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THE USE OF CHARTEK 7 INTUMESCENT COATING TO PROVIDE THERMAL PROTECTION TO TRANSPORT AND STORAGE PACKAGES FOR RADIOACTIVE MATERIALS

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Abstract

Traditional thermal insulation materials used in transport packaging's include materials such as cork, wood and foam. Such materials have intrinsically low thermal conductivity values that are largely temperature independent. Whilst they provide effective fire protection in the accident conditions of transport thermal test, these low values do not promote heat dissipation, for example from the package contents in normal conditions of transport. Chartek 7 is a very durable epoxy coating that 'intumesces' under the effects of high temperatures associated with exposures to accidental fires. Because it is applied at very low thickness and has a relatively high thermal conductivity at ambient temperatures, it offers low thermal resistance. This presents an option for efficient design of transport and storage packages, in particular where the dissipation of internal contents heat may be a design requirement. At high temperatures, due to the intumescence process the coating thickness increases by a factor of 10-12, and the thermal resistance significantly increases; limiting heat transfer into the package. These properties allow Chartek 7 to be utilised in packaging designs to augment or replace existing thermal insulation materials. In addition to its thermal properties, Chartek 7 has undergone the most stringent environmental and mechanical tests. These involve testing to the Norsok M501 standard, UL1709 external exposures and specific mechanical tests (impact, drop, penetration and punch tests). Further package related tests have been performed to simulate Accidental Conditions of Transport (ACT) as per the requirements of IAEA regulations pertaining to the safe transport of radioactive material. This paper will present the key thermal and mechanical properties, substantiated by testing, that may be of interest to transport and storage package designers and specifiers, in particular if specifying packages requiring heat dissipation as part of their performance criteria.

Introduction

Chartek 7 has been the dominant passive fire protection (PFP) material used to protect offshore structures from pool and jet fires for over 30 years. Chartek 7 was borne out of developments for the Apollo space program in the 1960's and 1970's's to protect the manned capsules from extreme temperatures during re-entry into the earth's atmosphere. The opportunity to use this advanced technology in the oil and gas sector was quickly identified and was launched as Chartek 59 in 1974. In response to the Piper Alpha disaster in 1988, Chartek 59 underwent further developments to meet the challenge of jet fire in the form of Chartek 3 and 4. These products have since undergone significant technical developments and testing to be the Chartek 7 it is today. Chartek 7 has been used to protect over 6.5m m^2 (70m ft^2) of structures globally.

What is Chartek 7?

Chartek 7 is a 2-part epoxy intumescent coating. The coating contains active components that undergo a non-reversible chemical reaction to create an insulating expanding char when exposed to fire conditions. These active components, Melamine, Pentaerythritol (PE) and Ammonium Polyphosphate (APP) are bound within an epoxy resin. It is this char that prevents or retards heat conduction to the substrate. The chemistry of these active components and degradation mechanism is shown below in figures 1 and 2.

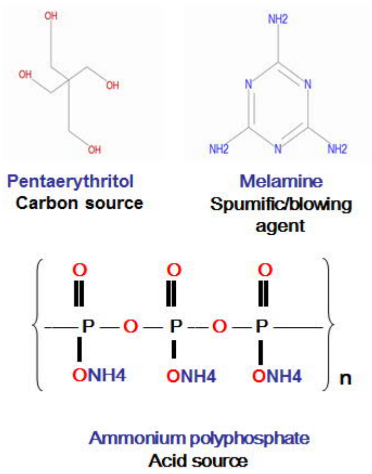


Figure 1 – Chemical structure of active components

Melt Phase	Char Formation	Melamine Degradation	Char Solidifies	Char Degradation
PE & binder system melt	PE & APP react together to start char process	Melamine degrades to evolve large amounts of gas Most char expansion happens	Char hardens to a black spongy mass Expansion rate reduces	As organic material burns off, char starts to degrade. Substrate temperature starts to rise

Figure 2 – Chartek degradation mechanisms

Chartek 7 is stable in the temperature range -40C to +80C, although testing has shown that temperatures of up to 100C for periods up to 4 hours show no detrimental effect on the coating. This range bounds the ambient conditions for transport - according to paragraphs 673(a)(vi) and 679 of the IAEA Transport Regulations, SSR-6, and paragraph 673.8 of the IAEA Transport Guidance, SSG-26, temperatures resulting from ambient conditions of –40°C to

+38°C should be considered unless the Competent Authority specifies otherwise in the certificate of approval.

