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Summary

The use of composite materials in a variety of applications has increased considerably in the last couple of decades. The main reason for this is that composite materials are lightweight materials with high strength and stiffness, which makes them interesting as an alternative to other materials, such as metals. For this reason, there is also significant activity on the development of composite materials at FFI, with the main aim of utilising them in applications that are relevant for military purposes. This also includes research on polymer nanocomposites, where nano-sized reinforcements are employed.

Epoxy polymers are widely used in composites and adhesives. For use in many applications, however, it is necessary to improve the material properties. One possible approach to improve the properties of the material is adding inorganic particles to the polymer. This reinforcement may modify mechanical properties, such as the stiffness, strength and toughness of the polymer material.

In this study, the effect of adding 20 nm silica nanoparticles to two different epoxy polymers, one amine-cured and one anhydride-cured, has been investigated. A commercial product of pre-dispersed silica nanoparticles in an epoxy resin, Nanopox F 400, containing as much as 40 wt% silica, was employed to reinforce the epoxies. The material properties of the neat epoxy polymers and the silica/epoxy nanocomposites were investigated by tensile testing, dynamic mechanical analysis (DMA), and indentation measurements. Particular emphasis was put on the measurement of the elastic modulus of the materials, and the elastic modulus obtained by the three different test methods was compared.

The elastic modulus of both epoxy polymers was increased by the addition of the silica nanoparticles. The relative increase in the measured mechanical properties was higher for the anhydride-cured polymer. For the amine-cured epoxy system, there was very good agreement between the elastic modulus measured by tensile testing and DMA. The elastic modulus from the indentation measurements was consistently higher. For the anhydride-cured epoxy system, there was also, in general, good agreement between the elastic modulus obtained from tensile testing and DMA. The indentation measurements, on the other hand, gave very different values. The measurements also showed that part of the increase in the elastic modulus for the anhydride-cured silica/epoxy nanocomposite could be due to a change in the polymer network structure, as indicated by a change in the glass transition temperature.

Sammendrag

Bruken av kompositttmaterialer i mange konstruksjoner har økt betydelig de siste par tiårene. Hovedgrunnen til dette er at kompositttmaterialer er lettvekts materialer med høy styrke og stivhet. Dette gjør dem interessante som erstatning for andre materialer, for eksempel metaller, som blir benyttet i dag. Av denne grunn utvikles det kompositttmaterialer ved FFI, og hovedmålet er anvendelser som er relevante for militære formål. Dette inkluderer også forskning på polymere nanokompositter, der forsterkninger av nano-størrelse blir benyttet.

Epoksypolymerer er mye brukt i kompositttmaterialer og lim. For mange bruksområder er det imidlertid nødvendig å forbedre materialegenskapene. En metode for å bedre materialegenskapene, er å tilsette uorganiske partikler til polymeren. Denne forsterkningen har potensiale til å endre mekaniske materialegenskaper, som for eksempel stivhet, styrke og bruddseighet.

I denne studien har effekten av å tilsette 20 nm silika nanopartikler til to ulike epoksypolymerer blitt undersøkt. En aminherdet og en anhydridherdet epoksypolymer ble benyttet. Et kommersielt produkt med dispergerte silika nanopartikler i et epoksyresin ble benyttet som forsterkningsmateriale. Dette produktet, Nanopox F400, inneholder så mye som 40 vektprosent silika. Materialegenskapene til de rene epoksypolymerene, og kompositter av silika og epoksy, ble studert ved hjelp av strekktesting, dynamisk mekanisk analyse (DMA) og indentering. Det ble lagt spesielt vekt på elastisitetsmodulen til materialene, og stivheten målt med de tre ulike teknikkene ble sammenliknet.

Stivheten til begge epoksyene økte ved tilsetning av silika nanopartikler. Den relative økningen i de målte mekaniske egenskapene var størst for den anhydridherdede polymeren. For den aminherdede polymeren var det god overensstemmelse mellom stivhet målt ved strekktesting og DMA. Stivhet målt ved indentering lå konsekvent høyere. For den anhydridherdede polymeren var det også relativt god overensstemmelse mellom stivhet målt ved strekktesting og DMA. Resultatene fra indentering var imidlertid svært forskjellige. Det ble også vist at stivheten til den rene anhydridherdede polymeren kan endres når blandingsforholdet mellom epoksy og herder endres – indikert ved endring i glassomvandlingstemperaturen. Dette kan ha hatt innvirkning på den målte stivheten til kompositten laget med denne polymeren.

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1 Introduction

Epoxy polymers are widely used in engineering adhesives and composites. Epoxies have many excellent material properties but for use in many applications, however, it is necessary to improve these properties. To fully exploit their potential, the epoxy polymers therefore often need to be reinforced by the addition of a particulate reinforcement. One such approach is to add inorganic particles to the polymer. Inorganic particles have a much higher stiffness and hardness than the polymer matrix, and they can be used to modify mechanical properties such as the stiffness, strength and toughness of the polymer material [1]. In general, the mechanical properties of particulate/polymer composites depend primarily on the particle size, the particle/matrix interface adhesion and the particle loading. The stiffness depends significantly on particle loading, while the strength and toughness are strongly affected by all three factors – particularly the particle/matrix adhesion and thus the stress transfer between the particles and the matrix.

An alternative for the reinforcement of epoxy polymers is inorganic silica particles. One commercially available product is the Nanopox from Evonik Hanse, which consists of 20 nm silica particles that are pre-dispersed in an epoxy resin. The particles are produced in-situ via a sol-gel process, and have a narrow particle size distribution. One main advantage of the Nanopox products is that the nanosilica particles are excellently dispersed in the epoxy resin. This helps in keeping the viscosity low, which is useful for processing and manufacturing purposes. A good dispersion of the reinforcing particles in a polymer is also regarded as beneficial for the optimisation of the mechanical properties of the composite. The presence of agglomerates will limit the positive effects of adding particles to the polymer. The excellent dispersion of the nanosilica from Nanopox F400 in an epoxy polymer is shown in Figure 1.1.

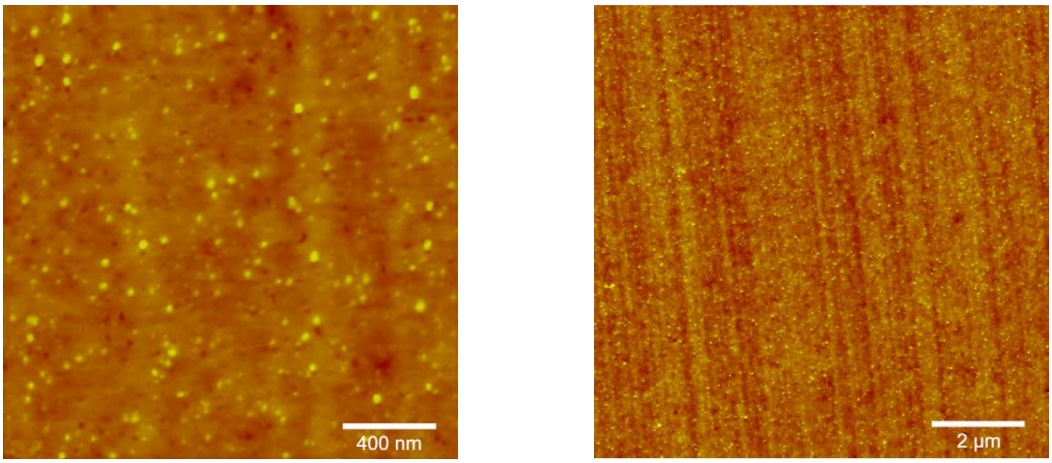


Figure 1.1 Atomic force microscopy images of an epoxy polymer containing 14.8 wt% nanosilica showing good dispersion of the nanoparticles [2].

The nanosilica in the Nanopox product range has been shown to efficiently improve the properties of epoxy polymers [2-8]. The toughness, the elastic modulus in tension and compression, and the fatigue properties can be significantly improved. Particular focus has been on the toughening effect of the nanosilica, and the toughening mechanisms that are acting.

Improvements in strength have also been observed, although not as dramatic as the improvements in toughness. The property improvements of epoxy materials have been reviewed by Sprenger [9]. The nanosilica has also been used to improve the polymer matrix properties of fibre-reinforced composites.

In this study, we have investigated the reinforcing effect of pre-dispersed silica nanoparticles in an epoxy resin. The material used was Nanopox F 400. In addition to the above mentioned advantages, unlike many other types of nanoparticles, one advantage of using Nanopox F400 is that composites with higher volume concentrations of inorganic particles can be produced. This is often not possible due to the high viscosity of other nanoparticle/polymer blends. The silica/epoxy nanocomposites that were produced here, were investigated by dynamic mechanical analysis, tensile testing, and indentation measurements. In all cases, the elastic modulus, i.e. Young's modulus, of the materials was determined, and the results of the three different test methods were compared. However, little effort has been made to explain the mechanisms behind the results that were obtained.

2 Materials

2.1 Epoxy resins

Two different epoxy polymers were investigated in this work; one amine-cured and one anhydride-cured system. The amine-cured polymer was Araldite LY 556/XB3473, while the anhydride-cured polymer was Araldite LY 556/Aradur 917/Accelerator DY 070. Both systems are from Huntsman.

LY 556 is a standard bisphenol A based epoxy resin with an epoxy equivalent weight (EEW) of 183-189 g/eq. XB3473 is an amine hardener containing two different diamines [10]. The active hydrogen equivalent weight (AHEW) of XB3473 is ≈43 g/eq, see the datasheet in Appendix A. Aradur 917 is methyltetrahydrophtalic acid anhydride hardener with an AHEW of 166.2 g/eq [11]. DY 070 is an imidazole accelerator. Data for the cure kinetics of the Araldite LY 556/Aradur 917/Accelerator DY 070 system are available [11].

2.2 Nanosilica

A colloidal sol of silica (silicon dioxide, SiO_2) nanoparticles in an epoxy resin was employed to produce silica/epoxy nanocomposites. The Nanopox F400 was supplied by Evonik Hanse, Geesthacht, Germany. The silica phase in Nanopox F400 consists of surface-modified silica spheres with an average particle size of 20 nm and a very narrow particle size distribution. The technical data sheet states a value of 295 g/eq for the EEW of Nanopox F400, see Appendix A.

The silica content in Nanopox F400 is 40 wt%, and the density of the silica is 2100 kg/m^3. The epoxy phase is a standard bisphenol A diglycidyl ether. Previous thermogravimetric analysis at FFI has confirmed that the silica content in Nanopox F400 stated by the manufacturer is correct

[12]. The silica/epoxy blend has a comparatively low viscosity due to the low degree of agglomeration of nanoparticles in the resin.

3 Experimental methods

3.1 Specimen preparation

Plates of both the neat epoxy polymer and silica/epoxy nanocomposites were prepared. Prior to preparing the composite plates, the effect of varying the amount of hardener was investigated (not shown here). From dynamic mechanical analysis (DMA), it was found that this could significantly alter the storage modulus and the glass transition temperature of the polymer phase of the composites. It was also found that the EEW of the F400 was of importance. For both polymer systems, it was the aim to avoid a large decrease in the glass transition temperature of the composites.

The mixing ratio of the different components was determined from the EEW of LY556 and F400, and from the AHEW of the two different hardener systems. Slightly different values of EEW and AHEW, which were based on manufacturer and literature values, were assumed for the two different polymer systems. (See Appendix A for recommended resin/hardener mixing ratios of the two epoxy systems.) For the amine-cured polymer, it was assumed that the EEW of LY556 was 187 g/eq, and that the AHEW of XB3473 was 43 g/eq. For the anhydride-cured polymer, it was assumed that the EEW of LY556 was 184.7 g/eq, and that the AHEW of Aradur 917 was 166.2 g/eq. On this basis, the formulations in Table 3.1 were established.

Plates of the neat epoxy polymers were prepared by first mixing the correct weight ratio of the epoxy resin and the hardener (and the accelerator in the case of the anhydride-cured system), according to the formulations in Table 3.1. After mixing, the blends were stirred manually using at spatula, thereafter heated to 80^{o}C and stirred thoroughly again. Finally, the blends were vacuum degassed and cast in preheated metal moulds that had been coated with a release agent. The plate thickness was 4 mm. The curing cycles that were employed were: (1) 2 hours at 120^{o}C, 2 hours at 140^{o}C, and 2 hours at 180^{o}C for the amine-cured system, and (2) 4 hours at 80^{o}C, and 8 hours at 140^{o}C for the anhydride-cured system.

The procedure for producing plates of the silica/epoxy nanocomposites was similar to that of the neat epoxy polymers, except that F400 was also mixed into the blends. Again, the formulations given in Table 3.1 were prepared, resulting in plates with four different loadings of silica for each polymers system. It was very difficult to remove all the air from the blend of the amine-cured system. This was increasingly difficult with increasing content of F400. However, although the blends contained some air, the cured composite plates seemed to be relatively free of air and blisters by visual inspection.

Table 3.1 Formulations used for preparation of the epoxy polymers and the composites.

Silica content		Parts by weight of components				
wt%	**vol%**[1]	**LY556**	**F400**	**XB3473**	**Aradur917**	**DY070**
Amine-cured system						
0.0	0.0	100	0	23.0	--	--
6.6	3.7	80	20	21.3	--	--
13.4	7.8	60	40	19.6	--	--
20.3	12.3	40	60	17.9	--	--
27.5	17.2	20	80	16.3	--	--
Anhydride-cured system						
0.0	0.0	100	0	--	90.0	1.00
4.4	2.5	80	20	--	83.0	0.92
9.1	5.4	60	40	--	75.9	0.84
14.2	8.6	40	60	--	68.9	0.77
19.8	12.3	20	80	--	61.9	0.69

3.2 Density

The density of the neat epoxy polymers and the composites were measured by immersion in water according to ASTM D 792-08 [13]. Further information about the procedure is given in [10]. The measurements were conducted with a Sartorius YDK01 Density Kit combined with a Sartorius analytical balance. Average values from six pieces were obtained. The pieces were taken from the each of the broken test specimens from the tensile testing (see Section 3.5).

The volume fraction of silica, V_{silica}, in the composite was calculated from

$$V_{silica} = W_{silica} \cdot \frac{\rho_{composite}}{\rho_{silica}} \tag{3.1}$$

where W_{silica} is the weight fraction of silica in the composite, $\rho_{composite}$ is the density of the composite, and ρ_{silica} is the density of silica. W_{silica} for each particular sample was calculated from the formulations given in Table 3.1. It was assumed that no air was present in the samples.

A theoretical value of the composite density, $\rho_{composite,calculated}$, can be calculated from

$$\rho_{composite,calculated} = \frac{1}{\frac{W_{polymer}}{\rho_{polymer}} + \frac{W_{silica}}{\rho_{silica}}} \tag{3.2}$$

where $W_{polymer}$ is the weight fraction of polymer in the composite, and $\rho_{polymer}$ is the density of the polymer.

[1] The volume concentrations of the cured materials in Table 3.1 are determined in Section 4.1.

The silica content by volume, V_{silica}, in %, is used to compare the test results in this report, since the change in e.g. the elastic modulus of a composite material is usually reported as a function of the volume fraction of the reinforcement.

3.3 Dynamic mechanical analysis

Dynamic mechanical analysis (DMA) was performed on a DMA 2980 Dynamic Mechanical Analyzer from TA Instruments. Rectangular specimens with the dimensions 3 mm × 10 mm × 60 mm were employed. The specimens were polished for correct thickness. The analysis was conducted in a three-point bending mode employing a low friction three-point bending clamp with a specimen free length of 50 mm. The oscillation frequency was 1 Hz, the pre-load force was set to 0.05 N, and the 'Force Track' was set to 150%. The specimens were heated from ambient to 200^{o}C at a heating rate of 3^{o}C/min. The value of the storage modulus, E', was measured at a temperature of 30^{o}C, and the value of the glass transition temperature, T_g, of the epoxy polymer was determined from the peak value of the loss modulus, E''. Average values of three replicate specimens are reported. Since a limited number of specimens were tested, the standard deviations given for the DMA-results should be examined critically since they may not be representative.

3.4 Crosslink density

The molecular weight between crosslinks, M_c, for an unmodified thermoset polymer can be determined from

$$\log_{10}(E_r / 3) = 6.0 + 293 \cdot \rho_{polymer} / M_c \qquad (3.3)$$

where M_c has the units of g/mol, and E_r, is the rubbery equilibrium tensile modulus in units of Pa, see [4] and the references therein. M_c for the neat epoxy polymers was determined. The value for E_r was determined from the plateau of the storage modulus in the DMA test at T_g + 40^{o}C. (As mentioned above, the T_g is here defined as the peak value of the loss modulus.)

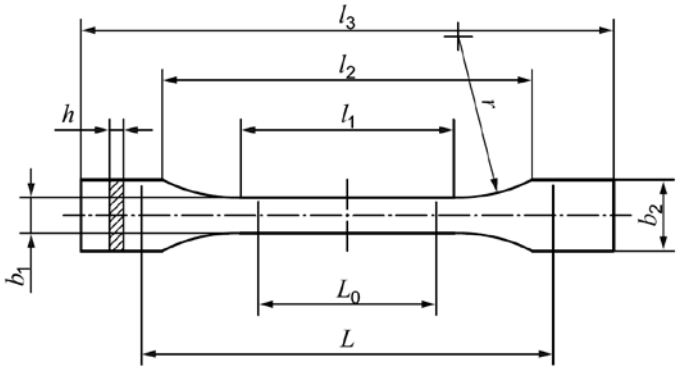
3.5 Tensile testing

Tensile testing was conducted on dumbbell specimens that were machined from the neat epoxy and silica/epoxy nanocomposite plates. The testing was conducted according to the relevant ISO standard [14;15] on a Zwick BZ2.5/TN1S material testing machine, employing specimens of type 1BA, see Figure 3.1 and Figure 3.2. The cross-sectional area of the narrow parallel-sided portion of the test specimen was 4 mm × 5 mm. The specimens of the amine-cured system were used without any further preparation. However, one side on some of the anhydride-cured specimens was uneven, and this side was therefore polished for uniform specimen thickness. The polishing did not have any influence on the intrinsic material properties.

The specimens were gripped in the broader ends and aligned so that the load-direction was parallel to the narrow portion. A clip-gauge extensometer, which was used for recording the strain, was thereafter attached, see Figure 3.3. The test speed was 1 mm/min. The most important

properties that were determined, were the tensile modulus of elasticity, E_t, the maximum tensile stress, or the tensile strength, σ_m, and the maximum elongation, or tensile strain at break, ε_b. E_t was determined from the linear part of the stress-strain curve in the strain range from 0.05% to 0.25%. Average values of six replicate specimens are reported. The ambient temperature during the testing was 22 ± 1°C.

