

EPOXY BASED MATERIALS FOR ELEVATED TEMPERATURE END USES: WHAT DO WE REALLY KNOW?

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SUMMARY: Epoxy phenolic based coatings have become the industry standard for CUI protection, widely respected for their temperature and corrosion resistance, while alternative epoxy technologies have received little attention. As a result epoxy phenolic technology takes a key place in the NACE SP0198_2010 CUI guideline offering protection across the critical CUI range of temperatures. This paper will take a fresh look at epoxy phenolic technology, reviewing chemistry, terminology and performance when subjected to a wide range of operating conditions, both insulated and un-insulated with a view to characterizing performance and understanding why it has become the first choice for many in terms of CUI protection. The paper will subsequently benchmark epoxy phenolic performance against that of alternative epoxy technologies in order to better understand their limitations in terms of application, temperature resistance and corrosion protection in both insulated and un-insulated service conditions. The paper will conclude with a review of the guidance offered in NACE SP0198 regarding conventional epoxy and epoxy phenolic technologies and the terminology used to describe them.

Keywords: Epoxy, Phenolic, Novolac, CUI, SP0198, benchmark, corrosion.

1. INTRODUCTION

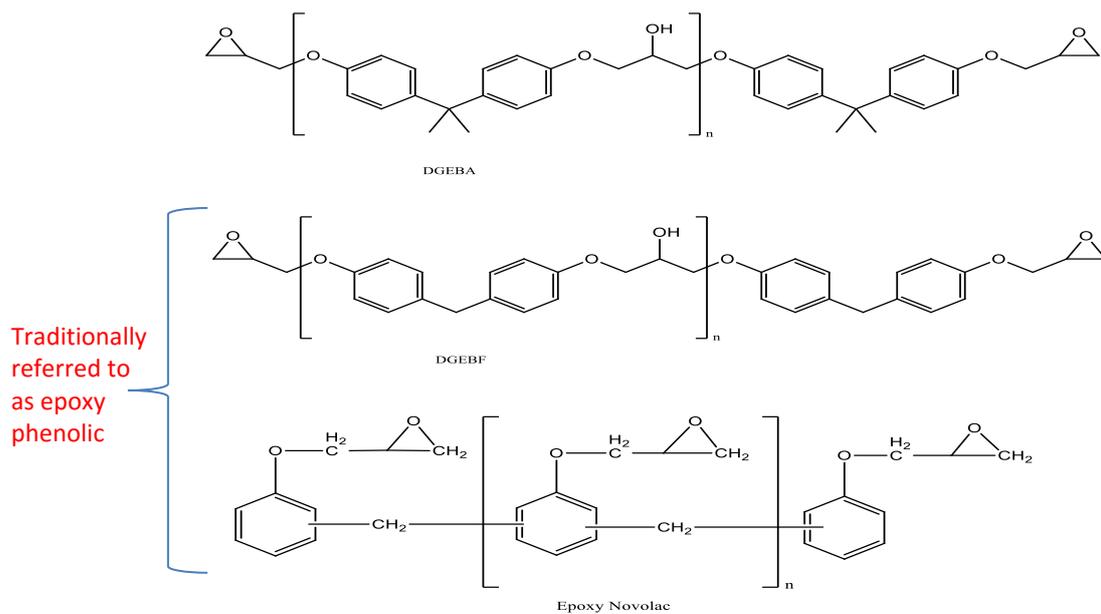
Epoxy based chemistry has been used in the coating industry over the past seven decades. The creation of specification positions has created a confusing number of generic chemistries such as epoxy, epoxy phenolic, epoxy novolac, epoxy phenolic novolac, etc. There are however significant structural and functional differences between these materials, as established in the published literature. Table 1 & Figure 1 below list some of these intricacies. In pursuit of these terminology driven specification positions, benefits from simpler (application benefits) and cost effective solutions are often overlooked. The primary focus of this paper is to review the performance of epoxy chemistry up to 232°C (450°F).

