EPOXY TECHNOLOGY

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Selecting Epoxies for Optical and Fiber Optic Applications

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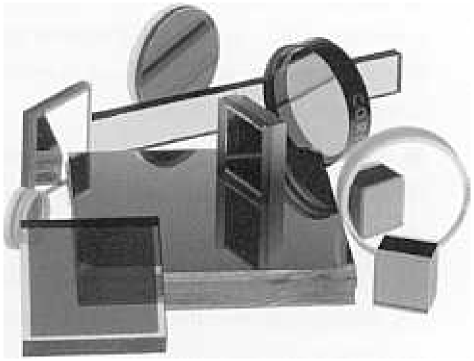
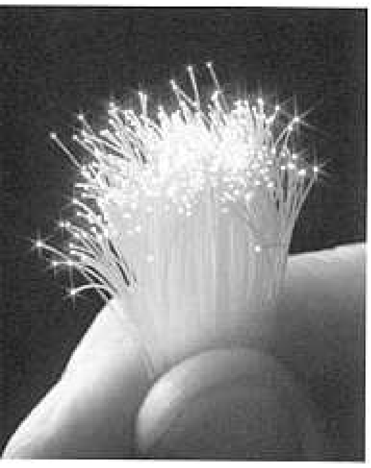
EPO-TEK® Epoxies for fiber optics, opto-electronics, optics, and lasers are used in a wide variety of bonding, coating, and encapsulating (potting) applications. They are valued for their excellent optical qualities, adhesive strength, durability and compatibility with a wide range of substrates, and solvent-free formulations. Epoxies can be formulated to fill gaps effectively, and they are easily applied and cured, making them an appropriate material for high-throughput production environments.

Because the uses and operating environments faced by epoxies are so varied, the attributes of specific epoxy formulations must also vary in many respects to produce optimal performance and reliability. As a result, many distinct epoxy formulations now exist that allow users to fine-tune the precise material for their specific needs.





This paper discusses the critical physical and performance characteristics of epoxies used in optical and fiber optic applications. The intent is to provide a perspective on the factors that should be considered in determining the most appropriate epoxy formulation for a given application. Epoxy properties relevant to both manufacturing techniques as well as adhesion and coating will be described. With this information, users can choose materials with reference to the variables that have the most impact on their requirements. After primary and secondary desirable attributes are defined, the optimal epoxy can then be selected or custom-designed.

Range of Applications

For telecommunications, data linking, cable television, medical, aerospace, and scientific instruments, advanced epoxies are continually called upon to connect, terminate, bond, coat, or encapsulate a wide range of components and devices. There are many applications when epoxies constitute the only viable option to achieve these functions. In other cases, epoxies are the preferred option for reasons that range from fabrication economy and efficiency to desirable performance characteristics.

Optically clear epoxies were first designed to add superior transparency to the other advantages of epoxy resins, such as toughness, durability, and low outgassing. To satisfy the need for highly reliable connections for single-fiber and bundled fiber optic applications, additional formulations were developed that surpassed Bellcore specifications and maximized thermal and moisture resistance, polishability, and adhesive strength. To meet the broadest range of requirements for both optics and fiber optics, additional epoxies have been formulated to exhibit special attributes such as UV transmission, biocompatibility, a low index of refraction, and curing by UV or visible light. New compounds are continually being formulated to meet the emerging needs of each of the industries that rely on optical grade epoxies.

The principal uses of epoxies for fiber optics and optics include:

■ Bonding optical fibers to connectors. After the fiber is stripped, it is coated with an epoxy adhesive, and then slipped into the connector (Fig. 1). An alternative method involves injecting the adhesive into the connector with a syringe. After heat curing, the end face of the connector and fiber is ground and polished. Note: To allow connector termination of PCS fibers using an epoxy, recladding of the stripped fibers may be accomplished with EPO-TEK 394, a low-index polymer. This eliminates attenuation due to stripping damage.

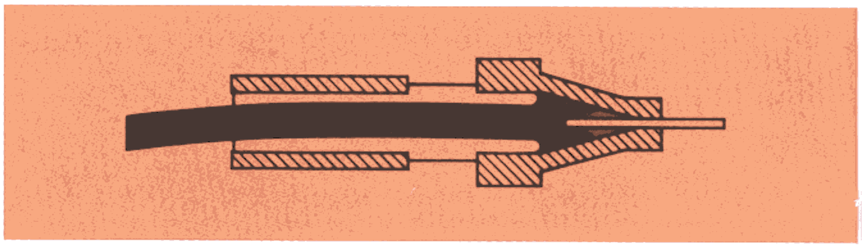


FIGURE 1

■ Splices, field repairs and modifications to fiber optic cable. Ease of use and reliability in field work is maximized by a color change that occurs with some epoxies that are heat cured. This color change indicates to a technician that the material is sufficiently cured for handling.

