TU1207

COST Action TU1207

Next Generation Design Guidelines for Composites in Construction

Short Term Scientific Mission

Mechanical and thermal characterization of the filament wound composites for construction

Final Report

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COST-STSM-TU1207-23020

STSM Grantee	Mechanical and thermal characterization of the filament wound composites for construction, Vineta Srebrenkoska				
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Contents

1. Summary

COST-STSM-TU1207-23020

Properties of fiber reinforced composites (FRP) arise as a function of its constituent materials, their distribution, and the interaction among them and as a result of it an unusual combination of material properties can be obtained. There are various methods of their manufacturing, but filament winding is a very important and widely used technique for FRP production related to civil engineering. The basis of this technology includes winding of resin-impregnated fibers into a tool and hardening of the wound structure. This technology enables the fibers to be placed into the direction of the load that may be expected during exploitation of construction elements. By varying the winding angle with respect to the mandrel axis, directional strength can be obtained by the loads, which will operate on the finished product. It is essential to know the mechanical and thermal characteristics of filament wound tubes in order to employ them in design applications.

The focus of this stay was to investigate the mechanical and thermal properties of different glass fiber reinforced composite pipes produced by filament winding technique. So, the research program of the STSM was divided into two tasks. The first one refers to the mechanical characterization, and the second one to the thermal characterization of the epoxy resin and filament wound pipes.

Based on the investigation in the fame of this STSM, it can be concluded that from the mechanical point of view there are significant differences in the filament wound pipes with different fiber orientation. Regarding the tensile properties, the bigger winding angle lead to higher hoop tensile properties of filament-wound tubular samples. But, for the transverse compression properties of composite specimens the lower winding angle lead to higher transverse compression properties of the samples. The optimal mechanical properties have the samples on the primary level winded with angle 45^0. It was noticed a slight influence of the fiber tension but the velocity of the filament winding doesn’t influence on the mechanical properties of the specimens.

From the results of thermal characterization, it can be concluded that all filament wound pipes have a good thermal stability and their weight loss was observed at temperature interval from 600 ^oC to 1000 ^oC. Based on the measurements for the glass transition and rate of cure, it can be concluded that crosslinking reaction between the resin and fibers in the filament wound pipes is already reached in all composites.

Figure 1. Representative Image/Figure of COST-STSM-TU1207-23020

2. Purpose of the STSM

This STSM is closely linked to the aims of working group 1 in the frame of the COST Action TU1207 and strongly supports the WG 1 activity and directly enables the development of FRP material characterization.

The fundamental structure of the STSM is based on designing of the filament wound tubes based on epoxy resin system and glass fibers in accordance with the final application in construction.

The purpose of the stay was focused on the realization of the mechanical and thermal analyses of the filament wound composite pipes in order:

- to find the most effective fiber strengthening configuration,
- to quantify the thermo-mechanical characteristics of the epoxy resin,
- to quantify the tension and compressive resisting of the different designed filament wound pipes,
- to better understand the interaction between fibers and matrix,
- to estimate the contribution of the fibers, matrix and designed composite systems for the mechanical properties,
- to estimate the contribution of the fibers, matrix and designed composite systems for the thermal properties.

The desired objectives of the STSM were achieved by the strong co-operation between Lodz University of Technology (TUL) and Goce Delchev University in Shtip, using the complementary competences of both research groups.

3. Description of the work carried out during the STSM

The focus of this stay was measurement of some mechanical characteristics: hoop tensile strength and transverse compressive properties of nine different models of filament wound pipes and thermal testing of the epoxy resin and of the filament wound tubes by DSC and TGA. So, the research program of the STSM was divided into two tasks. The first one refers to the mechanical characterization of the filament wound pipes, the second one to the thermal characterization of the epoxy resin and filament wound pipes.

The different models of filament wound pipes were produced by the home institution. For the production of the composites 10 bobbins of E-glass fiber roving 185P with 1200tex from Owens Corning were used. The glass fibers were impregnated into epoxy resin system Araldite LY564/Aradur 917/Accelerator 960-1 from Huntsman. The preparation of the composites was done by applying the 2^3 full factorial experimental design by using of three parameters and two levels of variation. The three parameters were: velocity of the filament winding, fibre tension and winding angle, for which were used two different levels of variation: for the first factor low and high levels were chosen to be 5, 25 m/min and 21 m/min, respectively, for the second factor – 34 N and 60 N, respectively and for the third factor – 10^0 and 90^0, respectively (Table 1). On basis on full factorial experimental design, nine test specimen configurations were made: eight from the matrix of full factorial experimental design and one on the primary level.

Table 1. Full factorial experimental design - 2^3

No. exp.	**Matrix of full factorial experimental design**							**Characteristics (conditions of the experiment)**		
	X_1	X_2	X_3	$X_1 X_2$	$X_1 X_3$	$X_2 X_3$	$X_1 X_2 X_3$	**X_1 (m/min)** *velocity of the filament winding*	**X_2 (N)** *fibre tension*	**X_3 (^{0})** *winding angle*
1	-1	-1	-1	+1	+1	+1	-1	21	60	90
2	+1	-1	-1	-1	-1	+1	+1	5,25	60	90
3	-1	+1	-1	-1	+1	-1	+1	21	34	90
4	+1	+1	-1	+1	-1	-1	-1	5,25	34	90
5	-1	-1	+1	+1	-1	-1	+1	21	60	10
6	+1	-1	+1	-1	+1	-1	-1	5,25	60	10
7	-1	+1	+1	-1	-1	+1	-1	21	34	10
8	+1	+1	+1	+1	+1	+1	+1	5,25	34	10

Primary level	X_1 = 13,125	X_2 = 47	X_3 = 50
Interval of variation	7,875	13	40
Lower level	5,25	34	10
Upper level	21	60	90

Samples with different winding designs were winded on iron mandrel with pins on the both sides with help of laboratory filament winding machine MAW FB 6/1 with six axes, roller type resin bath manufactured from Mikrosam A.D. Fibers pass through a resin bath after tensioning system and gets wet before winding operation.

After winding samples were cured with industrial heater at 80^{o}C and at 140°C, for four hours.

The tensile and compression testing of the filament wound pipes was carried out during the STSM as first part of the research plan.

For tensile tests each specimen was cut to obtain five split-disk test specimens according to ASTM D2290. Tensile tests of 45 split-disk samples were carried out at room temperature using universal testing machine Zwick/Roell Z050 with max load of 50kN and Zwick/Roell Z400 with max stress of 400MPa and loading speed of 0.3 in/min. For the compression test each specimen was cut to obtain three pipe test specimens according to ASTM D5449. Compression tests of 27 pipe specimens were carried out at room temperature using the same universal testing machine Zwick/Roell Z400 with max stress of 400MPa and loading speed of 0.05 in/min. Width and thickness of each split-disk specimen and pipe specimen were measured with a help of micrometre instrument by reading at least 0.0254mm. In this way prepared specimens were elongated and compressed till rupture with help of test fixtures for tensile and compression testing, made according to standards (Fig. 2).

a) split-disk test specimen and test fixture with specimen for tensile testing

b) pipe test specimen and test fixure with speciment for compression testing

Figure 2. Illustration of the assembled tensile and compression test fixture and specimens

The focus of the second part of work carried out during the STSM was the thermal characterization of the epoxy resin and filament wound pipes by using of Thermal Gravimetric Analysis - TGA and Differential Scanning Calorimetry - DSC methods.

The DSC measurements were performed with a Mettler – Toledo DSC instrument. The experiments were carried out under a constant flow of nitrogen of 50 ml/min. For the dynamic DSC measurements, the epoxy resin system and the composites were subsequently heated at a constant rate of 5, 10, 15 and 20 °C/min over a temperature range of 20 to 350 °C. The TGA measurements were performed with a Mettler – Toledo TGA instrument. About 20 mg of each sample was heated from 50 ^{o}C to 1000 ^{o}C at a heating rate of 20 K/min under argon/air flow of 50 ml/min (Fig.3).

a) Mettler – Toledo TGA instrument

b) Mettler – Toledo TGA instrument

Figure 3. TGA and DSC instruments

4. Description of the main results obtained

Part 1. Mechanical analyses

The results of the testing method of the split-ring specimens for determination of the apparent hoop tensile strength of the reinforced plastic are presented in Table 2.

Determination of hoop tensile properties of filament-wound composite tubular specimens by split disk method was one of the objectives of this STSM. Five specimens were tested from each testing group. Mainly, the ultimate hoop tensile strength of the specimens were determined. In addition, the average of these results were calculated for each group, and with the aid of this data, the general behavior of the specimens were determined. The apparent hoop tensile strength of the specimens were calculated by using the following equation:

$$\sigma = \frac{F_{\max}}{2 \cdot A_m} \qquad (1)$$

In equation (1) σ is ultimate hoop tensile strength, MPa, *Fmax* is maximum load prior to failure recorded in Newton (N), whereas *Am* is minimum cross-sectional area of the two reduced sections, d x b, mm^2 (Fig. 4).

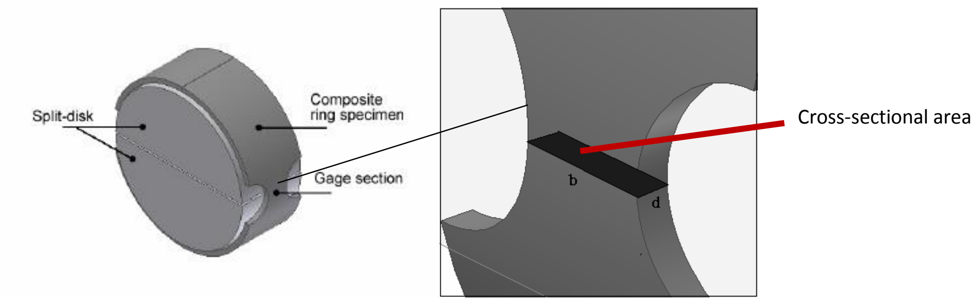


Figure 4. Cross-sectional area on which hoop tensile stress is applied

Table 2. Hoop tensile strength results of split-disk tests

Sample Designation		b (mm)	d (mm)	A $(mm)^2$	F (kN)	F average (kN)	σ (MPa)	σ average (MPa)
1	1-1	14,07	3,20	45,01	80,6	70,7	895,36	788,09
	1-2	14,01	3,19	44,76	68,5		765,19	
	1-3	13,99	3,20	44,77	65,7		733,75	
	1-4	14,14	3,20	45,28	61,5		679,11	
	1-5	14,05	3,17	44,52	77,20		867,03	
2	2-1	14,14	3,14	44,40	73,50	70,7	827,70	796,22
	2-2	14,14	3,12	44,12	64,70		733,23	
	2-3	14,17	3,15	44,64	73,90		827,73	
3	3-1	14,00	3,08	43,19	77,60	74,14	898,36	853,58
	3-2	14,07	3,09	43,49	74,10		851,92	
	3-3	14,08	3,09	43,51	74,60		857,27	
	3-4	14,08	3,09	43,51	71,40		820,50	
	3-5	14,02	3,10	43,46	73,00		839,85	
4	4-4	14,15	3,65	51,65	72,30	74,95	699,90	725,84
	4-5	14,14	3,65	51,61	77,60		751,79	
5	5-1	14,12	3,27	46,17	2,29	2,17	24,8	23,57
	5-2	14,2	3,27	46,43	2,40		25,84	
	5-3	14,19	3,27	46,40	2,25		24,25	
	5-4	14,17	3,20	45,34	1,97		21,72	
	5-5	14,17	3,24	45,91	1,95		21,24	
6	6-1	14,12	3,15	44,48	1,96	1,89	22,03	20,99
	6-2	14,08	3,20	45,06	2,03		22,52	
	6-3	14,09	3,20	45,09	1,92		21,29	
	6-4	14,16	3,20	45,31	1,82		20,08	
	6-5	14,04	3,20	44,93	1,71		19,03	
7	7-1	14,08	3,17	44,70	1,48	1,47	16,55	16,51
	7-2	14,10	3,15	44,42	1,55		17,45	
	7-3	14,07	3,17	44,60	1,13		12,67	
	7-4	14,00	3,17	44,38	1,72		19,38	
8	8-1	14,00	2,95	41,30	1,39	1,41	16,83	16,95
	8-2	14,00	2,95	41,30	1,43		17,07	
9	9-1	14,05	3,18	44,68	25,14	24,69	281,33	284,90
	9-2	14,01	3,00	42,03	24,25		288,48	

Figures 5 - 8 show a typical stress and deformation diagrams at ambient temperature for tested samples series 1, 2, 3 and 4 on universal testing machine Zwick/Roell Z400 with max stress of 400MPa and loading speed of 0.3 in/min. These composites were tested on testing machine with higher max load because of their higher tension strength than the composite samples series 5,6,7,8 and 9, which were tested on universal testing machine Zwick/Roell Z050 with max load of 50kN and with the same loading speed of 0.3 in/min.

