AdTech
European Harmonized Training for Adhesive Bonding Technology

EUROPEAN ADHESIVE ENGINEER

MODULE 4.2

THERMAL EFFECTS ON ADHESIVE JOINTS

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4.2 Thermal Effects on Adhesive Joints

Scope:

- ✓ Differential thermal expansion
- ✓ Thermal transition in adhesives
- ✓ Thermal degradation of adhesives
- ✓ Thermal conductivity of adhesives
- ✓ Temperature limits of adhesives

Differential thermal expansion [4]

Thermal expansion of adhesives:

- Linear thermal expansion coefficients (coefficients of thermal expansion – CTE) of adhesives are much greater than of a metallic adherends.
- Only if adherends are polymers, thermal expansion coefficient are approximately the same.
- When large areas are bonded, these differences can lead to significant and damaging interfacial stresses as the temperature changes.

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Differential thermal expansion [4]

- Expansion coefficients of adhesives (CTE) can be reduced by the addition of mineral fillers.
- Expansion coefficients of polymers are greater in the rubbery state than in the glassy state

Differential thermal expansion [4]

Thermal expansion of adhesives:

- Typical CTE values for polymer adhesives are 10-100 times higher than adherends from metallic materials.
- As long as the adhesive is below glass transition temperature *Tg* it will be compliant enough to relax and accommodate interphasal stresses.
- Once glass transition temperature *Tg* is reached (by heating), the adhesive is less compliant and stresses begin to built up.

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Differential thermal expansion [4]

Thermal stresses in a bonded joint depend on CTE differences between materials of adherends and adhesives.

For polymeric adherends the best way to minimize thermal stresses is to blend low and high CTE polymeric adhesives to match CTE of adherend.

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Differential thermal expansion [4]

Coefficients of linear thermal expansion CTE for some adhesives and polymer adherends

Polymer or adhesive type	CTE [10^{-6}/K]
Epoxy (above *Tg*)	190
Epoxy (below *Tg*)	70
Polymethymethacrylate PMMA (above *Tg*)	530
Polymethymethacrylate PMMA (below *Tg*)	260
Low density polyethylene (LDPE)	100
High density polyethylene (LDPE)	130
Polystyrene	60-80

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Differential thermal expansion [4]

The T_g is measured by methods such as Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analyzers (DMA) or Differential Thermomechanical Analyzers (DTA).

It is greatly affected, not merely by the chemical composition of the epoxy compound, but by cure conditions such as time, temperature, specific response to heating, amount of load applied, degree of orientation and rate of testing.

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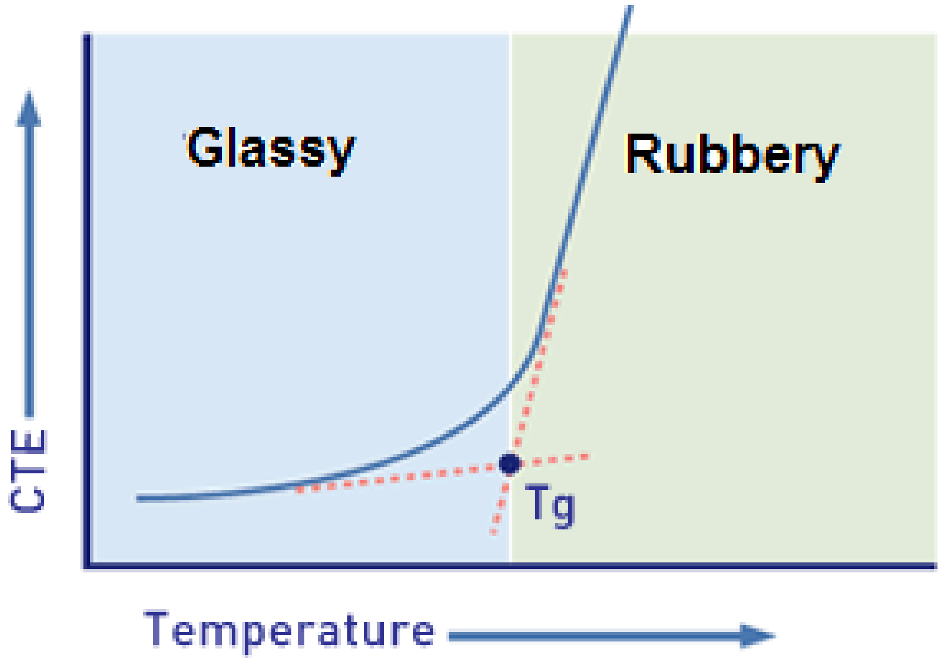
Differential thermal expansion [4]



Change of CTE of typical adhesive with a temperature and position of Glass transition temperature *Tg*

Differential thermal expansion [4]

Coefficients of linear thermal expansion CTE for some metallic and inorganic adherends

Adherend material, metallic or nonmetallic	CTE [10^{-6}/K]
Unalloyed steel	11-13
Aluminium	24-25
Titanium	8-9
Soda glass	8
Wood (along grain)	3-5
Wood (across grain)	35-60
Glass reinforced carbon	4-6

Differential thermal expansion [4]

Coefficients of linear thermal expansion CTE for some adhesives below and above *Tg*

Adhesive	CTE (below Tg) [10^{-6}/K]	Tg [° C]	CTE (above Tg) [10^{-6}/K]
Epoxide adhesives with hardeners			
BF3 amine	63	141	170
Diethylenetetramine	60	122	180
1,2-diaminobenzene	57	190	210
Methylene bis(1-chloroaniline)	83	149	190
Polymeric adhesives			
Polyviny acetate	210	30	600
Polymethymethacrylate (PMMA)	260	105	530

Differential thermal expansion [4]

Example 1: Calculate maximum rise of temperature for operating a bonded joint to reach yield strength of adhesive for the following data:

- Adherend: aluminium $CTE_{Al} = 25 \times 10^{-6} / K$
- Adhesive: epoxy $CTE_e = 70 \times 10^{-6} / K$; modulus of elasticy $E = 4000\ MPa$;

  yield strength $\sigma_y = 25\ MPa$; glass transition temperature $T_g = 150°\ C$

$\sigma_y = (CTE_e - CTE_{Al}) \times E \times \Delta T \rightarrow \Delta T_{max} = \sigma_y / ((CTE_e - CTE_{Al}) \times E)$

$\Delta T_{max} = 185\ K$ *or* $185°\ C > T_g$ ....yield strength of adhesive is reached above T_g where CTE_e is much higher, approximately $200 \times 10^{-6} / K$

THIS CALCULATION IS ONLY APPROXIMATE, SINCE MODULUS OF ELASTICITY OF ADHESIVE IS TEMPERATURE DEPENDENT!

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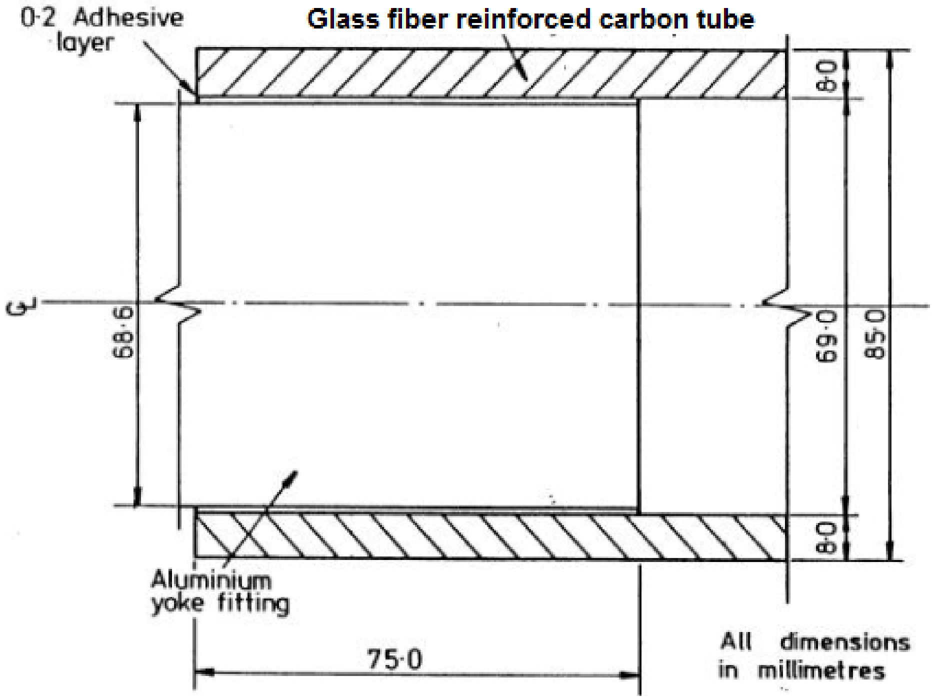
Differential thermal expansion [4]

Example 2: Shear and normal stress in a simple adhesive jointed axial bond between solid inner adherend from aluminium and outer tube adherend from glass reinforced carbon. The operating temperature is -130°C and axial loading of the joint is 20 kN. Length of a joint is 75 mm.