■ Bonding single-fiber and fiber optic bundles into ferrules. Epoxy adhesive is applied to the sheath of the fiber bundle by compressed air dispensing or dipping, and the sheath is then pushed into the sleeve.

■ Opto-electronic and optical devices. The myriad uses of optically transparent epoxies as bonds, coatings, and encapsulants include lasers, polarizing lenses, optical films, lenses, prisms, beam splitters, filters, laser windows, LEDs, photodiodes, optical filters, replicated optics, and encoders.

■ Medical devices. Medical uses for epoxies include fiber optic applications such as bundling and terminating fiber optics for light-guide applications, and bonding lenses and other components to fiber optic termini in endoscopes, as well as non-fiber-optic applications such as pacemakers, venous injection ports, and scalpel blades.

■ Compact discs. Newly developed UV-cure epoxies have proven effective and efficient as coatings for CDs as well as optical and electronic components and devices.

■ Miscellaneous. Besides optical materials such as glass and quartz, epoxies also adhere well to other types of materials, such as metals, ceramics, and many plastics. They have been used as a coating, impregnant or sealant in innumerable applications, including the restoration of valuable wooden artifacts and the preparation of thin rock sections or tissue specimens for microscopic studies.

Handling and Curing

Shelf Life and Pot Life

EPO-TEK Epoxies exist in both single-component and two-component formulations. One advantage of standard, two-component systems is a longer shelf life (the period of time for which the epoxy can be stored without any deterioration of physical, optical, or other properties). Typical shelf lives for two-component epoxies range from six months to one year at room temperature. Single-component formulations, on the other hand, are easier to work with because they do not need to be mixed in the correct proportions before they are used. During storage, however, the viscosity and thixotropy of a single-component material will gradually increase. However the shelf life of a single-component epoxy can usually be lengthened by freezing or refrigerating it according to the manufacturer's instructions. Two-component formulations typically can be preblended and frozen to yield single-component systems.

Pot life is the period of time during which a two-component epoxy can be used after mixing without causing significant variation in its consistency (viscosity and thixotropy). EPO-TEK 301, for example, has a shelf life of one year at room temperature, but once Part B (the hardener) is mixed with Part A (the resin), pot life is less than 1 hour. Toward the end of any epoxy's pot life, the material will thicken and become more difficult to use. Depending on the formulation, two-component adhesives can have a pot life that ranges from a few minutes to several days. The pot life value generally indicates a 10 percent increase from initial viscosity.

Epoxies designed for an exceptionally long pot life, such as EPO-TEK 354, may allow superior handling flexibility in production applications. When mixed and put into a syringe, EPO-TEK 354 can be used for up to five days without difficulty. Epoxies with lengthy pot lives also tend to provide higher peel strengths and a more versatile range of potential cure times and temperatures.

Some epoxies are available as both two-part and single-part systems. EPO-TEK 353ND, for example, while available as a conventional two-part system, is also provided as a single-part system that has been premixed, degassed and frozen in convenient-to-use syringes, which are useful for certain mechanical dispensing techniques, such as pneumatic or positive displacement methods. The shelf life of these syringes, which are available in several sizes, is six months at -40°, 30 days at -20° C. Pot life, after thawing, is four hours.

Solvent-free (100 percent solids) epoxies are the logical choice for potting or bonding applications because they do not suffer from the problem of voids that may form during curing as a result of solvent evaporation. However, some small bubbles can form due to the presence of minute amounts of dissolved air and water in the epoxy. To prevent this phenomenon, epoxies can be degassed before use. The two-component system is mixed and then placed in a vacuum for a time sufficient to remove any gases or entrapped air in the epoxy. Degassing is also done before bonding optical components to produce bonds that are completely free of minute bubbles.

