

Adaptation of COPRAS Algorithm for Reliable Epoxy Matrix Selection for Cryogenic Applications

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Submission: September 17, 2024; **Published:** November 07, 2024

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Abstract

This work aims to present an experimental framework for material selection purposes for different cryogenic applications that employ thermosetting epoxies. Overall, 9 commercially available epoxy candidates are chosen with varying viscosity and thermal properties. Each candidate is then cast into testing specimens according to ASTM D790 to be tested under three-point bending loading. Mechanical tests were performed both under room temperature and under a cryogenic environment to extract flexural modulus, strain, and strength values. The mechanical response of each candidate is explained case by case with the assistance of fracture surfaces obtained through cryogenic testing.

The last effort is to adapt the obtained mechanical response to well-accepted COPRAS material selection criteria to extract the best material candidates for prescribed engineering scenarios. Results suggested that 2 out of 9 candidates performed well both under RT and cryogenic environments. The presented work also aims to provide an example of the adaptation of COPRAS for material selection purposes via three-point bending tests considering the test variances frequently observed in the research conducted with thermosetting plastics.

Keywords: Epoxy-based adhesives; Material selection ; Cross-linking; Mechanical testing; Cryogenic Temperatures

Introduction

Cryogenic applications represent a frontier in modern engineering, spanning electronics, optics, and space. The high demands electronic, optical, and composite materials bring need of continuous development of material performance, particularly under cryogenic conditions where temperatures can reach to as low as -200°C. Among the various materials utilized in cryogenic environments, epoxy resins have arisen as multipurpose candidates due to their advantageous combination of mechanical strength, low outgassing characteristics, and ease of processing. In the cryogenic electronic applications, epoxy materials have a significant contribution on providing noise reduction, mechanical support and protection for electronic parts [1,2]. Furthermore, epoxy-based adhesives facilitate the bonding of superconducting

circuits and other cryogenic components, ensuring the reliability of cryoelectronic systems.

In optical applications, the demand for precision instruments operating at cryogenic temperatures has surged. Epoxy materials have been integral in the assembly of optical systems, particularly in space-borne telescopes and spectrographs, owing to their low thermal expansion, high optical transparency, and ease of bonding optical elements [3,4]. Cryogenic mechanical behavior studies generally focus on molecular structure effect. Hartwig [5] studied the low-temperature properties of epoxy resins and composites. The main factor that determines the low-temperature characteristics of epoxy resins is cross-linking. Cross-linking effect can be seen in thermal conductivity and thermal expansion

when the dominant phonon wavelengths of interchain modes and cross-link distances are in the same order for corresponding temperatures. On the other hand, cross-linking has no effect on the elastic mechanical characteristics or specific heat that may be represented by harmonic approximations. The structural independence of Poisson's ratio contradicts the theory that cross-linking has a significant impact on the free volume between chains. The fracture energy varies significantly according to cross-linking [5].

In the study conducted by Chen [6], the cryogenic performance of a resin system involving diglycidyl ether of bisphenol A (DGEBA) and acid anhydride hardener impregnation was tested. This resin system was blended with multifunctional epoxies, specifically tetraglycidyl dibenzmethyl diamine and triglycidyl benzylamine. The conclusion drawn from the study was that the multifunctional epoxy, particularly with amine groups in the molecular structure and at high stoichiometric ratios, cannot be effectively employed to enhance the typical cryogenic shear strength of the matrix. Nobelen [7] examined the impact of elastomer distribution on the mechanical behavior of carbon-epoxy composite laminates undergoing cryogenic cycling. The findings suggest that the presence of rubber particles enhances fracture toughness and reduces micro-crack density. However, it also leads to a decrease in interlaminar shear strength in the laminates subjected to cryogenic cycling. The author attributed this enhanced microcrack resistance to the elevated rubber concentration in the matrix.

Sawa [8] investigated various type of epoxies to establish a baseline for developing a molecular design applicable to cryogenic conditions. They utilized bisphenol-A (DGEBA) and tetraglycidyl metu-xylene diamine (TGMXDA) as the main constituents, with acid anhydride serving as the hardener. Hybrid material systems with different reactive groups were obtained to achieve higher fracture toughness values at cryogenic conditions. The results indicate that the crack tip stresses can be reduced with larger molecular weights across cross-linking. Fracture toughness dependent on intermolecular forces and stress relaxation at the crack tip. Ueki [9] expanded on the previous work of Sawa [8] by curing di-epoxies or multifunctional epoxies using various hardeners, such as anhydride, amine, or phenol. These were then blended with polycarbonate, carboxyl-terminated butadiene acrylonitrile copolymer or phenoxy. The results reveal that two-dimensional network-structured epoxies (phenoxy) exhibit high fracture toughness values even at cryogenic conditions.