In Appendix B, a figure showing typical stress-strain curves for different plastic materials is displayed. It is indicated how the different tensile properties are determined.



		Dimensions in mm
l_3	Overall length	100
l_1	Length of narrow parallel-sided portion	30.0 ± 0,5
r	Radius	≥30
l_2	Distance between broad parallel-sided portions	58 ± 2
b_2	Width at ends	10.0 ± 0.5
b_1	Width at narrow portion	5.0 ± 0.5
h	Thickness	≥2
L_0	Gauge length	25.0 ± 0.5
L	Initial distance between grips	--

Figure 3.1 Dimensions of type 1BA test specimen in ISO 527-2:2012.

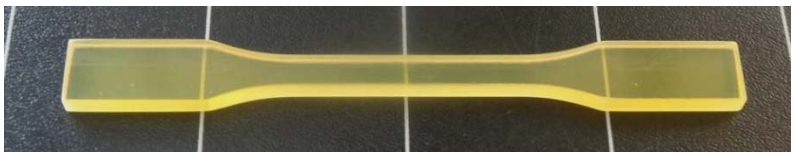


Figure 3.2 Tensile test specimen, type 1BA. The distance between the parallel lines is 30 mm.

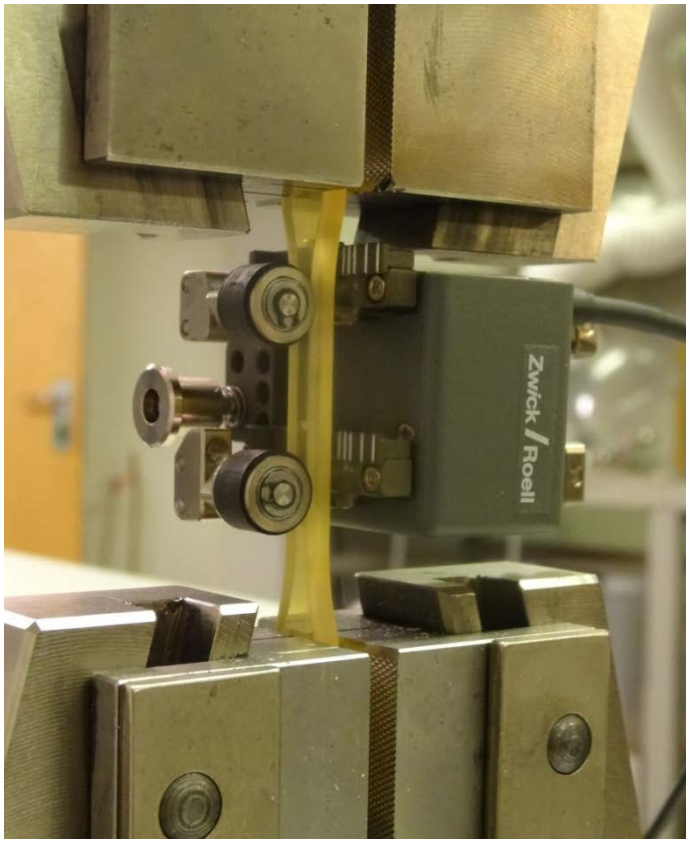


Figure 3.3 Experimental set-up for the tensile testing with the extensometer attached.

3.6 Indentation measurements

The indentation measurements were conducted on a DUH-211 Shimadzu Dynamic Ultra-micro Hardness Tester. Prior to indentation measurement, a specimen of approximate size 10 mm × 10 mm was embedded in an epoxy matrix and polished until a smooth surface was obtained. An initial rough polishing removed the top layer of the specimen, so that the bulk material was exposed. The surface was then fine-polished for high smoothness.

Three different steps were followed during the indentation measurement. First, the specimen was loaded at a rate of 0.5 mN/s up to the maximum load of 10 mN. The specimen was then held at the maximum load for 30 s. Finally, the specimen was unloaded at a rate of 0.5 mN/s, and held at 0 mN for 5 s. The resulting indent in the specimen was inspected for any irregularities. The indenter tip was made of diamond and had the shape of a Berkovich triangle.

Three properties from the indentation measurements are report here. The first is the indentation modulus, E_{it}, which is determined from the slope of the tangent of the unloading curve. The second is the indentation hardness, H_{it}, which is the projected area of contact between the indenter and the sample. H_{it} is a measure of the permanent resistance to deformation. The third, which is also a measure of the hardness, is the Vickers hardness, *HV**, which can be calculated from the H_{it}:

$$\mathrm{HV^*} = 0.0924 \cdot \mathrm{H_{it}} \tag{3.4}$$

Average values of at least five parallel measurements are reported. (For the anhydride-cured system, up to 27 parallel measurements were performed.) The ambient temperature during the testing was 22 ± 1°C.

It is referred to the user manual for further explanation of the test method and the measured properties [16]. A typical load-displacement curve is shown in Appendix C.

4 Results

4.1 Density measurements and crosslink density

The density of the amine-cured epoxy polymer was 1159 kg/m^3, see Table 4.1. The addition of silica gradually increased the density to 1314 kg/m^3 for the composite containing 27.5 wt%. The results were very reproducible, with a low standard deviation, thus indicating a uniform density for the produced composite plates. The volume fraction of silica, here given as vol% in Table 4.1, could then be calculated. Equations for calculation of volume fraction of silica and composite density are given in Section 3.2.

The density of the anhydride-cured epoxy polymer was 1201 kg/m^3, gradually increasing to 1312 kg/m^3 for the composite containing 19.8 wt% silica. The graphs in Figure 4.1 show that the density increase was relatively similar for the two systems.

The measured density of the composites was very similar to the calculated density. This indicates that the silica density of 2100 kg/m^3, which is stated by the manufacturer, can be assumed to be correct. Nevertheless, the calculated density is consistently slightly higher than the measured density, which may indicate that the silica density is slightly lower than the stated value. The difference is, however, negligible. (The measured densities of the neat epoxy polymers were employed in the theoretical calculations.)

The molecular weight between crosslinks, M_c, was higher for the anhydride-cured epoxy compared to the amine-cured epoxy, see Table 4.2. Thus, the crosslink density was higher for the amine-cured epoxy. This is as expected, since the amine hardener has a higher functionality. It can also be seen that a higher glass transition temperature, T_g, generally indicates a higher crosslink density. (E_r for the composites will be affected by the presence of the silica particles, and not only by the epoxy polymer network. Hence, M_c for the composites was not calculated.)

Table 4.1 *Measured densities, ρ, and calculated densities, $\rho_{calculated}$, and silica content, in wt % and vol%, for the epoxy polymers and the composites.*

Amine-cured system				Anhydride-cured system			
wt% silica	ρ (kg/m^3)	$\rho_{calculated}$ (kg/m^3)	vol% silica	wt% silica	ρ (kg/m^3)	$\rho_{calculated}$ (kg/m^3)	vol% silica
0.0	1159 ± 1	n/a	0.0	0.0	1201 ± 2	n/a	0.0
6.6	1191 ± 3	1194	3.7	4.4	1222 ± 1	1224	2.5
13.4	1228 ± 5	1233	7.8	9.1	1244 ± 1	1250	5.4
20.3	1270 ± 1	1275	12.3	14.2	1275 ± 2	1279	8.6
27.5	1314 ± 1	1322	17.2	19.8	1304 ± 1	1312	12.3

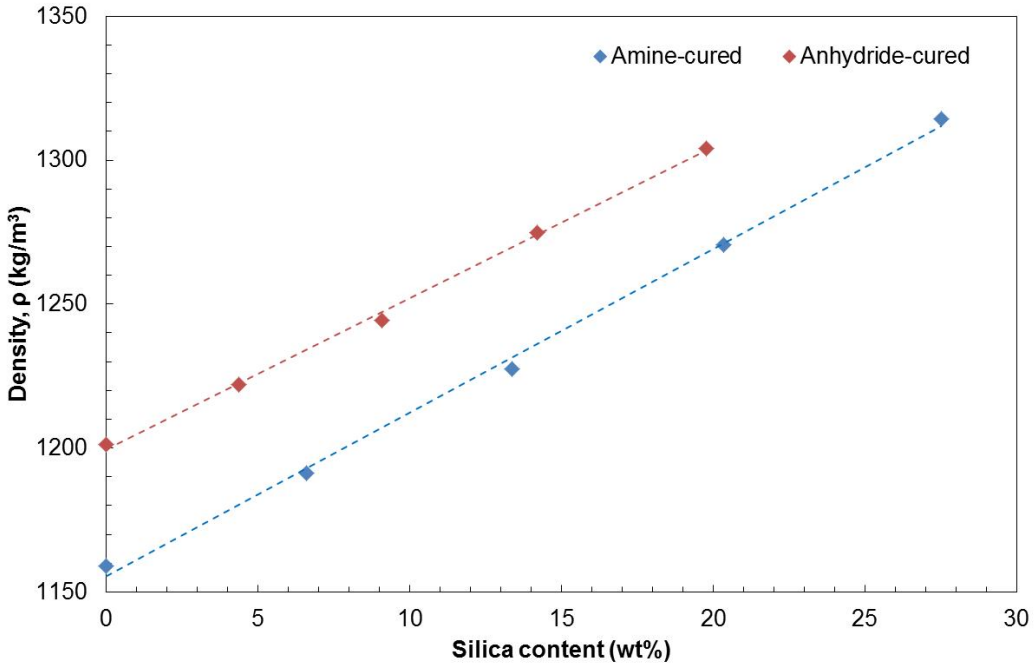


Figure 4.1 *Density, ρ, versus silica content, in wt%, for the amine-cured and the anhydride-cured system.*

Table 4.2 *Glass transition temperature, T_g, density, ρ, tensile storage modulus in the rubber plateau region, E_r, and molecular weight between crosslinks, M_c, for the neat epoxy polymers.*

Epoxy polymer	T_g (°C)	ρ (kg/m^3)	E_r (MPa)	M_c (g/mol)
Amine-cured	186 ± 2	1159 ± 1	39.7 ± 1.4	303
Anhydride-cured	155 ± 0	1201 ± 2	32.2 ± 3.4	341

4.2 Dynamic mechanical analysis

The DMA showed a linear increase in the storage modulus, *E*', with increasing amounts silica in the amine-cured composite, see Table 4.3 and Figure 4.4. The value increased from 2610 MPa for the neat epoxy polymer, to 3700 MPa for the composite containing 17.2 vol% nanosilica. The T_g remained almost constant at around 186°C. However, a small decrease in T_g was observed for the composite containing the highest amount of silica. Examples of DMA-curves are given in Figure 4.2.

A linear increase in the storage modulus was also observed for the anhydride-cured composite, as shown in Table 4.3 and Figure 4.5, where the value increased from 2990 MPa for the neat epoxy polymer, to 3970 MPa for the composite containing 12.3 vol% silica. However, for this system there was also a linear decrease in the T_g with increasing silica content. The decrease in T_g was from 155°C to 143°C. Examples of DMA-curves are given in Figure 4.3.

The decrease in T_g for the anhydride-cured composite indicates a lower crosslink density, which may contribute to some of the increase in *E*'. This effect was further investigated, and the results are reported in Section 4.3. Also, the results from an under-cured composite with presumably lower crosslink density are given in Appendix D.

Table 4.3 Storage modulus, E', and glass transition temperature, T_g, of the epoxy polymers and the composites.

Amine-cured system			Anhydride-cured system		
vol% silica	**E' (MPa)**	**T_g (°C)**	**vol% silica**	**E' (MPa)**	**T_g (°C)**
0.0	2610 ± 30	186 ± 2	0.0	2990 ± 40	155 ± 0
3.7	2870 ± 50	186 ± 2	2.5	3240 ± 40	154 ± 1
7.8	3090 ± 30	187 ± 1	5.4	3410 ± 80	151 ± 0
12.3	3370 ± 60	185 ± 1	8.6	3750 ± 60	146 ± 1
17.2	3700 ± 50	182 ± 1	12.3	3970 ± 100	143 ± 1

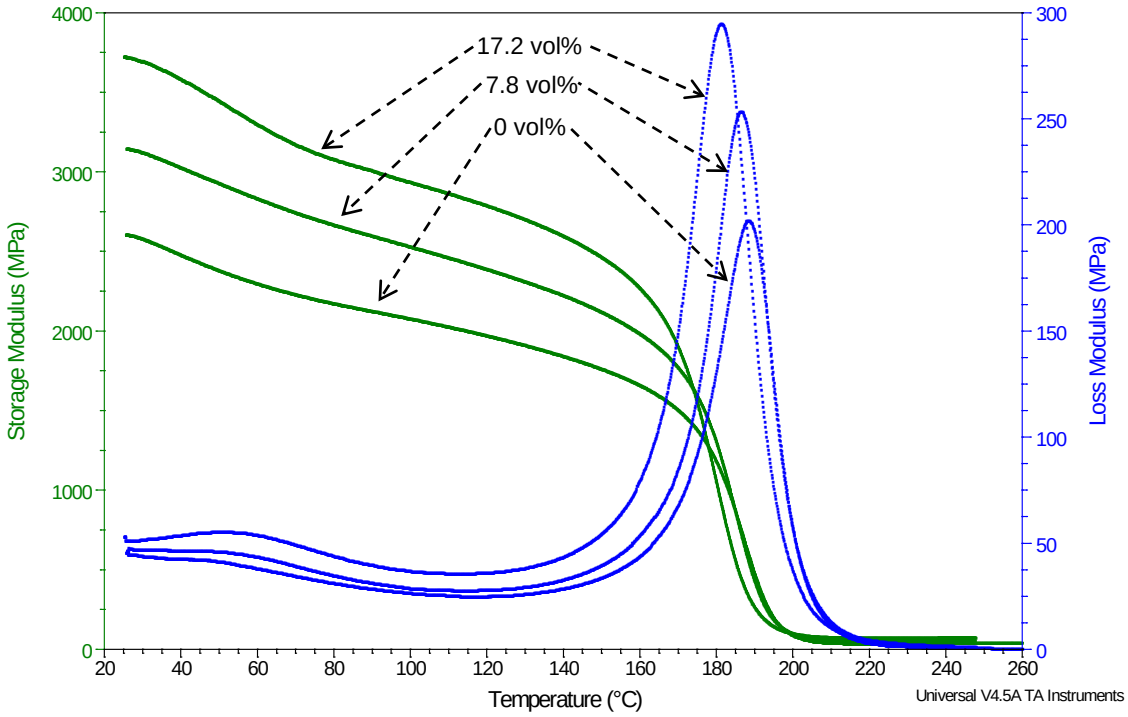


Figure 4.2 Typical DMA-curves for the amine-cured system.

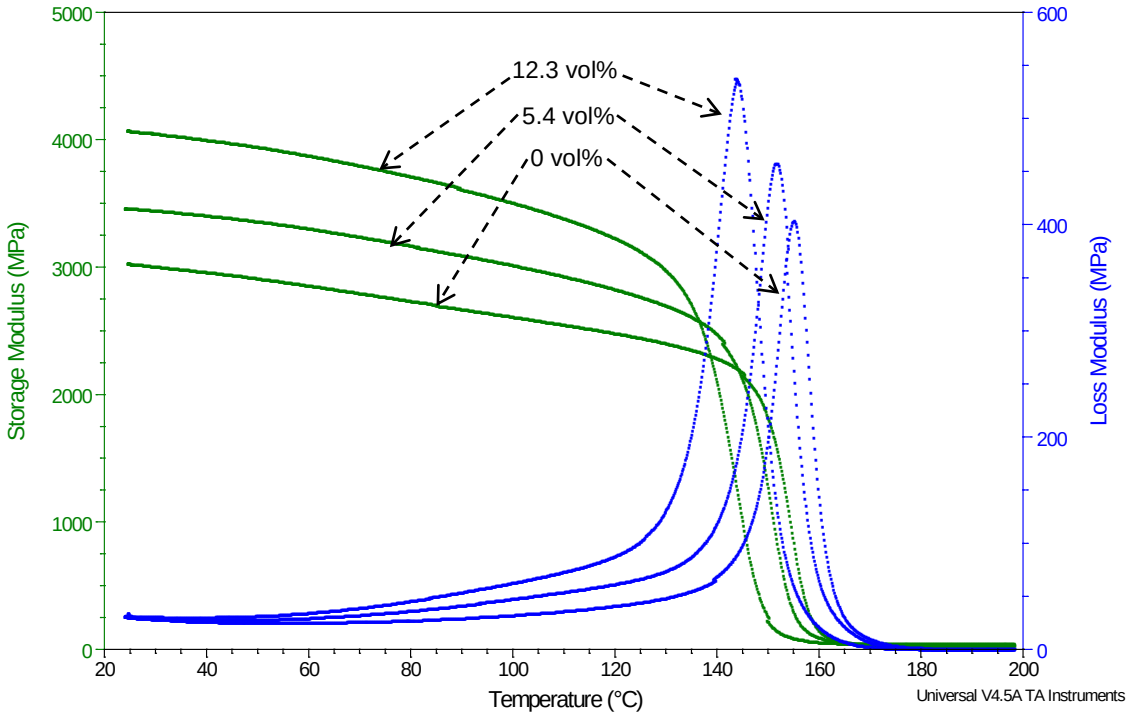


Figure 4.3 Typical DMA-curves for the anhydride-cured system.

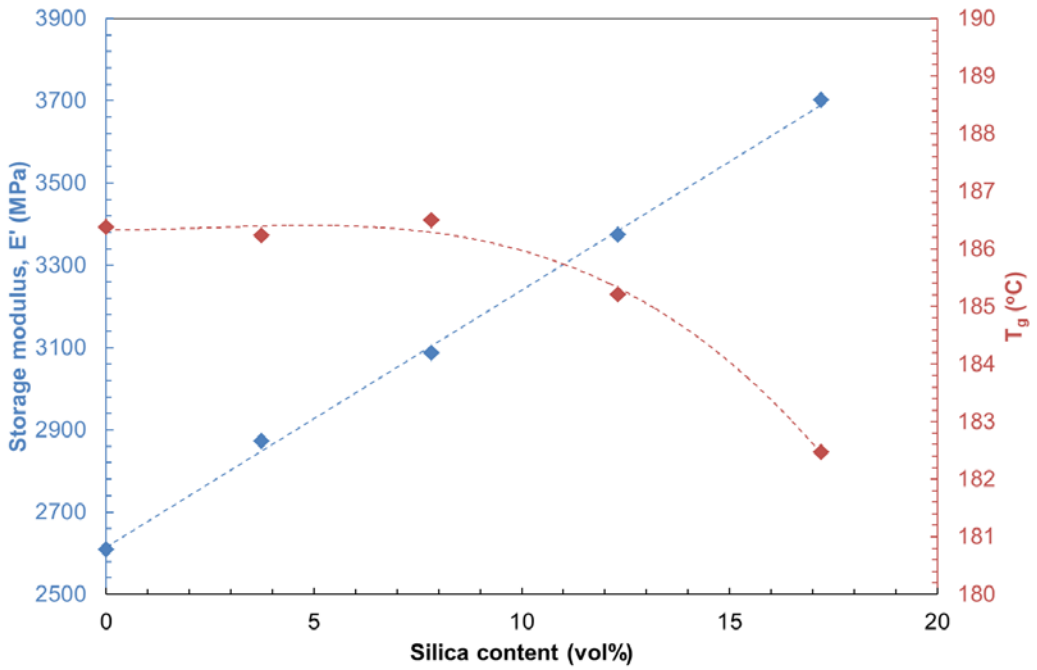


Figure 4.4 Storage modulus, E', and glass transition temperature, T_g, of the amine-cured system.