Figures 9-13 shows a typical force and displacement curves at ambient temperature for tested samples series 5,6,7,8 and 9.

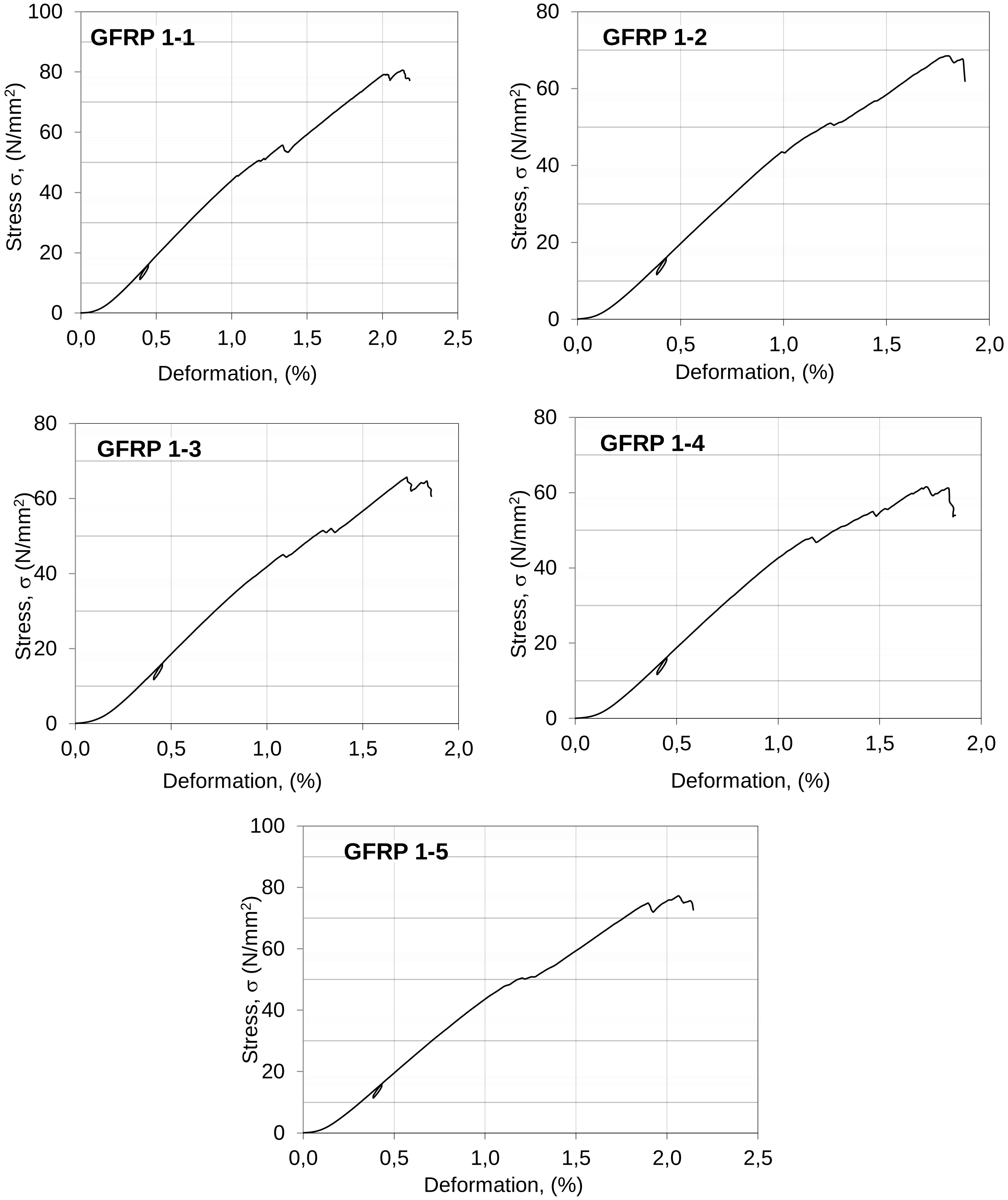


Figure 5. Stress and deformation graphs of split disk samples – **series 1** from Zwick/Roell Z400 universal tensile testing machine

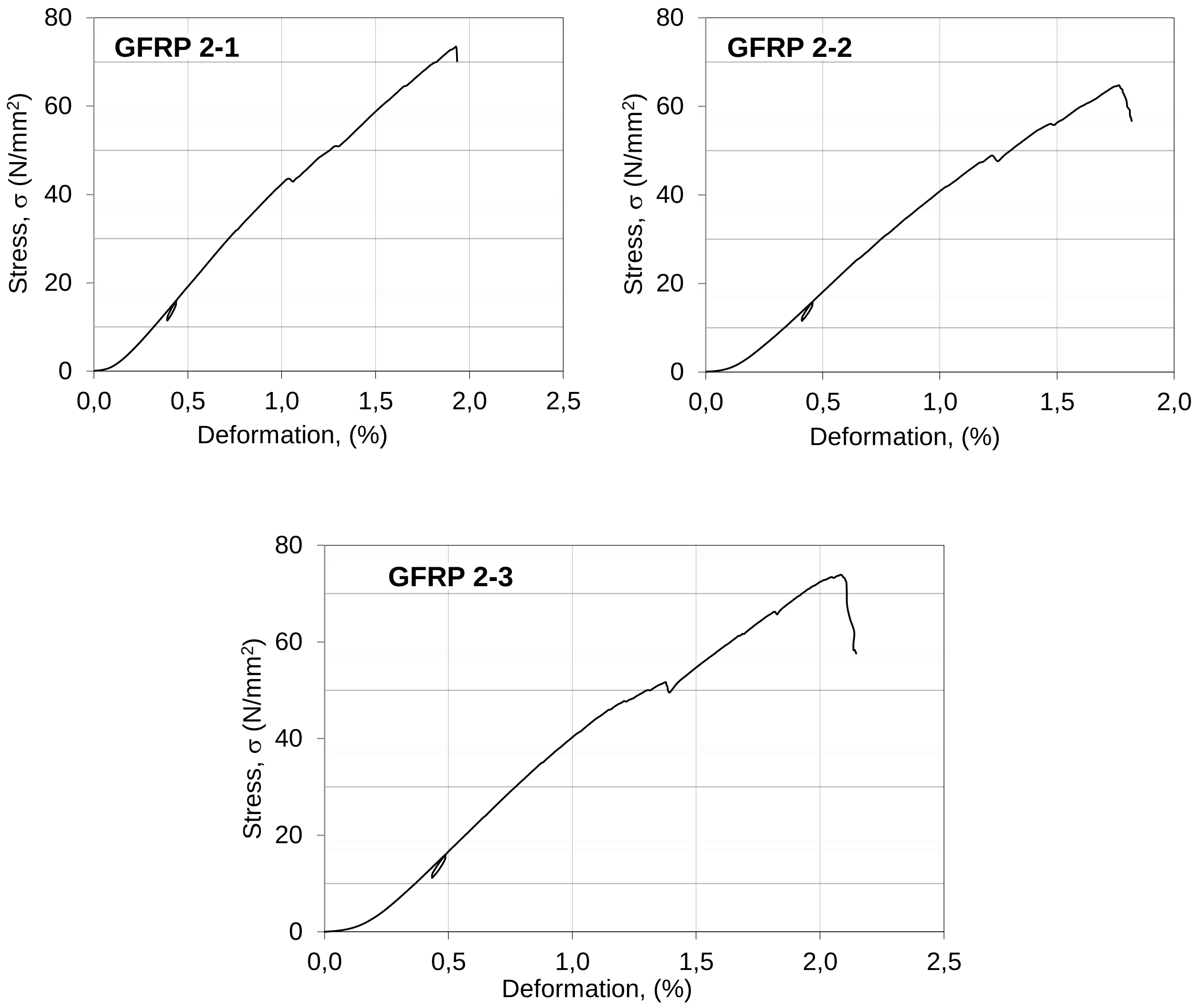
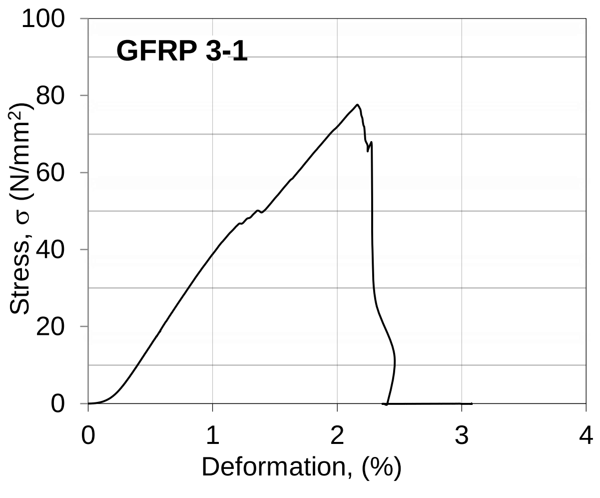
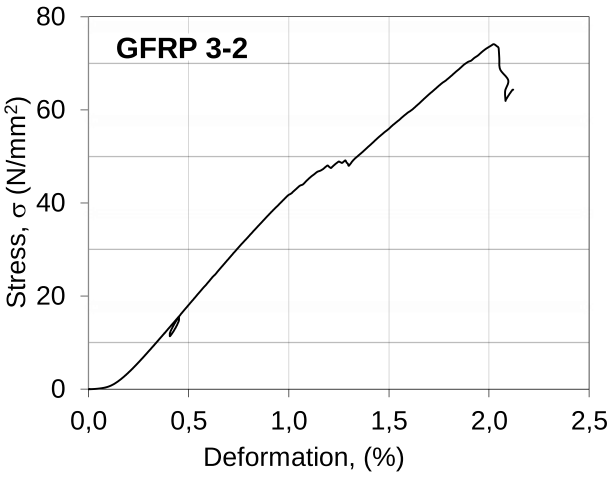
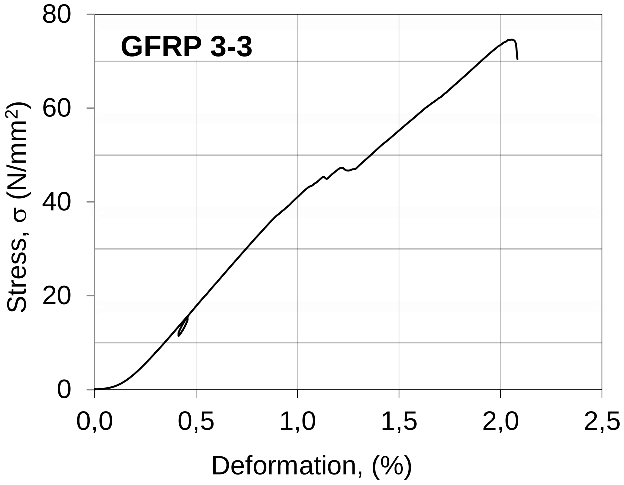
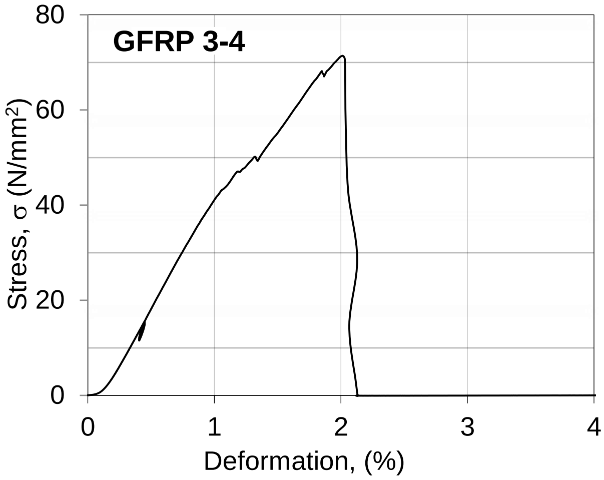


