

ENGINEERING, PRODUCTION AND LIFE-CYCLE MANAGEMENT FOR THE COMPLETE CONSTRUCTION OF LARGE-LENGTH FIBRE-BASED SHIPS

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EXECUTIVE SUMMARY

Today, fibre reinforced polymer (FRP) materials are extensively used for building lightweight hull structures of vessels with length up to about 50 metres, whereas in longer vessels their use is limited to secondary structures and components. In the European FIBRESHIP research project, innovative FRP materials are evaluated, new design and production procedures and guidelines are elaborated, and new validated software analysis tools are developed. As a result of the project, a comprehensive set of methods will be compiled, enabling the building of the complete hull and superstructure of over 50-metre-long ships in FRP materials. The results enhance significantly the use of FRP materials in shipbuilding and strengthen the competitiveness of the European shipbuilding industry on the world market.

In Task 2.4 of the FIBRESHIP project, an extensive experimental campaign was performed in two phases to characterize the fire performance of FRP materials and solutions.

For the first phase, seven commercially available resins or resin systems were selected for examination of fire performance. Laminates with glass fibre reinforcement and cured resins without reinforcement were produced for cone calorimeter tests and thermogravimetric analyses, respectively. From these seven candidates, two materials were down-selected on the basis of the mechanical performance, manufacturability and impact (including cost, claimed fire retardancy, worker health impact and recyclability).

The two material solutions chosen to continue to the second phase were LEO vinylester resin system and SR1125 epoxy resin system. The fire tests of the first phase showed that an intumescent coating on the surface of these laminates is essential for providing adequate fire performance.

In the second phase, a more comprehensive evaluation of thermal and fire properties was performed by carrying out more cone calorimeter tests, as well as dynamic mechanical thermal analysis, microscale combustion calorimetry, differential scanning calorimetry and transient plane source tests. Simultaneously, data for pyrolysis modelling, thermomechanical modelling and fire simulation was produced.

In cone calorimeter test at the irradiance of 50 kW/m², the times to ignition of coated LEO and SR1125 were 75 and 52 seconds on the average, respectively. The maximum heat release was 261 kW/m² for SR1125, but only 69 kW/m² for LEO indicating good reaction-to-fire performance. The total heat release and the total smoke production were ca. 40 MJ/m² and 9 m², respectively, for both systems.

Cone calorimeter tests of coated laminate specimens were run also at the irradiance levels of 25 and 35 kW/m² and for specimens representing different production batches. The results were not consistent in all cases. In addition, DSC tests revealed changes of the glass transition temperature when the specimens were re-heated, referring to incomplete curing in the manufacturing process. These observations highlight the importance of repeatable and well-controlled manufacturing process. The whole process must be carefully instructed, monitored and reported. The laminates and coatings must be of uniform quality to ensure the fire performance claimed on the basis of fire tests performed. Precise specifications and quality control play a key role in securing the fire safety of materials and products.

Thermogravimetric analyses and micro-scale combustion calorimetry showed that the mass loss of cured resins typically starts slightly above 300 °C both in inert (N₂) and oxidative (air) atmosphere. The reactions in these atmospheres differ, the oxidative atmosphere revealing reactions such as char oxidation. At about 300 °C, however, a structure made of these FRP materials starts to produce combustible gases and contribute to fire.

Dynamic mechanical thermal analysis showed that the glass transition temperature is ca. 111 °C for LEO, and ca. 95 °C for SR1125. In general, the glass transition temperatures of FRP materials are typically about 100 °C. At this temperature, the material softens and loses its loadbearing capacity.

Structures made of FRP materials have a tendency to heat up locally, due to their relatively low thermal conductivity. In the case of a local fire, combustible gas production and heat release are the main concerns in terms of fire safety. If the fire threatens a large structure, like in the case of a compartment fire, the main problem is the softening of the material and the loss of the loadbearing capacity.

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NOMENCLATURE/ ACRONYM LIST

Acronym/symbol	Meaning
C_p	Specific heat capacity
DMTA	Dynamic Mechanical Thermal Analysis
DSC	Differential Scanning Calorimetry
FRP	Fibre Reinforced Polymer
HRR	Heat Release Rate
HRR_{max}	Maximum value of heat release rate
k	Thermal conductivity
MCC	Micro-scale Combustion Calorimetry
MS	Mass Spectrometry
$R@1000^{\circ}C$	Residual mass in % at the final temperature of the test (1000 °C)
STA	Simultaneous Thermal Analysis
$T_{50\%}$	Temperature at which the specimen has lost 50% of its initial mass
T_g	Glass transition temperature
TGA	Thermogravimetric Analysis
THR	Total Heat Release
t_{ig}	Time to ignition
$T_{onset, 10\%}$	Temperature at which the specimen has lost 10% of its initial mass
TPS	Transient Plane Source
TSP	Total Smoke Production
α	Thermal diffusivity
$\Delta H_{c, eff}$	Effective heat of combustion
Δm	Mass loss

1. INTRODUCTION

This document describes the results of the fire performance tests performed in Task 2.4: Fire performance of composites, as part of WP2: Fibre based materials and joining solutions for large-length ships.

In Task 2.4, an extensive experimental campaign was performed in two phases to characterize the fire performance of fibre reinforced polymer (FRP) materials and solutions. In Phase 1, seven candidate materials were tested. From these candidates, two materials were down-selected for more comprehensive studies in Phase 2. The test methods used and their outcome are introduced in Table 1.

TEST METHOD	PHASE	OUTCOME
Cone calorimeter	1 & 2	time to ignition, heat release and smoke production data per unit area, mass loss
TGA	1	mass loss as a function of temperature
MCC	2	heat release as a function of temperature
DMTA	2	temperature dependency of key mechanical properties (storage modulus, loss modulus), glass transition temperature
DSC	2	specific heat capacity
TPS	2	thermal conductivity

Table 1 - Test methods and their outcome

The cone calorimeter test ISO 5660-1 [1] was used for the assessment and comparison of the reaction-to-fire performance of the material candidates. The tests also produced data for modelling and simulation in other work packages of the FIBRESHIP project.

Thermogravimetric analysis (TGA) provides information on the thermal stability of the material tested. The data was used for the assessment of the material's potential applicability, determining the temperature at which significant thermal decomposition begins. TGA tests were performed both in N₂ and in air, in order to get information on the role of oxidation reactions, and on the number of reactions in each environment. TGA data can also be used to create kinetic pyrolysis models.

Micro-scale Combustion Calorimetry (MCC) reveals how much combustible gases evolve and how much energy is released in the pyrolysis of the specimen tested. MCC data is also valuable for pyrolysis modelling purposes.

Dynamic Mechanical Thermal Analysis (DMTA) provides information on how the polymer matrix softens at elevated temperatures, which is crucial for evaluating their thermomechanical behaviour. In Task 2.1, key mechanical properties of the FRP laminates were studied in various lay-ups at normal conditions, but not at higher temperatures. Thus,

DMTA tests gave added value to the experimental campaign by providing data for thermomechanical modelling.

Additionally, Differential Scanning Calorimetry (DSC) and Transient Plane Source (TPS) tests were performed to determine the thermal properties of the materials downselected for Phase 2. These tests provided specific heat capacity (C_p) and thermal conductivity (k), respectively.

It is noted that the purpose of the experimental campaign was the comparison of candidate materials, serving the downselection. The tests were not intended for an absolute evaluation of the fire properties of the materials studied, or for classification or product approval purposes.

2. MATERIALS

2.1. Phase 1 materials

For the first phase, seven commercially available resins or resin systems were selected for examination of fire performance. Laminates with glass fibre reinforcement and cured resins without reinforcement were produced for the tests. The specimens were manufactured by ULIM, and the manufacturing process is presented in the FIBRESHIP deliverable D2.1 [2]. Phase 1 materials are introduced in Table 2.

Resin class	Resin details	Notes
Vinylester	LEO system	Tested both with and without a system-specific topcoat
Urethane acrylate	Crestapol 1210	
Epoxy	Prime 27	
Epoxy	SR1125	Tested both with and without SGi 128 topcoat
Bio-epoxy	Super Sap CLR	
Phenolic	Cellobond J2027X	
Thermoplastic	Elium	

Table 2 - Phase 1 materials

In addition to the materials shown in Table 2, an intumescent coating manufactured by CIMNE was studied. In this case, the specimens were stainless steel plates coated with the intumescent coating. A practical guide of application of the CIMNE coating is presented in Annex 4.

2.2. Phase 2 materials

Two of the resin systems introduced above were down-selected for more detailed studies in Phase 2. The down-selection procedure is described in the FIBRESHIP deliverable D2.1 [2]. The

properties taken into account were mechanical performance in dry condition, manufacturability and impact (cost, claimed fire retardancy, worker health impact and recyclability).

The two resin systems chosen to continue to Phase 2 were:

- LEO vinylester resin system
- SR1125 epoxy resin system

It is noted that when the down-selection was made, all Phase 1 fire test results were not available due to a delay in the specimen preparation and delivery to VTT. The evaluation of fire performance was based on the fire retardancy claimed by the resin manufacturer.

The production of laminate test samples for Phase 2, including the lay-up and orientation of glass fabric plies, is described in D2.1 [2].

3. EXPERIMENTAL

3.1. Cone calorimeter

3.1.1. Test method

The cone calorimeter test is a bench-scale fire test method to assess the contribution of the product tested to the rate of evolution of heat during its involvement in fire. The main parts of the apparatus are a cone-shaped radiant electrical heater with a temperature controller, a spark igniter, a weighing cell, a specimen holder, and an exhaust gas system. A schematic picture of the cone calorimeter is presented in Figure 1. The test method is described in detail in the ISO 5660-1 standard [1].

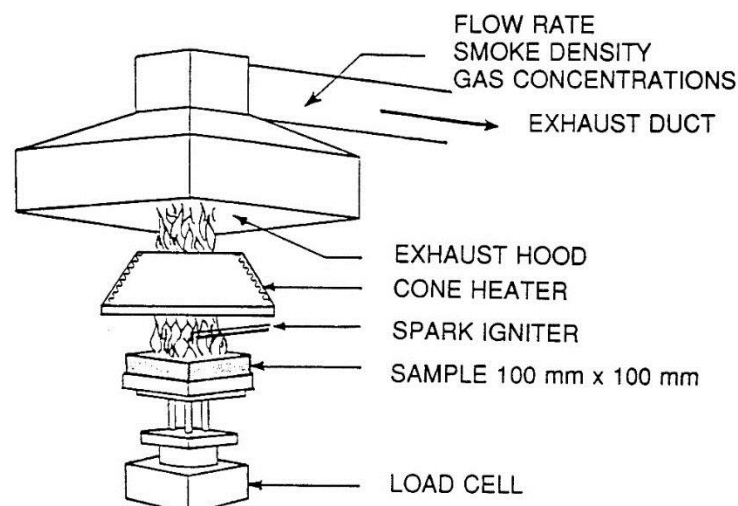


Figure 1 - Schematic picture of cone calorimeter

The test specimen has an area of 100 mm × 100 mm and maximum thickness of 50 mm. In a standard test, the specimen in the specimen holder is positioned under the cone-shaped heater

on the weighing cell. The orientation of the specimen can be either horizontal or vertical, the horizontal orientation being more common in standard testing. The combustion products flow through the top opening of the heater into the hood and the exhaust duct from which gas sample is taken for gas analysis. The main measurements in the test are the time to ignition, the mass loss, and the rate of heat release based on oxygen consumption calorimetry.

In the FIBRESHIP project, the cone calorimeter tests were performed at the premises of VTT Expert Services Ltd using iCone® cone calorimeter manufactured by Fire Testing Technology.

3.1.2. Conditioning of specimens

Before the tests, the composite laminate specimens were conditioned to constant mass in the temperature of (23 ± 2) °C and the relative humidity of (50 ± 5) % RH. The specimens with CIMNE intumescent coating were not conditioned since they could not be left unattended to the premises of the subcontractor VTT Expert Services Ltd for confidentiality reasons.

3.1.3. Specimens and their preparation

3.1.3.1. Composite laminate specimens

The specimens were glass fibre reinforced composite laminates with a thickness in the range of 3–4 mm. For the cone calorimeter tests, the bottom side and the edges of the composite laminate specimens were wrapped to aluminium foil. The specimens were placed on a specimen holder, with layers of refractory fibre blanket (density 64 kg/m³, layer thickness 13 mm) as insulation under the specimens. A retainer frame was used in the tests. The tests were performed in two replicates in horizontal orientation at the irradiance level of 50 kW/m². Specimens of materials in a specimen holder before a test are shown in Figure 2.



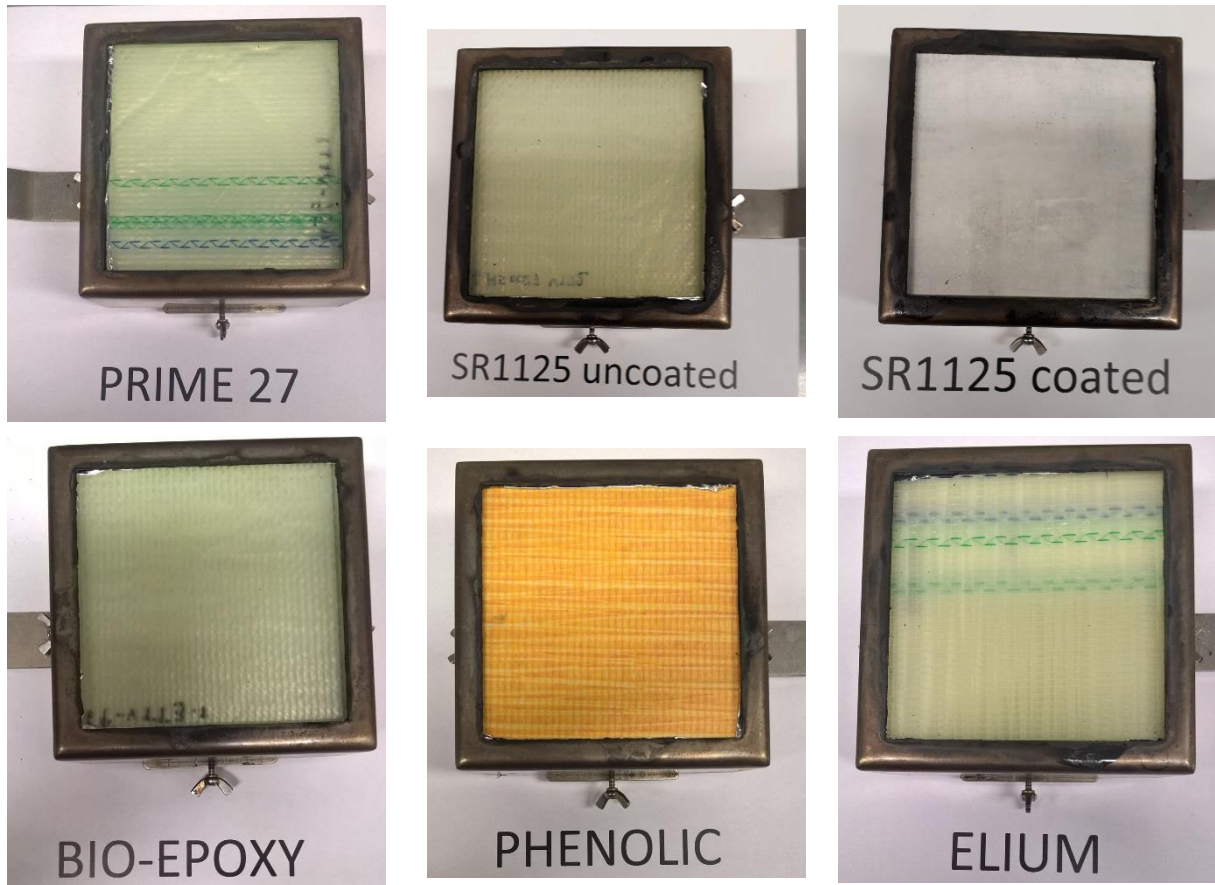


Figure 2 - Cone calorimeter specimens of composite laminates before tests. From left to right: top row LEO uncoated, LEO system with topcoat, Crestapol 1210; middle row Prime 27, SR1125 uncoated, SR1125 with SGI 128 topcoat; bottom row Super Sap CLR, Cellobond J2027X, Elium.

3.1.3.2. Cured resin specimens

Cured resin specimens for cone calorimeter tests (Phase 2) were prepared from cylindrical blocks with a diameter of ca. 100 mm. Slices with a thickness of 3–4 mm were sawn, and the surfaces were polished to make the specimen surfaces smooth. The bottom side and the edges of the circular cured resin specimens were wrapped to aluminium foil. The specimens were placed on a specimen holder, with layers of refractory fibre blanket (density 64 kg/m³, layer thickness 13 mm) as insulation under the specimens. A grid and retainer frame were used in the tests. The grid was taken into use to protect the igniter spark and the cone heater since SR1125 cured resin exhibited extensive intumescence in a preliminary test. Cured resin specimens in a specimen holder before a test are shown in Figure 3.

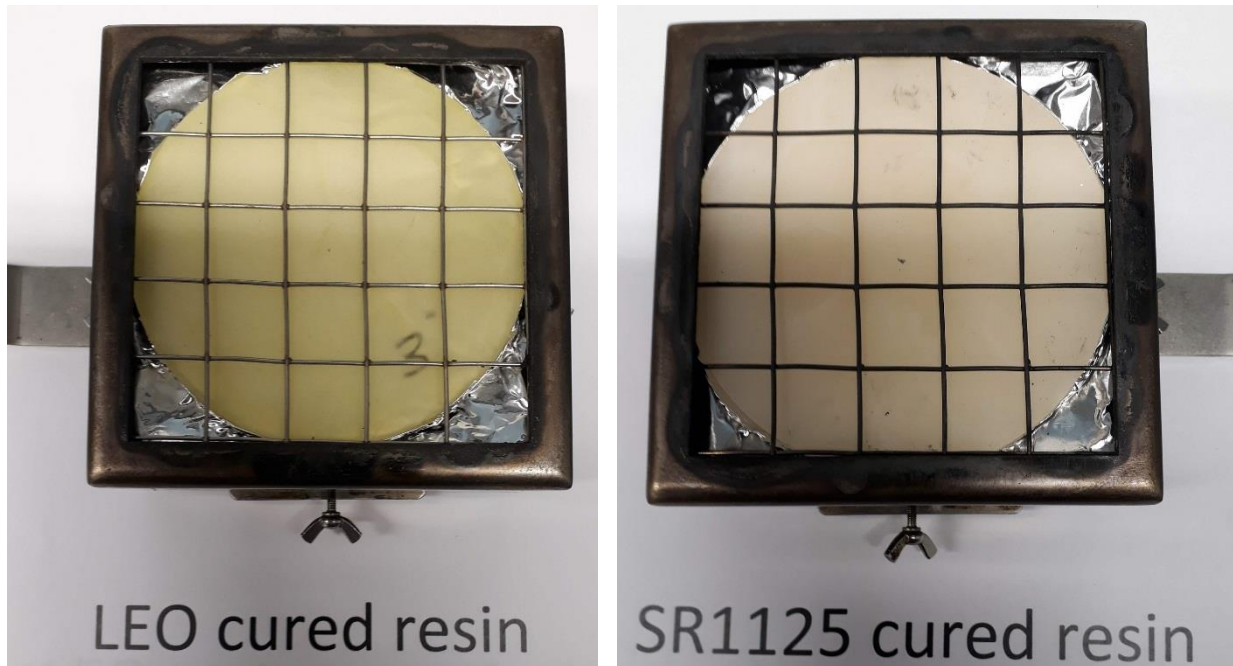


Figure 3 - Cone calorimeter specimens of cured resins before tests. Left: LEO, right: SR1125.

3.1.3.3. Specimens with CIMNE intumescent coating

The specimens including the CIMNE intumescent coating consisted of a stainless steel plate with a thickness of 3.2 mm and the coating on top of it. The total thickness of the coated specimens was 5.0 mm. One uncoated stainless-steel plate was used as a reference specimen. Plate thermocouples of type K (wire thickness 0.5 mm) were glued on the uncoated side of the specimens (TC 1 in Figure 4) two days before the tests.

For the tests, the bottom side and the edges of the specimens were wrapped to aluminium foil. The specimens were placed on a specimen holder and layers of refractory fibre blanket (density 64 kg/m³, layer thickness 13 mm) as shown in Figure 4. In total, four layers of refractory fibre blanket were used, the topmost of which was partly thinned in the middle for specimen positioning, to prevent direct heat transfer to the steel plate through its edges. The total thickness from the bottom edge of the specimen holder to the top surface of the specimen was ca. 48 mm. Another thermocouple (TC 2) was positioned on top of the lowermost refractory fibre blanket as shown in Figure 4. The photograph in Figure 5 illustrates the specimen preparation.

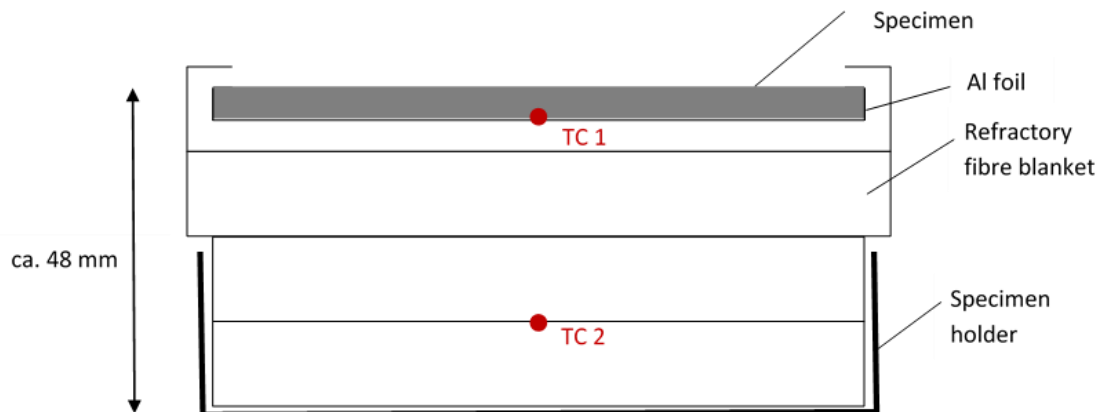


Figure 4 - Schematic picture of specimen preparation (side view, not to scale).

