LOCTITE®

Examining the Effect of Sterilization on Bulk Adhesive Properties

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Any changes in the structural integrity of a disposable device after sterilization are related to the interaction of the adhesive and the substrate materials.

Sterilization can greatly influence the integrity of a medical device, especially those bonded with adhesives. Therefore, it is critical that device manufacturers select the adhesive best suited for the substrate materials, end-use environment, and sterilization technique.

The data shown in **Charts 1** and **2** reflect the post-sterilization performance of three light cure acrylic adhesives used to bond 22-gauge (ga) stainless steel cannulas to either polycarbonate or plasma-treated polypropylene hubs.

The pull strength data from this study on actual devices shows post-sterilization performance similar to that of the untreated control group. Often there is actually an improvement in strength. This improvement may be the result of elevated temperatures enhancing the adhesive's cross-linking reaction, or of annealing that actually relaxes stress on the component.

But what happens when we eliminate both substrates and joint design from the test and look only at the properties of the adhesive itself and how it is affected by sterilization? To answer this question, let's focus on the performance of typical light cure acrylic and light cure silicone adhesives when exposed to autoclave, ethylene oxide and gamma irradiation sterilization.

Adhesives Studied

This study tested the Loctite® light cure acrylic adhesives referenced in **Charts 1** and **2**:

- **3921™**, a highly fluorescent, 150 cP (centipoise) adhesive that provides bond strength on dissimilar materials. This material is commonly used in needle bonding applications.

Autoclave samples were exposed for 6 minutes to 120°C temperatures at 0.103 MPa.

- **Tensile strength** at break measured the strength of the adhesive in terms of force per unit area. For this study, tensile strength was the point on the stress strain curve where the adhesive sample failed rather than where the sample yielded.
- **Elongation**, reported as a percentage, measures how far the adhesive can be stretched prior to breaking.

Study Results

The results of this study are represented in **Charts 3, 4** and **5**. **Chart 3** reports modulus results and illustrates that gamma, ethylene oxide, and control results are very consistent. Autoclave exposure, however, reduced the rigidity of 3972 by more than 50%. The end-use application truly determines whether this loss of rigidity is critical for the success of the device.

Chart 4 shows the results of tensile strength at break testing. The tensile strength of Loctite® 3921™ increased after autoclave while its modulus decreased. The tensile strength of Loctite® 3933™, Loctite® 5240™ and Loctite® 5056™ was very consistent across all sterilization methods.

Chart 5 shows the results of elongation testing. After autoclave, the 3921™ exhibited an improved ability to stretch under stress. The remaining adhesives retained their ability to stretch or elongate even after sterilization, regardless of the sterilization method employed.

Depending on the adhesive chemistry and sterilization method selected, the study data shows similar performance in all three areas – modulus, elongation and tensile strength – to performance recorded before sterilization. Put simply, the adhesives tested did not change dramatically as a result of sterilization.

As a result of this study, engineers designing disposable medical devices should feel confident that the bulk properties of the adhesives will not change significantly when sterilized via ethylene oxide or gamma irradiation. Any changes to the structural integrity of a medical device after sterilization is related to the interaction of the adhesive and the substrate materials, not to changes to the bulk properties of the adhesive.

Engineers designing non-disposable medical devices should be aware of the difficulties that autoclave presents to adhesives. While the silicones tested in this study performed well under autoclave, designers must understand that this study exposed the adhesives to only one cycle of autoclave sterilization. A typical non-disposable medical device is exposed to hundreds of autoclave cycles over its usable life, a factor that will undeniably affect the long-term performance of the adhesive.

Regardless of the type of medical device being designed and manufactured, the relationship between the end use of the device, the substrates specified, the adhesive chemistry, and the actual design of the bond joint can all impact the strength of the final assembly, yielding different results for each device. To ensure a robust device design, engineers must thoroughly test the assembly under both manufacturing and sterilization conditions.

