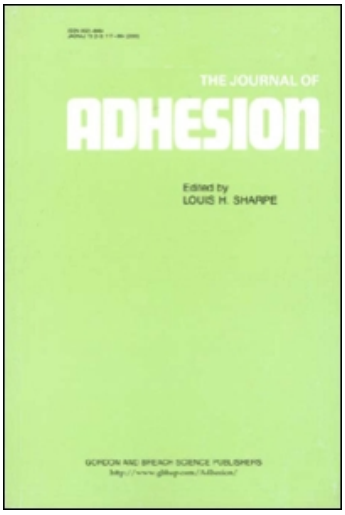

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Autographs from Peeling Pressure Sensitive Adhesives: Direct Recording of Fracture-induced Photon Emission

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Autographs from Peeling Pressure Sensitive Adhesives: Direct Recording of Fracture-induced Photon Emission

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Katisha: "Volcanoes have a splendour that is grim, And Earthquakes only terrify the dolts, But to him who's scientific, There is nothing that's terrific, In the falling of a flight of thunderbolts!"
KoKo: "Yes, In spite of all my meekness, If I have a little weakness, It's a passion for a flight of thunderbolts! . . ."

Gilbert and Sullivan, The Mikado

It is well known that visible light is emitted during the peeling of adhesives from various substrates. The major source of light has been identified as small gaseous discharges that result from the intense charge separation accompanying detachment of dissimilar materials. In this paper, we describe an experimental technique which produces clear images of the photons created by peeling pressure sensitive adhesives directly from the surface of a film emulsion or from the surface of a glass fiber optic face plate in contact with the film. The resulting autographs of the emitted light show in considerable detail the spatial structure of the photon emission which in turn reveals the mechanical and electrical behavior of these materials during the peeling process.

KEY WORDS Pressure Sensitive Adhesives; Adhesive Failure, Fracto-Emission, Triboluminescence, Electrical Charge Separation, Imaging.

I INTRODUCTION

Accompanying the deformation and fracture of materials, particles and radiation are emitted (collectively known as fracto-emis-

sion[1–16]). The types of particles that have been observed include electrons, ions, neutral species, photons (often referred to as triboluminescence), and long wavelength electromagnetic radiation (radiowaves). These emissions can often serve as sensitive probes of bond breaking, locus of fracture, crack velocity, and other properties of the fracture process. In previous work,[1–16] we have presented experimental studies of the photon emission (phE), electron emission (EE), positive ion emission (PIE), and the neutral emission (atoms and molecules —NE) accompanying fracture. This includes studies of composites, metal/inorganic interfaces, and the peeling of pressure sensitive adhesives from several substrates, in various gases, and in vacuum.

When an adhesive is peeled in air and other gases,[16] the time variations of the phE detected with a fast photomultiplier tube could be characterized as having two components:

a) very fast bursts (peak width on the order of 50 ns or less), followed by
b) characteristic non-first order decay lasting 50 to 100 μs.

The bursts of light were found to be in coincidence with bursts of long wavelength electromagnetic radiation. These observations supported the view that as small regions of the adhesive detach from the substrate, patches of electrical charge are created which lead to gaseous breakdown (micro-"thunderbolts") in and near the crack tip. This light is very likely similar in wavelength to a weak discharge in air which is dominated by molecular nitrogen emission lines. We have attributed the slower-decaying component to a phosphorescence-like emission from the adhesive and/or substrate which is excited *by the discharge, i.e.*, the resultant particle bombardment of the surface(s), created by the discharge and accelerated by the charged surfaces. In polymers, the relaxation from such stimuli is multicomponent in nature, consisting of fluorescence decay (the decay of excited electronic states created at $t = 0$) and photons from the recombination of charge carriers, defects, and/or free radicals. The latter is rate limited by diffusion of "reactants" (the mobile species frequently being electrons) and is therefore a thermally activated process.[17]

In this paper, we present a novel method of examining the characteristics of these light emissions. Early attempts using a camera equipped with fast film and a large aperture lens were not successful due to insufficient light intensity. However, by placing the adhesive joint in close proximity to the film while the adhesive was undergoing failure we were able to obtain images. In an early experiment we peeled tapes from a glass slide in contact with the film. Although the resulting images were poorly resolved due to the spreading of the light through the thickness of the glass, this experiment demonstrated that photons from peeling (as opposed to electrons, X-rays, or chemical reactions) did indeed produce images on photographic film. As we shall show, extremely clear images were obtained by peeling adhesives directly from the surface of the film emulsion or from a fiber optic face plate in contact with the film. Furthermore, we also show that details of the adhesive bonding and the peeling mechanics greatly influence the resulting images.