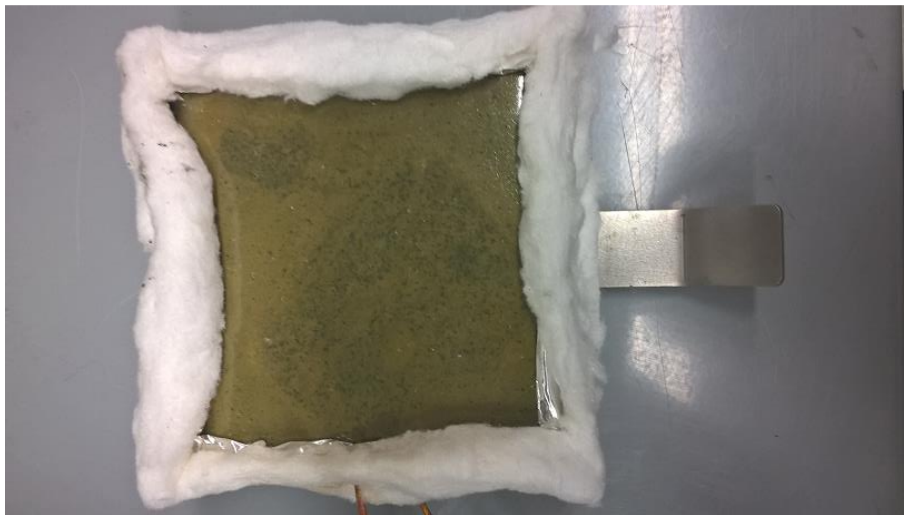


Figure 5 - Specimen "Plate 4" before testing (top view).

The tests for the CIMNE intumescent coating were performed in two replicates in horizontal orientation at irradiance levels of 25 and 50 kW/m². One reference specimen without the coating was tested at 25 kW/m². The specimen IDs for the test in the following chapter are "Reference" for the uncoated steel plate, "Plate 4" and "Plate 5" for the coated specimens tested at irradiance of 25 kW/m², and "Plate 6" and "Plate 7" for the coated specimens tested at irradiance of 50 kW/m². The tests were run for ca. 15 minutes.

It is noted that the cone calorimeter test is mainly meant for specimens of more or less combustible materials. In a specimen consisting of an intumescent coating on a steel plate, there should be practically nothing combustible. The specimen is not expected to ignite, and heat release rate, smoke production rate and mass loss are expected to be very small. So the most significant information in the tests performed was anticipated to be the data from the thermocouple attached on the unexposed side of the steel plate, showing the effect of the coating to the temperature rise. This kind of tests cannot be considered standard tests, but they give some indication on the protective capability of the coating.

3.2. Thermogravimetric analysis

3.2.1. Test method

Thermogravimetric analysis (TGA) is a commonly used small-scale experiment for evaluating the thermal stability of materials. It uses a small furnace filled with either air or inert purge gas (often nitrogen). The sample is inside a small crucible that is placed over a load cell. During the experiment, the sample mass is measured. The experiment can be performed either isothermally (i.e., at a constant temperature) or non-isothermally (with temperature increasing linearly). The non-isothermal experiment is often more convenient for determining reaction kinetics, since it also provides information about the reaction temperatures and requires fewer experiments. The heating rates are relatively low (2–30 K/min), in order to keep the sample in thermal equilibrium with the furnace [3, 4]. TGA data can be used in pyrolysis modelling for the estimation of kinetic parameters.

The TGA instrument used in the FIBRESHIP tests is NETZSCH STA 449F1, shown in Figure 6. The instrument performs simultaneous thermal analysis (STA), i.e. a combination of TGA and differential scanning calorimetry (DSC), along with mass spectrometry (MS).

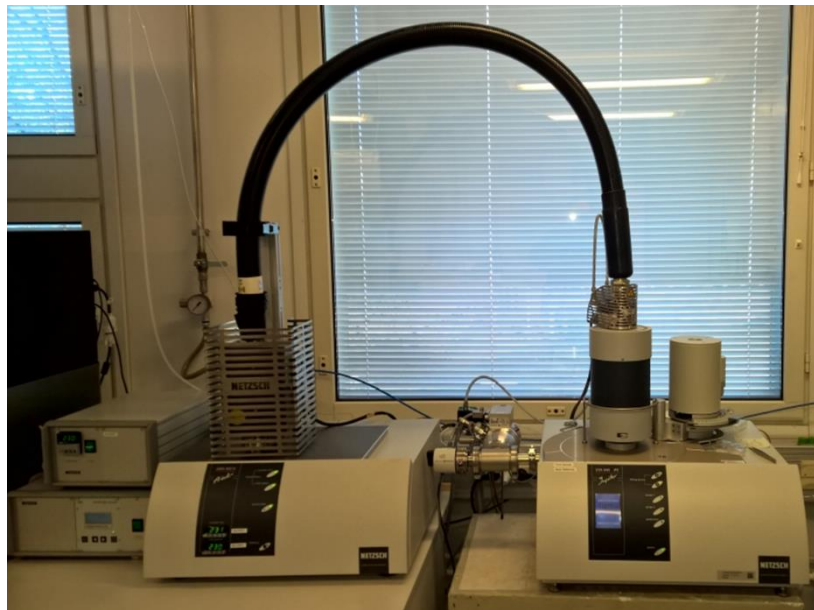


Figure 6 - NETZSCH STA 449F1 instrument for STA

The TGA tests were run both in N₂ and in air to probe the effect of an oxidative environment, and with two heating rates, 5 and 20 K/min, to probe the sensitivity to heating conditions. The temperature range was 40–1000 °C. Two replicate tests were made for each sample in each condition.

3.2.2. Specimens and their preparation

The TGA specimens were solid pieces or powder. The mass of the specimens varied in the range of 5–15 mg.

The cured resins were tested as solid pieces, with the exception of the phenolic resin Cellobond J2027X which was tested as powder due to the tendency of pieces to jump out of the crucible during the tests.

The first TGA trial of the CIMNE coating was made with small, single pieces. However, the samples jumped out of the crucible at the temperature of ca. 300 °C. This was probably caused by internal tensions in the resin matrix. Therefore, only ground powder was used in the actual TGA tests. The powder did not cause any problems in the tests. Figure 7 shows the CIMNE coating test series loaded onto an automatic sample changer.



Figure 7 - CIMNE coating test series in automatic sample changer before (top) and after (bottom) TGA runs

3.3. Micro-scale combustion calorimetry

Micro-scale combustion calorimetry (MCC), also known as Pyrolysis combustion flow calorimetry, is an experimental method for measuring the heat release rate of a small sample as a function of temperature. The method operates in similar manner to thermogravimetric analysis. A small (~ 5 mg) sample is linearly heated inside a furnace, following a constant heating rate (typically 20-60 K/min). The furnace atmosphere is either inert (N₂) or oxidative (air). The released pyrolysis gases are conducted to the combustion chamber that has high temperature and high enough oxygen level to ensure complete combustion [5]. The MCC used is the Govmark MCC2, and it is owned by VTT.

3.4. Dynamic mechanical thermal analysis

Dynamic mechanical thermal analysis (DMTA) is used to measure the mechanical and viscoelastic properties of materials as a function of temperature, time and frequency when they are subjected to a periodic stress. The sample is fixed directly to the force sensor and subjected to a sinusoidal periodic stress with fixed frequency, amplitude and temperature program. Both force and

displacement and well as their phase shift are measured and dynamical material properties: storage modulus, loss modulus and loss factor (tan delta) can be calculated.

Dynamic properties of selected fibre based materials (LEO system and SR1125, both without topcoat) were measured as function of temperature with 1 Hz excitation frequency with Dynamic Mechanical Analyzer (Mettler Toledo DMA/SDTA861^e, see Figure 8) in shear mode and in three point bending mode. Shear mode was used for resin systems samples and three point bending mode for samples with reinforcements.



Figure 8 - Mettler Toledo DMA/SDTA861^e.

Samples were prepared, approximately 5 mm x 5 mm x 1.9 mm for shear and approximately 100 mm x 10 mm x 2.8 mm for three point bending tests. DMTA samples for three point bending tests are presented in Figure 9, showing the fibre orientation. The temperature range was 0 – 160 °C and the heating rate was 2 °C/min. Two parallel measurements were made for each tested material.

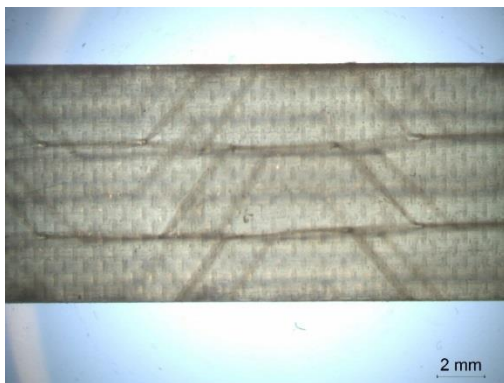


Figure 9 - DMTA samples for three point bending tests. Left: LEO system, right: SR1125. Loading in vertical direction.

3.5. Differential scanning calorimetry

The specific heat capacity (C_p) of LEO and SR1125 cured resins was determined by using temperature modulated differential scanning calorimetry (TM-DSC) between -50°C and 260°C with a heating rate of $5^{\circ}\text{C}/\text{min}$. The equipment was TA Instruments MDSC 2920. The determination of C_p is described in standards ISO 11357-4 and ASTM E 1269. The size of the specimens was $4\text{ mm} \times 0.5\text{ mm}$. Two parallel measurements were made for each tested material.

3.6. Transient plane source method

The measurement of thermal conductivity was done by using transient plane source method, which is described in detail in standard ISO 22007-2. The instrument was Hot Disk TPS 2500S with Kapton sensor and was placed in constant atmosphere room at 24°C and 50 %RH. The samples of size $50\text{ mm} \times 50\text{ mm} \times 2.5\text{ mm}$ were stored in the same atmosphere. There were two sample pieces per material, one surface ground to planar. The planar surfaces were placed against each other and the sensor was placed in between. The sample pieces were pressed tightly together by a screw to achieve as good thermal contact as possible. The heating power was 55 mW for 5 s.

4. RESULTS AND CONCLUSIONS

4.1. Results of Phase 1 tests

4.1.1. Cone calorimeter tests

The results of the cone calorimeter tests of Phase 1 are presented as heat release rate and smoke production rate curves as a function of time in Annex 1, and in Tables 3 and 4. The scalars reported in the tables are t_{ig} = time to ignition, HRR_{max} = maximum heat release rate, THR = total heat release, TSP = total smoke production, and $\Delta H_{\text{c, eff}}$ = effective heat of combustion.

4.1.1.1. Laminates with glass fibre reinforcement

A summary of the cone calorimeter test results of the composite laminates in Phase 1 is shown in Table 3. The tests in Phase 1 were performed at the irradiance level of $50\text{ kW}/\text{m}^2$. Heat release rate and smoke production rate graphs as functions of time are shown in Annex 1.

Cellobond J2027X showed the best fire performance in terms of both time to ignition, heat release and smoke production. Its ignition behaviour was exceptional and differed clearly from other specimens: first, a small local flame appeared close to the spark igniter, and then the flames spread gradually over the specimen surface.

The specimens based on epoxy or bio-epoxy resin, i.e. Prime 27, SR1125 without topcoat and Super Sap CLR, behaved rather similarly. The times to ignition were relatively long, but heat release and smoke production were high.

Crestapol 1210 showed intermediate results in terms of time to ignition, heat release and smoke production. LEO system without a topcoat exhibited results comparable to Crestapol 1210, with the exception of higher smoke production.

Elium had the shortest time to ignition. Its maximum heat release rate was intermediate but the total heat release was high. The smoke production was low. The combustible material of Elium specimens was completely consumed during the tests, only glass fibres remaining.

The measured effective heats of combustion were in the range of 19–23 MJ/kg for all composite laminates tested.

Two of the composite laminates, LEO system and SR1125, were tested both with and without an intumescent topcoat. The coating had a significant influence on the fire performance. In the case of LEO system, the time to ignition increased from 50 to 75 seconds in average due to the coating. A notable change was seen in the shape of heat release rate and smoke production rate curves (see Annex 1). For the coated LEO system specimens, the curves were rather flat on a very low level. In the case of SR1125, the coated specimens exhibited two maxima and an intermediate plateau. The times to ignition of coated and uncoated SR1125 specimens were similar. For both LEO system and SR1125, the total heat release values were of the same order for coated and uncoated specimens, but the total smoke production was reduced due to the coating.

Specimen		t_{ig} (s)	HRR_{max} (kW/m ²)	THR (MJ/m ²)	TSP (m ²)	$\Delta H_{c, eff}$ (MJ/kg)
LEO system without topcoat	Test 1	53	330	36.0	16.0	20.6
	Test 2	47	341	31.0	14.2	20.3
	Average	50	336	33.5	15.1	20.4
LEO system with topcoat	Test 1	75	69	42.2	8.5	19.6
	Test 2	74	68	42.3	9.1	19.6
	Average	75	69	42.3	8.8	19.6
Crestapol 1210	Test 1	43	320	36.2	9.7	20.8
	Test 2	44	308	34.6	9.0	20.6
	Average	44	314	35.4	9.3	20.7
Prime 27	Test 1	60	494	40.1	10.9	22.1
	Test 2	59	498	38.7	10.5	21.6
	Average	60	496	39.4	10.7	21.9
SR1125 without topcoat	Test 1	50	507	43.8	13.9	21.1
	Test 2	55	585	41.1	13.0	21.0
	Average	53	546	42.5	13.5	21.1
SR1125 with SGi 128 topcoat	Test 1	50	267	43.7	9.6	21.6
	Test 2	53	255	37.6	9.0	20.8
	Average	52	261	40.7	9.3	21.2
Super Sap CLR	Test 1	60	498	41.2	11.9	22.3
	Test 2	62	541	42.7	12.1	23.6
	Average	61	520	42.0	12.0	23.0
Cellobond J2027X	Test 1	86 *)	74	9.9	0.4	19.3
	Test 2	115 *)	67	9.9	0.3	19.0
	Average	101 *)	71	9.9	0.4	19.1
Elium	Test 1	23	251	41.2	1.8	22.7
	Test 2	22	258	40.1	1.8	23.0
	Average	23	255	40.7	1.8	22.9

*) The first flame of sustained flaming, close to the spark igniter. The flames spread gradually over the whole specimen surface.

Table 3 - Cone calorimeter test results of composite laminates, irradiance 50 kW/m²

4.1.1.2. Intumescent coating by CIMNE

A summary of the cone calorimeter test results of the intumescent coating by CIMNE is shown in Table 4. The char layer thickness was measured using a slide gauge after the test. The minimum, maximum and “typical” values are based on visual observation of the char layer.

Specimen ID	t _{ig} (s)	HRR _{max} (kW/m ²)	THR (MJ/m ²)	TSP (m ²)	Δm (g)	Char layer thickness (mm)		
						min	max	typical
Reference	NI	4	0.3	0.04	8.59	–	–	–
Plate 4	NI	4	0.9	12.4	12.98	14.2	19.0	18.2
Plate 5	NI	6	2.2	8.5	10.42	11.1	18.0	15.4
Plate 6	60 ¹⁾	188	53.6	8.2	21.85	21.2	29.5	25.0
Plate 7	42 ²⁾	125	37.8	6.0	16.17	13.2	20.1	19.0

NI = no ignition; ¹⁾ unpiloted ignition; ²⁾ piloted ignition

Table 4 - Cone calorimeter test results of CIMNE coating

The specimens were not expected to ignite in the cone calorimeter tests, and this was the case at the irradiance level of 25 kW/m². At the irradiance level of 50 kW/m², however, the specimen “Plate 6” ignited 60 seconds after the start of the heat exposure even though the spark igniter was not present; that is, unpiloted ignition occurred. For the test of the specimen “Plate 7”, the spark igniter was in position. In this case, the specimen ignited in 42 seconds. Due to the different ignition condition, the test results of “Plate 6” and “Plate 7” are not fully comparable. Figure 10 shows “Plate 6” burning, and Figure 11 illustrates the char layer formed during the test.

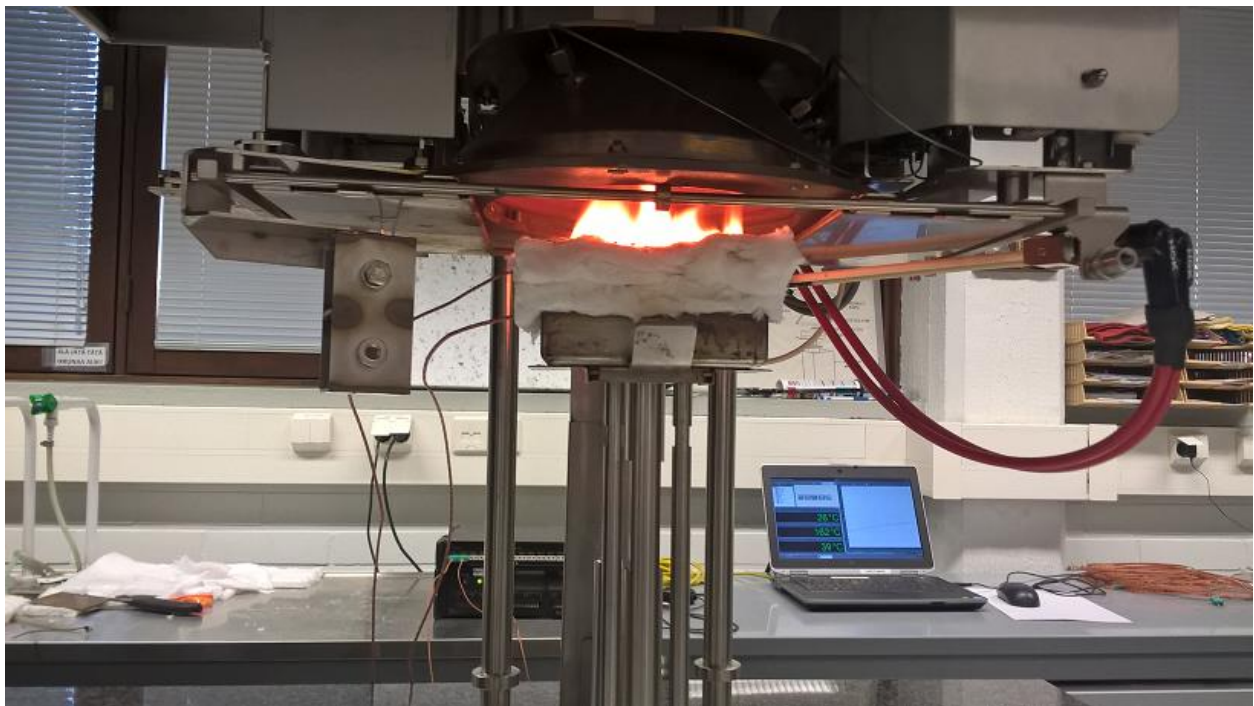


Figure 10 - Specimen “Plate 6” burning during cone calorimeter test at 50 kW/m²



Figure 11 - Char layer of specimen “Plate 6” after cone calorimeter test at 50 kW/m²

The heat release rate and smoke production rate as a function of time are shown in Annex 1, Figure A1-2 for irradiance levels of 25 and 50 kW/m². At 25 kW/m², heat release was practically zero, but notable smoke production was observed. At 50 kW/m², both heat release and smoke production were significant.

The results of the temperature measurements are shown in Figures 12 and 14 for the irradiance level of 25 and 50 kW/m², respectively. Figure 13 illustrates the effect of the intumescent coating to the temperature rise on the unexposed side of the specimen (TC 1) and on top of the lowermost refractory fibre blanket layer (TC 2), showing the difference between the temperatures measured in the reference test and the intumescent coating tests at the irradiance level of 25 kW/m². The temperature difference on the unexposed side of the specimen in the end of the tests was 78 °C in average. For 50 kW/m², similar data is not available since a reference test was not performed due to lack of uncoated specimens of the specific steel plate.

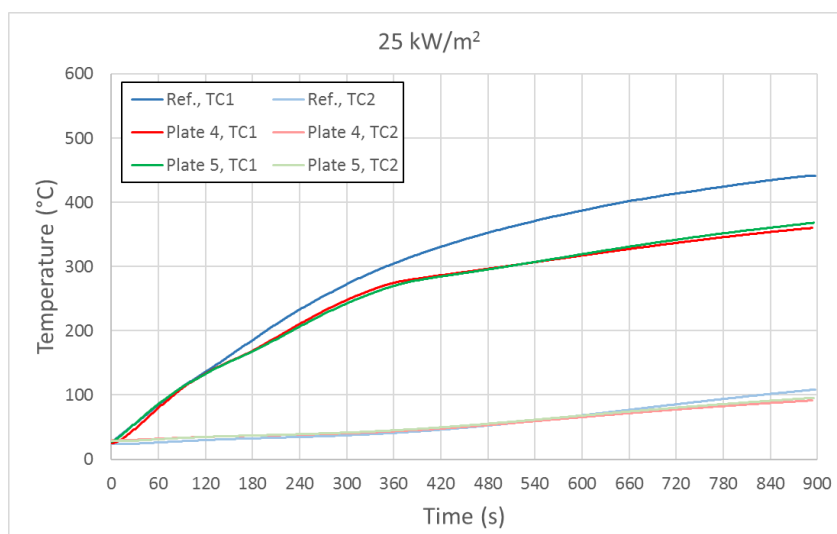


Figure 12 - Results of temperature measurements for irradiance level of 25 kW/m²

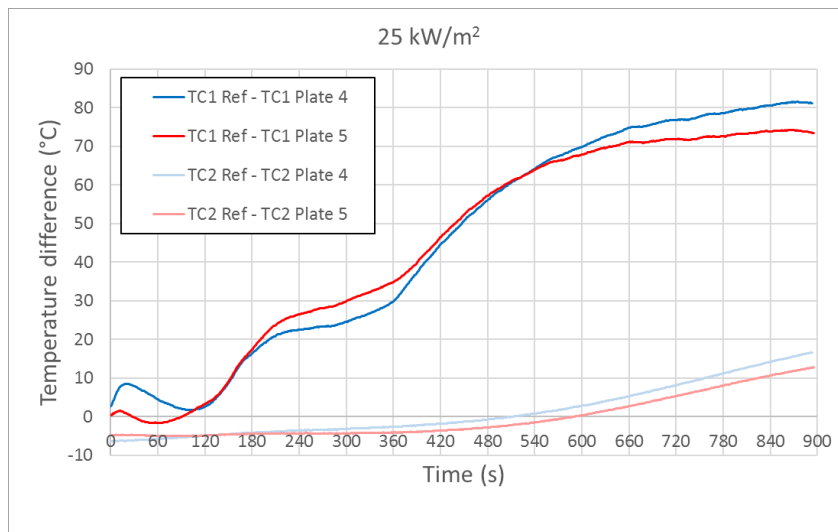


Figure 13 - Difference between temperatures measured in reference test and intumescent coating tests for irradiance level of 25 kW/m².