Consistency

The consistency of the mixed, uncured epoxy is often an important consideration for efficient production. The viscosities of epoxies range from the watery and free-flowing to the thick and thixotropic. EPO-TEK 301, for instance, has very low viscosity at only 100-200 centipoise (cps) @ 23°C/100 RPM, whereas EPO-TEK 353ND-T is a nonflowing thixotropic epoxy with a viscosity of 9,000 to 15,000 cps @ 23°C/20 RPM.

The viscosity should be carefully matched to the choice of application method, which may include compressed-air dispensing, centrifuging, screening, stamping. roller coating, dipping, or brush application. Low-viscosity materials tend to provide the widest possible choice of application methods, such as dispensing, roller coating, dipping, or brush. Low-viscosity materials are especially desirable for applications such as bundling optical fibers, when the material needs to flow easily between the individual fibers. On the other hand, when bonding components where excessive flow or wicking cannot be tolerated, epoxies with nonflowing properties are desired.

Viscosity is temperature-dependent: As temperatures rise, the viscosity of the material declines, and lower temperatures result in higher viscosity levels. This tradeoff between temperature and viscosity can be used to optimize epoxy application. When optical fibers are being bundled into ferrules, for example, an epoxy with a low viscosity is used so that it will flow well between the fibers and completely fill the spaces between them. But it is also essential for the material not to flow between fibers beyond the ferrule. Typical solutions include heating the ferrule before the epoxy is applied, so that viscosity is lowered and the material flows freely through the bundled fibers before it begins to cure and blocks the flow of the material beyond the desired point. The flow of the epoxy through the fibers may also be enhanced at the start of an oven-curing process, when the elevated temperatures cause an interval of low viscosity before curing begins.

Thermosetting Epoxies

Most epoxies are cured by exposing them to a specified temperature for an adequate time for setting to take place. A wide range of cure temperatures and times are recommended for various thermosetting epoxy formulations. Cure temperature may range from room temperature to 150° C or more. Furthermore, users typically have a choice of several different combinations of temperatures and time periods that may be successfully used as curing alternatives for the same adhesive. Some epoxies offer the choice of curing at room temperature or elevated temperatures, while those formulated for high-temperature operating environments, like EPO-TEK 353ND, usually offer only heat-curing options. This epoxy takes 90 minutes to cure at 60° C, but the cure time for the same material at 100° C is 5 minutes, and it takes only 1 minute to cure at 150° C. In some applications, an epoxy that cures at low temperatures or even at room temperature may be desired. For example, EPO-TEK 302 is an optically transparent adhesive that has been designed for fast curing at room temperature. Curing of this material takes only 1 hour at room temperature, compared to 12 to 48 hours for other optical formulations. On the other hand, higher-temperature curing requirements generally mean that the material itself is more heat-resistant. And, to some extent, by curing at the highest temperature possible, the heat resistance of a material (particularly its glass transition temperature), can be elevated due to the tighter cross linking of the polymer.

Shrinkage and Outgassing

Solvent-free epoxy formulations are essential for optics and fiber optics applications, because shrinkage is reduced to minimal levels when no solvent is released during the curing process, and low shrinkage is vital to reduce stress on the component or device being bonded or coated. The optical-grade epoxy EPO-TEK 301-2 is an especially low-stress material, causing linear shrinkage of only 1.4 percent during curing. Similarly, EPO-TEK 354 is recommended for bonding large core (100μ or 140μ) optical fibers. When the epoxy is cured at 100° C for 2 hours, there is very little stress, eliminating any possibility of cracking the glass fiber, a common problem with large-core fibers.

The absence of solvents and thinners also means no problems with outgassing in aerospace or vacuum applications. Based on outgassing test results by NASA, EPO-TEK 301 has been approved for space flight programs, demonstrating a total weight loss of 1.08 percent. The high-temperature formulation EPO-TEK 354 demonstrates only 0.31 percent outgassing to 300° C. In general, the higher the temperature used for curing, the less outgassing the material will demonstrate after cure.

UV and Visible-Light-Curing Epoxies

UV-curing epoxies are used for extremely fast and economical processing in applications where the adhesive, coating, or encapsulant can be directly exposed to the UV light source or where the substrate is clear and adequately transmits UV radiation. EPO-TEK UVO-114 , for example, can be used to bond quartz crystal to tips of fiber.

UV-curing epoxies cure upon exposure to ultraviolet radiation at specified wavelengths for an adequate time interval. The curing of these materials depends on the total amount of UV radiation (which, in turn, is a function of intensity and time) and wavelength. For example, EPO-TEK UVO-114 will cure at 340 mW/cm2 at 1" for 60 seconds at 365 nm.

