Review Article

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Epoxy Coatings at Cryogenic Temperatures with and without Nano-fillers – A Review

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ABSTRACT

The study of cryogenic coatings has garnered substantial interest from engineers due to their wide range of applications, particularly in the aerospace and chemical industries. Polymers with their advantages of good mechanical properties and low weight are being considered for coating on both metallic and non-metallic substrates working at different ranges of temperatures, from high to very low temperatures. Very low temperatures, usually below -150°C to absolute zero, are termed cryogenic temperatures. This report compiles the studies on the tribological and mechanical properties of polymer coatings developed for cryogenic environments. The primary focus is on the epoxy resin (DGEBA) and its enhanced variants incorporating additives like graphene nanoplatelets (GNP), graphene and carbon nanotubes (CNT).

Keywords: Polymers; Epoxy; Coating; Cryogenic temperature; Nanoparticles.

1. INTRODUCTION

Cryogenic temperature refers to the temperatures below -150 ºC (123.15K) where gases like nitrogen, hydrogen, and helium can be stored in liquid phase. Cryogenics is the science that deals with the effects of these temperatures on materials working at these temperatures (Zohuri *et al.* 2018). There are many applications in fields like engineering, aerospace, construction, and medical where the materials work in extremely low temperatures (Weisend *et al*. 1998). In the case of space applications, satellites, and rockets, these temperatures are experienced in the outer space. Low temperatures can also be observed in places like Antarctica, the Arctic, and nearby areas. Rocket and missile fuel, liquefied gas storage and transportation (Aziz *et al.* 2021), cryosurgeries (Rubinsky *et al*. 2000), advancements in quantum computing (Oyeniran *et al*. 2023), superconducting technologies (Ganni *et al*. 2012), and medical technologies (Jha, 2006) are some specific applications of cryogenics.

Materials like rubber, plastics, and metals undergo a change in their properties at low temperatures. Rubber becomes more rigid and brittle at temperatures below zero degrees centigrade. It also enters the glass transition phase. However, plastics show an increase in strength at low temperatures, but their ductility decreases. Some metals show a ductile to brittle transition at these temperatures. It has been observed that metals with face-centered cubic structures (Lead, Aluminum, Gold, Austenitic stainless steel, etc.) do not undergo this transition at any temperature and retain their ductility at lower temperatures. Metals with body-centered cubic (BCC) and hexagonal close-packed (HCP) structures (except high-purity Titanium) exhibit ductile to brittle transition at low temperatures (Berto and Lazzarin, 2014). Using these materials without proper caution can lead to failure of the material, which leads to the failure of the component or system, which is catastrophic in some cases.

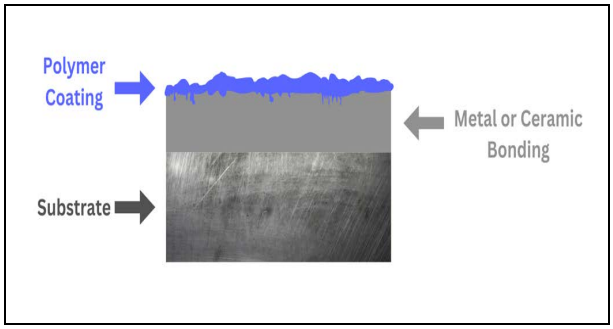


Fig. 1: Substrate coated with polymer coating

One of the solutions to overcome this problem and reduce the effect of very low temperatures on the performance of the metals/composites is applying polymer coatings (Chhipa *et al*. 2024). Polymer coating means applying a layer of polymer on any substance that is acting as a substrate (El-Sherif *et al*. 2017) as shown in Fig. 1. These are done by using various coating methods like Solution coating (Joung *et al*. 1982), Dip coating (Lu *et al*. 1997), Spray coating (Susanna *et al* 2011), Spin coating (Hall *et al*. 1998), and Doctor Blade technique (Chang *et al.* 2009).