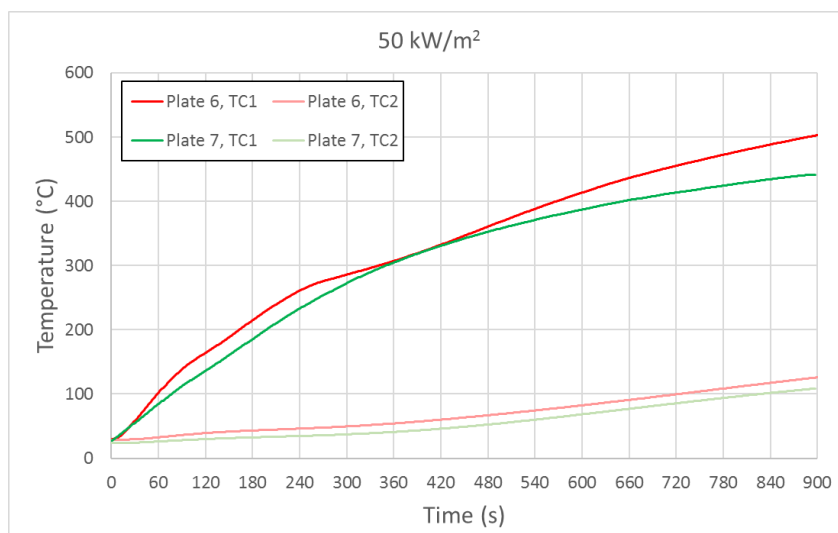


Figure 14 - Results of temperature measurements for irradiance level of 50 kW/m²

The cone calorimeter tests of the CIMNE intumescent coating on stainless steel plate indicated that the coating can delay and decrease the temperature rise of the underlying material. At the irradiance level of 25 kW/m², the temperature difference on the unexposed side of the specimen in the end of the tests of 15 minutes was 78 °C in average. At the irradiance level of 50 kW/m², the coating layer ignited to sustained flaming and burned thoroughly to char, which refers to limited capability to effectively protect underlying combustible material layers. It is noted, however, that the test results are indicative, and the protective ability of the coating on top of FRP products cannot be fully assessed on this basis.

4.1.2. Thermogravimetric analysis

The results of the thermogravimetric analyses are presented as mass loss curves as a function of temperature in Annex 2, and in Tables 5 and 6. The scalars reported in the tables are $T_{\text{onset}, 10\%}$ = temperature at which the specimen has lost 10% of its initial mass, $T_{50\%}$ = temperature at which the specimen has lost 50% of its initial mass, and $R@1000\text{ °C}$ = residual mass (in %) at the final temperature of the test (1000 °C). $T_{\text{onset}, 10\%}$ describes temperature at which the material starts to degrade, produce combustible gases, and contribute to fire.

4.1.2.1. Cured resins

The results of the thermogravimetric analyses of cured resins are presented in Table 5. The best performance is shown by the phenolic resin Cellobond J2027X. It has the highest $T_{\text{onset}, 10\%}$, over 400 °C in N_2 and 365–385 °C in air, and the highest residual mass, over 50 % in N_2 and 6–7 % in air. The thermoplastic resin Elium represents the other extreme, with $T_{\text{onset}, 10\%}$ clearly below 300 °C and residual mass close to 0 %. The other resins are in-between, thermal degradation starting at ca. 300 °C or soon after that, both in N_2 and in air. For all resins, the curve shapes measured in N_2 and in air are different, due to oxidation reactions taking place in air (see Annex 2).

Specimen	Test condition		$T_{\text{onset}, 10\%}$ (°C)	$T_{50\%}$ (°C)	$R@1000\text{ °C}$ (%)
LEO system	N ₂ , 5 K/min	Test 1	332.5	387.5	11.9
		Test 2	332.9	387.9	12.1
		Average	332.7	387.7	12.0
	N ₂ , 20 K/min	Test 1	360.8	410.8	10.7
		Test 2	360.8	410.8	10.4
		Average	360.8	410.8	10.6
	air, 5 K/min	Test 1	329.3	394.3	1.5
		Test 2	329.2	389.2	1.5
		Average	329.3	391.8	1.5
	air, 20 K/min	Test 1	360.5	415.5	1.2
		Test 2	360.9	410.9	1.4
		Average	360.7	413.2	1.3
Crestapol 1210	N ₂ , 5 K/min	Test 1	328.1	393.1	9.9
		Test 2	326.7	396.7	9.9
		Average	327.4	394.9	9.9
	N ₂ , 20 K/min	Test 1	353.6	423.6	9.1
		Test 2	353.1	418.1	8.6
		Average	353.3	420.8	8.8
	air, 5 K/min	Test 1	326.1	396.1	0.8
		Test 2	324.6	399.6	0.4

		Average	325.3	397.8	0.6	
	air, 20 K/min	Test 1	350.7	420.7	0.4	
		Test 2	355.1	420.1	0.4	
		Average	352.9	420.4	0.4	
Prime 27	N ₂ , 5 K/min	Test 1	323.1	358.1	6.0	
		Test 2	322.1	357.1	6.4	
		Average	322.6	357.6	6.2	
	N ₂ , 20 K/min	Test 1	353.3	393.3	6.3	
		Test 2	348.3	383.3	5.2	
		Test 3	347.5	382.5	5.6	
		Average	349.7	386.4	5.7	
	air, 5 K/min	Test 1	320.8	360.8	0.8	
		Test 2	320.0	365.0	0.4	
		Average	320.4	362.9	0.6	
	air, 20 K/min	Test 1	346.1	381.1	0.4	
		Test 2	345.4	385.4	0.3	
		Average	345.7	383.2	0.3	
	SR1125	N ₂ , 5 K/min	Test 1	291.7	336.7	12.7
			Test 2	296.6	336.6	12.3
Average			294.1	336.6	12.5	
N ₂ , 20 K/min		Test 1	315.7	355.7	12.3	
		Test 2	315.8	360.8	11.8	
		Average	315.7	358.2	12.0	
air, 5 K/min		Test 1	294.9	394.9	5.0	
		Test 2	294.4	379.4	5.2	
		Average	294.6	387.1	5.1	
air, 20 K/min		Test 1	315.8	365.8	5.6	
		Test 2	314.1	364.1	5.2	
		Average	314.9	364.9	5.4	
Super Sap CLR		N ₂ , 5 K/min	Test 1	328.0	358.0	5.3
			Test 2	326.5	356.5	5.2
			Average	327.3	357.3	5.2
	N ₂ , 20 K/min	Test 1	353.3	383.3	4.6	
		Test 2	353.3	383.3	4.4	
		Average	353.3	383.3	4.5	
	air, 5 K/min	Test 1	320.5	355.5	0.6	

		Test 2	325.1	355.1	0.5	
		Average	322.8	355.3	0.6	
		air, 20 K/min	Test 1	345.9	380.9	0.5
		Test 2	345.4	385.4	0.5	
Cellobond J2027X (powder)	N ₂ , 5 K/min	Test 1	408.1	NR *)	54.8	
		Test 2	401.7	NR *)	53.1	
		Average	404.9	–	54.0	
	N ₂ , 20 K/min	Test 1	433.1	NR *)	54.3	
		Test 2	433.2	NR *)	54.0	
		Average	433.1	–	54.1	
	air, 5 K/min	Test 1	365.9	445.9	8.3	
		Test 2	365.1	445.1	5.0	
		Test 3	364.8	444.8	5.2	
		Average	365.3	445.3	6.2	
	air, 20 K/min	Test 1	384.4	494.4	8.1	
		Test 2	385.1	490.1	6.6	
		Average	384.8	492.3	7.3	
	Elium	N ₂ , 5 K/min	Test 1	243.0	353.0	0.0
			Test 2	276.9	356.9	0.0
			Average	260.0	355.0	0.0
N ₂ , 20 K/min		Test 1	273.0	373.0	0.0	
		Test 2	282.6	372.6	0.0	
		Test 3	282.1	372.1	0.0	
		Average	279.2	372.5	0.0	
air, 5 K/min		Test 1	265.6	310.6	0.6	
		Test 2	285.1	310.1	0.5	
		Test 3	279.8	309.8	0.4	
		Test 4	280.3	310.3	0.4	
		Average	277.7	310.2	0.5	
air, 20 K/min		Test 1	275.9	335.9	0.5	
		Test 2	285.2	345.2	0.5	
		Average	280.6	340.6	0.5	

*) NR = not reached, i.e. total mass loss < 50%

Table 5 - Results of thermogravimetric analyses of cured resins.

4.1.2.2. Intumescent coating by CIMNE

The results of the thermogravimetric analyses of the CIMNE coating are presented in Table 6. The purpose of the tests was, on one hand, to obtain information on the thermal degradation of this coating, and on the other hand, to serve the development of modelling of coatings in general. The CIMNE coating has $T_{onset, 10\%}$ in the range of 215–260 °C depending on the conditions. The residual mass was ca. 36–37 % in N_2 and ca. 24–25 % in air.

Specimen	Test condition		$T_{onset 10\%}$ (°C)	$T_{50\%}$ (°C)	$R@1000$ °C (%)
CIMNE coating (powder)	N_2 , 5 K/min	Test 1	227.5	522.5	36.3
		Test 2	223.2	518.2	35.8
		Average	225.3	520.3	36.0
	N_2 , 20 K/min	Test 1	243.7	513.7	35.7
		Test 2	238.4	548.4	38.4
		Test 3	237.5	527.5	36.9
		Average	239.9	529.9	37.0
	air, 5 K/min	Test 1	215.4	580.4	25.7
		Test 2	214.6	579.6	24.1
		Average	215.0	580.0	24.9
	air, 20 K/min	Test 1 ^{*)}	274.8	–	–
		Test 2	245.0	630.0	24.0
		Average	259.9	630.0	24.0

^{*)} Test failed after $T = 300$ °C

Table 6 - Results of thermogravimetric analyses of CIMNE coating (powder)

4.2. Results of Phase 2 tests

4.2.1. Cone calorimeter tests

4.2.1.1. Laminates with glass fibre reinforcement

Summaries of the cone calorimeter test results of the composite laminates in Phase 2 are shown in Table 7 for the irradiance of 25 kW/m² and in Table 8 for the irradiance of 35 kW/m². Heat release rate and smoke production rate graphs as functions of time are shown in Annex 3.

Specimen		t_{ig} (s)	HRR_{max} (kW/m ²)	THR (MJ/m ²)	TSP (m ²)	$\Delta H_{c, eff}$ (MJ/kg)
LEO system without topcoat	Test 1	158	316	39.6	14.7	23.3
	Test 2	158	316	39.6	14.7	23.3
LEO system with topcoat	Test 1	NI *)	16	11.9	1.9	15.1
SR1125 without topcoat	Test 1	125	353	32.9	9.0	24.0
	Test 2	132	351	33.8	9.6	23.3
	Average	129	352	33.3	9.3	23.7
SR1125 with SGI 128 topcoat	Test 1	143	114	36.5	6.1	20.9
	Test 2	150	121	27.4	4.5	21.3
	Test 3	136	94	28.1	3.8	19.5
	Average	143	110	30.7	4.8	20.6

*) NI = no ignition to sustained flaming

Table 7 - Cone calorimeter test results of composite laminates, irradiance 25 kW/m²

Specimen		t_{ig} (s)	HRR_{max} (kW/m ²)	THR (MJ/m ²)	TSP (m ²)	$\Delta H_{c, eff}$ (MJ/kg)
LEO system without topcoat	Test 1	92	298	40.3	15.3	22.7
	Test 2	90	308	44.6	15.6	23.5
	Average	91	303	42.4	15.5	23.1
LEO system with topcoat	Test 1	38	230	75.4	29.7	20.6
	Test 2	46	239	77.4	28.6	21.0
	Average	42	234	76.4	29.2	20.8

Table 8 - Cone calorimeter test results of composite laminates, irradiance 35 kW/m²

LEO system with topcoat did not ignite to sustained flaming at the irradiance of 25 kW/m². Therefore, only single tests were run for this material at this irradiance level (Table 7). Since the reaction-to-fire performance of the material during flaming combustion is more useful for pyrolysis modelling purposes, LEO system was tested also at the irradiance of 35 kW/m² (Table 8). The comparison of these test results to those obtained in Phase 1 at the irradiance of 50 kW/m² (Table 3) reveals unexpected behaviour: at 35 kW/m², t_{ig} is shorter and heat release and smoke production is higher than at 50 kW/m². Normally, lower irradiance level should result in longer t_{ig}

and lower heat release and smoke production. Furthermore, the comparison of LEO system with and without topcoat at 35 kW/m², the coated specimens exhibit inferior fire performance: their t_{ig} is considerably shorter and THR and TSP are substantially higher than the values of the uncoated specimens.

LEO system specimens with the topcoat after cone calorimeter tests at different irradiance levels are shown in Figure 15. It can be seen that at 50 kW/m², the coating of the specimen exhibited notable intumescence, but at 35 kW/m² no intumescence occurred. The view angle of the photograph of the specimen tested at 25 kW/m² is different, but it shows that some intumescence took place at this irradiance level.



Figure 15 - LEO system with topcoat after cone calorimeter tests with an irradiance of 25 kW/m² (left), 35 kW/m² (centre) and 50 kW/m² (right).

In a recent experimental study [6], three different experimental setups corresponding to three different fire scenarios were used to investigate how different heating conditions and heating rates affect the behaviour of two different thin intumescent coatings (a solvent-based and a water-based paint). The results showed that the water-based paint performed better at low heating rates, while the tested solvent-based paint performed better at high heating rates and did not activate or provide proper insulation at very low heating rates. The LEO topcoat is solvent-based, with styrene, phosphoric acid ester and methyl methacrylate as the three most abundant components. However, it is unclear whether the observations of Ref. 6 are applicable to this particular case.

The fire behaviour of SR1125, both with and without the SGi 128 topcoat, at irradiance levels of 25 and 50 kW/m² is logical: at 25 kW/m², t_{ig} is longer and heat release and smoke production are lower than at 50 kW/m². For the specimens with the topcoat, three tests were run at 25 kW/m². The first specimen was from another production batch than the second one. Their heat release rate and smoke production rate curves were different (see Annex 3). In Test 1, the curves first show a short-termed maximum, followed by decrease and then another, more gradual maximum. In Test 2, only one broad maximum is seen. Since Tests 1 and 2 were not similar, a third test was performed. Test 3 showed results similar to Test 2.

The average mass of coating per unit area for different production batches of SR1125 and LEO system composite laminates are presented in Table 9. For both systems, the average mass of

coating per unit area in the second production batch was less than in the first production batch: -14 % for SR 1125 and -10 % for LEO. The LEO specimens tested at 25 and 50 kW/m² were from the first batch, and those tested at 35 kW/m² were from the second batch. Of the SR1125 specimens tested at 25 kW/m², the specimen of Test 1 was from the first batch, and the specimens of Test 2 and 3 were from the second batch. It is unclear whether the differences in the average mass of coating per unit area could explain the differences in the fire performance of the specimens.

System	Date	Mass of coating (g)	Mass of coating per unit area in average (kg/m ²)
SR1125	9 Feb 2018	220	1.26
	9 Apr 2018	190	1.09
LEO system	30 Nov 2017	200	1.14
	7 Jun 2018	180	1.03

Table 9 - Coating information of SR1125 and LEO system composite laminates. The area of the laminates was 300 mm × 500 mm.

The observations on the fire performance of coated laminate specimens of different production batches highlight the importance of repeatable manufacturing process. The whole process must be carefully instructed, controlled and reported. The laminates and coatings must be of uniform quality to ensure the fire performance claimed on the basis of fire tests performed. Precise specifications and quality control play a key role in securing the fire safety of materials and products.

4.2.1.2. Cured resins

A summary of the cone calorimeter test results of the cured resins in Phase 2 is shown in Table 10. Heat release rate and smoke production rate graphs as functions of time are shown in Annex 3.

LEO cured resin was tested in two replicates at 35 and 50 kW/m², since these two irradiance levels were used also for LEO composite laminate specimens. For the same reason the irradiance levels of 25 and 50 kW/m² were selected for SR1125 cured resin. In the case of SR1125, however, only single tests were run to protect the igniter spark and the cone heater since SR1125 cured resin exhibited extensive intumescence even though a grid was used on top of the specimen (see Section 3.1.3.2).

The fire performance of LEO and SR1125 laminates without a topcoat and cured resins in the cone calorimeter tests at the irradiance level of 50 kW/m² can be compared on the basis of Tables 3 and 10, respectively. In the case of laminates (Table 3), LEO shows lower heat release: HRR_{max} is 336 kW/m² for LEO and 546 kW/m² for SR1125, and THR is 33.5 MJ/m² for LEO and 42.5 MJ/m² for SR1125. When looking at cured resins (Table 10), the situation is opposite: HRR_{max} is

724 kW/m² for LEO and 291 kW/m² for SR1125, and THR is 74.3 MJ/m² for LEO and 59.0 MJ/m² for SR1125. This can be explained by the intumescence behaviour of the cured resins. SR1125 resin had a strong tendency to intumescence, forming a porous, insulating layer on the surface of the specimen which protected the underlying material. LEO resin did not show intumescence, which led to more severe burning. In the case of the laminates, the reinforcing fibres stabilize the matrix, and no intumescence occurs for either of the materials studied. In this case, LEO shows better heat release performance than SR1125.

Specimen	Irradiance (kW/m ²)		t _{ig} (s)	HRR _{max} (kW/m ²)	THR (MJ/m ²)	TSP (m ²)	ΔH _{c, eff} (MJ/kg)
LEO	35	Test 1	83	682	69.8	22.2	22.0
		Test 2	87	766	78.9	23.1	26.8
		Average	85	724	74.3	22.6	24.4
	50	Test 1	52	893	77.0	24.8	20.6
		Test 2	53	840	90.3	26.8	20.3
		Average	53	866	83.7	25.8	20.4
SR1125	25	Test 1	151	139	17.2	5.9	10.2
	50	Test 1	42	291	59.0	14.7	17.5

Table 10 - Cone calorimeter test results of cured resins; irradiances 25, 35 and 50 kW/m²

The cone calorimeter test results of cured resins support pyrolysis modelling and thermomechanical analyses, and give input to fire simulations to be performed in WP4.

4.2.2. MCC tests

Eight MCC experiments were performed for both sample materials: Two nominal heating rates (20 and 60 K/min), two purge gases (inert and oxidative), and two repeated experiments for each test. The specimens were cured resins, without fibre reinforcement and coating.

4.2.2.1. Leo system

The experimental MCC results for LEO System can be seen in Figure 16 and the comparison between inert and oxidative pyrolysis in Figure 17. The numerical results (total heat release (Q), effective heat of combustion (Q_{eff}) and total mass loss (Δm)) are listed in Table 11. There are no significant differences between the two repetitions. The reactions occur slightly earlier at the lower heating rate, which is typical for MCC results. In inert atmosphere (N₂) the heat release has two peaks, but the experiments with oxidative atmosphere (air) show an additional peak at high temperatures (500–700 °C). This indicates char oxidation. The total heat release seems to be a bit higher for the experiments in N₂ than in air.

	Q (MJ/kg)	Q _{eff} (MJ/kg)	Δm (%)
N ₂ 20 K/min	26.01	29.43	88 %
N ₂ 60 K/min	26.37	27.50	96 %
Air 20 K/min	25.60	26.51	97 %
Air 60 K/min	25.71	25.71	100 %

Table 11 - Numerical MCC results for LEO system.

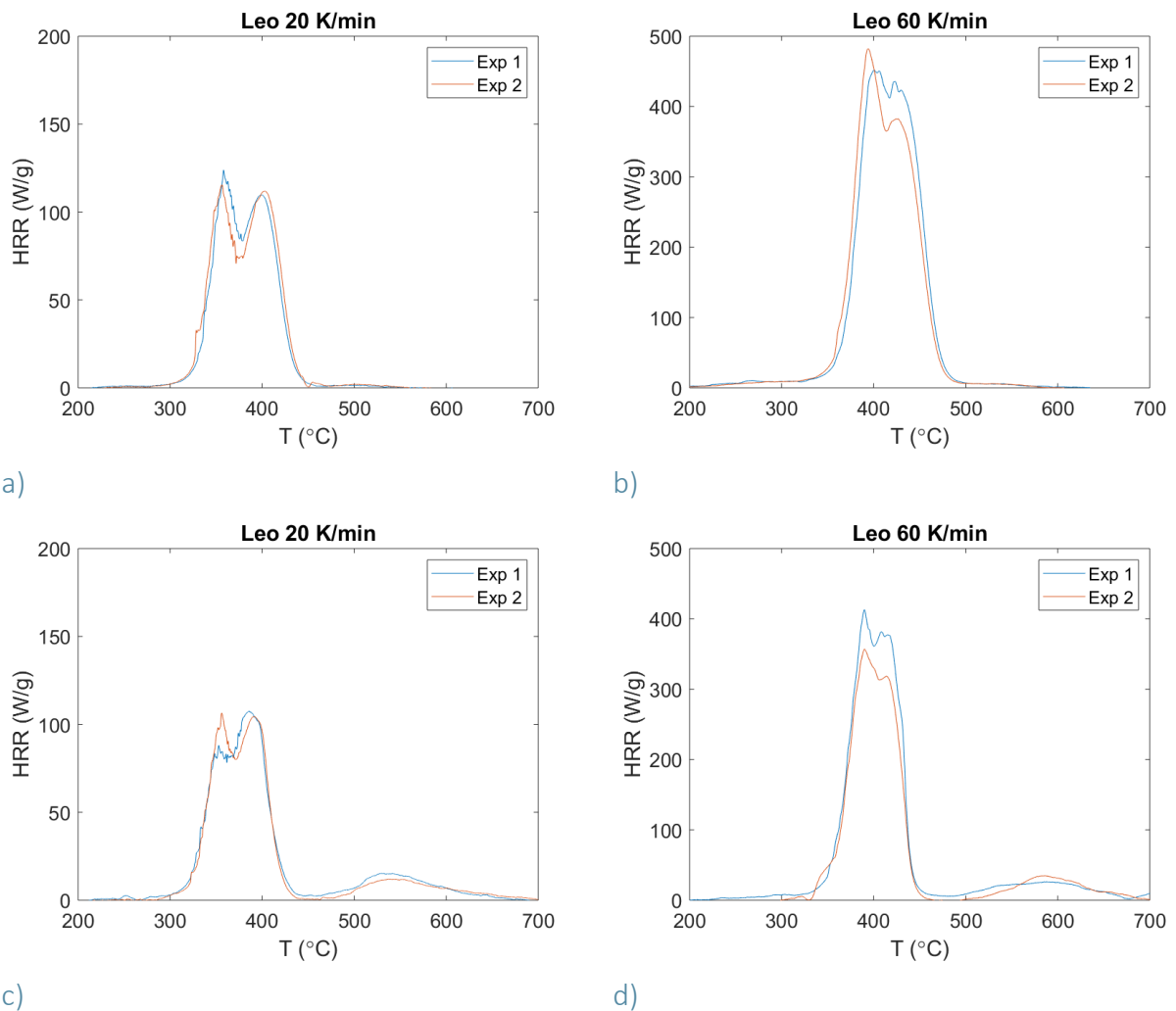


Figure 16 - MCC results for LEO system: a) 20 K/min in N₂, b) 60 K/min in N₂, c) 20 K/min in air, d) 60 K/min in air.