Differential thermal expansion [4]

Material data for the calculation:

- Inner solid adherend from aluminium:

CTE_{Al} = 25 ×10^{-6} / K

modulus of elasticy E_{Al} = 70000 MPa

- Outer adherend from GRC (tube):

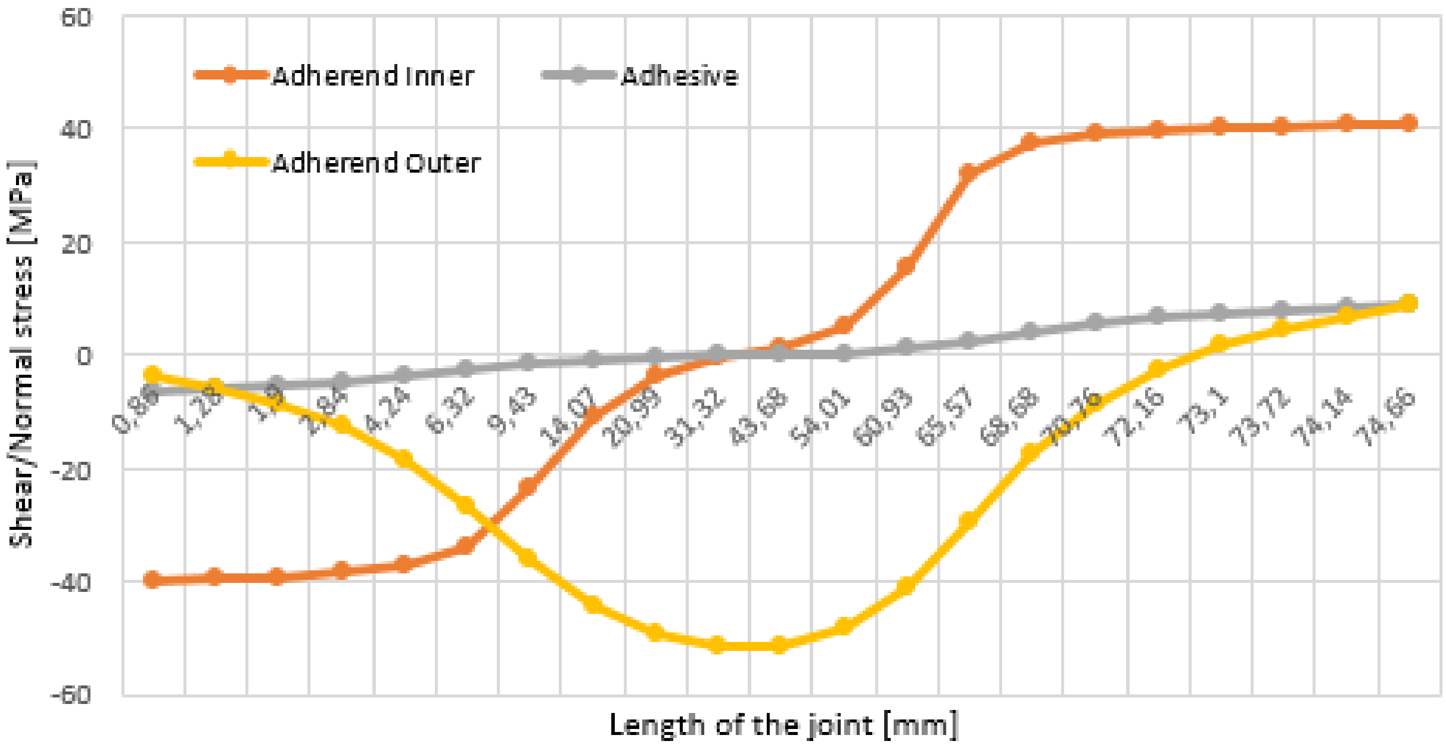
$CTE_{GFR\text{-}C}$ = 5,4 ×10^{-6} / K

modulus of elasticy $E_{GFR\text{-}C}$ = 29000 MPa

- Adhesive with a thickness of 0.2 mm:

Shear modulus at operating temperature G_A = 1400 MPa; Yield shear strength τ_y = 27 MPa

Differential thermal expansion [4]



Relationship between shear/normal stress versus length of the adhesive bonded joint

Differential thermal expansion [4]

Conclusions from the Example 2:

- Maximal normal stress in the Inner Adherend (Al) occurs at the edges of the bonded joint
- Maximal normal stress in the Outer Adhered (GRC) occurs in the middle of the joint
- Peak values of normal stress occurs at the ends of the joint and causes an origin of the failure
- Shear stress in the adhesive is distributed lineary along the length of the joint
- Shear stress in the adhesive is always smallar than elastic limit at operating tmeperature

Thermal Transition in Adhesives [4]

Definition of Glass Transition Temperature T_g of adhesives

Temperature at which molecular mobility changes dramatically as the polymer goes from glassy (glass like) to rubbery (rubber like) behavior. At low temperatures all polymers are in the glassy state, by which it is meant that they are relatively hard and inflexible. At some temperature, which is characteristic for each polymer, the material becomes soft and flexible, and so enters the rubbery state. This change is the glass transition, and it occurs at the glass transition temperature T_g. Most polymeric adhesives are amorphous rather than crystalline, and the glass transition is a property of the amorphous phase.

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Thermal Transition in Adhesives [4]

Both glassy and rubbery polymers are used as adhesives

Examples of glassy adhesives:

- epoxides
- phenolics

Examples of rubbery adhesives:

- reactive acrylics

Important:

The glass transition temperature of a cured adhesive is of critical importance, crossing T_g in service is not acceptable. Due to large changes in mechanical properties, it is unacceptable for the glass transition temperature to exist in the same region of adhesive bonded joint operating temperature.

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Thermal Transition in Adhesives [4]

Glass transition temperatures and brittle points of some adhesives

Adhesive (with brittle points)	Tg [° C]
Polydimethysiloxane (silicone)	-120
Poly cis-1,4 isoprene (natural rubber like)	-73, -70, -69
Polybutadiene polyol	-70
Polychloroprene	-46
Polymethyacrylate	9, 10, 11, 19
Polyvinyl acetate	30
Styrene-butediene rubber	61
Polymethymethacrylate (PMMA)	105
Polysodium acrylate	230

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Thermal Transition in Adhesives [4]

Optimizing the Glass Transition Temperature T_g

When a polymer system such as an epoxy adhesive, potting compound or sealant is heated, significant changes occur in mechanical strength properties and thermal, electrical and chemical resistance which do not necessarily recover upon cooling. While these changes generally take place over a limited temperature range, a single temperature called the glass transition temperature (T_g) is generally selected for convenience.

Thermal Transition in Adhesives [4]

Polymeric compounds, which are exposed to a temperature below the T_g, exhibit much higher physical strength and stiffness, as well as greater electrical insulation, dimensional stability and chemical resistance than they do above their T_g.

Furthermore, performance upon structural loading is greatly enhanced. For convenience, the T_g is often considered the maximum sustainable operating temperature for a polymer system, especially in structural applications.

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Thermal Transition in Adhesives [4]

Measurement of the Glass transition temperature T_g

The T_g is measured by methods such as Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analyzers (DMA) or Differential Thermomechanical Analyzers (DTA). The DSC automatically plots a chart from which can be calculated approximate *Tg* value.

