles of operating the automated apparatus and processing data in real time. To test practical performance of the apparatus, the relation between peel force and rate of peeling was investigated for various pressure-sensitive adhesive (PSA) tapes over a wide peel rate range. The respective curves in peel force versus peel rate in the increasing and decreasing rate processes are consistent with each other in the rate regions where cohesive or interfacial failures occur; while in the transition region between their failures, it appears that a peel hysteresis exists. Furthermore, the conventional testing under constant rate was repeated, and good agreement with the results from the present apparatus was obtained.

INTRODUCTION

Peel adhesion properties of pressure-sensitive adhesive (PSA) tapes have been reported by several investigators¹⁻⁶ using the conventional tests of constant rate or constant load. Fukuzawa² reported from the results of the peel testing under the constant rate that cohesive failure takes place at low pulling rates and interfacial failure at high pulling rates, and also that a peak of the peel force appears in the intermediate region where the transition from one failure to the other occurs. The existence of a similar peak was reported by Gent et al.³

In constant rate testing, stick-slip phenomenon has been observed for PSA tapes; from the viewpoint of viscoelasticity, the stick-slip behavior occurs when adhesives change from rubbery to glass states on peeling. Aubrey et al.⁵ reported that such behavior sometimes appears when viscoelastic properties of adhesives change from viscous to rubbery states or vice versa.

Andrew et al.⁷ have recently developed a modified peel test method (soft-machine testing) in which a spring was inserted between the test machine cross-head and the peeling strip, so the pulling rate was determined only by the adhesive properties and the instantaneous load. In tests using PSA, this soft machine gave results that differed significantly from those obtained with a conventional testing of constant rate.

The purpose of the present study is to develop the peel adhesion apparatus using the rotation of the compound pendulum. Furthermore, to test practical performance of the apparatus, the peel force as a function of peel rate is measured in two kinds of processes, for increasing and for decreasing rates of peeling.

APPARATUS

Figure 1 shows the schematic diagram of a peel adhesion apparatus constructed of four measuring systems: The first is a mechanical system in which the torque of the compound pendulum is trans-

* To whom correspondence should be addressed.

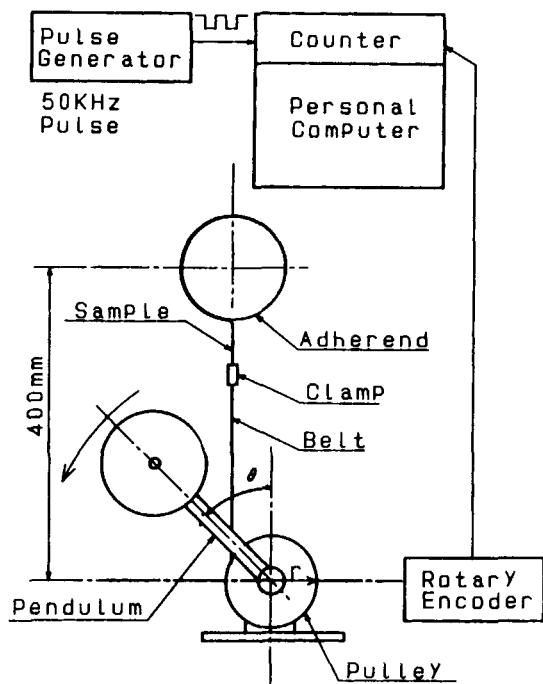


Figure 1 Schematic diagram of peel adhesion apparatus. The radius r of the pulley is 15 mm. The direction of the pendulum rotation is counterclockwise.

formed via the pulley into a tensile force for application to the sample. The second is the drum-type adherend to which tapes as samples are adhered; the third is a rotary encoder to detect the rotational angle of the pendulum; and the fourth is the computer system to operate the automated apparatus and to calculate the peel force and the peel rate. The procedure of the measurement is briefly given here: At the outset, tapes are adhered to glass adherends; then the pendulum is allowed to rotate from the highest potential position by release of a stopper. The variation in rotational angle is measured with elapsed time; the details of this variation depend on the force balance between the motion of the pendulum and the peel behavior of the tapes. The data is stored in memory and processed in real time for displaying the peel force versus peel rate relation.

