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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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To cite this Article Murata, Norio and Nakamura, Kouzaburo(1991) 'UV-Curable Adhesives For Optical Communications', The Journal of Adhesion, 35: 4, 251 – 267 **To link to this Article: DOI:** 10.1080/00218469108041012

URL: http://dx.doi.org/10.1080/00218469108041012

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UV-Curable Adhesives For Optical Communications*

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UV-curable optical adhesive systems featuring refractive indices in the 1.45 to 1.59 region controllable to within 0.005 are developed using new fluoro-epoxies and fluoro-epoxy (meth) acrylates. These adhesives possess excellent refractive index matching with optical glass and optical fibres, and the joints exhibit high bonding strength and good durability. These high performance adhesives are readily applicable as optical adhesives in fabricating optical components, attaching fibres to optical waveguides, and splicing optical fibres for optical communications.

KEY WORDS UV-curable fluorinated epoxy adhesives; UV-curable fluorinated epoxy (meth)acrylate adhesives; refractive index matching; optical cable components and splicing; durability; return loss and transparency.

1 INTRODUCTION

UV-curable adhesives which set in a short time are widely used, mainly in the manufacture of electronics. In the field of optical communications, their suitability for use in the assembly of optical components and the splicing of optical fibres is currently being studied.^{1–5} It is hoped that the adhesives used in these applications, particularly adhesives used in optical circuits, will not only perform the function of adhesive bonding, but will also have the high degree of light transmittance and the properties required to form a bond most suitable from the point of view of optics. In order to optimize the efficiency of this bond, it is necessary to match the refractive index of the adhesive to that of the materials to be bonded. Furthermore, greater durability is required of adhesives. In addition, various other properties are desirable, such as ease of handling. This includes mixing from one liquid type, with no need for degassing and low viscosity so that it is easy to work with. They should also have good durability, and be capable of setting within a short period of time at room temperature.

We are undertaking a series of experiments in order to develop high performance adhesives for use in optical communications applications. Recently, by mixing

^{*}Translated from the Japanese and published with the kind permission of The Adhesion Society of Japan. Originally published in *Nihon Setchaku Kyokaishi* [J. Adhesion Soc. Japan] **26**(5), 179–87 (1990).

fluoro-epoxies and fluoro-epoxy acrylates⁶ which have low refractive indices, we have developed adhesives in which the refractive index can be controlled.⁷⁻⁸

In this article, as well as summarizing the special properties of UV-curable adhesives for optical communications applications which have had their refractive indices designed and produced as optical adhesives, we also report on the results of studies into their durability, reactivity and waterproofness, as well as practical results of splicing optical fibres and assembling optical components.

2 DESIGN AND CONTROL OF REFRACTIVE INDEX IN OPTICAL ADHESIVES

2.1 Designing the Refractive Index

Adhesives used to join components through which light passes are required to minimize the reflection of light. When part of the beam of light is reflected, particularly when this occurs close to the light source, it gives rise to interference. In order to reduce reflection occurring at the junction, it is necessary to use an adhesive which has a refractive index close to that of the materials to be joined, such as optical fibres, or lenses. The following equation gives the Fresnel reflection coefficient rfrom the join surface when optical components of refractive index n_0 and n_2 , respectively, are bonded using an optical adhesive of refractive index n_1 .

$$r = \frac{\left(\frac{n_2 - n_1}{n_2 + n_1}\right)^2 + \left(\frac{n_1 - n_0}{n_1 + n_0}\right)^2 + 2\left(\frac{n_2 - n_1}{n_2 + n_1}\right)\left(\frac{n_1 - n_0}{n_1 + n_0}\right)\cos\left(\frac{4\pi n_1 d_1}{\lambda}\right)}{1 + \left\{\left(\frac{n_2 - n_1}{n_2 + n_1}\right)\left(\frac{n_1 - n_0}{n_1 + n_0}\right)\right\}^2 + 2\left(\frac{n_2 - n_1}{n_2 + n_1}\right)\left(\frac{n_1 - n_0}{n_1 + n_0}\right)\cos\left(\frac{4\pi n_1 d_1}{\lambda}\right)}$$
(1)

The Fresnel reflection coefficient depends on the thickness of the adhesive, but this is very difficult to control. If we seek the relationship between return loss (Pmax) and the refractive index of the adhesive for the greatest value of the Fresnel reflection coefficient, we obtain the following equation:

$$P_{\max} = 10 \log \left[\frac{(n_2 - n_1)/(n_2 + n_1) \pm (n_1 - n_0)/(n_1 + n_0)}{1 \pm (n_2 - n_1)/(n_2 + n_1) \cdot (n_1 - n_0)/(n_1 + n_0)} \right]$$
(2)

Taking into consideration the dependency of the refractive index on wavelength and temperature, by using equation 2 we can design the most suitable refractive index range for adhesives used to join all kinds of optical components.⁸

Here we will show the most suitable refractive index range for adhesives used to join the end surfaces of optical fibres or optical couplers. Figure 1a shows the construction of an optical fibre termination.