A wide variety of polymers like different grades of epoxies, urethanes, Teflon, acrylics and cellulose can be used as coatings based on the working conditions of the substrate (Wang *et al.* 1994). These coatings give an advantage over metals as they have high strength and low weight ratio compared to metals (Sethi *et al.* 2013). These coatings offer protection from wear and corrosion of the surface of metals at normal temperatures. However, not all polymer coatings are effective at cryogenic temperatures. Research has shown that thermoset polymers are comparatively strong and stiff and show good dimensional stability compared to thermoplastics (Madhav *et al.* 2019). Among the polymers used for structural applications, epoxy has shown good mechanical properties, both as a matrix and a coating. They commonly protect components at cryogenic temperatures like spacecraft, cryogenic tanks and satellites. Also, these epoxy coatings are used in tanks, pipelines, and other storage vessels that transport or contain cryogenic fluids.

2. EPOXY MICROSTRUCTURE AND PROPERTIES

Epoxy coatings are widely recognized for their strength, durability, and adaptability across various industries. These coatings are created through a two-component system, combining resin and hardeners to form a robust and chemically resistant layer. They are extensively used to protect surfaces such as metal, concrete, and composite materials, providing excellent adhesion, chemical resistance, and mechanical stability. Typical applications include corrosion prevention, waterproofing, and enhancing surface finishes. 'Epoxy' refers to a chemical group consisting of two carbon atoms and one oxygen atom bonded together. The simplest structure of epoxy is a three–member ring shown in Fig. 2 (Park *et al.* 2011).

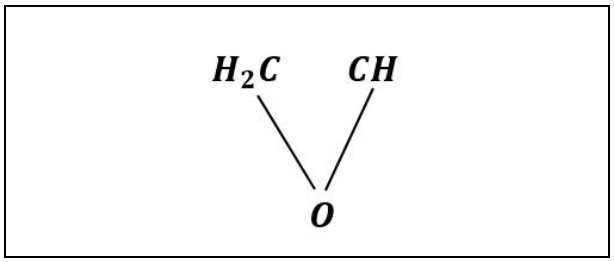


Fig. 2: Simplified chemical structure of alpha epoxy

Table 1. Typical properties of a neat epoxy system

Property	Value
Specific Gravity	1.1 -1.2
Viscosity (m Pas)	9000 – 12000
Tensile Strength (MPa)	70 -80
Flexural Strength (MPa)	140 -150
Tensile Modulus (GPa)	4.0 - 4.8
Expansion Coefficient (10^{-6} K^{-1})	45 - 55

Table 1 shows the properties of neat epoxy resin (L12 grade, ATUL Make) at room temperature. Epoxy resins typically form a cross-linked structure after curing. Hence, this resin is generally used as a matrix to make composite laminates using different manufacturing techniques like filament winding, pultrusion, etc. In some epoxy systems, a phase separation phenomenon can be observed during curing due to adding certain modifiers like styrene–butadiene–styrene (SBS). As the epoxy cures, the components segregate into distinct regions, resulting in different microstructure morphologies like spherical particles, layers, co-continuous structures, etc. This phase separation phenomenon improves properties of epoxy like fracture toughness, impact resistance, and thermal stability based on the modifier used. (Jiang *et al.* 2018).

In general, epoxies are classified as three types which are bisphenol – A, bisphenol – F and novolac grades (Bobby *et al.* 2017). Table 2 shows the epoxy formulations after mixing them with amines. Amines are the solidifier components that drive the curing of epoxy.

Table 2. Monomers to synthesize different epoxies (Studer *et al.* 2024)

Name	Abbreviations	F (Functionality)	Epoxy / Amine Equivalent (*g/mol*)
bisphenol: A diglycidyl ether	DGEBA	2	172
meta phenylene diamine	MPD	4	27
n-octylamine	OA	2	64.6
n-butylamine	BA	2	36.6
tert-octylamine	t-OA	2	64.6
2-heptylamine	2-HA	2	57.6

Among these epoxy types, bisphenol – A is the most used resin for coating applications owing to its good adhesion, chemical and wear properties (Durig *et al.* 2000, Pulikkalparambil *et al.* 2018). The chemical structure of Diglycidylether of bisphenol-A abbreviated as DGEBA, is shown in Fig. 3, obtained by reacting the bisphenol – A with epichlorohydrin.