Mechanical System

A side view of the mechanical system is shown in Figure 2. The rotational shafts of the compound pendulum, the pulley, and the rotary encoder are coaxial. The torque of the pendulum is transformed via the pulley into tensile force, which is applied to the belt connected to the sample by the clamps. The moments of force and inertia of the pendulum can be varied by changing the mass attached to it. The

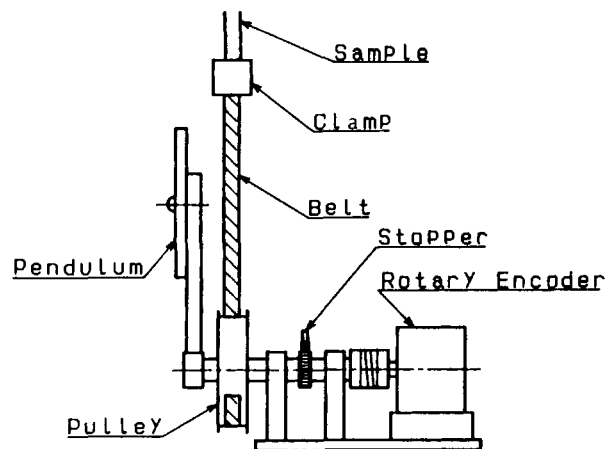


Figure 2 Side view of peel adhesion apparatus.

stopper consisting of a set of gear and pawl is used for holding the sample fixed or for starting the run by the release of the set.

Adherends

Two sorts of adherends, made of 2-mm-thick piece of Pyrex glass, were provided; for 90° peeling, a drum with the size of 120 mm diameter and 50 mm wide (Fig. 1), and for 180° peeling, a plate with the size of 50×120 mm (Fig. 3). To realize the 90° peeling approximately, a drum with a sufficiently large radius was used in comparison with the curvature radius at the separation point and the distance between the center of the pulley and the point.

Rotary Encoder

The rotational angle was measured by means of a rotary encoder that can generate 600 pulses per rotation, i.e., having the resolution in angle of 0.6° ; the initial torque is 2.5×10^{-4} Nm. A universal joint was used for connecting the rotational shaft of the encoder with that of the pendulum.

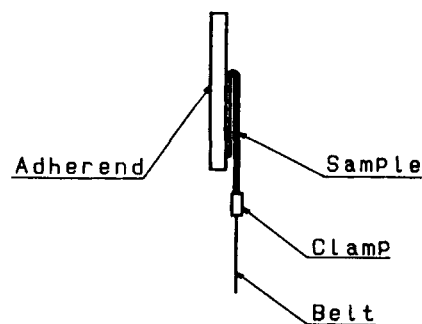


Figure 3 Plate device for 180° peel testing.

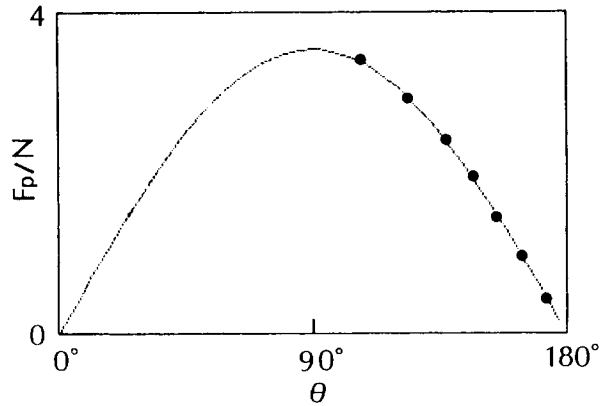


Figure 4 F_p as test weight against rotation angle θ in the uniform circular motion of the compound pendulum. Solid curve, calculated from $C_2 \sin \theta = F_p$ with $C_2 = 3.5$ N; solid circles, experimental.

Computer System

The estimate of the peel force and peel rate from the results of the rotational angle with respect to time is performed using the computer system. This consists of a personal computer with a 10-MHz clock pulse, two integral counters, and a 50-kHz pulse generator. One of the counters, which has a reversible function, is used for detecting angle variation, and the other is used as a timer for counting 50-kHz pulses. The encoder generates trigger pulses necessary to input the data into computer memory; it notes that the actual data is input as the time elapsed in the regular angle interval of 0.6° .

Motion of the Compound Pendulum

The equation of motion of the compound pendulum in the presence of tapes adhered to adherends is

$$C_1 \frac{d^2\theta}{dt^2} + C_2 \sin \theta = F_p \quad (1)$$

where C_1 and C_2 are the constants relating to the moments of inertia and force of the pendulum, respectively; the constants are empirically determined

later, θ is the rotational angle from the highest position of the pendulum, and the variation of θ as a function of the elapsed time t is associated with the peel force F_p and the peel rate at each instantaneous time for the adhered tape. The first term in the left side of Eq. (1) acts as the internal force and the second as the driving force of peeling.