The NACE SP0198 ¹ guideline clearly defines the background to many of the topics touched on in this paper and itself provides evidence to support the discussion. ¹Field experience has shown many instances of CUI on equipment operating at temperatures up to 175°C (350°F), and it has been recognized that carbon steel operating with a skin temperature in the temperature range of -4°C (25°F) to 175°C (350°F) has the greatest likelihood of CUI. Equipment that operates continuously below -4°C (25°F) usually remains free of corrosion. Corrosion of equipment operating above 175°C (350°F) is reduced because the carbon steel surface is warm enough to remain dry. However, corrosion tends to occur at those points of water entry into the insulation system where the temperature is below 175°C (350°F) and when the equipment is idle.

Table 1 – typical epoxy resin types and key property

Epoxy type	Typical functionality	Relative viscosity
Bisphenol A	1.9	Medium
Bisphenol F	2.1	Low
Epoxy phenol novolac	2.6 – 3.5	High
Epoxy cresol novolac	2.7 – 5.4	Very high
Resorcinol DGE	~ 2	Very Low

Figure 1 – molecular structure differences for key epoxy resin types



Variability in functionality affects CUI performance significantly, however there are numerous other intricacies to consider. The confusion is compounded by industry standards and established commercial products available off-the-shelf. See Table 2 for some examples of the confusion in the market.

Table 2 – variation in industry standards for high temperature epoxy products

Standard	Epoxy Product Specified	Specified Temperature
NACE SP0198	Novolac	205°C
NACE SP0198	Phenolic	150°C
CUSTOMER A	Phenolic	200°C
NORSOK M501	Phenolic	150°C
CUSTOMER B	Phenolic	200°C
CUSTOMER B	Novolac	200°C

The key factor to note is within the terminology of ‘Epoxy Phenolic’ where there is variability in the temperature range. This sends mixed signals to the owner / specifier and raises the question ‘what material is being supplied?’. While it is true that chemistry type can give an indication of performance there are many other formulation factors that will impact performance, e.g. filler package, additives used to react/fuse into the polymer network and type of amine curing agent.

2. EXPERIMENTAL

In order to maintain consistency in testing and evaluation surface preparation and application was kept identical. Both pipe sections and flat panels were utilized for testing. Performance evaluation was carried out using three test protocols as listed below –

1. Dry heat testing
2. Corrosion under insulation (CUI)
3. High heat tolerance testing

All tests were performed on carbon steel substrate and surface preparation was done by grit blasting to Sa 2.5 with a profile of 50 – 65 microns (2 – 2.6 mils). Coating material was airless spray applied to a thickness as specified on published product data sheets.

2.1 Dry heat testing

The objective of this test is to identify the maximum dry temperature resistance that a coating can withstand. The coating to be tested was applied on carbon steel pipe sections of 60 cm length and 4mm thickness. The pipe was insulated to minimize heat loss. The hot plate was set at a desired temperature to create a temperature gradient along the pipe. The test was run for 30 days continuously with no temperature cycling. After 30 days

the pipe was removed and the coating evaluated for discolouration, cracking, flaking and disbondment. Figure 2 shows a typical set up while Figures 3 and 4 show various specimens after 30 days of dry heat testing.

Figure 2 - Typical pipe test set up for dry heat testing



Figure 3 – Comparison of epoxy Novolacs after dry heat testing



Figure 4 – Comparison of Bis A Epoxies after dry heat testing



2.2 Figure 3 shows two different products, both based upon epoxy novolac technology. From the calibration temperature noted up the right hand side of the pipe it can be seen that the left hand pipe fails before the 213°C mark, while the right hand pipe maintains its integrity up to 223°C. This simple test demonstrates that relying upon generic chemistry type alone will not give an accurate picture of maximum temperature resistance. The same picture is presented in Figure 4 where two different Bisphenol A based epoxy coatings are tested to the same method and here you can see that the left hand pipe demonstrates much higher temperature resistance than the right one.