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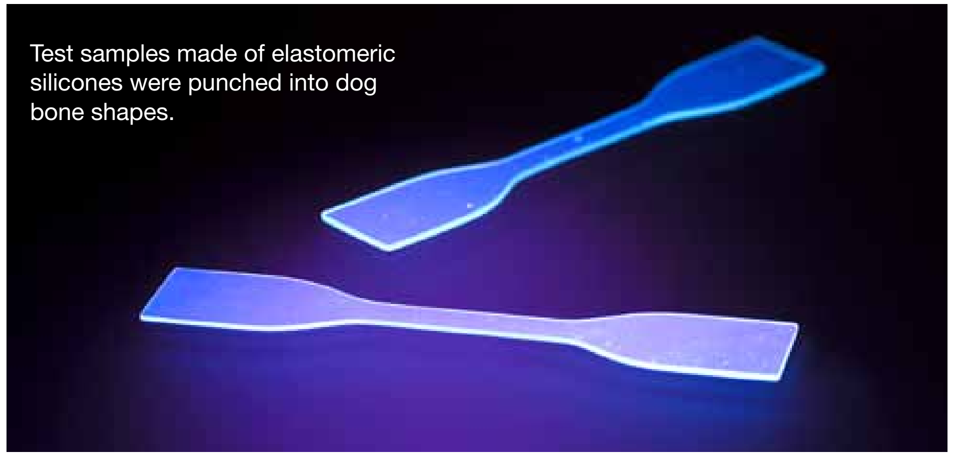


Test samples made of elastomeric silicones were punched into dog bone shapes.

- **3933™**, a 3,250 cP adhesive that provides bond strength to polycarbonate and other thermoplastics with minimal stress cracking. This material is typically used for bonding thermoplastic substrates in fluid devices such as housings for filters and fittings.
- **3972™**, a 4,500 cP adhesive that provides tack-free curing capabilities at wavelengths greater than 254 (nm) including the visible wavelengths in excess of 405 nm. This material is used in devices where there is significant adhesive exposed in the bond line, for example in tube fittings where the bond line is not encapsulated between two substrates and a tack-free surface is required to prevent contamination.

These light cure acrylic adhesives were selected primarily due to their glass transition temperature (T_g) and their ability to be cured under ultraviolet (UV) and/or visible (Vis) wavelengths. T_g is the temperature at which a substance changes from a glassy solid to a rubbery soft material. Loctite® 3921™ has a T_g of 82˚C, while 3933™ and 3972™ have T_gs of 54˚C and 49˚C respectively.

Glass transition temperature is critical to the performance of a medical device exposed to sterilization. An adhesives is far less likely to support a load once its T_g has been exceeded as the material becomes soft and pliable, losing its rigidity and strength. While the adhesive's ability to support a load returns after cooling down, any assembly that is under stress during the sterilization cycle may fail at sterilization temperatures.

The study also included testing two silicone adhesives formulations. For this, the cure method was the most important performance criteria in the selection process:

- **5056™**, a 2,200 cP light cure silicone, offers superior heat and moisture resistance and bonds well to silicone tubing and polycarbonate or thermoplastic fittings. This traditional light cure adhesive will not cure in areas that are not exposed to light of the appropriate wavelength and intensity during the curing process.
- **5240™**, a 25,000 cP dual cure silicone, cures on exposure to light and moisture and offers high tear strength. The secondary moisture cure mechanism allows the adhesive to cure in shadowed areas where light cannot reach.

Both of these adhesives would typically be used for bonding respiratory devices such as masks or breathing circuits, or for assembling components made from silicone substrates.