In an optical fibre termination, in order to prevent reflection from the termination, BK-7 optical glass with anti-reflection film (refractive index n_2) is attached to the end of the optical fibre (refractive index n_0) by means of an optical adhesive (refractive index n_1). In the case of the optical coupler shown in Figure 1b, the prism



FIGURE 1 Optical components.

(refractive index n_2) and the dielectric multi-layer film (refractive index n_2) are bonded using an optical adhesive (refractive index n_1).

Figure 2 shows the relationship between the refractive index of an adhesive at a wavelength of $1.3\mu m$ and maximum return loss for an optical fibre termination and



FIGURE 2 Relationship between the refractive index of an adhesive at 1.3 µm and maximum return loss. (A) Fiber/Adhesive/Glass; (B) Glass/Adhesive/Glass.

an optical coupler, as calculated using equation 2. To make return loss exceed 34dB in the optical fibre, it is necessary to make the refractive index of the adhesive fall within the range 1.450–1.504 as shown by curve (A) in Figure 2. In the case of the junction between the prism and optical glass in an optical coupler, in order to make return loss exceed 34dB, the refractive index of the adhesive must be in the range 1.474–1.534, as curve (B) shows.

2.2 Refractive Index Control

In general, the refractive index of a material is determined by its molecular refraction [R] and its molecular volume V

$$n = \frac{2[\mathbf{R}]/\mathbf{V} + 1}{1 - [\mathbf{R}]/\mathbf{V}}$$
(3)

Compared with hydrogen molecules, [R]/V for fluorine molecules is small, and their refractive index is lower. This means that the refractive index can be lowered by introducing fluorine system substituents. Accordingly, it is possible to control the refractive index by mixing materials which have a comparatively high refractive index with materials having a high fluorine content and a low refractive index.

Table I shows the molecular structure, refractive index and fluorine content of fluoro-epoxy systems and fluoro-epoxy (meth) acrylate systems synthesized for the purpose of refractive index control. Because the refractive indices of cyclohexane and glycol compounds are low, if these compounds are used as the base resins, it is possible to design adhesives in which the refractive indices can be controlled over a wide range.

3 OPTIMIZATION OF COMPOUND COMPOSITION FOR STORAGE STABILITY AND WATERPROOFNESS

3.1 Test Materials

As shown in Table I, fluoro-epoxy systems such as Bisphenol AF epoxy (AFEp) and Cyclohexane epoxy (CHEp), epoxy acrylate resins such as Bisphenol AF epoxy acrylate (AFEpA) and Cyclohexane epoxy acrylate (CHEpA), and bisphenol AD epoxy (ADEp) (commercially-available epoxy resins) were used as base resins. Diluents, flexibilizers and silane coupling agents were added, and then onium salt system cationic polymerisation initiators and radical polymerisation initiators were used to form UV-curable adhesive compounds.

3.2 Test Procedures

Viscosity was measured at 25C using a Brookfield type viscometer. Refractive index was measured with an Abbe refractometer, and light transmittance was measured with a UV photospectrometer (Hitachi U-3400) using 1mm thick sheets.

UV-CURABLE ADHESIVES

 TABLE I

 Molecular structure, refractive index and fluorine content of epoxy (A) and epoxy(meth) acrylate (B)

$$\begin{array}{c} CH_2 CHCH_2 O - Rf - OCH_2 CHCH_2 \\ \searrow \\ O \\ \end{array} \tag{A}$$