Nasab [10] provided a review paper analyzing over 100 scientific articles, highlighted COPRAS and TOPSIS as effective methods for addressing material selection challenges. The study found that combining these MCDM techniques provides a simple and effective framework, while also considering the role of DEA in material selection, though it cannot fully replace MCDM approaches. Recent studies, such as the one by Ramachandran [11], have utilized the COPRAS (Complex Proportional Assessment) method to evaluate and rank CFRP composites based

on multiple performance criteria, demonstrating its effectiveness in composite material assessment. In another recent work, a novel 3D braided/unidirectional (3D/UD) hybrid composite was developed to enhance both in-plane (tensile, compression, bending) and interlaminar (shear, impact) properties for rail transit components.

Through the COPRAS method, the optimal configuration (UD6/3D/UD6) was identified, improving the comprehensive mechanical properties by 53.9% compared to pure 3D composites, offering a balanced solution for complex working conditions [12]. This article aims to present a framework study where thermosetting epoxy adhesives with different thermal and mechanical properties are tested for their performance under cryogenic conditions. In total 9 commercially available epoxy adhesives are tested for their flexural performance under room temperature and under cryogenic environments. Changes in the mechanical behavior of each epoxy type is underlined by the comparison of RT behavior and cryogenic behavior. Flexural stiffness, flexural strain and flexural strength values recorded for each sample case are then provided as beneficial properties for material selection algorithm COPRAS (Complex PROportional Assessment). The average variance on all measured properties is selected as a non-beneficial term to select the resin candidates with superior and reliable properties under both operating conditions.

Materials and Methods

Experimental Procedures

Epoxy systems can be categorized based on their usage area, viscosity, glass transition temperature, and hardener type. Consequently, six different epoxy systems were procured from two distinct manufacturers (EPO-TEK® Epoxy Technology, USA; Huntsman International LLC, UK) each possessing various thermomechanical properties. The selection of epoxy types was constrained to six due to cost considerations and accessibility. Details of the epoxy systems are presented in Table 1. Resin types suitable for electronic and optical system applications were chosen for their low glass transition temperature (T_g) and varying viscosity properties.

While the structural systems share the same resin, they differ in terms of hardener type and component numbers. The data sheets for the provided resin systems (Table 1) were taken basis during the preparation of resin mixtures. Prior to stoichiometric mixing, all samples were gently mixed for 15 minutes to enhance the homogeneity of resin/hardener/initiator components. These mixtures were then carefully introduced into pre-prepared silicon molds shaped according to ASTM D790-17 standard [13]. The nominal sample dimensions are 56x12.8x3.2 mm. Three-point bending tests were performed according to ASTM D790-17 [13].

These tests were performed under two conditions: room temperature and liquid nitrogen (LN₂) temperature, with 8

specimens for each case. This allowed for a comparison of the mechanical performance of epoxies at both room and cryogenic temperatures. The tests were carried out using the 10kN Shimadzu AGS-X universal testing machine (UTM). Cryogenic

three-point bending systems were implemented by submerging the specimens into liquid nitrogen, with UTM heads modified for the cryogenic submerged system.

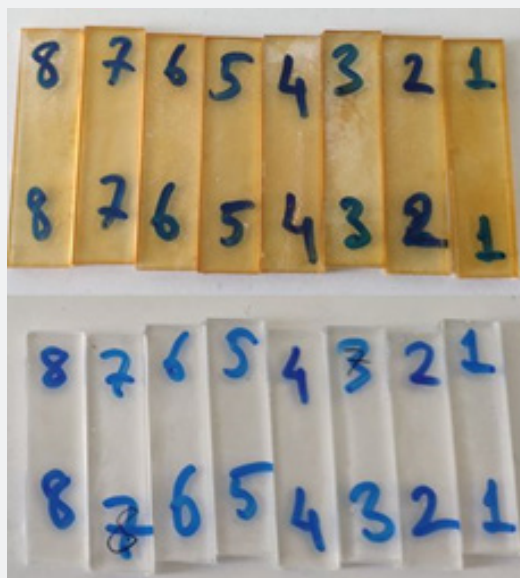


Figure 1: As manufactured three point bending specimens

These tests helped identify the cryogenic behavior of the epoxies. Through this testing method, mechanical properties such as flexural strength, flexural strain, and tangent modulus of elasticity were obtained according to ASTM D790-17 [13], for both room and cryogenic temperatures. Both cryogenic and normal three-point bending tests of cast epoxy samples were performed at a 5 mm/min crosshead displacement rate. Force vs. displacement values were recorded and later used in the calculation of maximum flexural strain (ϵ_{max}) and maximum flexural stress (σ_{max}) using the following relations:

$$\sigma_{max} = \frac{3FL}{2Bd^2} \quad (1)$$

$$\epsilon_{max} = \frac{6Dd}{L^2} \quad (2)$$

In these calculations, F represents the maximum force recorded in force-displacement curves, L is the half span length, B is the specimen width, d is the thickness of the sample and D is the deflection of the beam center [13]. For each test case, at least 8 specimens were tested. The bending tests in the cryogenic environment were carried out by designing a liquid nitrogen container for the test grips. To contain the liquid nitrogen in the container, insulation was provided by covering the container with XPS foam (Figure 2).

Results and Discussion

Low Tg Epoxy Adhesives (T7110-T7109-19)

When subjected to cryogenic temperatures, the T7109-19 resin specimens exhibited a significant increase in stiffness (+14800%), strength (+31%), and reduced flexibility (-82%) compared to room temperature tests (Table 2). Moreover, T7110 specimens displayed changes of (+3300%), (+390%), and (-96%), respectively (Table 2). This observation suggests that the T7109-19 resin is more susceptible to cryogenic embrittlement than the T7110, an expected result related to the restricted movement of the formed 3D network structures in extremely cold environments [5,14,15]. Notably, the stress-strain curves reveal distinctive low flexural stress values for T7109-19 (Figure 3).

In order to understand the significant differences between the flexural strength values of both resins, the fracture surfaces of the failed samples under cryogenic conditions were investigated. For each set of samples, three fracture surface images are provided to formulate a general comment on the type of failure. As seen in Figure 4, the fracture surfaces of T7109-19 and T7110 samples were very smooth, suggesting a very fast crack propagation during failure. This observation clearly highlights the amplified embrittlement effect for these resin types. One significant difference between the two resins was the increased amount

of air bubbles and shape inhomogeneities for T7109-19 resin. This is attributed to the extremely high viscosity of T7109-19 resin, causing manufacturing-related defects. Overall, the T7109-19 resin is challenging to process, susceptible to microcracking

under cryogenic conditions, and may hinder its use in cryogenic applications. However, these resin types provide the highest flexural modulus among other resin samples, which may be preferred.



Figure 2: Three-point bending test of T7109-19 epoxy system on RT (left) and T7110 epoxy system on LN2 (right).

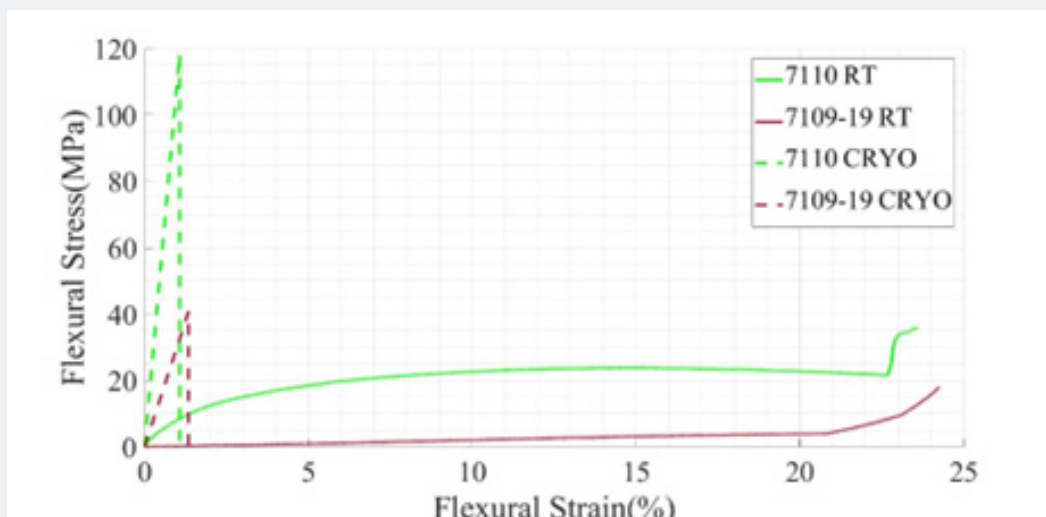


Figure 3: Representative flexural stress vs. flexural strain curves for T7110 and T7109-19 samples tested under RT and LN2 conditions.