The melt phase will commence at temperatures of around 100C when components within the coating start to react. These temperature ranges can be extended with the use of thermally insulating materials but this aspect of performance is not covered in this paper.

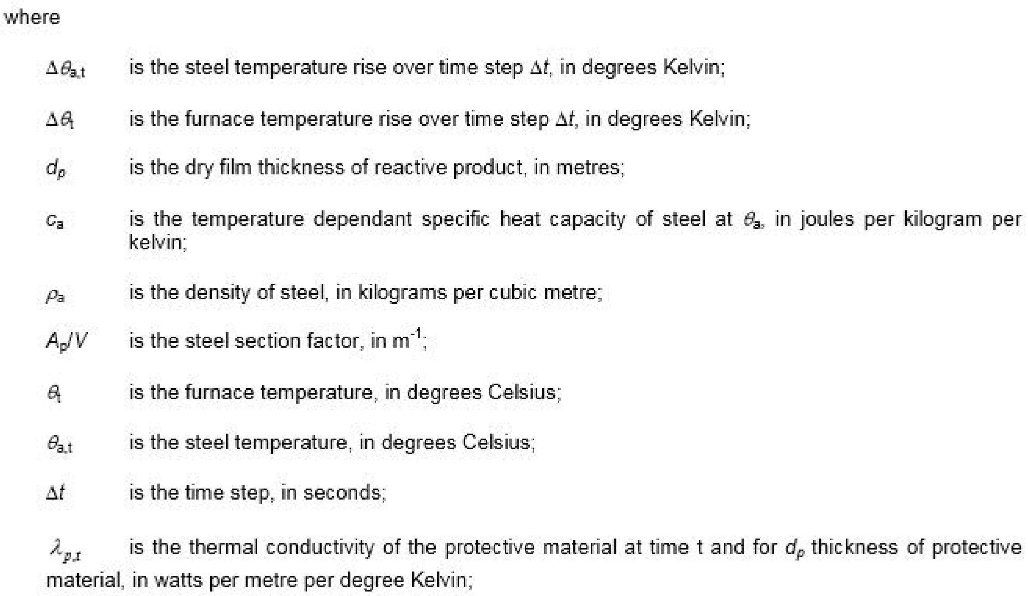
Thermal Properties

The values for a material's thermal inertia properties are typically used in heat transfer modelling. Thermal inertia is defined as:-

$$\boldsymbol{I = \sqrt{k\rho c}}$$

Ambient values of Chartek 7 for these 3 properties are easily obtained, however due to the physical changes of the coating as it chars (expands), some of these properties become temperature dependent and change significantly. For assessment purposes in EN fire test standards, it is permitted to assume values of 1000 J/kg.K and 100 kg/m^3 respectively for specific heat and density respectively. As the value for thermal conductivity dominates in heat transfer modelling and recognising that this value cannot be physically measured (only at ambient using the hot plate method), values for thermal conductivity must be derived by calculation from actual fire testing. The method to do this follows the standard heat transfer equation for a protected element from Eurocode 3[i]:-

$$\Delta\theta_{a,t} = \frac{\lambda_{p,t}}{d_p} \times \frac{A_p}{V} \times \frac{1}{c_a \times \rho_a} \times (\theta_t - \theta_{a,t}) \times \Delta t$$



where

$\Delta\theta_{a,t}$	is the steel temperature rise over time step Δt, in degrees Kelvin;
$\Delta\theta_t$	is the furnace temperature rise over time step Δt, in degrees Kelvin;
d_p	is the dry film thickness of reactive product, in metres;
c_a	is the temperature dependant specific heat capacity of steel at θ_a, in joules per kilogram per kelvin;
ρ_a	is the density of steel, in kilograms per cubic metre;
A_p/V	is the steel section factor, in m^{-1};
θ_t	is the furnace temperature, in degrees Celsius;
$\theta_{a,t}$	is the steel temperature, in degrees Celsius;
Δt	is the time step, in seconds;
$\lambda_{p,t}$	is the thermal conductivity of the protective material at time t and for d_p thickness of protective material, in watts per metre per degree Kelvin;