Figure 6. Stress and deformation graphs of split disk samples – **series 2** from Zwick/Roell Z400 universal tensile testing machine









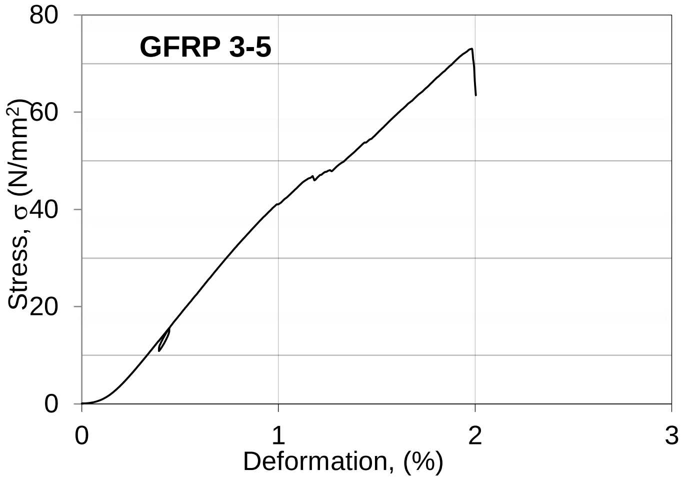
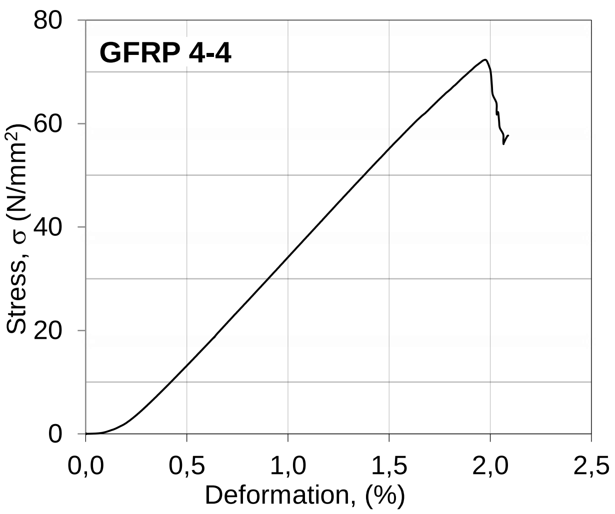


Figure 7. Stress and deformation graphs of split disk samples – **series 3** from Zwick/Roell Z400 universal tensile testing machine



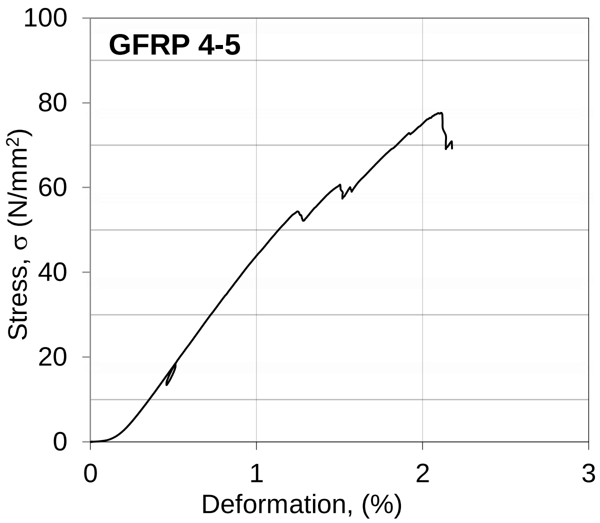


Figure 8. Stress and deformation graphs of split disk samples – **series 4** from Zwick/Roell Z400 universal tensile testing machine

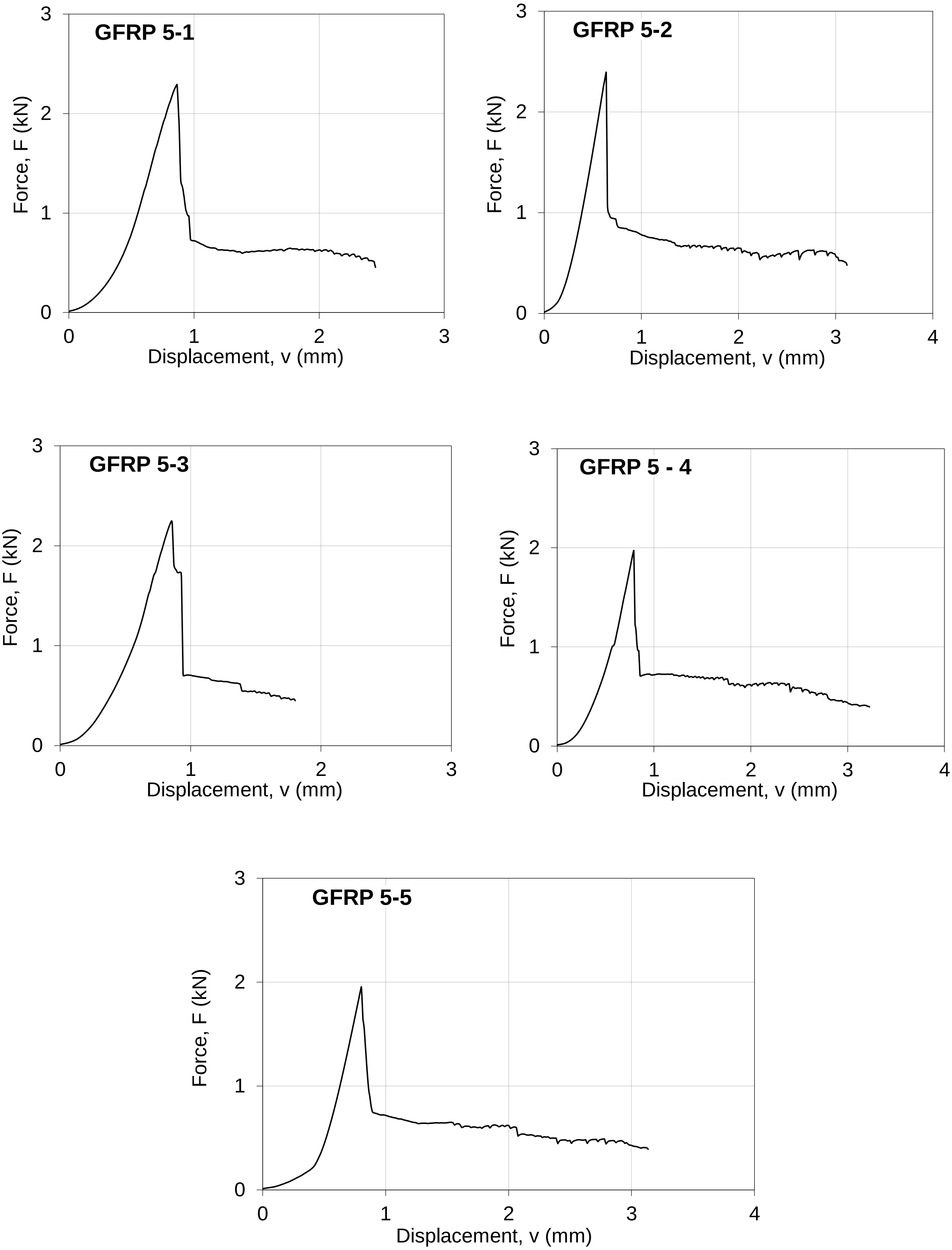


Figure 9. Force and displacement graphs of split disk samples – **series 5** from Zwick/Roell Z050 universal tensile testing machine

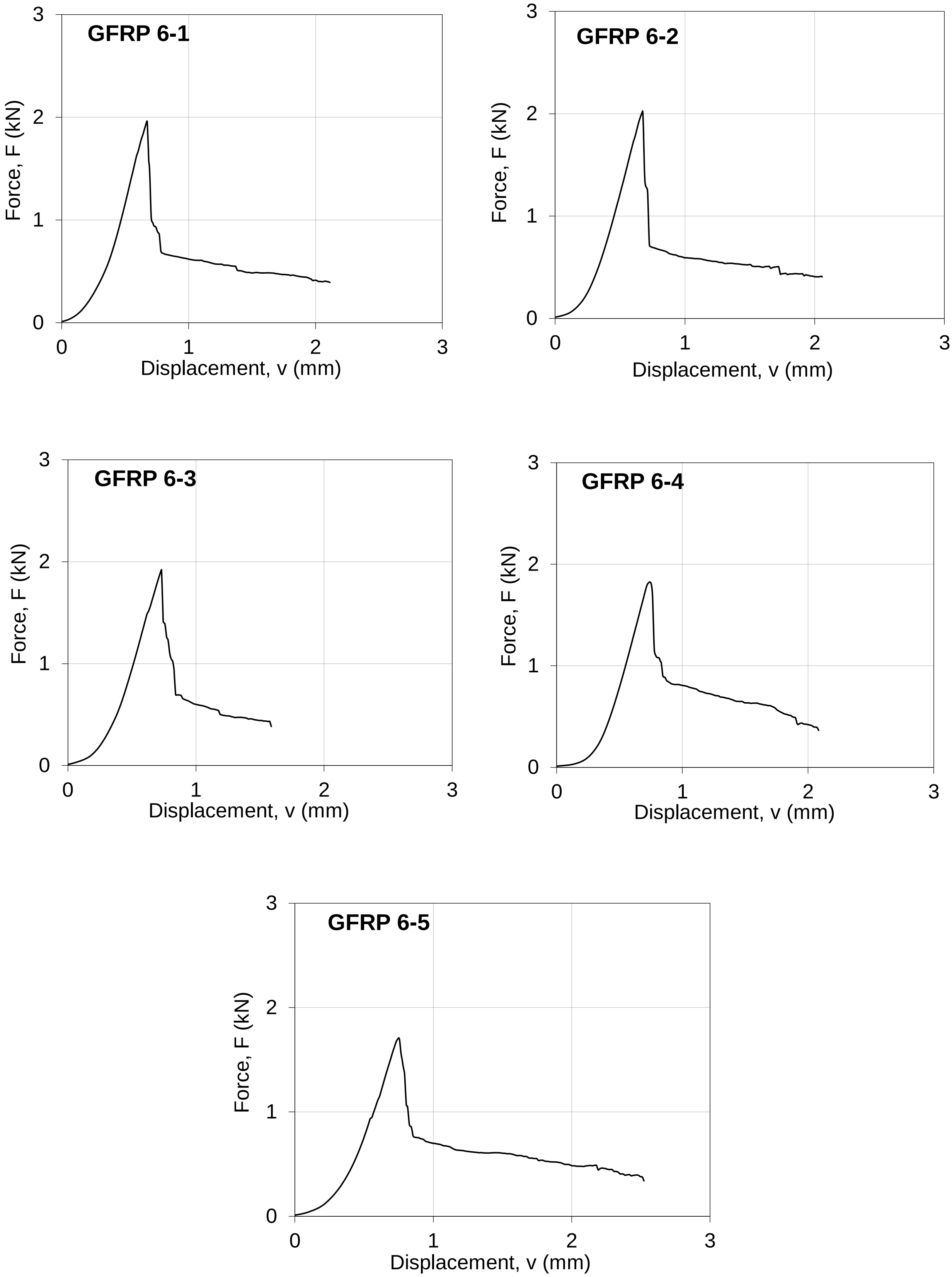


Figure 10. Force and displacement graphs of split disk samples – **series 6** from Zwick/Roell Z050 universal tensile testing machine