Medium Tg Epoxy Adhesive Systems

Exposure to cryogenic temperatures resulted in notable changes in the mechanical properties of the 301-2 and 301-2FL specimens (Figure 5). In the case of 301-2FL, there was a considerable increase in stiffness (+240%), strength (+215%), and a decrease in flexibility (-26%) compared to the tests conducted at room temperature (Table 2). When compared with the previous

case (electronic grades),

The main difference between 301-2 and 301-2FL is their difference in Tg values. The highest Tg of 301-2 suggests that the macromolecular structure of this resin is harder to disturb. This situation is reflected in room temperature flexural modulus values of both resins, with 301-302 having a higher one (Figure 5).

Table 1: Selected Epoxy Adhesive Candidates and Characteristics.

Epoxy System	Supplier	Hardener Type and Mix Ratio by Weight	Tested Tg	Curing Cycle	Viscosity (23°C - 100rpm)
301-2	EPOTEK	Amine (100:35)	67	80°C - 3h	225-425cPs
301-2FL	EPOTEK	Amine (100:35)	40	80°C - 3h	100-200cPs
T7110	EPOTEK	Amine (10:1)	24	80°C - 2h	1400-2200cPs
T7109-19	EPOTEK	Amine (100:15)	15	80°C - 2h	40000-70000cPs
1565 / 1971 / 700	CET Composites	Anhydride (100:90:0.5-2)	101.1	90°C - 3h+ 90°C - 4h+ 8h 120°C	600-900 cPs
11546 / 13447	CET Composites	Amine (100:26)	109.3	80°C - 1h+ 120°C - 4h	350-450 cPs
105025 / 105025	CET Composites	Polyamines (100:38)	75.7	23°C - 1d + 90°C - 15h 50°C	500-700 cPs
1564-3474	HUNTSMAN	Amine (100:26)	118	80°C - 1h+ 120°C - 4h	500-700cPs
1564-917-960	HUNTSMAN	Anhydride (100:98:3)	107	80°C - 4h + 120°C - 4h	350-450cPs

Table 2: Mechanical properties measured for each candidate and weight of each property employed in COPRAS algorithm. v. denotes the % variance in each measurement

Type Condition	B		B		B		NB	B		B		B		NB
	RT							CRYO						
	σ_{flex}	v	ϵ_{flex}	v	E_{flex}	V	Avg v.	σ_{flex}	v	ϵ_{flex}	v	E_{flex}	v	Avg v.
Epoxy System	MPa	%	%	%	MPa	%	%	MPa	%	%	%	MPa	%	%
301-2	90	0.07	7.4	0.23	2674	0.18	0.16	157	0.13	4.6	0.24	6298	0.15	0.17
301-2FL	59	0.02	7.7	0.03	1984	0.23	0.09	185	0.14	5.7	0.14	6745	0.1	0.12
T7110	23	0.04	22.3	0.03	365	0.13	0.07	116	0.06	1	0.2	12396	0.09	0.12
T7109-19	33	0.23	7.3	0.01	31	0.23	0.16	42	0.24	1.3	0.46	4622	0.28	0.33
1565 / 1971 / 700	102	0.17	9.9	0.26	2109	0.16	0.2	73	0.3	2.6	0.27	4180	0.23	0.27
11546 / 13447	100	0.04	13.2	0.08	765	0.08	0.07	146	0.18	4.5	0.24	3180	0.03	0.15
105025 / 105025	83	0.11	8.4	0.19	1012	0.12	0.14	139	0.23	3.8	0.24	3643	0.02	0.16
1564-3474	99	0.03	12.4	0.09	1853	0.13	0.08	136	0.28	4.4	0.25	5741	0.22	0.25
1564-917-960	52	0.33	3.4	0.41	2596	0.18	0.31	48	0.1	1.5	0.13	4445	0.12	0.12
Weights (Modulus Oriented)	0.2		0.2		0.4		0.2	0.2		0.2		0.4		0.2
Weights (Deformation Oriented)	0.2		0.4		0.2		0.2	0.2		0.4		0.2		0.2
Weights (Strength Oriented)	0.4		0.2		0.2		0.2	0.4		0.2		0.2		0.2

Table 3: Material rankings obtained from COPRAS algorithm maximizing each property with minimum variance.

Material Ranking	Eflex RT	ϵ flex RT	σ flex RT	Eflex CRYO	ϵ flex CRYO	σ flex CRYO
301-2	2	5	4	3	3	3
301-2FL	4	6	5	2	1	1
T7110	7	1	6	1	2	2
T7109-19	9	9	9	9	9	9
1565 / 1971 / 700	3	4	3	8	8	8
11546 / 13447	5	3	2	4	4	4
105025	8	7	7	6	5	5
1564-3474	1	2	1	7	7	7
1564-917-960	6	8	8	5	6	6