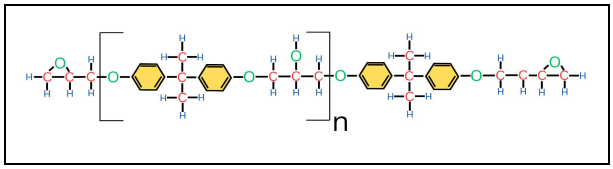


Fig. 3: Chemical structure of DGEBA

Epoxy coating systems are classified as liquid-based epoxy (LBE) systems in which epoxy is in liquid form and fusion bonded epoxy (FBE) coating systems in which epoxy is in powder form. Their tribological properties can assess the performance of coatings. It was observed that the pristine epoxy, when modified with

other polymers or fluorinated and siloxane compounds, impart good tribological properties for the coating (Yan *et al*. 2013, Kim *et al.* 2011). Another method studied by researchers to improve the coating performance is by modifying epoxy with polyepoxides containing silicon and bioactive coordination compounds. These improve the anti-friction coats' scratch resistance (Barbakadze *et al*. 2018). Dan Liu *et al.* (Liu *et al.* 2015) studied the effect of curing agents on the tribological properties of epoxy coatings. Three curing agents were taken: benzene shaped m – phenylenediamine (m-PDA), ring with side chain-shaped isophorone diamine (IPDA), and chain-shaped diethylenetriamine (DETA) respectively, of which using DETA resulted in obtaining the lowest friction coefficient. Anwar *et al.* 2024 have comprehensively reviewed the usage dynamics of high-quality epoxy resin for corrosion applications.

The performance of the polymer coatings can be analyzed by different types of tests (listed in Table 3) based on the application intended.

Table 3. Various tests for evaluation of the performance of polymer coatings

Property	Method	Standard	Reference
Adhesion	Tape Test	ASTM D3359	(Sabau *et al.* 2020)
	Cross Hatch Test	ASTM D3359	(Wang *et al.* 2022)
	Pull - off Adhesion Test	ASTM D4541	(Liddel *et al.* 2023)
Abrasion Resistance	Taber Abrasion Resistance	ASTM D1044, ASTM D4060	(Turri *et al.* 2010)
	Pin–on-Disc	ASTM D4060	(Jakab *et al.* 2021)
Impact Resistance	Falling Weight Test	ASTM D2794	(Zhang *et al.* 2022)
Hardness	Pencil Hardness	ASTM D3363	(Molera and Sue, 2019)
	Shore Hardness	ASTM D2240	(Yan *et al.* 2020)
Scratch Resistance	Scratch tester	ASTM D7027	(Molera and Sue, 2019)
Flexibility	Conical Mandrel Bend Test	ASTM D522	(Deyab *et al.* 2021)
Thickness	Non – destructive testing	ASTM D6132-13	(Zargarnezhad *et al.* 2024)
Chemical Testing	Salt Spray Test	ASTM B117	(Muhammad et al. 2019)
	UV exposure test	ASTMG154 – 155	(Kamde et al. 2020)
	Chemical Resistance	ASTM G85	(Bahrami *et al.* 2017)
	Moisture Resistance	ASTM D570	(Acikbas *et al*. 2024)
Thermal	Differential Scanning Calorimetry (DSC)	-	(Trzaskowska 2018)
	Thermal gravimetric analysis (TGA)	-	(Parra *et al*. 2002)
	Dynamic Mechanical Thermal Analysis (DMTA)	-	(Molera and Sue, 2019)
	Vicat Softening Point	-	(Kiattipornpithak *et al*. 2021)
	Heat Distortion Temperature (HDT)	-	(Sapinski *et al*. 2024)
Microsctru- cture / Defects	Fluorescent Penetrant Inspection (FPI)	-	(Dhole *et al.* 2020)

	Scanning Electron Microscopy (SEM)	-	(Beaugendre *et al.* 2019)
	X-Ray Fluorescence (XRF)	-	(Vandi *et al.* 2013)
	Electrochemi-cal Impedance Spectroscopy (EIS)	-	(Wang *et al.* 2023)