II EXPERIMENTAL

In these experiments, two types of pressure sensitive adhesives were used: 3M Scotch Brand Magic Tape [No. 810] and 3M Brand Filament Tape [No. 893]. We refer to these tapes as Magic Tape and Filament Tape, respectively. Magic Tape consists of a cellulose acetate backing which carries a back release coating having a critical surface tension for wetting of approximately 21 dynes/cm. The adhesive is a long-side-chain alkyl acrylate. Filament Tape has an adhesive of natural rubber combined with varying amounts of a tackifying agent which is a hydrocarbon resin, apparently terpene based. More tackifier is used on the face of the adhesive and less in the saturating layer binding the glass filaments to the backing. In Filament Tape, the polyester tape backing has been treated with a release coating having a critical surface tension for wetting of approximately 21 dyne/cm.

In the work presented here, Polaroid films were used exclusively; the following films were used to record the images:

Film Emulsion Type (Polaroid)	ISO (ASA)	Coating
146	200	Uncoated
47	3000	Gelatin
107C	3000	Gelatin
612	20000	Proprietary

These films are all available in flat or roll format and were loaded in the appropriate Polaroid camera back which made the emulsion side of the film surface conveniently available for experimenting. A dark slide protected the film when room light was on. Experiments were conducted in a darkroom with typical times of exposure to the "background" light being a minute or two. No evidence of fogging from background light was observed.

Experiments were carried out by pressing the tape down by hand on the chosen substrate and then stripping by hand, usually in a 90° peel geometry. Approximate peel speeds were estimated by timing the duration of the peel and measuring the distance peeled from the image itself. Of course, instantaneous peel speeds varied considerably. After each experiment was completed, the Polaroid films were developed in a standard fashion, producing a "print" in a few seconds. Materials placed between the adhesive and the film emulsion were useful in demonstrating the nature of the emitted light. To determine if residue of the adhesive left on the surface of the film following peeling might prevent subsequent normal development, we exposed the film to light before *or* after peeling. These films showed normal sensitivity in the region where the tape was attached and removed proving that the images were not altered by a possible thin contamination layer.

III RESULTS AND DISCUSSION

The results to be presented in this section involve presentation of the resulting images from various substrates and peeling configurations. In general, in the figures shown, the peeling was from left to right and (unless otherwise stated) in 90° peel geometry.

Glass slide as substrate

As mentioned previously, the peeling of various tapes from glass slides held in contact with the Polaroid film was investigated. Figures 1 and 2 show the resulting images from Magic Tape and Filament Tape peeled from the glass slide at approximately 15 cm/s, using Type 107C Film and Type 612 Film. The images are somewhat blurred because the glass slide allows the light to spread. Although this results in images with poor resolution, the glass assured us that only *visible light* reached the film, excluding charged particles, reactive chemical species, and ultraviolet light. Since peeling tape directly from the film produced images of a similar nature, it implies that these clearer images [autographs] are indeed due to photon emission. Note that the image intensities in fact

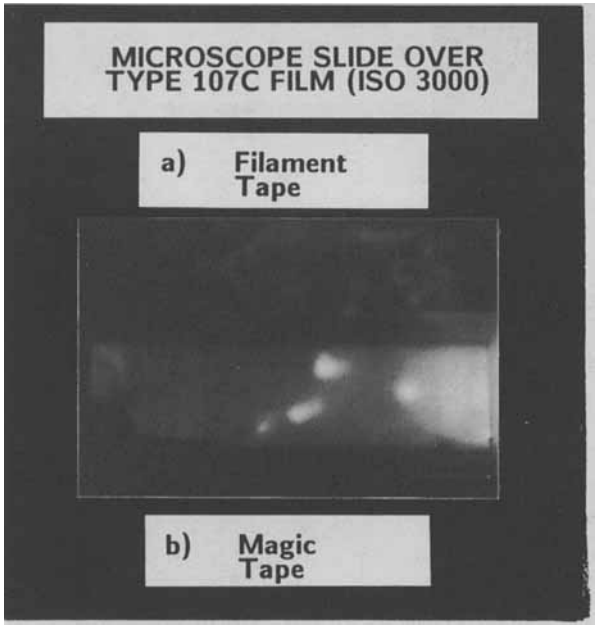

FIGURE 1 Images from peeling a) 3M Magic Tape and b) 3M Filament Tape (below) from glass slides placed directly upon the emulsion of Polaroid Type 107C Film, where peeling started at ~1 mm/sec and was accelerated. Here, and in all other cases, the tape was peeled from left to right. Unless otherwise stated, the peels were in a 90° geometry.

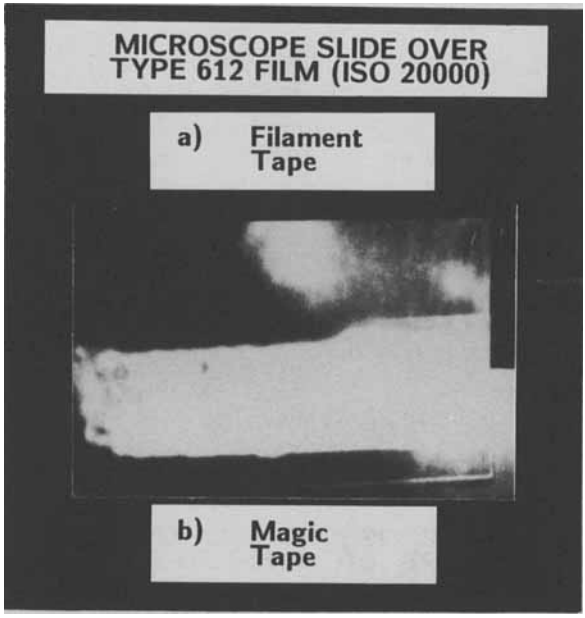

FIGURE 2 Images from peeling a) 3M Magic Tape and b) 3M Filament Tape from glass slides placed directly upon the emulsion of Polaroid Type 612 film.