Unlike most UV-cure adhesives, EPO-TEK UVO-114 is not an acrylic formulation. Compared to other UV-curable acrylic adhesives, UV-curable epoxies like EPO-TEK UVO-114 exhibit superior moisture resistance, chemical resistance, and tolerance of high-temperature conditions. UV-curable epoxies also offer improved adhesion to a wide variety of surfaces, including glass, quartz, ceramic, metals, and most plastics. There is no significant shrinkage during cure.

New epoxy formulations that combine UV and visible-light curing promise improvements in both curing speeds and in the range of application of curing by light. The addition of visible-light curing compensates for the inability of many clear substrates to transmit UV light, and improves curing of shadowed areas.

Curing by Color Change

For ease of use in making fiber optic connections, some EPO-TEK epoxy formulations will change color during cure. EPO-TEK 353ND, for example, changes from amber to dark red in the course of curing. This feature allows the users to judge when curing is complete simply by watching for the color change, instead of attempting to calibrate precise temperatures and timing in uncontrolled field conditions where a heat gun is used to complete the curing.

Performance Characteristics

Optical Properties

Optically clear epoxies are now available which offer excellent transparency over a wide range of wavelengths. EPO-TEK 301, for example, displays greater than 97 percent transmission between 3,200 and 9,000 angstroms. This degree of transparency results in excellent transmission in thin bond lines. The exceptional stability of optical-grade epoxies, including UV-cure formulations, means that this optical clarity can be maintained throughout the operating life of the device.

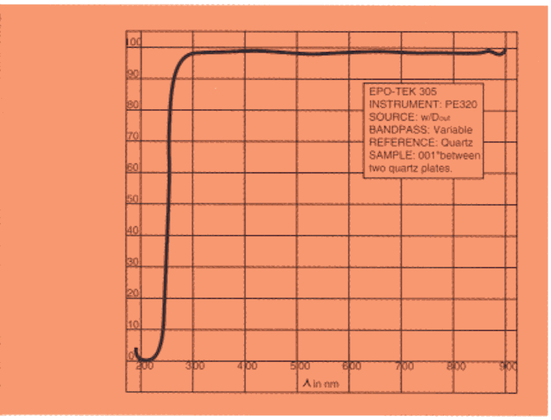


FIGURE 2

EPO-TEK 305 is an optical adhesive with a unique feature: It starts transmitting in the ultraviolet region, with 60 percent transmission at 220 nm, 85 percent transmission at 250 nm, and more than 94 percent transmission between 260 nm and 2.5 microns, at an adhesive film thickness of .0005 inch. Figure 2 shows similar transparency results for EPO-TEK 305 at a film thickness of .001 inch. This material also performs exceptionally well in replication processes.

For coating or potting some optically sensitive components, total opaqueness to light is of primary importance. EPO-TEK 320 has been formulated as a black, optically opaque epoxy, and a film of it only 0.0005 inches thick will transmit less than 0.0001 percent of light over a wavelength range of 3,000 angstroms to 1 micron.

Most epoxies have indexes of refraction in the middle of the 1.50-1.57 range. An exception is EPO-TEK 314, an epoxy with a low index of refraction of 1.4939. It is designed for high-temperature applications in optical or fiber optic assemblies where a low refractive index is required.

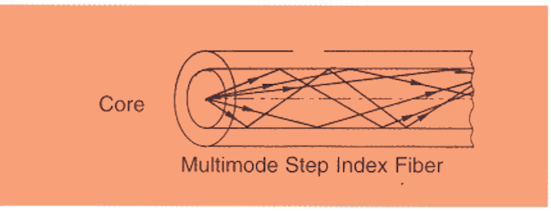


FIGURE 3

EPO-TEK 394 is a cladding polymer with a low index of refraction of only 1.394, designed for cladding quartz optical fibers. This material demonstrates greater than 95 percent transmission through a .0005-inch-thick sample from 2,600 to 9,000 angstroms, which will allow for excellent performance for UV-type light guides. A modified version of EPO-TEK 394 may be used as a cladding material over the entire length of a quartz fiber (Fig. 3). Fibers coated with EPO-TEK 394 exhibit superior transmission in UV (690 dB/Km @ 200 mm).

It is important to note that while optical clarity is not needed for most fiber optic connections, splice coupling does require an optical connection.