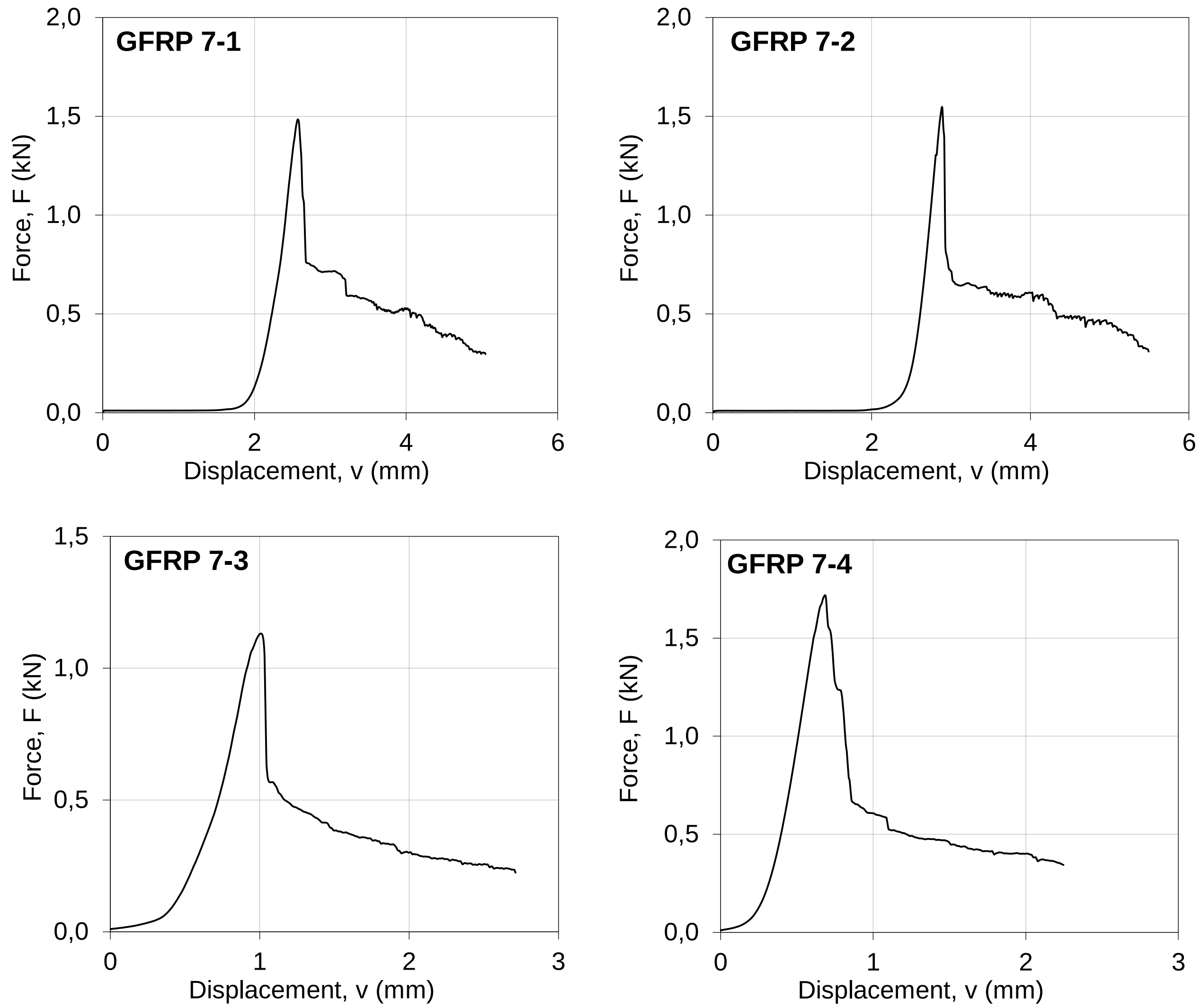


Figure 11. Force and displacement graphs of split disk samples – **series 7** from Zwick/Roell Z050 universal tensile testing machine

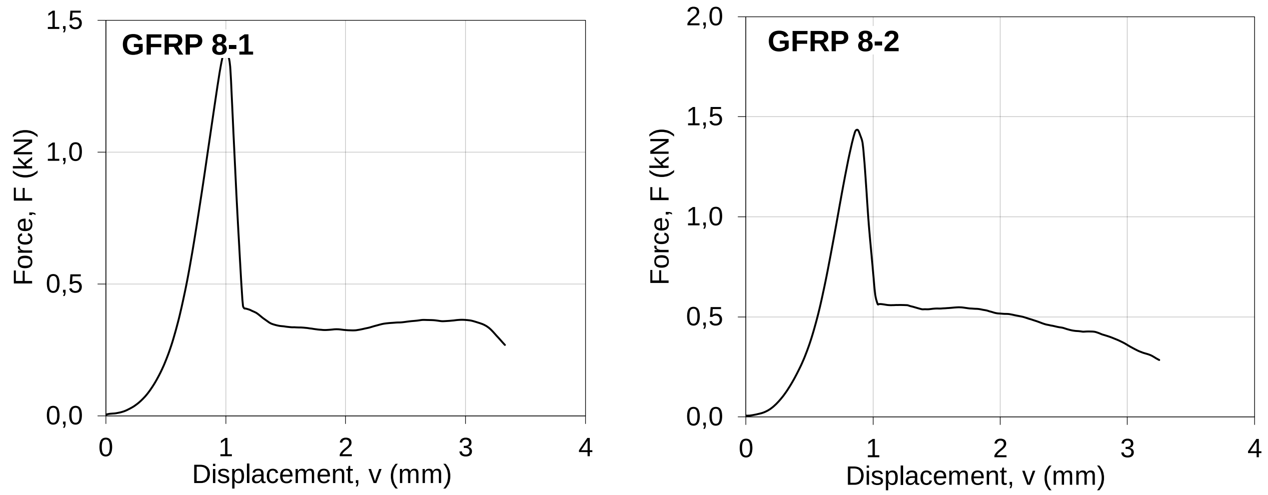


Figure 12. Force and displacement graphs of split disk samples – **series 8** from Zwick/Roell Z050 universal tensile testing machine

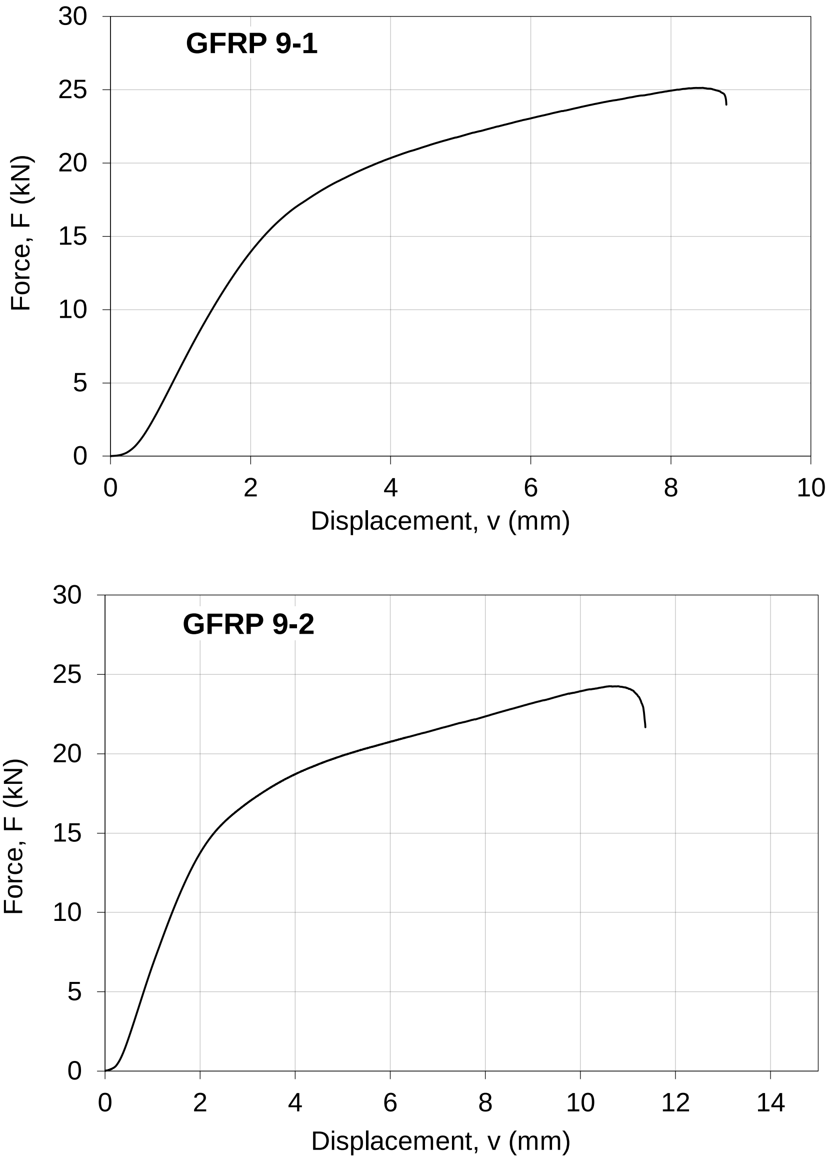


Figure 13. Force and displacement graphs of split disk samples – **series 9** from Zwick/Roell Z050 universal tensile testing machine

From the results shown in table 3 and from curves on figures can be observed that split-disk specimens 1-4 had shown the best results. These samples were winded with angle 90^0. In contrary, specimens 5 - 8 winded with angle 10^0 had shown much lower value than tensile strength from samples 1 - 4. The split-disk specimens 9 winded with angle 45^0 had shown ten times better tensile strength from samples 5 – 8 but less than samples 1 – 4. From received results it can be noticed that tensile properties of composite specimens depended from winding angles in filament winding technology. Namely, the bigger winding angle lead to higher hoop tensile properties of filament-wound tubular samples. Also, it can be noticed that fiber tension and velocity of the filament winding don’t influence the tensile strength of the specimens. Some pictures of the failure surfaces obtained during tensile tests are presented on the figure 14.

a) 10^{o} winding angle

b) 90^{o} winding angle

c) 45^{o} winding angle

Figure 14. Damages made during tensile tests of the split-disk specimens with different winding angle of the glass fibers

The results of the testing method of the pipe specimens for determination of the transverse compressive properties of the filament wound pipes are presented in Table 3.