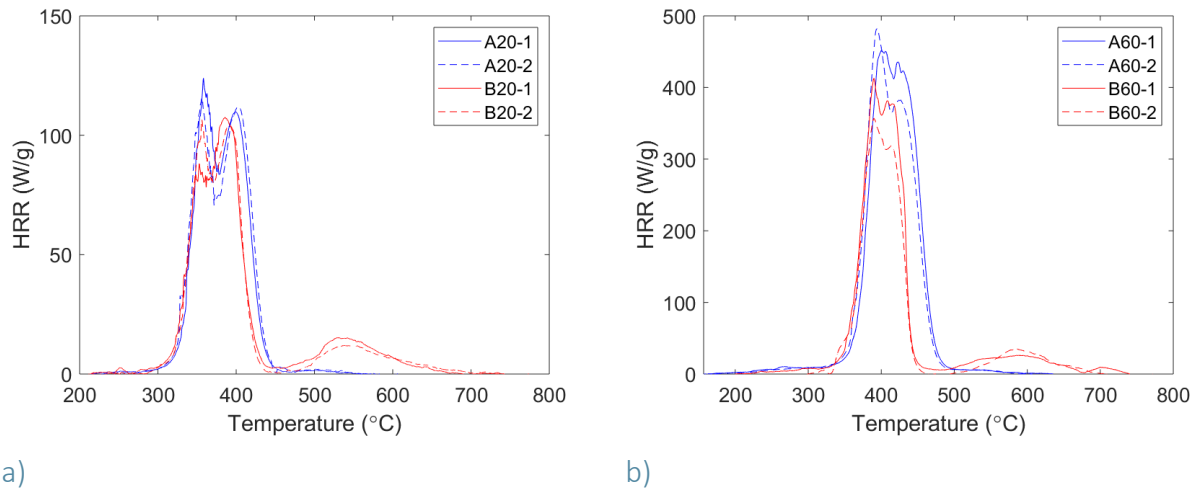


Figure 17 - Comparison of LEO system MCC results in N₂ (A) and air (B): a) 20 K/min, b) 60 K/min.

4.2.2.2. SR1125

The experimental MCC results for SR1125 can be seen in Figure 18 and the comparison between inert and oxidative pyrolysis in Figure 19. The numerical results (total heat release (Q), effective heat of combustion (Q_{eff}) and total mass loss (Δm)) are listed in Table 12. For 60 K/min in N₂ and 20 K/min in air the results look a bit different between the repetitions. In inert atmosphere (N₂) the heat release has one higher peak, and a lower tail which indicates another reaction, but the experiments with oxidative atmosphere show 1–2 additional peaks at high temperatures (500–900 °C). This indicates char oxidation. The values for total heat release seem to be the same order of magnitude for the other results, except for 60 K/min in air, for which the total heat release is significantly higher than for the other experiments.

	Q (MJ/kg)	Q _{eff} (MJ/kg)	Δm (%)
N ₂ 20 K/min	21.84	26.27	83 %
N ₂ 60 K/min	21.78	24.79	88 %
Air 20 K/min	20.06	20.99	95 %
Air 60 K/min	28.79	29.41	98 %

Table 12 - Numerical MCC results for SR1125.

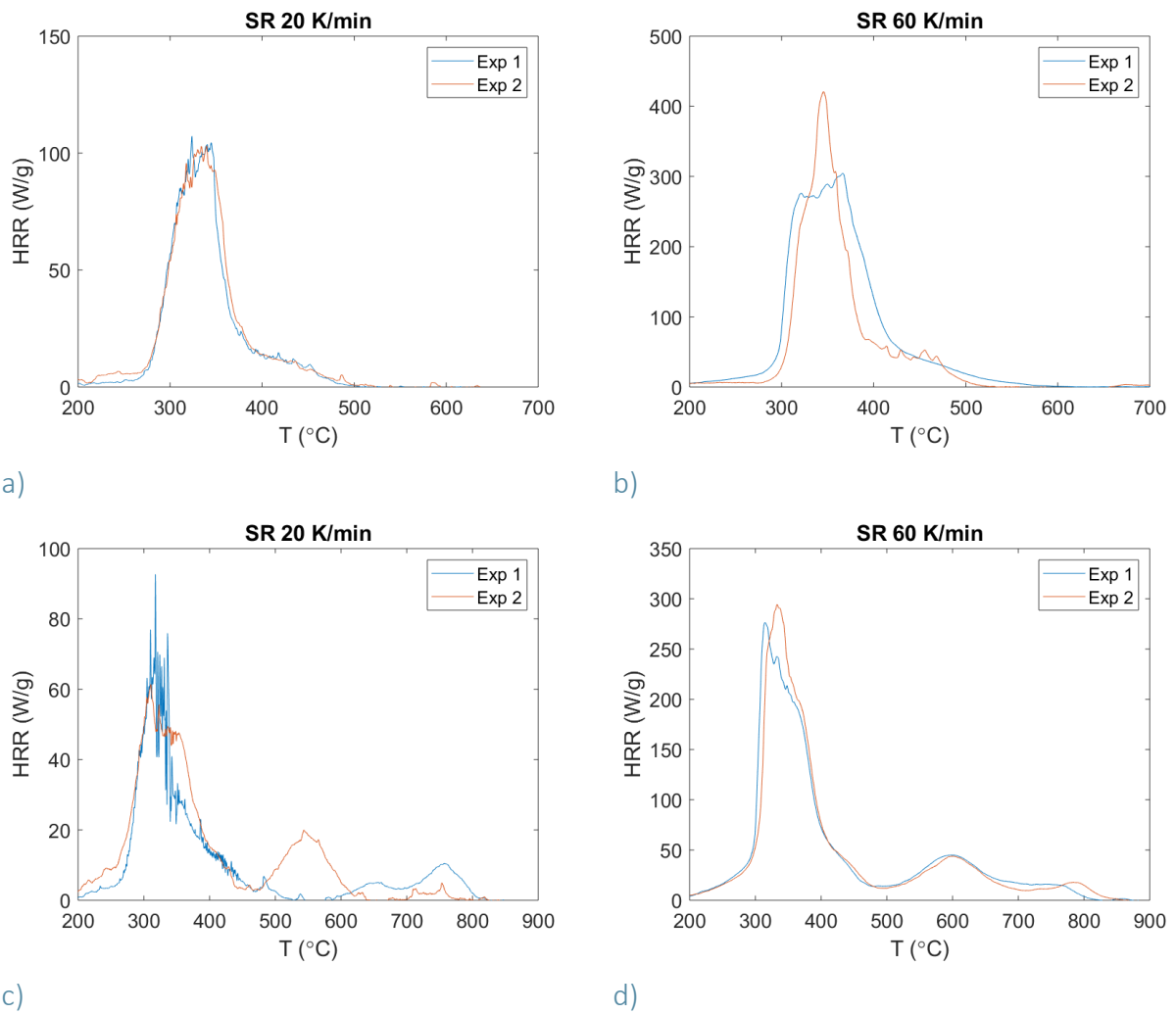


Figure 18 - MCC results for SR1125: a) 20 K/min in N₂, b) 60 K/min in N₂, c) 20 K/min in air, d) 60 K/min in air.

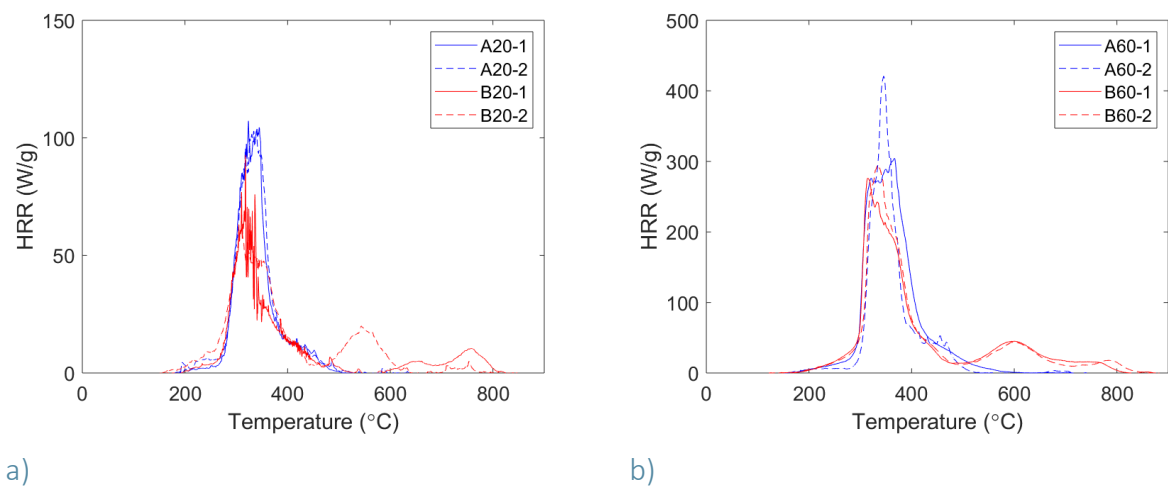


Figure 19 - Comparison of SR1125 MCC results in N₂ (A) and air (B): a) 20 K/min, b) 60 K/min.

4.2.3. DMTA tests

Dynamic Mechanical Thermal Analysis results: shear storage modulus and loss factor (tan delta) as the function of temperature with 1 Hz frequency are presented in the following figures for resin systems, LEO system (Figure 20) and SR1125 (Figure 21).

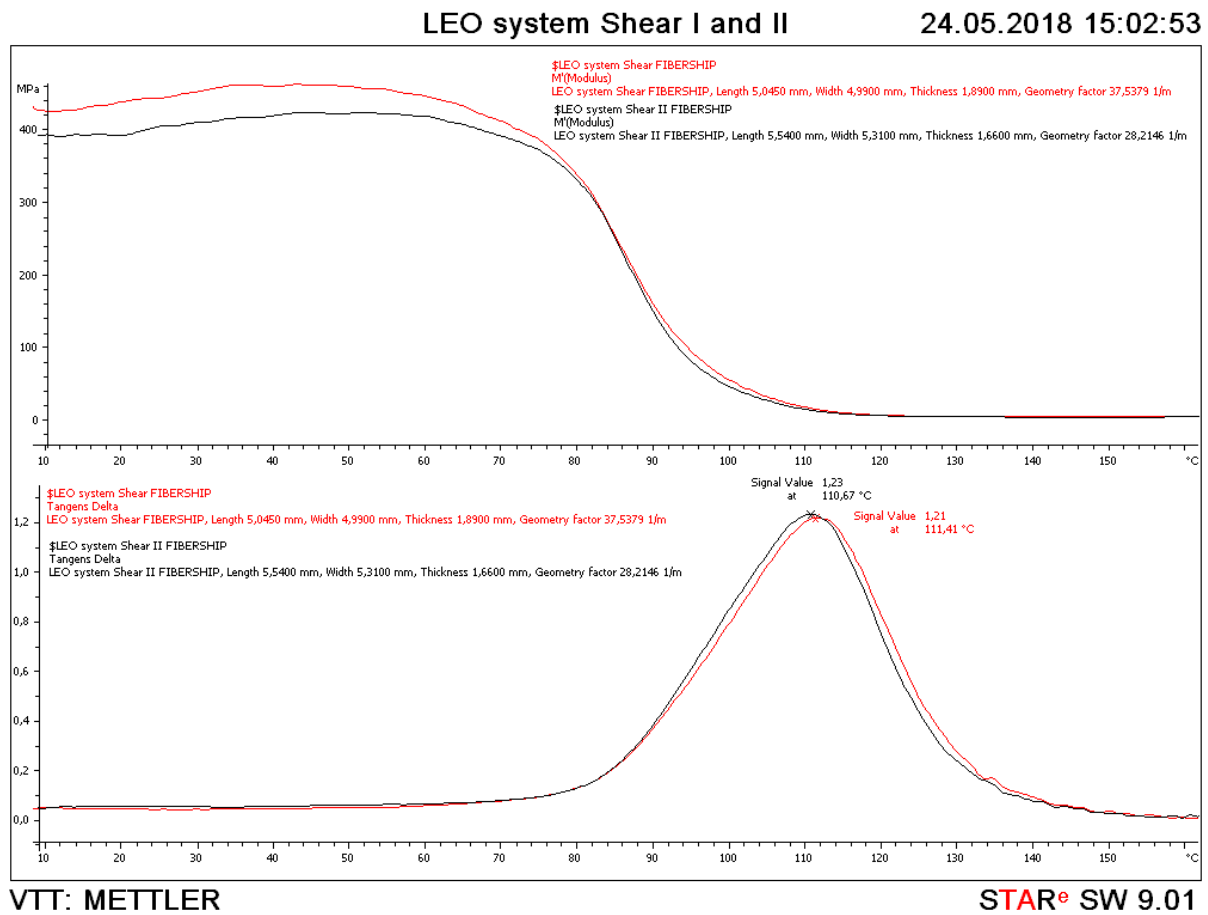


Figure 20 - Storage modulus (in MPa) and loss factor (tan delta) values as the function of temperature with 1 Hz frequency for LEO System samples in shear.

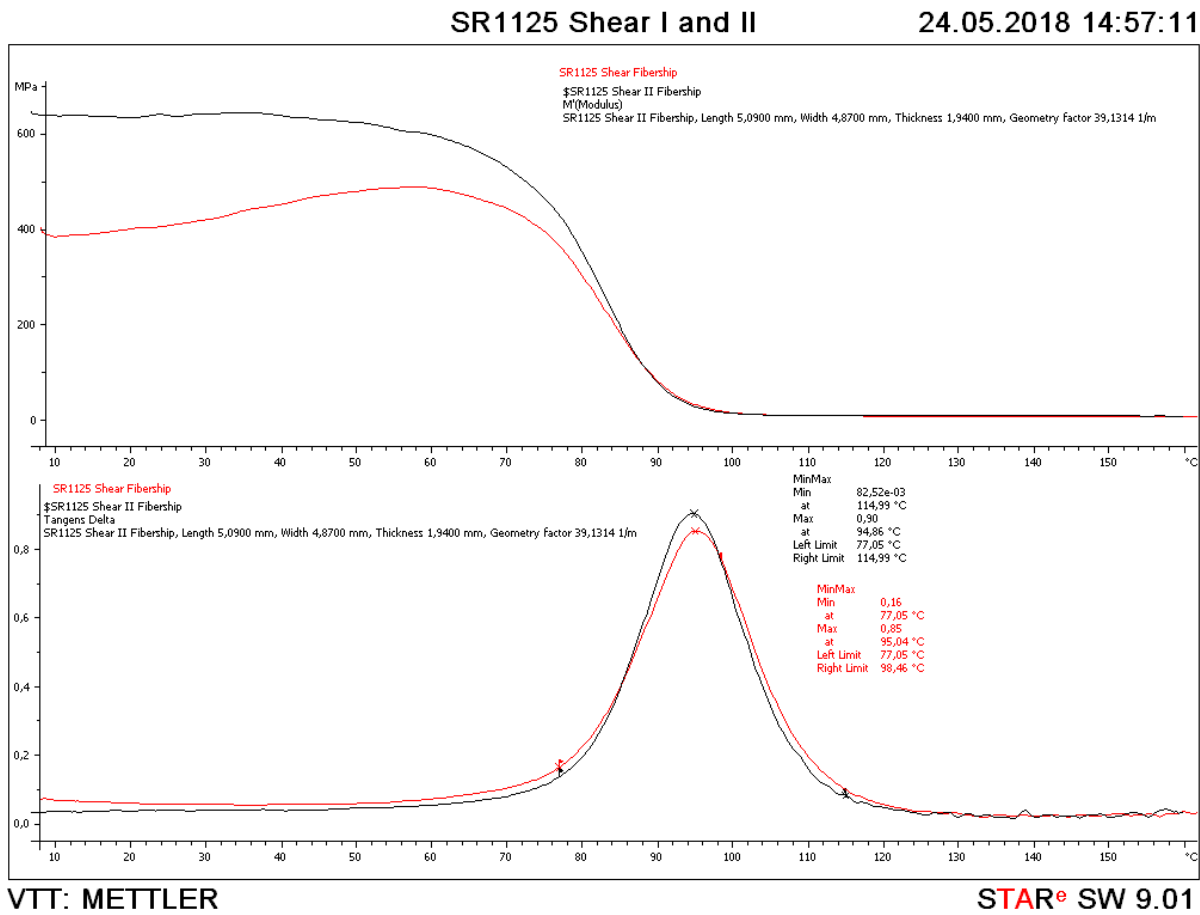


Figure 21 - Storage modulus (in MPa) and loss factor (tan delta) values as the function of temperature with 1 Hz frequency for SR1125 samples in shear.

Results show the glass transition temperature (T_g) for LEO system at approximately 111°C determined as peak value in tan delta curve. For SR1125, T_g is lower, peak temperature detected at approximately 95°C. For SR1125 there is variation between the samples in storage modulus values below glass transition temperature. This is most probably caused by insufficient sample tightening or some mismatch in sample dimensions for sample 2 (caused by uneven loading).

Dynamic Mechanical Thermal Analysis results for reinforced systems are shown in Figure 22 and Figure 23 for LEO system and in Figure 24 for SR1125 measured in three point bending mode.

LEO system 3-point bending

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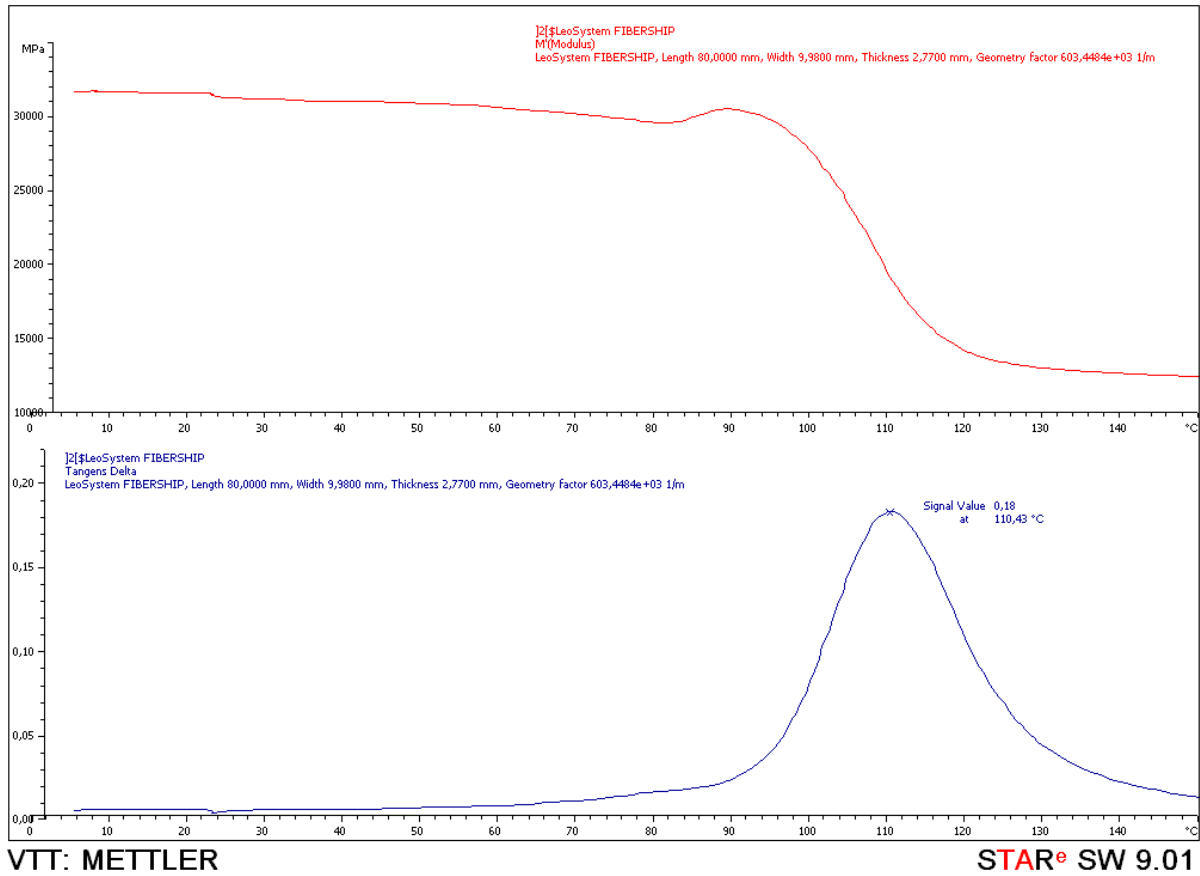
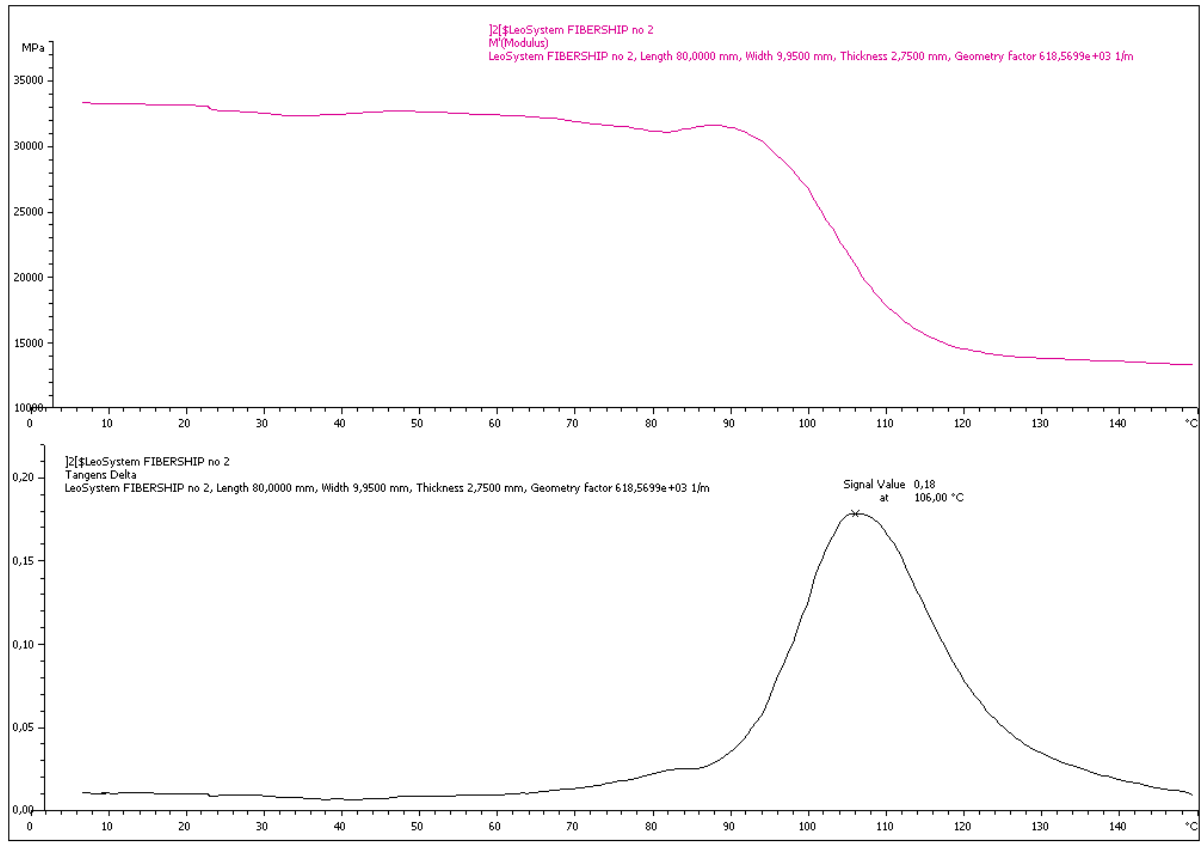


Figure 22 - Storage modulus (in MPa) and loss factor (tan delta) values as the function of temperature with 1 Hz frequency for LEO system sample measured in three point bending.