Thermal Properties

Two thermal properties are especially important to consider when an epoxy's ability to perform well in high-temperature conditions is being evaluated. *Maximum continuous operating temperature*, of course, is the highest temperature that the cured epoxy can endure for a lengthy interval without deteriorating. Optical epoxies generally have maximum continuous operating temperatures of 125° C or higher. This temperature can usually be exceeded for finite intervals without causing any yellowing or deterioration of the material. EPO-TEK 314, for example, is an optical-grade epoxy with a low index of refraction that was designed for high-temperature applications. Its maximum continuous operating temperature is 150° C, but it can tolerate intermittent intervals at 300° C. This material is suitable for products that will be subjected to autoclave conditions such medical instruments.

EPO-TEK 353ND, also designed for high-temperature applications, is an adhesive often used for bonding fiber optics and manufacturing medical devices such as catheters and surgical scalpel blades. While the maximum continuous operating temperature of EPO-TEK 353ND is 200° C, it will endure temperatures between 300 and 400° C for several hours (Fig. 4). Another formulation, EPO-TEK 377, is an ultra-high-temperature epoxy. Metals bonded with this material have been subjected to temperatures as high as 500° C for 10 hours without bond failure.

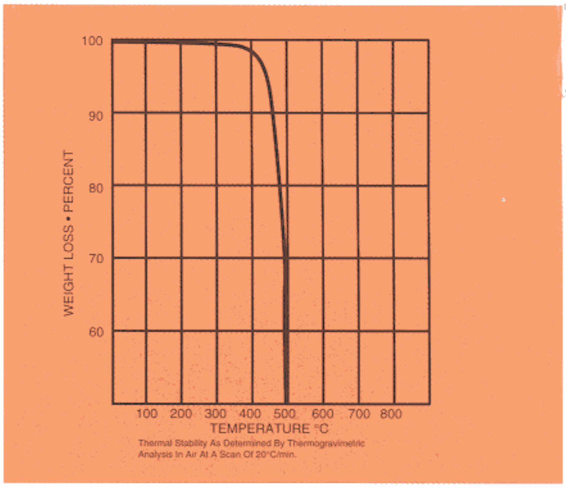


FIGURE 4

Glass transition temperature (Tg) is defined as the midpoint of the temperature region where the physical properties of an amorphous polymer (such as an epoxy) shift from those of a hard, glassy state to those of a soft and rubbery condition. An epoxy's glass transition temperature, while below the maximum continuous operating temperature, it is important to consider because it affects the physical characteristics of the material, including shear strength, thermal coefficient of expansion (CTE) and elasticity. At temperatures above the Tg, CTE and elasticity are increased, while the strength of the bond formed by the adhesive is diminished. When cooled below its Tg, the epoxy will regain its original characteristics. For applications where the epoxy bond will be subjected to severe stresses, the Tg of the epoxy should be above the projected use temperature.

While EPO-TEK 353ND has, in addition to a high maximum temperature, a high glass transition temperature (>100° C, when cured at 150° C for one hour), a subsequent version of this material was formulated to provide an even higher Tg. Known as EPO-TEK 353ND-4, this product provides a Tg in excess of 150° C, allowing extremely reliable bonds in fiber optic connector terminations that may be subjected to unusually high temperature conditions.

It is important to note that Tg is affected both by the formulation of the epoxy and by how it is cured. Other factors being equal, the use of higher curing temperatures will tend to result in a higher Tg. To maximize Tg, it is also important to cure the epoxy for an adequate period of time. Although the material may appear to be cured after intervals well short of the recommended cure duration, Tg will continue to increase as the curing process continues until a maximum level is reached (Fig. 5). Note: The thermal mass (amount of epoxy) also influences the degree of cure.