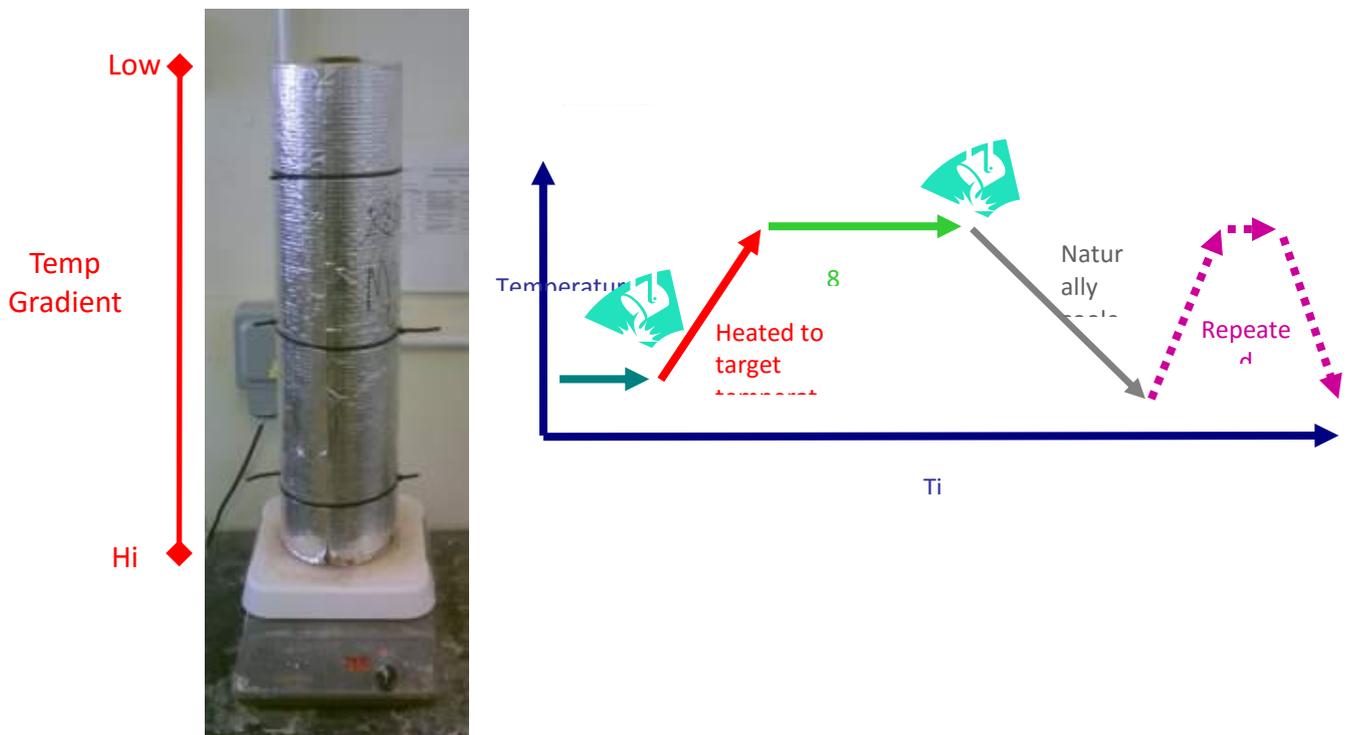
3. CORROSION UNDER INSULATION TESTING

3.1 Using a similar test set-up shown previously, pipe sections were prepared and subjected to cycling testing. The pipes were insulated to minimize heat loss. The insulated pipe was then subjected to the following cycle:

- Cycle description:
 - Add 1 litre of salt water (1% NaCl Solution) to the thermal insulation
 - Apply heat for 8 hours to produce a thermal gradient
 - Add one more litre of salt water
 - Allow to cool to ambient for 16 hours

Figures 5 and 6 show the set up and cyclic process carried out. The test was continued for 30 days, i.e. 30 cycles, and then as previously the pipes were evaluated for discolouration, cracking, flaking and disbondment.