correlate as expected with the ISO numbers. Similar patterns could be created using other transparent substrates (*e.g.*, PMMA). Note that the outline of the slide can be seen clearly from the light scattered along the edges into the film by internal reflection.

Film emulsions as substrates

Placing the adhesive in direct contact with the film emulsion and peeling it off within a few seconds of application produced autographs of the highest resolution. In Figure 3, we see typical patterns from the peeling of both Filament and Magic Tapes from Type 146 Polaroid Projection Film, where the peel was from left to right in a 90° geometry. The peel was accelerated during the peeling from a speed of ~1 mm/s to ~5 cm/s. Both adhesives show striations or stripes in the emission pattern for both fast and slow peels. This is a consequence of the stick-slip behavior characteristic

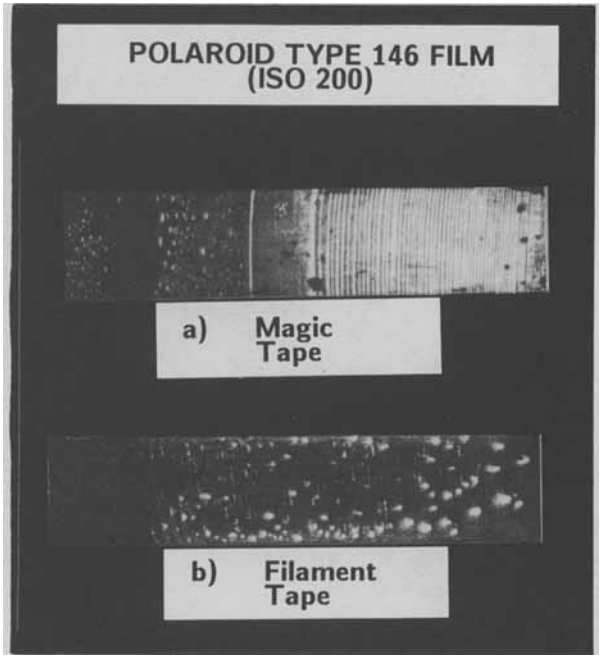

FIGURE 3 a) Magic Tape and b) Filament Tape peeled from the negative emulsion of Polaroid Type 146 Film. For scale we note that the tapes are 19 mm wide. In this case the peel speed was accelerated during peeling beginning at ~1 mm/sec and proceeding to ~5 cm/sec.

of peeling with an elastic backing material. Presumably the brightest regions occur during the "slip", *i.e.*, during the fastest detachment. The darker regions are not totally dark, particularly in the case of Magic Tape (Fig. 3a). The sharpest features seen for both tapes must occur quite close to the emulsion surface. The larger, more diffuse features most likely occur at slightly greater distances from the film. However, it was shown earlier[16] that when Filament Tape was peeled off of its own backing, some light was emitted even during the minima in intensity (the stick-slip peeling behavior shows up also as intensity variations in time). We also note that preliminary measurements of the charge density remaining on substrates *after* peeling show similar spacings in the charged patches (approximately 1.0 mm in "wavelength" for similar peel velocities).

Also, consistent with previous results,[16] the numerous bright spots seen in Figure 3b (Filament Tape) correspond to the very fast and intense bursts that we showed were due to the more intense microdischarge events accompanying peeling. In the case of Filament Tape, we see from the images that the intensity and number of these stronger arcs increase with peel speed. When these intense spots are examined under magnification, there is considerable structure suggesting breakdown paths parallel to the film surface. Also, it should be noted that the number density of these bright bursts at fast peel speeds is roughly 1 arc/mm^2 which is consistent with the number of radiowave bursts detected during comparable peeling.[16]

Smaller, individual arcs are also observable for Magic Tape/Type 146 Film and are most evident at slower peel speeds. At higher speeds, large numbers of these arcs merge to form a nearly continuous line across the width of the tape image. In the original photographs viewed with magnification, it is also evident that the regions of lower intensity emission are in fact the result of many low intensity discharge events.

The spacing of the striations is governed by the geometry of peeling, by the viscoelastic properties of the adhesive, and the elastic properties of both the tape backing and the film substrate. One can effectively modify the stiffness of the tape backing by applying one or two layers of electrical tape to the back of the

FIGURE 4 Magic Tape stiffened by two layers of electrical tape then peeled from Type 107C Film.