CH2=CCOO	DCH ₂ CHCH ₂ OR	f-OCH ₂ CHCH ₂ OC	СОС=СН	$_{2}$ (X = H, CH ₃)	(B)
	I				
X	ОН	OH	Х		

	· · · · · · · · · · · · · · · · · · ·	Refractive index (n_D^{20})			Fluorine content (wt%)		
Structures	Rf	Ep1	EpA ²	EpMA ³	Ер	EpA	ЕрМА
Cyclohexane	$-C(CF_3)_2$ $-C(CF_3)_2$	1.405	1.413	1.417	43.2	33.8	32.5
Diphenylether	$-C(CF_3)_2$ $-O$ $-O$ $-C(CF_3)_2$ $-$	1.465	1.469	1.473	37.1	30.1	29.0
Bisphenol AF		1.518	1.512	1.512	25.4	19.3	18.4
Benzene	$-C(CF_3)_2$ $-\bigcirc$ $C(CF_3)_2$ -	1.433	1.440	1.443	43.7	34.2	32.9
Glycol	CH ₂ (CF ₂) ₆ CH ₂	1.385	1.416	1.418	48.1	36.9	35.3
Bisphenol AD ⁴		1.573	_	_	0	_	—

¹Epoxy

²Epoxyacrylate

³Epoxymethacrylate

⁴Low-viscosity, conventional epoxy

The sheets used in the measurement of refractive index and light transmittance were constructed by using a 1mm thick Teflon spacer and Teflon FEP mould-releasing film, placed between two plates of quartz glass and cured using UV irradiation. Curing was done using a UV light source (400W metal halide lamp Phillips HPA-400W) at a distance of 40cm. UV radiation intensity was approximately 10mW/cm² (at 350nm).

Curing was evaluated from changes occurring in the adhesive strength of cladding glass as a result of UV irradiation. As Figure 3 shows, adhesive strength was measured by placing BK-7 cladding glass shear adhesive strength test plates in a sample holder and using an Instron test machine at a shear speed of 5mm/min at 25C. Waterproofness was assessed by subjecting the cladding plates shown in Figure 3 to wet heat treatment and boiling for a fixed time, and then measuring adhesive strength at 25C. Storage stability was assessed by observing changes in quality such as gelling and increases in viscosity of the adhesive after a fixed period in a thermostatic bath at 60C, as well as by the decrease in waterproofness shown by observa-



FIGURE 3 Method of tensile shear strength measurement.

tions of bubble formation and peeling on the adhesive interface surface caused by boiling on cladding glass plates as shown in Figure 4, using adhesive which had been stored for a fixed time.

3.3 Experimental Results and Comment

Table II shows the results concerning waterproofness and storage stability for UVcurable adhesive compounds made up of different types of base resins and silane coupling agents. For all the compounded adhesives, epoxy silane (GPS) additive compound displays the best waterproofness and storage stability. Vinyl silane (VS), methacryloxy silane (MPS), amino silane (APS) and mercapto silane (MCP) had inferior storage stability. It is thought that the reactivity of the alkoxyl groups which control waterproofness and storage stability is influenced by the epoxy groups in the compound resins, by the concentration of free acids in acrylate resins, and by acids formed by UV irradiation of epoxy photoinitiator. It can be seen that by skilful use of this influence it is possible to reconcile the two properties of storage stability before use, and reactivity at the time of adhesion, which usually stand in opposition to one another.

Figure 5 shows the change in infra-red absorption spectrum before and after UV irradiation of epoxy silane coupling agent and cationic photoinitiator systems. As a result of 30 seconds of UV irradiation, absorption at the wavelength of 3400cm⁻¹ attributable to silanol groups increased. This shows that the acid formed in the process converted alkoxyl silanes to silanol.

Conversely, when stored in a cool, dark location, the reaction described in equation 4 does not occur, the reaction described in equation 5 becomes difficult, and



FIGURE 4 Bonding layer defect test specimen after boiling in water.

		Waterproofness ²	Storage time ³ (weeks)			
UC-curable adhesives	Silane ¹		2	4	8	
AFEp/CHEp system ⁴		Δ			— ×	
	MPS GPS	0 O	Δ O	Δ	Δ O	
ADEp/CHEp system ⁴	GPS	0	0	0	\triangle	
AFEpA/CHEpA system ⁴	– VS MPS APS GPS MCPS		04000		△ △ × ○ △	
Conventional adhesives	_	⊙~ ×	0~×	$\triangle \sim \times$	$\triangle \sim \times$	

TABLE II Waterproofness and storage stability (60°C)

¹VS: Vinyl-tris (2-methoxyethoxy)-silane

MPS: y -Methacryloxypropyl-trimethoxy-silane

APS: γ -Aminopropyl-trimethoxy-silane

GPS: γ -Glycidoxypropyl-trimethoxy-silane

MCPS: y -Mercaptopropyl-trimethoxy-silane

² \odot : Excellent, \bigcirc : Good, \triangle : Fair, \times : Poor (After 5h boiling)

