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The Journal of Adhesion

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713453635>

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S. Wasserman^a; M. Snir^a; H. Dodiuk^a; S. Kenig^a

^a RAFAEL, Haifa, Israel

To cite this Article Wasserman, S. , Snir, M. , Dodiuk, H. and Kenig, S.(1989) 'Transmission and Mechanical Properties of Optical Adhesives', The Journal of Adhesion, 27: 2, 67 – 81

To link to this Article: DOI: 10.1080/00218468908050594

URL: <http://dx.doi.org/10.1080/00218468908050594>

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J. Adhesion, 1989, Vol. 27, pp. 67–81
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Transmission and Mechanical Properties of Optical Adhesives

S. WASSERMAN, M. SNIR, H. DODIUK, S. KENIG

RAFAEL, P.O. Box 2250, Haifa 31021, Israel

(Received April 4, 1988; in final form July 19, 1988)

The optical, mechanical and durability performance of selected epoxy, polyester, UV-curable acrylic, cyanoacrylate and silicone adhesives were evaluated and measured for bonding applications of optically transparent glasses in the visible and infra-red regions of the electromagnetic spectra.

From the initially selected adhesives only the UV-curable modified acrylic, two-component silicone and room temperature cured epoxy, were found to be of high performance characteristics, having good transmission properties and enhanced endurance in a combination of heat and humidity and following thermal cycling.

Sodium chloride substrates served as adherends for the transmission characterization of the optical adhesives, due to their high transmission properties in the 0.4–10 μm spectral range. A modified lap shear specimen was designed for studying the mechanical properties and failure mechanisms of the adhesives and their durability in a humid and hot environment. Finally, a two-piece glass doublet was used for investigating the opto-mechanical characteristics of the optical adhesive following environmental conditioning and thermal shock cycling.

Due to the inherent C—C bond, polymer adhesives are limited in utility, as far as transparency is concerned, close to 3.5 μm and in most of the 8–12 μm spectral range.

KEY WORDS Optical adhesives; optical properties; mechanical properties; durability; transmittance.

INTRODUCTION

Adhesives are commonly used to combine dissimilar materials and components to constitute a more complicated assembly, as an alternative to mechanical fastening. In the present case, the cohesive mechanical properties of the adhesive, surface preparation of the adherends and the interfacial adhesion are the most important factors for the product designer and end user.

The advantages offered by adhesives, such as an improved stress distribution, the possibility to join adherends with rough surface finish and still obtain a permanent seal between them, are very attractive in another potential application—adhesive bonding of optically transparent components. In this case, the optical properties of the adhesive are the principal ones for the designer, in addition to the conventional mechanical characteristics.

Typical applications of adhesives for joining optical parts are bonding of windows, prisms and lenses to obtain a composed optical component with defined specific characteristics such as refractive index, dispersion and transmission. In recent years, transparent adhesives have been introduced in optics, both in commercial as well as military equipment, due to the cost savings that could be realized by producing relatively simple subcomponents and assembling them into a complicated component by means of transparent adhesives.

The role of the optically transparent adhesive is to avoid the air gap that is obtained when two components are attached.¹ The occurrence of an air gap results in optical losses as a result of reflection of the transmitted light in passing from high to low (air) refractive index and back to a high index medium. The optical adhesive with its elevated refractive index may remedy the situation to a large extent, provided it is transparent in the relevant range of the electromagnetic spectrum.

Apart from their optical properties,^{1-4,6} optical adhesives should have appropriate moduli and thermal expansion coefficients that match those of the substrates to be joined for adequate stress relaxation,⁵ minimal shrinkage during curing to avoid formation of residual stresses at the adhesive interface, adjusted viscosity⁵ for bond thickness control and to ensure good wetting of the adherends and, finally, good bond durability for long term service and mechanical and optical integrity of the bonded assembly.⁷

Though the potential applications of optically transparent adhesives are unquestionable, the technical data regarding their optical and mechanical properties as well as their durability characteristics, are relatively sparse. Consequently, the present work is aimed at evaluating and measuring the optical, mechanical and durability behavior of selected epoxy, polyester, cyanoacrylates, ultraviolet curing acrylics and silicone adhesives for applications in the visible (0.4–0.7 μm) and infra red (0.9–1.2 μm 3–5 μm and 8–12 μm) regions of the spectrum.

EXPERIMENTAL

Methodology

The experimental methodology that was followed throughout the present study included two basic stages.