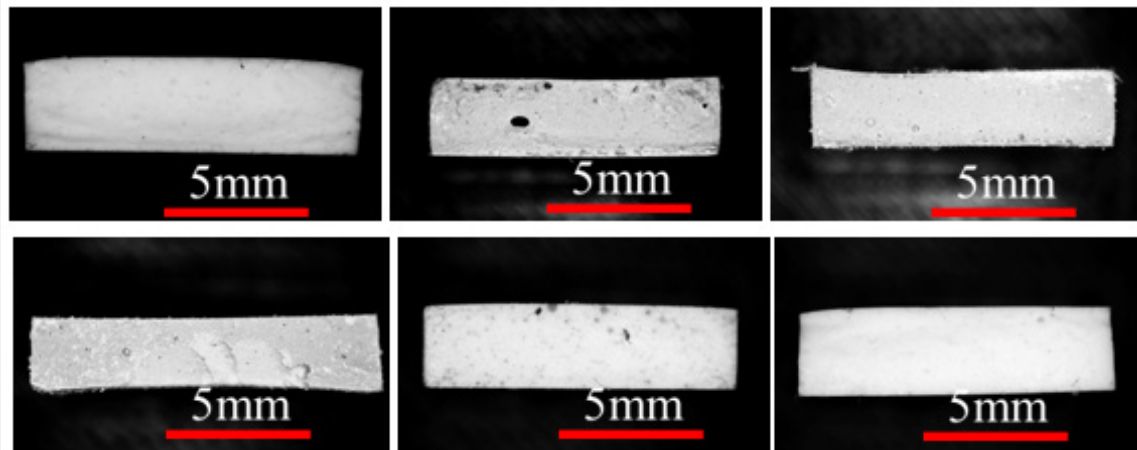


Figure 4: Fracture surfaces of selected samples from T7109-19 (top row) and T7110.

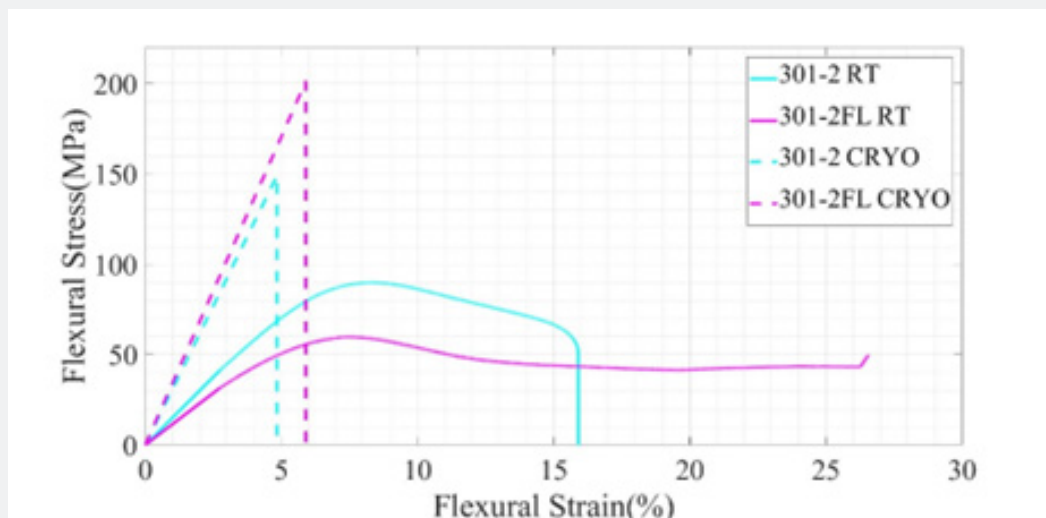


Figure 5: Representative flexural stress vs. flexural strain curves for 301-2 and 301-2FL samples tested under RT and LN2 conditions.

When subjected to cryogenic temperatures, 301-2 specimens exhibited relatively smaller changes: +136% in flexural modulus, +74% in strength, and -38% in flexural strain (Table 2). In comparison with 301-2FL, 301-2 resin experienced a significant reduction in flexural strain and did not show similar increases in strength and modulus. The rationale behind different changes in

the mechanical response has been evaluated by comparing the fracture surfaces of tested samples. As depicted in Figure 6, the failure surfaces of the investigated samples are divided into two sections, namely the compression and tension regimes, due to the applied bending load.