This equation is re-arranged to find the 'effective' thermal conductivity values:-

$$\lambda_{p,t} = d_p \times \frac{V}{A_p} \times c_a \times \rho_a \times \frac{1}{(\theta_t - \theta_{a,t}) \times \Delta t} \times \Delta\theta_{a,t}$$

Whilst Chartek applications are predominantly in hydrocarbon extraction and processing industries, previously fire testing has been undertaken on package designs for transportation of radioactive materials. In these tests the package containers were coated with a calculated thickness of Chartek 7 and subject to a regulatory 30 minute fire test in accordance with International Atomic Energy Agency (IAEA) regulation SSR-6. The tests were undertaken in the Akzo Nobel centre for fire testing in the UK in a 1.5m^3 gas-fired furnace.

Graph 1 below shows the temperature rise on the external surface of the package container. It is important to note that the 'critical' temperatures are taken to be those recording temperature rise on the surface of the internal containment vessel (CV) - these temperatures were significantly lower at t=30 (max 96ºC)

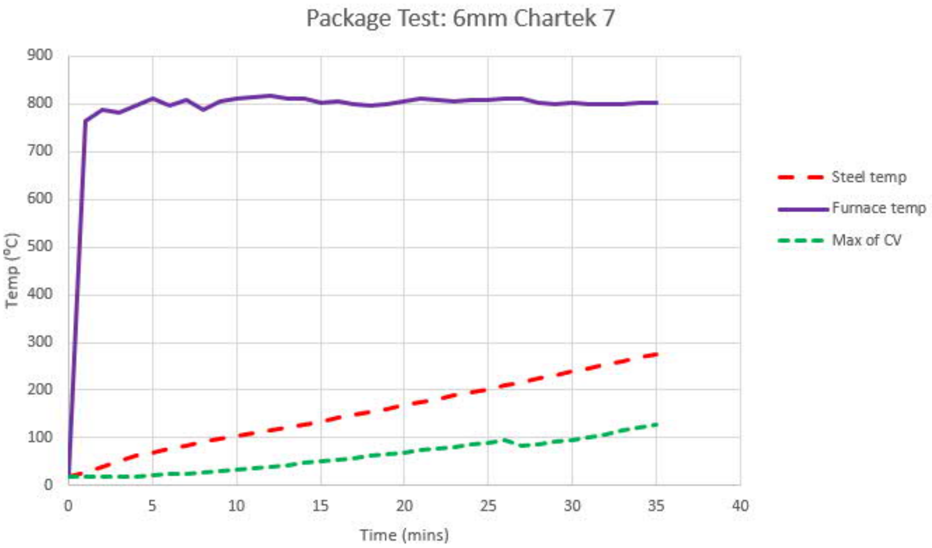


Figure 3 – Temperature rise of external package & internal CV

The measured temperatures from this package test have been used to calculate the 'effective' temperature dependent thermal conductivity values for the external surface of the vessel. These are presented in the graph below clearly demonstrating the rapid reduction in thermal conductivity as the char expands. Also presented are the calculated values from a recent fire test on a Chartek protected steel column (4.5mm dft) showing very close agreement with the values in the vessel package test. Note in this column test, the rise in thermal conductivity at ~550ºC represents the start of char degradation due to furnace gas turbulence, overall performance is a function of Chartek thickness – as the thickness increases the substrate temperatures are lower for a longer period of time.