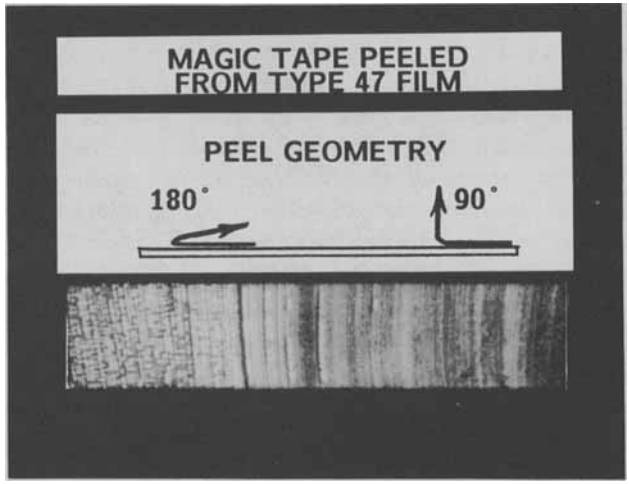

FIGURE 5 Effect of peel geometry. First 180° then 90° peel angle, Magic Tape and Type 47 Film.

adhesive tape being tested prior to peeling. In Figure 4, we show the resulting image for modified Magic Tape. The striations now have a wave length of 2 mm as compared to 1 mm for peeling unbacked Magic Tape at about the same speed. Note also the reduction in intensity and larger width of each striation due to changes in the mechanics of peeling.

We also tested the effect of peel geometry on emission patterns. In Figure 5 the peeling of Magic Tape from type 47 film was started at approximately 180° (parallel to the film), then the peel angle was changed to about 90° at approximately the same peel speed. The striations are considerably closer together for the 90° peel angle with fewer high intensity "thunderbolts". The latter may well be due to the distribution and density of charge favoring higher intensity discharges in the case of the 180° peel.

When a different film is used as a substrate, there are considerable differences in the resulting patterns. In spite of a factor of 10 increase in ISO, the peeling of tape from Type 47 and Type 107C Films does not produce as bright an image as from Type 146 Film, in contrast to the observations in Figures 1 and 2. We show the records for Magic Tape peeled at three different peeling speeds

from Type 107C Film in Figure 6, which results in visible but less intense images. This means that considerably less light per unit area was produced during the peeling. The likely explanation for the reduced emission is differences in the substrate surfaces which result in less contact charging [*i.e.*, in the case of Type 107C Film], thus producing less electrostatic discharge activity upon peeling. This demonstrates the important role played by the interface in creating the conditions which produce these emissions.

At higher speeds, Figure 6c, one sees aligned and imbedded in the less intense vertical striations of light, the appearance of a

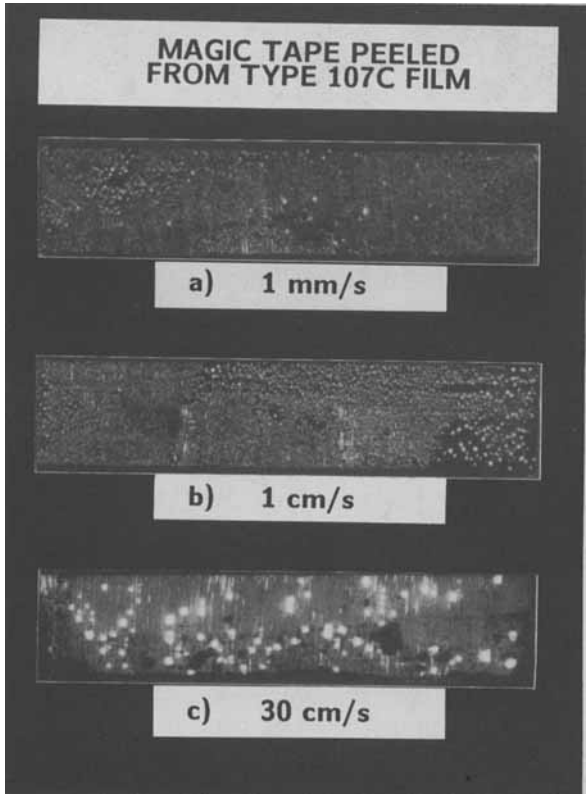

FIGURE 6 Magic Tape peeled from Type 107C Film at speeds a) 1 mm/s, b) 1 cm/sec, and c) 30 cm/s.

number of very bright discharges, occasionally several per strip. The very dark circles are due to bubbles inadvertently formed when applying the tape. Flaws in the adhesive produce very similar features. Frequently, extremely sharp, enhanced images of the tape edges are produced. The mechanics of peeling the tape is known to produce a positive pressure rise along these edges as the crack tip approaches. This momentarily improves the contact between the adhesive and substrate which appears to assist the resulting emission, visible in the bottom of Figure 6c. As we show below, if we purposely produce high pressure regions during the attachment of