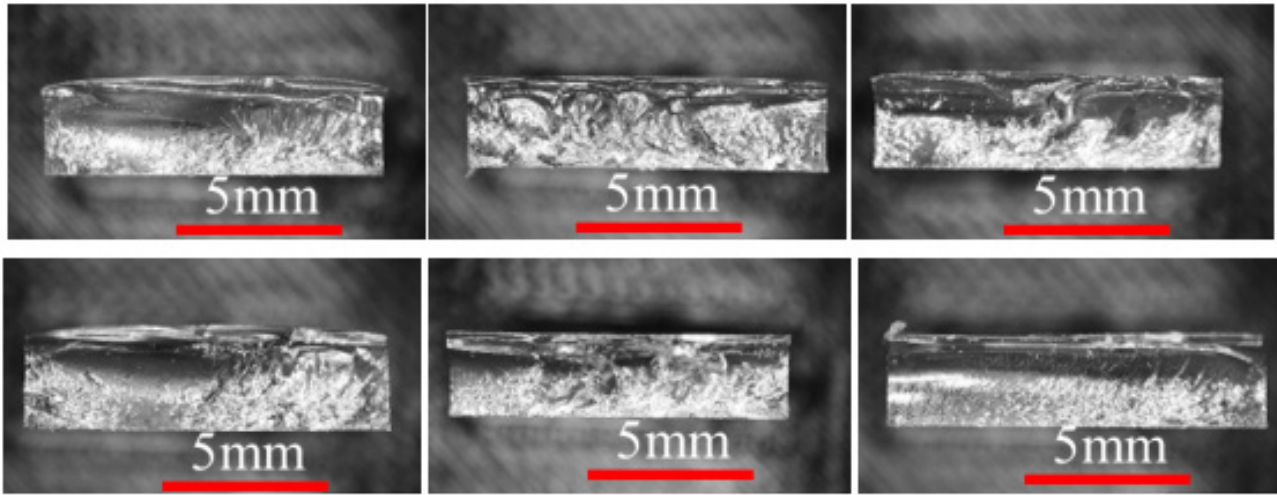


Figure 6: Fracture surfaces of selected samples from 301-2FL (top row) and 301-2 (bottom row).

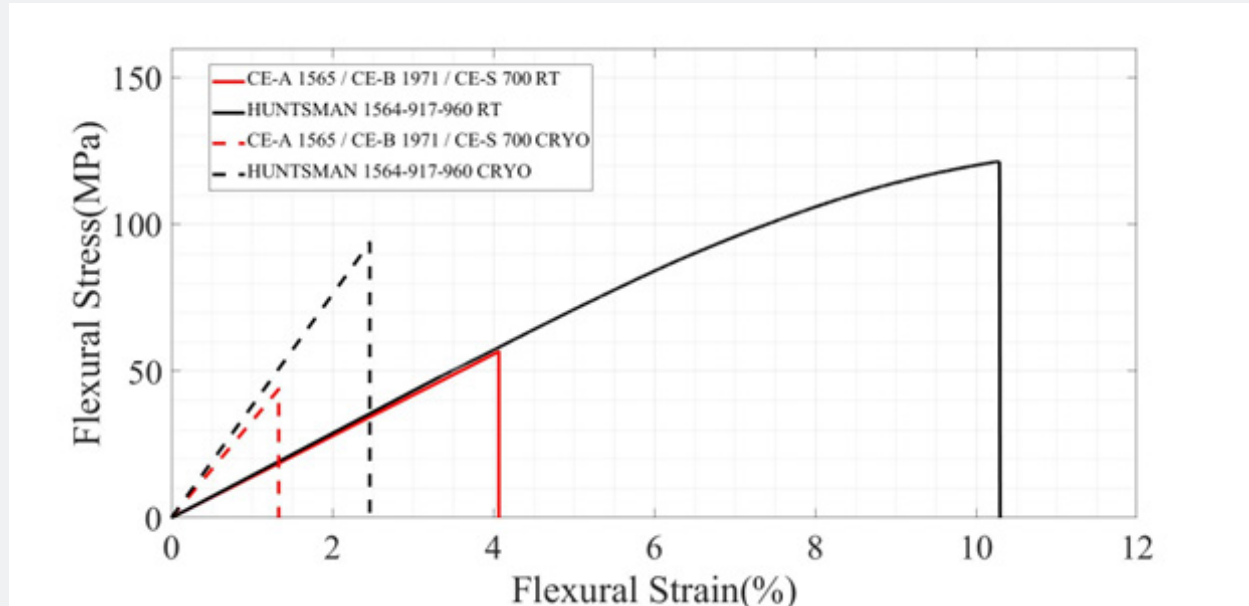


Figure 7: Representative flexural stress vs. flexural strain curves for 3 component structural resin systems tested under RT and LN2 conditions.