Determination of transverse compressive properties of filament-wound composite tubular specimens was the second mechanical characteristic which was determined in frame of this STSM. For this test method three specimens: filament wound cylinders with 100 mm in diameter and 140 mm in length bonded into two end fixtures were tested from each testing group. The transverse compressive strength was determined from the maximum load carried before failure. Mainly, the ultimate compressive strength of the specimens were determined. In addition, the average of these results were calculated for each group, and with the aid of this data, the general behavior of the specimens were determined. The transverse compressive strength of the specimens were calculated by using the following equation:

$$\sigma = \frac{F \max}{A} \quad (2)$$

where:
A is the cross-sectional area,

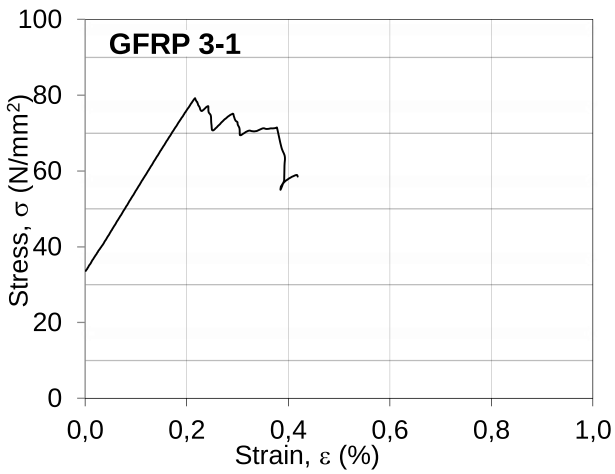
$$A = \frac{\pi}{4}\left(OD^2 - ID^2\right)$$

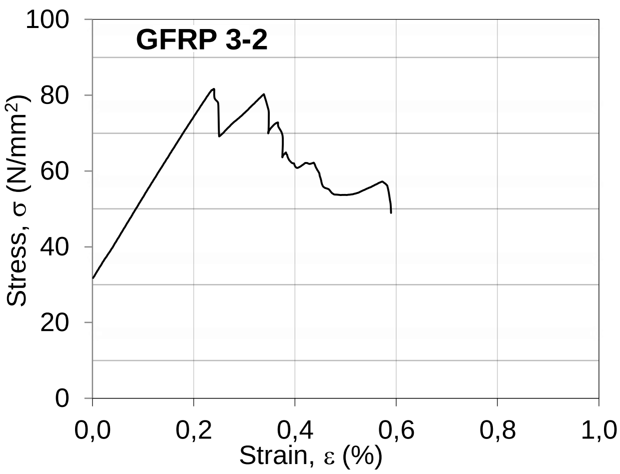
and ID and OD are the average inner and outer diameters respectively.

Table 3. Transverse compressive strength results of pipe specimens

Sample Designation		l (mm)	OD (mm)	ID (mm)	A $(mm)^2$	F_{max} (kN)	F average (kN)	σ (MPa)	σ average (MPa)
1	1-1	140,00	106,20	99,90	10,20	913,92	1029,21	89,60	102,67
	1-2	140,00	106,20	100,00	10,03	1127,97		112,46	
	1-3	139,80	106,10	100,00	9,87	1045,73		105,95	
3	3-1	140,00	106,20	100,00	10,03	792,37	856,60	79,00	83,86
	3-2	140,00	106,30	99,90	10,37	817,78		78,86	
	3-3	140,10	106,20	100,00	10,30	959,65		93,71	
4	4-1	140,00	106,85	100,10	10,95	1070,58	1019,99	97,77	93,58
	4-2	140,10	106,60	100,00	10,70	962,36		89,94	
	4-3	140,00	106,90	100,10	11,04	1027,05		93,03	
5	5-1	139,90	106,65	99,60	11,46	2558,90	3156,61	223,29	274,54
	5-2	140,00	106,60	99,50	11,54	3518,43		304,89	
	5-3	140,10	106,70	99,50	11,71	3392,50		289,71	
6	6-1	140,00	106,20	99,50	10,87	3263,93	3025,21	300,27	271,49
	6-2	140,00	106,60	99,60	11,37	3514,24		309,08	
	6-3	139,90	106,40	99,50	11,20	2297,46		205,13	
7	7-1	140,05	106,20	100,00	10,03	2358,45	2241,28	235,14	216,59
	7-2	139,90	106,50	99,90	10,70	2582,23		241,33	
	7-3	140,00	106,35	100,00	10,29	1783,15		173,29	
8	8-2	140,10	106,40	100,00					
	8-3	140,10	106,30	100,00					
9	9-1	140,10	106,60	99,50	11,54	1512,78	1539,93	131,09	131,82
	9-2	139,90	106,80	99,45	11,96	1600,01		133,78	
	9-3	140,10	106,60	99,50	11,54	1507,01		130,59	

Figures 15 - 19 show a typical stress and strain diagrams at ambient temperature for some tested samples series on universal testing machine Zwick/Roell with max stress of 400MPa and loading speed of 0.3 in/min.





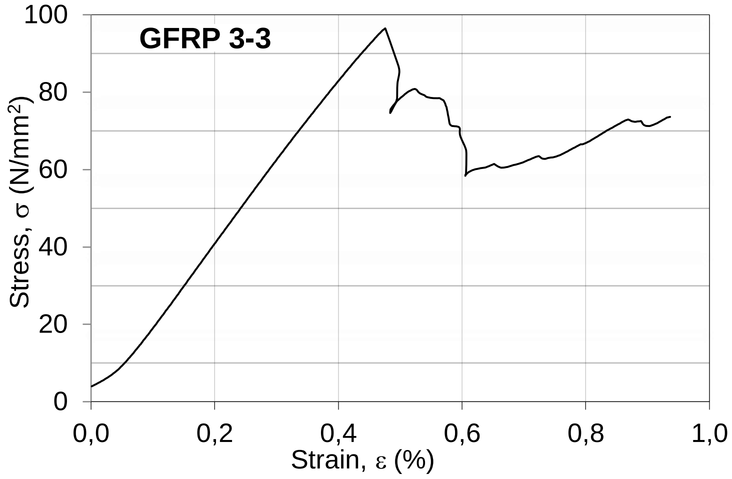


Figure 15. Stress and strain curves of pipe samples – **series 3** from transverse compressive testing

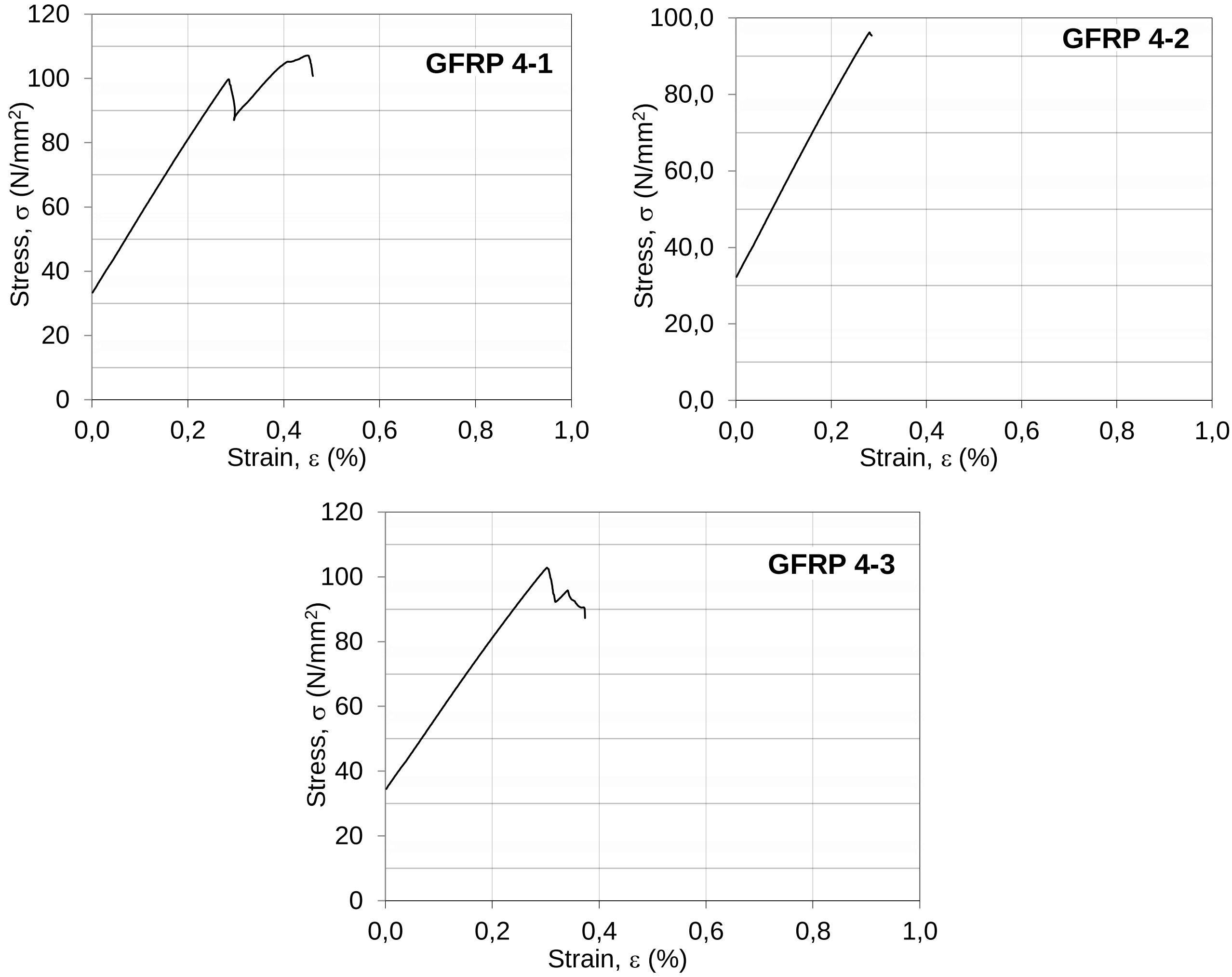


Figure 16. Stress and strain curves of pipe samples – **series 4** from transverse compressive testing