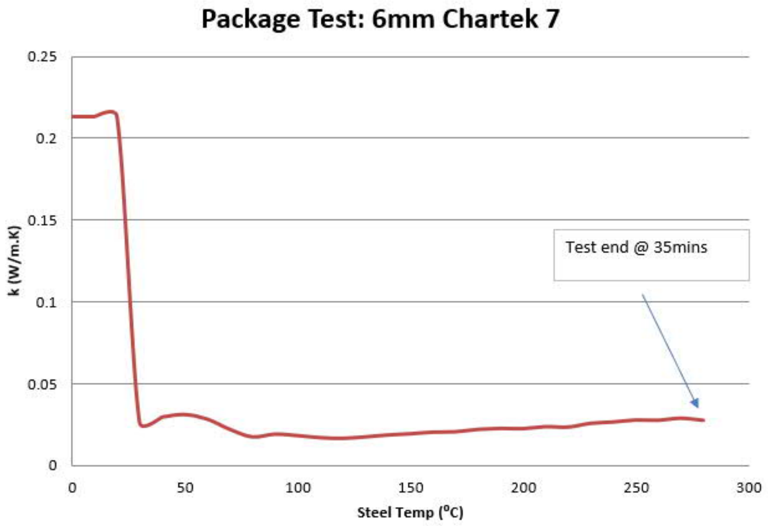


Figure 4 – Derivation of ‘effective’ thermal conductivity values

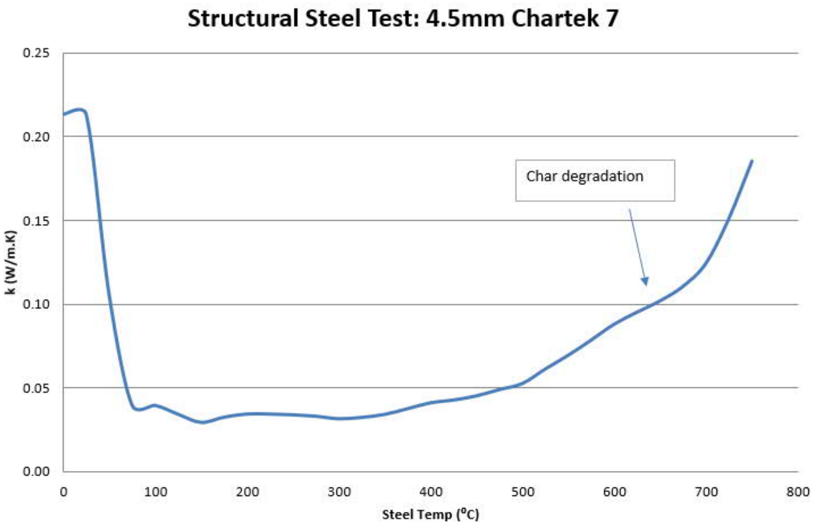


Figure 5 –‘Effective’ thermal conductivity values from a steel column test

Heat Dissipation

Where package designs require heat dissipation from internal contents under normal transport conditions, it is beneficial to utilise materials with low thermal resistance. Owing to the relatively high (ambient) thermal conductivity value of Chartek 7 (0.213 W/m.K), its thermal resistance (R) is therefore only 0.028 m^2.K/W (or U value 35.7 W/m^2.K). The benefits of this

are demonstrated in the 2 graphs below comparing the heat flux and temperature gradients with 6mm Chartek 7 against 50mm cork insulation. Assume internal content temperature 50ºC, ambient 0ºC and still air.