To measure the adhesive and cohesive properties of the coatings, different types of peel tests are performed. Wei *et al.* 2020 has given a comprehensive review of different methods to improve the bonding properties of the epoxy coatings with the substrates and the methods to be used to study the adhesive behaviour of epoxy. It was also found that the adhesive test results are very sensitive to the procedural discrepancies. Failure mode analysis and statistical analysis of the results will provide an accurate understanding of the substrate and coating bonding (Khambampati *et al*. 2023). The wear performance of the coatings in during the function / working of the component is studied using Abrasion test, the most popular one being the pin – on – disc test. Apart from the abrasion tests mentioned in the Table, falling sand abrasion test (amount of sand required to remove the coating is measured as abrasive resistance) (Wang *et al.* 2023), wear resistance test (done by subjecting the surface to sliding abrasive particles), abrasive jet erosion test (done by subjecting the coated surface to a high-speed jet of abrasive particles) are performed. Apart from the conventional techniques listed in the Table, many advanced techniques are also available for the study of performance of epoxy coatings. One of the techniques is LaSAT, acronym for Laser shock adhesion testing uses high intensity laser shock waves onto the coating surface to evaluate its strength. The critical value of the laser energy required to cause delamination is taken as the measure of the adhesion strength. One of the main features of this test is to identify different coating and substrate adhesion strength levels (Arrigoni *et al.* 2008, Kimoto *et al.* 2021, Sma *et al*. 2024).

To measure the free volume and porosity in epoxy coatings, Positron annihilation lifetime spectroscopy (PALS) technique, is used. This is an effective technique for defect characterization of coatings (Macqueen *et al*. 1996, Wang *et al*. 2010, Hughes *et al*. 2012). An advanced technique to study the porosity, defects, free volume etc. is Infrared Thermography. It is a non- destructive method which monitors the coating quality by analyzing the thermal radiation maps obtained after thermal exposure of the coating surface (Goffin *et al.* 2020). Laser scanning Confocal Microscopy (LSCM) is another non-destructive technique to generate 3D images of the coating surface topography and to identify the defects (Sung *et al.* 2004, Hu *et al.* 2010).

3. EPOXY COATINGS AT CRYOGENIC TEMPERATURES

Epoxy (Chen *et al.* 2021), Teflon (*Ferrick et al*. 2008), Polytetrafluoroethylene (PTFE) (Burris *et al.* 2008), Polyethylene (PE) (Lan *et al.* 2018), Ultrahigh molecular weight polyethylene (UHMWPE) (Deenoi and Dechjarern, 2019) and Polychlorotrifluoroethylene (PCTFE) (Cui *et al*. 2023) are some of the polymers used as coatings at cryogenic temperatures. Table 4 shows the properties of some such polymers at cryogenic temperature from the literature.

Table 4. Properties of various coatings for cryogenic applications

Materials	Compression Strength *(MPa)*	Melting temperature *(°C)*	Density *(g/cm³)*	Dynamic viscosity *(Pa. s)*	References
Polyethylene-engineered cementitious composite (PE-ECC)	59.1	140	0.97	-	(Mahmoudi *et al*. 2022; Lingfei *et al*. 2024)
UHMWPE	12(HB26) 8(HB50) 340(SK76)	130	0.93	10^8	(Attwood *et al.* 2015; Li *et al.* 2019)
Polyethylene Terephthalate, PET	87	243	1.38	-	(Ichetaonye *et al*. 2023)
Polystyrene, PS	-	175	1.055	5×10^4	(Abdel-Goad *et al.* 2018)
Epoxy	4 -8	80-120	1.18	10 -100	(Samad *et al*. 2017)

Among these polymer coatings, epoxies provide a wide range of applications, as discussed in previous sections, and the same applies at cryogenic temperatures. Alberts *et al*. 2017, tested the tensile and shear strength of 15 different grades of epoxy for bonding multilayer insulation standoffs to cryogenic storage tanks. Cryobond 621, Masterbond EP29LPSP and Scotchweld 2216 have demonstrated best results among those tested.

Epoxies could withstand 18Kg of mass with zero failures for all the metal surfaces tested. Pascal *et al.* 2024, developed low-viscosity epoxy systems by adding short–chain alkylamines with DGEBA + metaphenylene diamine (MPD). The different types of chain extenders used are n-octylamine (OA), n-butylamine (BA), tert-octylamine(t-OA), and 2 – heptylamine (2 – HA). Fig. 4 indicates significant improvement in epoxy fracture toughness values upon addition of short- chain alkylamines.