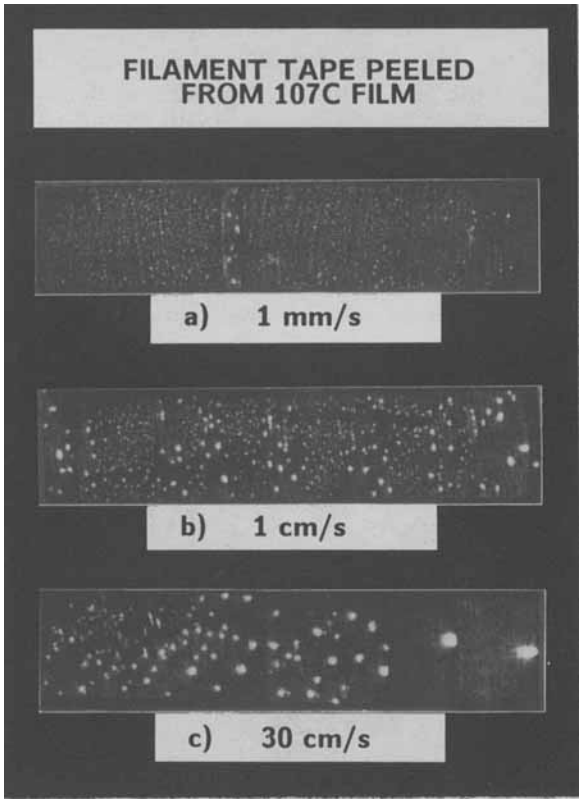

FIGURE 7 Filament Tape peeled from Type 107C Film at speeds a) 1 mm/s, b) 1 cm/sec, and c) 30 cm/s.

the adhesive to the emulsion surface, higher intensity emission is observed.

Figure 7 shows similar results for three peel speeds of the Filament Tape peeled from the surface of Type 107C Film. Again, note the diminished images relative to Type 146 Film. As the peel speed increases, the striations become less noticeable and the individual discharges, although fewer, are considerably stronger.

Figure 8 shows one of the more intricate patterns of surface arcs observed. During the initial slow part of the peel, there is considerable evidence of longitudinal streamers. When the peel speed was increased, several large star-shaped arcs occurred and subsequently many longitudinal surface arcs are found adjacent to the lines of intense emission. Because these arcs are very clear (particularly in the original photograph) it means that they occurred on the surface of the film emulsion. Note that at the higher speeds (to the right), the total light emission actually diminished.

Figure 9 shows slow peeling from the surface of a much faster film, Polaroid Type 612 Film, which is a high contrast film used for recording traces on a cathode ray tube (oscilloscope). With this film there tends to be no gray scale in terms of brightness; nevertheless, considerable detail is still observable. Obviously, the higher ISO (20,000) greatly increases the sensitivity and could be useful for

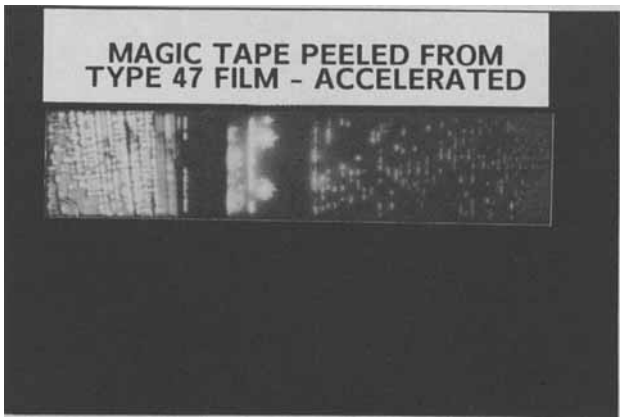

FIGURE 8 An example of complex discharge activity during peeling. Magic Tape peeled from Type 47 Film at an increasing speed.

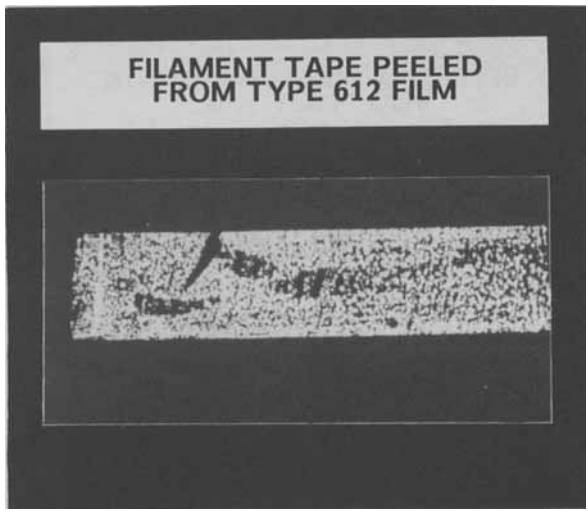

FIGURE 9 Filament Tape peeled from Type 612 Polaroid Film.

interfaces that are less emissive. The dark regions are due to creases in the tape where there was no adhesive in contact with the film.

Effect of application pressure

As mentioned above, contact pressure can influence the intensity of the emission. Figure 10 illustrates such a situation, where during attachment greater pressure was used on the bottom half to produce a better bond between the Magic Tape and Type 107C Film. The region where the highest pressure was applied yields more emission.