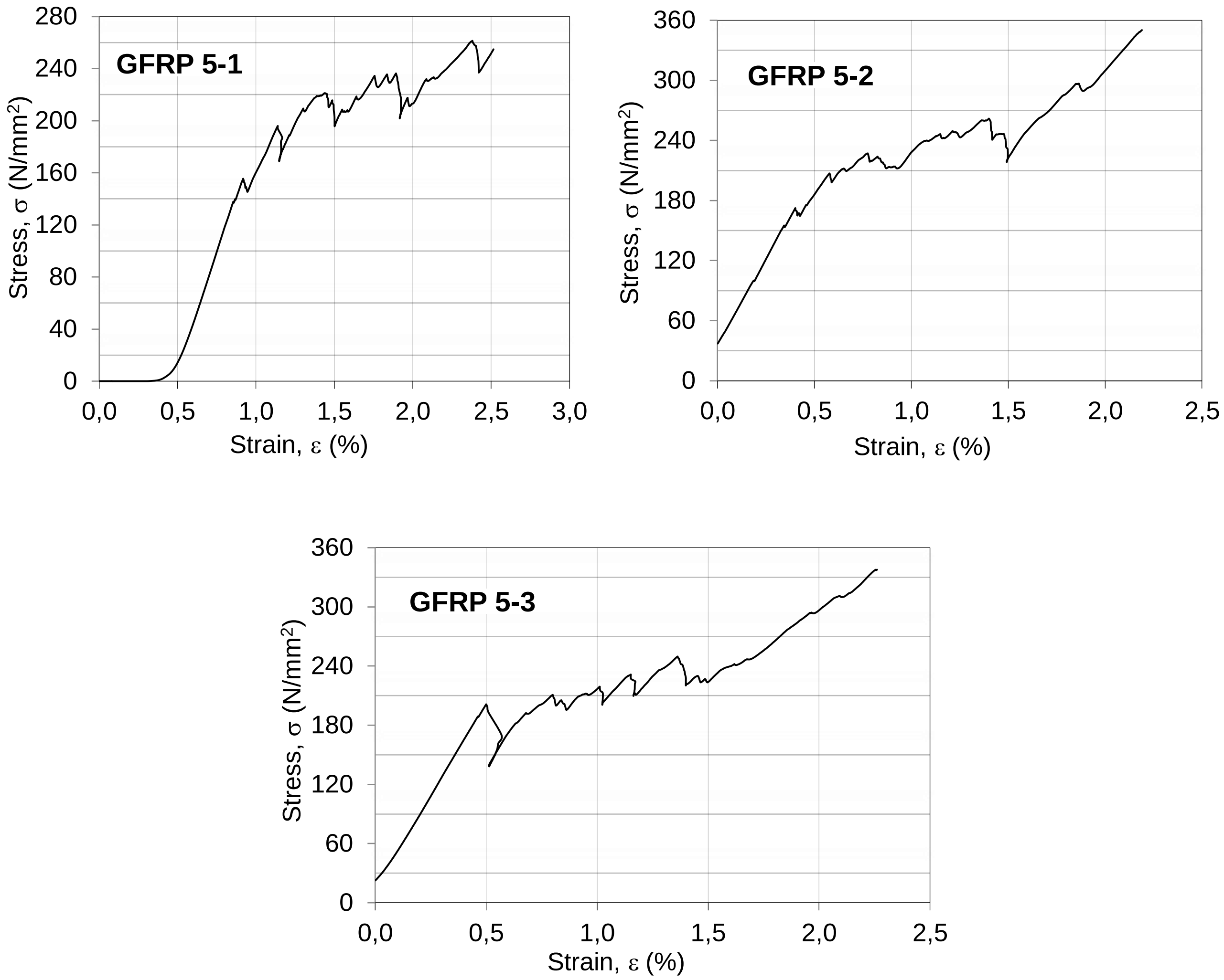
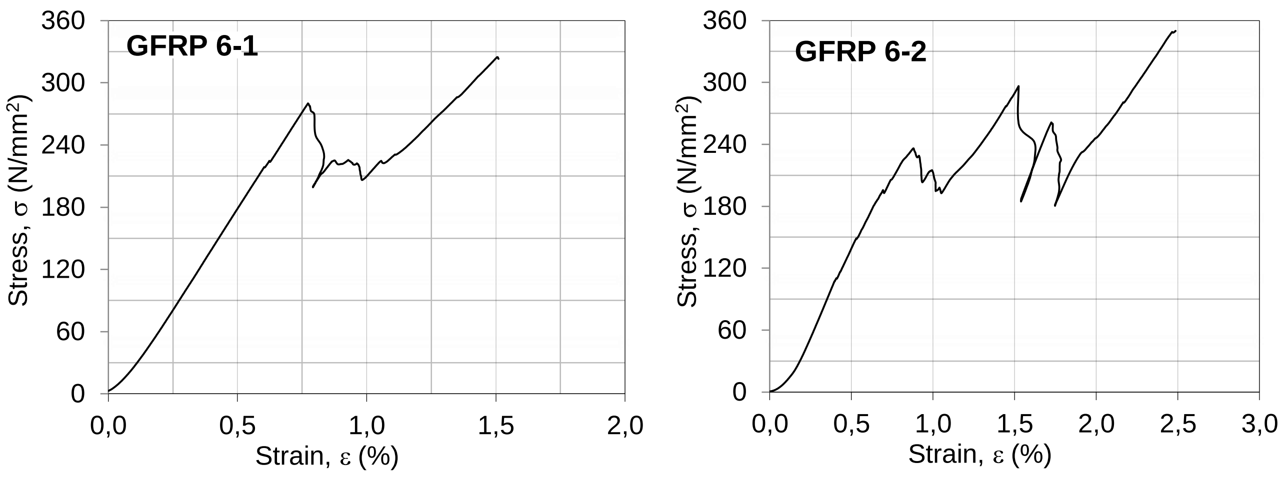


Figure 17. Stress and strain curves of pipe samples – **series 5** from transverse compressive testing



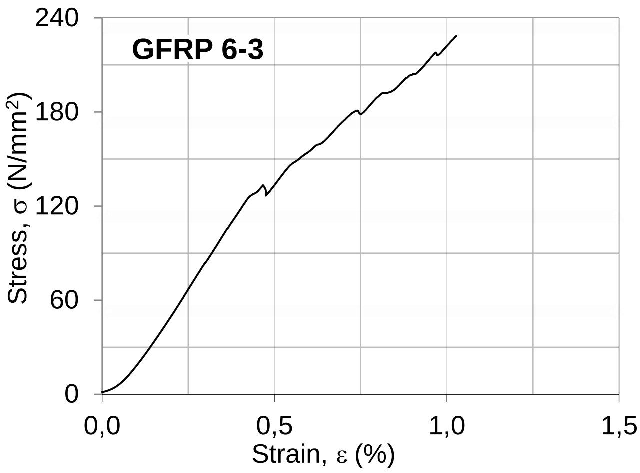
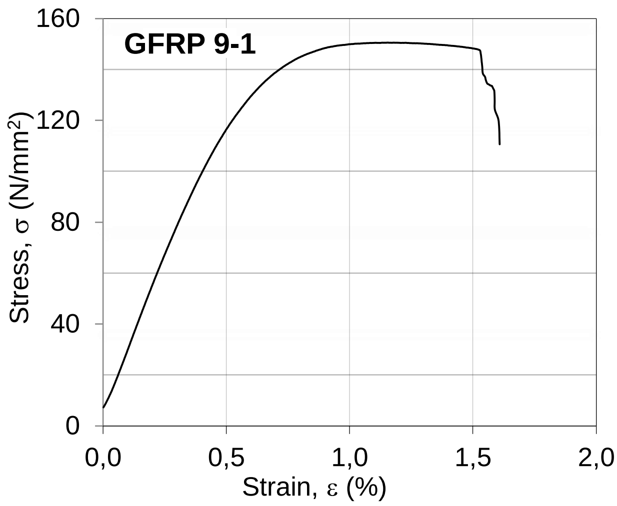
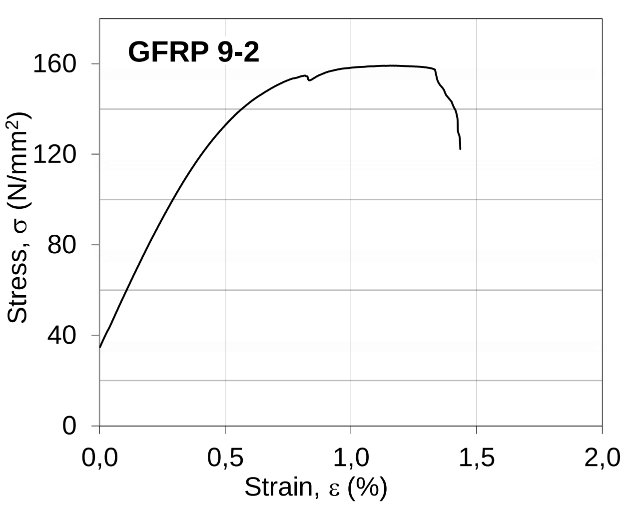


Figure 18. Stress and strain curves of pipe samples – **series 6** from transverse compressive testing





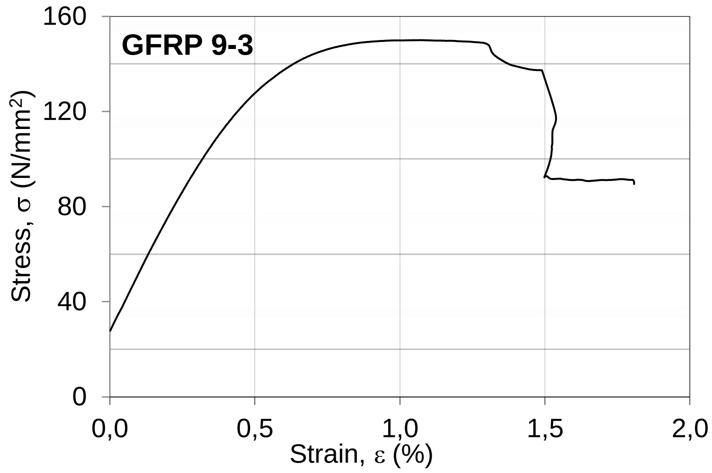
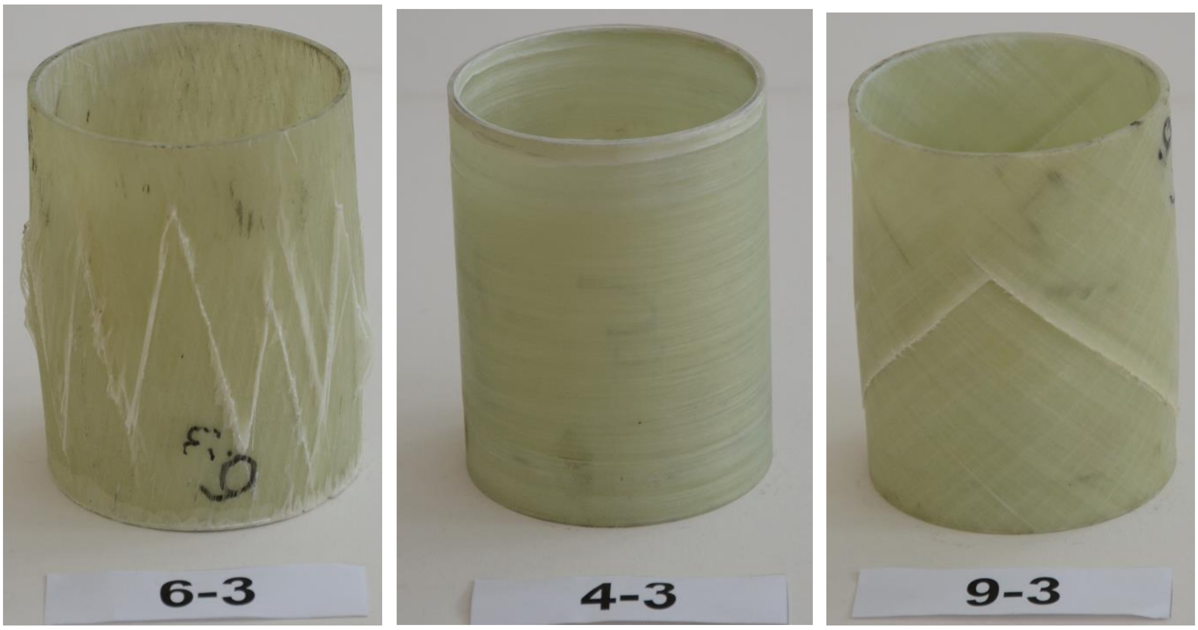


Figure 19. Stress and strain curves of pipe samples – **series 9** from transverse compressive testing

From the results shown in table 3 and from the curves can be noticed, that specimens 5-8 had shown the highest values for compressive strength. These samples were winded with angle 10^0. On contrary, specimens 1 - 4 winded with angle 90^0 had shown much lower value almost for 50%. The pipe specimens series 9 winded with angle 45^0 had shown better transverse compressive strength than specimens 1-4 but less strength of almost 50% than specimens 5-8. From received results it can be noticed that transverse compression properties of composite specimens depended from winding angles in filament winding technology. Namely, the lower winding angle lead to higher transverse compression properties of filament-wound tubular samples. Also, it can be noticed slight influence of the fiber tension on the compression strength. Namely, the higher fiber tension leads to higher value of the compression strength. The velocity of the filament winding doesn't influence the compressive strength of the specimens. Some pictures of the failure surfaces obtained during compression tests are presented on the figure 20.



a) 10^o winding angle b) 90^o winding angle c) 45^o winding angle

Figure 20. Damages made during compression tests of the pipe specimens with different winding angle of the glass fibers

Part 2. Thermal analysis

The thermal characteristics of the epoxy resin and of the different models of filament wound glass fibers/epoxy composites were measured using both Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analyses (TGA) methods.