LEO system 3-point bending II

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Figure 23 - Storage modulus (in MPa) and loss factor (tan delta) values as the function of temperature with 1 Hz frequency for LEO system sample measured in three point bending.

SR1125 3-point bending I and II

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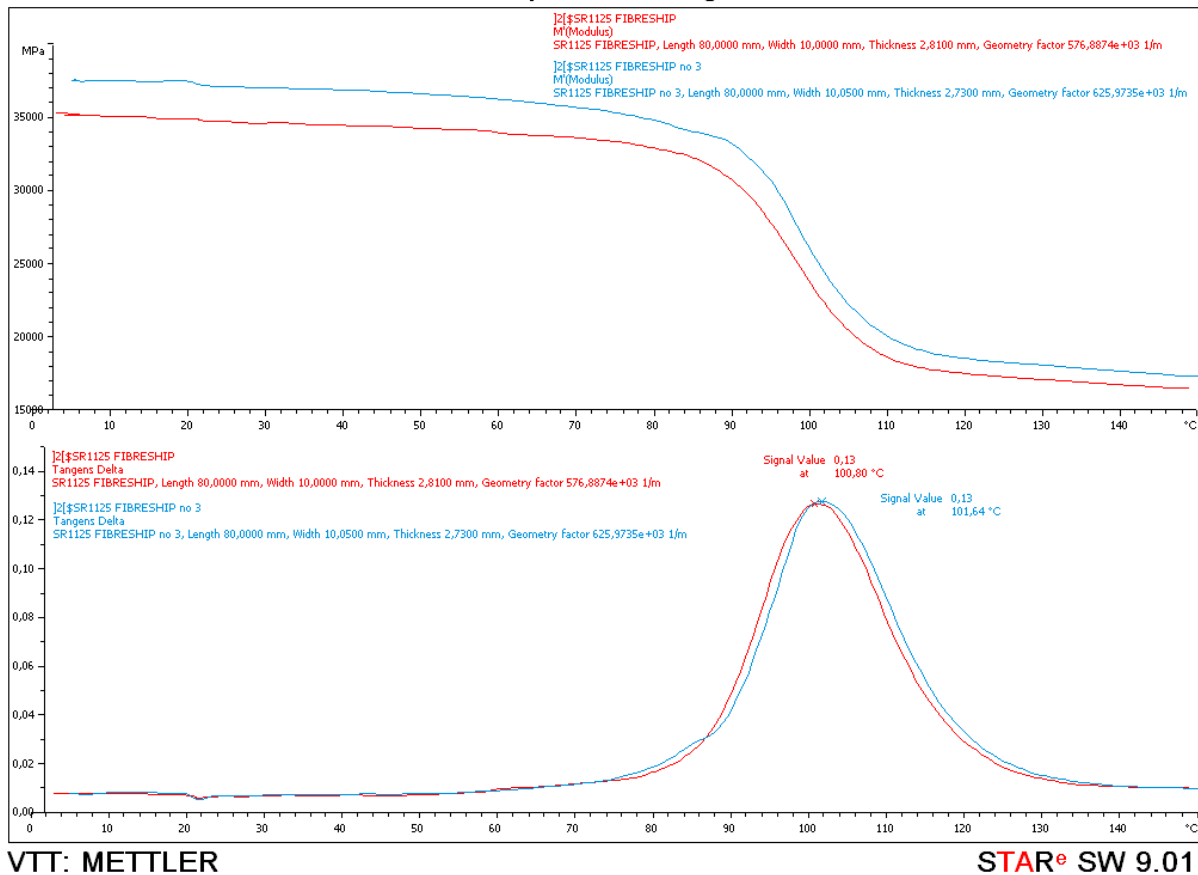


Figure 24 - Storage modulus (in MPa) and loss factor (tan delta) values as the function of temperature with 1 Hz frequency for SR1125 samples measured in three point bending.

4.2.4. DSC tests

The results of the DSC tests of the cured resins are presented in Figures 25 and 26 for LEO and SR1125 cured resins, respectively.

Glass transition temperatures T_g were determined on the basis of reverse heat flow curves of DSC measurements. T_g values obtained were ca. 69 °C for LEO and ca. 80 °C for SR 1125. Since these results differ significantly from the values obtained in DMTA measurements (111 °C for LEO and 95 °C for SR1125, see Section 4.2.3), a second DSC run was performed for one specimen of both materials; i.e. the same specimen which had gone through heating in DSC was heated another time. From these second runs, T_g values of ca. 112 °C for LEO and ca. 108 °C for SR1125 were determined. The observation can refer to incomplete curing in the manufacturing process, emphasizing the importance of manufacturing process control.

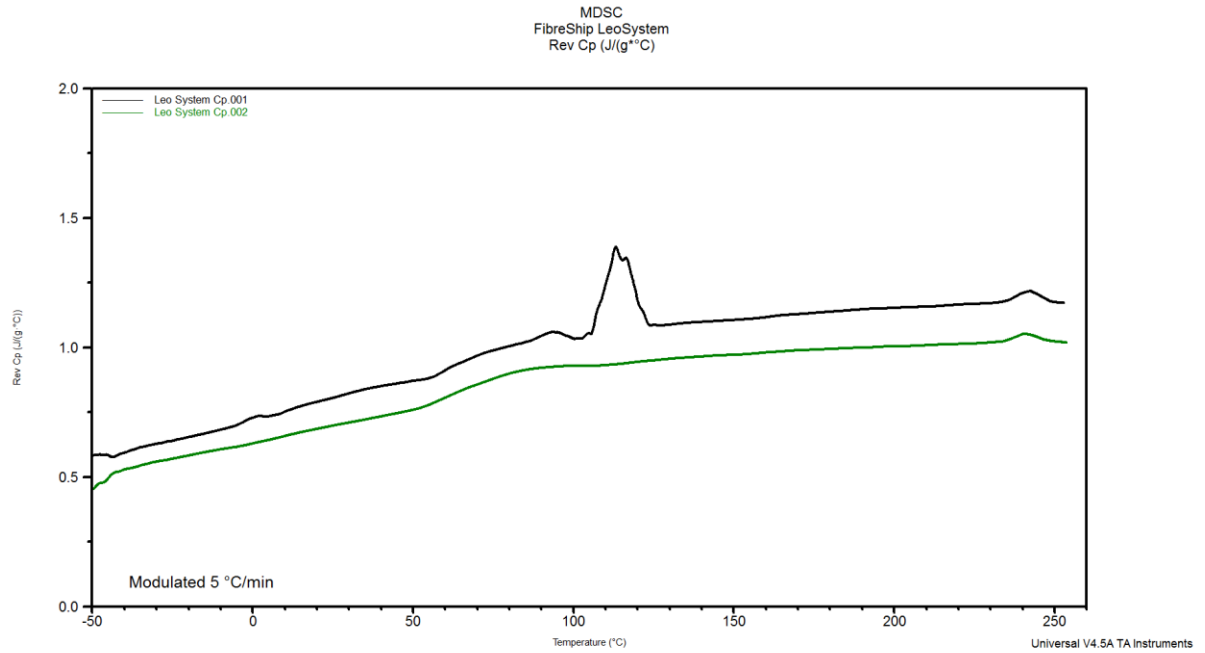


Figure 25 - Specific heat capacity Cp (J/(g °C)) as a function of temperature (°C) for LEO resin. The peak in the upper curve is a measurement artefact.

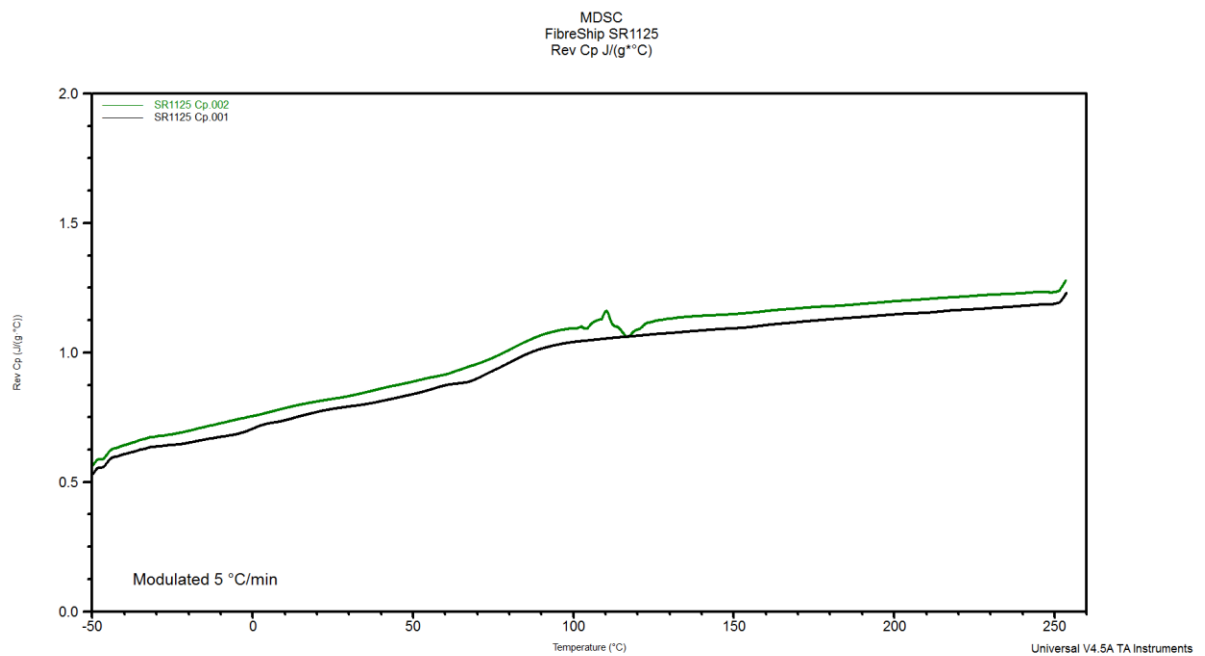


Figure 26 - Specific heat capacity Cp (J/(g °C)) as a function of temperature (°C) for SR1125 resin.

4.2.5. TPS tests

The results of TPS tests of the composite laminates are summarized in Table 13. Previously measured density and volumetric heat capacity were used for calculating anisotropic axial (through the sample) and radial (along the sample) thermal conductivities and diffusivities.

Quantity	LEO	SR1125
Isotropic measurement		
Thermal conductivity (W/(m K))	0.49	0.52
Thermal diffusivity (mm ² /s)	0.32	0.34
Volumetric heat capacity (MJ/(m ³ K))	1.53	1.52
Anisotropic measurement		
Axial thermal conductivity (W/(m K))	0.5135	0.4881
Axial thermal diffusivity (mm ² /s)	0.3522	0.3081
Radial thermal conductivity (W/(m K))	0.4560	0.5424
Radial thermal diffusivity (mm ² /s)	0.3127	0.3424

Table 13 - TPS test results of composite laminates.

5. CONCLUSIONS

In Task 2.4 of the FIBRESHIP project, an extensive experimental campaign was performed in two phases to characterize the fire performance of fibre reinforced polymer materials and solutions.

For the first phase, seven commercially available resins or resin systems were selected for examination of fire performance. Laminates with glass fibre reinforcement and cured resins without reinforcement were produced for cone calorimeter tests and thermogravimetric analyses, respectively. From these seven candidates, two materials were down selected on the basis of the mechanical performance, manufacturability and impact (including cost, claimed fire retardancy, worker health impact and recyclability).

The two material solutions chosen to continue to the second phase were LEO vinylester resin system and SR1125 epoxy resin system. The fire tests of the first phase showed that an intumescent coating on the surface of these laminates is essential for providing adequate fire performance.

In the second phase, a more comprehensive evaluation of thermal and fire properties was performed by carrying out more cone calorimeter tests, as well as dynamic mechanical thermal analysis, microscale combustion calorimetry, differential scanning calorimetry and transient plane source tests. Simultaneously, data for pyrolysis modelling, thermomechanical modelling and fire dynamics simulations was produced.

In cone calorimeter test at the irradiance of 50 kW/m², the times to ignition of coated LEO and SR1125 were 75 and 52 seconds in average, respectively. The maximum heat release was 261 kW/m² for SR1125, but only 69 kW/m² for LEO indicating good reaction-to-fire performance. The total heat release and the total smoke production were ca. 40 MJ/m² and 9 m², respectively, for both systems.

Cone calorimeter tests of coated laminate specimens were run also at the irradiance levels of 25 and 35 kW/m² and for specimens representing different production batches. The results were not consistent in all cases. In addition, DSC tests revealed changes of the glass transition temperature when the specimens were re-heated, referring to incomplete curing in the manufacturing process. These observations highlight the importance of repeatable and well-controlled manufacturing process. The whole process must be carefully instructed, monitored and reported. The laminates and coatings must be of uniform quality to ensure the fire performance claimed on the basis of fire tests performed. Precise specifications and quality control play a key role in securing the fire safety of materials and products.

Thermogravimetric analyses and micro-scale combustion calorimetry showed that the mass loss of cured resins typically starts slightly above 300 °C both in inert (N₂) and oxidative (air) atmosphere. The reactions in these atmospheres differ, the oxidative atmosphere revealing also oxidative reactions. At about 300 °C, however, a structure made of fibre reinforced polymer materials starts to produce combustible gases and contribute to fire.

Dynamic mechanical thermal analysis showed that the glass transition temperature T_g is ca. 111 °C for LEO, and ca. 95 °C for SR1125. In general, the glass transition temperatures of fibre reinforced polymer materials are typically about 100 °C. At this temperature, the material softens and loses its loadbearing capacity.



Structures made of fibre reinforced polymer materials have a tendency to heat up locally, due to their relatively low thermal conductivity. In the case of a local fire, combustible gas production and heat release are the main concerns in terms of fire safety. If the fire threatens a large structure, like in the case of a compartment fire, the main problem is the softening of the material and the loss of the loadbearing capacity.

6. REFERENCES

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- [3] Drysdale, D. (2011) An Introduction to Fire Dynamics. 3rd Edition. John Wiley & Sons, Ltd, UK.
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ENGINEERING, PRODUCTION AND LIFE-CYCLE MANAGEMENT FOR THE COMPLETE CONSTRUCTION OF LARGE-LENGTH FIBRE-BASED SHIPS

Annexes to D2.4 (WP2)

Report and database on the results of the fire performance
experiments

Responsible Partner: VTT

Contributor(s): CIMNE, ULIM, BV, LLOYDS, RINA



LIST OF ANNEXES

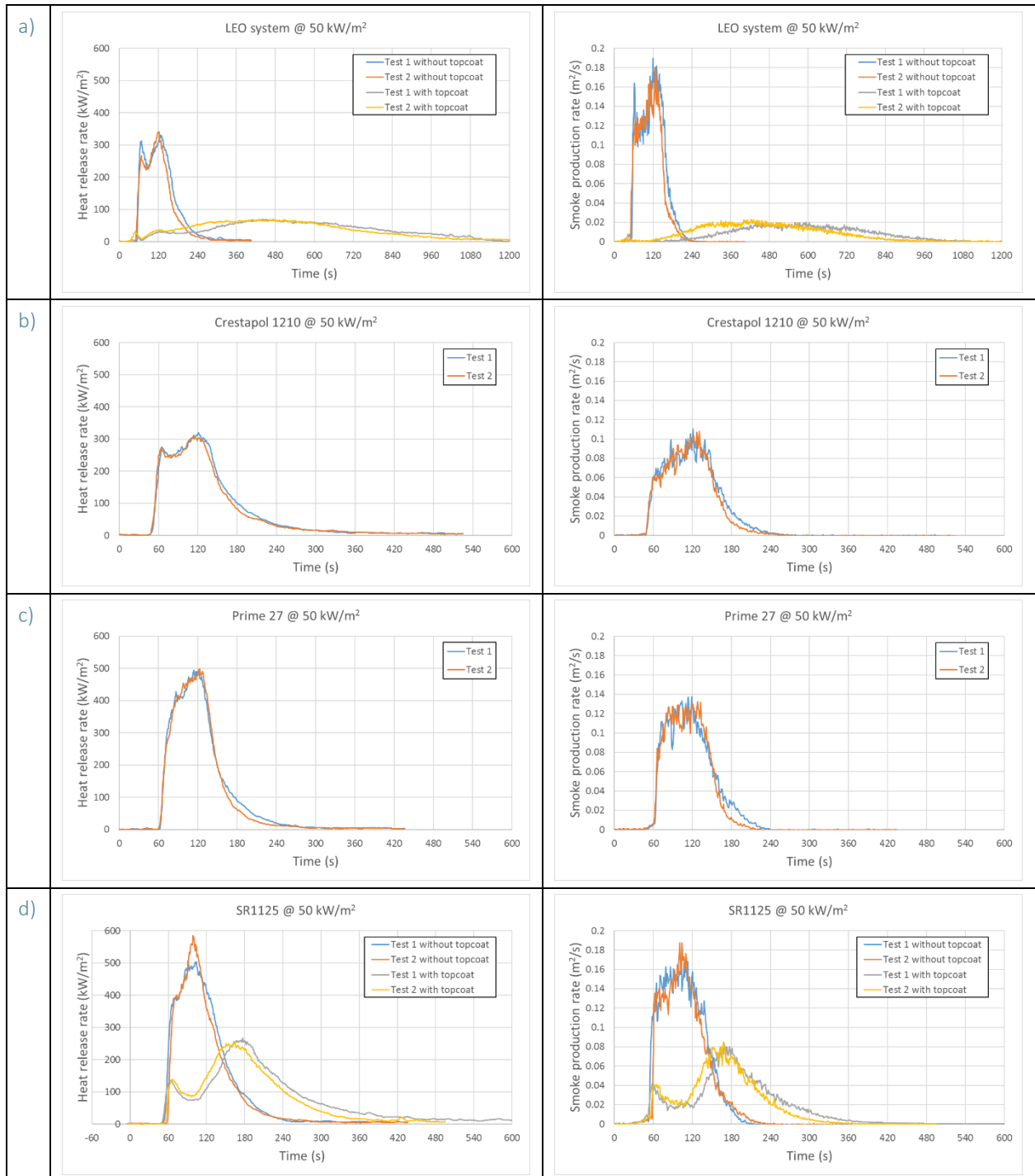
ANNEX 1: Phase 1 cone calorimeter graphs

ANNEX 2: Phase 1 TGA graphs

ANNEX 3: Phase 2 cone calorimeter graphs

ANNEX 4: Practical Guide of Application of Coating KCICF

ANNEX 1: Phase 1 cone calorimeter graphs



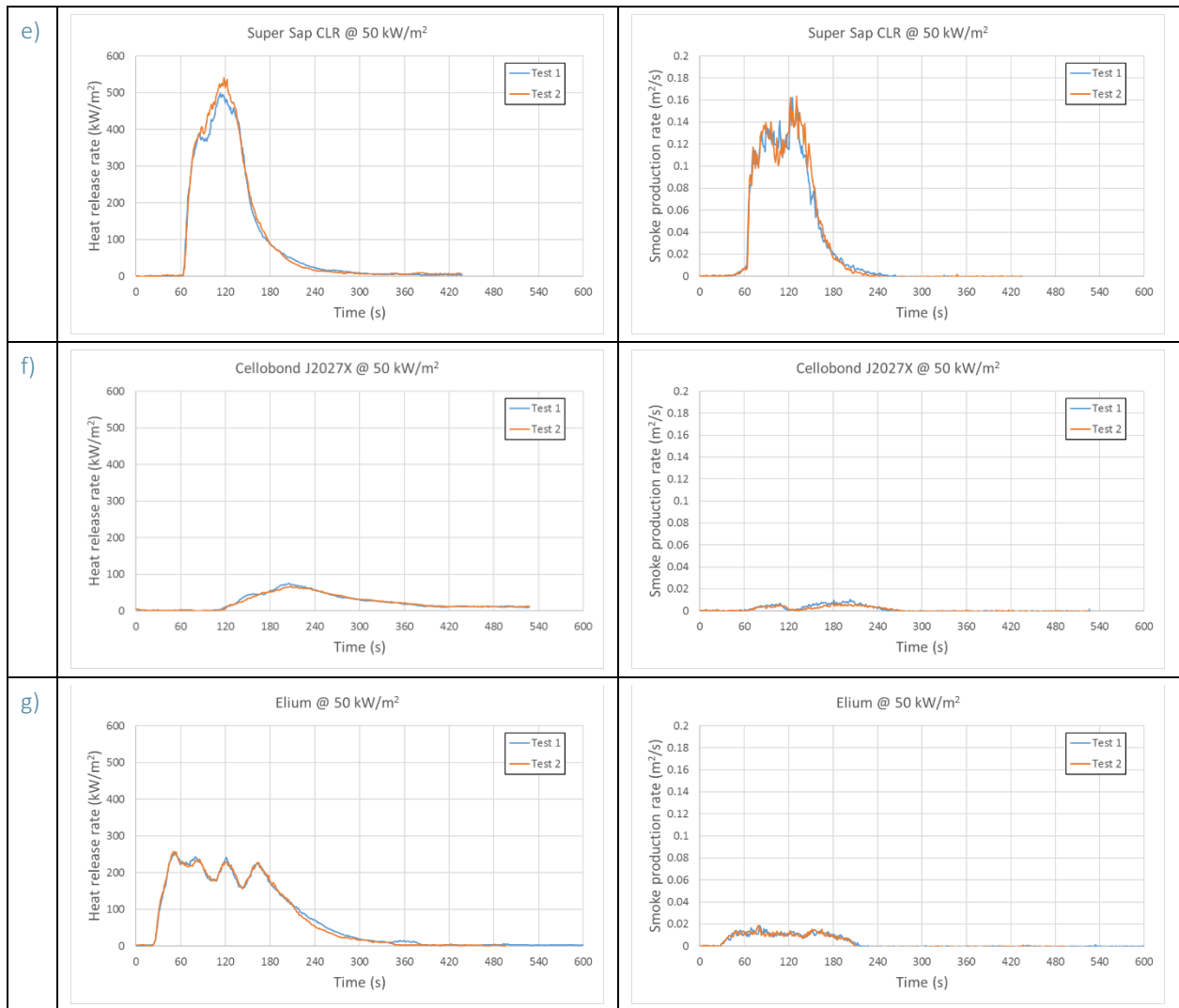


Figure A1-1. Heat release rate (left) and smoke production rate (right) results of composite laminates in cone calorimeter tests with an irradiance of 50 kW/m²: a) LEO system with and without a topcoat, b) Crestapol 1210, c) Prime 27, d) SR1125 with and without a topcoat, e) Super Sap CLR, f) Cellobond J2027X, and g) Elium. Note that the time scale of LEO system graphs is double compared to other graphs.