Tape as a Substrate

To vary the type of failure (cohesive *vs* adhesive), we first attached a layer of Scotch Double Stick Tape to the film emulsion and used this as a platform for attaching various substrates with little loss of resolution due to the thin tape backing. Once a test was performed, this platform lifted from the film without additional photon emission by wetting the adhesive with methanol applied by a cotton swab adjacent to the peel zone. The methanol appears to fill the capillary

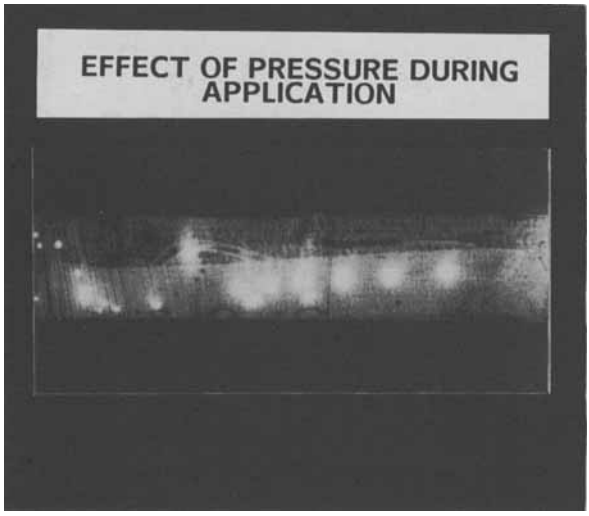

FIGURE 10 The consequence of increased pressure during attachment of the adhesive to the film emulsion (Type 107C Film).

crack, and either provides a conducting path which suppresses micro-arcs or simply destroys the adhesive bond. Following removal of this platform layer, the film could be developed in the ordinary fashion.

In photomultiplier experiments as well as electron and positive ion emission experiments (performed in vacuum), we have found that the emission intensity from adhesive failure is considerably more intense than from cohesive failure.[11–14] For illustration, we arranged the peeling of surfaces of Magic Tape so that the block letters *W S U* peeled in an adhesive fashion (adhesive from backing) and surrounding the letters, only adhesive-from-adhesive was peeled. Where the adhesive was peeled from the backing, much less force was required than the cohesive failure of the adhesive. The top of Figure 11 shows the actual sample (reattached) used. The resulting image (light passing through various tape layers to the film) is shown on the bottom of Figure 11; the light came from the adhesive-from-backing separation, *i.e.*, from the regions where adhesive failure occurred. The cohesive failure of polymers *can* produce photon emission,[15] although it is typically orders of

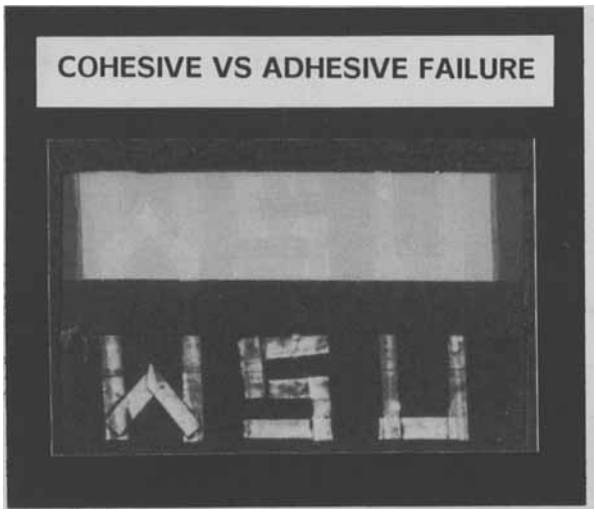

FIGURE 11 Cohesive-adhesive failure. A sandwich of Magic Tape was separated while it was held in close contact with the Type 107C Film by Double Stick Tape. The letters *W S U* were regions where the magic Tape peeled from its backing, surrounded by adhesive-from-adhesive peeling.

magnitude lower in intensity and in this case was not visible on the developed film.

Release Agents

We noted frequently that where the adhesive side of the tape had been touched by a finger, we could see the finger print in the image, where the print was *dark* relative to the normal light emission. The oils from the fingers were possibly serving as a release agent (or a conducting path—thus suppressing the discharges). Figure 12 shows a finger print (EED's) deliberately put on the adhesive tape prior to attaching to Type 146 Film. A careful examination of some of the other figures may reveal inadvertent fingerprints on the edges or ends of the images (a sure way to identify stolen data).

Similarly, we marked on the adhesive side of the tape (*or* on the film surface) with a felt tip pen, allowed the ink to dry, then applied the tape to Type 107C film and peeled. The ink layer produced a peel strength about 1/5 that of untreated Magic Tape. In Figure 13

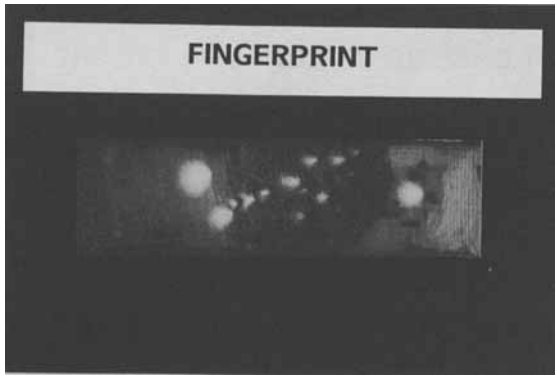

FIGURE 12 A fingerprint (the *dark* pattern) produced by imprinting the adhesive side of Magic tape before attaching to Type 146 Film. The oil from the finger serves as a release agent.