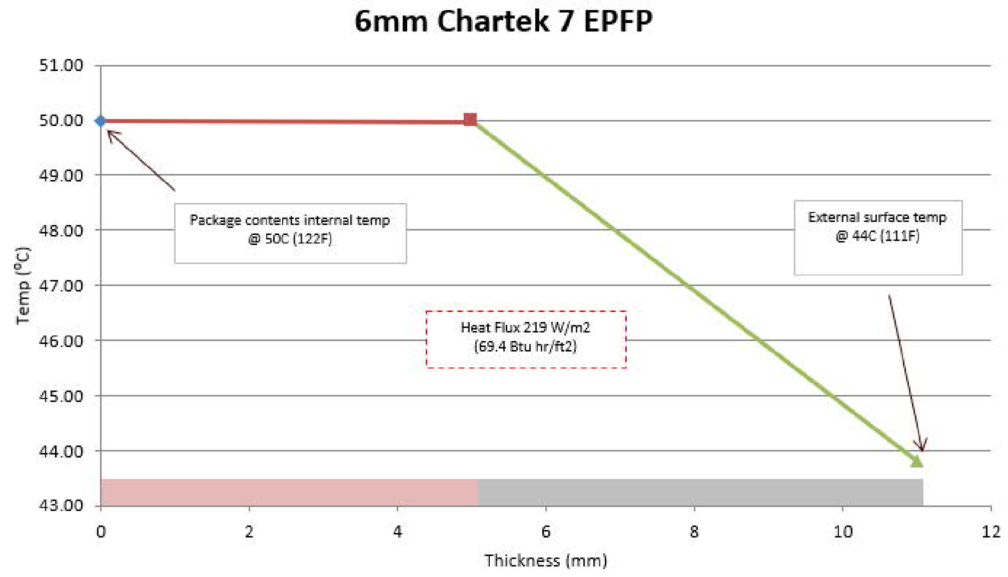


Figure 6 – Steady state temperature gradient through 5mm steel & 6mm Chartek 7

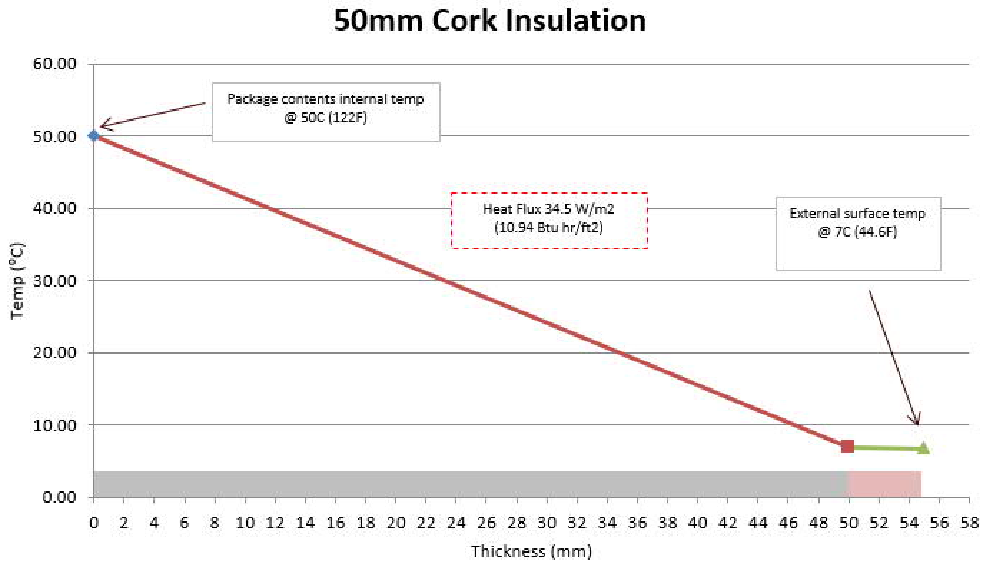


Figure 7 – Steady state temperature gradient through 5mm steel & 50mm cork insulation

Variable Temperature Tests

To permit understanding of the effects of the char integrity under different environmental conditions and at a range of different (lower) fire exposure temperatures, additional thermal tests were undertaken on steel plates with Chartek at 2 different thicknesses'. A number of plates were additionally chilled down to -20C prior to testing and a number after water immersion for a minimum of 72 hours. Figures 8 and 9 below show the test selection and plots of the measured steel temperatures at 4 different fire temperatures.

Coating dft (mm)	Surface Temperature (°C)	Heat Flux (kW/m^2)	Pre-test conditions
6	200	6	Dry, ambient
6			Wet, 72hr pre-test soak in tap water
3.5			Dry, ambient
6	400	16	Dry, ambient
6			Wet, 72hr pre-test soak in tap water
3.5			Dry, ambient
6	600	37	Dry, ambient
6			Wet, 72hr pre-test soak in tap water
3.5			Dry, ambient
6	800	75	Dry, ambient
6			Wet, 72hr pre-test soak in tap water
3.5			Dry, ambient
6	200	6	Dry, plate temp at -20°C
	400	16	
	600	37	
	800	75	