Figure 5 - Typical pipe test set up for CUI testing **Figure 6** – Test cycle for CUI testing

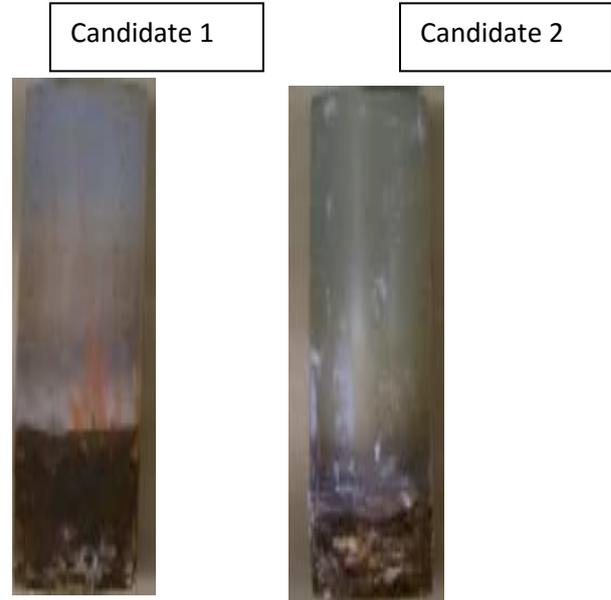


Figures 7 and 8 show the performance of two different epoxy chemistries tested under identical conditions. In Figure 7 both systems are epoxy novolac based but candidate 2 outperforms candidate 1 in this corrosion under insulation test. Both formulations differ in curing agent (amine) chemistry and subsequently cure characteristics. In Figure 8 both formulations are based on Bis A epoxy but candidate 2 significantly outperforms candidate 1.

Figure 7 - Comparison of epoxy Novolacs after CUI testing



Figure 8 - Comparison of Bis A Epoxies after CUI testing



Epoxy Novolac Systems

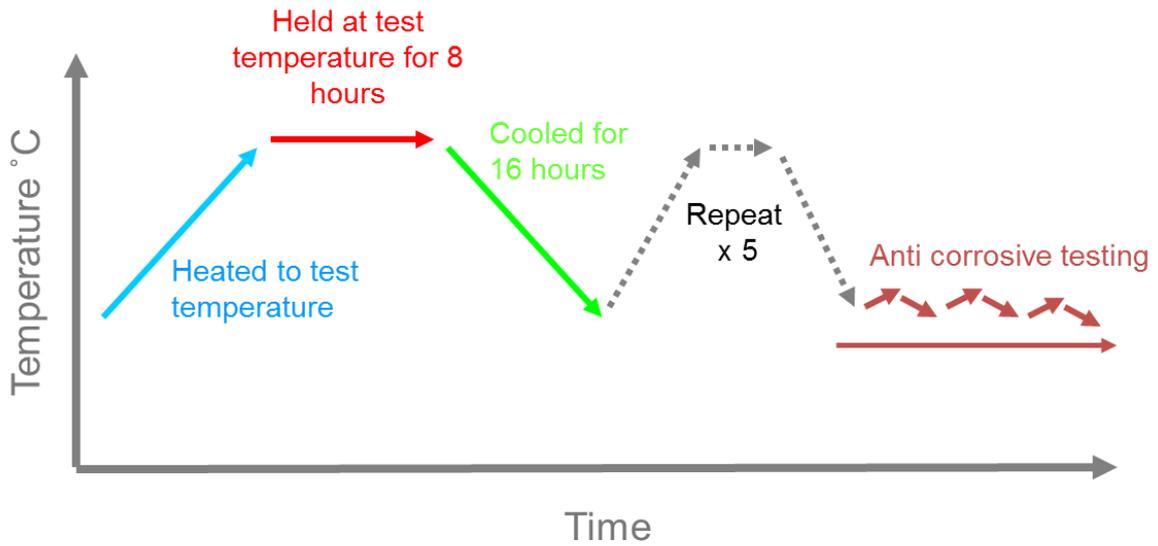
Bis A based Systems

The above work shows that careful consideration of coating chemistry is necessary prior to selection of a suitable coating.