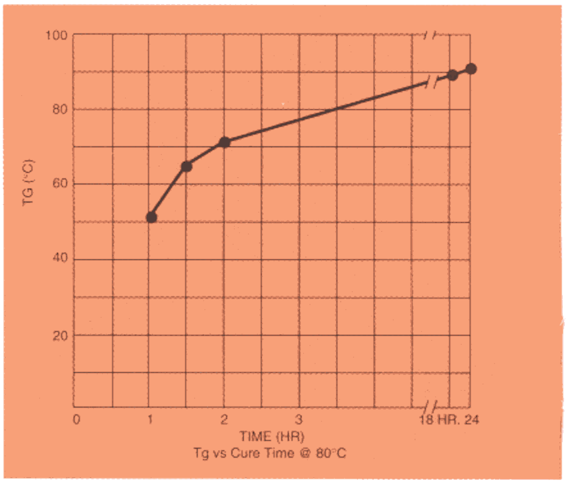


FIGURE 5

Strength and Hardness

The most common measurement used to gauge the strength of the bond provided by an epoxy adhesive is shear strength, defined as the maximum force that can be applied to two bonded plates parallel to the bonded surface without separation occurring. Epoxy adhesives offer excellent bond strength, with lap shear values ranging as high as 2,000 psi or more. Most epoxies used in fiber optics are also formulated to cure to an extremely hard finish. EPO-TEK 353ND, for example, with a Shore D hardness of 87, is easily polished to a flat face after curing for optimal fiber optic connections. This material also provides an extremely strong and durable bond, which prevents the signal-damaging phenomenon of fiber "creep" away from the connector.

EPO-TEK 310, on the other extreme, is a flexible material, with a Shore D hardness of only 22 and a glass transition temperature at subambient temperatures. For some applications, such as lens and prism bonding, this flexible material is ideal for bonding glass to glass, glass to metal, and metal to metal.

Moisture Resistance

Moisture resistance is measured in terms of the percentage weight increase of the material caused by water absorption when placed underwater or in a highly humid atmosphere for a given period of time. Moisture resistance becomes a critical factor when the epoxy will often be subjected to moisture, such as in autoclave sterilization for medical devices, where the combination of heat and moisture poses an especially difficult environmental challenge. The ability of the epoxy to resist moisture is also critical when the material is being used to bond or coat a component or device which is vulnerable to moisture, as in certain fiber optic applications. For example, EPO-TEK 353ND and 377 have extremely low sensitivity to moisture and easily pass 85/85 humidity aging tests as well as autoclaving. EPO-TEK 353ND, 353ND-T and 354 exhibits a weight gain of 0.03 percent after 7 days in 96 percent relative humidity, passing MIL SPEC MIL-1-16923-D. These adhesives also demonstrate excellent resistance to many types of solvents and chemicals.

EPO-TEK 302-3M has the lowest water-absorption of any standard EPO-TEK optical formulation. It shows only 0.075 percent weight gain after 24 hours immersion in fresh water, and 0.095 percent gain after the same interval in salt water. EPO-TEK 314 also demonstrates excellent moisture resistance. After 48 hours at 30°C in saline solution, the weight gain is 0.22 percent; in fresh water the gain is 0.18 percent.

Toxicity

The use of solvent-free adhesives in medical devices continues to increase due to safety considerations and consequent legislation. Several of the solvent-free EPO-TEK epoxy formulations have passed tests by independent testing laboratories for compliance with U.S. Pharmacopeia (USP) Class VI biocompatibility standards. Meeting these standards is an indication that these materials are nontoxic and will likely prove safe when used in medical instruments and components that are introduced inside the human body. USP Class VI compliance is considered essential for all materials employed in producing devices that require FDA approval for medical or dental use.

TYPICAL APPL CATIONS AND EPO-TEK ADHESIVE RECOMMENDAT ONS

APPLICATIONS / EPOXY	301	301-2	302	302-3M	305	310	314	320	353ND	354	360	377
Bond 125µ glass fiber to fiber optic connector	x	x	x	x	x		x		x	x	x	x
Bond 125 or 140µ fiber to fiber optic connector		x								x		
Bond fiber optic cable jacket to non-crimp type fiber connect									x	x	x	
Glass fiber potting with good visual indication of wicking									x	x	x	
Plastic & glass fiber potting or lens & prism bonding	x	x							x	x	x	
Coating or potting optically sensitive components								x				
Casting or potting optically clear components & indicators	x	x	x	x	x		x					
Excellent spectral transmission in thin bond lines	x	x	x	x	x		x					
Fast, room temperature curing	x		x									
High temperature requirements							x		x	x	x	x
No electrical resistance change within high humidity environ.												x
Yag laser (UV transmission)					x							
Autoclavable									x			x
Medical Applications	x	x							x			x
Potting I.R. Detectors		x										
Flexible: Coating or bonding stress sensitive components		x				x						
Optical Replication		x										
Fiber optic bundling in ferrules	x	x		x			x		x	x	x	x
Low stress on large core glass fibers in connectors		x								x		

About the authors

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