Figure 8 – plate tests, variable exposure conditions

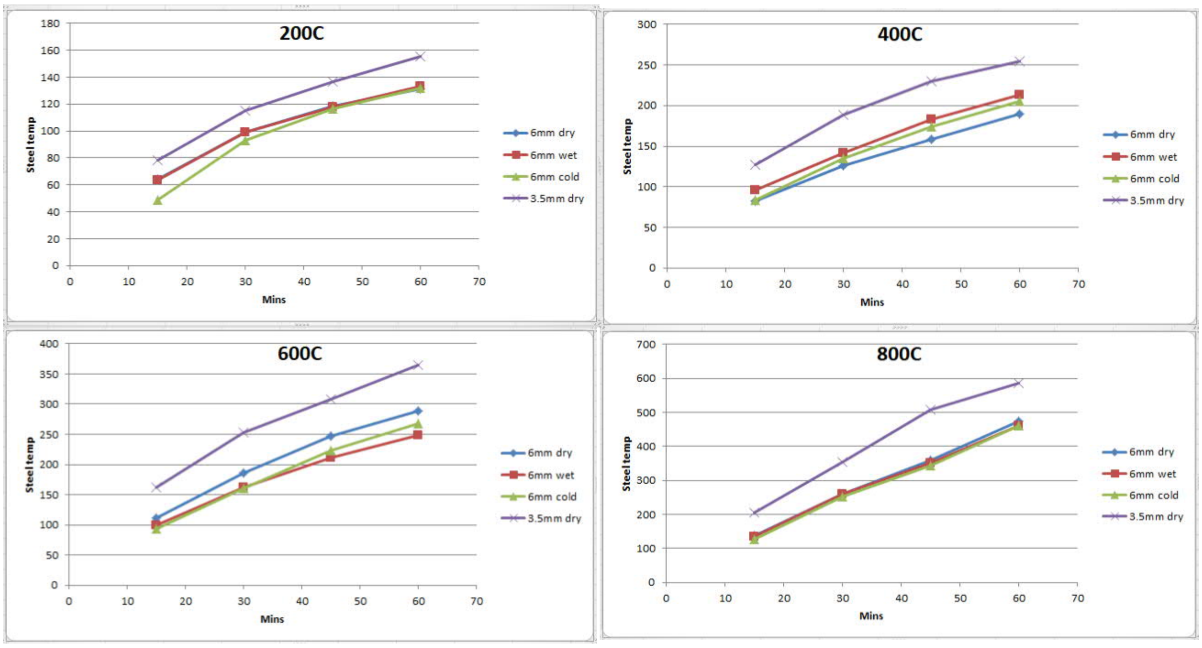


Figure 9 – graphs of measured steel temperatures

Mechanical Properties

IAEA performance requirements demand rigorous tests to demonstrate package designs are resistant to mechanical damage. As the Chartek coating provides a vital element in overall package performance, these tests are used to determine coating integrity. Specifically will such tests lead to loss of thermal performance when placed under fire test conditions? Chartek has been tested under the following conditions with respect to the SSR-6 requirements for type B(U) packages:-

Impact tests were performed upon full scale test packages for the normal conditions of transport (NCT) and accident conditions of transport (ACT). The test specimens were subjected to the cumulative effects of the 4 impact tests listed below:-

- 1.2m drop test
- 1.0m penetration test
- 9.0m drop test
- 1.0m punch test

For the impact test programme two test packages were utilised; one for the lid edge impact orientation and one for the side impact orientation. Due to the cumulative effects of performing the previous 9m ACT impact test, the punch test was found to be most damaging, with coating removed from the initial impact, but also the sliding movement of the package over the punch initiating a shearing mechanism within the coating.

However, when considering the package as a whole, the local Chartek damage was a relatively small area of the package surface area. This was considered acceptable to proceed to thermal testing.

The above tests were then followed by a successful leak test of the CV before proceeding to a regulatory thermal test of the damaged package previously damaged in the regulatory impact testing. The thermal test is designed to achieve:-

- 800°C flame temperature for 30 minutes duration.

The results of the above tests were successfully utilised in contributing to package qualification. Furthermore, the successful test with a damaged coating allowed a determination of the package damage tolerance, and firm recommendations to be made with respect to maintenance and serviceability.

Resistance to accidental impact damage must be considered as a key material property for a thermal protection medium that must demonstrate superior resistance to cracking and possible detachment of the coating. This requirement has been evaluated by extensive testing to ASTM D2794-93[ii] with modifications to the standard to generate more onerous loading conditions, notable increasing the impact weight from the standard 4kg to 15kg. These tests are used to determine at what impact energy the coating will suffer this cracking and detachment. Tests were undertaken in the following conditions:-

- Chartek average dft 11mm
- Drop heights 0.7m to 1.5m
- Impact weight 15.3kgs
- Plate temperatures 20C and -20C
- Impact point on coated and reverse faces

The results show:-

- Up to 105J impact energy, ambient temperature – no cracking of the coating
- Up to 150J impact energy, ambient temperature – visible surface cracking but no coating detachment*[note]
- Up to 105J impact energy, -20C – visible surface cracking, no coating detachment*[note]

Note – in terms of cracking, full depth cracks are deemed worse case as these are often associated with shear failure detachment at the steel/coating interface - visible surface cracking of widths up to approximately 3mm Chartek intumescent coating has been shown to ‘self-heal’ during the char expansion process, thus ‘closing off’ a route for possible heat transfer to the steel substrate.