³O: No change of properties

 \triangle : Waterproofness decreased a little

×: Viscosity increased or waterproofness decreased

⁴Base Resins/Diluents/Photoinitiator/Silane = 70-80/20-43/3/5



FIGURE 5 Change in IR spectrum with UV-irradiation of epoxy-silane coupling agent and photoinitiator system (silane/initiator = 100/3). (A) Original; (B) UV-irradiation for 30 min.

the silane coupling agent is not activated. Accordingly, it is thought that because storage in a cool, dark place lowers reactivity and makes it difficult, storage stability is good.

Figure 6 shows the results of boiling tests on glass test plates concerning the effect of silane coupling agents on waterproofness. Glass test plates using adhesive compounds with 2wt% of epoxy silane coupling agents added (epoxy base resins/diluents/epoxy silane coupling agent/photoinitiator = 100/20/2/2 parts by weight) maintained an adhesive strength of $200kgf/cm^2$ even after boiling for 24 hours. It is thought that the effect of this coupling agent is that the adhesive interface becomes difficult to destroy by the intrusion of water because the bonding force between the adhesive and the glass is extremely powerful, and a strong hydrogen bond and siloxane mutual bond is formed. In contrast to this, in the case of glass plates using adhesives which did not contain silane coupling agents (epoxy base resins/diluents/photoinitiator = 100/20/2 parts by weight), after 15 hours of boiling, the initial adhesive strength of $90kgf/cm^2$ was reduced by 25%. It was found that by using an appropriate silane coupling agent both waterproofness and adhesive strength could be improved to an extraordinary degree.

PHOTOINITIATOR $\xrightarrow{h \nu} H^+$ (Brönsted acid) (4) = Si-OR $\xrightarrow{H^+}$ = Si-OH (5) (silane)

$$\equiv Si - OH + OH - Si \equiv \longrightarrow \equiv Si - O, H, O - Si \equiv (6)$$
(silane) (glass)

$$\implies$$
 \equiv Si-O-Si \equiv (7)



FIGURE 6 Effect of water on tensile shear strength at 25°C (AFEp/CHEp system). ○ with 2 wt% silane coupling agent; ● without silane coupling agent.

MAIN PROPERTIES OF THE NEW ADHESIVES AND THEIR APPLICATION 4 TO OPTICAL COMMUNICATIONS

4.1 Main Properties

Applying the experimental results above, we developed epoxy system and epoxy acrylate system UV-curable optical adhesives. Table III shows the formulations of the new optical adhesives.

In Table IV the properties of the new epoxy system and epoxy acrylate system UV-curable adhesives are compared with those of commercially-available acrylate system UV-curable adhesives and epoxy system two-liquid heat-curable optical adhesives. The new adhesives have many superior properties. The main properties are discussed below.

(1) Control of refractive index

Figure 7 shows the refractive indices of the hardened new adhesives bisphenol AF epoxy (AFEp)/cyclohexane epoxy (CHEp) system, bisphenol AD epoxy (ADEp)/cyclohexane epoxy (CHEp) system, and bisphenol AF epoxy acrylate (AFEpA)/cyclohexane epoxy acrylate (CHEpA) system. By adjusting the fluorine content, the refractive index (n_D^{25}) can be controlled within the range 1.45 to 1.59 with a tolerance of 0.005. Because the refractive index can easily be matched to those of optical glasses such as quartz glass $(n_D^{25} = 1.475)$ and BK-7 $(n_D^{25} = 1.52)$, when used in the assembly of optical components and the splicing of optical fibres, it is possible to keep light reflection from the adhesive section to within theoretical limits, making possible the design of high performance optical components.

(2) Viscosity

Viscosity can be controlled within the range 300–2000 cps by means of a diluent. Because of their low stickiness and clean separation, these adhesives do not spread to the surrounding area and are easy to work with.