Also, adding extra chains reduces the glass transition temperatures and cross-linking density, as seen in Fig. 5. This property helps minimize the thermal stress buildup while reducing the temperature to a cryogenic level. Gravit *et al.* 2021 have applied epoxy coatings for liquified LNG spillage protection of steel structures. The epoxy coatings proved effective in delaying the fire exposure by reaching the critical temperature after 120 min in case of a cryogenic exposure. Epoxy systems are also used to coat sensors like Fiber Bragg grating (FBG) sensors. Cai *et al.* 2021 used a predictive model to estimate the performance of epoxy coated and bare FBG sensors at cryogenic temperatures. The results have shown an improvement in linear characteristics and sensitivity as compared to the bare sensors. Ren *et al.* 2024 an experimental study on epoxy-coated FBGs at cryogenic temperatures varying the size of the sensors and thickness of coatings. Comparing wavelength responses of FBGs at temperatures ranging from 25°C to -180°C, epoxy-coated FBGs demonstrated a sensitivity of 48pm/°C, ten times more than bare sensors (Sampath *et al.* 2018). In the case of temperature sensors, epoxy polymer coatings have shown a sensitivity of -0.024 dB/K, and a good repeatability, stable robust sensing capability in the temperature range of 90 -298K (Sampath *et al.* 2023). In the case of large aerospace vehicles, epoxy has been used as an internal coating to store propellants at very low temperatures (Liu *et al.* 2021).

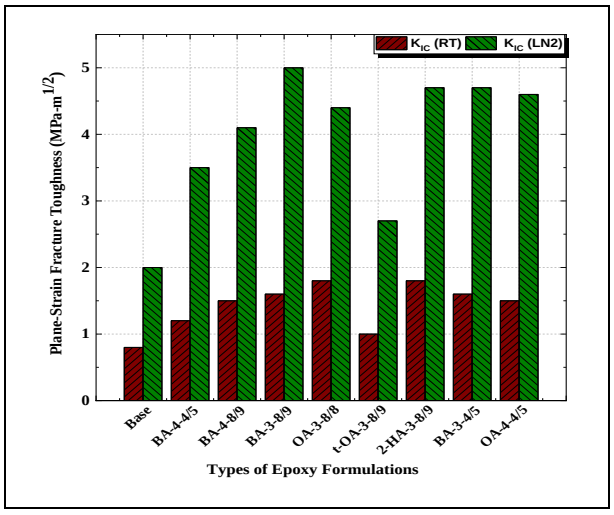


Fig. 4: Plane – strain fracture toughness values at RT and LN2 for different epoxy formulations (Schwegler *et al.* 2024)

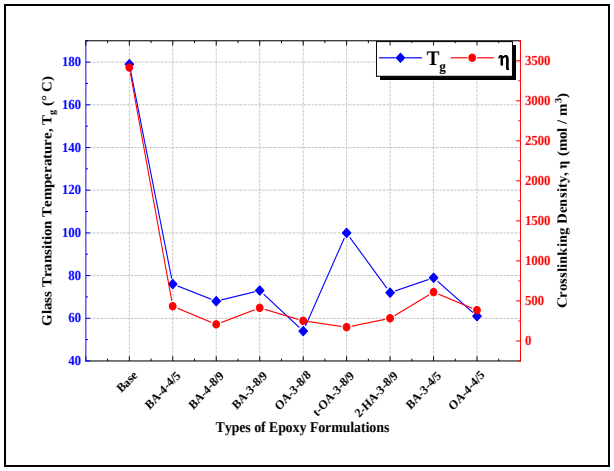


Fig. 5: Glass transition temperature and crosslinking density for different epoxy formulations (Studer *et al.* 2024)

3.1 Epoxy with Fillers

Micro/nano – scale reinforcements are added to the neat epoxy resin to improve the performance of epoxy coating systems at cryogenic temperatures. Carbon-based fillers like Graphene, Graphene Oxide (GO), Carbon nanotubes (CNT), fullerenes, etc., are the most researched combinations of epoxy (Marrivada *et al.* 2022, Marrivada *et al.* 2023, Ojha *et al.* 2019, Huang *et al.* 2002). Fig. 6 shows the effect of addition of commonly used carbon – based nano fillers to epoxy on its tensile strength at room and cryogenic temperatures.