4. HIGH HEAT TOLERANCE TESTING

This test program included high temperature cycling with 5 cycles up to 200°C (392°F). For panels that appeared intact following the high temperature cycling further anti-corrosive testing was conducted to assess the ability of the temperature cycled coating to continue to offer corrosion protection. Figure 9 details the test procedure and durations. Flat panels were utilized. Sample preparation and application was done as per the previously described method.

Figure 9 – test cycle for high heat tolerance testing



4 different products were put through this test. As before two different commercially available materials described as epoxy novolac based and two commercially available materials described as Bis A epoxy resin based were tested (see Figures 10 and 11, respectively). Both the epoxy novolac and the Bis A materials were applied at the same dfts.

Figure 10 - Comparison of epoxy Novolacs high heat tolerance testing

Figure 11 - Comparison of Bis A Epoxies after high heat tolerance testing

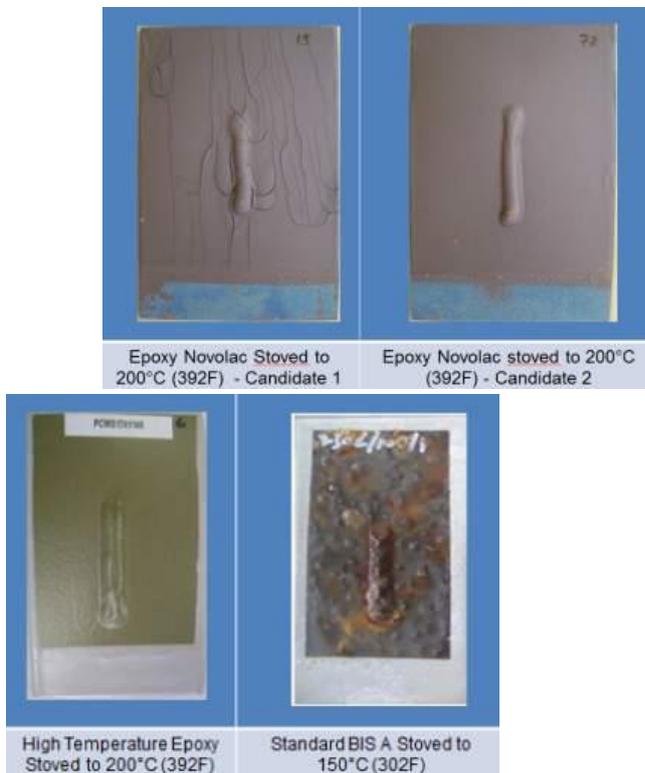


Figure 10 clearly demonstrates that although generically the same, under a simple cyclic temperature test Candidate 2 offers superior elevated temperature resistance compared to candidate 1. Figure 11 demonstrates that for two products based upon Bis A epoxy resin technology, the left hand panel which has been specifically formulated to withstand elevated temperature vastly outperforms a standard Bis A epoxy based technology. What is more, the high temperature epoxy in Figure 11 outperforms the candidate 1 Epoxy Novolac in Figure 10!

5. CONCLUSIONS

From the extensive work carried out in the laboratory, it has been demonstrated that expectations of elevated temperature performance cannot be based upon resin chemistry alone. There are numerous other ways by which a material's temperature resistance can be improved outside of just generic resin type choice. As such this once again raises the issue of generic resin based coating specifications such as those offered in SP0198, where following correct generic resin type guidance can lead to the use of materials which are not fit for purpose.

To answer this issue thorough lab and field evaluation of a given product is essential to provide confidence in performance for a given elevated temperature scenario. Furthermore consideration should be given to new ways of thinking and approaching coating formulation development that challenges the conventions based upon generic resin type and pushes the boundaries of other materials in order to deliver the next generation of elevated temperature coatings.

6. REFERENCES

1. NACE SP0198-2010 - Control of Corrosion Under Thermal Insulation and Fireproofing Materials—A Systems Approach