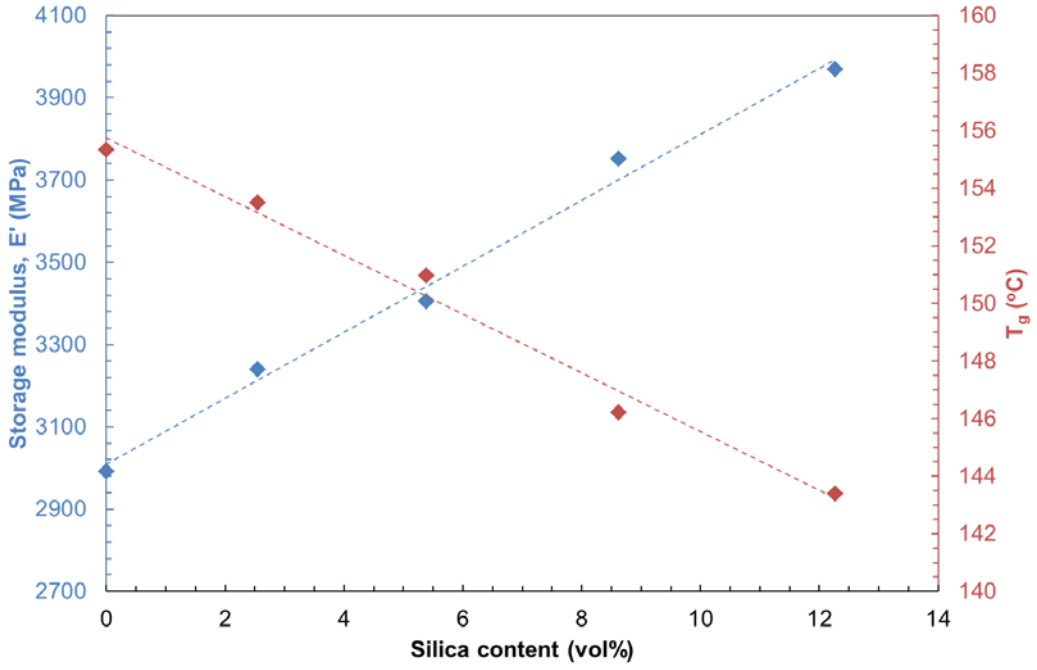


Figure 4.5 Storage modulus, E', and glass transition temperature, T_g, of the anhydride-cured system.

4.3 Effect of resin/hardener ratio

The resin/hardener mixing ratio of the neat anhydride-cured epoxy polymer (Araldite LY 556/Aradur 917/Accelerator DY 070), was varied to investigate the effect on the elastic modulus and the T_g. The active hydrogen equivalent weight (AHEW) of Aradur 917 is 166.2 g/eq, and the recommended mixing ratio given by Huntsman is 100:90:1. On this basis, an epoxy equivalent weight (EEW) of 184.7 g/eq was calculated for LY556, and this was used as a starting point for the formulations given in Table 4.4. The results obtained from DMA and density measurements of three replicate specimens are also given in the table.

The variation of the storage modulus, *E*', and the glass transition temperature, T_g, as a function of the amount of hardener, given in per hundred resin (phr), is shown in Figure 4.6. (The two 'extreme' formulations marked 'NN' in Table 4.4, have not been included in the figure.) As can be seen from the figure, there is a linear increase of E' with increasing amount of hardener, while T_g is reduced when non-stoichiometric amounts of hardener are used. The T_g has a maximum which is slightly below the assumed stoichiometric hardener content of 90 phr, at around 86 phr. The value of this maximum may indicate that the stoichiometric amount of hardener is actually slightly lower than 90 phr. Also, an excess of hardener results in broadening of the peak of the loss modulus, *E*'', see the DMA-curves in Figure 4.7.

If an excess of hardener is used, the result will be a reduction of the T_g and an increase of *E*'. For example, in the case that the T_g is reduced by 10^{o}C, this will coincide with an increase of *E*' by around 150 MPa.

It has also been shown in the literature that the properties of epoxy polymers could be significantly affected by the curing cycle and/or the resin/hardener ratio, see e.g. [17-19] and the references therein, and that a change in the value of the T_g may be correlated to a change in the elastic modulus. A lower T_g and higher modulus is often explained in terms of 'free volume'. A lower degree of curing can result in lower crosslink density, i.e. higher molecular weight between crosslinks, which could result in higher packing of the molecular chains. In the results in Table 4.4, there is no obvious correlation between the molecular weight between crosslinks, M_c, and the amount of hardener. There might be a small tendency of increasing M_c, that is, lower cross-link density, with increasing amount of hardener. However, the scatter in the data is too large to make any certain assumptions. There is, however, a tendency of decreasing density with increasing amount of hardener.

Table 4.4 *Formulations used for preparation of the neat anhydride-cured epoxy polymers with varying resin/hardener ratio. The results of the measured storage modulus, E', glass transition temperature, T_g, tensile storage modulus in the rubber plateau region, E_r, density, ρ, and molecular weight between crosslinks, M_c, are given.*[2]

EEW	Parts by weight of components			E' (MPa)	T_g (°C)	E_r (MPa)	ρ (kg/m^3)	M_c (g/mol)
	LY556	Aradur917	DY070					
NN	100	112.5	1.22	3350	124	27.5	1210	368
164.7	100	100.9	1.12	3240	142	29.7	1203	354
174.7	100	95.1	1.06	3200	150	33.5	1201	336
182.5	100	91.1	1.01	3070	153	29.7	1204	354
184.7	100	90.0	1.00	2990	155	32.2	1201	341
187.0	100	88.9	0.99	3020	155	32.8	1201	339
194.7	100	85.4	0.95	2990	156	35.0	1198	329
204.7	100	81.2	0.90	2920	153	30.8	1198	347
NN	100	67.5	0.79	2920	146	31.2	1198	345

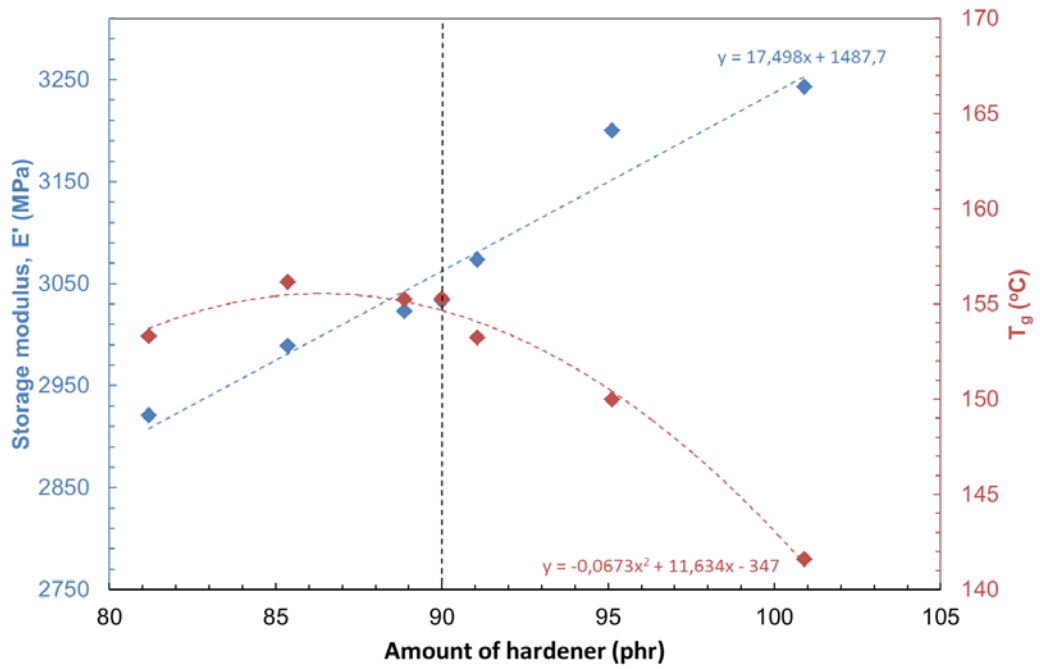


Figure 4.6 *Storage modulus, E', and glass transition temperature, T_g, as a function of the amount of hardener in the anhydride-cured system.* [3]

[2] For some formulations, only two replicate specimens were tested. Standard deviations have therefore been omitted from the table.

[3] The results for the two 'extreme' formulations marked 'NN' in Table 4.4 have not been included in the figure.

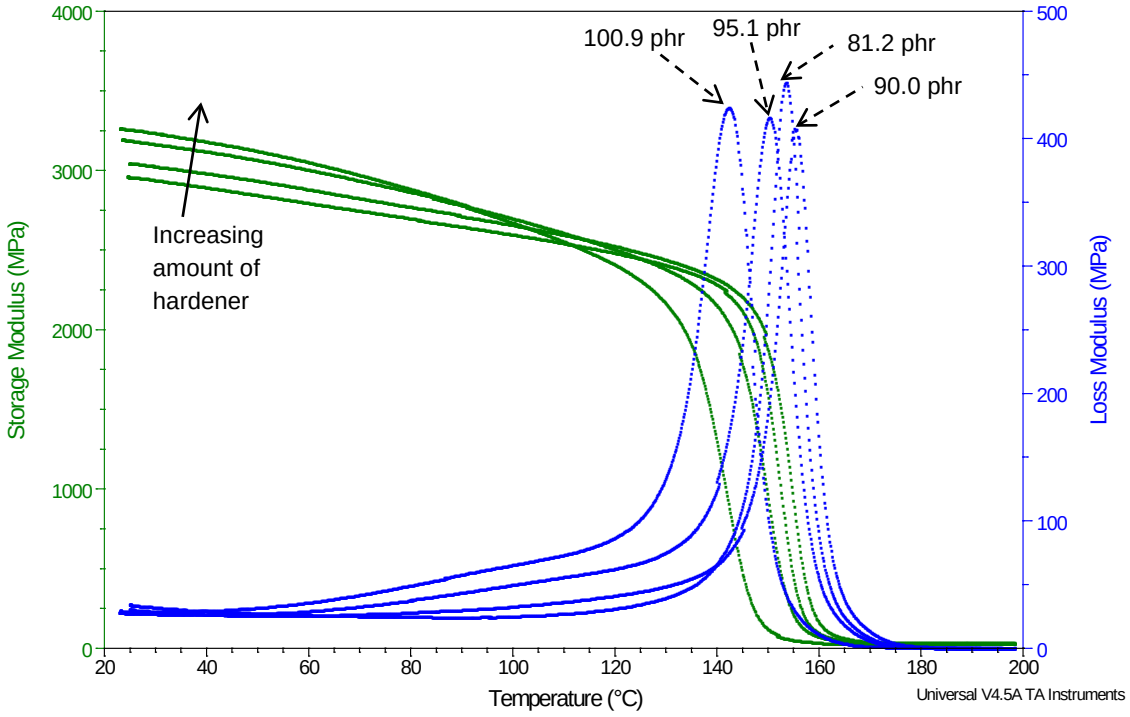


Figure 4.7 DMA-curves for the neat anhydride-cured epoxy polymer with varying resin/hardener ratio. Hardener contents of 81.2, 90.0, 95.1, and 100.9 phr are plotted.

4.4 Tensile testing

The tensile testing showed a linear increase in the elastic modulus, E_t, with increasing silica content in the amine-cured composite, see Table 4.5. The value increased from 2610 MPa for the neat epoxy polymer, to 3680 MPa for the composite containing 17.2 vol% silica. The tensile strength, σ_m, remained relatively constant, although the results show that the strength may have been slightly improved at the highest silica contents. The tensile strain at break, ε_b, on the other hand, is significantly reduced by the addition of the silica nanoparticles. The yield strength, σ_y, could not be determined since the specimens broke without yielding. Typical stress-strain curves of the amine-cured system are shown in Figure 4.8. All test results are given in Appendix E. Also, the tensile properties of the neat epoxy polymer are very similar to those obtained earlier using a different experimental test set-up with a different specimen geometry [10].

A linear increase in the elastic modulus, E_t, with increasing silica content was also observed for the anhydride-cured composite, see Table 4.5. The value increased from 3030 MPa for the neat epoxy polymer, to 4150 MPa for the composite containing 12.3 vol% silica. Thus, the elastic modulus was higher for the anhydride-cured system. Also, the tensile strength, σ_m, was gradually improved with addition of silica to the polymer, whereas a minor reduction in the strain at break,

ε_b, was observed. Typical stress-strain curves of the anhydride-cured system are shown in Figure 4.9.

On the whole, the anhydride-cured system showed a more positive effect of the addition of silica, particularly with respect to the increased elastic modulus, compared to the amine-cured system. This is illustrated by the relative increase in the elastic modulus of the two systems, as shown by the $E_t/E_{t,0}$ graphs in Figure 4.10, where $E_{t,0}$ is the elastic modulus of the neat epoxy polymer.

Table 4.5 Tensile test results for the epoxy polymers and the composites.

Amine-cured system				Anhydride-cured system			
vol% silica	E_t (MPa)	σ_m (MPa)	ε_b (%)	vol% silica	E_t (MPa)	σ_m (MPa)	ε_b (%)
0.0	2610 ± 30	69 ± 4	4.8 ± 0.6	0.0	3030 ± 70	85 ± 1	5.8 ± 0.5
3.7	2800 ± 50	68 ± 2	4.1 ± 0.2	2.5	3280 ± 50	89 ± 1	5.7 ± 0.4
7.8	3040 ± 70	66 ± 2	3.5 ± 0.3	5.4	3530 ± 100	89 ± 2	4.9 ± 0.7
12.3	3310 ± 80	69 ± 1	3.3 ± 0.2	8.6	3800 ± 90	88 ± 5	4.3 ± 1.1
17.2	3680 ± 110	67 ± 4	2.7 ± 0.3	12.3	4150 ± 80	93 ± 0	5.1 ± 0.3

Some of the tensile test results for the anhydride-cured system were considered erroneous and have not been included when the average values in Table 4.5 were calculated. This had no effect on the E_t-values. Examples of data that were considered erroneous are e.g. specimens that gave unproportional low strain at break values. Although it has been assumed previously in this report that no voids are present in the composites, a likely explanation for this behaviour is the presence of defects or voids, or possibly surface defects, in the specimens.

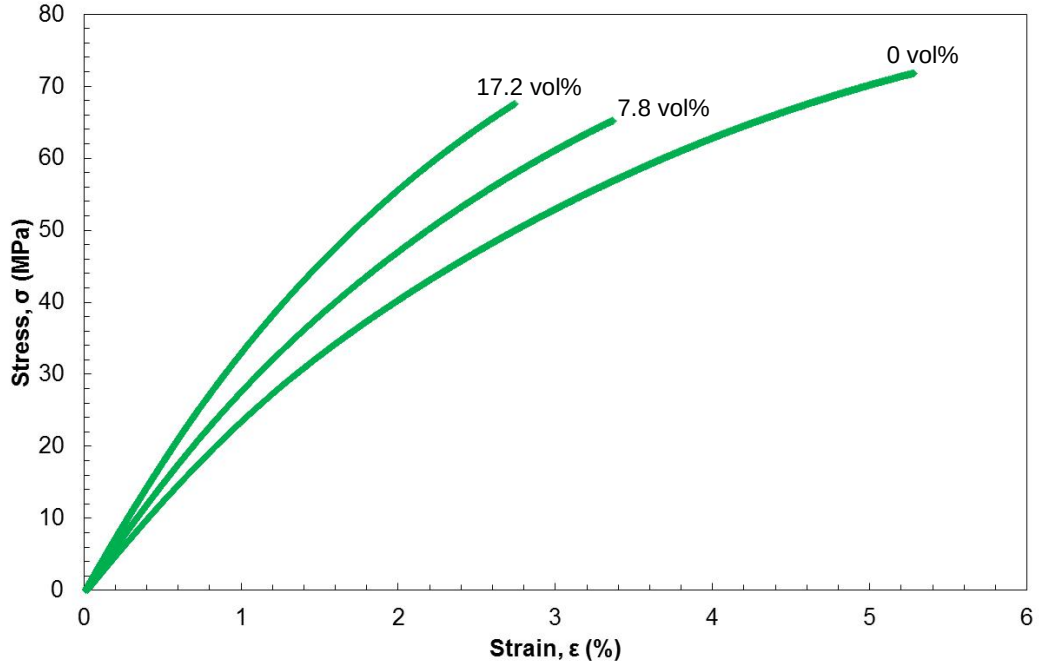


Figure 4.8 Typical stress-strain curves for the amine-cured system. The silica content is indicated.