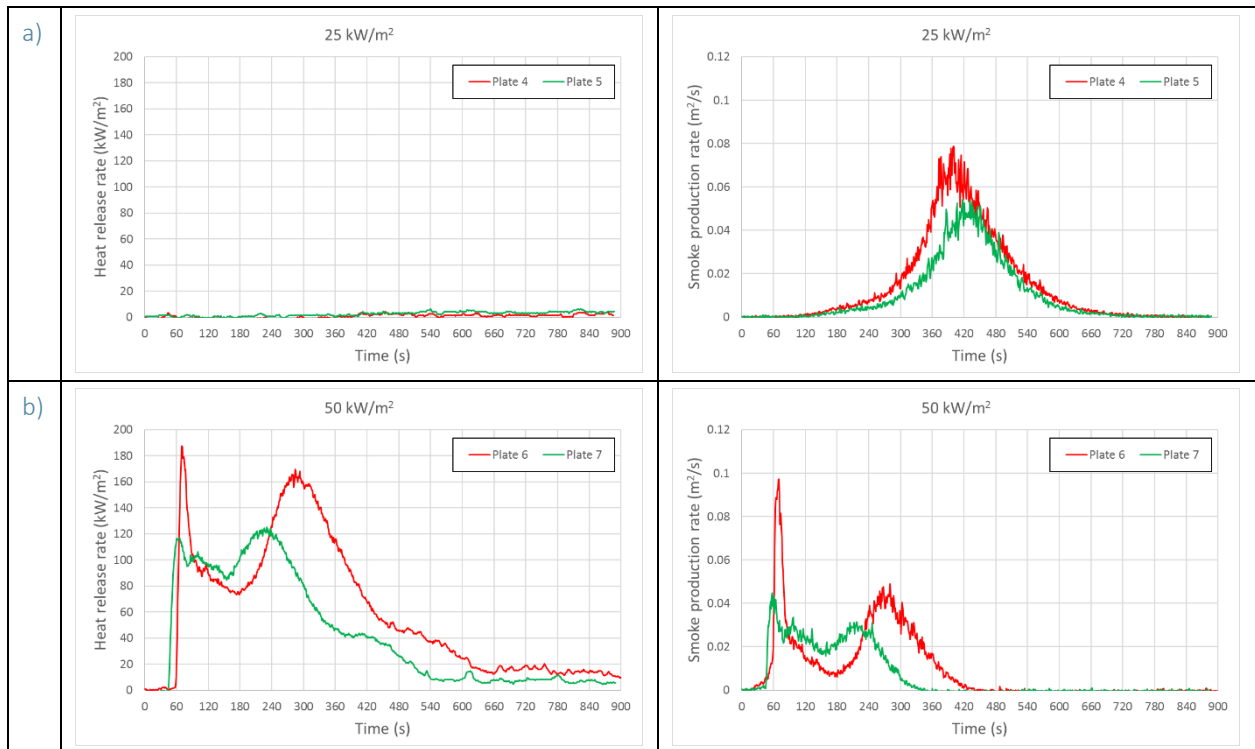
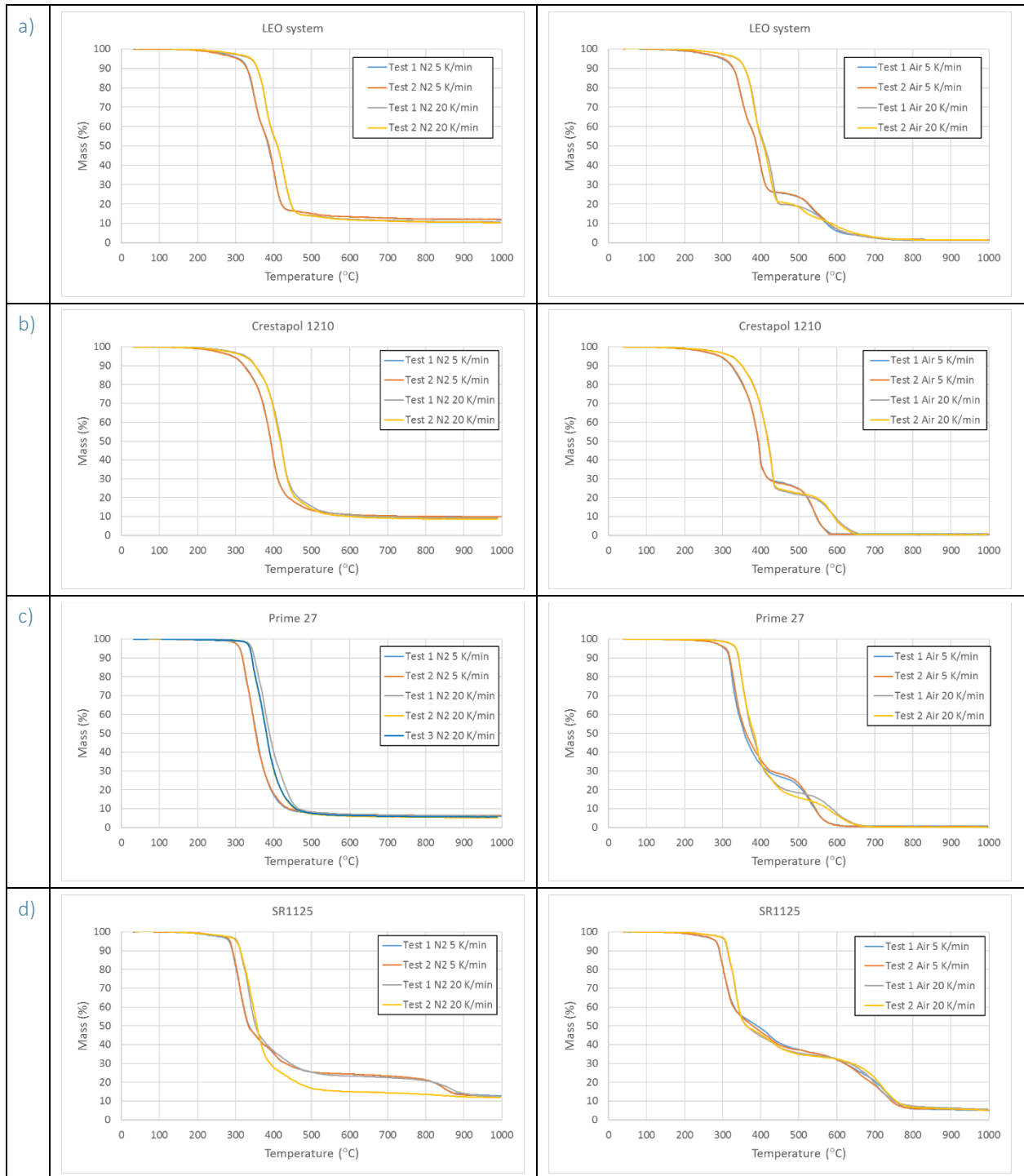


Figure A1-2. Heat release rate (left) and smoke production rate (right) results of CIMNE coating in cone calorimeter tests with an irradiance of a) 25 kW/m² and b) 50 kW/m².

ANNEX 2: Phase 1 TGA graphs



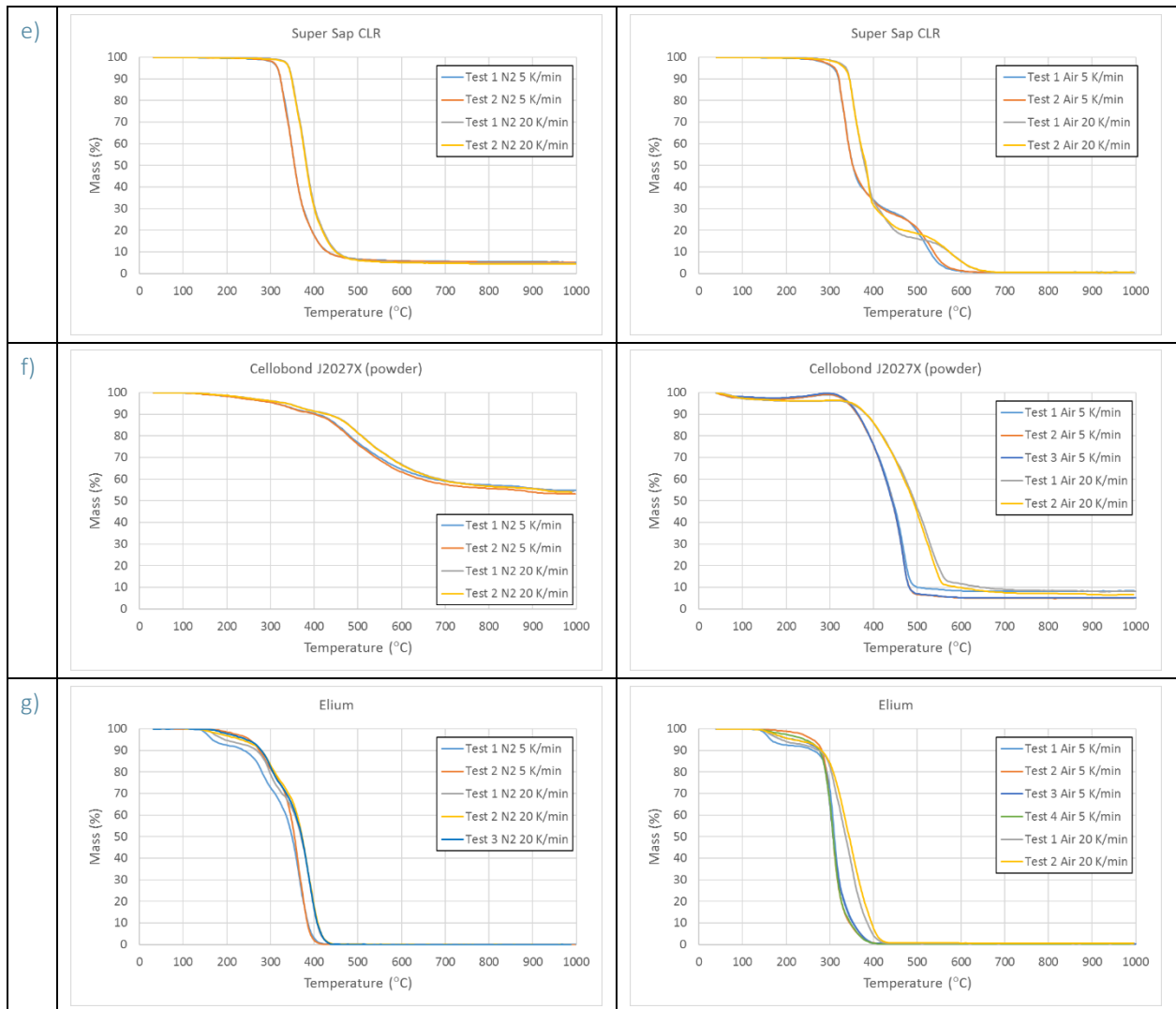


Figure A2-1. TGA curves of cured resins in N₂ and in air at heating rates of 5 and 20 K/min: a) LEO system, b) Crestapol 1210, c) Prime 27, d) SR1125, e) Super Sap CLR, f) Cellobond J2027X, and g) Elium.

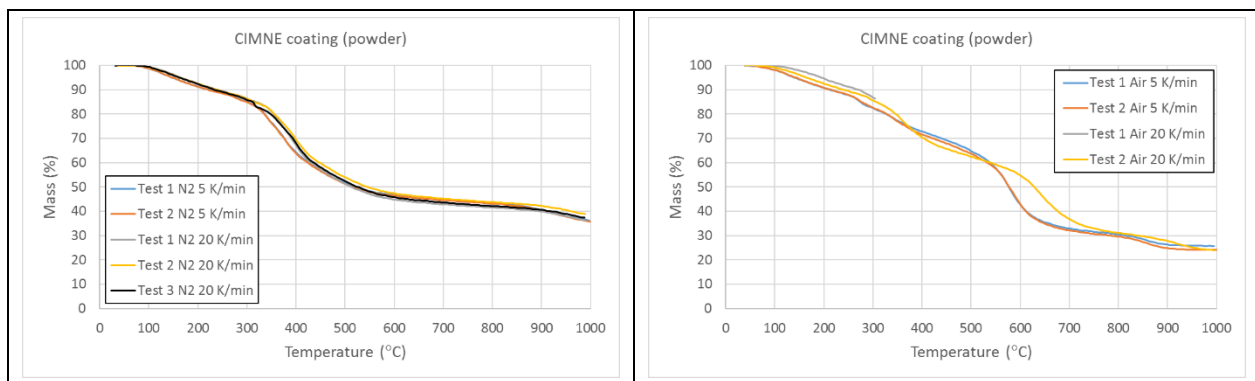
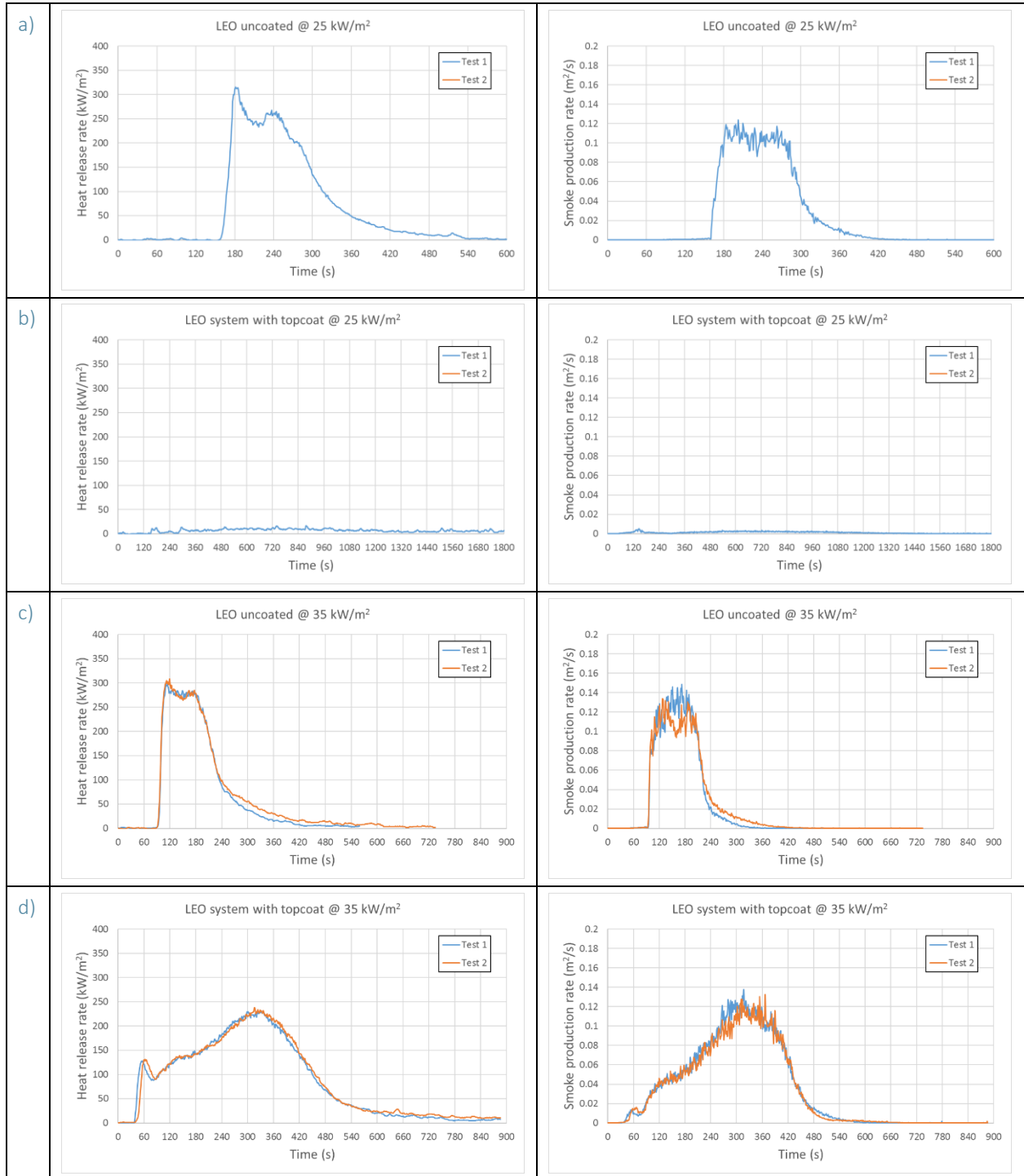


Figure A2-2. TGA curves of CIMNE coating powder in N₂ (left) and in air (right) at heating rates of 5 and 20 K/min. Test 1 in air at 20 K/min failed after T = 300 °C.

ANNEX 3: Phase 2 cone calorimeter graphs



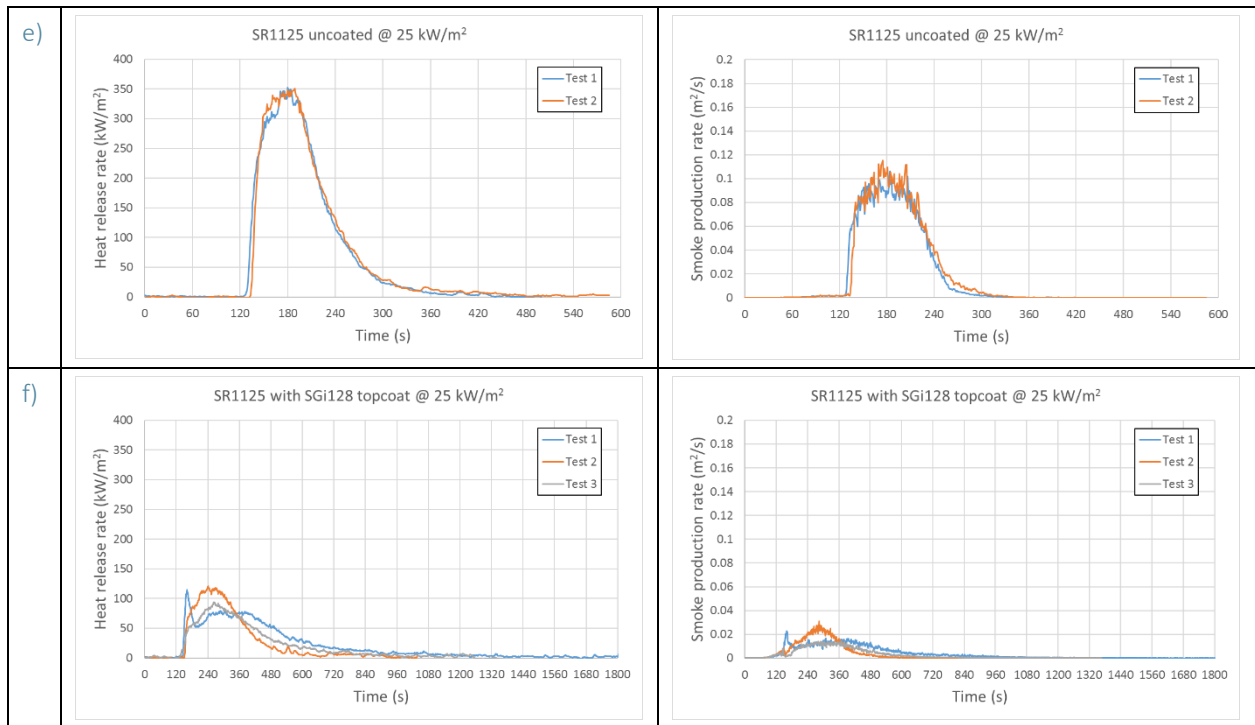


Figure A3-1. Heat release rate (left) and smoke production rate (right) results of composite laminates in Phase 2 cone calorimeter tests: a) LEO system without a topcoat at 25 kW/m², b) LEO system with a topcoat at 25 kW/m², c) LEO system without a topcoat at 35 kW/m², d) LEO system with a topcoat at 35 kW/m², e) SR1125 without a topcoat at 25 kW/m², and f) SR1125 with SGi 128 topcoat at 25 kW/m².

Note that the time scales of the graphs vary.