It is greatly affected, not merely by the chemical composition of the adhesive compound, but by cure conditions such as time, temperature, specific response to heating, amount of load applied, degree of orientation and rate of testing.

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Thermal Transition in Adhesives [4]

Importance of T_g from the designers view

- It is an important measurement if a conscientious design engineer wants to assess how his adhesive bonded joint is going to operate within a service temperature range and how it could have an impact on other materials, which are bonded.
- For example, an automotive application bonding dissimilar materials (metal to a composite), the application is likely to see temperatures of below freezing during wintery conditions and then in summertime temperatures, a sealed cabin can reach in excess of +60° C, and even much higher temperatures in the engine compartment. It is important that automotive adhesive can withstand these extremes, whilst still offering high strength, impact and vibration resistance.

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Thermal Transition in Adhesives [4]

Operating of the adhesives above the *T_g* temperature

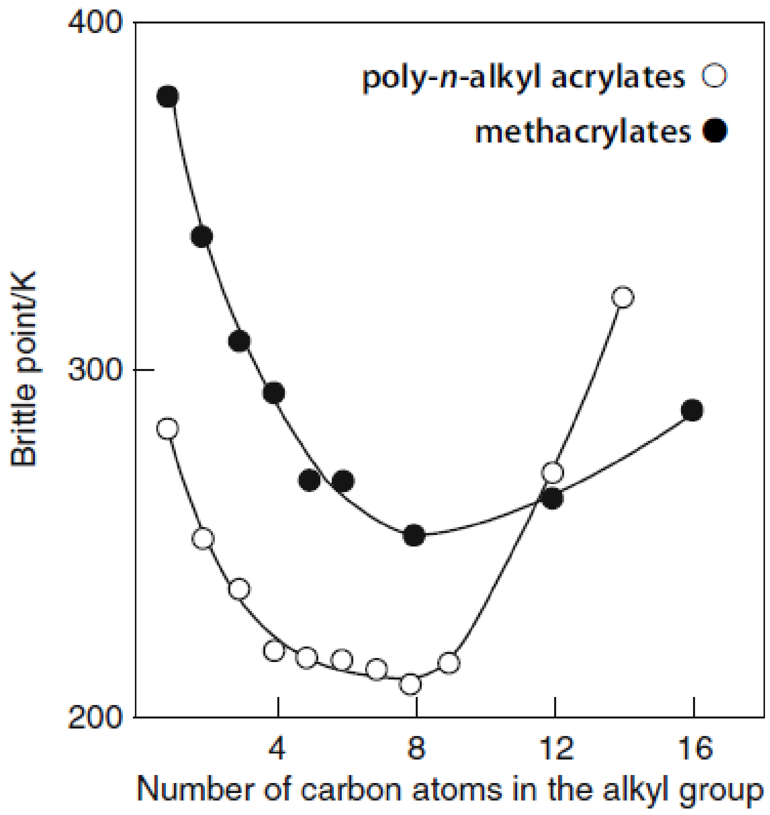
Structural adhesives such as epoxides, acrylate based adhesives and PMMA continue to work well above the *Tg* temperature. They become slightly more flexible which actually improves certain features such as impact and vibration resistance as well as slightly higher peel strength.

Low temperatures and freezing conditions make adhesives more brittle. If there are bonded two dissimilar materials with different coefficients of thermal expansion (CTE), a toughened, more flexible adhesive is recommended.

Thermal Transition in Adhesives [4]

Brittle points for some polyacrylates and polymethylacrylates:

Lower brittle point is prefered for this group of adhesives, since this mean that rubbery behaviour will last to the temperatures below 0° C.



Thermal Transition in Adhesives [4]

Impact of the glass transition temperature of epoxy adhesives:

- The T_g is strongly dependent on the cure schedule. Low temperature cures such as room temperature will result in the lowest possible T_g of all for that chemistry. Very high T_g values are not achievable by room temperature curing. If the same material is cured at an elevated temperature, a higher T_g will result.

- As an example an adhesive could have a T_g between +60° C and +110° C, based on the cure schedule. This is why it is important to maintaining tight cure temperature control.

Thermal Transition in Adhesives [4]

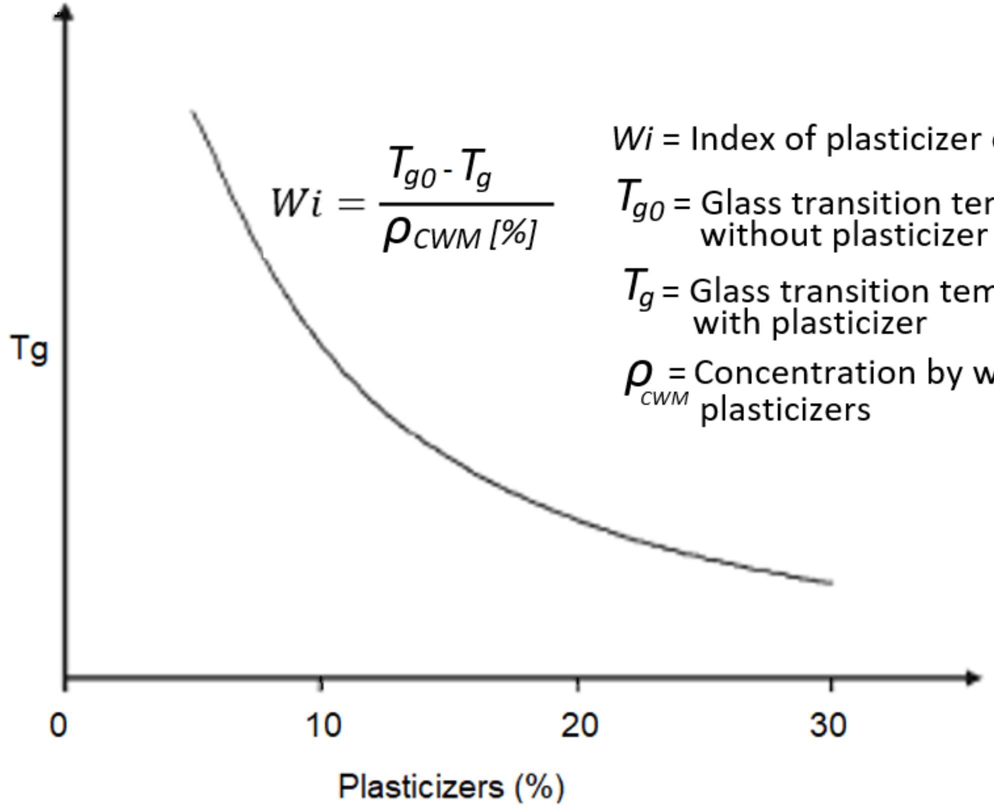
Impact of the glass transition temperature of epoxy adhesives:

- The glass transition temperature T_g of epoxides can be significantly reduced by moisture absorption, a factor which should be considered when designing for humid applications.
- Adhesives with the highest T_g have the best heat resistance and therefore deliver the best tensile properties at high temperature. Unless there are significant exotherm reactions associated with the cure process, as a general rule, a T_g cannot be significantly higher than the highest temperature during curing process.

Thermal Transition in Adhesives [4]

Lowering the glass transition temperature of adhesives by adding a plasticizers:

- paraffinic oils,
- phthalate esters,
- polybutenes,
- lanolin,
- chlorinated hydrocarbons,
- aliphatic hydrocarbons,
- mineral oils.



$$Wi = \frac{T_{g0} - T_g}{\rho_{CWM}\ [\%]}$$

Wi = Index of plasticizer effect

T_{g0} = Glass transition temperature without plasticizer

T_g = Glass transition temperature with plasticizer

ρ_{CWM} = Concentration by weight of plasticizers

Thermal Degradation of Adhesives [4]

Introduction

Polymers are the major components of all adhesives, and thus have a dominant influence on their properties. There is relatively little specific information in the literature on the thermal properties of adhesives; therefore, we must depend on the properties of the polymer to know about an adhesive.

Thermal degradation of adhesives changes mechanical properties like:

- stress relaxation (constant strain is applied to polymer at fixed temperature and time-dependent stress is expressed as relaxation modulus $E(t)$),
- modulus of elasticity E,
- tensile strength.