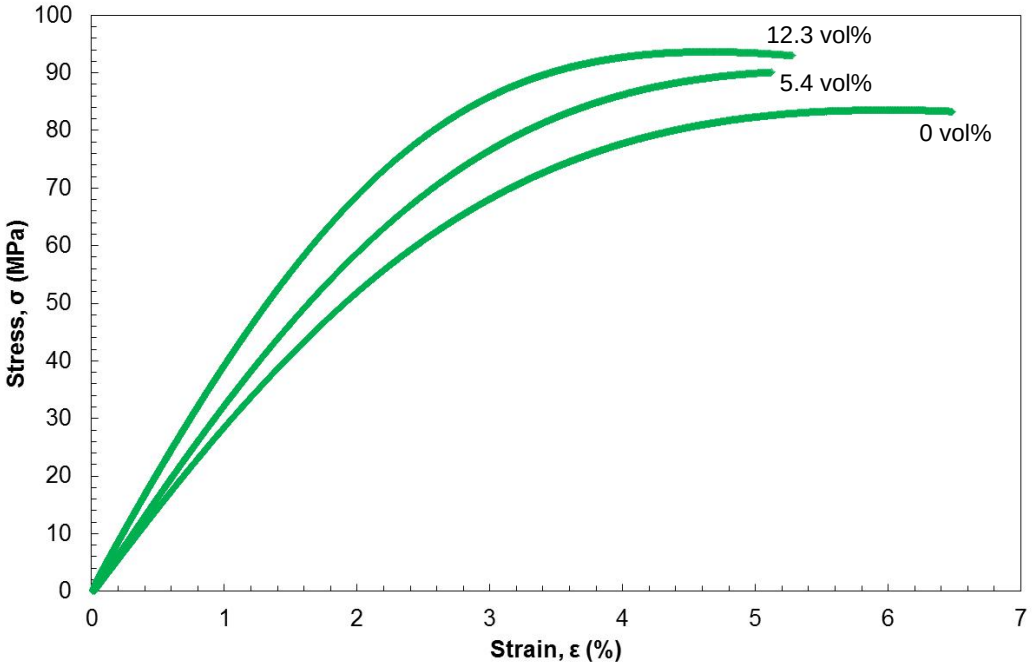


Figure 4.9 Typical stress-strain curves for the anhydride-cured system. The silica content is indicated.

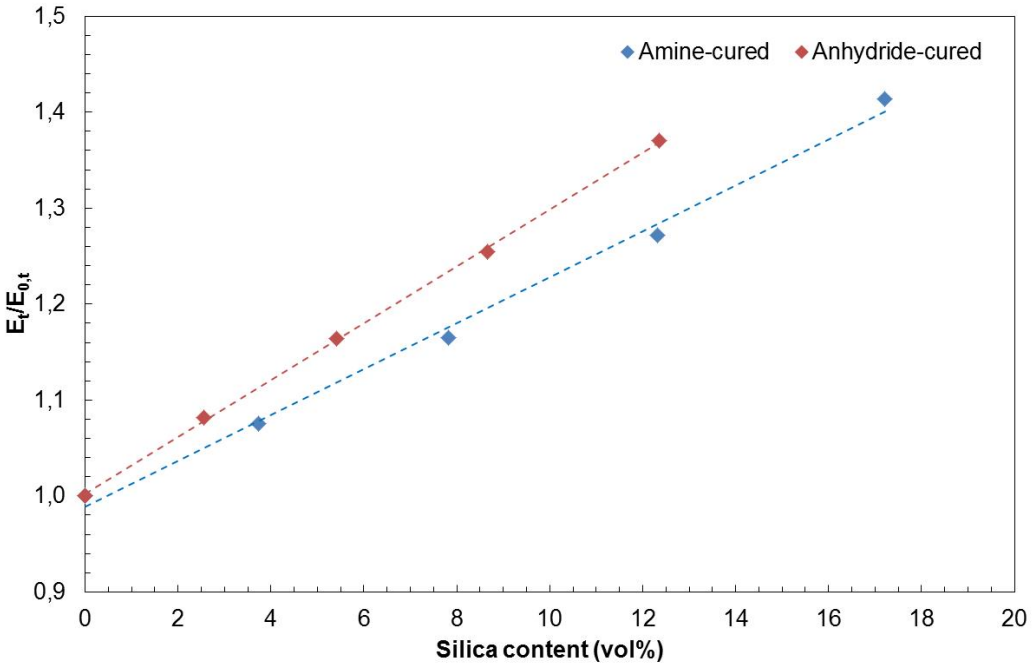


Figure 4.10 $E_t/E_{t,0}$ versus silica content, in vol%, for the amine-cured and the anhydride-cured system.

4.5 Indentation measurements

The indentation measurements of the amine-cured system showed an increase in the indentation modulus, E_{it}, with increasing silica content in the composite, see Table 4.6. The increase in E_{it} was close to linear, but there was a small tendency of an incremental increase with increasing silica content. The value of E_{it} was 2820 MPa for the neat epoxy polymer, increasing to 3940 MPa for the composite containing 17.2 vol% silica.

For the anhydride-cured system, the development of E_{it} with increasing silica content is different, as illustrated in Figure 4.11. Initially there is a plateau value at around 3500 MPa, and it is noteworthy that the value for the neat epoxy polymer and the composite containing 2.5 vol% is practically identical. Thereafter, E_{it} dramatically increases to a maximum value of 4420 MPa for the composite containing 12.3 vol% silica. The reason for this behaviour is unknown.

There were some variations in the measured hardness values for the two systems, see Table 4.6. The hardness at low silica contents did not differ significantly from the neat epoxy polymers. However, the hardness was generally increased at the highest silica contents. All the available test data are given in Appendix F.

Table 4.6 Results from indentation measurements of the epoxy polymers and the composites.

Amine-cured system				Anhydride-cured system			
vol% silica	E_{it} (MPa)	H_{it} (MPa)	HV*	vol% silica	E_{it} (MPa)	H_{it} (MPa)	HV*
0.0	2820 ± 30	226 ± 8	20.8 ± 0.8	0.0	3500 ± 100	247 ± 17	22.8 ± 1.5
3.7	3010 ± 20	222 ± 8	20.6 ± 0.8	2.5	3530 ± 20	250 ± 10	23.1 ± 0.9
7.8	3260 ± 40	241 ± 8	22.3 ± 0.7	5.4	3730 ± 20	260 ± 5	24.0 ± 0.4
12.3	3560 ± 10	229 ± 9	21.1 ± 0.8	8.6	4140 ± 40	279 ± 16	25.8 ± 1.4
17.2	3940 ± 20	263 ± 13	24.3 ± 1.2	12.3	4420 ± 60	290 ± 26	26.8 ± 2.4

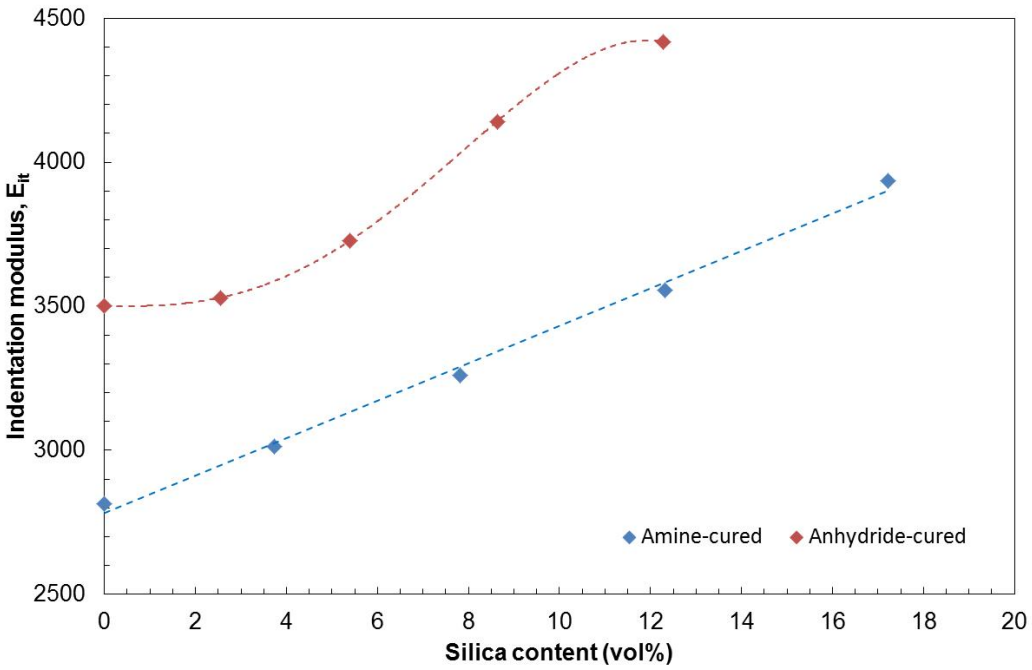


Figure 4.11 Indentation modulus, E_{it}, versus silica content, in vol%, for the amine-cured and the anhydride-cured system.

4.6 Comparison of elastic modulus

The elastic modulus of the silica/epoxy composites was obtained by three different experimental techniques, namely tensile testing, DMA and indentation measurements. The results are summarised in Table 4.7. For the amine-cured system, there was very good agreement between E_t, measured by tensile testing, and E', measured by DMA. Practically identical values were obtained, when considering the experimental scatter in the measurements. However, it should be noted that the tensile testing was conducted at room temperature, i.e. 22 ± 1°C, while *E*' from the DMA analysis was determined at 30°C. This has little effect on the anhydride-cured system. However, due to the slope of the *E*'-curve, the value of *E*' may be up to 50 MPa lower at 30°C (compared to 22°C), for the amine-cured system. This effect should be taken into account when evaluating the results. The E_{it} value, measured by indentation, conducted at 22°C, gave a value that was consistently around 200 MPa higher than the E_t and *E*' values.

For the anhydride-cured system, there was in general good agreement between the modulus values obtained from the tensile testing and the DMA measurements. There were, however, some minor differences in the values for two of the composite specimens. The indentation measurements, on the other hand, gave modulus values that were very different from the two other techniques. For example, for the neat epoxy polymer, a value of 3500 MPa was obtained in the indentation measurements, while a value around 3000 MPa was obtained in the tensile and DMA measurements. The reason for this high value in the indentation measurements is unknown.

Table 4.7 Comparison of the elastic modulus obtained by tensile testing (elastic modulus, E_t), DMA (storage modulus, E'), and indentation measurements (indentation modulus, E_{it}).

Amine-cured system				Anhydride-cured system			
vol% silica	E_t (MPa)	E' (MPa)	E_{it} (MPa)	vol% silica	E_t (MPa)	E' (MPa)	E_{it} (MPa)
0.0	2610 ± 30	2610 ± 30	2820 ± 30	0.0	3030 ± 70	2990 ± 40	3500 ± 100
3.7	2800 ± 50	2870 ± 50	3010 ± 20	2.5	3280 ± 50	3240 ± 40	3530 ± 20
7.8	3040 ± 70	3090 ± 30	3260 ± 40	5.4	3530 ± 100	3410 ± 80	3730 ± 20
12.3	3310 ± 80	3370 ± 60	3560 ± 10	8.6	3800 ± 90	3750 ± 60	4140 ± 40
17.2	3680 ± 110	3700 ± 50	3940 ± 20	12.3	4150 ± 80	3970 ± 100	4420 ± 60

For the anhydride-cured composites, some of the increase in the elastic modulus could be attributed to differences in the resin/hardener ratio, and not merely as an effect of the silica reinforcement. The DMA measurements in Section 0, showed that there was a decrease in the glass transition temperature, T_g, with increasing amount of silica. In Section 4.3, DMA measurements on the neat anhydride-cured polymer showed that the storage modulus was increased when the T_g was decreased, as the result of excess hardener. A T_g-reduction of 10°C gave an increase of E' of around 150 MPa. Hence, it is likely that the T_g-reduction for the composites indicate that there is a change in the polymer network structure. It follows that part of the increase of the elastic modulus of the composites is due to the altered polymer structure, and not directly related to the silica reinforcement. This effect should be considered when evaluating the elastic modulus of the anhydride-cured system. Nevertheless, the main part of the increase of the elastic modulus will be a result of the silica reinforcement.

A similar effect is shown in Appendix D, for the composite that was under-cured. For the same silica content, the under-cured composite have a lower T_g, higher E' and E_t, and it is expected to have a lower crosslink density, compared to the fully cured composite.

5 Conclusions

The material properties of two different silica/epoxy nanocomposite systems were determined by tensile testing, DMA and indentation measurements. The elastic modulus of both epoxy polymers was increased by the addition of silica nanoparticles. As a whole, the relative increase in material properties was higher for the anhydride-cured system, compared to the amine-cured system.

There was, in general, relatively good agreement between the values of the elastic modulus obtained by tensile testing and DMA. Thus, only one of the techniques, e.g. DMA, could be used to get a reliable estimate of the elastic modulus of an unmodified or modified epoxy polymer.

The indentation measurements, on the other hand, gave values that were either slightly higher, or very different, from the values obtained by the two other techniques. Indentation measurements are suitable for measuring the elastic modulus of small specimens, much smaller than what is

required for tensile testing or DMA, or small areas in the specimens. However, when doing so on epoxy polymers, the result should be critically examined before drawing any conclusions.

Part of the increase in the elastic modulus of the anhydride-cured composites is probably related to an altered polymer network structure, as indicated by the change in the glass transition temperature. This factor should be considered when evaluating the elastic modulus of these composites.

Acknowledgements

Stephan Sprenger at Evonik Hanse is acknowledged for supplying the nanomaterial, and for giving valuable advice with respect to the formulation of the nanocomposites. The authors also wish to thank Tom Thorvaldsen and Finn Knut Hansen for reading the report and for suggesting improvements.

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Appendix A Material data sheets

A.1 Araldite LY 556 / Hardener XB 3473

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Advanced Materials

Araldite® LY 556* / Hardener XB 3473*

HOT CURING EPOXY SYSTEM

Araldite® LY 556 is an epoxy resin
Hardener XB 3473 is a formulated amine hardener

APPLICATIONS	• Industrial composites • Structural composites		
PROPERTIES	Laminating system		
PROCESSING	• Filament Winding • Resin Transfer Moulding (RTM) • Pressure Moulding • Pultrusion		
KEY DATA	**Araldite® LY 556**		
	Aspect (visual)	clear, pale yellow liquid	
	Colour (Gardner, ISO 4630)	≤ 2	
	Viscosity at 25 °C (ISO 12058-1)	10000 - 12000	[mPa s]
	Density at 25 °C (ISO 1675)	1.15 - 1.2	[g/cm^3]
	Flash point (ISO 2719)	> 200	[°C]
	Storage temperature (see expiry date on original container)	2 - 40	[°C]
	Hardener XB 3473		
	Aspect (visual)	clear yellow to brown liquid	
	Viscosity at 25 °C (ISO 12058-1)	95 - 145	[mPa s]
	Density at 25 °C (ISO 1675)	0.99 - 1.02	[g/cm^3]
	Flash point (ISO 2719)	121	[°C]
	Storage temperature (see expiry date on original container)	2 - 40	[°C]
STORAGE	Provided that Araldite® LY 556 and Hardener XB 3473 are stored in a dry place in their original, properly closed containers at the above mentioned storage temperatures they will have the shelf lives indicated on the labels. Partly emptied containers should be closed immediately after use.		

* *In addition to the brand name product denomination may show different appendices , which allows us to differentiate between our production sites: e.g , BD = Germany, US = United States, IN = India, CI = China, etc.. These appendices are in use on packaging, transport and invoicing documents. Generally the same specifications apply for all versions. Please address any additional need for clarification to the appropriate Huntsman contact.*

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PROCESSING DATA

MIX RATIO

Components	*Parts by weight*	*Parts by volume*
Araldite® LY 556	100	100
Hardener XB 3473	23	27

We recommend that the components are weighed with an accurate balance to prevent mixing inaccuracies which can affect the properties of the matrix system. The components should be mixed thoroughly to ensure homogeneity. It is important that the side and the bottom of the vessel are incorporated into the mixing process.

When processing large quantities of mixture the pot life will decrease due to exothermic reaction. It is advisable to divide large mixes into several smaller containers.

INITIAL MIX VISCOSITY (CONE/PLATE VISCOSIMETER)

	[°C]	*[mPa s]*
LY 556/XB 3473	at 25	5200 - 6000
	at 40	700 - 900

POT LIFE (TECAM, 23°C, 65 % RH)

	[g]	*[h]*
LY 556/XB 3473	100	32 - 37

GEL TIME (HOT PLATE)

	[°C]	*[min]*
LY 556/XB 3473	at 120	68 - 78
	at 140	35 - 43
	at 160	18 - 23
	at 180	9 - 13

The values shown are for small amounts of pure resin/hardener mix. In composite structures the gel time can differ significantly from the given values depending on the fibre content and the laminate thickness.

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PROPERTIES OF THE CURED, NEAT FORMULATION			
GLASS TRANSITION TEMPERATURE	*Cure:*	T_g	*LY 556* *XB 3473*
(IEC 1006, DSC, 10 K/MIN)	4 h 80°C + 4 h 160°C	[°C]	162 - 168
	2 h 120°C + 4 h 180°C	[°C]	185 - 194
	2 h 120°C + 2 h 160°C + 2h 200°C	[°C]	185 - 193
	2 h 120°C + 2 h 160°C + 2h 200°C + 4h 220°C	[°C]	187 - 195
GLASS TRANSITION TEMPERATURE	*Cure:*	T_g	*LY 556* *XB 3473*
(ISO 6721, DMA, 2 K/MIN)	2 h 120°C + 2 h 140°C + 2h 180°C	[°C]	175 - 185
FLEXURAL TEST (ISO 178)	*Cure:* 2 h 120°C + 2 h 140°C + 2 h 180°C		
	Flexural strength	[MPa]	110 - 120
	Elongation at flexural strength	[%]	5,5 - 6,5
	Ultimate strength	[MPa]	110 - 120
	Ultimate elongation	[%]	5,5 - 6,5
	Flexural modulus	[MPa]	2700 - 2900
FRACTURE PROPERTIES BEND NOTCH TEST (PM 258-0/90)	*Cure:* 2 h 120°C + 2 h 140°C + 2 h 180°C		
	Fracture toughness K_{1C}	[MPa$\sqrt{m}$]	0,70 - 0,85
	Fracture energy G_{1C}	[J/m^2]	190 - 220

PROPERTIES OF THE CURED, REINFORCED FORMULATION			
	Short beam: Laminate comprising 12 layers unidirectional E-glass fabric (425 g/m^2) Laminate thickness t = 3.1 - 3.3 mm Fibre volume content: 63 - 65 %		
INTERLAMINAR SHEAR STRENGTH (ASTM D 2344)	*Cure:* 2 h 120 °C + 2 h 140 °C + 2 h 180 °C		
	Shear strength	[MPa]	62 - 66
FLEXURAL TEST (ISO 178)	*Cure:* 2 h 120 °C + 2 h 140 °C + 2 h 180 °C		
	Flexural strength	[MPa]	1050 - 1250
	Ultimate elongation	[%]	2.4 - 2.8
	Flexural modulus	[MPa]	40000 - 44000

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HANDLING PRECAUTIONS

Personal hygiene	
Safety precautions at workplace	
protective clothing	yes
gloves	essential
arm protectors	recommended when skin contact likely
goggles/safety glasses	yes
Skin protection	
before starting work	Apply barrier cream to exposed skin
after washing	Apply barrier or nourishing cream
Cleansing of contaminated skin	
	Dab off with absorbent paper, wash with warm water and alkali-free soap, then dry with disposable towels. Do not use solvents
Disposal of spillage	
	Soak up with sawdust or cotton waste and deposit in plastic-lined bin
Ventilation	
of workshop	Renew air 3 to 5 times an hour
of workplaces	Exhaust fans. Operatives should avoid inhaling vapours

FIRST AID

Contamination of the *eyes* by resin, hardener or mix should be treated immediately by flushing with clean, running water for 10 to 15 minutes. A doctor should then be consulted.