FIGURE 13 A release coating of ink from a felt tip pen was applied to the adhesive surface before the joint was made (Staedtler Lumocolor pen was used). The ink decreased the adhesion by a factor of five and greatly suppressed the light emission. The reconstructed sample is shown on the top.

we show the reconstructed sample on top (viewed from the adhesive side), and the resulting photon emission from peeling (bottom). Clearly, wherever there was ink the light emission was negligible compared to the surrounding, normal peel. Thus, such release surface modifications can greatly influence the resulting emission. We point out that similar results were obtained when the ink was applied to the film emulsion.

Metal present at Interface

The production of charge separation during the peeling should lead to strong electric fields. If a conductor is placed in the region between the film substrate and the adhesive, then as the peel front approaches the metal, sufficient charge can be induced on the metal to cause breakdown from the metal edges to the surrounding dielectric (the nearby film surface). In effect, sharp edges of the conductor can act as a charge density "concentrator". The image shown in Figure 14a was made by placing several pieces of aluminum foil on the adhesive of Magic tape. This tape was then pressed on the surface of Type 107C Film with finger pressure (reconstructed sample shown on top). Subsequent peeling (>10 cm/s) of the tape resulted in the usual emission where adhesive failure was occurring (bottom of Fig. 14a). Surrounding the foil there is a dark area which is due to bridging of the tape between the film and the foil, thus producing a moat of no contact. Along the trailing edge of the aluminum foil, we observe that there are clear images due to sharp discharges occurring along the trailing edge of the aluminum foil, occasionally along the sides of the foil, and never along the leading edge. These patterns suggest that as the peel line advances, the charge created on the *tape* (which is lifting the back of the aluminum foil) induces the same *SIGN* charge on the opposite edges of the conducting foil. When sufficient charge is induced so that the breakdown voltage of air is reached, the "sparks" occur.

To show that it is not necessary to peel the tape completely past the region where breakdown occurs, we used a triangular piece of foil, with the sharp tip which concentrates induced charge pointing away from the advancing peel (reconstructed sample shown in bottom of Fig. 14b). The resulting breakdown at the tip of the metal where the peel was stopped 7 mm from the tip is seen in the top of

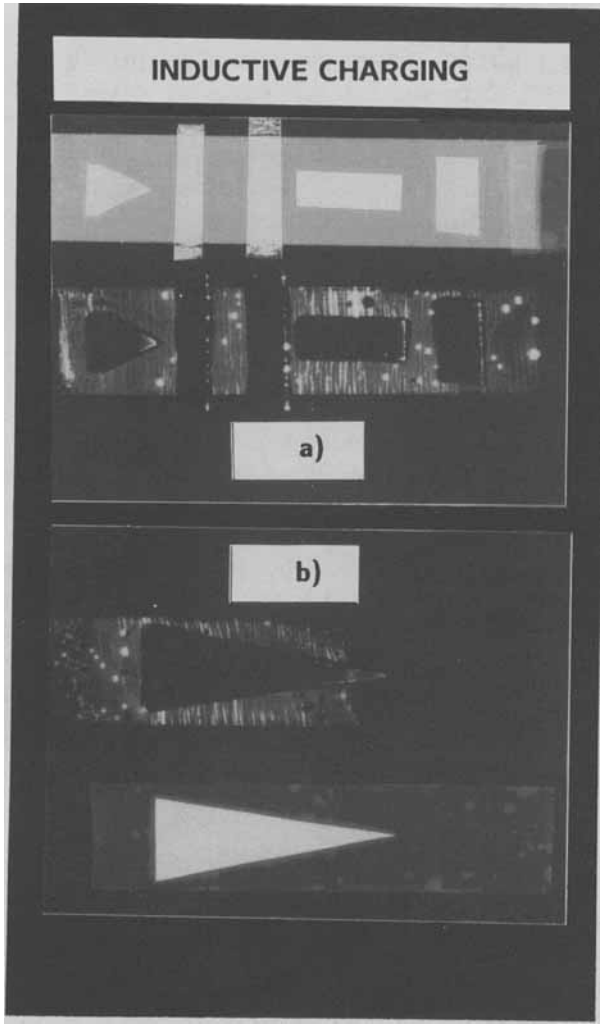

FIGURE 14 The consequence of peeling in the presence of aluminum foil between the adhesive and the film. a) Various strips of aluminum foil placed across the width of the tape; b) a pointed piece of foil oriented "downstream" from the advancing peel line. The charge induced on the metal is the cause of the breakdown at the metal edges. The corresponding reconstructed samples are shown at the top and bottom, respectively.