If uniform motion of the pendulum is assumed, since the internal force term is neglected, it holds that $C_2 \sin \theta = F_p$. In order to estimate C_2 under such conditions, the moment of force of the pendulum was balanced with a test weight equivalent to F_p attached to the pulley. Figure 4 shows the different test weights F_p against the rotational angle θ , in the case of a pendulum mass of 50 g. It is obvious, as shown in the solid curve in Figure 4, that the experimental results follow the expression $C_2 \sin \theta = F_p$ with $C_2 = 3.5$ N. Values of C_2 were in the range of 1–6 N, dependent on the mass attached to the pendulum.

The constant C_1 was estimated from the circular motion of the pendulum in the absence of samples; the variation of θ in the range of $\theta = 0^\circ$ to 180° with elapsed time was measured. From Eq. (1) with $F_p = 0$ and using C_2 estimated above, values of C_1 were estimated in the range from -0.02 to -0.06 Ns^2 , dependent on the pendulum mass attached; for example, $C_1 = -0.051$ Ns^2 at the pendulum mass of 50 g.

The rate of peeling v_p at the instantaneous time is obtained from the variation of θ against t :

$$v_p = \frac{r d\theta}{dt} \quad (2)$$

where r is the radius of the pulley. The actual estimate of the peel rate is made from the time period necessary to rotate by 0.6° of regular angle interval. After the pendulum was released from the highest potential at $\theta = 0^\circ$, it is obvious that the peel rate increases with change in θ , and reaches a maximum value near $\theta = 90^\circ$, and then decreases until $\theta = 180^\circ$; however, the manner of the change in v_p is complicated because of cooperative interaction with the peel force of the peeling tape.

Table I Characteristics of PSA Tapes Used

Samples (PSA Tapes)	Adhesives	Thickness of Adhesives (μm)	Backing of Tapes*
A	Crosslinked (acrylic polymer)	30	PET
B	Noncrosslinked (rubber)	30	PET

* Thickness is 25 μm .

The influence of stretching of the belt on the peel rate may be regarded to be negligible within an error of at most 10%; let us consider the case that the maximum value in tensile force used in the present study, i.e., 6 N, was applied to the belt; then a distortion of about 0.016 mm in regular angle interval of 0.6° resulted. Since the peel length per angle interval is equal to 0.16 mm considering the diameter of the pulley used, the error in the peel rate is estimated to be less than 10%, if the influences due to creep of the belt and tape were not considered.

EXPERIMENTAL

The pressure-sensitive adhesive (PSA) tapes used had a backing of polyethylene terephthalate (PET) films of 25 μm thick, with two types of adhesives: crosslinked (acrylic polymer) and noncrosslinked (rubber). The sample tape was cut into 10-mm-wide strips. Table I gives the brief specification of the tape samples used. The measurement was carried out at room temperature, in a half cycle of the rotation of the pendulum.

The surface of adherends was wiped with a soft cloth impregnated with toluene, then washed with a water-detergent mixture, and afterward under a flow of water, and finally dried in a warming furnace. The PSA tapes adhered to adherends were kept at room temperature for about 3 h after they had been annealed at 313 K for 3 h because of stationary adhesion.

RESULTS AND DISCUSSION

A blank test, in the absence of samples, to determine the available rate range of peeling in the present

measuring system was done. The variation of time period in every 0.6° was read in, and from the numerical data, the peel force F_p and the peel rate v_p were calculated according to Eqs. (1) and (2). Figure 5 shows the resulting relation between F_p and v_p ; consequently F_p is approximated to be zero at rates below 1×10^2 mm/s. Therefore on the basis of $F_p = 0$, we regard the value of 1×10^2 mm/s as the upper limit in v_p that can be measured.

Some scatter in F_p was recognized at rates above the upper limit value; this results from the turn around time of data processors used here: The total time necessary for the interrupt in every 0.6° and data input is at least 1.6 ms. The larger the rate of peeling becomes, the shorter the interrupt period does, and it turns out that lack of time to process data brings about the scatter from $F_p = 0$. If the upper limit rate is calculated from the practical interrupt period of 1.6 ms, its value is consistent with the experimental value of 1×10^2 mm/s. The upper limit rate is raised by use of higher speed processors or direct memory access (DMA) interface. On the other hand, the lower limit of the rate of peeling is concerned with the initial torque of the encoder and friction of the bearings of the rotational shafts because in general the peel force decreases as the peel rate is decreased. Frictional forces of the bearings in the present apparatus is about 5×10^{-3} N, and therefore the lower limit rate is controlled by the friction, rather than the initial torque.