Thermal Degradation of Adhesives [4]

Principles of high-temperature stability

- A serious limitation to the use of organic polymers in general and of adhesives, in particular, is their poor resistance to thermal degradation. Considerable effort has been put into the development of High-temperature adhesives (Polybenzimidazole, Polyether ether ketone, Polyimide, Polyphenylquinoxaline).
- A very wide range of reactions is involved in polymer degradation, depending on the polymer concerned and the environment. Main chain reaction occurs in many polymers. This often involves chain scission, but sometimes cross-linking results.

Thermal Degradation of Adhesives [4]

Principles of high temperature stability

Some molecular features giving thermal stability to a polymer can be described. They are based on the recognition that degradation must start with bond breaking somewhere in the structure and that this will often initiate a series of reactions leading to a loss of desirable properties:

1. Only strong bonds should be incorporated in the structure;
2. No easy pathways for rearrangement should be present;
3. Maximum use should be made of delocalization (resonance) stabilization;
4. Where possible, polybonding should be used.

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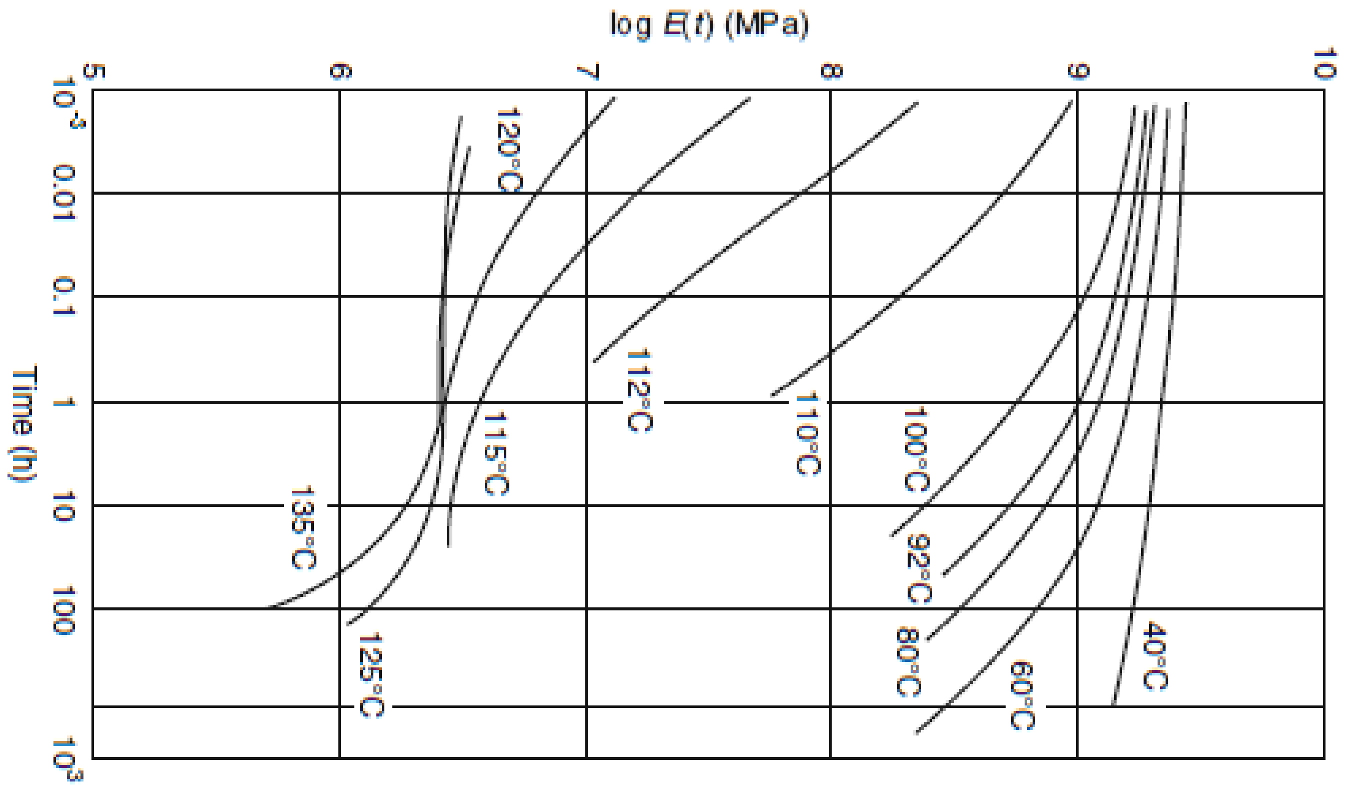
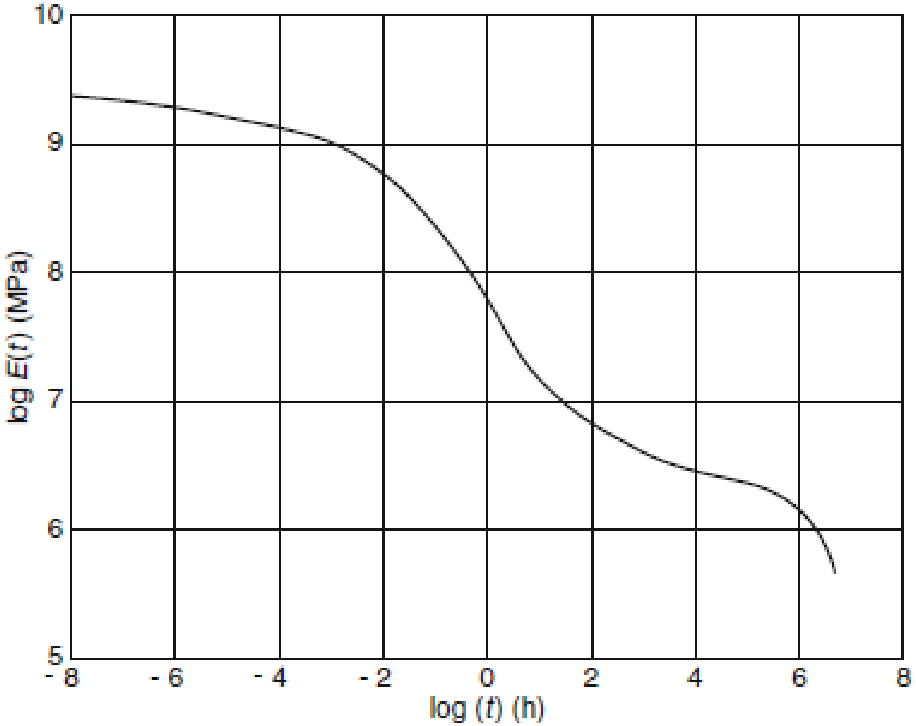
Thermal Degradation of Adhesives [4]

Dependence of relaxation modulus *E (t)* upon time for PMMA at temperatures +40°C to +135 °C



Thermal Degradation of Adhesives [4]

Dependence of relaxation modulus *E(t)* upon time for PMMA at fixed temperature +110 °C for shorter and longer time range