In the first stage thirteen different adhesives from five polymer families were characterized with respect to their optical transmission properties in the 0.4 to 12 μm spectral range. Only the best performing adhesives were further evaluated, in a second stage, with regard to their mechanical properties following hygrothermal exposure, using a modified lap shear set-up. In addition, the opto-mechanical performance of the adhesives was investigated using a doublet made of two glass discs bonded by the selected adhesives. In this part of the study, the adhesively bonded doublets were thermally cycled and conditioned in a hot-humid environ-

ment. Subsequently, the doublets were examined for possible surface irregularities and development of cracks and defects due to stresses built up during curing and their release upon exposure to the extreme conditions.

Adhesives

Five different families of adhesives were studied, namely, epoxies (both room-temperature and elevated-temperature-curing formulations), polyester adhesives based on styrene or acrylate cross-linking, cyanoacrylates, UV-curable acrylic and silicone elastomer adhesives.

Details concerning the thirteen different adhesives investigated are given in Table I.

Bond line thickness of the adhesive varied between 0.05 to 0.1 mm for all specimen types used in the present study.

Transmission Testing

Specimens for optical transmission characterization were prepared from sodium chloride substrates that served as adherends. The NaCl substrates were part of the spectrophotometer windows, 1 inch in diameter and 0.2 inch thick, polished on both sides. These NaCl substrates have a refractive index of 1.54 and are transparent in a wide spectral range (0.2 to 15 μm). Consequently, they were used in the transmission measurements.

The adhesives (refractive index ~ 1.5) were applied between the NaCl substrates using a special bonding fixture to ensure optical alignment throughout the curing process. Following bonding, care was exercised to prevent contamination of the substrate surfaces and to avoid moisture absorption by the bonded assembly. A sealed desiccator containing silica-gel powder was used for this purpose.

The transmission measurements were carried out using a Beckman Acta IV Spectrophotometer for the visible (0.4–0.7 μm) and near I.R. (0.9–1.2 μm) regions, and a Perkin Elmer 283B Spectrophotometer for the 3 to 12 μm region.

Mechanical and Opto-mechanical Testing

Two types of specimens were used for the mechanical and the opto-mechanical characterization of the adhesives.

The first one constituted a modified lap joint according to MIL-A-48611,⁸ see Figure 1, for evaluation of shear strength before and after exposure to heat (50°C) and humidity (95% R.H.). The modified specimen included a central borosilicate glass (BK-7), 25 \times 25 mm square of 0.2 mm thickness. The central glass, which is commonly used for production of lenses and windows, was degreased in methylene chloride and isopropyl alcohol and then bonded on both sides to aluminum adherends 25 mm wide, 100 mm long and 2 mm thick. The aluminum substrates were chromic acid anodized to ensure proper adhesion.

TABLE I
Adhesives investigated

| No. | Adhesive type | Curing conditions | Trade name | Manufacturer |
|-----|----------------------|---------------------------------|--------------------------------|---------------------------|
| 1 | Epoxy, flexible | 24 hours/R.T. | Epotek 310 | Epoxy Technology |
| 2 | Epoxy, low shrinkage | 24 hours/R.T. | Sira Optical Cement | Sira |
| 3 | Epoxy | 8 hours/R.T. | Stycast 1266 | Emerson & Cuming |
| 4 | Epoxy | 1½ hours/60°C | Epotek 353ND | Epoxy Technology |
| 5 | Epoxy | 4 hours/100°C | M—Bond 610 | Vishey Measurements Group |
| 6 | Polyester—styrene | 4 days/R.T. or 1 hour/70°C | Lensbond M-62 | Summers |
| 7 | Polyester—styrene | 24 hours/R.T. | Lensbond F-65 | Summers |
| 8 | Polyester—acrylate | U.V. cure/5 min. | Lensbond UV-71 | Summers |
| 9 | Cyanoacrylate | 30 seconds/R.T. | Zipbond | Tescocom Corporation |
| 10 | Cyanoacrylate | 30 seconds/R.T. | Permabond 910 (Eastman 910) | Permabond |
| 11 | Modified acrylic | U.V. cure/5 min | Noa 61 | Norland |
| 12 | Silicone Rubber | 24 hours/R.T. + 4 hours/65°C | DC-93500 (without primer) | Dow Corning |
| 13 | Silicone Rubber | 7 days/R.T. | R.T.V. 108 | General Electric |

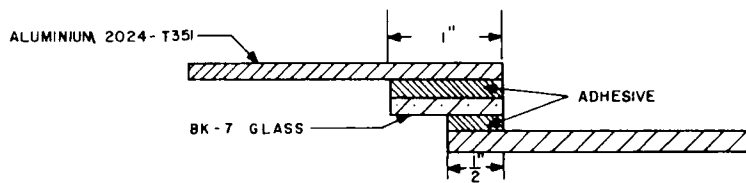


FIGURE 1 Schematic description of modified single lap joint.