The relation between the peel force and the peel rate for sample A with crosslinked adhesives, using 180° peeling of the plate adherend, is shown in Figure 6. Interfacial failure was observed at the overall rates, including the increasing and decreasing rates of peeling. Furthermore, the peel force at a peel rate

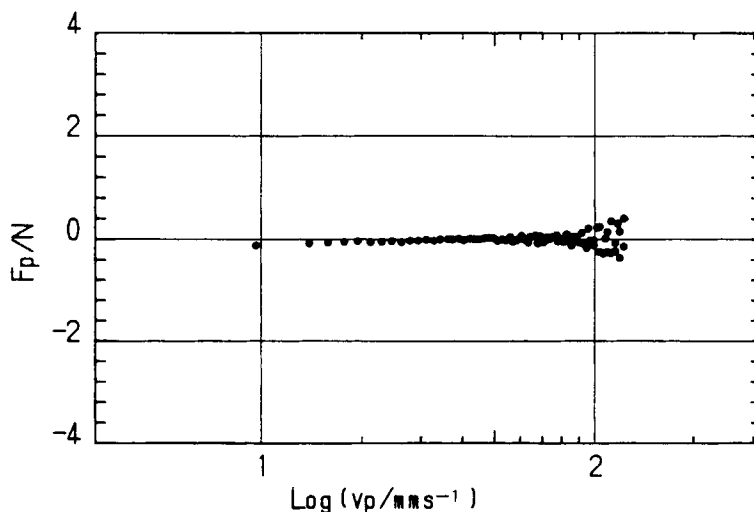


Figure 5 Peel force F_p vs. rate of peeling v_p in the blank test.

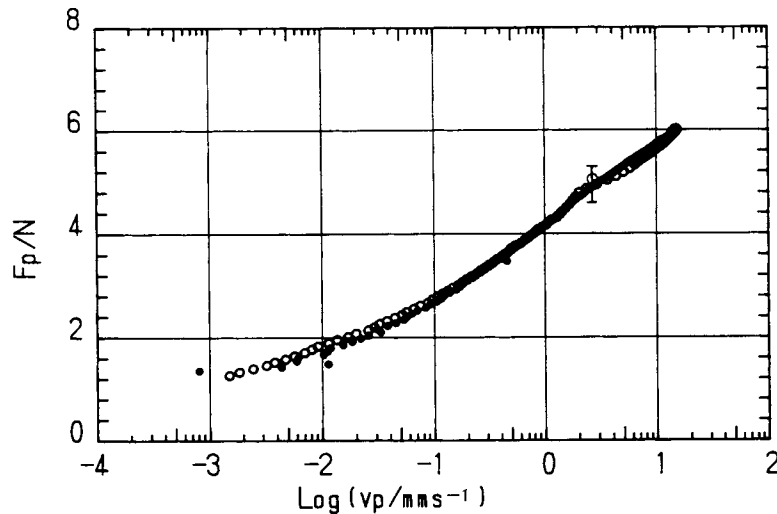


Figure 6 Peel force F_p vs. rate of peeling v_p for sample A with crosslinked adhesives, using 180° peel testing device. Solid circles, increasing rate; open circles, decreasing rate; bar, conventional testing at 2.5 mm/s. Tape width is 10 mm. The pendulum mass attached is 100 g.

of 2.5 mm/s, measured by means of the conventional peeling tester, is in good agreement with that obtained using the present apparatus, as the bar in Figure 6 shows.

Figure 7 shows the peel force F_p against the peel rate v_p for sample B with noncrosslinked adhesives, adhered to a plate testing device of 180° peeling. The F_p - v_p characteristics are qualitatively explained in terms of marks A to G given in Figure 7: The pendulum at $\theta = 0^\circ$ starts to rotate at the position near A, and F_p rises along B, C, and D as the peel

rate is increased, and then F_p lowers with decreasing the peel rate, via E, F, and G. In other words, marks A, D, and G correspond approximately to $\theta = 0^\circ$, 90° , and 180° , respectively.