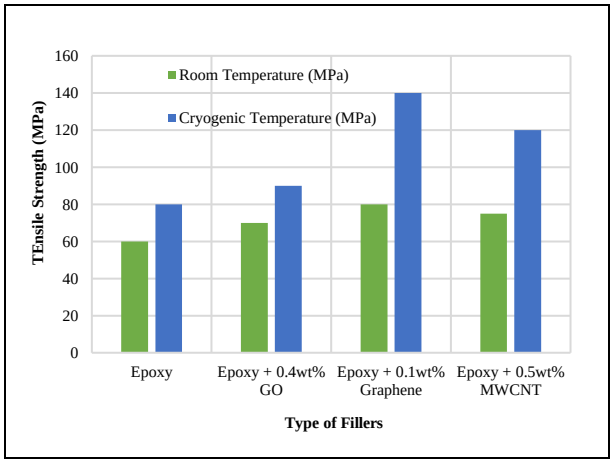


Fig. 6: Comparison of tensile strength for different epoxy-filler blends (56-59)

The addition of graphene significantly increased the tensile performance of epoxy, followed by the addition of multi-walled CNTs (MWCNT) epoxy. With the increase in the MWCNT, the adhesive strength improved, which was revealed by atomic force microscopy and blister tests. The tribological performance of the fillers can be estimated from Table 5. The percentage of wear of graphene cannot be measured, and that of CNTs is unavailable.

Table 5. Tribological parameters of carbon-based fillers + epoxy blends

Filler	Wt % of the Filler	Coefficient of Friction	Wear Rate (mm^2 / N-m)	% Wear	Ref.
Graphene Sheets	0.5	0.48	7.10×10^{-6}	85	(Chen *et al.* 2017)
CNT	1.5 – 2.5	0.2	-	-	(Howson *et al.* 2012)
MWCNT	0.5	0.27	-	-	(Khun *et al.* 2014)
Fullerene C60	0.5	0.6	6.94×10^{-4}	60	(Liu *et al.* 2016)
Graphene	0.1	0.17	-	-	(Avilés *et al.* 2020)

Fig. 7 shows the percentage reduction in the coefficient of friction for blended coatings compared to neat epoxy. The addition of Graphene to epoxy has shown a significant reduction in the coefficient of friction, thereby predicting less wear for those coatings.

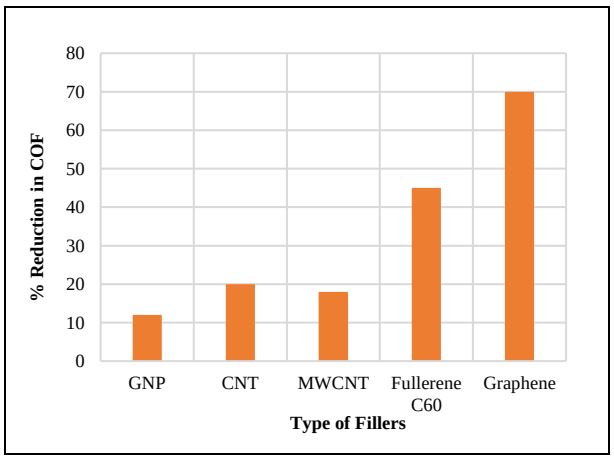


Fig. 7: Percentage reduction in COF (Le *et al.* 2012; Khun *et al.* 2014; Liu *et al.* 2016; Aviles *et al.* 2020)

Fan *et al.* (2022) observed the failure mechanism in GO toughened epoxy's wear tests. GO samples with varying oxidation levels were synthesized using a modified Hummers' method. Analytical techniques including TG, titration, XRD, XPS, and Raman spectroscopy confirmed the enhanced oxidation of GO. Improved dispersion homogeneity of highly oxidized GO in epoxy indicates stronger interfacial interactions. Fig. 5 shows the wear rate of Epoxy GO blends, where it can be observed that the blends have shown better wear performance than neat epoxy.

Nanofillers-based resins significantly improved tensile properties as GO oxidation increased, reaching optimal performance at moderate oxidation levels at room temperature and cryogenic conditions—however, excessive oxidation led to reduced mechanical properties due to GO sheets' compromised strength and modulus. Crack deflection and crack pinning were identified as primary toughening mechanisms at room and cryogenic temperatures, while GO sheet pull-out contributed to toughness at LNT. The degree of oxidation also influenced crack patterns on fractured surfaces.

In the case of powder coatings, a moderate addition of graphene to pure epoxy powder demonstrated a dense, defect-free microstructure. The adhesion between the coating and the substrate was observed to be improved by adding 0.2–0.6 wt% of graphene nanosheets and it also enhanced the impact resistance. Furthermore, results from sliding friction tests indicated that the average value of the coefficient of friction and the rate of wear of the epoxy powder blend coating with 0.4 wt% graphene nanosheets were reduced by 23.8% and 80.29%, respectively, compared to the pure epoxy coating (Zhang *et al.* 2022).