Material smeared or splashed on the *skin* should be dabbed off, and the contaminated area then washed and treated with a cleansing cream (see above). A doctor should be consulted in the event of severe irritation or burns. Contaminated clothing should be changed immediately.

Anyone taken ill after *inhaling* vapours should be moved out of doors immediately.

In all cases of doubt call for medical assistance.

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A.2 Araldite LY 556 / Aradur 917 / Accelerator DY 070

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Advanced Materials

Araldite® LY 556* / Aradur® 917* / Accelerator DY 070*

HOT CURING EPOXY MATRIX SYSTEM

Araldite® LY 556 is an epoxy resin
Aradur® 917 is an anhydride hardener
Accelerator DY 070 is an imidazole accelerator

APPLICATIONS	High performance composite parts		
PROPERTIES	Anhydride-cured, low-viscosity standard matrix system with extremely long pot life. The reactivity of the system is adjustable by variation of the accelerater content.The system is easy to process, has good fibre impregnation properties and exhibits excellent mechanical, dynamic and thermal properties. It has an excellent chemical resistance especially to acids at temperatures up to 80 °C. This epoxy system fulfills MIL specifications R 9300.		
PROCESSING	Filament Winding Pultrusion Pressure Moulding		
KEY DATA	**Araldite® LY 556**		
	Aspect (visual)	clear, pale yellow liquid	
	Colour (Gardner, ISO 4630)	≤ 2	
	Epoxy content (ISO 3000)	5.30 - 5.45	[eq/kg]
	Viscosity at 25 °C (ISO 12058-1)	10000 - 12000	[mPa s]
	Density at 25 °C (ISO 1675)	1.15 - 1.20	[g/cm^3]
	Flash point (ISO 2719)	> 200	[°C]
	Aradur® 917		
	Aspect (visual)	clear liquid	
	Colour (Gardner, ISO 4630)	≤ 2	
	Viscosity at 25 °C (ISO 12058-1)	50 - 100	[mPa s]
	Density at 25 °C (ISO 1675)	1.20 - 1.25	[g/cm^3]
	Flash point (ISO 2719)	195	[°C]
	Accelerator DY 070		
	Aspect (visual)	clear liquid	
	Colour (Gardner, ISO 4630)	≤ 9	
	Viscosity at 25 °C (ISO 12058-1)	≤ 50	[mPa s]
	Density at 25 °C (ISO 1675)	0.95 - 1.05	[g/cm^3]
	Flash point (ISO 2719)	92	[°C]
	Storage temperature (see expiry date on original container)	2 - 40 °C	[°C]

* *In addition to the brand name product denomination may show different appendices , which allows us to differentiate between our production sites: e.g , BD = Germany, US = Unied States, IN = India,CI = China, etc.. These appendices are in use on packaging, transport and invoicing documents. Generally the same specifications apply for all versions. Please address any additional need for clarification to the appropriate Huntsman contact*

STORAGE	Provided that Araldite® LY 556, Aradur® 917 and Accelerator DY 070 are stored in a dry place in their original, properly closed containers at the above mentioned storage temperatures they will have the shelf lives indicated on the labels. Partly emptied containers should be closed immediately after use. Because Aradur® 917 is sensitive to moisture, storage containers should be ventilated with dry air only. Araldite® LY 556 which has crystallized and looks cloudy can be restored to its original state by heating to 60 - 80 °C.

PROCESSING DATA

MIX RATIO

Components	*Parts by weight*	*Parts by volume*
Araldite® LY 556	100	100
Aradur® 917	90	86
Accelerator DY 070	0.5 - 2	0.6 - 2.4

We recommend that the components are weighed with an accurate balance to prevent mixing inaccuracies which can affect the properties of the matrix system. The components should be mixed thoroughly to ensure homogeneity. It is important that the side and the bottom of the vessel are incorporated into the mixing process. When processing large quantities of mixture the pot life will decrease due to exothermic reaction. It is advisable to divide large mixes into several smaller containers.

PROCESSING RECOMMENDATIONS

To simplify the mixing process the resin can be preheated to about 30 °C to 50 °C before adding the cold hardener. Hardener and accelerator can be premixed, thus allowing the use of two component mixing/metering equipment. The mix of hardener and accelerator has a shelf life of several days.

The processing of the system at elevated temperatures of 30 °C to 40 °C shows the best results. The gelation temperature should not be higher than absolutely necessary. A high gelation temperature induces high shrinkage and generates internal stresses.

INITIAL MIX VISCOSITY (HOEPPLER, ISO 12058-1B)

[°C]	*[mPa s]*
at 25	600 - 900
at 40	200 - 300
at 60	< 75

VISCOSITY BUILD-UP (HOEPPLER, ISO 12058-1B)

Components [pbw]	*System 1*	*System 2*	*System 3*
Araldite® LY 556	100	100	100
Aradur® 917	90	90	90
Accelerator DY 070	0.5	1	2

[°C]	*[mPa s]*				
at 25	to 1500	*[h]*	10 - 12	3.5 - 4.5	1.5 - 2
	to 3000	*[h]*	33 - 37	16 - 18	6 - 7
at 40	to 1500	*[h]*	19 - 21	7 - 8	3 - 4
	to 3000	*[h]*	23 - 26	9 - 10	4 - 5
at 80	to 1500	*[min]*	95 - 105	52 - 57	32 - 35
	to 3000	*[min]*	105 - 115	60 - 65	35 - 38
at 90	to 1500	*[min]*			14 - 16
	to 3000	*[min]*			15 - 17

POT LIFE (TECAM, 65 % RH, 100 G) 10 KG METAL CONTAINER

[°C]		*System 1*	*System 2*	*System 3*
at 23	*[h]*	165 - 175	95 - 105	48 - 54
at 40	*[h]*	5 - 7	4 - 5	-

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GEL TIME (HOT PLATE)	*[°C]*		*System 1*	*System 2*	*System 3*
	at 80	*[min]*	230 - 270	140 - 160	65 - 75
	at 100	*[min]*	65 - 75	35 - 45	18 - 22
	at 120	*[min]*	21 - 25	10 - 12	5 - 7
	at 140	*[min]*	7 - 9	3 - 5	1 - 3
	at 160	*[min]*	2 - 4	1 - 2	-
	The values shown are for small amounts of pure resin/hardener mix. In composite structures the gel time can differ significantly from the given values depending on the fibre content and the laminate thickness.				
TYPICAL CURE CYCLES	Gelation either				2 - 4 h at 80 °C
	or				1 - 3 h at 90 °C
	Post-cure either				4 - 8 h at 120 °C
	or				2 - 8 h at 140 °C
	or				2 - 8 h at 160 °C
	Cure temperatures in excess of about 130 °C cause brown discolouration but do not impair the properties of the product.				

PROPERTIES OF THE CURED, NEAT FORMULATION

Unless otherwise stated, the processing schedule for the samples tested was gelation for 4 hours at 80 °C and post-cured for 8 hours at 140 °C.

GLASS TRANSITION TEMPERATURE (T_G) (IEC 1006, 10 K/MIN)	*Cure:*		*T_G DSC [°C]*	*T_G TMA [°C]*
	4 h 80 °C + 4 h 120 °C		140 - 144	125 - 128
	4 h 80 °C + 8 h 120 °C		144 - 148	125 - 128
	4 h 80 °C + 4 h 140 °C		145 - 150	130 - 135
	4 h 80 °C + 8 h 140 °C		148 - 153	135 - 145
	4 h 80 °C + 4 h 160 °C		150 - 155	140 - 145
	4 h 80 °C + 8 h 160 °C		150 - 155	140 - 145
TENSILE TEST (ISO 527)	Tensile strength	[MPa]		83 - 93
	Elongation at tensile strength	[%]		4.2 - 5.6
	Ultimate strength	[MPa]		80 - 90
	Ultimate elongation	[%]		5.0 - 7.0
	Tensile modulus	[MPa]		3100 - 3300
FLEXURAL TEST (ISO 178)	Flexural strength	[MPa]		125 - 135
	Deflection at maximum load	[mm]		10 - 18
	10 days in H_2O 23 °C	[MPa]		110 - 120
	Flexural strength	[mm]		8 - 18
	Deflection at maximum load			
	60 min in H_2O/100 °C			
	Flexural strength	[MPa]		125 - 135
	Deflection at maximum load	[mm]		10 - 18
FRACTURE PROPERTIES BEND NOTCH TEST (PM 258-0/90)	Fracture toughness K1C	[MPa√m]		0.56 - 0.6
	Fracture energy G_{1C}	[J/m^2]		88 - 96
WATER ABSORPTION (ISO 62)	*Immersion:*			
	1 day H_2O 23 °C	[%]		0.10 - 0.15
	10 days H_2O 23 °C	[%]		0.30 - 0.40
	30 min H_2O 100 °C	[%]		0.10 - 0.15
	60 min H_2O 100 °C	[%]		0.15 - 0.20
COEFFICIENT OF LINEAR THERMAL EXPANSION (DIN 53 752)	*Mean value:*			
	α from 20 - 100 °C	[10^{-6}/K]		55 - 57
	α from 100 - 130 °C	[10^{-6}/K]		67 - 70
POISSON'S RATIO		[μ]		0.35

PROPERTIES OF THE CURED, REINFORCED FORMULATION

Unless otherwise stated, the figures given are for pressed laminate samples comprising 16 layers (4 mm) of E-glass fabric 1:1, 280 - 300 g/m^2, fibre volume content 42 - 47 %.

FLEXURAL TEST (ISO 178)	Flexural strength	[MPa]	520 - 550
	Deflection at maximum load	[mm]	5 - 6
	Flexural modulus	[MPa]	16500 - 16700
	10 days in H_2O 23 °C		
	Flexural strength	[MPa]	390 - 410
	Deflection at maximum load	[mm]	4 - 5
	60 min in H_2O/100 °C		
	Flexural strength	[MPa]	460 - 480
	Deflection at maximum load	[mm]	5 - 6
TENSILE TEST (ISO 3268 - 1978)	Tensile strength	[MPa]	345 - 375
	Ultimate elongation	[%]	1 - 2
	Tensile modulus	[MPa]	25500 - 26000
INTERLAMINAR SHEAR STRENGTH (ASTM D 2344)	Short beam: E-glass unidirectional specimen Laminate thickness t = 6.4 mm Fibre volume content: 60 %		
	Shear strength:	[MPa]	75 - 77
WATER ABSORPTION (ISO 62)	*Immersion:*		
	1 day H_2O 23 °C	[%]	0.15 - 0.20
	10 days H_2O 23 °C	[%]	0.25 - 0.30
	30 min H_2O 100 °C	[%]	0.01 - 0.05
	60 min H_2O 100 °C	[%]	0.03 - 0.07

TENSILE, COMPRESSIVE AND TORSIONAL TEST (TCT)	E-glass	Roving	E-glass roving, 1200 tex, silane finish
		Fibre volume content	67 %
		Gelation temperature	90 °C
		Post-cure	8 h at 140 °C
	Carbon HT	Roving	Carbon fibre high tensile, Torayca T 300 B - 6000 - 50 B
		Fibre volume content	64 %
		Gelation temperature	90 °C
		Post-cure	8 h at 140 °C

		E-Glass	*Carbon HT*
Transverse tensile test			
Tensile strength	[MPa]	48 - 55	77 - 85
Tensile strain	[%]	0.25 - 0.33	0.9 - 1.0
Elastic modulus	[MPa]	18000 - 20000	9300 - 9900
Transverse compressive test			
Compressive strength	[MPa]	165 - 175	190 - 206
Compressive strain at brak	[%]	1.2 - 1.4	2.7 - 3.4
Elastic modulus	[MPa]	20000 - 22000	9700 - 9900
Torsional test			
Shear strength	[MPa]	77 - 82	76 - 80
Shear angle	[%]	2.7 - 3.1	3.3 - 4.0
Shear modulus	[MPa]	6100 - 7100	6000 - 6300

HANDLING PRECAUTIONS

Personal hygiene	
Safety precautions at workplace	
protective clothing	yes
gloves	essential
arm protectors	recommended when skin contact likely
goggles/safety glasses	yes
Skin protection	
before starting work	Apply barrier cream to exposed skin
after washing	Apply barrier or nourishing cream
Cleansing of contaminated skin	
	Dab off with absorbent paper, wash with warm water and alkali-free soap, then dry with disposable towels. Do not use solvents
Disposal of spillage	
	Soak up with sawdust or cotton waste and deposit in plastic-lined bin
Ventilation	
of workshop	Renew air 3 to 5 times an hour
of workplaces	Exhaust fans. Operatives should avoid inhaling vapours

FIRST AID

Contamination of the *eyes* by resin, hardener or mix should be treated immediately by flushing with clean, running water for 10 to 15 minutes. A doctor should then be consulted.

Material smeared or splashed on the *skin* should be dabbed off, and the contaminated area then washed and treated with a cleansing cream (see above). A doctor should be consulted in the event of severe irritation or burns. Contaminated clothing should be changed immediately.

Anyone taken ill after *inhaling* vapours should be moved out of doors immediately.

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A.3 Nanopox F 400

Technical data sheet

Nanopox F 400

Nanopox F 400 is a high performance, versatile, silica reinforced bisphenol A based epoxy resin for the use in fiber composites. The silica phase consists of surface-modified synthetic SiO_2 nanospheres of very small size (average diameter of 20 nm) with a narrow particle size distribution (maximum diameter 50 nm). Despite the high SiO_2 content of 40 wt%, Nanopox F 400 has a comparatively low viscosity due to the agglomerate-free colloidal dispersion of the nanoparticles in the resin.

Technical data (no specification)

Property	Units	Typical Values
Base resin		bisphenol A diglycidyl ether
Appearance		opaque liquid
SiO_2-content	[wt%]	40
Density @ 20 °C	[g/ml]	1,4
Viscosity @ 25 °C	[mPa·s]	60000
Epoxy equivalent weight	[g/eq]	295
Shelf life	[months]	6 (if stored in the original unopened container)

Processing instructions

Nanopox F 400 can be used as any other bisphenol A diglycidyl ether. However, the colloidal silica in Nanopox products tends to agglomerate if the stabilisation is affected by inappropriate formulation components like hydrocarbon solvents (e.g. xylene). Therefore the compatibility between Nanopox F 400 and all other formulation components should be tested separately before starting formulation development.

Handling and storage

Nanopox F 400 should be handled in accordance with good industrial practice. Detailed information is provided in the Material Safety Data Sheet.

Keep container(s) tightly closed when not in use!

Nanopox F 400 tends to crystallize at ambient temperatures. The product can be easily remelted by heating it up to 70 °C for a short period of time.

The information and data contained in this data sheet are believed to be correct an our today's knowledge of the characteristics and properties of our products. Therefore, any information is given upon condition that the customer shall make his own assessment to determine the suitability of our product for his particular purpose. Since the conditions of use are beyond our control, we accept no liability for any system or application in which our products are utilized unless otherwise stated by us. No warranty of freedom any patent is to be inferred.

April 2006

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Technical Bulletin

NANOPOX® F Products

NANOPOX® products are high-performance concentrates of nanosilica in epoxy resins and have been specially designed for fiber-reinforced composite applications. The silica phase consists of surface-modified SiO_2 nanospheres with a defined size of 20 nm and a very narrow particle-size distribution.

NANOPOX® products are used to replace part of the epoxy resin in existing formulations, typically in the range of 20—30%. Some applications, however, require higher concentrations of nanosilica. They can be blended with all commercially available epoxy resins and cured with all commercially available hardeners.

Introducing nanosilica in the epoxy formulation improves various properties of the fiber-reinforced composite part:

- increased strength, modulus, and hardness
- improved toughness (fracture energy, fracture toughness, impact resistance)
- significantly improved fatigue performance
- drastically increased compressive properties
- no change in Tg

Using NANOPOX® products can significantly improve the performance of fiber-reinforced composite parts.

Because the viscosities of NANOPOX® products and their blends are rather low; these materials are highly suitable for all infusion and injection processes.

With a size of 20 nm, the silica particles penetrate even tightly meshed fabrics easily.

As the particles are completely transparent, the NANOPOX® products can be used for visible parts as well.

Using NANOPOX® F 631, UV-curable systems can be formulated.

NANOPOX® FW 404, the aqueous emulsion of NANOPOX® F 400 is designed for fiber sizing applications. Just a thin layer of this material on the fiber surface is enough to improve the mechanical properties of the composite part.

Areas of Application

Some examples of applications

- pre-pregs
- pultrusion, filament winding, pull winding, and so forth.
- RTM, VARTM, VARI, and so forth.

Technical data NANOPOX® products (no specification)

Product	Base resin	SiO_2 content (wt%)	Viscosity (mPas)	Epoxy equivalent weight	Product specific
NANOPOX® F 400	DGEBA	40	60,000	295	standard grade
NANOPOX® F 440	DGEBA/DGEBF	40	45,000	290	crystallization-free
NANOPOX® F 520	DGEBF	40	20,000	275	standard grade
NANOPOX® F 631	cycloaliphatic epoxy	40	4,000	220	standard grade
NANOPOX® F 640	HDDGE	40	200	245	low viscosity
NANOPOX® FW 404	aqueous emulsion of NANOPOX® F 400	40 (on solid)	7,000	295 (solid)	56% solids content
NANOPOX® F 700	Epoxidized Novolac	40	20,000 (at 50°C)	310	high performance

Packaging

Steel drum, 25 kg
Steel drum, 250 kg
Other types of packaging available on request

Shelf-life

Six months if stored in the original unopened container.

Application Recommendations

Part of the epoxy resin used in the formulation to be improved is replaced with a NANOPOX® F product (up to 100%). The amount of hardener is reduced in proportion to the new epoxy equivalent of the resin blend. For some non-stochiometric hardeners like dicyandiamide a change in the hardener amount is unnecessary.

Fillers and other ingredients of the formulation are used as usual. If the viscosity of NANOPOX® products prove too high for the formulating procedure, we recommend preheating the product to 60-80 °C. The viscosity will be decreased below 10,000 mPas.