For durability studies the specimens were subjected to a combination of 50°C and 95% relative humidity for periods of up to 28 days, using a Tenney Benchmaster temperature/humidity cabinet Model BTR. The shear strength of the modified lap shear specimens was obtained by loading them to failure in an Instron Mechanical Tester (Model 1185), with crosshead speed of 0.2 cm/min.

A second type of specimen was designed to investigate the development of irregularities, debonding of the adhesives and the resulting optical performance. The doublet-type specimens were composed of two BK-7 glass discs, 40 mm in diameter and 2 mm thick, which were degreased and then bonded with the adhesives. The specimens were then subjected to a thermal shock, by exposing them to three thermal cycles between -20 to 70°C. At each extreme temperature the samples were soaked for 24 hours. Besides thermal cycling, another set of samples were conditioned at 50°C and 95% R.H. for 28 days. The consequences of stress release of the adhesives, and their effect on the optical performance of the doublets, were determined by examining the surface irregularities of the doublets and the debonding state of the adhesives. A Zygo Mark I or Mark II interferometer was used for examining the optical irregularities before and after thermal shock. Visual inspection every 7 days was practised to examine debonding of the environmentally conditioned specimens followed by interferometer measurements after 28-day exposure.

Of the initial thirteen adhesives, only ten were evaluated for their mechanical and opto-mechanical properties. The three adhesives that were eliminated did not exhibit acceptable transmission properties. The ten remaining adhesives included: the UV-curable (Noa 61 and Lensbond UV-71), the silicone elastomer (DC 93500), the cyanoacrylate adhesive (Zipbond), four epoxy adhesives (Sira, Stycast 1266, Epotek 310 and Epotek 353ND) and finally the two polyesters (Lensbond M-62 and Lensbond F-65).

RESULTS AND DISCUSSION

Transmission Properties

Tables II, III, IV and V summarize the transmission properties of the studied adhesives for the visible (0.4–0.7 μm), near I.R. (0.9–1.2 μm), 3–5 μm and 8–12 μm I.R. regions, respectively. It should be emphasized that measurements in each region were taken at 4 to 5 points and that transmission level is given in

TABLE II
Percent transmission at 0.4–0.7 μm

| Wavelength | 0.4 μm | 0.5 μm | 0.6 μm | 0.7 μm | Min. | Max. | Average |
|------------------------|-------------------|-------------------|-------------------|-------------------|------|------|---------|
| Adhesive | | | | | | | |
| Epotek 310 | 88.6 | 93.0 | 95.1 | 97.8 | 88.6 | 97.8 | 93.6 |
| Sira Optical Cement | 82.6 | 87.5 | 89.7 | 90.8 | 82.6 | 90.8 | 87.6 |
| Stycast 1266 | 91.8 | 94.7 | 96.2 | 97.3 | 91.8 | 97.3 | 95.0 |
| Epotek 353ND | 68.0 | 92.4 | 96.7 | 97.8 | 68.0 | 96.7 | 88.7 |
| M-bond 610 | 81.5 | 85.3 | 87.0 | 88.0 | 81.5 | 88.0 | 85.5 |
| Lensbond M-62 | 71.7 | 80.4 | 85.8 | 89.1 | 71.7 | 89.1 | 81.7 |
| Lensbond F-65 | 90.7 | 94.0 | 95.6 | 97.3 | 90.7 | 97.3 | 94.4 |
| Lensbond UV-71 | 65.2 | 72.8 | 77.2 | 81.5 | 65.2 | 81.5 | 74.2 |
| Zipbond | 96.2 | 96.7 | 96.7 | 97.8 | 96.2 | 98.8 | 96.7 |
| Permabond 910 | 90.2 | 93.5 | 95.1 | 97.3 | 90.2 | 97.3 | 94.0 |
| Noa 61 | 90.2 | 94.6 | 96.7 | 97.8 | 90.2 | 97.8 | 94.8 |
| DC 93500 | 86.4 | 90.8 | 92.9 | 94.6 | 86.4 | 94.6 | 91.2 |
| RTV 108 | 73.9 | 82.6 | 87.0 | 89.1 | 73.9 | 90.2 | 83.1 |

percentage relative to the transmission of NaCl (91.24%). Each table points out, in addition, the minimum, maximum and average transmission levels for each adhesive. Figures 2 and 3 depict the transmission spectra of a polyester-styrene and an epoxy, respectively. It is clear that the polyester exhibits higher transmission levels, in all the relevant spectral regions, than the epoxy.

Generally, results have indicated that no single adhesive, out of the thirteen adhesives studied, is "universal" as far as its transmission properties in the entire 0.4 to 12 μm spectral range. Each spectral region has its best-performing adhesives.