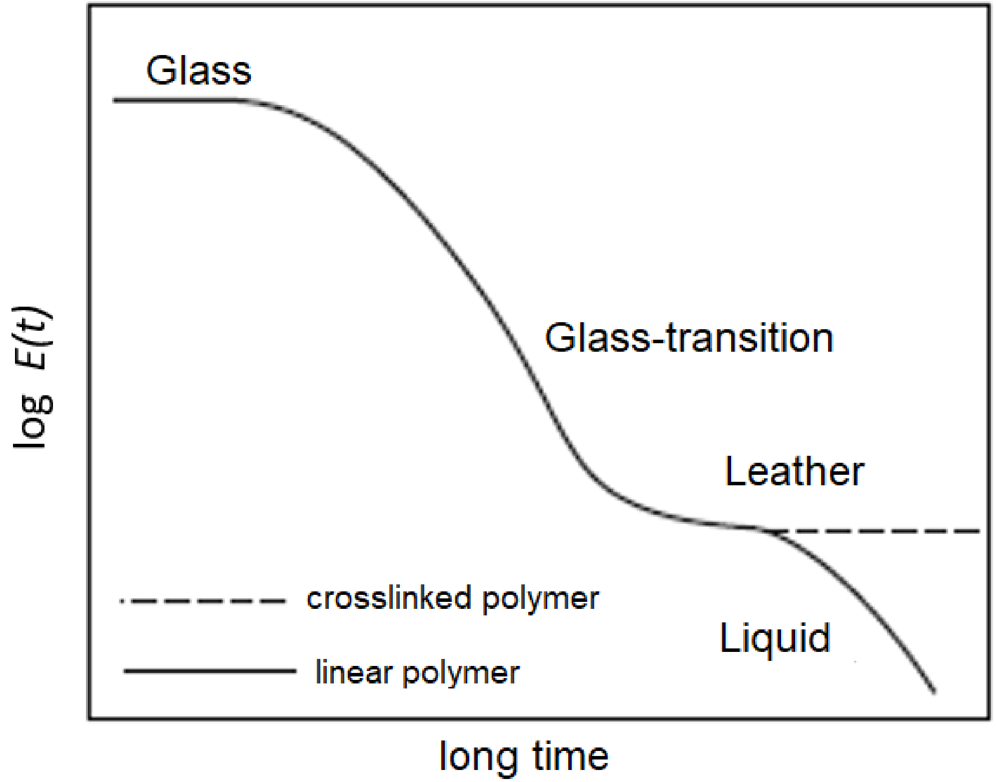
Thermal Degradation of Adhesives [4]

Difference in relaxation modulus *E(t)* between linear and crosslinked polymers

Linear polymer has 4 regions of behaviour:

- Glass
- Glass-transition
- Leather
- Liquid

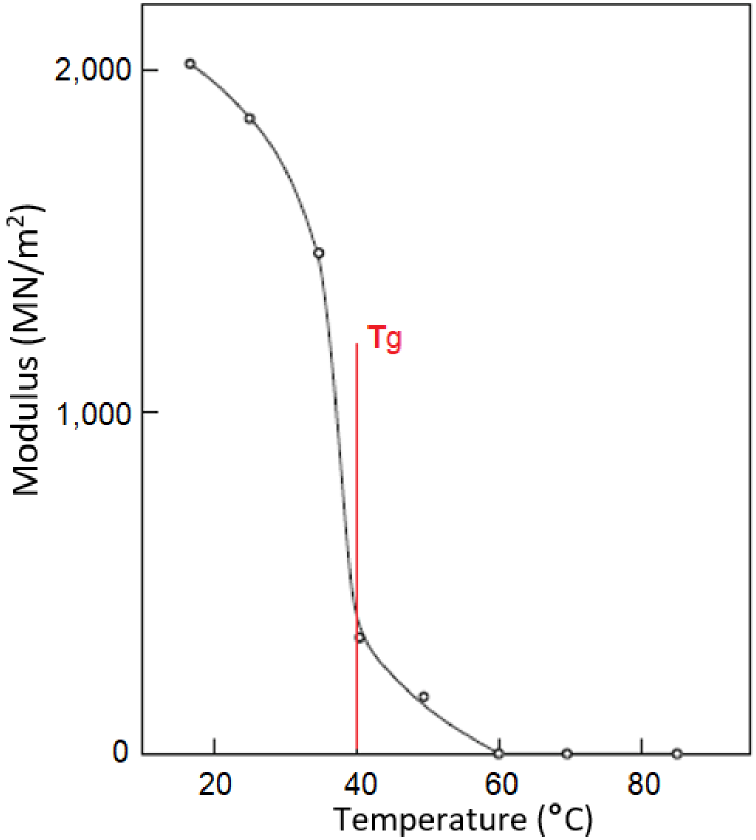
Cross-links in crosslinked polymer prevent flowing at higher temperatures.



Thermal Degradation of Adhesives [4]

Effect of temperature on the modulus of elasticity *E* of the epoxy-polyamide adhesive

Rapid fall of elasticity modulus *E* begin at temperatures that are 20 K below T_g.
This is typical viscoelastic behaviour.
From glass transition temperature $T_g = +40°C$ to the melting temperature of adhesive, modulus of elasticity *E* fall lineary to the value 0.



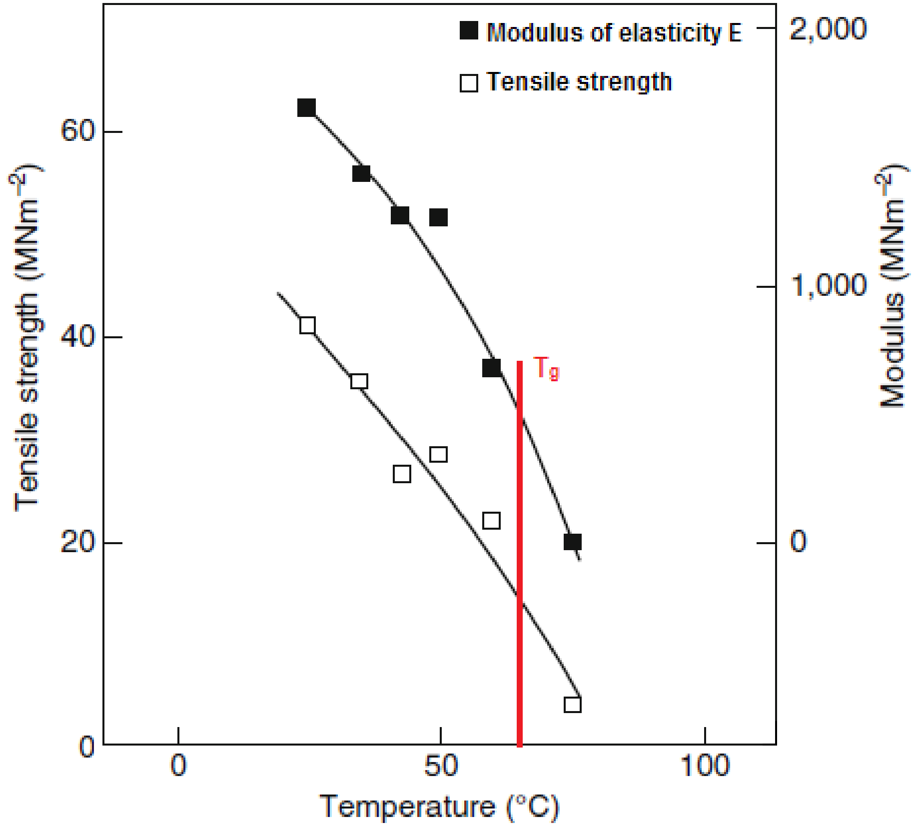
Thermal Degradation of Adhesives [4]

Mechanical properties of lap bonded joint in dependence of temperature

Adhesive: aliphatic amine-cured epoxide

Glass transition temperature: $T_g = +65°C$

Tensile strength and modulus of elasticity falling in the same manner with the increasing temperature, even at temperatures higher than glass transition temperature.