Figure 14b. The remaining portion of the attached tape was lifted off of the film without producing additional light emission by wetting the adhesive with methanol. The tip being in intimate contact with the film produces very sharp images from microdischarges in the high charge density portion of the tip. Microscopic asperities along the cut edges are the likely sources of the individual arcs observed. With the use of an electrometer, we found that a negative charge $|Q| > 10^{-8}$ C is induced on the foil as the crack tip advances along the edges of the conductor, which is quite sufficient for producing breakdown in a number of gaseous environments, including Pullman air.

Fiber Optics Face Plate as Substrate

With the use of a fiber optic face plate, one can transmit light from a substrate above the film, *e.g.*, the top surface of the glass plate itself. The face plate was circular 4 cm in diameter, 4.4 mm thick, and consisted of 6 μm diameter fibers. The transmission of the face plate was only a few percent so we used the sensitive Type 612 Film. Figure 15 shows the images produced by peeling Magic Tape from the top surface of the face plate. The resulting images are similar to Figure 6 except that with this rigid substrate the striations of stick slip failure are not as prevalent and are really strong only at the highest peel speeds. The microarcs are very distinctly seen with high resolution. The advantage of using a fiber plate is that thin films can be deposited on it and any substrate that transmits light can be studied. These include thin films of polymers and perhaps even metals. Furthermore, the fiber bundle could easily be coupled with an image intensifier, then to film, thereby obtaining even more sensitivity.

IV CONCLUSION

Spatial images in the form of autographs of the photon emission accompanying the peeling of pressure sensitive adhesives from various substrates have been produced. The best images were created either by peeling tapes directly from Polaroid emulsion surfaces or from the top surface of a glass fiber optics face plate.

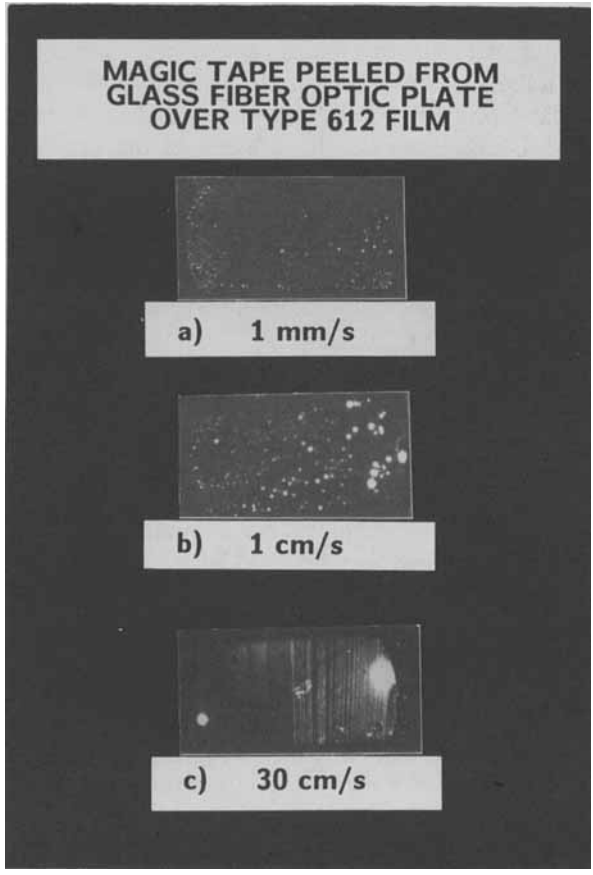

FIGURE 15 Magic Tape peeled from the top surface of a glass fiber optic face plate which was held in close contact with the surface of Type 612 Film.

Several features were consistent with other measurements: *e.g.*, a) striations which correspond to periodic variations in time of the phE intensities, b) corresponding variations in charge density on similar insulating substrates, and c) the number density of large discharges from Filament Tape as seen in these images agreed in magnitude with the observed frequency of the large radiowave bursts accompanying peeling. Also, the increase in emission intensity with

peeling speed[3] and the appearance of large individual bursts of light at higher speeds was observed in the photomultiplier experiments.[16] The observations of the influence of the mechanics of peeling (stick-slip, edge effects, role of release agents) may be of interest in studying details of the peeling process. The use of a fiber optics plate removes the need to use the emulsion as a substrate to create clear, distinct images and allows the introduction of a variety of substrates for further study. Finally, we mention that we have detected in composite structures photon emission coming from *inside* glass fiber-epoxy specimens[18] and have shown that it is due to interfacial failure between the fiber and the matrix. With the use of image intensifiers it may be possible to create images of this type of emission and pinpoint the location of yet another type of adhesive failure. Also, color slide films are available (*e.g.*, Fuji Film, Type DX-400) which can be pushed to ISO 1600 by special developing techniques. This sensitivity was found sufficient to produce faint but distinct blue images of both 3M tapes studied here when peeled from the film emulsion.

V Acknowledgments

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