TABLE III
Percent transmission at 0.9–1.2 μm

| Wavelength | 0.9 μm | 1.0 μm | 1.1 μm | 1.2 μm | Min. | Max. | Average |
|------------------------|-------------------|-------------------|-------------------|-------------------|------|------|---------|
| Adhesive | | | | | | | |
| Epotek 310 | 94.2 | 94.7 | 94.7 | 94.7 | 94.2 | 94.7 | 94.6 |
| Sira Optical Cement | 91.6 | 91.6 | 91.6 | 91.6 | 91.6 | 91.6 | 91.6 |
| Stycast 1266 | 98.9 | 98.9 | 99.0 | 99.0 | 98.9 | 99.0 | 98.9 |
| Epotek 353ND | 95.8 | 95.8 | 95.8 | 95.8 | 95.8 | 95.8 | 95.8 |
| M-bond 610 | 83.5 | 84.2 | 84.7 | 84.2 | 82.6 | 85.3 | 84.1 |
| Lensbond M-62 | 91.0 | 92.1 | 93.1 | 92.1 | 91.0 | 93.1 | 92.7 |
| Lensbond F-65 | 92.6 | 92.6 | 93.7 | 93.7 | 92.6 | 93.7 | 93.1 |
| Lensbond UV-71 | 82.6 | 84.2 | 86.3 | 87.4 | 82.6 | 87.4 | 85.1 |
| Zipbond | 93.7 | 93.7 | 94.2 | 94.2 | 93.7 | 94.2 | 94.1 |
| Permabond 910 | 94.2 | 95.8 | 95.8 | 95.8 | 94.7 | 95.8 | 95.5 |
| Noa 61 | 95.3 | 95.8 | 95.8 | 96.3 | 94.7 | 96.3 | 95.8 |
| DC 93500 | 93.5 | 94.2 | 94.7 | 95.3 | 93.5 | 95.3 | 94.4 |
| RTV 108 | 93.7 | 94.7 | 95.3 | 95.3 | 93.7 | 95.3 | 94.7 |

TABLE IV
Percent transmission at 3–5 μm

| Wavelength Adhesive | 3 μm | 4 μm | 5 μm | Min. | Max. | Average |
|------------------------|------|------|------|------|------|---------|
| Epotek 310 | 59.5 | 88.0 | 88.0 | 23.0 | 91.0 | 78.5 |
| Sira Optical Cement | 70.4 | 65.0 | 72.0 | 3.0 | 78.0 | 48.0 |
| Stycast 1266 | 23.0 | 70.0 | 81.5 | 3.0 | 82.5 | 58.2 |
| Epotek 353ND | 77.5 | 72.0 | 79.5 | 30.0 | 81.0 | 76.3 |
| M-bond 610 | 72.5 | 79.5 | 79.5 | 68.5 | 80.0 | 77.2 |
| Lensbond M-62 | 82.0 | 88.5 | 89.0 | 46.0 | 90.0 | 86.5 |
| Lensbond F-65 | 62.5 | 82.0 | 84.0 | 10.0 | 85.0 | 76.2 |
| Lensbond UV-71 | 54.0 | 70.0 | 72.5 | 6.5 | 80.0 | 65.5 |
| Zipbond | 90.0 | 90.0 | 90.0 | 72.5 | 91.0 | 90.0 |
| Permabond 910 | 90.0 | 90.5 | 90.0 | 72.0 | 91.0 | 90.1 |
| Noa 61 | 82.5 | 84.0 | 83.5 | 18.0 | 85.0 | 83.3 |
| DC 93500 | 90.0 | 91.0 | 89.0 | 5.0 | 92.5 | 90.0 |
| RTV 108 | 72.0 | 78.0 | 62.0 | 3.0 | 88.0 | 70.7 |

Visible Range (0.4–0.7 μm)

In the visible region, most of the studied adhesives have good transmission properties. Best optical transmissions were demonstrated by the UV-curable acrylic adhesive, room temperature cured epoxy, polyester-styrene and cyanoacrylate formulations. These compositions exhibited higher than 90% transmission, while the single component silicone elastomer, polyester-acrylate and high-temperature-cured epoxy showed transmissions below 70%.