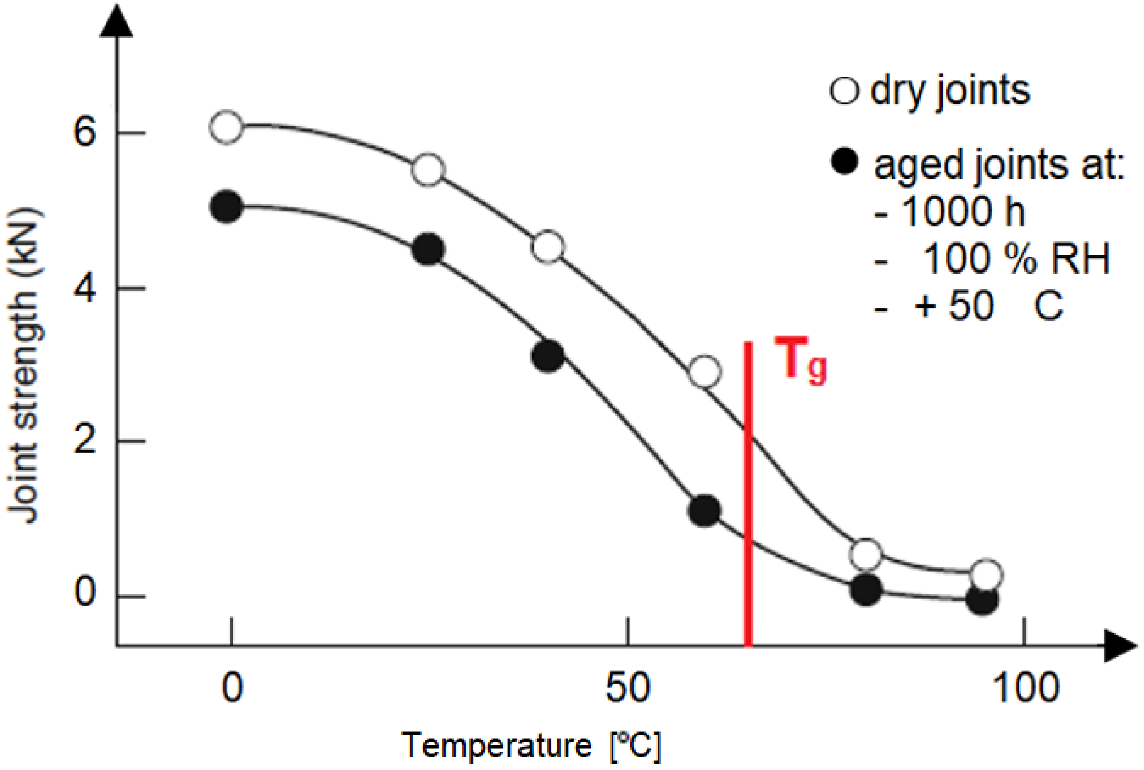


Thermal Degradation of Adhesives [4]

Strength of lap bonded joint in dependence of temperature

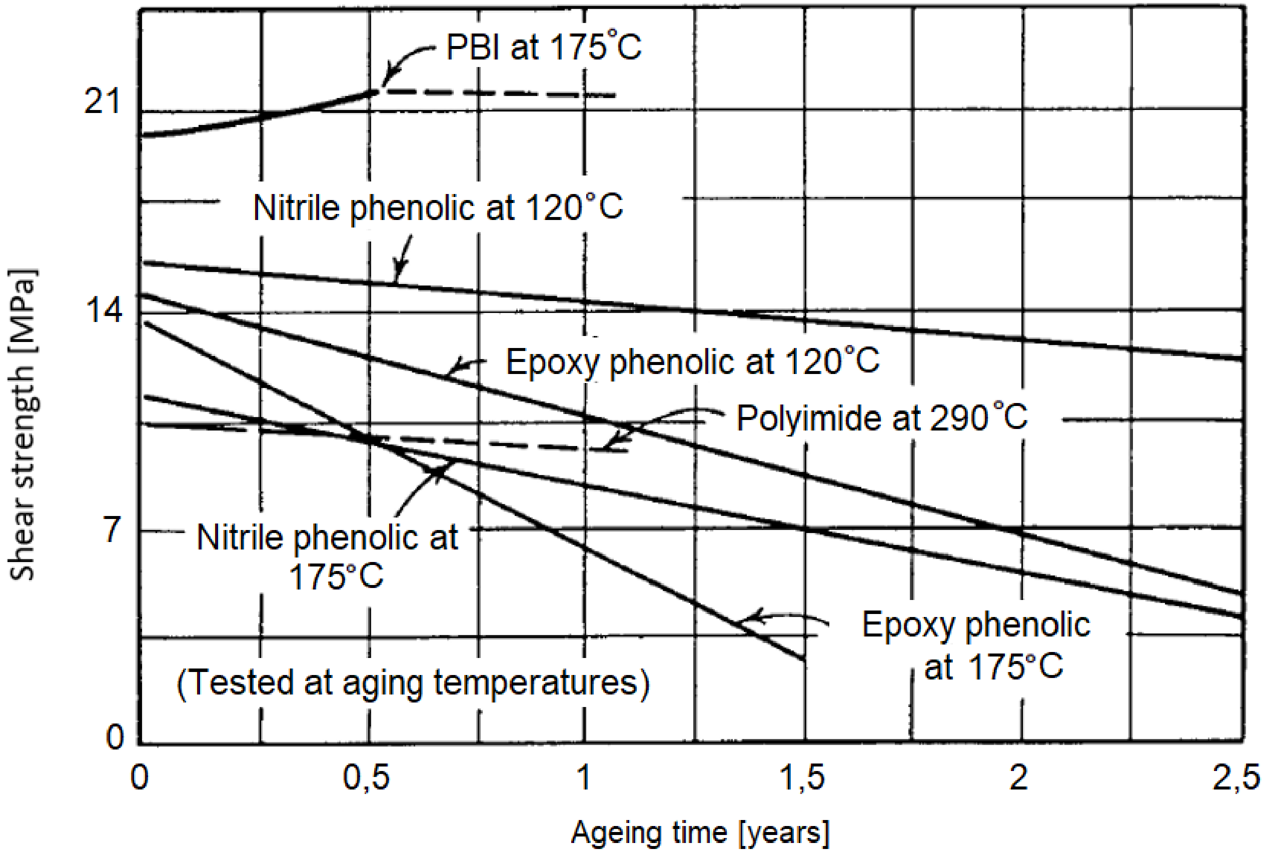
<u>Adhesive:</u> aliphatic amine-cured epoxide
<u>Glass transition temperature:</u> $T_g = +65°\,C$

Aged joints show lower joint strength in comparison to the dry joints. Otherwise, above the glass transition temperature joint strength falling rapidly.



Thermal Degradation of Adhesives [5]

Shear strength of high temperature adhesives regarding to ageing time



Thermal Degradation of Adhesives [4]

Oxidative degradation at higher temperatures

- Although all polymers will degrade at elevated temperature in the absence of air, the rate of degradation is generally increased when there is a supply of oxygen.
- In adhesive joints, the supply of oxygen will depend on the oxygen permeabilities of both the adhesive and the adherends, and on the distance that oxygen must diffuse in either of these.
- Metallic, ceramic and glass adherends are impermeable to oxygen and therefore represent a complete barrier to it, but composites with polymeric matrices are permeable to atmospheric gases.

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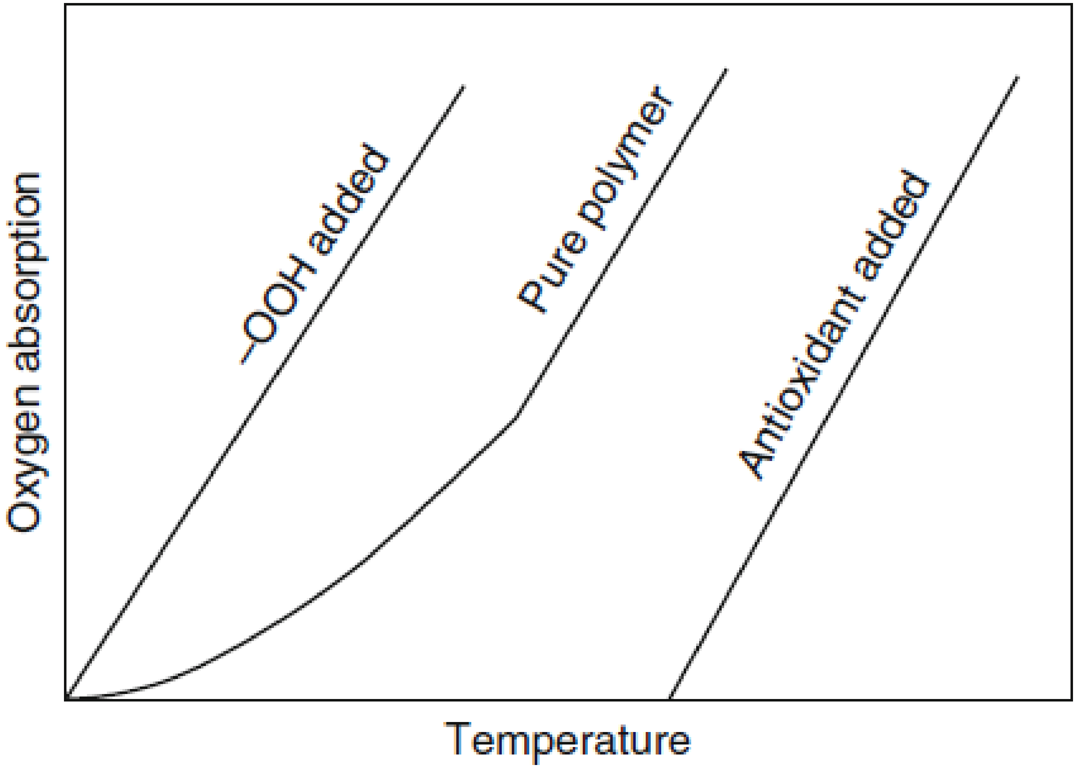
Thermal Degradation of Adhesives [4]

Oxidative degradation at higher temperatures

- The rate of oxygen diffusion into adhesives at elevated temperatures will probably be sufficiently high for impermeable adherends to offer little protection against oxidative degradation.
- Oxygen is absorbed by the polymer to form a hydroperoxide that subsequently decomposes to give radicals that then initiate degradation.