The tensile failure regime is typically characterized by the appearance of mirror-mist-hackle formations [16]. In the provided micrographs, this region is the bottom part, where

linear hackle lines originating from mirror regions are clearly distinguishable. The compressive regime, on the other hand, is the top part, where a very fast crack propagation from the top of

the surface towards the tensile failure regime is present. Due to the nature of the bending test, such features usually coincide in the middle of the specimen (neutral axis). One major difference between the top (301-2FL) and the bottom (301-2) is the location where the tensile regime and compressive regime coincide. In the case of 301-2FL, such a location is clearly visible for all of the samples, which is not the case in 301-2. For 301-2 samples, the tensile failure regime is highly dominant, suggesting that its microstructure is more prone to generate tensile micro-cracks, which can decrease the flexural strain.

High Tg Epoxy Adhesive Systems

Three-component structural grade epoxy systems (1565 / 1971 / 700 and 1564-917-960): The comparison of these two epoxy systems must be approached differently than other systems due to their distinct curing mechanism involving three components. In three-component epoxy systems, the curing reaction is typically initiated by an initiator at an elevated temperature [17]. Anhydrides are commonly used as bulk hardener agents, becoming reactive beyond 120°C [18]. The

crosslinking reaction is usually initiated by a smaller and more reactive amine-based component [17].

Given the three-component nature, the microstructure of these resins is more dependent on manufacturing conditions than two-component systems. Curing enthalpies and ideal maximum Tg values are similar for both resin systems. However, when subjected to manufacturing conditions, it becomes evident that the 1564-917-960 system has a significantly lower Tg compared to the CE-1565 system, suggesting that the 3D network structure of the former is rather loosely connected. In terms of mechanical performance at room temperature, both resin systems exhibited a similar flexural modulus, whereas 1564-917-960 was significantly less strong and less flexible. When tested under cryogenic conditions (Figure 7), it is evident that the system drastically suffers from chain immobility, resulting in a reduction in strength, which is dissimilar to previously reported cases. In contrast, the CE system is less affected in terms of flexural strength while losing flexibility, underscoring an important mechanical response that can be achieved from three-component epoxy systems (Figure 7).

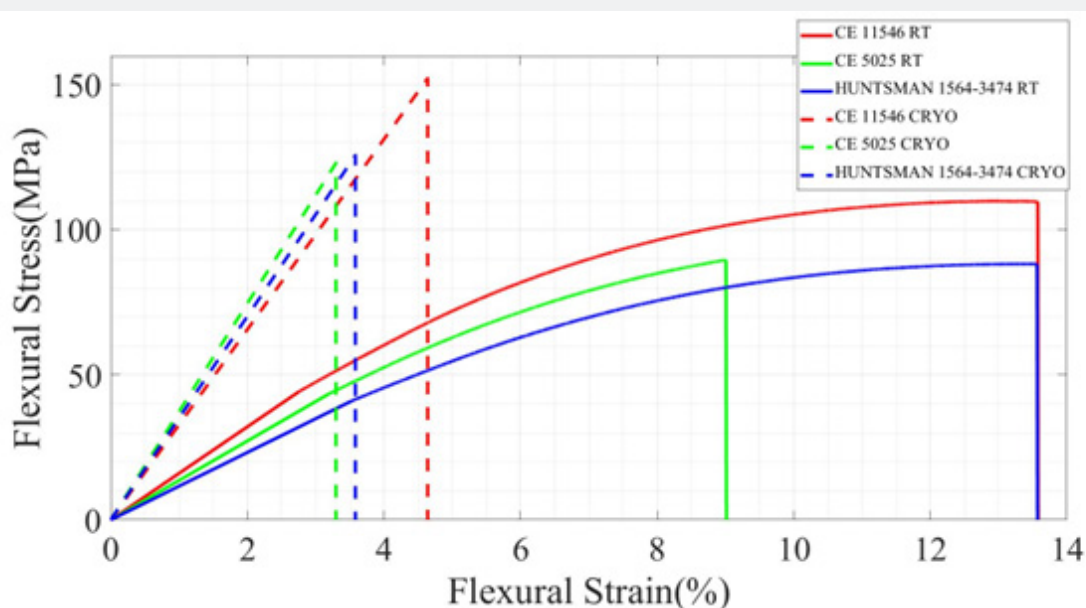


Figure 8: Representative flexural stress vs. flexural strain curves for two component structural resin systems tested under RT and LN2 condition.

Two-component structural grade epoxy systems (11546/13447, 1564-3474 and 105025 / 105025): The 11546/CE-B 13447 and 1564-3474 systems exhibit similar behavior under thermal analysis, sharing identical curing profiles. Furthermore, the mechanical behaviors of the two systems are consistent under both room and cryogenic conditions (Figure 9). In cryogenic conditions, strength increased by around 40%, flexural modulus increased by around 250%, and elongation capability decreased by around 65%. The observed brittle

behavior in cryogenic conditions is evident in these epoxies. Overall, these two epoxy systems demonstrate promising results in cryogenic bending tests. This system incorporates several different amine hardeners to provide curing capability even at low curing temperatures, allowing curing in a wide range of temperatures from 23°C to 130°C. The behavior of this epoxy changes drastically according to the curing temperature cycle. The mechanical behavior of this epoxy is average for both room and cryogenic conditions when compared to other tested epoxies.