TABLE V
Percent transmission at 8–12 μm

| Wavelength Adhesive | 8 μm | 9 μm | 10 μm | 11 μm | 12 μm | Min. | Max. | Average |
|------------------------|------|------|-------|-------|-------|------|------|---------|
| Epotek 310 | 30.0 | 12.0 | 55.0 | 58.0 | 65.0 | 12.0 | 73.0 | 44.0 |
| Sira Optical Cement | 3.0 | 3.0 | 3.0 | 12.5 | 3.0 | 3.0 | 26.5 | 4.9 |
| Stycast 1266 | 3.0 | 15.0 | 38.0 | 12.0 | 30.0 | 3.0 | 54.0 | 19.6 |
| Epotek 353ND | 5.5 | 11.0 | 43.0 | 52.0 | 43.0 | 3.0 | 66.0 | 30.9 |
| M-bond 610 | 55.0 | 62.5 | 73.0 | 73.0 | 75.0 | 50.5 | 77.0 | 67.7 |
| Lensbond M-62 | 30.0 | 33.0 | 55.0 | 66.0 | 74.0 | 10.0 | 80.5 | 51.6 |
| Lensbond F-65 | 3.0 | 6.5 | 19.0 | 37.0 | 45.5 | 3.0 | 59.5 | 22.2 |
| Lensbond UV-71 | 3.0 | 3.0 | 25.0 | 33.0 | 30.0 | 3.0 | 33.0 | 19.4 |
| Zipbond | 17.0 | 72.0 | 73.0 | 87.0 | 70.0 | 15.0 | 88.0 | 63.8 |
| Permabond 910 | 30.0 | 78.5 | 83.5 | 88.0 | 72.0 | 26.0 | 90.0 | 70.4 |
| Noa 61 | 5.0 | 35.0 | 23.0 | 46.0 | 63.0 | 3.0 | 63.0 | 34.4 |
| DC 93500 | 3.0 | 33.0 | 33.0 | 40.0 | 3.5 | 3.0 | 63.5 | 10.5 |
| RTV 108 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 |

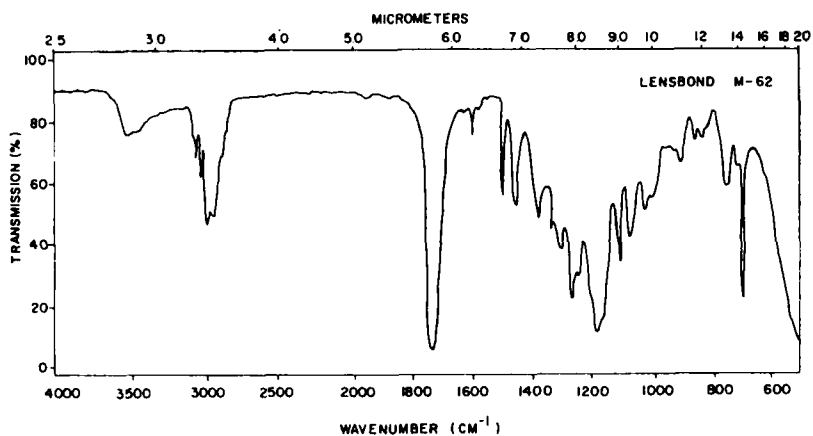


FIGURE 2 I.R. spectrum of Lensbond M-62.

Near I.R. Range (0.9–1.2 μm)

As in the visible range, in this region most of the adhesives are transparent and show transmissions above 90%, while some of them show transmissions above 95%.

I.R. Range (3–5 μm)

The 3 to 5 μm region includes inherent absorbance peaks which are characteristic of polymers and especially of the C—C bond (see Figures 2 and 3). Hence, the calculated average transmission value has no real meaning. The most pronounced absorbance is in the neighborhood of 3.5 μm . The best performing adhesives in this region are the UV-curable acrylic adhesive, the cyanoacrylates, the polyester-

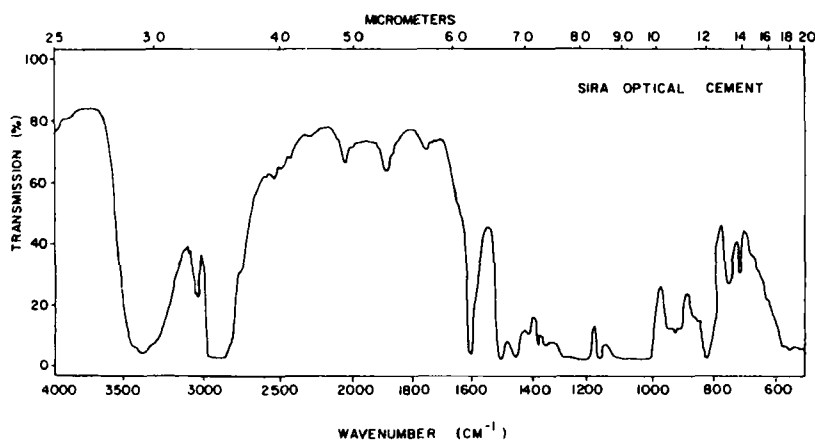


FIGURE 3 I.R. spectrum of Sira epoxy adhesive.

styrene formulation and the two-component silicone elastomer. All these adhesives were transparent to levels above 80%. Figure 3 depicts a low transparency epoxy adhesive in this spectral range.