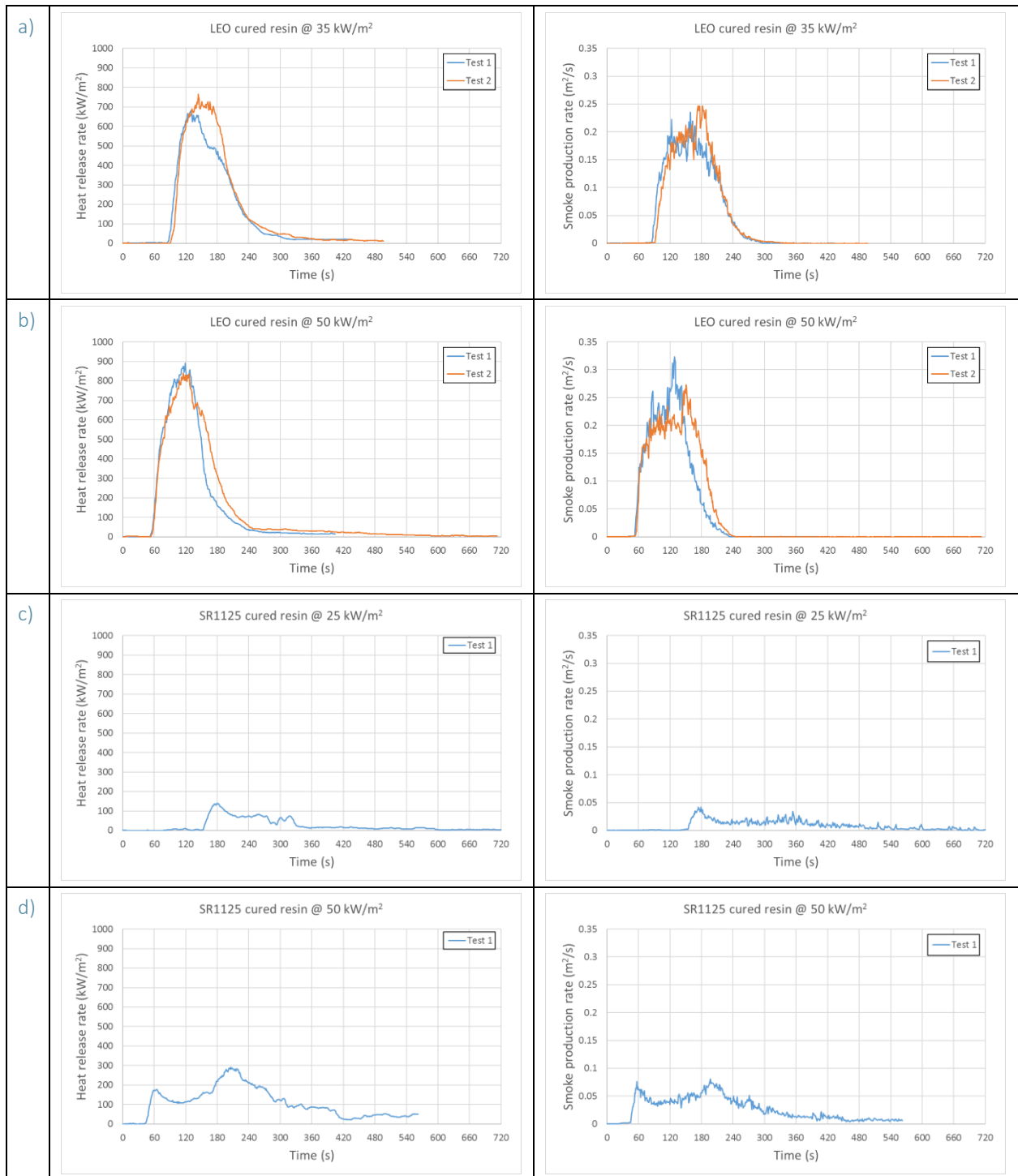


Figure A3-2. Heat release rate (left) and smoke production rate (right) results of cured resins in Phase 2 cone calorimeter tests: a) LEO at 35 kW/m², b) LEO at 50 kW/m², c) SR1125 at 25 kW/m², and d) SR1125 at 50 kW/m².

ANNEX 4: Practical Guide of Application of Coating KCICF

PRACTICAL GUIDE OF APPLICATION OF COATING KCICF



EXECUTIVE SUMMARY

This document is a practical guide of application of coating KCICF that has been developed by CIMNE and tested by VTT. This document describes the advice on application of KCICF intumescent coating.

NOTICE AND STATEMENTS

Fire protection performance and pre-fire durability are critically dependent on the correct application of the system. These guidance notes are intended to assist applicators and are for guidance only; CIMNE accepts no liability for the acts or omissions of any applicators of Cimne KCICF. It's assumed that applicators of Cimne KCICF understand the terminology associated with epoxy intumescent coatings and the various pieces of spray equipment, hand/trowel and application techniques. The installation of Cimne KCICF shall be performed only by contractor personnel trained or qualified in the installation of these materials.

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1. INTRODUCTION

This document describes the advice on application of KCICF intumescent coating. This document may be used by applicators as a guideline on which to base their own quality control procedure. CIMNE can aid in compliance with this practical guide however are not accountable as project inspectors. The accountability for inspection and quality control is the responsibility of the applicator and fabricator quality control departments.

Both fire protection performance and pre-fire durability critically depend on the correct application of the fireproofing system. A scope of the present practical guide is to define the activities required to verify that the work is carried out.

This document is subject to the following:

1. Compliance with standards concerning fire resistance
2. All equipment being suitable and fully serviced prior to use for Cimne applications
3. All applicators being trained, competent and certified in the application of fire protection epoxy intumescent coatings
4. All Health & Safety recommendations of the equipment manufacturer being followed

This practical guide is verified by Cimne, and the contents are to be considered effective from the revision date.

The registration of the verification and approval of every edition of this practical guide and the original document are held in the archives of the office at Barcelona, which has the responsibility of conserving the document in its approved state and of distributing copies that conform to the last deposited revision. The previous editions are also held in the archives, separately, and conserved for possible consultation.

This practical guide is distributed internally and externally with the aim of providing consistent information to all parties involved in the application of Cimne KCICF.

This practical guide uses metric (SI).

The information provided in this practical guide is not intended to be exhaustive; any person applying the product by any method other than that specifically recommended in this practical guide without first obtaining our written confirmation as to the suitability of the proposed method does so at their own risk. Cimne tries very hard to ensure that all advice Cimne gives regarding application of KCICF (whether in this practical guide or otherwise) is correct but we have no control over either the quality or condition of the substrate or the many factors affecting application of the product. In the event of any doubts or misunderstandings, Cimne should be consulted for clarification.

2. GENERAL CONDITIONS

2.1. SCOPE

This practical guide describes the requirements for the application of the coating KCICF to composite material for vessel market. For the application of Cimne KCICF to other substrates, also is detailed on this document.

2.2. QUALITY PRACTICAL GUIDE

2.2.1. QUALIFICATIONS OF APPLICATORS/RESPONSIBILITIES OF PERSONNEL

The application shall be performed by a Qualified Applicator having EU certification and training with proper equipment and experience.

2.2.2. REQUIREMENTS

In order to qualify, an Applicator shall:

- (a) Have an EU certificate on intumescent coating application.
- (b) Be experienced in the application of thick film coatings, preferably epoxy based.
- (c) Have the necessary approved spray application equipment and recommended quality control instrumentation.
- (c) Understand and recognize their statutory obligations with regard to health and safety.

2.3. SAFETY PRECAUTIONS

The Applicator shall follow standard industrial hygiene practices for the handling of chemical coatings and shall conform to applicable codes of practice, regulations, and Owner safety rules in all respects.

Where power tools, hand tools, spray equipment or other mechanical equipment are being used, the proper operating procedures for each tool or piece of equipment, as well as eye, hearing and respiratory protection should be followed. Any injury caused by high pressure liquids can be serious and immediate medical attention should be sought.

2.4. STORAGE

Cimne KCICF not in immediate use shall be stored off the ground in a covered area assigned for that purpose. The materials in storage shall be protected from temperatures above 38°C and below 0°C.

During storage, higher temperatures may be tolerated, however this may result in a significantly shorter shelf life. Product subject to higher than recommended conditions should be sprayed/applied with all parties present to verify acceptability of spray properties and surface appearance prior to use. To achieve the correct temperatures it may be necessary to use heated storage units, especially in cold and temperate climates.

Prior to use with plural or single component equipment, Cimne KCICF shall be heated to a minimum of 21°C and a maximum of 43°C.

2.5. AMBIENT CONDITIONS

Suitable application conditions are of critical importance for the successful application of KCICF.

Consequently, it should only be applied when the ambient conditions are within the following parameters:

- Air temperature: minimum 10°C, maximum 50°C
- Relative humidity: maximum 85%
- Surface temperature: at least 3°C above dew point temperature, less than 50°C for heated plural application and less than 60°C for hand application

KCICF may be applied at an air temperature minimum 5°C using plural spray equipment only, recognizing that the time to cure will be extended in such applications. This should only be undertaken where there is no alternative to modify the ambient conditions. Additionally, there may also be a higher propensity for amine bloom formation which would affect over-coating with further layers of KCICF, where required, and may affect over-coating with top-coats. For such low air temperature applications, plural equipment is the only recommended dispensing method.

In climates where there is direct sunlight with temperatures up to 50-60°C or there is a likelihood of condensation or airborne particulate contamination, application should be conducted in an encapsulated area and dehumidification equipment provided where required.

2.6. PROTECTION OF ADJACENT SURFACES

The applicator shall mask off all adjacent areas and equipment from material overspray during the application. Overspray shall be removed promptly before material has cured. When applying these coatings in windy conditions, additional precautions to control overspray should be undertaken.

3. MATERIAL DESCRIPTION

KCICF is a two component, thermally activated, intumescent epoxy coating designed to protect substrates from fire and corrosion. When exposed to flame, the material volatilizes at fixed temperatures, exhibits a volume increase through formation of a multicellular matrix and absorbs and blocks heat to protect the substrate material. Depending on the fire type, rating and design, KCICF may require reinforcement.

KCICF provides effective fire protection to a wide range of structures, vessels and equipment in almost any fire scenario. In a fire, it will intumesce (expand) to form an insulating layer of char. It is this char and its formation that protects the substrate from the effects of fire.

KCICF has been extensively tested to industry recognised standards to demonstrate its ability to withstand weather and environmental exposure. KCICF is able to be coloured with any

scheme and finish requested by the project. KCICF has a high hardness against scratches. KCICF has a brilliant and smooth finishing surface; the finishing surface quality is the highest possible.

4. EXTRA MATERIALS

Cimne KCICF is applied directly to blast cleaned substrates. But it's possible that due to the environmental factors such as temperature, time and humidity may make it necessary to prime the substrate, to prevent deterioration of the blast. Correct primer type and thickness are required to ensure optimal bonding between KCICF and the substrate under this environmental extreme factors.

In some circumstances carbon fibre mesh, fibreglass mesh or metal mesh reinforcement may be specified. The general method of mesh attachment and retention is by bonding pins to the composite substrate or welding pins to the metallic substrate.

Cimne KCICF doesn't need topcoat; but where the project requires a topcoat due to specific circumstances, topcoat could be used.

4.1. PRIMER

4.1.1. ZINC SILICATE

Generally, zinc silicate is discouraged due to their inherent cohesive weakness and inconsistency of film, but several zinc silicates are accepted for use under KCICF, but it's preferred that an epoxy tie coat is applied to these prior to application of KCICF. It is recommended that a solvent rub test, ASTM D4752 is carried out to assess the state of cure of the zinc silicate prior to overcoating. A minimum value of 4 should be obtained.

4.1.2. EPOXY

KCICF admits the epoxy primers use. KCICF recommends the use of bi-component epoxy primer.

The following list is a tentative of epoxy primer accepted by KCICF: Carboguard 888, Carboguard 890, Carboguard 893 SG, Carbomastic 15, Carbomastic 242, Intergard 251, Interzinc 52 and Intergard 269. However, this list is not exhaustive. Care should be taken to ensure that primer systems have reached sufficient levels of cure prior to over-coating with KCICF. A 14-hour cure period is good general practice for all epoxy primers which have been applied at temperatures greater than 15°C.

4.2. MESH

4.2.1. FIBREGLASS

The fibreglass mesh is a woven strand mesh with continuous filament of E-glass fibre. It is used as a reinforcing mesh KCICF when applied to structural steel beams and columns which are subjected to ASTM-E119/UL 263 or ANSI/UL 1709 fire environments (Underwriters Laboratories UL). The fibreglass mesh is installed into the wet KCICF.

4.2.2. CARBON FIBRE

Carbon fibre mesh is woven carbon fibre fabric mesh with 99+% carbon content as an open leno weave, usually with a coated finish to enhance handling. It is used as a reinforcing mesh KCICF when applied to structural steel beams and columns, bulkheads, underdecks and jet fire applications which are subjected to ASTM-E119/UL 263 (cellulosic), UL 1709 (hydrocarbon) or jet fire environments, and tubular steel beams and columns also.

4.2.3. STEEL

Steel mesh is a steel galvanised wire mesh, with square opening or hexagonal opening. It is used as a reinforcing mesh KCICF where it is required by the project. A stainless steel wire mesh is preferred for jet fire applications, and where the substrates is stainless steel also.

4.3. TOPCOAT

Topcoat is the last coat of a project, it's used for provide to specific colour schemes and finish requirements.

In the offshore platforms is recommended the topcoat use in areas exposed to periodic immersion such as the upper surfaces of horizontal decks and flanges and areas in the splash. Also, for aesthetic purposes on coastal locations or areas of high UV exposure – like all epoxy products, to avoid KCICF chalk when exposed to high levels of UV and moisture for prolonged periods. And when KCICF sprayed is coarse because the stippled finish collects airborne contamination and results in shading KCICF surfaces.

The following list is tentative and not exhaustive of topcoat accepted by KCICF: Carboguard 890 series, Carboguard 690, Carboguard 60, Carboguard 1340, Carbothane 133 HB, Carbomastic 94 series, Interzone 954 or Interthane 990.

5. EQUIPMENT

5.1. RECOMMENDED PLURAL COMPONENT EQUIPMENT

The following recommended plural component units (without holding tanks) are suitable for the application of KCICF shall be supplied from:

Equipment Model No:	Manufacturer
CoverCat 352 CoverCat 452	CoverCat Hyperion Equipment Ltd. 5 Orde Wingate Way Primrose Hill Industrial Estate Stockton-on-Tees, TS19 0GA, UK
Duomix 333 (PFP)	WIWA Wilhelm Wagner GmbH & Co. KG Gewerbestraße 1–3 D-35633 Lahnau, Germany
2K HV-PFP fixed ratio	Ecco Finishing Supplies Units 5-10 Letitia Industrial Estate Middlesbrough, TS5 4BE, UK
ES-430 FR PFP	Equipment Supplies and Consultancy Services 2 South Tees Freight Park Middlesbrough Road East Middlesbrough, TS6 6TZ, UK
397-884 SQ/GRACO H.D. Fixed Ratio	Graco Graco Distribution BVBA Industrieterrein Oude Bunders Slakweidestraat 31 B-3630 Maasmechelen, Belgium
SP5000 Fireproofing Systems	Spray Pump Inc. 719 East Pierce Street Baytown, TX 77520, USA
398105C	Spray-Quip, Inc. 1754 Des Jardines Houston, TX 77023, USA

It is essential that all the equipment used by the Applicator is properly maintained. It is necessary to regularly change items such as seals, gun tips, packing and hoses. Every Applicator shall obtain a parts list and/or manual from the manufacturer for the proper operation of the equipment and its maintenance.

Any company that has not utilized the specific pump prior to applying KCICF shall be trained in the proper operation of the pump by the pump manufacturer.

Poorly maintained equipment will not function properly and may cause the ratio of components (set at 1:1), to vary. The permitted ratio tolerance of components is $\pm 5\%$ (by volume). The Applicator shall perform at least 2 ratio checks (by volume) per shift collecting a minimum of 10 litres of components during each check.

Ratio checks shall only be performed by pumping both components into separate pails simultaneously.

5.1.1. PLURAL COMPONENT EQUIPMENT STARTUP PROCEDURE

(i) The pump and all lines shall be clean and free from any contamination.

(ii) Turn off air to main pump. Be sure all pressure is removed from lines. Load heated material under follower plates. Lower follower plates on to buckets and bleed off trapped air and then close bleed valves.

Note: If using pump with holding tanks, use transfer pumps and begin filling unit with material

Turn on the main pump air to assist in the movement of material into the material lines. Continue pumping Part A and Part B until a steady flow of material is present at the end of each fluid delivery hose. Turn off all air.

(iii) Turn on fluid and line heaters and adjust temperature of the heaters to 60°C. Wait at least 30 minutes for material to heat thoroughly or recirculate the material until the temperature of the material reaches the temperatures stated above.

(iv) The pump shall be set for a 1:1 ratio and it is important to check this ratio before beginning any work. Hose Bundle: $\frac{3}{4}$ " (30m maximum), Whip Hose: $\frac{1}{2}$ " (6m maximum).

(v) The air pressures needed for properly delivering material may vary from pump to pump, and adjustments may vary from the stated pressures herein.

(vi) With the air still turned off to the main pump, adjust the air pressure to the "A" component transfer pump to 3 bar. Adjust the air pressure to the "B" component transfer pump to 3 bar. While directing the discharge into separate, clean and equally sized containers, turn the air on to the main pump. Keep pumping until both or one of the containers are full. Insure that the transfer pumps are not continuously cycling. If this is so, turn up the main pump to increase the back pressure or reduce the pressure on the transfer pumps (see note below).

(vii) If containers contain equal volumes, the 1:1 ratio has been achieved. If not, check for restrictions in the air or material flow and material temperature. Repeat above procedure. If ratio is still not correct, inspect equipment and pump packings and replace if needed. Repeat the above procedure until a 1:1 ratio is achieved.

It is required that when using plural component pumps, which have holding tanks and recirculation capabilities, the material shall not be recirculated more than one time, and the temperature of the holding tanks shall be set to 60°C. Tank mixers should be used at minimum revolutions.

Material shall be pre-heated to a minimum of 38°C prior to introduction to the pumping units. Thoroughly mix Parts A and B well - separately - before using.

Note: If the transfer pump pressure is adjusted too high, it will over pressure the proportioning unit, and affect the ratio.

5.2. RECOMMENDED SINGLE COMPONENT EQUIPMENT

The following recommended single component unit is suitable for the application of KCICF:

Airless Spray Pump	Fluid to Air Ratio of 45:1 or greater. Hopper Feed required – ¾" outlet on high pressure side with an output of 12.5 litres/minute (minimum)
Gun	Binks IM Mastic Gun with Graco Tip Adapter
Tip Size	0.33 - 0.9 mm Graco Heavy duty RAC non- diffuser tips Fan
Size	150 – 250mm
Material Hose	Solvent Resistant: ¾" I.D. 15 m max. ½" I.D. Whip hose 7 m max.
Pressure at gun	170-210 bar
Air requirements	70 -170 m ³ /h @ 7 bar per unit. Use moisture and oil traps
Static mixer	Standard 12 turn ¾" mixer

5.2.1. SINGLE COMPONENT EQUIPMENT STARTUP PROCEDURE

Material shall be pre-heated to a minimum of 21°C and maximum of 30°C. It is important that the pump and all lines are clean and free from any contamination.

If half-filled containers have been supplied, add thinner into part B and mix thoroughly. Stage Part B onto Part A and mix thoroughly. Thinning will decrease the amount of material which can be applied in one coat and decrease the rate of cure.

If full containers are supplied, split containers in half, making sure that there are equal volumes of each. Follow instructions listed above.

Pour mixed material into hopper. Remove spray gun from the end of the hose. Turn pump pressure up only high enough to move material to the end of the delivery line. Turn off air

and replace spray gun. Increase air pressure until appropriate fan pattern is achieved. Apply material in accordance with the procedures detailed herein.

6. PREPARATIONS PREVIOUS TO KCICF APPLICATION

6.1. DEGREASING, BLASTING AND PRIMING

6.1.1. DEGREASING

All surfaces (FRP, GRP, steel, galvanised steel stainless steel and aluminium) shall be cleaned and degreased previous to any action over surface based on Solvent Cleaning (SP 1) requirements of The Society for Protective Coatings (SSPC) that includes: hot water washing, steam cleaning, detergents or emulsions/alkaline cleaners followed by steam or fresh water wash to remove detrimental residues.

The presence of surface oil or grease prevents a coating from properly adhering to the substrate and can lead to rapid failure of the whole system. For this reason, all visible oil, grease and other soluble contaminants must be removed before the application of both primer and coating. Degreasing is also important before blast cleaning activities.

The applicator should select the method most appropriate to the situation, giving due consideration to relevant health and environmental regulations. Heavy oil or grease deposits should be removed first by scraper, followed by detergent washing (or solvent cleaning) and a thorough fresh water rinse.

When it is necessary to clean the composite (FRP or GRP) surface before applying coating, primer or top coating, solvent wiping with Toluene or Plasite Thinner #19 is recommended.

When selecting a cleaning method, the primer manufacturer's and project recommendations must be adhered to.

6.1.2. BLASTING

All blasting abrasive shall be dry, clean and free from contaminants.

Abrasive blast cleaning is widely accepted as being the best way of preparing a metallic substrate (steel, stainless steel and aluminium) before application of a protective coating, and it is a very important part of the whole application process. A non-metallic blasting medium should be chosen for aluminium and stainless steel substrates.

The general requirement is Commercial Blast Cleaning (SSPC SP6), with a sharp angular profile being obtained. The blast profile (Rz) should be minimum 50 microns for steel substrates and the recommended method for measuring the blast profile is with replica tape or blast profile comparators.

Aluminium and stainless steel substrates should be prepared by abrasive blasting using a non-metallic abrasive. The blast profile (Rz) should be a minimum of 50 microns.

Where grit is used, contact primer manufacturer for specific details on acceptable blast abrasive and other industry standards.

6.1.3. BRUSH-OFF BLASTING

Galvanised substrates should be prepared by brush-off abrasive blasting to provide a roughened surface, to a standard similar to Brush-Off Blast Cleaning (SSPC SP 7). The blast profile (Rz) should be 50 to 75 microns. For the abrasive sweep blast or brush blast, blast material particle size should range between 200 and 600 microns and be propelled by a blast pressure of just 350 kPa. Garnet with (30/60 mesh) is to be used to generate a suitable blast profile.

The surface should be clean, dry and grease free. Degreasing of most galvanized surfaces requires some effort to obtain a clean surface. Any white zinc corrosion products should be removed by high pressure fresh water washing, or fresh water washing with scrubbing. If sweep blasting is used to remove surface contaminants, it is still advisable to fresh water wash to remove soluble zinc salts. Many coatings based on non-saponifiable polymers can be applied directly to galvanized surfaces prepared in this way.

6.1.4. POWER TOOL CLEANING

The power tool cleaning could be used on FRP and GRP to remove glaze.

Hydro-blasting may be an acceptable method of surface preparation of previously coated substrates. However, this is conditional on an adequate surface profile being revealed as detailed above. Where a suitable surface profile is not revealed, then brush-off blasting or abrasive blasting will be required. A qualified wet blast primer must be used with this method of surface preparation.

Power tool cleaning is not an acceptable primary surface preparation method for large areas of steel in new construction. In scenarios where blasting is not permitted, power tool surface preparation is acceptable provided a suitable surface profile and cleanliness is generated.

For these areas power tool cleaning methods must achieve a clean, roughened surface in accordance with Bare Metal Power Tool Cleaning (SSPC SP 11), power tool cleaning. A minimum profile of 50 microns must be maintained. For areas less than 1 m², a surface preparation yielding ST3 surface cleanliness is acceptable.