Changes in mechanical properties when subjected to cryogenic conditions are around +67% in strength, -55% in elongation, and 260% in flexural modulus. The mechanical behavior of the 105025system is illustrated in (Figure 8).

Application of COPRAS and material selection

Complex Proportional Assessment (COPRAS) algorithm for material selection criteria was applied per described in [19-21]. Beneficial terms are labeled as "B" whereas non-beneficial terms are highlighted as NB. All of the measured material properties under room temperature and cryogenic environment are considered as beneficial terms. The average variance on the performed experimental efforts is considered as a non-beneficial term. The weight of each beneficial term is tuned according to objective property that is to be maximized (Table 3).

Resin elasticity is one of the primary material property that is searched for in any structural engineering design [22,23]. The elasticity of the employed resin type also affects the anisotropic response in reinforced composite applications [24,25]. From this perspective the maximization of the matrix stiffness is typically sought. When ranked according to their Eflex at room temperature 1564-3474 (High Tg), 301-2 (Medium Tg) and three component 1565-1971-100 (High Tg) systems had the highest ranking among other candidates. However, when subjected to cryogenic temperatures this ranking drastically changes especially for high Tg systems like 1564-3474 (High Tg) and 1565-1971-100 (High Tg) that ranked 7th and 8th respectively. This observation suggests that high Tg systems with relatively low viscosity are prone to micro-structural changes appearing under cold environment. This finding is further supported with their low ranking at oflex CRYO and eflex CRYO rankings.

On the contrary 301-2 system found to perform well under both conditions and provides more reliability on experimental errors for stiffness based designs. Under cryogenic environment, the reduction in material deformation is one of the major concerns. Hence maximum strain bearing capacity is often considered as a desired property especially for the investigated thermosetting materials that goes to serious embrittlement under cryogenic environments [14,26,27]. 301-2-FL, T7110 and 301-2 types appeared as the best candidates for maximum deformation with minimum experimental error under cryogenic environment.

Among these candidates, the behavior of T7110 resin presents an important point for low Tg resins that are commonly employed in electronics industries. Different than structural applications in composite materials, in electronics such resins are applied in very small amounts which makes structural irregularities that may cause strength reductions less important. From this perspective, experimental reliability and cryogenic performance of T7110 resin could be desirable. Another point of interest in the

material rankings according to eflex CRYO is that all of the high Tg structural resin types has low rankings. This observation suggests for the need of advanced resin solutions that are less susceptible to cryogenics environments [28].

In the case of strength based evaluation it should be noted that rankings almost coincides with the eflex case. This is an expected result since failure analysis performed suggested for significant tensile cracking in the samples especially under cryogenic environments. The root cause of this cracking is known to be reduction of micro-structure mobility hence the strain development in the material [9,14,15]. A similar observation can be made for the comparison of oflex RT and oflex cryogenics rankings. This observation suggests that the root-cause of failure under three point bending does not effected from the testing environment and if a resin is susceptible to cracking under RT it is also susceptible to cracking under cryogenic environment.

Conclusion

Flexural performance of 9 different off the shelf thermosetting epoxy candidates are evaluated by room temperature and cryogenic environments. Presented results suggested that cryogenic embrittlement reducing the deformation capabilities of epoxide resins heavily depends on the type of resin and their thermal properties. Strength of the resins on the other hand relies on the generation of micro-cracks and ability of resin system to endure such micro-cracking. Material selection via COPRAS enabled for effective material selection considering the contribution of experimental errors combined with mechanical performance.

Selection process suggested that even though low Tg resins are found be non-desired candidates their cryogenic properties may be of advantage as long as the high viscosity problem is overcome. Best cryogenic properties are found to be provided by medium Tg resins that neither suffers from extreme embrittlement (as in the case of low Tg resins) nor from the high crosslinking that disables the cryogenic adaptation (as in the case of high Tg resins). Hence a candidate for electronics applications where small amount of adhesive is used and a candidate for large scale structural applications has been selected for further engineering efforts. In overall, presented framework also provided an example for adaptation of COPRAS via three point testing efforts that is more quick in terms of experimentation.

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DOI: [10.19080/AJOP.2024.06.555690](https://doi.org/10.19080/AJOP.2024.06.555690)

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