Best improvements in composites properties are found usually in the range of 8-10% silica nano-particles.

Formulation example:

	Original formulation	5% SiO_2	10% SiO_2	15% SiO_2
Standard Bisphenol-A epoxy resin (EEW 185)	100	87.5	75	62.5
NANOPOX® F 400 (EEW 295)	–	12.5	25	37.5
Total mass parts	100	100	100	100
EEW	185	194	204	215

07/2014

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(Status: April 2008)

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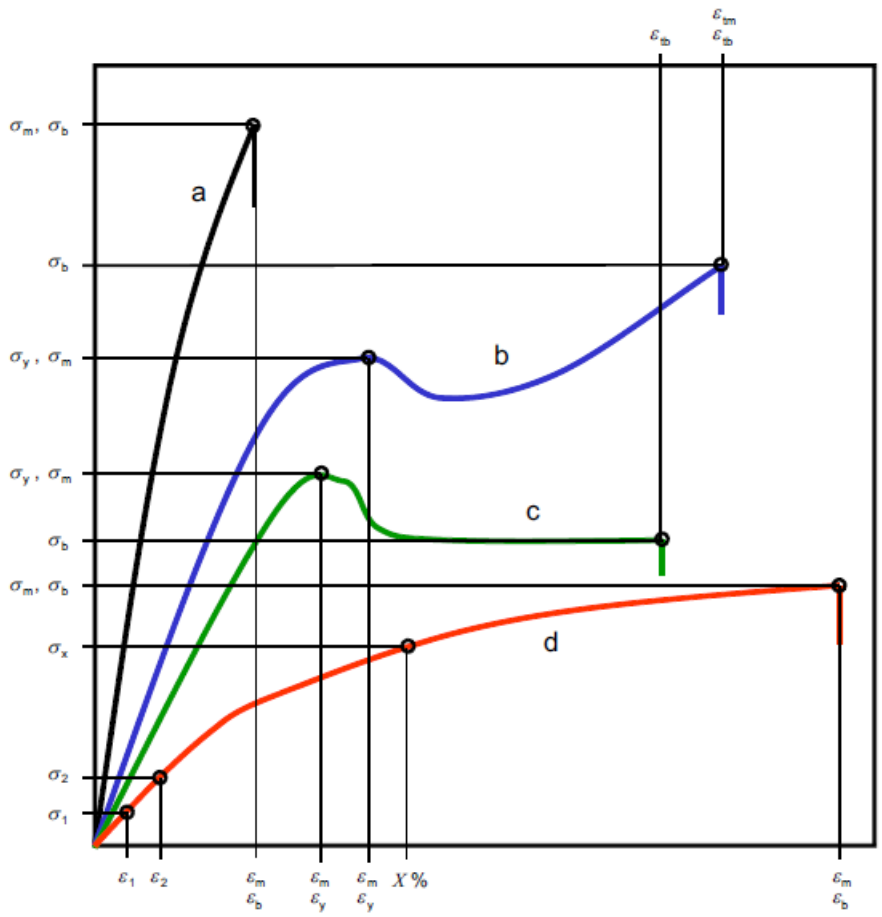
EVONIK INDUSTRIES

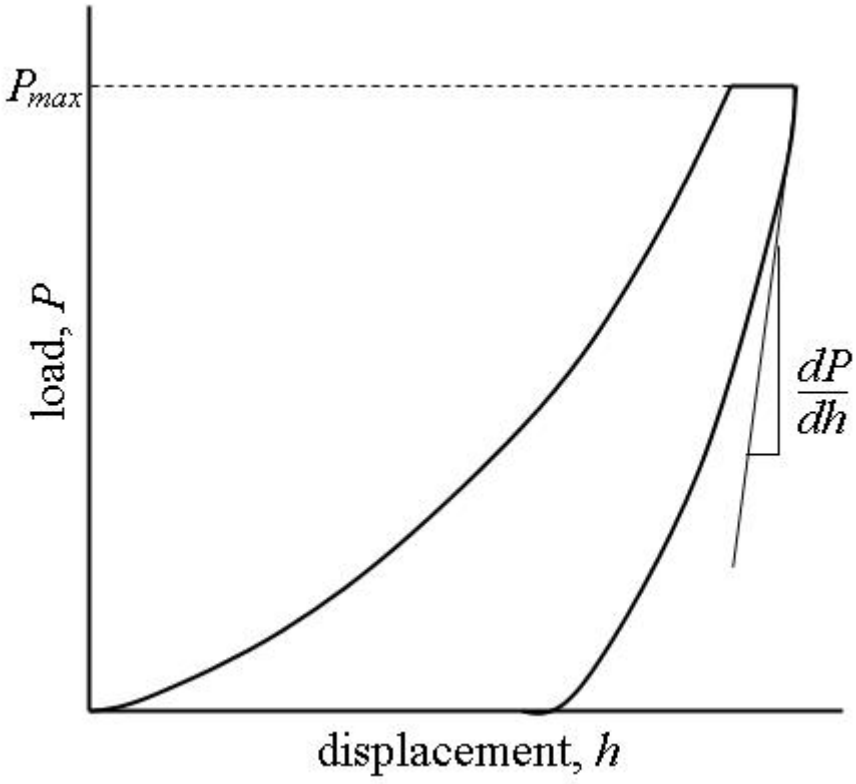
Appendix B Typical stress-strain curves for plastic materials

Typical stress-strain curves for plastic materials. The figure is taken from *ISO 527-1:2012 Plastics – Determination of tensile properties – Part 1: General principles*. It indicates how the different material properties are found from the stress-strain curves.



Appendix C Typical indentation load-displacement curve

Load-displacement curve for a typical indentation measurement cycle. The specimen is loaded to P_{max}, held at P_{max} for some time, and then unloaded to zero load. The elastic modulus of the specimen is determined from the tangent of the unloading curve, dP/dh.



Appendix D Anhydride-cured composite that was under-cured

During the production of the anhydride-cured plate containing 9.1 wt% silica, the oven used for curing failed overnight when the curing cycle was running. Thus, the oven shut down, the temperature dropped, and the curing of the plate was not completed. This resulted in a composite that was under-cured. It is not known at which point during the curing cycle the oven failed. However, the polymer matrix was, as a minimum, cured beyond the gel point.

A new, fully cured plate was produced. The measured properties of the fully cured and the under-cured plates are compared in the table below.

	Fully cured	**Under-cured**
Silica content (wt%)	9.1	9.1
Silica content (vol%)	5.4	5.4
ρ (kg/m^3)	1244 ± 1	1253 ± 1
T_g (^{o}C)	151 ± 0	133 ± 0
E' (MPa)	3410 ± 80	3780 ± 90
E_r (MPa)	39.8 ± 1.2	38.2 ± 0.2
E_t (MPa)	3530 ± 100	3940 ± 50
σ_m (MPa)	89 ± 2	74 ± 4
ε_b (%)	4.9 ± 0.7	2.3 ± 0.2

A much lower T_g, which would indicate a lower crosslink density of the epoxy polymer, and higher values for E' and E_t were observed for the under-cured composite. Much lower values of σ_m and ε_b were also observed, indicating that the under-cured composite is much more brittle than the fully cured composite.

Appendix E Tensile test results

E.1 Amine-cured system

LY556/XB3473, neat epoxy polymer

Zwick / Roell

30.10.14

Test report

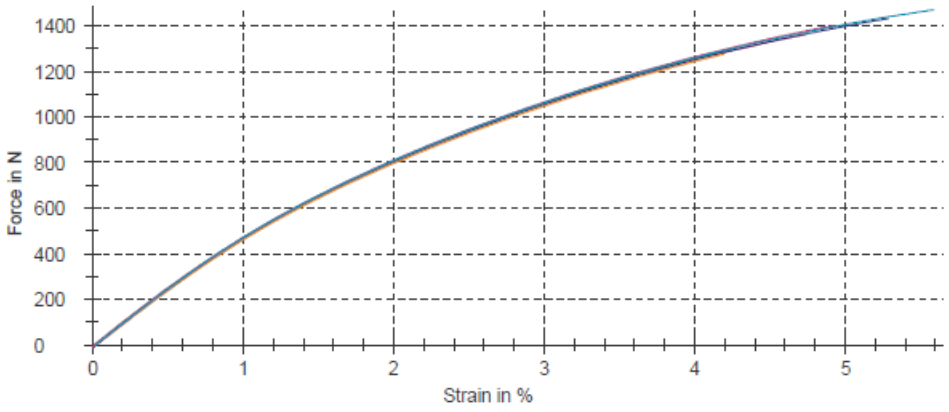
Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of	:	Note	:
Material	:	Machine data	:
Specimen removal	:		

Speed, tensile modulus	:	1	mm/min
Test speed	:	1	mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	Neat	2610	-	68,1	4,7	68,0	4,7	3,94	5,07	19,98
2	Neat	2560	-	64,7	4,2	64,7	4,2	3,96	5,06	20,04
3	Neat	2620	-	71,8	5,3	71,8	5,3	3,945	5,05	19,92
4	Neat	2570	-	64,6	4,2	64,6	4,2	3,92	5,03	19,72
5	Neat	2640	-	70,3	4,9	70,3	4,9	3,95	5,04	19,91
6	Neat	2630	-	73,2	5,6	73,2	5,6	3,96	5,07	20,08

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	2600	-	68,8	4,8	68,8	4,8	3,946	5,053	19,94
s	33,1	-	3,62	0,6	3,63	0,6	0,01497	0,01633	0,13
v	1,27	-	5,27	11,54	5,27	11,55	0,38	0,32	0,64

LY556-XB3573-100-23.zs2 **Page 1/1**

Zwick / Roell

30.10.14

Test report

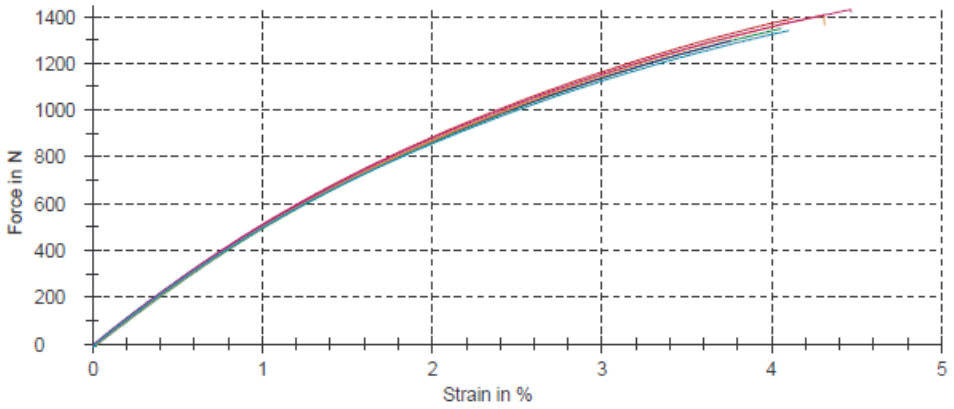
Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of	:	Note	:
Material	:	Machine data	:
Specimen removal	:		

Speed, tensile modulus	:	1	mm/min
Test speed	:	1	mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1		2770	-	68,1	4,1	68,1	4,1	4,05	5,05	20,45
2		2740	-	67,0	4,1	67,0	4,1	3,98	5,06	20,14
3		2870	-	64,9	3,8	64,9	3,8	3,96	5,04	19,96
4		2780	-	69,3	4,3	67,1	4,3	4,03	5,04	20,31
5		2810	-	71,1	4,5	70,6	4,5	4	5,03	20,12
6		2840	-	67,8	4,1	67,8	4,1	3,93	5,03	19,77

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	2800	-	68,0	4,1	67,6	4,1	3,992	5,042	20,12
s	46,6	-	2,08	0,2	1,84	0,2	0,04446	0,01169	0,24
v	1,66	-	3,05	5,82	2,72	5,83	1,11	0,23	1,21

LY556-F400-XB3573-80-20-21,3.zs2 **Page 1/1**

Zwick / Roell

27.11.14

Test report

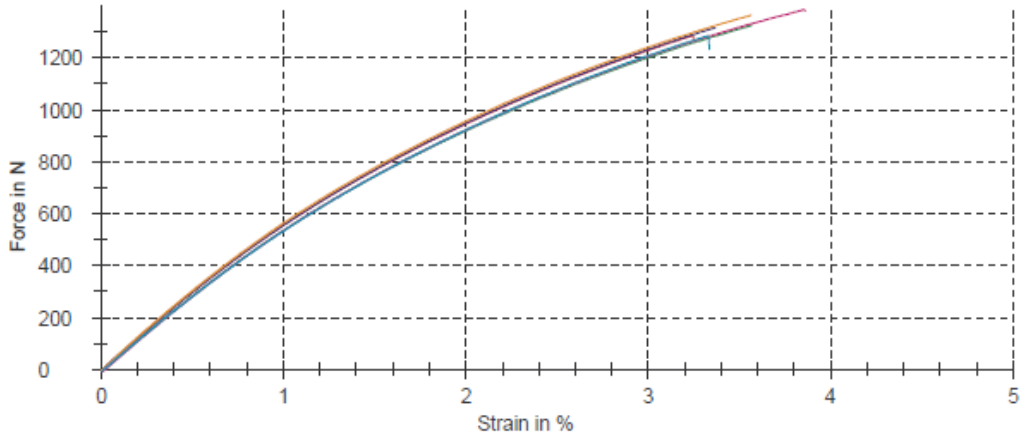
Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of	:	Note	:
Material	:	Machine data	:
Specimen removal	:		

Speed, tensile modulus	:	1	mm/min
Test speed	:	1	mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1		3070	-	63,6	3,2	62,6	3,2	4,01	5,04	20,21
2		3000	-	66,1	3,6	66,1	3,6	3,985	5,03	20,04
3		3070	-	65,2	3,4	65,1	3,4	4	5,04	20,16
4		3140	-	67,5	3,6	67,5	3,6	4,005	5,04	20,19
5		3000	-	69,0	3,9	68,8	3,9	3,99	5,03	20,07
6		2930	-	63,7	3,3	61,1	3,3	4	5,04	20,16

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3040	-	65,9	3,5	65,2	3,5	3,998	5,037	20,14
s	73,9	-	2,14	0,2	2,89	0,2	0,009309	0,005164	0,07
v	2,43	-	3,25	6,38	4,44	6,35	0,23	0,10	0,33

LY556-F400-XB3573-60-40-19,6.zs2

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LY556/F400/XB3473, 12.3 vol% silica

Zwick / Roell

27.11.14

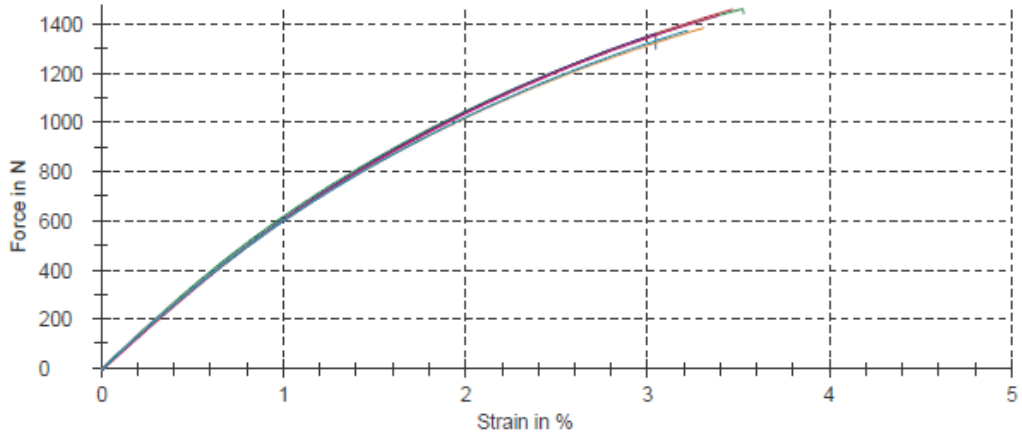
Test report

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1		3190	-	70,1	3,5	70,1	3,5	4,12	5,05	20,81
2		3390	-	71,9	3,5	70,9	3,5	4,03	5,04	20,31
3		3270	-	66,0	3,0	63,0	3,0	4,08	5,05	20,60
4		3410	-	69,3	3,3	69,3	3,3	3,95	5,05	19,95
5		3270	-	69,9	3,4	69,9	3,4	4,06	5,05	20,50
6		3350	-	68,1	3,2	68,1	3,2	4	5,04	20,16

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3310	-	69,2	3,3	68,5	3,3	4,04	5,047	20,39
s	84,4	-	2,01	0,2	2,87	0,2	0,06033	0,005164	0,31
v	2,55	-	2,91	5,29	4,18	5,28	1,49	0,10	1,53

LY556-F400-XB3573-40-60-17,9.zs2 **Page 1/1**

Zwick / Roell

27.11.14

Test report

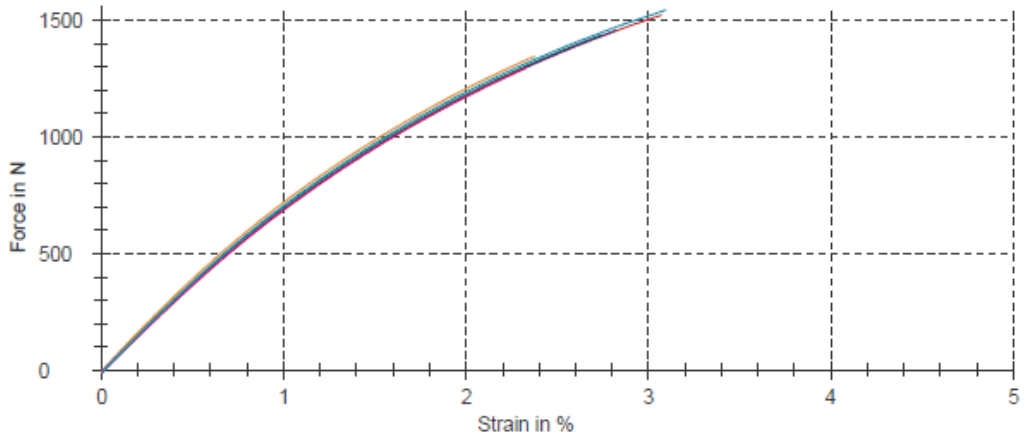
Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of	:	Note	:
Material	:	Machine data	:
Specimen removal	:		