Ultimately all these findings will help industry when the coating materials can be commercialized and mass produced. The handling of the materials and maintenance at room temperatures is considerably easier compared to extreme temperature applications. At cryogenic temperatures, apart from material property degradation of both substrate and coating, special equipment and accessories are required for applying the coatings (Sapi *et al.* 2020). Also, reliable standards are yet to be developed for evaluating properties of materials at cryogenic temperatures. Also, the long-term durability of the coatings at very low temperatures should be studied for commercialization of epoxy coatings. The mass production of epoxy coatings with fillers is another challenge. Mixing huge quantities of resin with fillers and storing it is a big obstacle that has to be addressed (Jin *et al.* 2025).

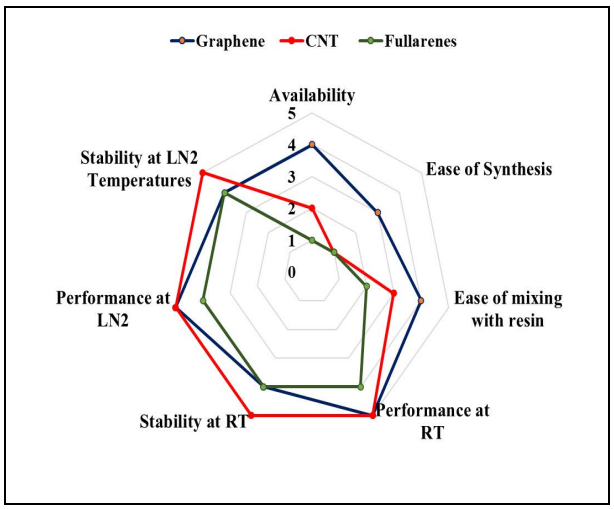


Fig. 8 Comparison of performance of commonly used carbon-based fillers with epoxy

Among the fillers / nano-particles used for making resin blends, mineral based and natural fillers are easily available and of low cost (Sienkiewicz *et al*. 2020,). Carbon based fillers have different price ranges depending upon the manufacturing process and their properties. Fullerenes and CNTs fall under high-cost category whereas carbon black, bio char and some grades of graphene are available at low cost (Khan *et al*. 2017). Fig. 8 gives a brief summary of the comparison of the commonly used carbon – based fillers in terms of cost and performance.

Fig. 8 depicts the performance on a scale of 1 to 5 , 1 being low and 5 being the highest. Among the compared fillers, graphene is economical and offers good cost savings in the long run by improving the performance of the coatings both at room temperature (RT) and cryogenic (LN2) temperatures.

4. CONCLUSION

In this article, an attempt has been made to summarize the studies on the application of polymer coatings mainly epoxies at cryogenic temperatures. However, it has been observed that the research on using polymer coatings including epoxies at cryogenic temperatures is minimal. Owing to the good mechanical and tribological properties of epoxies, they are preferred as a coating material for a wide range of applications like biomedical, membranes, packaging, corrosion protection, sensors, diodes, etc. biomedical, membranes, packaging, corrosion protection, sensors diodes, etc. at room temperature. Also, the properties of polymer coatings were further improved by adding additional fillers/reinforcements at the nanoscale. Among the carbon-based fillers, EP/Graphene demonstrates good tensile strength at both room and cryogenic temperatures. Graphene nanoparticle emerges as the most effective additive, significantly improving both UTS and fracture toughness across temperature ranges. It improves the corrosion resistance which is one of the main disadvantages of metallic coatings. The sliding behaviour of graphene particles improve the abrasion resistance of the coatings as compared to regular polymer and metallic coatings. Moreover the durabiltiy and stability of graphene based coatings at extreme temperatures make it a good choice for the cryogenic temperature applications.

More studies have to be performed on new combinations of polymers and polymer blends to overcome the disadvantages of material property deterioration at low temperatures. Additionally, establishing global standards for testing coatings at cryogenic temperatures, along with designing specialized equipment and accessories for such testing, remains a crucial area of focus. To make the polymer blend coatings to be commercially viable, focus should be on development of mass scale production methods and storage solutions.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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