The thermal measurements of the epoxy resin and composites are useful in determining on key physical and chemical characteristics of the resin and of the composites, including: glass transition or softening temperature (Tg), onset and completion of cure, heat of cure, maximum rate of cure, percent cure, heat capacities (Cp). These properties can be used to address some of problems: establishment of optimal processing conditions, estimation of percent cure of end products, analyses of competitive materials etc.

The results from the thermogravimetric analyses of an uncured epoxy resin are presented in Figure 21.

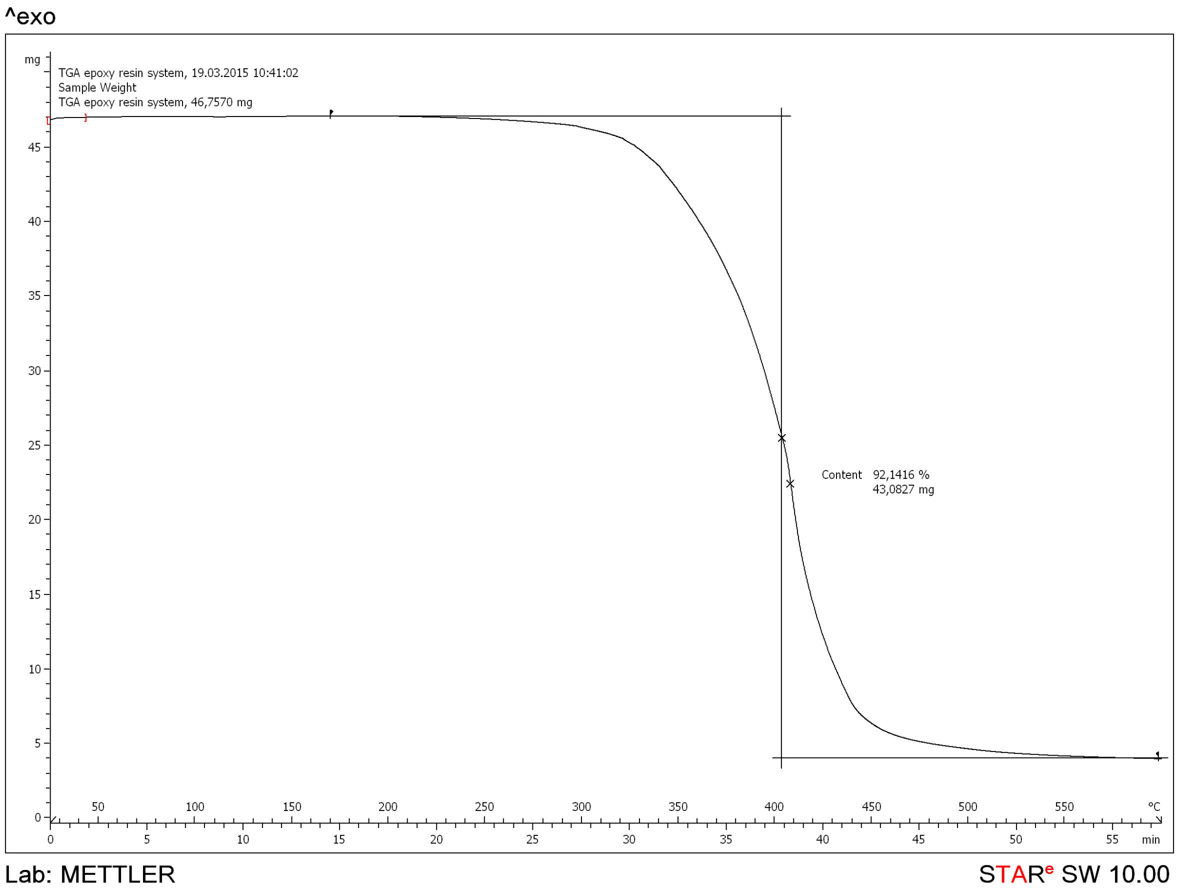
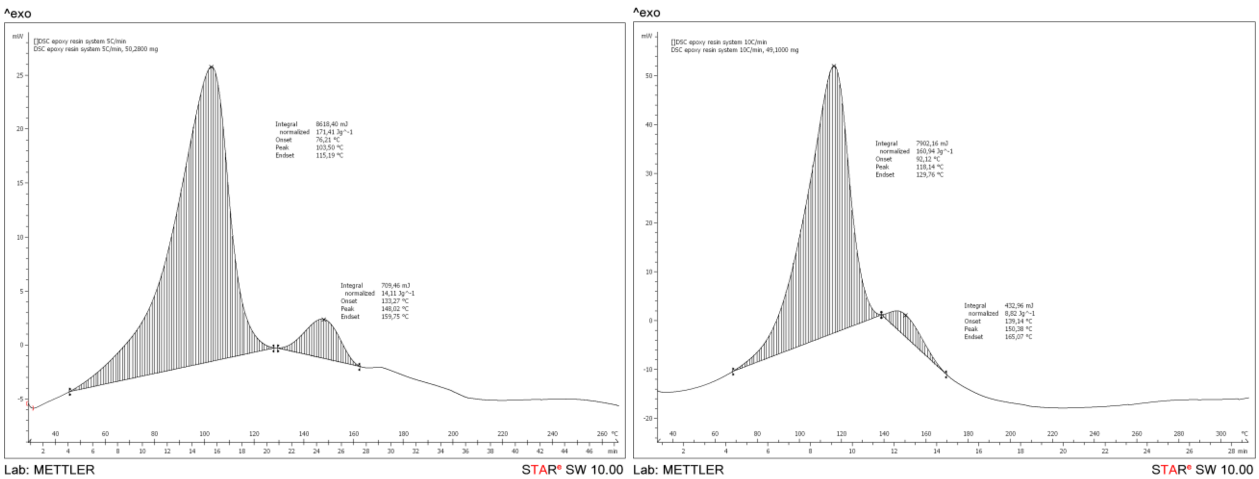


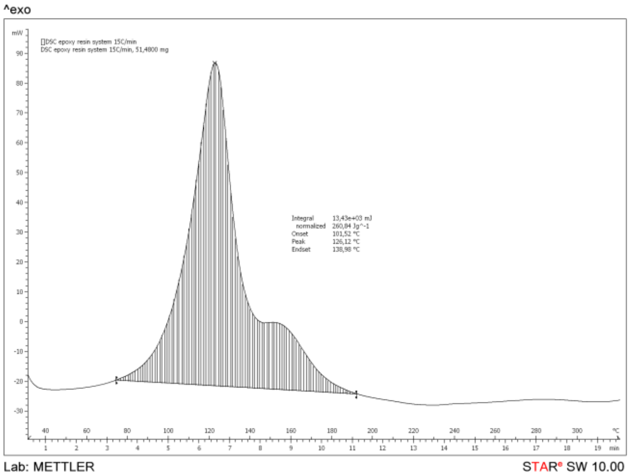
Figure 21. Thermogravimetric curve of an uncured epoxy resin system: weight loss (mg) versus temperature

An uncured epoxy resin system loses about 8% of its weight until 350 °C, followed by ongoing 90% weight loss to 450–500 °C. After that, the weight loss continues with a slower degradation rate. It should be noted that at a temperature of 600 °C, the epoxy resin system exhibit residual weight of about 8%.

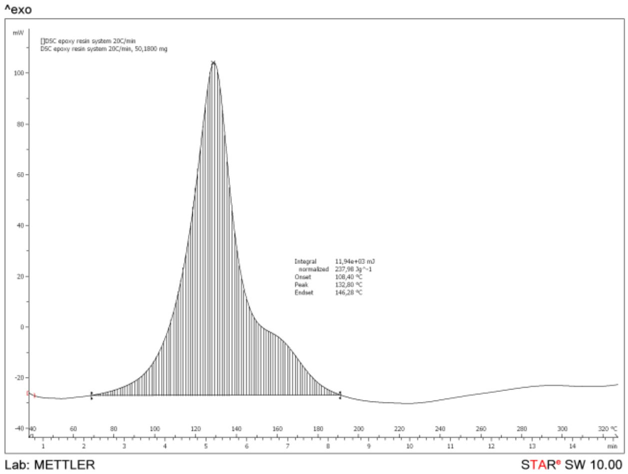
DSC results of an uncured epoxy resin system are obtained by heating an uncured epoxy resin system at a rate of 5, 10, 15 and 20 °C/min over a temperature range of 20 to 350 °C under a constant flow of nitrogen of 50 mL/min (Figure 22).



a) heating rate 5°C/min b) heating rate 10°C/min



a) heating rate 15°C/min



b) heating rate 20°C/min

Figure 22. DSC results on uncured epoxy resin system

The graphs show the heat flow as a function of the sample temperature. The glass temperature event (Tg) is observed as a stepwise increase in the heat flow or heat capacity. The onset of cure is the temperature at which the heat flow deviates from the linear response and the peak temperature reflects the maximum rate of curing of the resin. At the completion of curing or crosslinking, the DSC heat flow returns to a quasilinear response. The area of the peak can be integrated to give the heat of cure, ΔHcure (J/g).
As a thermosetting resin cures or crosslinks, two main things happen: Tg increases and heat of cure decreases.

Table 4. DSC analysis data obtained by heating an uncured epoxy resin system at a rate of 5, 10, 15 and 20 °C/min

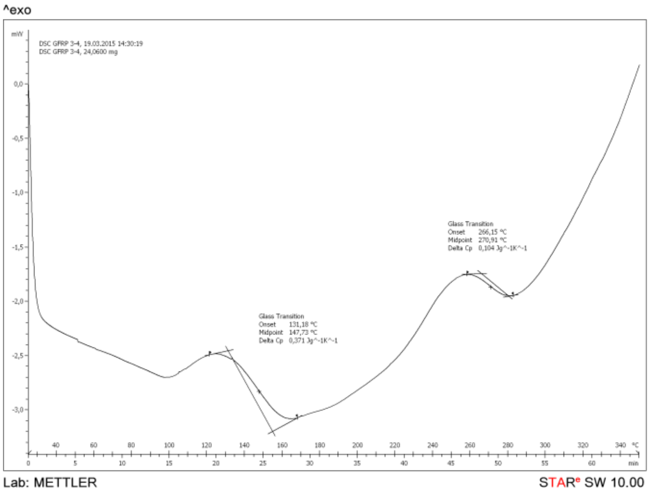
	Heating rate of an uncured epoxy resin system					
	5°C/min		**10°C/min**		**15°C/min**	**20°C/min**
ΔHcure (Jg^{-1})	171,41	14,11	160,94	8,82	260,84	237,96
Onset (°C)	76,21	133,27	92,12	139,14	101,52	108,40
Peak (°C)	103,50	148,02	118,14	150,38	126,12	132,80
Endset (°C)	115,19	159,75	129,76	169,07	138,98	146,28

Based on the DSC scans for an uncured epoxy resin system at different heating rate and from the DSC analysis data shown on Table 4, it can be noticed that, two exothermic peaks were observed at heating rate 5 °C/min and 10 °C/min . In the first peak the glass transition temperature (Tg) is observed at a temperature of 103,50°C and 148,2°C/min respectively on the heating rate 5 and 10 °C/min, whereas in the second peak glass transition temperature (Tg) has been increased to 118,14°C and 150,38°C/min, respectively. But, from DSC analysis data obtained by heating an uncured epoxy resin system at a rate of 15 and 20 °C/min, one exothermic peak was observed at 126,12°C and 132,80°C respectively.