TABLE III

	Composition (parts by weight)			
Ingredient	(Epoxy system)	(Epoxyacrylate system)		
Base resins ¹	100	100		
Diluents ²	20-30	40-70		
Flexibilizer ³	10-20	_		
Silane coupling agent ⁴	2-5	2-5		
Photoinitiator	2-3	2-5		

²Monoepoxides or acrylates

³Diepoxide

⁴Epoxy silane

	New ¹	Conventional		
Property	(UV-curable)	(UV-curable) ²	(Thermosetting) ³	
Viscosity (cps)	300-2000	3000	600	
Curing time	<1 min (Room temp.)	<1 min (Room temp.)	4 h (65°C)	
Refractive Index (n ²⁵)	1.45-1.59	1.53	1.56	
Tensile shear strength (kgf/cm ²) Dry Wet (1) Wet (2)	90 to >200 180 to >200 110 to >200	107 0 5	>200 92 17	

TABLE IV Properties of optical adhesives

¹Epoxy and epoxyacrylate adhesives

²Acrylate adhesive

³Epoxy adhesive

Wet (1): After immersion in water at 60°C for 15 days

Wet (2): After exposure to 85% RH at 85°C for 15 days



FIGURE 7 Refractive index of new adhesive sheet. ○ AFEp/CHEp system; ● ADEp/CHEp system; □ AFEpA/CHEpA system.

(3) Curing

Figure 8 shows the effect of UV irradiation on adhesive strength for AFEp/CHEp systems as a function of time. With one minute of UV irradiation, tensile shear strength reaches 90kg/cm². Even in the case of epoxy system UV-curable adhesives, which have a relatively slow curing speed, curing occurs in under one minute using a UV irradiator with an output of approximately 10mW/cm². Furthermore, with a one-hour post-cure at 60C, tensile shear strength increased to a level in excess of untreated BK-7 glass' fracture point of 200kgf/cm². It is thought that the reason for the enormous increase in tensile shear strength in the post-cure is the reduction in internal distortion in the cured adhesive and increase in cross-linking density. Because UV-curable adhesives are capable of speedy adhesion and fixation inside one minute at room temperature with the application of UV radiation, it is easy to align optical components when assembling them. In contrast to this, the heat curable adhesives currently in use take four hours to cure completely at a temperature of 65C.



FIGURE 8 Effect of UV-irradiation on tensile shear strength (AFEp/CHEp system).

(4) Adhesiveness and waterproofness

As far as initial adhesive strength is concerned, there is no great difference between the newly developed epoxy and epoxy acrylate UV-curable adhesives and the heat-curable optical adhesives used up until now. However, after 15 days immersion in water at 60C and 15 days of 85C, 85%RH atmosphere treatment, the adhesiveness of the current epoxy system heat-curable adhesives deteriorated markedly. On the other hand, the newly developed epoxy and epoxy acrylate UV-curable adhesives showed superior waterproofness, with little deterioration in adhesiveness after 60C water immersion treatment and 85C, 85%RH atmosphere treatment, and no signs of peeling or bubbling on the adhesive surface.

(5) Light transmittance

The new epoxy and epoxy acrylate adhesives are colourless and transparent in both the cured and uncured states. However, as Figure 9 shows, the light transmittance of epoxy (AFEp/CHEp) adhesive sheet of 1mm thickness decreases for wavelengths in the region below 0.4μ m, reaching zero at 0.37μ m. Moreover, at wavelengths of 1.2μ m and 1.44μ m, absorption attributable to C—H and O—H oscillation was observed. At the wavelengths used for optical communications, viz. 0.8μ m, 1.3μ m and 1.55μ m, the light transmittance of sheets of the epoxy (AFEp/ CHEp) adhesive of 1mm thickness is 89%, 90% and 88% respectively.



FIGURE 9 Transmittance spectra of new adhesive sheet (AFEp/CHEp system).

The light transmittance of epoxy acrylate (AFEpA/CHEpA) sheets at wavelengths $0.8\mu m$, $1.3\mu m$ and $1.55\mu m$ was between 80% and 90%. This shows that light transmittance is sufficient for practical use as an optical communications adhesive, where an adhesive layer thickness of $10-50\mu m$ has a light absorption loss in the adhesive layer that is small enough to be ignored.

(6) Storage stability

As Table II shows, the newly developed epoxy (AFEp/CHEp) and epoxy acrylate (AFEpA/CHEpA) adhesives with the addition of the relatively stable epoxy silane displayed excellent storage stability, with no alteration to the adhesive's waterproofness even after 28 days in environmental conditions of 60C. In contrast to this, amongst commercially available UV-curable acrylate systems and ene-thiol systems, high reactivity types are prone to increased viscosity, colour change and a deterioration in waterproofness during storage at 60C. Additionally, the new adhesive stored in a brown glass container showed no colour change and no deterioration in waterproofness even after six months, and when it was cured, the silane was activated by means of UV irradiation, and excellent waterproofness was displayed.