Chart 1 : 22 ga Needle Pull Strength on Polycarbonate

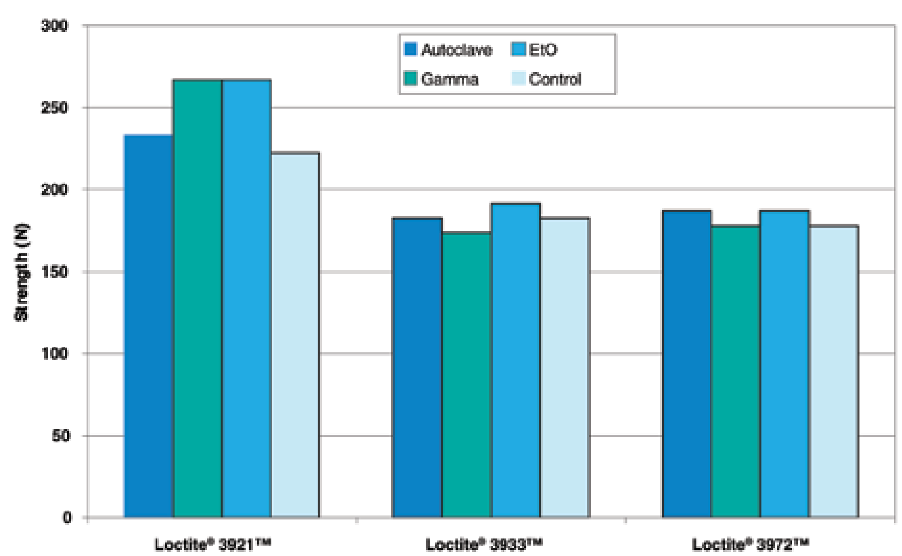


Chart 2: 22 ga Needle Pull Strength on Treated Polypropylene

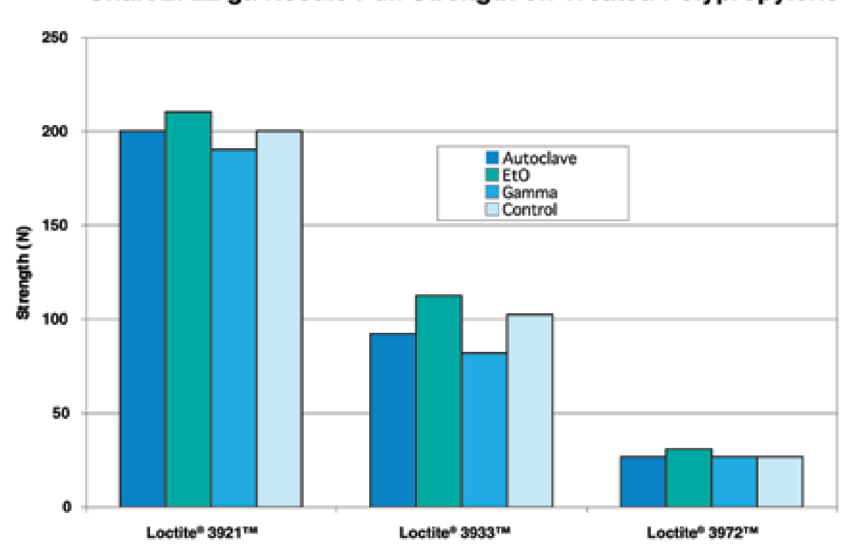


Chart 3: Modulus Results

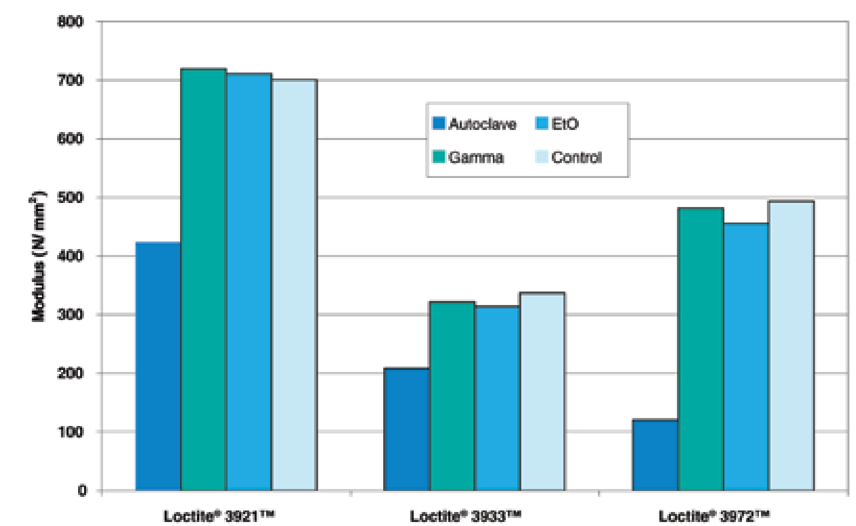


Chart 4: Tensile Strength at Break

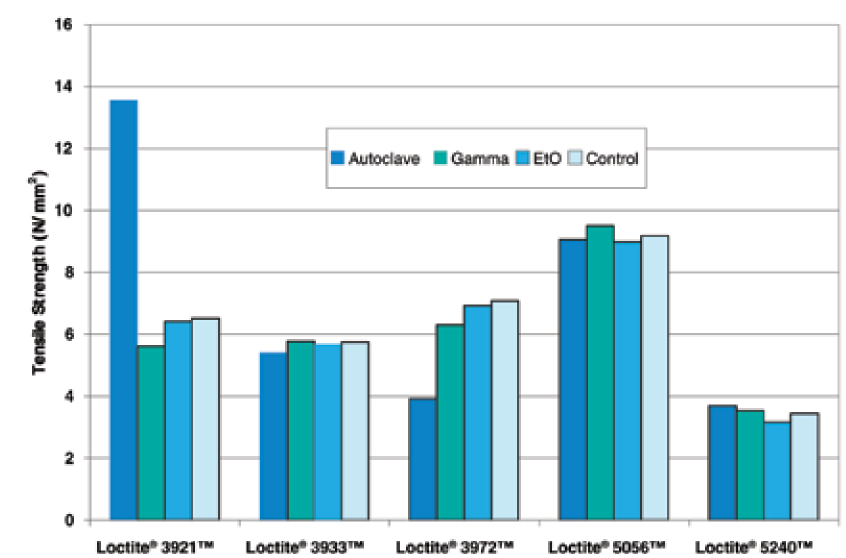
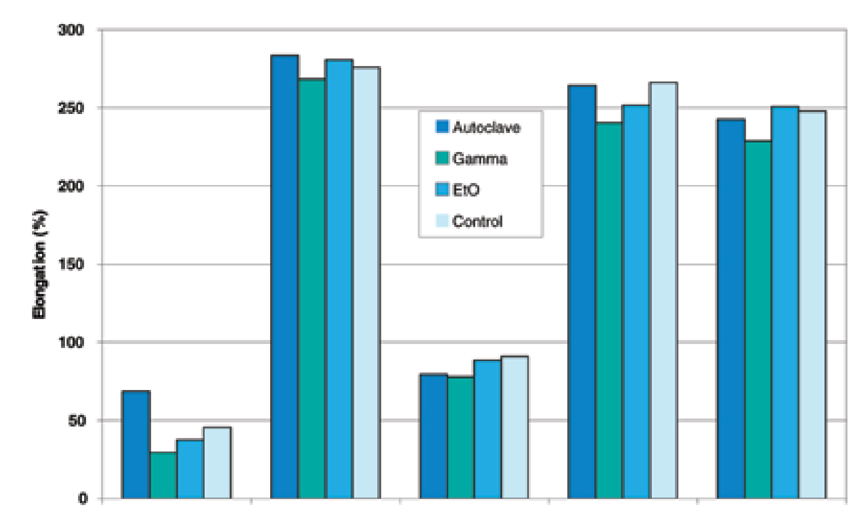


Chart 5: Elongation Results



Test Method

Glass transition temperature was not a critical factor in the selection of silicone adhesives as the T_g of silicones is typically in the -40ºC range and all of the tested sterilization methods operated above the T_g of silicones.

For this study, Henkel's lab manufactured bubble-free films from all the test adhesives. These films were made in molds that allowed light transmission and generated films with consistent thicknesses of 0.8 mm for the light cure acrylics and 2 mm for the silicones.

Post-cure, the adhesive films returned to ambient conditions and were prepared into test samples. The cured light cure acrylic films were machined into tensile bars with dimensions of 150 mm by 6 mm. The elastomeric silicones were punched into dog bone shapes with dimensions of 25 mm wide at the jaws of a mechanical properties tester and 6 mm wide at the neck and an overall length of 115 mm. This shape allowed the force to be concentrated in the neck area, forcing failure away from the jaws.

Once the specimens were prepared, gamma irradiation, ethylene oxide and autoclave sterilization processes began. The gamma irradiation took place for 108 minutes between 27.3 KiloGray (kGy) to 30.5 kGy. The ethylene oxide specimens were exposed to sterilant for 6 hours at 15.2 in Hg absolute at 54.4˚C. The autoclave samples were exposed for 6 minutes to 120˚C temperatures at 0.103 megapascal (MPa).

Once the sterilization processes were completed, the samples were tested in a mechanical properties tester for elastic modulus, tensile strength at break, and elongation at break. Samples were placed in the mechanical properties tester and pulled until they broke. Different measurements were taken for each attribute tested.

- **Elastic modulus**, for this study, is defined as the ratio of stress over strain. This attribute is more relevant for rigid materials such as light cure acrylics than for flexible materials such as silicones. For this reason, silicones were not included in the modulus testing.