Thermal Degradation of Adhesives [4]

Oxygen absorption by a polymer



Oxygen absorption by a polymer

- The pure polymer has a build-up period in which the hydroperoxide is formed, but this is absent in the polymer in which hydroperoxide groups have previously been added.
- In the presence of an antioxidant, there is a long induction period in which the stabilizer scavenges any free radicals.
- The end of the induction period marks consumption of the stabilizer.
- Oxidative degradation is a complex series of chain reactions.

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Thermal Degradation of Adhesives [4]

Resistance to oxidative degradation

Due to their high content of aromatic groups, high-temperature adhesives such as polyimides, polyquinoxalines, and polybenzamidazoles (PBI) are excellent resistant to oxidative degradation.

Combined effects of UV radiation and oxygen

These combined effects are particularly damaging. However, unless one adherend is transparent to UV rays, this is not a factor in the weakening of polymer adhesives.

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Thermal Conductivity of Adhesives [4]

Basic physical backgrounds

- Adhesives are poor conductors of heat and electricity, but both can be increased by filling with powdered metals, especially silver.
- To increase thermal conductivity alone, metal oxide and metal nitride fillers can be used.
- The most effective of these is beryllium oxide BeO, which is both toxic and expensive; aluminum oxide Al_2O_3 is a practical alternative.

Thermal Conductivity of Adhesives [4]

Electrical conductive adhesives are filled with:

- silver (Ag)
- copper (Cu)
- graphite (C)

Fillers for increasing a thermal conductivity are:

- alumina, aluminium oxide Al_2O_3
- aluminium nitride AlN
- boron nitride BN

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Thermal Conductivity of Adhesives [4]

Adhesives with good electrical conductivity for electronic industry

Material or adhesive	Thermal conductivity λ [W/mK]
Silver (Ag)	410
Copper (Cu)	370
Graphite (C)	300-400
Epoxy with 75 % aluminium	4,5
Epoxy with 50 % aluminium	1,7-3,4
Epoxy with 50 % silver	2,4-2,6
Epoxy with 35 % graphite	1,9-2,0
Epoxy with 50 % copper	1,8

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Thermal Conductivity of Adhesives [4]

Adhesives with good thermal conductivity

Thermally nonconductive

Material or adhesive	Thermal conductivity λ [W/mK]
Cyanoacrylate	0,3
Unfilled epoxy	0,15-0,7
Epoxy with 25 % Al_2O_3	0,35-0,5
Epoxy with 35 % BN	1,0-1,4
Epoxy with 50 % Al_2O_3	1,3-1,5
Epoxy with 75 % Al_2O_3	1,3-1,7
Silicone rubber	2,3
Epoxy with 35 % AlN	2,6-2,9
Epoxy with 60 % AlN	3,5
Aluminium oxide Al_2O_3	34

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Thermal Conductivity of Adhesives [4]

Definition of high temperature limit

The temperature at which adhesive will survive in service for a period of 1 year.

Main points an engineer should consider when selecting an adhesive for high temperature applications:

- Many adhesives can withstand even +300° C for a few seconds, but if that exposure time is increased to hours, days or even months, the choice of suitable adhesive products is heavily reduced.

Temperature Limits of Adhesives [4]

- It is important to focus on applications that must withstand sustained temperatures of more than +180°C, what is chosen to be high temperature application of adhesive.

- Adhesives with high temperature resistance (limit) also have high glass transition temperature T_g values meaning they are rigid across their operating temperature range.

Temperature Limits of Adhesives [4]

Exceptions to the relationship between heat resistance and a high T_g:

Certain silicones and B-staged flexible epoxies do not necessarily follow these rules. B-staged epoxies (epoxy-nylon) has a T_g = +35°C and service temperature limit up to +260°C. T_g gives a good indication of good design practice in high temperature applications. When an adhesive is selected, design engineers should consider the expected maximum service temperature, T_g and mechanical properties requirements.

Temperature Limits of Adhesives [4]

Role of glass transition temperature in cold temperature extremes:

- In cold temperature applications the T_g does not provide the same clear window into adhesive performance as it does in high temperature applications.
- The dip in temperatures below the *Tg* causes adhesives to become highly brittle and more susceptible to low failure stresses. Theoretically, that reasoning would seem to favor flexible adhesives with low T_g values for the coldest applications.
- Epoxy adhesives do not experience a considerable loss of properties even at cryogenic temperatures, implying they work best in a rigid state that extends from their T_g temperature into far colder temperatures.

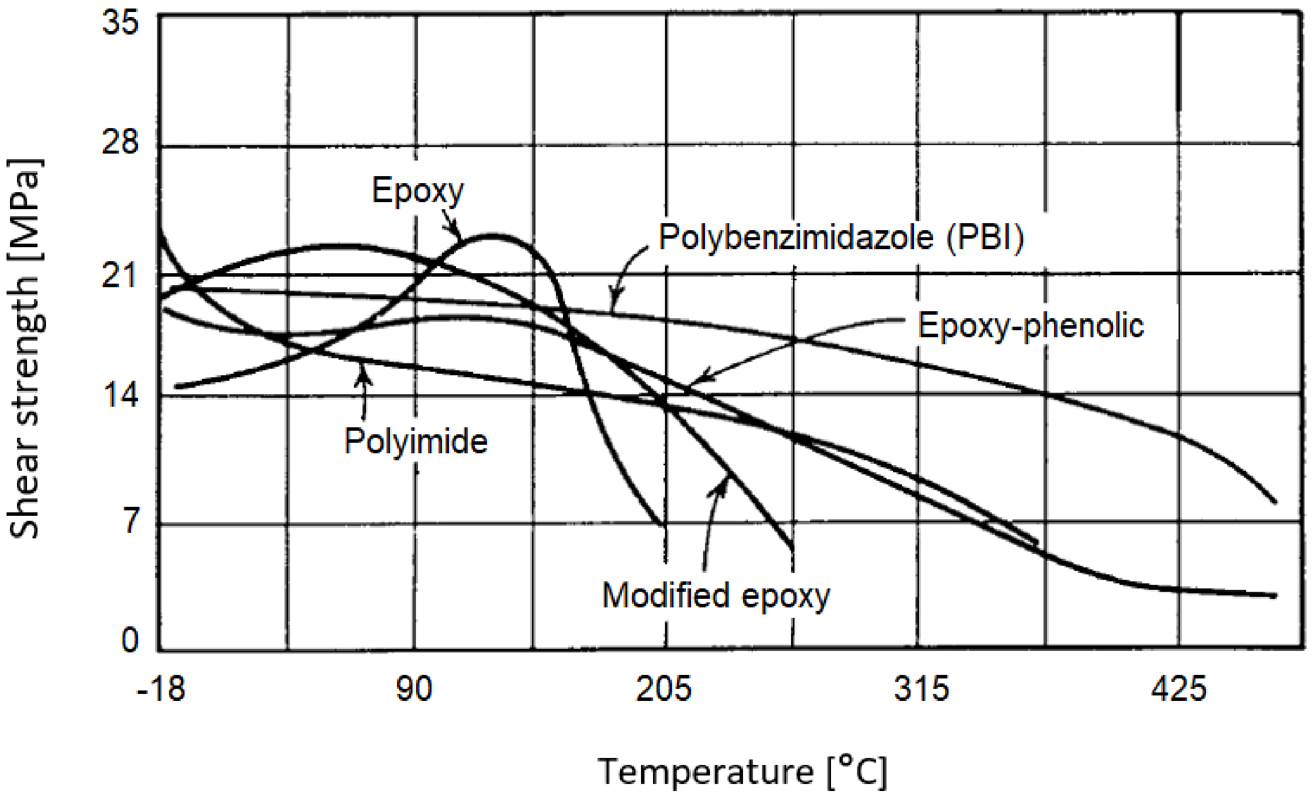
Temperature Limits of Adhesives [5]

Adhesives for high temperature service

- High temperature adhesives are usually characterized by a rigid polymeric structure, high softening temperature and stable chemical groups. The same factors also make these adhesives very difficult to process.
- Only epoxy-phenolic, bismaleimide, polyimide, and polybenzimidazole based adhesives can withstand long-term service temperatures higher than +180°C. modified epoxy and even certain cyanoacrylate adhesives have moderately high short term temperature resistance. Silicone adhesives also have excellent high temperature permanence, but exhibit low shear strength.