(7) Heat resistance

Figure 10 shows the effect over time of 120C heating on the refractive index for the new epoxy (AFEp/CHEp) adhesive sheets and commercially-available UVcurable acrylate adhesive sheets. The new epoxy adhesive preserves its original refractive index even after 70 days. In contrast, the commercial UV-curable acrylate adhesive's refractive index increases up until day 20. Furthermore, the light transmittance of the new epoxy adhesive sheet (thickness 1mm) maintains its original level of 86–88% (1.3μ m), even after 70 days of 120C heat treatment, showing excellent heat resistance.

4.2 Applications in Optical Communications

4.2.1 Assembly of optical components

Using the new UV-curable epoxy acrylate (AFEpA/CHEpA) adhesive $(n_D^{25} = 1.50 \text{ for use with BK-7 optical glass and quartz glass})$, an optical fibre termination (Fig. 1a) was constructed using BK-7 optical glass with anti-reflective film. A glass block with an optical filter attached was also constructed.

The return loss at the optical fibre termination was greater than 30dB, and the amount of light reflected was extremely small, from which the excellence of the results can be seen. Additionally, even after 500 cycles of 6 cycles per day of heat cycles from +60C to -20C, and 20 weeks of exposure to wet heat atmospheric conditions of 85C and 85% RH, there were no signs of deterioration of return loss in the optical fibre termination, no slippage between the optical filter and the glass block, and no bubbling or peeling of the adhesive surface. The superior durability of the newly developed UV-curable optical adhesives was thus confirmed in practice with real optical components.



FIGURE 10 Effect of 120°C heating on refractive index at 25°C. ○ New adhesive (AFEp/CHEp system); ● Conventional UV-curable optical adhesive.

4.2.2 Splicing optical fibres

Using the UV-curable epoxy (AFEp/CHEp) optical adhesive with the same refractive index as optical fibre $(n_D^{25} = 1.475)$, a nylon-silicone sheathed single mode optical fibre (core/clad diameter 9/124µm) was spliced by means of a precision glass capillary splicer, as shown in Figure 11. The average splicing loss was 0.04dB, and the increase in loss in the temperature range -160C to +70C was extremely small, being less than 0.01dB. Moreover, in 20 cycles of heat cycle testing involving 6 cycles/day from -30C to +60C, the greatest variation in loss was extremely small, at 0.01dB. The average strength of the spliced section was 1.5kgf, which is higher than the splicing strength of current adhesive techniques. The suitability of the new UV-curable adhesive for optical fibre splicing was thus confirmed.

5 CONCLUSIONS

It has been demonstrated that by using fluoro-system base resins with low refractive indices, it is possible to control the refractive index of an adhesive over a wide



FIGURE 11 Precision glass capillary splicer.

range, and by using an appropriate silane coupling agent, adhesive compounds displaying both superior waterproof property and adequate storage stability can be developed. Using these techniques, epoxy and epoxy acrylate adhesives were developed as UV-curable optical adhesives for use in optical communications. The main feature of these new adhesives is that their refractive indices can be controlled, with a tolerance of ± 0.005 in the range 1.45-1.59, with a high light transmittance of 80-90% (at 1.3μ m). Adhesive fixation can be obtained within 1 minute at room temperature, adhesive strength in the case of glass is high, at 90-200kg/cm², and waterproofness is superior to that of the heat curable adhesives currently in use. These new adhesives are suitable not only for use in optical circuitry for joining and assembling optical components such as lenses and glass, and the splicing and fixing of optical fibre, but also have applications in the sealing and protective coating of optical and electronic components, and the fixing of small components of all kinds used in optical and electronic communications.

The assembly and splicing of optical communications components is an important technology in the construction of optical communication systems, and even higher properties are desired for the adhesives used in such applications. For example, when it is necessary to assemble optical components with a micro order of precision, there are still many areas of inadequacy in organic system adhesives, such as curing shrinkage and the coefficient of linear expansion being comparatively high, so it is hoped that even higher performance adhesives will be developed.

Acknowledgment

We would like to express our thanks to Toru Maruno, senior research engineer at the NTT Applied Electronics Laboratories for his assistance and advice concerning the research described in this paper. Parts of this report were presented at the 27th annual meeting of the Adhesion Society of Japan. (23 June 1989, Tokyo).

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