I.R. Range (8–12 μm)

The 8 to 12 μm range is the least transparent region for polymeric adhesives. This region of the spectrum has wide absorbance bands. The best performance is exhibited by the two cyanoacrylates investigated. However, their transmission is limited to 60% only. Much is left to be desired for proper optical adhesives in this region. Figure 2 illustrates the transparency of the polyester-styrene formulation which exhibits a reasonable behavior while the epoxy adhesive (Figure 3) shows very low transparency in this region.

MECHANICAL PROPERTIES

Shear Strength and Durability

The modified lap-shear specimen, as applied in the present work,⁸ is not symmetric and, consequently, the stress distribution developed during loading is complicated. The asymmetric configuration was chosen following preliminary testing with a symmetrical lap-shear specimen. During the initial stage failure of the joint occurred primarily in the BK-7 glass. Hence, to force the failure to take place in the adhesive layer, the adhesive surface area was reduced on one side of the glass substrate. Consequently, the reported results should be analyzed in qualitative terms.

The shear strengths before and following exposure to heat and humidity of the modified shear specimens are given in Table VI. As can be seen, the adhesives can be divided into five groups with respect to their durability.

The first group includes the UV-curable modified acrylic, the two-component silicone adhesive and room-temperature-cured epoxy. These adhesives exhibit almost no degradation in shear strength following exposure to the hot-wet conditions after 28 days, with respectable absolute shear strength of 30 to 40 kg/cm². Examination of the fractured specimen indicated that failure was adhesive in nature at the glass interface. Only the silicone adhesive failed at the aluminum interface. This may be due to the relatively different stress distribution that developed during curing and upon mechanical loading and also to the compatibility of the adhesive and substrate interface. The low modulus elastomeric adhesive based on silicone rubber may chemically bond to the glass and may give rise to the different failure mechanism compared to the other, more rigid adhesives.

The second class of adhesives constitutes the room temperature epoxy cement (Sira), the polyester-styrene and polyester-acrylate compositions. The shear strengths of these three adhesives are in the 20 kg/cm² range before conditioning, and they degrade by 20 to 30% following 14 to 28 days of conditioning.

TABLE VI
Shear Strengths and Durability at 50°C, 95% R.H.

| Adhesive | Aging Duration | Unaged (kg/cm ²) | 7 days (kg/cm ²) | 14 days (kg/cm ²) | 28 days (kg/cm ²) |
|---------------------|----------------|------------------------------|--|--|-------------------------------|
| Epotek 310 | | 20.6 ± 2.6 ^a | 9.8 ± 0.7 ^a | 7.7 ± 3.0 ^a | 6.3 ± 1.6 ^a |
| Sira Optical Cement | | 22.5 ± 2.7 ^a | 21.6 ± 5.2 ^a | 18.0 ± 5.5 ^a | 14.1 ± 2.3 ^a |
| Stycast 1266 | | 21.9 ± 1.8 ^a | 23.3 ± 1.2 ^a | 33.7 ± 9.4 ^a | 33.6 ± 9.0 ^a |
| Epotek 353ND | | 11.0 - 1 sample ^b | 19.6 ± 3.1 ^b (2 samples— broken along the glass) | 36.8 - 1 sample ^b | 17.5 ± 2.9 ^a |
| Lensbond M-62 | | 19.7 ± 2.7 ^a | 9.7 ± 5.7 ^a | 10.5 ± 2.4 ^a | 15.3 ± 8.4 ^a |
| Lensbond F-65 | | 22.9 ± 7.0 ^a | 10.6 ± 1.1 ^a | 5.8 ± 2.5 ^a | 6.4 ± 2.2 ^a |
| Lensbond UV-71 | | 17.7 ± 0.9 ^a | 20.7 ± 2.4 ^a | 17.2 ± 5.5 ^a | 6.4 ± 2.8 ^a |
| Zipbond | | 75.9 ± 8.2 ^a | ^d | ^d | ^d |
| Noa 61 | | 45.7 ± 9.6 ^a | 43.3 ± 4.3 ^a | 45.3 ± 10.6 ^a (Ad. to glass) | 38.8 ± 7.1 ^a |
| DC 93500 | | 21.9 ± 0.5 ^c | 20.7 ± 3.9 ^c | 19.4 ± 3.9 ^c | 27.6 ± 4.2 ^c |

^a interfacial failure (glass/adhesive).

^b glass fractured.

^c interfacial failure (Aluminum/adhesive).

^d samples debonded before testing.