6.1.5. DEBRIS REMOVAL

All surfaces (FRP, GRP, steel, galvanised steel stainless steel and aluminium) would be able to be removed from debris using vacuum system.

6.1.6. PRIMER

The primer shall be applied in accordance with the manufacturer's and project's specification. Refer to previous proposed primers list. It is the applicator's responsibility to ensure that the primed surface and the primer itself are in an acceptable condition for over-coating with KCICF. Factors such as ageing of the primer (degradation and surface chalking), contamination, zinc salt formation, rust, bloom, etc. shall be considered when determining a surface's suitability before over-coating with KCICF.

In all situations the surface of the primer must be clean, dry and free of all contaminants immediately prior to over-coating with KCICF. Consult the relevant primer product data sheet for minimum over-coating times.

Any primer surface which shows indications of UV degradation (such as surface chalking) or has exceeded its maximum over-coating period will need to be treated by abrasion (e.g.

sweep-blasting) prior to over-coating. Areas of rust breakthrough should be abrasive blasted back to Commercial Blast Cleaning (SSPC SP6) requirements and the primer re-applied.

Amine bloom formation on the primer surface can be removed by high pressure water-washing at a minimum of 170 bar.

If epoxy zinc primers are exposed to humid or outdoor conditions prior to application of KCICF, zinc salts may be formed on the surface which is detrimental to adhesion of KCICF. These will need to be removed prior to application of KCICF. Typically, this is achieved by high pressure water-washing at a minimum of 170 bar. If stubborn salts persist then brushing or another abrasive method will need to be included with the water-washing. Alternatively, an epoxy tie coat may be applied over the epoxy zinc primer to prevent salts forming.

Prior to over-coating the applied primer, dry film thickness (DFT) checks should be made to ensure that the following tolerances are not exceeded: Epoxy 75 microns max., Epoxy zinc 75 microns max., Epoxy zinc plus tie coat 110 microns max.; and in overlap areas no primer 150 microns max.

To ensure that KCICF has been applied to an appropriate primed galvanised surface, pull off tests (ISO 4624) and shear tests should be used appropriately to ensure sufficient strength at all interfaces as well as the cohesive strength of the products applied.

Pull off dollies may be applied to the primed galvanised steel after 3 days cure. KCICF should be used as the glue for the dolly. The primer should then be left for a total of 7 days prior to conducting the pull off tests. A pull off value greater than 5 MPa should be achieved.

If the pull off test results in a failure at the primer/KCICF interfaces a further sweep blast should be undertaken to ensure a robust interface with KCICF. Further tests are then required to ensure adhesion of KCICF to the primer as outlined above. If the primer fails at the galvanised steel interface with the primer, then the primer should be removed by blasting, a full solvent wash be undertaken to ensure there is no oil and grease contaminants followed by re-instatement of the primer. Again further tests should be undertaken to ensure KCICF will be adherent to the primed galvanised surface. If the galvanising layer fails, then steel should be rejected as blasting will not assure the integrity of the galvanised layer.

Inorganic zinc silicate primers are especially susceptible to the formation of white zinc salts on external exposure and these must be removed prior to overcoating, as per recommendations above.

6.1.6.1. PRIMER THICKNESS AND ADHESION MEASUREMENTS

Controlling the thickness of applied primers is very important. The following method will be mandatory:

- (i) Use a flat polished steel plate to calibrate the measuring device to zero.
- (ii) Use the measuring device to measure individual primer coats and multi-coat thicknesses.
- (iii) Record measurements at the rate specified by the project.
- (iv) Thickness of primer must be sufficient to cover blast profile.

Optimal bonding is achieved when the primer DFT is sufficient to just cover the peaks of the blast profile and maintain a rust free condition prior to application of KCICF. Where KCICF is

in service in cold climates, optimal performance to maximise the lifetime of the asset is expected by adherence to this principle. Maximum primer DFT varies by primer, and careful monitoring and measurement of primer thickness is required. DFT measurement should be carried out in accordance with ISO 2808 or Procedure for Determining Conformance to Dry Coatings Thickness (SSPC PA 2).

The maximum allowable primer system DFTs are summarised again:

- (a) Epoxy 75 microns in normal areas, and 150 microns in overlap areas
- (b) Epoxy zinc 75 microns in normal areas, and 150 microns in overlap areas
- (c) Epoxy zinc plus tie coat 110 microns in normal areas, and 150 microns in overlap areas

6.1.6.2. PRIMER REACTIVATING

If multiple primer coats are required, care must be taken to ensure that the manufacturers recommended maximum overcoating time has not been exceeded.

6.1.6.3. REDUCTION OF EXCESSIVE PRIMER THICKNESS

Excessive primer thickness should be reduced to the DFTs indicated. The preferred method to be used is abrasive sweep blasting followed by thorough clean air blowing to remove contaminants from the surface. Sanding with P80-P100 grade aluminium oxide abrasive paper may be suitable for small areas less than 1 m²; however, care should be taken to prevent polishing of the surface which would lead to inadequate adhesion of KCICF. Frequent changes of the abrasive paper should be made. After primer reduction, surfaces should be cleaned of dust and contaminants and thoroughly dry prior to the application of KCICF.

6.1.6.4. PRIMER ADHESION

The coating inspector for the project must be satisfied that the adhesion values of the primer system meet the project specification.

6.2. SURFACE PREPARATION WHERE AN EXISTING PRIMER WAS INSTALLED

This applies to areas where an existing primer system has been installed and the existing surface was prepared according to Commercial Blast Cleaning (SSPC SP6) with a minimum of 50 microns profile. If these conditions are not available, this section will not apply.

Ultra high pressure water jetting may be employed to remove a primer or other coating where no dry blast surface preparation is permitted.

Thoroughly water jet the surface until the original blasted surface has been revealed. The cleaned surface should be blown dry with clean, dry compressed air to remove water residues. Immediately after, the clean surface shall be primed with an approved wet blast type primer, in accordance with the manufacturer's and project's specification.

6.3. GALVANIZED SURFACE PREPARATION

Galvanizing requires a roughened surface for optimum adhesion/performance of high build epoxies. Remove any contaminants per Solvent Cleaning (SSPC SP 1) requirement; ensure there are no chemical treatments that may interfere with adhesion; and abrade the surface

to establish a suitable roughness per Brush-Off Blast Cleaning (SSPC SP 7) with a minimum angular anchor profile of 50 microns.

6.4. METALIZED SURFACE PREPARATION

All steel surfaces shall be blasted to Brush-Off Blast Cleaning (SSPC SP 7), with a minimum angular anchor profile of 50 microns.

6.5. STAINLESS STEEL SURFACE PREPARATION

All stainless steel surfaces shall be blasted to Commercial Blast Cleaning (SSPC SP6), with a minimum angular anchor profile of 50 microns.

6.6. WELD CUT BACK

It is often necessary to block out areas on the structure before the application of KCICF to allow for future welding.

As heat is generated during the welding process, either during a preheating stage and/or during the welding itself, it is important that a suitable distance is left around the weld area to prevent damage to the adjacent KCICF.

For small weld attachments such as the addition of clips and hangers, a cutback distance of 50 mm on each side of the weld areas should be sufficient. When carrying out welding close to KCICF, a discoloration may be noted. This discoloration denotes that the KCICF has received too much heat and the bond to the steel is likely to have been affected. When this happens, the discoloured KCICF must be removed as described in this document. For larger welds, i.e. full girth, a cutback of 300 mm each side of the weld area should be sufficient.

In the case of allowance of cutback for welding of structural members and structural members themselves which are to be pre-coated with KCICF prior to assembly, the following recommendation is made:

- No weld preheat: > 100 mm either side of weld
- Weld preheat 100°C: duration 4-8 hour will be 0.75m free distance; and duration 9-12 hour will be 1.00m free distance
- Weld preheat 150°C: duration 4-8 hour will be 0.75m-1.00m free distance; and duration 9-12 hour will be 1.00m-1.25m free distance

The above figures are expected to be conservative, actual distance is a combination of factors, including mass of steel at weld junction and method of preheat.

7. KCICF APPLICATION PROCEDURES

KCICF shall be applied in good weather, following good painting practices. In general, the substrate and ambient temperature shall be no less than 5°C, and 3°C above dew point. The humidity shall be 85% or less. All attempts shall be made to maintain dry conditions throughout the application process, through the topcoat application. The Applicator shall apply Cimne KCICF over a properly cured approved primer if the primer is required. The material shall be applied in as many passes required to provide the desired film thickness.

While not a requirement, applying KCICF in a thickness no greater than 3mm will provide an improved surface appearance. Allow applied material to cure sufficiently to support the weight of subsequent coats. The thickness which can be applied in a single coat will depend on the temperature, humidity, applicator technique, etc. The time between coats is largely dependent upon ambient temperatures and in general the following values should be used:

Temperature (°C)	10	25	30	35	43
Minimum minutes	60	30	30	30	30
Maximum hours	72	72	48	48	24

If the overcoat time has been exceeded, the surface of KCICF must be reactivated. If the time to reactivated is greater than 72 hours but less than 7 days the surface of KCICF must be solvent wiped (Toluene or Plasite Thinner #19) to remove contaminants and to reactivate KCICF. If the time to reactivate is greater than 7 days or beyond the surface of KCICF must be brush-off blasted or mechanically abraded to remove any contamination or particulate. The freshly blasted surface must then be solvent wiped (Toluene or Plasite Thinner #19) to remove contaminants and to reactivate KCICF.

During application and curing, the surface should be protected from moisture (e.g. rain, high humidity, etc.) as required. These conditions could affect film integrity and inter-coat adhesion with subsequent coats. KCICF must be protected from direct rain until it has reached sufficient cure. If water contamination occurs to uncured KCICF, any uncured material must be removed and reapplied. The material must be clean and dry prior to applying subsequent coats of KCICF or topcoating. The following actions will be carried out based on contamination type as is described:

- Cured with fresh water contamination: acts drying and wiping solvent
- Cured with salt water contamination: acts applying warm water (max. 640°C), and then dry and wipe a solvent
- Uncured with any contamination: acts removing and replacing all uncured material

Topcoating shall be applied in accordance with the project specification and manufacturer's recommendations. The maximum allowable time to topcoat KCICF is 7 days at 21°C. If this time is exceeded, the area should be brush-off blasted or mechanically abraded with 40 grit paper, then solvent wiped, prior to topcoating. The minimum topcoating time is 10 hours to 48 hours.

The Applicator shall take frequent thickness measurements during application, using a penetrating measuring device to ensure that the coating is uniformly applied at the required film thickness. The recommended method of measuring wet thickness is to use a pre-cut bridge gauge. Typically made from a putty knife, widths of 40-100 mm have been found to be most appropriate. Notch or pin gauges are not acceptable because they limit the measurement to one point. The gauge should just touch the rolled surface in the middle and the firm base layer at the edges. It's strongly recommends the continuous use of the bridge gauge by all members of the application team (sprayer, trowellers and rollers), as this is the only way of ensuring the design thickness will be applied to the whole surface.

Care shall be taken to keep the fan pattern at an angle of 90° to the surface and at 300 mm to 450 mm away from the surface. A brush and/or roller can be used to improve the surface quality.

Excessive build-up of KCICF in angle areas can be removed or leveled by rolling the surface with a solvent resistant foam or short napped mohair roller. Use Toluene or Plasite Thinner #19 to keep roller moist which will prevent roller from sticking to the material. It's recommends the use of solvent resistant spray bottles to minimize the use of solvents. Do not use excessive solvent during rolling, as this will retard the surface cure of the material.

Special care should be taken when spraying flange edges on structural steel members to ensure complete coverage and a consistent thickness. The normal spray pattern on the outside and the inside surfaces of the flanges should cause the material to flow and wrap around the edge of the flange. If the coating on the flange edge is uneven after application, the edge shall be rolled lightly in order to provide an even surface.

7.1. HAND/TROWEL APPLICATION PROCEDURE

KCICF is able to be applied manually using plasterers' trowels. This has the advantage of very little waste and virtually eliminates the need for masking. Special care must be taken during hand trowel applications when coating connection joints that a uniform thickness and required surface finish is achieved.

To facilitate application by trowel KCICF material should be maintained at a temperature of 21-27°C for a minimum of 24 hours prior to use. Mixing should be carried out. A maximum amount of solvent for hand application is 0.5 litres for 20 kg for temperatures less than 30°C. For temperatures greater than 30°C no solvent is required to thin KCICF.

After KCICF has been mixed, it may be spread out on flat clean boards or surfaces for each applicator to work with. This prevents the material from curing too quickly and allows sufficient working time. Alternatively, the material may be applied directly from the pail.

7.2. PLURAL COMPONENT AND SINGLE COMPONENT APPLICATION PROCEDURE

KCICF is able to be applied using Plural Component Equipment and Single Component Equipment (also known as Plural Spray and Single Leg Airless Spray).

To application procedure with these equipments please consult above the full description.

7.3. INSTALLATION OF MESH ON STRUCTURAL STEEL I & H SECTIONS

When a mesh on structural steel I & H sections is required to be applied, once the first coat of KCICF has been applied, the mesh is placed in position as detailed UL1709 Designs: Design No. XR 618, Design No. XR 620, and Design XR 621 and its time application criteria.

Ensure that the proper mesh reinforcement is utilized based on the project specific UL1709 Design (Design No. XR 618 with FP-Fiberglass mesh, Design No. XR 620 with High Temp mesh, Design No. XR 621 High Temp mesh), the mesh shall be overlapped 25mm on all longitudinal seams running down the wide flange member. Rolled hollow section (RHS), tubular and other hollow steel sections are reinforced in a similar manner except that the entire surface is reinforced by using one or two pieces of mesh; the mesh shall be overlapped 25mm on all seams.

7.4. FINAL MESH APPLICATION

After the mesh has been installed, apply KCICF to the final required thickness in as many coats as is necessary. Any overcoating shall be done within 72 hours of the prior coat. If the time is exceeded, the surface must be prepared as stated in previous sections. It is mandatory that imbedded mesh is covered with 1 mm of KCICF prior to daily shutdown.

7.5. FINAL THICKNESS

The final thickness(es) shall be specified in project drawings and Owner specifications. In mesh applications, UL thicknesses for KCICF are outlined in UL Design Nos. XR 618, XR 620, XR 621 and are based on average thickness. However, on some projects, a minimum thickness may be specified from the owner or specific engineering firm.

The thickness measurement, the number of thickness checks and other issues regarding this topic are found in ISO 2808 or Procedure for Determining Conformance to Dry Coatings Thickness (SSPC PA 2).

In any case here, it's shown some recommended methods to measure the Dry Film Thickness (DFT):

- Drill a small hole approximately 2 mm in diameter and checking the thickness with a depth gauge (care must be taken not to damage the substrate and to refill holes with KCICF as soon as possible).
- Use an eddy current thickness gauge (care must be taken to correctly calibrate the gauge on a smooth calibration plate immediately before taking any readings).

Where KCICF is supplied as part of a cryogenic spill protection system, thickness must only be determined by means of an eddy current thickness gauge.

It is recognized that the applied KCICF layer will never be perfectly even and that areas of lower than specified design thickness ("valleys") are compensated by areas of higher than design thickness ("peaks"). For protection against cryogenic releases combined with fire exposure, the minimum thickness requirement will be the specified thickness at all points. For non-cryogenic scenarios, unless specifically stated otherwise in project documentation, the DFT limits at all points should be:

Design DFT	Minimum DFT	Maximum DFT
<10mm	0.85 x Design DFT	1.15 x Design DFT
≥10mm	Design DFT – 1.5mm	

All matters relating to the thickness(es) shall be decided between the owner and the applicator prior to the startup of the job.

After KCICF has cured, an approved thickness gauge shall be used to ensure the thickness requirements have been met. Thicknesses below specification shall be built up to specified thickness by the application of additional KCICF.

7.6. SAMPLE INSTALLATION

Prior to actual production work, a sample test area shall be prepared following all specified procedures and approved finish / surface quality. This sample must then be approved by representatives of the owner, applicator and any others having a vested interest in the installation. The actual production work must follow, and conform to, the standards and approved finish / surface quality of the site sample. The site sample is a mandatory requirement and shall be made available to all parties throughout the completion of the project.

7.7. EQUIPMENT CLEANUP / FLUSHING

The plural component equipment with hot water flush components must be flushed using 66°C water (minimum). The plural component equipment with solvent flush components must be flushed with Toluene or Plasite Thinner #19. The spray gun, static mixer and block assembly must be hand cleaned at the end of each spray period using Toluene or Plasite Thinner #19.

7.8. RECORD KEEPING

The maintaining of proper records is an essential requirement for all KCICF projects. The minimum requirements will be established by the project.

8. ADHESION TESTING

Verification of system integrity should be undertaken. Once suitable reference areas have established good adhesion to the structure and between KCICF and the primer (using primer method test, see above sections), pull-off adhesion testing may then be requested as a project-specific requirement for acceptable system integrity (cohesive strength not adhesive strength from substrate). Pull-off tests should be conducted in accordance with ISO 4624, using 2 cm dollies and a minimum value of 5 MPa should be attained. Refer to above sections for information on primer suitability.

9. CLEAN-UP

The application area shall be maintained in a clean and orderly condition. Following the application, all overspray, debris, and equipment shall be removed and the area left in a condition acceptable to the Owner and Main Contractor.

10. REMOVAL AND REPAIR PROCEDURES

10.1. REMOVAL

The preferred method is to cut through KCICF at right angles to the substrate with a disc saw around the area to be removed. Make sure that the steel substrate is not damaged. A power chisel can then be used to “chip” away the KCICF from the substrate.

It is essential that proper safety precautions are taken during this operation

10.2. REPAIRS - GENERAL

Repair of damaged or failed areas, in the fabrication yard or during service life, and cutback areas should follow the procedure below. Application conditions must conform to specified conditions for primer top-coat and KCICF application:

- (i) Check the 'border' area of surrounding KCICF for soundness and adhesion in the joint area.
- (ii) For guidelines on how to replace and/or abut against epoxy fire proofing, refer previous sections.
- (iii) Mark out the area to be repaired. Masking should be done around the area to be repaired in a square or rectangular shape. This will give the patch a clean appearance once completed.
- (iv) Using a disc grinder, cut through KCICF (take care not to damage substrate).
- (v) Using chosen tools (hammer and chisel or pneumatic chisel), cut into patch to remove damaged KCICF. Make sure all uncured material is removed.
- (vi) Small areas, for example welds and local repair areas, may be prepared by power tools to achieve a clean, roughened surface in accordance with Bare Metal Power Tool Cleaning (SSPC SP 11) and maintaining a minimum profile of 50 microns. Power tool cleaning should not be used as a primary surface preparation method for large areas.
- (vii) Abrade adjacent surfaces for a distance of 150 mm from edge of repair area. After roughening, a suitable solvent should be used to ensure that the area is clean.
- (viii) Reinstall primer to specification, if required. For repairs, KCICF may also be applied directly to blast cleaned steel.
- (ix) Reinstall reinforcement to specification. If the repair area is less than 150x150 mm, reinforcement is not necessary, except for jet fires.
- (x) Reinstall KCICF to specification, taking care to smooth over the repair area edges. Trowel application of KCICF is acceptable for repair areas.
- (xi) Top-coat replacement and repair should follow top-coat manufacturer's instructions.

11. IN SHOP APPLICATION – PRE-ERECTION

KCICF lends itself to being a material which can be applied off site and later shipped to the job site. This allows for a more consistent application of the material off site and elimination of interference with other trades which may affect application flow. In general, the shop application of KCICF follows the previous guidelines in this practical guide. The following additional requirements are necessary.

11.1. BLOCK OUT AREAS

Prior to application of the shop fireproofing, define all areas of the structures that do not receive KCICF until after the erection phase of the construction. These areas which are not

to receive KCICF shall be masked off in the proper dimensions, prior to application. The size of the block out shall be determined by the applicator, owner or engineering firm. KCICF has high bond strength, and cannot be easily removed. For in shop application, the specified topcoat system shall be applied to all KCICF surfaces. After piece is erected, the coat shall be mechanically removed from the edges and back approximately 25mm from the edge of the block out.

Application to the block out areas may now continue, utilizing spray or trowel methods. KCICF finish appearance must be approved by the owner.

In case of mesh use, the specified mesh type shall be installed and butted up to the existing mesh (no overlap required). The final specified thickness of KCICF can then be applied.

The blocked out edges can be left as butt joints to a 90° angle or beveled to a 45° angle.

The specified topcoat system shall be applied, based on the original specification.

11.2. HANDLING, STORAGE AND ERECTION

11.2.1. UNLOADING

Generally the fireproofed structure is loaded and unloaded with a crane to permit maximum loading of each truck. It is not advisable to unload fireproofed structure with forklifts, as the structure is nested in ways that forklifts do not easily accommodate and damage to fireproofing may occur.

11.2.2. RIGGING

Wherever possible, structure should not be rigged to the fireproofed portion of the member. Shackles at end holes are a generally acceptable practice and provide the best method for unloading members. Follow all site specific safety standards for handling the structures.

11.3. DUNNAGE

Members should be stored on proper dunnage (on the block out areas) to avoid exposure to the ground and moisture.

11.3.1. ORIENTATION OF MEMBERS

Members should be stacked with webs vertical to avoid ponding of water and snow. Elevate one end of the structure member to provide water run off.

11.3.2. STACKING

Where members must be double stacked, avoid placing dunnage on the fireproofed surfaces.

11.4. RIGGING

Avoid using chokers against fireproofed surfaces. Wherever possible, add erection tabs or bolt holes at the beam ends to allow top rigging without using chokers around the fireproofing. Follow all site specific safety standards for handling structures.

11.4.1. SWING RADIUS

Where infill beams use framing angles, the erector must be aware of the swing radius of the member impacting the fireproofing. This type of connection has a blockout on each side of

the holes on the primary member. When the block out dimension is insufficient, the erector is advised to chip out a minimal amount of fireproofing on “one side only”. It is better to chip a minimal amount of fireproofing than to make all the connections unnecessarily large. Follow all site specific safety standards.

11.4.2. CONSIDER GROUND ASSEMBLY

On repetitive structures like pipe racks, by pre-assembling the bents it will minimize the damage to the fireproofing.

11.4.3. LIFTING

All pre-erection projects will require the structure to be lifted onto trucks or into place on the jobsite. All lifting shall be done utilizing the block out areas for strapping, shackling to erection tabs or bolt holes. Lifting should be performed from a 2-point lift so to insure the straps are flat against the structure, so that no slipping and tearing can occur.

11.5. DAMAGE

Any damage to the coating system shall be repaired as per above sections of this guide.