Speed, tensile modulus	:	1	mm/min
Test speed	:	1	mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1		3600	-	71,0	3,1	71,0	3,1	4,22	5,08	21,44
2		3730	-	67,6	2,7	67,6	2,7	4,17	5,1	21,27
3		3600	-	68,1	2,8	68,1	2,8	4,21	5,08	21,39
4		3860	-	63,2	2,4	63,2	2,4	4,19	5,08	21,29
5		3580	-	61,3	2,3	61,3	2,3	4,15	5,09	21,12
6		3730	-	72,2	3,1	72,2	3,1	4,21	5,08	21,39

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3680	-	67,2	2,7	67,2	2,7	4,192	5,085	21,31
s	111	-	4,25	0,3	4,25	0,3	0,02714	0,008367	0,11
v	3,03	-	6,32	12,03	6,32	12,03	0,65	0,16	0,54

LY556-F400-XB3573-20-80-16,3.zs2

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E.2 Anhydride-cured system

LY556/ Aradur917/DY070, neat epoxy polymer

Zwick / Roell

27.11.14

Test report

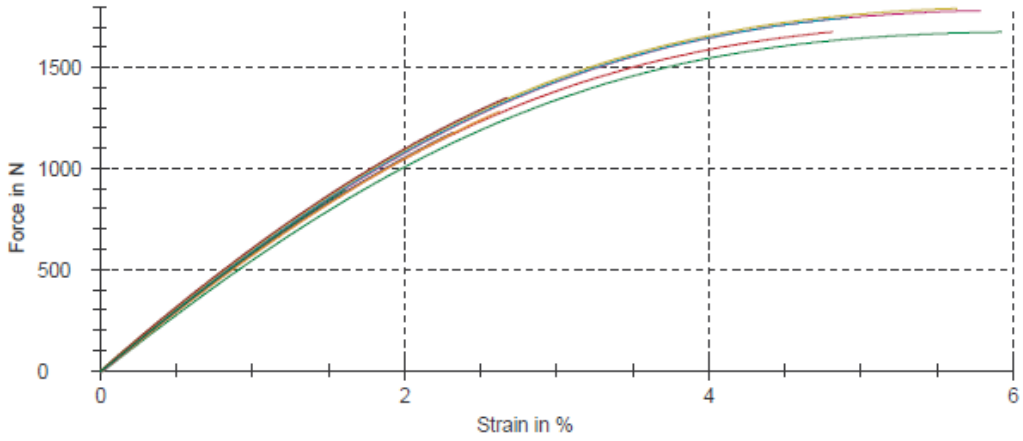
Job no. : LY556-Aradur917-100-90
Material : LY556-Aradur917-100-90
Tester : Marie

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	LY556-Aradur917-100-90	3230	-	84,8	4,8	84,8	4,8	3,98	4,97	19,78
2	LY556-Aradur917-100-90	3020	-	86,9	5,9	86,8	5,9	3,89	4,96	19,29
3	LY556-Aradur917-100-90	3070	-	58,7	2,3	58,7	2,3	4,01	5,01	20,09
6	LY556-Aradur917-100-90	2960	-	62,9	2,6	62,9	2,6	4,09	4,99	20,41
7	LY556-Aradur917-100-90	3020	-	86,8	5,8	86,8	5,8	4,135	4,97	20,55
8	LY556-Aradur917-100-90	3120	-	85,3	4,9	85,3	4,9	4,11	4,99	20,51
9	LY556-Aradur917-100-90	3120	-	87,0	5,6	86,9	5,6	4,14	4,98	20,62
10	LY556-Aradur917-100-90	3180	-	65,8	2,7	65,8	2,7	4,12	4,99	20,56
11	LY556-Aradur917-100-90	3010	-	44,4	1,6	44,4	1,6	4,03	5,01	20,19

Series graph:



Statistics:

Series n = 9	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3080	-	73,6	4,0	73,6	4,0	4,056	4,986	20,22
s	88,4	-	16,0	1,7	16,0	1,7	0,08484	0,0174	0,44
v	2,87	-	21,70	42,07	21,67	42,19	2,09	0,35	2,19

LY556-Aradur917-100-90.zs2 **Page 1/1**

Zwick / Roell

28.11.14

Test report

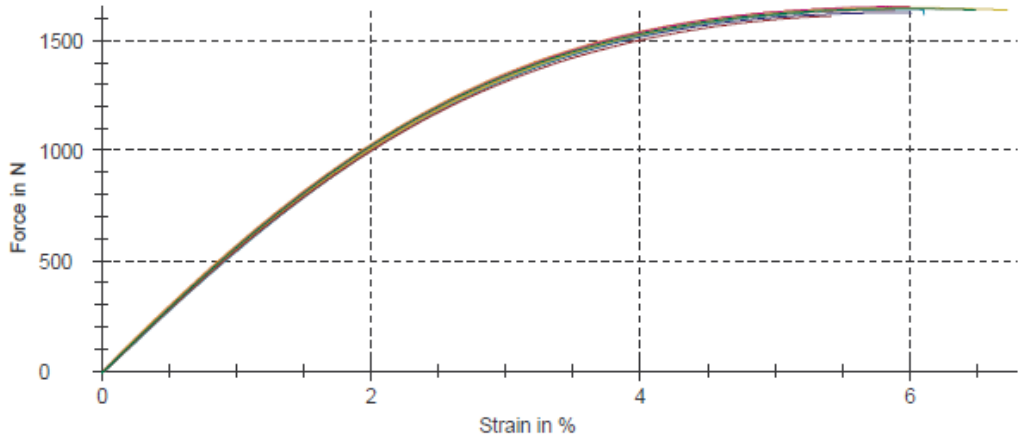
Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of	:	Note	:
Material	:	Machine data	:
Specimen removal	:		

Speed, tensile modulus	:	1	mm/min
Test speed	:	1	mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1		3030	-	83,9	5,9	83,9	5,9	3,88	5,03	19,52
2		2970	-	82,9	5,2	82,9	5,2	3,89	5,04	19,61
3		2990	-	84,0	5,9	83,9	6,0	3,87	5	19,35
4		3150	-	84,3	5,8	84,3	5,8	3,91	5	19,55
5		3040	-	84,2	5,9	84,2	6,0	3,93	4,99	19,61
6		2890	-	84,0	5,9	82,7	6,1	3,9	5,01	19,54
7		3060	-	84,4	6,1	83,9	6,7	3,88	5,03	19,52
8		3000	-	82,8	5,4	82,8	5,4	3,88	5,01	19,44
9		3020	-	83,6	6,0	83,3	6,5	3,95	4,98	19,67

Series graph:



Statistics:

Series n = 9	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
x	3020	-	83,8	5,8	83,5	6,0	3,899	5,01	19,53
s	68,6	-	0,580	0,3	0,618	0,5	0,02667	0,02	0,10
v	2,28	-	0,69	4,71	0,74	7,84	0,68	0,40	0,49

LY556-Ar917-0.zs2

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Zwick / Roell

27.11.14

Test report

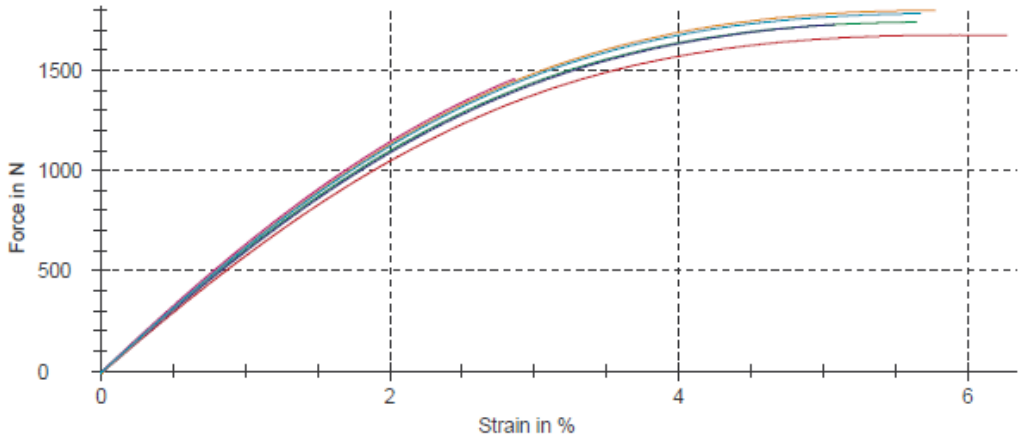
Material : LY556-F400-Aradur917-80-20-83
Tester : Marie

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	LY556-F400-Ar917-80-20-83	3240	-	89,3	5,9	89,1	6,3	3,77	4,98	18,77
2	LY556-F400-Ar917-80-20-83	3320	-	88,9	5,6	88,9	5,6	3,92	4,99	19,56
3	LY556-F400-Ar917-80-20-83	3260	-	88,3	5,1	88,3	5,1	3,92	4,99	19,56
4	LY556-F400-Ar917-80-20-83	3250	-	89,2	5,8	89,2	5,8	4,04	4,99	20,16
5	LY556-F400-Ar917-80-20-83	3360	-	71,9	2,9	71,4	2,9	4,06	4,99	20,26
6	LY556-F400-Ar917-80-20-83	3240	-	88,8	5,7	88,8	5,7	4,02	4,99	20,06

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3280	-	86,1	5,1	86,0	5,2	3,955	4,988	19,73
s	49,7	-	6,96	1,2	7,16	1,2	0,1088	0,004082	0,56
v	1,52	-	8,08	22,39	8,33	23,27	2,75	0,08	2,82

LY556-F400-Aradur917-80-20-83.zs2 **Page 1/1**

Zwick / Roell

27.11.14

Test report

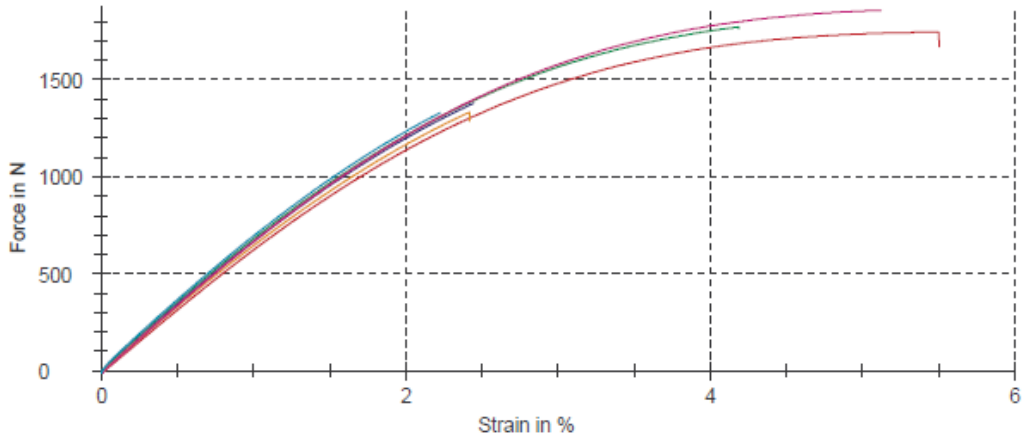
Material : LY554-F400-Aradur917-60-40-75,9
Tester : Marie

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	LY554-F400-Ar917-60-40-75,9	3380	-	90,1	5,4	86,3	5,5	3,88	4,99	19,36
2	LY554-F400-Ar917-60-40-75,9	3590	-	87,4	4,2	87,1	4,2	4,07	4,98	20,27
3	LY554-F400-Ar917-60-40-75,9	3580	-	68,5	2,4	68,5	2,4	4,04	4,98	20,12
4	LY554-F400-Ar917-60-40-75,9	3590	-	67,5	2,4	65,1	2,4	3,97	4,98	19,77
5	LY554-F400-Ar917-60-40-75,9	3410	-	90,1	5,1	90,1	5,1	4,14	4,98	20,62
6	LY554-F400-Ar917-60-40-75,9	3600	-	64,9	2,2	64,9	2,2	4,11	4,99	20,51

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3530	-	78,1	3,6	77,0	3,7	4,035	4,983	20,11
s	99,9	-	12,3	1,5	12,0	1,5	0,09607	0,005164	0,47
v	2,83	-	15,73	40,10	15,60	40,38	2,38	0,10	2,35

LY554-F400-Aradur917-60-40-75,9.zs2

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Zwick / Roell

27.11.14

Test report

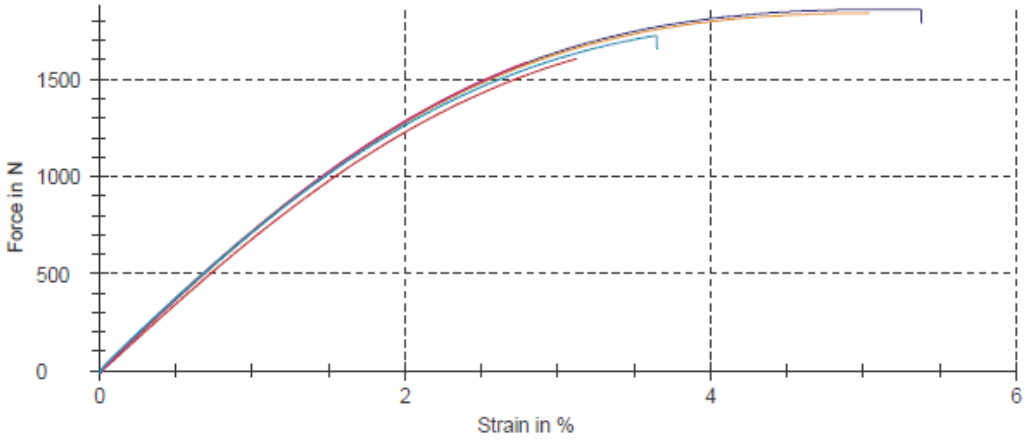
Material : LY556-F400-Aradur917-40-60-68,9
Tester : Marie

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	LY556-F400-Ar917-40-60-68,9	3640	-	82,2	3,1	82,2	3,1	3,92	4,98	19,52
2	LY556-F400-Ar917-40-60-68,9	3750	-	79,2	2,9	79,2	2,9	4,05	4,98	20,17
3	LY556-F400-Ar917-40-60-68,9	3830	-	91,8	5,2	88,2	5,4	4,07	4,98	20,27
4	LY556-F400-Ar917-40-60-68,9	3870	-	91,7	5,0	91,7	5,0	4,03	4,98	20,07
5	LY556-F400-Ar917-40-60-68,9	3830	-	78,3	2,8	78,3	2,8	4,07	4,98	20,27
6	LY556-F400-Ar917-40-60-68,9	3870	-	87,7	3,6	84,3	3,6	3,95	4,98	19,67

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	3800	-	85,2	3,8	84,0	3,8	4,015	4,98	19,99
s	88,5	-	6,09	1,1	5,22	1,1	0,06442	0,000	0,32
v	2,33	-	7,15	28,40	6,21	29,67	1,60	0,00	1,60

LY556-F400-Aradur917-40-60-68,9.zs2

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Zwick / Roell

27.11.14

Test report

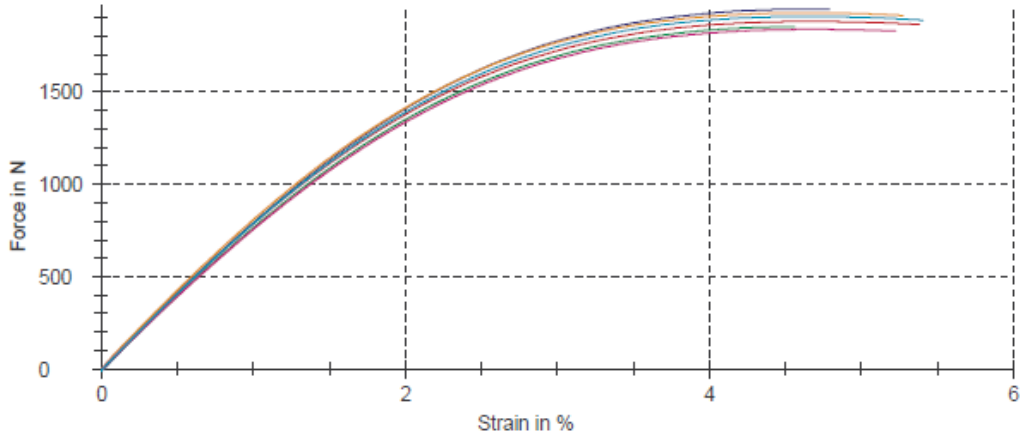
Material : LY556-F400-Aradur917-80-20-61,9
Tester : Marie

Speed, tensile modulus : 1 mm/min
Test speed : 1 mm/min

Test results:

Nr	Specimen ID	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
1	LY556-F400-Ar917-20-80-61,9	4260	-	93,3	4,7	92,5	5,4	4,05	4,98	20,17
2	LY556-F400-Ar917-20-80-61,9	4190	-	93,0	4,6	92,9	4,6	3,99	5	19,95
3	LY556-F400-Ar917-20-80-61,9	4100	-	93,8	4,7	93,7	4,8	4,18	4,97	20,77
4	LY556-F400-Ar917-20-80-61,9	4190	-	93,7	4,7	93,0	5,3	4,14	4,975	20,60
5	LY556-F400-Ar917-20-80-61,9	4050	-	93,2	4,7	92,7	5,2	3,97	4,97	19,73
6	LY556-F400-Ar917-20-80-61,9	4100	93,6	93,6	4,6	92,6	5,4	4,1	4,975	20,40

Series graph:



Statistics:

Series n = 6	E_t MPa	σ_Y MPa	σ_M MPa	ε_M %	σ_B MPa	ε_B %	h mm	b mm	A_0 mm²
$\bar{x}$	4150	93,6	93,4	4,6	92,9	5,1	4,072	4,978	20,27
s	77,7	-	0,322	0,0	0,452	0,3	0,08329	0,01125	0,40
v	1,87	-	0,34	1,02	0,49	6,82	2,05	0,23	1,95

LY556-F400-Aradur917-20-80-61,9.zs2

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Appendix F Indentation measurements

F.1 Amine-cured system

LY556/XB3473, neat epoxy polymer

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.06	1.5808	0.4886	1.1742	131.421	124.190	222.568	2.843e+003	6.065	47.835	20.565	9.734	16.997	XB3473_100_2(1)
2	10.06	1.5443	0.4497	1.1229	137.748	129.912	240.214	2.856e+003	4.882	51.238	22.196	9.641	17.330	XB3473_100_2(2)
3	10.05	1.5926	0.5085	1.1775	129.376	124.709	220.475	2.772e+003	5.425	49.210	20.372	9.990	16.123	XB3473_100_2(3)
4	10.06	1.5875	0.4989	1.1768	130.322	127.507	221.290	2.809e+003	5.698	48.886	20.447	10.075	15.867	XB3473_100_2(4)
5	10.05	1.5832	0.4628	1.1692	130.870	127.137	223.394	2.797e+003	5.604	49.331	20.642	9.607	17.428	XB3473_100_2(5)
Average	10.06	1.5777	0.4817	1.1641	131.947	126.691	225.588	2.815e+003	5.535	49.300	20.844	9.809	16.749	
Std. Dev.	0.007	0.019	0.025	0.023	3.329	2.314	8.254	34.127	0.433	1.233	0.763	0.211	0.713	
CV	0.065	1.216	5.130	2.001	2.523	1.827	3.659	1.212	7.831	2.501	3.659	2.150	4.255	