Additional Tests

- Water Deluge:-

  Chartek has been successfully tested to the requirements of NFPA 58 AnnexH[iii]. This 50 minute torch fire test runs concurrently with a pressurised (30 psi) water hose stream for 10 minutes and the test shall show no detrimental effects on the char formation during water deluge and its ability to retard the rate of heating to the underlying steel substrate.
- Blast Testing:-

  Chartek has shown resistance to blast tests producing a peak overpressure of 4bar[iv] in an explosion chamber measuring 9m long by 4.5m in width and opening height. These tests subject the circular test specimens to both overpressures and drag loading.
- Weathering Testing

  The integrity of an epoxy type intumescent coating such as Chartek is highly dependent on the ability of the resin binder to ‘protect’ the active components from effects of extreme environments, such as high salinity through to arctic exposures. The most demanding test regime used to evaluate the performance of coating systems is Norsok M501[v]. This standard defines those tests that are necessary to perform based on ISO 20340[vi]:-

- Cycling testing - 25 cycles of 168hrs, including:-
    - Wet/dry cycling, UV and water immersion - 72 hours
    - Salt spray – 72 hours
    - Freeze/thaw – 24 hours

After exposure, panels evaluated for:-
- Disbondment
- Blistering
- Rusting (corrosion creep from a scribe line)
- Cracking
- Chalking
- Flaking
- Adhesion

Post weathering samples are then subject to a hydrocarbon fire test and compared to 'control' specimens that have not been aged or weathered. A maximum 10% reduction in fire resistance is permitted compared to the controls tested in the same furnace. Chartek 7 is Norsok M501, Rev 6 certified.

The Underwriters Laboratories (UL) 1709[vii] standard further defines a test regime to determine coating fire integrity after different environmental exposure conditions:-

- Accelerated ageing at 70°C for 270 days
- High humidity at 97-100% for 180 days
- Industrial atmosphere, SO_2 and CO_2 for 30 days
- Salt spray for 90 days
- Wet, freeze and dry cycling – combined cycling for 72 hours, repeated 12 times
- Acid and solvent spray

Chartek 7 is UL certified under design listing XR617.

Conclusions

Thin film epoxy intumescent coating technologies, such as Chartek represent a novel approach to providing or augmenting existing thermal protection to package design for the transportation or storage of radioactive materials. This paper has outlined performance of the Chartek coating in the regulatory impact and thermal tests of the IAEA transport regulations and how these properties could be used to demonstrate or enhance package performance in NCT and ACT.

In addition further information is presented upon the key mechanical and environmental testing that has been performed on Chartek to underpin its durability including compliance with global test standards required in the oil and gas industry.

The unique attributes that this technology brings to the transport packaging designer offer enhanced thermal performance and safety which can be considered alongside or in place of conventional thermal insulating materials in radioactive material packaging designs. Most importantly, Chartek coating offers highly insulating properties under fire conditions, whilst under normal ambient conditions, the low thermal resistance allows heat dissipation from the internal containment vessel contents.

Acknowledgements

The author wishes to thank Croft Ltd for providing assistance in the development of a test package that meets the IAEA requirements, SSR-6. Further gratitude is extended to the author's colleagues in the Felling fire and insulation laboratory.

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[i] EN 1993-1-2:2003, Eurocode 3: 'Design of Steel Structures, Part 1.2: General Rules, Structural Fire Design'

[ii] ASTM D2794-93 (2019): 'Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)'

[iii] NFPA 58, Liquified Petroleum Gas Code: 'Annex H Procedure for Torch Fire and Hose Stream Testing of Thermal Insulating Systems for LP Gas Containers'

[iv] DNV GL, Spadeadam Test Procedure 108

[v] Norsok Standard M501 Edition 6, 'Surface Preparation and Protective Coating'

[vi] ISO 20340 2003, Paints and Varnishes: 'Performance Requirements for Protective Paint Systems for Offshore and Related Structures'

[vii] UL1709 Edition 4, June 2011: 'Standard for Rapid Rise Fire Tests of Protection Materials for Structural Steel'