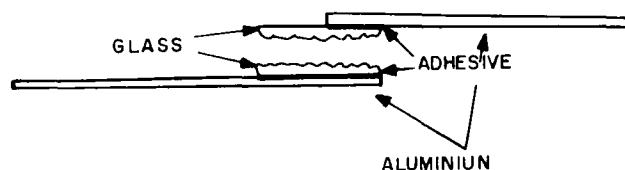


FIGURE 4 Schematic description of the failure mode obtained with the high-temperature-curing epoxy.

The third group consists of the flexible room-temperature-curing epoxy and the fast-curing polyester-styrene adhesives (Lensbond F-65). These two adhesives showed high rates of strength decrease (50%) after one week of hot-wet exposure.

The fourth category is composed of the room-temperature cyanoacrylate adhesive that, even though it demonstrated very high shear strength above 75 kg/cm^2 before conditioning, failed after short exposure to heat and humidity.

The fifth and last group includes the high-temperature-curing epoxy, which showed a unique failure along the glass substrate (see Figure 4) not observed in the other adhesives. Furthermore, this unique rigid epoxy exhibited a strengthening effect after 7 and 14 days, followed by a decrease after the final 28 days exposure to the environmental conditions.

Thermal Shock Endurance

For testing the thermal shock endurance of the adhesives with the borosilicate glass, two doublets were prepared for each of the ten adhesives. As mentioned earlier, both interferometric measurements of irregularities and visual inspection were used to study the response of the adhesives to cyclic thermal shock. Table VII summarizes the observed results in terms of cracks and blisters developed and irregularities.

As shown in Table VII, the UV-curable acrylic, the high-temperature epoxy, the two-component silicon adhesive, and the polyester acrylate cement exhibited no development of damage. Very slight optical irregularity and debonding took place in the case of the room-temperature-curing flexible epoxy and the other room-temperature-curing epoxies and polyester-styrene based adhesives.

All other adhesives exhibited inferior optical response after thermal shock cycling.

Environmentally-Induced Optical Degradation

The exposure of the adhesively bonded glass doublets to the 50°C and 95% R.H. environment induced debonding and optical irregularities in some of the adhesive-glass systems.

As can be seen in Table VIII and Figure 5, under these environmental conditions the UV-curable acrylic adhesive (Figure 5a), the high-temperature-

TABLE VII
Visual inspection and interferometric results of doublets exposed to thermal shock

| Adhesive | Visual inspection | Interferometric results |
|---------------------|----------------------------|--|
| Epotek 310 | Slight wrinkles in edges | No change |
| Sira Optical Cement | Few bubbles in center | Slight irregularity in center |
| Stycast 1266 | Tiny bubbles in edges | Slight irregularity in center |
| Epotek 353ND | No change | Doublet very irregular before thermal shock. |
| | | No change |
| Lensbond M-62 | Tiny bubbles in edges | No change |
| Lensbond F-65 | Slight separation in edges | Irregularity in edge |
| Lensbond UV-71 | No change | No change |
| Zipbond | Few bubbles in edges | Doublet irregular before and after thermal shock |
| Noa 61 | No change | No change |
| DC 93500 | No change | Slight irregularity in center |

curing epoxy, room-temperature curing epoxy and the fast-curing polyester-styrene adhesive demonstrated the best performance. Wrinkles and tiny blisters developed in the case of the flexible room-temperature epoxy, the two-component silicone adhesive and the regular polyester-styrene adhesive. Figure 5b indicates the development of slight irregularity (curving of the lines) due to deformation caused by stress development in the adhesive-glass interface. Figure 5d shows another kind of stress development (compared to Figure 5c) which results in separation of the substrate from the adhesive layer (the white patches on the edges). Large separations were observed in the case of the Sira epoxy and polyester acrylate cement. The room-temperature cyanoacrylate adhesive showed complete haziness and large areas of debonding following the environmental exposure.

CONCLUSIONS

Optical adhesives offer to the designer and end-user of optical components the means to combine simple and relatively low cost subcomponents, to form a composite optical assembly, provided that the adhesive can meet both optical and mechanical requirements.

With respect to the mechanically-induced stresses resulting from thermal shock and temperature and humidity environments on the one hand, and the inherent optical transparency on the other hand, optical adhesives may be divided into two major categories: Those that exhibit enhanced optical transparency and good mechanical properties in an aggressive environment, and those that demonstrate good optical properties and durability only under mild environmental conditions.