LY556/F400/XB3473, 3.7 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.06	1.5872	0.5878	1.2153	130.390	132.616	211.697	3.032e-003	7.507	43.553	19.561	9.428	18.123	Epoksy_80_b(1)
2	10.05	1.5363	0.5160	1.1453	139.041	138.744	234.295	3.036e-003	5.521	48.037	21.649	9.543	17.670	Epoksy_80_b(2)
3	10.05	1.5615	0.5538	1.1753	134.571	142.239	223.754	3.004e-003	6.346	46.257	20.675	9.289	18.647	Epoksy_80(1)
4	10.06	1.5728	0.5577	1.1892	132.754	137.524	219.330	2.992e-003	6.678	45.609	20.266	9.468	17.961	Epoksy_80(2)
5	10.06	1.5690	0.5516	1.1860	133.417	137.149	220.539	3.003e-003	6.515	45.771	20.378	9.346	18.438	Epoksy_80(3)
Average	10.06	1.5654	0.5534	1.1822	134.035	137.654	221.923	3.013e-003	6.514	45.845	20.506	9.415	18.168	
Std. Dev.	0.005	0.019	0.026	0.025	3.188	3.459	8.210	19.483	0.712	1.604	0.759	0.100	0.386	
CV	0.048	1.196	4.610	2.145	2.379	2.513	3.700	0.647	10.930	3.498	3.700	1.063	2.124	

LY556/F400/XB3473, 7.8 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.05	1.5316	0.5458	1.1723	139.885	137.900	227.223	3.253e+003	7.316	43.653	20.995	9.304	18.588	XB3473_60(1)
2	10.07	1.4886	0.5440	1.1242	148.271	140.882	245.382	3.332e+003	6.821	44.740	22.673	9.403	18.221	XB3473_60(2)
3	10.06	1.4950	0.5112	1.1211	146.962	143.855	245.616	3.254e+003	6.201	46.846	22.695	9.185	19.093	XB3473_60(3)
4	10.06	1.5036	0.5302	1.1294	145.277	140.776	242.259	3.222e+003	6.483	45.671	22.385	8.952	20.099	XB3473_60(4)
5	10.05	1.4988	0.5321	1.1239	146.071	142.729	244.122	3.235e+003	6.243	46.304	22.557	8.952	20.079	XB3473_60(5)
Average	10.06	1.5035	0.5326	1.1342	145.293	141.228	240.920	3.259e+003	6.613	45.443	22.261	9.159	19.216	
Std. Dev.	0.006	0.017	0.014	0.022	3.221	2.267	7.772	42.882	0.464	1.271	0.718	0.204	0.855	
CV	0.063	1.105	2.600	1.897	2.217	1.605	3.226	1.316	7.018	2.796	3.226	2.232	4.449	

LY556/F400/XB3473, 12.3 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.05	1.5301	0.5697	1.2114	140.155	140.045	217.139	3.570e+003	9.765	38.190	20.064	8.671	21.398	XB3473_40_2(2)
2	10.06	1.5121	0.5720	1.1866	143.644	143.481	225.335	3.570e+003	8.934	39.758	20.821	8.833	20.643	XB3473_40_2(3)
3	10.06	1.5096	0.5682	1.1799	144.163	144.816	227.417	3.542e+003	8.543	40.462	21.013	8.375	22.968	XB3473_40_2(4)
4	10.05	1.4982	0.5580	1.1645	146.222	146.049	232.475	3.542e+003	8.191	41.297	21.481	8.448	22.549	XB3473_40_2(5)
5	10.03	1.4752	0.5479	1.1374	150.492	146.526	241.979	3.560e+003	8.096	41.922	22.359	8.962	19.997	XB3473_40_2(6)
Average	10.05	1.5050	0.5632	1.1760	144.935	144.183	228.869	3.557e+003	8.706	40.326	21.147	8.658	21.511	
Std. Dev.	0.012	0.020	0.010	0.027	3.796	2.597	9.180	13.900	0.678	1.448	0.848	0.249	1.251	
CV	0.121	1.343	1.789	2.330	2.619	1.801	4.011	0.391	7.786	3.592	4.011	2.871	5.814	

LY556/F400/XB3473, 17.2 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.04	1.3876	0.4920	1.0624	170.261	168.299	276.469	3.962e+003	6.505	44.032	25.546	8.592	21.778	XB3473_20_2(1)
2	10.04	1.4499	0.5857	1.1421	155.879	156.626	243.249	3.917e+003	8.911	38.845	22.476	9.014	19.778	XB3473_20_2(2)
3	10.04	1.4173	0.5440	1.1014	163.165	163.399	259.481	3.952e+003	7.649	41.492	23.976	8.764	20.924	XB3473_20_2(3)
4	10.04	1.4073	0.5219	1.0841	165.506	163.456	266.452	3.907e+003	7.265	42.329	24.620	9.133	19.271	XB3473_20_2(4)
5	10.04	1.4004	0.4888	1.0779	167.160	163.550	269.403	3.944e+003	7.096	42.946	24.893	8.523	22.127	XB3473_20_2(5)
Average	10.04	1.4125	0.5265	1.0936	164.394	163.066	263.011	3.936e+003	7.485	41.929	24.302	8.805	20.776	
Std. Dev.	0.002	0.024	0.040	0.031	5.416	4.164	12.618	23.247	0.897	1.957	1.166	0.263	1.236	
CV	0.015	1.667	7.622	2.789	3.294	2.553	4.797	0.591	11.989	4.667	4.797	2.992	5.949	

F.2 Anhydride-cured system

LY556/Aradur917/DY070, neat epoxy polymer

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.06	1.4679	0.6956	1.1286	152.476	143.422	246.135	3.585e+003	8.396	37.571	22.743	9.265	18.769	Aradur_100_d(1)
2	10.07	1.4883	0.6271	1.1416	148.326	147.773	240.216	3.479e+003	7.122	39.891	22.196	9.701	17.118	Aradur_100_d(2)
3	10.06	1.4952	0.5719	1.1362	146.954	147.625	241.128	3.360e+003	6.389	42.025	22.280	9.514	17.799	Aradur_100_d(3)
4	10.07	1.4740	-0.2434	1.1106	151.231	148.198	251.086	3.391e+003	6.465	43.069	23.200	9.299	18.634	Aradur_100_d(4)
5	10.05	1.4857	0.6979	1.1421	148.703	175.205	240.097	3.501e+003	8.155	40.078	22.185	9.397	18.228	Aradur_100c(2)
6	10.05	1.4812	0.6881	1.1363	149.553	169.354	242.169	3.494e+003	7.906	40.177	22.376	9.315	18.541	Aradur_100c(3)
7	10.07	1.4739	0.6539	1.1243	151.288	165.054	246.924	3.493e+003	7.323	41.382	22.816	9.806	16.761	Aradur_100c(4)
8	10.05	1.4803	0.6681	1.1347	149.689	166.767	242.660	3.497e+003	7.106	41.049	22.422	9.160	19.170	Aradur_100c(5)
9	10.06	1.4748	0.6546	1.1262	151.043	163.734	246.159	3.492e+003	6.734	41.866	22.745	9.032	19.749	Aradur_100c(6)
10	10.05	1.5545	0.8213	1.2239	135.763	134.283	211.995	3.415e+003	9.652	34.442	19.588	9.656	17.255	Aradur_100(5)
11	10.05	1.5076	0.7375	1.1602	144.356	141.228	232.735	3.407e+003	8.127	38.349	21.505	9.473	17.928	Aradur_100(6)
12	10.05	1.4012	0.6546	1.0450	167.023	146.536	281.258	3.659e+003	7.535	40.439	25.988	9.930	16.310	Aradur_100(7)
13	10.07	1.4141	0.6852	1.0672	164.384	147.778	272.329	3.691e+003	7.679	39.180	25.163	10.101	15.799	Aradur_100(8)
14	10.07	1.4427	0.6939	1.0879	157.911	146.643	261.885	3.546e+003	7.625	39.732	24.198	9.695	17.146	Aradur_100(9)
Average	10.06	1.4744	0.6147	1.1261	151.336	153.114	246.913	3.501e+003	7.587	39.946	22.815	9.524	17.801	
Std. Dev.	0.008	0.038	0.253	0.042	7.812	12.341	16.765	95.918	0.859	2.157	1.549	0.305	1.128	
CV	0.084	2.552	41.196	3.756	5.162	8.060	6.790	2.740	11.320	5.400	6.790	3.203	6.339	

LY556/F400/Aradur917/DY070, 2.5 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.06	1.4691	0.6638	1.1227	152.154	150.301	247.701	3.528e+003	6.825	40.438	22.888	9.472	17.950	Aradur_80_2(1)
2	10.04	1.4827	0.6830	1.1381	149.121	146.618	241.288	3.490e+003	7.508	38.993	22.295	9.133	19.274	Aradur_80_2(2)
3	10.04	1.4813	0.6806	1.1421	149.316	147.895	240.100	3.539e+003	7.463	39.112	22.185	9.649	17.255	Aradur_80_2(3)
4	10.04	1.4411	0.6278	1.0876	157.835	151.691	261.458	3.546e+003	6.473	41.501	24.159	9.393	18.216	Aradur_80_2(4)
5	10.05	1.4480	0.6183	1.0959	156.419	150.946	258.086	3.547e+003	6.541	41.645	23.847	9.276	18.691	Aradur_80_2(5)
Average	10.05	1.4644	0.6547	1.1173	152.969	149.490	249.727	3.530e+003	6.962	40.338	23.075	9.385	18.277	
Std. Dev.	0.009	0.019	0.030	0.025	4.013	2.146	9.689	23.702	0.496	1.263	0.895	0.196	0.762	
CV	0.089	1.302	4.584	2.198	2.623	1.435	3.880	0.671	7.124	3.132	3.880	2.084	4.167	

LY556/F400/Aradur917/DY070, 5.4 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.06	1.4642	0.6614	1.1408	153.249	153.788	242.837	3.743e+003	7.475	37.949	22.438	——	——	Aradur_60(3)
2	10.04	1.4063	0.6017	1.0632	165.643	159.834	273.731	3.747e+003	5.614	42.175	25.293	——	——	Aradur_60(4)
3	10.05	1.4260	0.6430	1.0888	161.311	162.588	262.943	3.739e+003	5.828	41.073	24.296	——	——	Aradur_60(6)
4	10.04	1.4288	0.6451	1.0913	160.516	161.112	261.494	3.722e+003	5.919	40.918	24.162	——	——	Aradur_60(7)
5	10.04	1.4331	0.6496	1.0998	159.576	157.160	258.252	3.742e+003	6.226	40.145	23.862	——	——	Aradur_60(8)
6	10.04	1.4300	0.6422	1.0940	160.261	157.703	260.514	3.732e+003	6.380	40.418	24.071	——	——	Aradur_60(9)
7	10.06	1.4353	0.6482	1.1007	159.475	156.808	258.351	3.731e+003	6.167	40.221	23.872	——	——	Aradur_60(10)
8	10.04	1.4311	0.6433	1.0975	160.020	156.284	259.244	3.751e+003	6.282	40.282	23.954	——	——	Aradur_60(11)
9	10.05	1.4296	0.6443	1.0956	160.488	156.804	260.234	3.753e+003	6.348	40.416	24.046	——	——	Aradur_60(12)
10	10.04	1.4314	0.6395	1.0945	159.931	156.045	260.138	3.717e+003	6.317	40.481	24.037	——	——	Aradur_60(13)
11	10.04	1.4269	0.6388	1.0885	160.943	157.784	262.658	3.720e+003	6.135	40.750	24.270	——	——	Aradur_60(14)
12	10.05	1.4385	0.6518	1.1017	158.478	155.820	257.224	3.698e+003	6.390	40.141	23.768	——	——	Aradur_60(15)
13	10.04	1.4286	0.6403	1.0940	160.618	157.421	260.722	3.754e+003	6.305	40.534	24.091	——	——	Aradur_60(16)
14	10.05	1.4251	0.6346	1.0857	161.473	157.570	264.005	3.720e+003	6.268	40.816	24.394	——	——	Aradur_60(17)
15	10.04	1.4323	0.6447	1.0943	159.758	157.575	260.168	3.706e+003	6.281	40.534	24.039	——	——	Aradur_60(18)
16	10.04	1.4298	0.6378	1.0895	160.319	157.992	262.039	3.699e+003	6.269	40.746	24.212	——	——	Aradur_60(19)
17	10.04	1.4306	0.6446	1.0966	160.126	155.596	259.596	3.745e+003	6.407	40.160	23.987	——	——	Aradur_60(20)
18	10.05	1.4288	0.6432	1.0924	160.645	156.122	261.359	3.732e+003	6.395	40.408	24.150	——	——	Aradur_60(21)
19	10.07	1.4306	0.6362	1.0886	160.572	156.934	262.950	3.692e+003	6.245	40.949	24.297	——	——	Aradur_60(22)
20	10.04	1.4317	0.6403	1.0946	159.857	155.842	260.067	3.716e+003	6.312	40.524	24.030	——	——	Aradur_60(23)
21	10.04	1.4331	0.6422	1.0966	159.565	156.974	259.299	3.713e+003	6.386	40.246	23.959	——	——	Aradur_60(24)
22	10.07	1.4438	0.6559	1.1129	157.663	159.287	253.617	3.744e+003	6.459	39.736	23.434	——	——	Aradur_60(25)
23	10.07	1.4334	0.6399	1.0954	160.011	156.094	260.483	3.715e+003	6.344	40.490	24.069	——	——	Aradur_60(26)
24	10.07	1.4331	0.6446	1.0947	160.043	157.054	260.701	3.713e+003	6.375	40.517	24.089	——	——	Aradur_60(27)
25	10.04	1.4371	0.6508	1.1060	158.674	155.230	255.806	3.752e+003	6.527	39.804	23.636	——	——	Aradur_60(28)
26	10.07	1.4329	0.6409	1.0945	160.072	155.445	260.764	3.707e+003	6.304	40.483	24.095	——	——	Aradur_60(29)
27	10.06	1.4362	0.6483	1.1008	159.201	157.719	258.121	3.721e+003	6.321	40.258	23.850	——	——	Aradur_60(30)
Average	10.05	1.4322	0.6427	1.0960	159.944	157.207	259.901	3.727e+003	6.307	40.414	24.015	——	——	
Std. Dev.	0.012	0.009	0.010	0.012	1.910	1.833	4.845	18.563	0.304	0.672	0.448	——	——	
CV	0.123	0.628	1.574	1.121	1.194	1.166	1.864	0.498	4.822	1.664	1.864	——	——	

LY556/F400/Aradur917/DY070, 8.6 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.05	1.3309	0.5544	0.9988	185.309	192.569	309.369	4.120e+003	4.808	44.239	28.586	9.239	18.857	Aradur_40b(1)
2	10.05	1.3993	0.6604	1.0931	167.551	177.275	264.515	4.121e+003	7.527	37.906	24.441	8.813	20.712	Aradur_40b(2)
3	10.05	1.3844	0.6560	1.0797	171.184	175.745	270.901	4.192e+003	7.764	37.592	25.031	8.743	21.047	Aradur_40b(3)
4	10.04	1.3969	0.6518	1.0897	168.038	176.128	265.807	4.117e+003	7.300	38.084	24.561	8.702	21.230	Aradur_40b(4)
5	10.04	1.3823	0.6350	1.0720	171.574	179.116	273.659	4.143e+003	6.718	39.163	25.286	8.875	20.409	Aradur_40b(5)
6	10.04	1.3756	0.6161	1.0602	173.260	178.735	278.758	4.109e+003	6.274	40.176	25.757	8.812	20.706	Aradur_40b(6)
7	10.04	1.3549	0.5824	1.0401	178.591	178.148	288.893	4.195e+003	6.381	40.271	26.694	8.790	20.804	Aradur_40b(7)
Average	10.05	1.3749	0.6223	1.0619	173.644	179.674	278.843	4.142e+003	6.682	39.633	25.765	8.854	20.538	
Std. Dev.	0.005	0.024	0.041	0.033	6.319	5.825	15.801	36.251	1.003	2.298	1.460	0.178	0.786	
CV	0.048	1.773	6.515	3.129	3.639	3.242	5.667	0.875	15.011	5.798	5.667	2.016	3.828	

LY556/F400/Aradur917/DY070, 12.3 vol% silica

Load-unload Result

SEQ	Fmax	hmax	hp	hr	HMT115	HMs	Hit	Eit	Cit	nit	HV*	Length	HT115	Data name
	[mN]	[um]	[um]	[um]	[N/mm2]	[N/mm2]	[N/mm2]	[N/mm2]	[%]	[%]		[um]		
1	10.04	1.3762	0.6450	1.0874	173.029	175.339	268.838	4.402e+003	7.510	36.338	24.841	8.430	22.612	Aradur_20(3)
2	10.05	1.2693	0.5183	0.9528	203.630	192.077	339.832	4.537e+003	4.042	43.947	31.400	8.862	20.485	Aradur_20(4)
3	10.04	1.3374	0.6028	1.0347	183.305	182.097	293.353	4.397e+003	5.859	39.588	27.106	8.513	22.183	Aradur_20(5)
4	10.05	1.3501	0.6124	1.0494	180.040	179.389	286.263	4.374e+003	6.158	39.040	26.451	8.468	22.444	Aradur_20(6)
5	10.04	1.3610	0.6318	1.0679	176.971	179.280	277.665	4.416e+003	6.824	37.312	25.656	8.728	21.100	Aradur_20(7)
6	10.05	1.3736	0.6498	1.0818	173.850	175.980	271.346	4.385e+003	7.204	36.675	25.072	8.795	20.791	Aradur_20(8)
Average	10.05	1.3446	0.6100	1.0457	181.804	180.694	289.549	4.418e+003	6.266	38.816	26.754	8.633	21.602	
Std. Dev.	0.005	0.040	0.048	0.050	11.361	6.101	26.287	59.815	1.254	2.827	2.429	0.185	0.919	
CV	0.053	2.948	7.945	4.741	6.249	3.377	9.079	1.354	20.009	7.283	9.079	2.140	4.255	