Cohesive failure was observed in the A to B and the F to G regions; while in all other regions, except in the B to C region in which the transition from cohesive to interfacial failures occurs, the interfacial failure was observed. In the transition region, the peel behavior was different in the increasing or decreasing rates of peeling; there is a hysteresis of

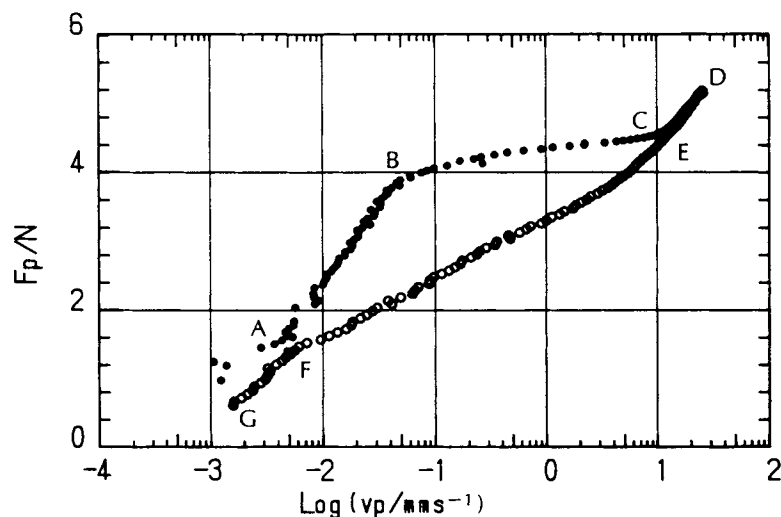


Figure 7 Peel force F_p vs. rate of peeling v_p for sample B with noncrosslinked adhesives, using 180° peel testing device. Solid circles, increasing rate; open circles, decreasing rate. Tape width is 10 mm. The pendulum mass attached is 100 g.

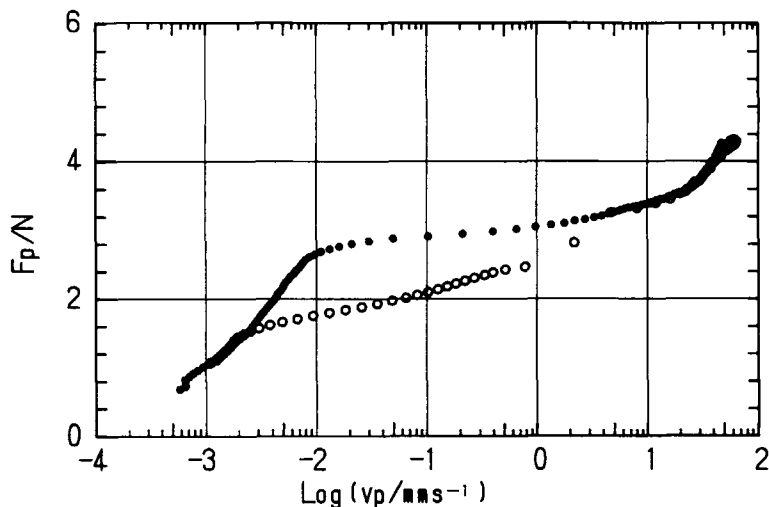


Figure 8 Peel force F_p vs. rate of peeling v_p for sample B with noncrosslinked adhesives, using 90° peel testing device. Solid circles, increasing rate; open circles, decreasing rate. Tape width is 10 mm. The pendulum mass attached is 70 g.

peeling where a change, discrete or occasionally accompanied by cohesive failure, occurred in the increasing rate of peeling; while in the decreasing process, interfacial failure was observed.

The dependence of the peel behavior on the moment of force of the pendulum was investigated. The results indicate that the peel force in cohesive and interfacial failure regions, except the transition region, was independent of the moment of force. In the transition region, the peel force increased with increase in the moment.

Figure 8 shows the peel force and the peel rate for sample B adhered to the drum of 90° peeling. The peel adhesion behavior, accompanied by a peel hysteresis, indicated the same tendency as that in 180° peeling of the plate for the same tape, although the magnitude of the peel force was lower.

The peel behavior measured using the present apparatus is not in steady state, in contrast to stationary peeling of the conventional testings under constant rate or constant load. However, in view of the facts described above, it seems that the deviation from stationary peeling is not large. The peel behavior from the present apparatus gives additional information of the peel hysteresis in the transition region from cohesive and interfacial failures. It is also characterized that the peel spectrum in a wide rate range can be readily obtained in a half cycle of the pendulum.

CONCLUSION

A peel adhesion apparatus using the circular motion of the compound pendulum was developed to investigate the peel adhesion behavior in the peeling processes of increasing and decreasing rates. Practical testing of the apparatus was carried out over the wide rate range from 10^{-3} to 10^2 mm/s for various PSA tapes; the results showed a good agreement with those of the conventional testing at a constant rate of 2.5 mm/s. The peel hysteresis was observed in the transition region between cohesive and interfacial failures. The present apparatus is expected to be available for practical testing to investigate the peel adhesion behavior of PSA tapes.

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