In the first class, only three adhesives performed satisfactorily. They were the UV-curable modified acrylic adhesive, the two-component silicone elastomer and the room-temperature-cured epoxy. These adhesives performed well in terms of

TABLE VIII
Visual (a) and interferometric (b) Inspections of doublets after humidity exposure

| Adhesive | Duration | 7 days ^a | 14 days ^a | 21 days ^a | 28 days ^a | 28 days ^b |
|-----------------------------------|----------|----------------------------------|-------------------------|------------------------|----------------------|-------------------------------|
| Epotek 310 Sira Optical Cement | | No change | No change | No change | No change | No change |
| | | Wrinkles and separation in edges | Wrinkles and separation | Larger separation | No further change | Irregularity in debonded area |
| Stycast 1266 Epotek 353ND | | No change | No change | No change | No change | No change |
| | | No change | No change | No change | No change | Deformations in edges |
| Lensbond M-62 | | Slight wrinkling in edges | No further change | No further change | No further change | No change |
| Lensbond F-65 | | No change | No change | No change | No change | Slight irregularity in edges |
| Lensbond UV-71 | | Wrinkles and separation in edges | Wrinkles 1/3 of area | Wrinkles 1/2 of area | No further change | Large deformations |
| | | Separation in edges | Bubbles and separation | Bubbles and separation | No further change | Large deformations |
| Zipbond | | No change | No change | No change | No change | No change |
| | | No change | Bubble in center | No further change | No further change | No change |
| Noa 61 | | No change | | | | No change |
| DC 93500 | | No change | | | | No change |

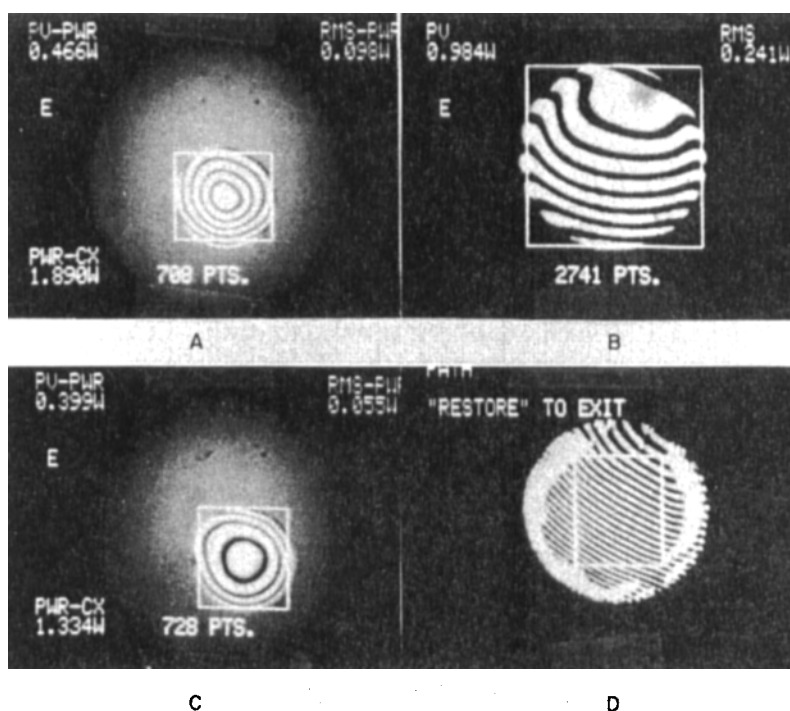


FIGURE 5 Interferograms of doublets bonded with Noa 61 prior to (a) and after (b) humidity exposure, Lensbond F65 prior to (c) and after (d) humidity exposure. (Interferogram of b was taken at a magnification of 2).

their thermal and hygrothermal endurance both optically and mechanically and showed a correlation with their respective shear properties. Furthermore, the adhesives showed high transparency in the visible (above 95%) and near infra-red regions (above 95%). In the 3–5 μm range both the UV-curable acrylic and the silicone adhesive showed more than 80% and 90% transparency, respectively, while the epoxy resin exhibited above 80% transmission only in the vicinity of 5 μm .

The second group of optical adhesives include the Sira epoxy cement, the room-temperature-curing flexible epoxy and the polyester-based adhesives. This group developed slight imperfections when exposed to humidity and temperature. However, they endured thermal shock conditions. In the visible and near I.R. regions these adhesives demonstrated a high level of transparency. They also gave acceptable transmission in the 3–5 μm I.R. range.

All other studied adhesives, although they exhibited acceptable or even high transmission levels (depending on the optical region), developed high stresses during curing that resulted in shear strength degradation, debonding and severe blisters and distortions upon exposure to thermal or humidity environments.

In the spectral region studied (0.4–12 μm), good optical transmission levels were obtained with most adhesives. However, due to their characteristic

absorbance, polymer adhesives are limited in performance in the 3.5 μm range and are far from satisfactory in the 8 to 12 μm region.

Acknowledgment

The authors would like to thank Mr. E. Perlshtein for this technical assistance.

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