Temperature Limits of Adhesives [5]

Shear strength of high temperature adhesives depending on temperature



Temperature Limits of Adhesives [4], [5]

Mixed temperature limits (extreme heat and extreme cold-cryogenic temperatures):

- This is often the case in aerospace and spacecraft applications.
- Adhesives with a low T_g that function well in a cold environment tend to exhibit large coefficients of thermal expansion (CTE) as they heat up which can lead to thermal stress problems.
- Adhesives with a high T_g have lower, more manageable CTE across their entire operating temperature range and may be more suitable for these mixed extreme environments.

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Temperature Limits of Adhesives [4], [5]

Factors that determine the strength of adhesive over wide temperature ranges:

- Difference of CTE between adhesive and adherend.
- Elastic modulus *E* of adhesive at the service temperature.
- Thermal conductivity of adhesive.

Temperature Limits of Adhesives [4], [5]

Adhesives for low temperature service

Many applications for adhesives and sealants require service life at very low temperatures. Adhesives for superconducting components in advanced machines should maintain their mechanical performance in the range of -269°C (liquid helium) to -196°C (liquid nitrogen).

There are also certain applications that require the adhesive or sealant not only to resist extremely low temperatures but also to resist extremely high temperatures. These applications exist primarily in the space industry. Bonded structures on space vehicles could be exposed to cold when oriented away from the sun and to heat when oriented directly at the sun.

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Temperature Limits of Adhesives [4], [5]

Factors affecting low temperature performance of adhesives

The factors that determine the strength of an adhesive when used over wide temperature ranges are:

- The difference in coefficient of thermal expansion (CTE) between adhesive and adherend;
- The elastic modulus of the adhesive at the service temperature;
- The thermal conductivity of the adhesive.

Temperature Limits of Adhesives [4], [5]

Factors affecting low temperature performance of adhesives

- At low service temperatures, the difference in thermal expansion is very important, especially since the elastic modulus of the adhesive generally increases with falling temperature.
- The thermal conductivity of adhesive is also important in minimizing transient stresses during cooling. This is why thinner bond thicknesses and adhesives with higher levels of thermal conductivity have better cryogenic properties than thicker ones.
- In addition to external stress, the final bonding strength at a given temperature is affected by internal stress arising mainly from shrinkage during cure and differences in thermal expansion and contraction.

Temperature Limits of Adhesives [4], [5]

Factors affecting low temperature performance of adhesives

- Residual stresses resulting from thermal expansion or contraction are due to the differences in the coefficient of thermal expansion (CTE) between adhesive and adherend and also due to temperature distribution in the joint due to differences in thermal conductivity.
- At room temperature, a standard low elastic modulus adhesive may readily relieve stress concentration by deformation. At cryogenic temperatures the modulus of elasticity may increase to a point where the adhesive can no longer effectively release the concentrated stresses.

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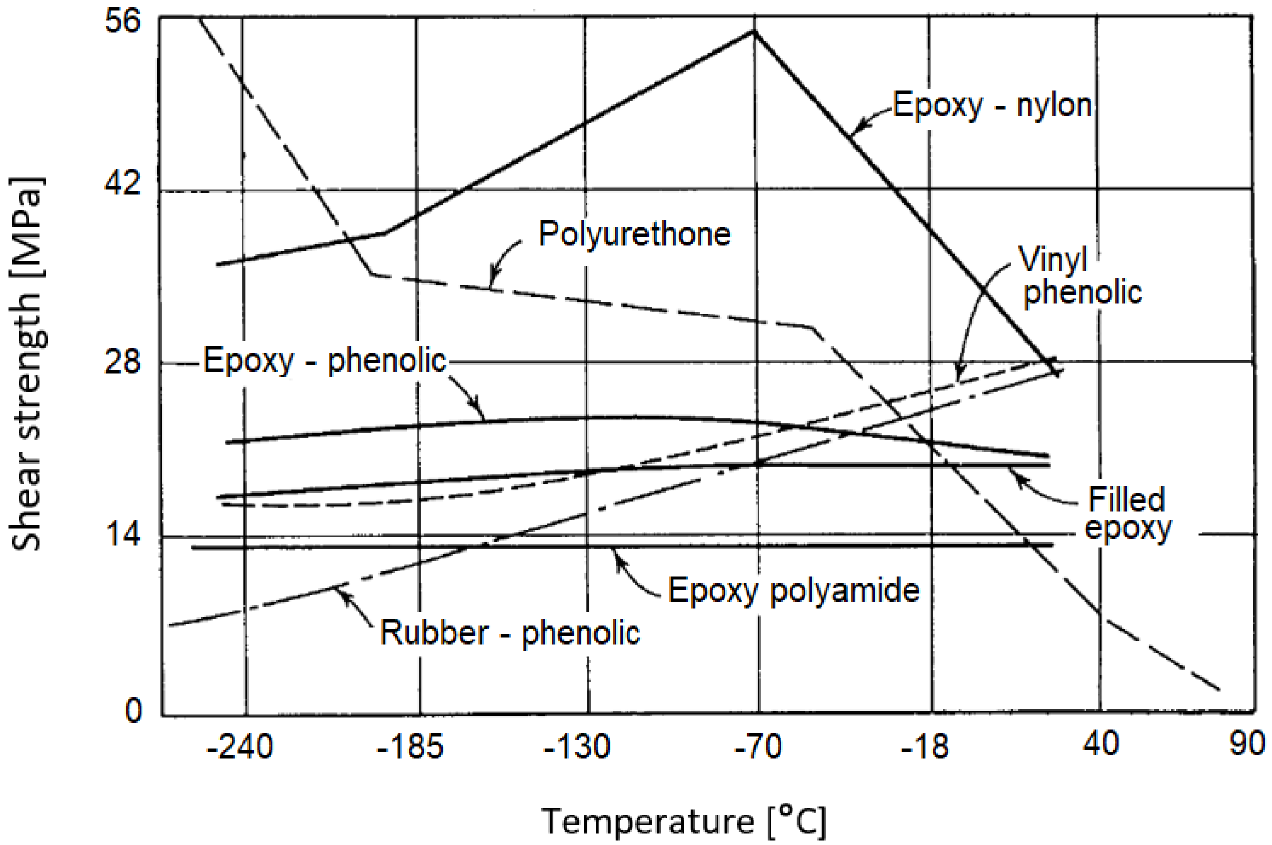
Temperature Limits of Adhesives [5]

Shear strength of cryogenic adhesives depending on temperature



Temperature Limits of Adhesives [4], [5]

Typical maximum and minimum temperature limits for adhesives

Adhesive (high temperature use)	Max. temperature limit [° C]	Min. temperature limit [° C]
epoxy + polyamide	+65	/
acrylate	+80	-40
cyanoacrylate	+80	/
epoxy + aliphatic amines	+100	-50
epoxy + acid anhydrides	+150	-50
silicones	+200	-70
polyimides	+260	/
vulcanizing silicone rubber	+260	-70
B-staged epoxy-nylon	+260	-150
polyimide	+290	-250
epoxy-phenolic	+300	-250
sodium silicate	+370	/

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