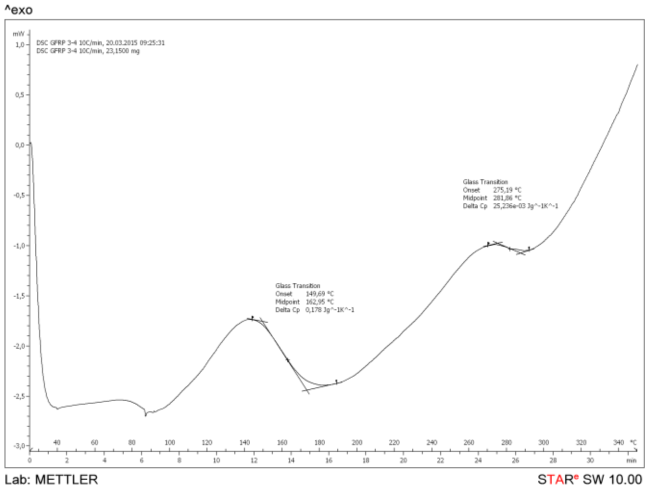
From all configuration of filament wound pipes (glass fiber reinforced plastic – GFRP) the thermal analysis (DSC and TGA) were measured for the composites:
- with winding angle 90^o, fiber tension of 34N and with lower and upper level of velocity of winding (samples 3-4 and 4-4, respectively),
- with winding angle 10^o, fiber tension of 34N and with lower and upper level of velocity of winding (samples 7-4 and 8-4, respectively),

- on the primary level: winding angle 45°, fiber tension of 47N and velocity of winding 13,125 m/min (sample 9-4).

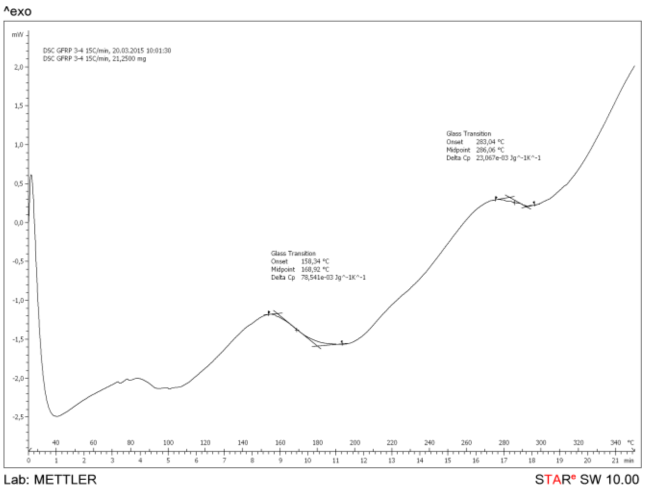
The results of DSC analyses of the filament wound pipes samples 3-4, 4-4, 7-4, 8-4 and 9-4 are presented on Figures 23-27. Also, DSC results of the analyzed filament wound pipes are summarized in Table 5.



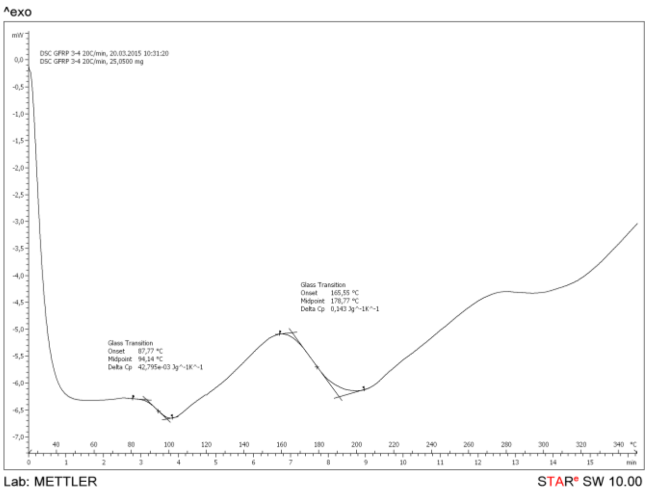
a) heating rate 5°C/min



b) heating rate 10°C/min

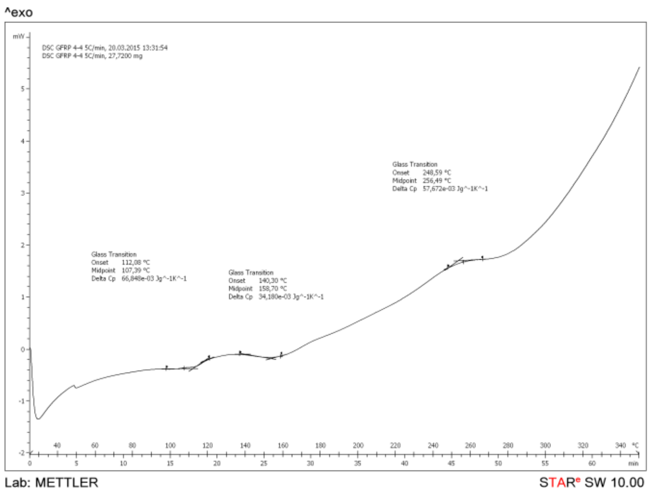


a) heating rate 15°C/min

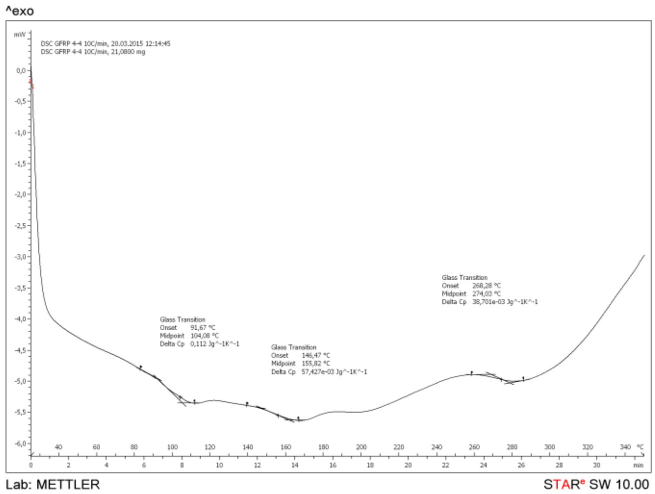


b) heating rate 20°C/min

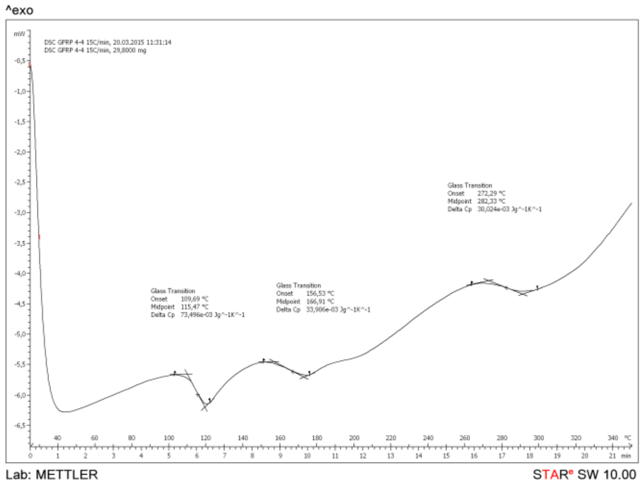
Figure 23. DSC results on filament wound pipe – **GFRP 3 – 4**



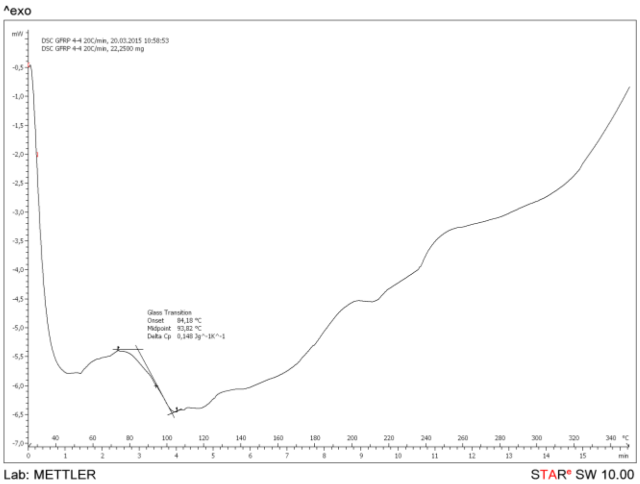
a) heating rate 5°C/min



b) heating rate 10°C/min

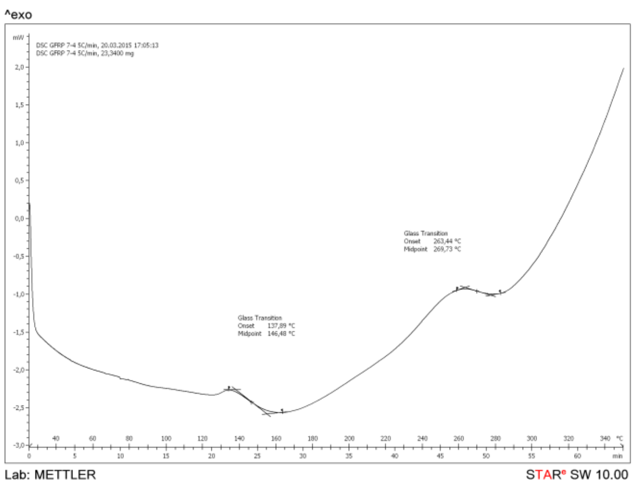


a) heating rate 15°C/min

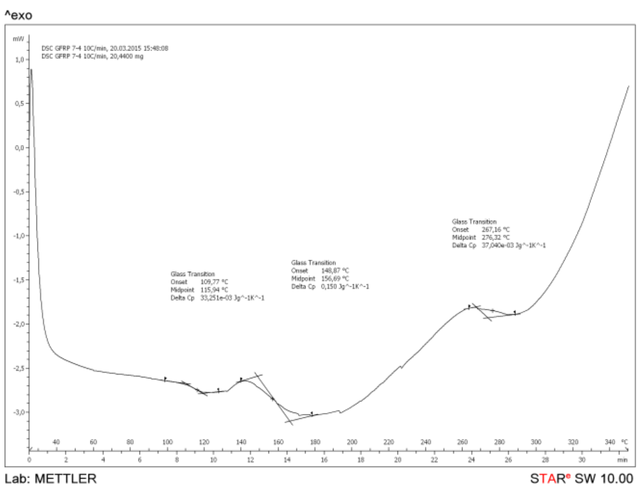


b) heating rate 20°C/min

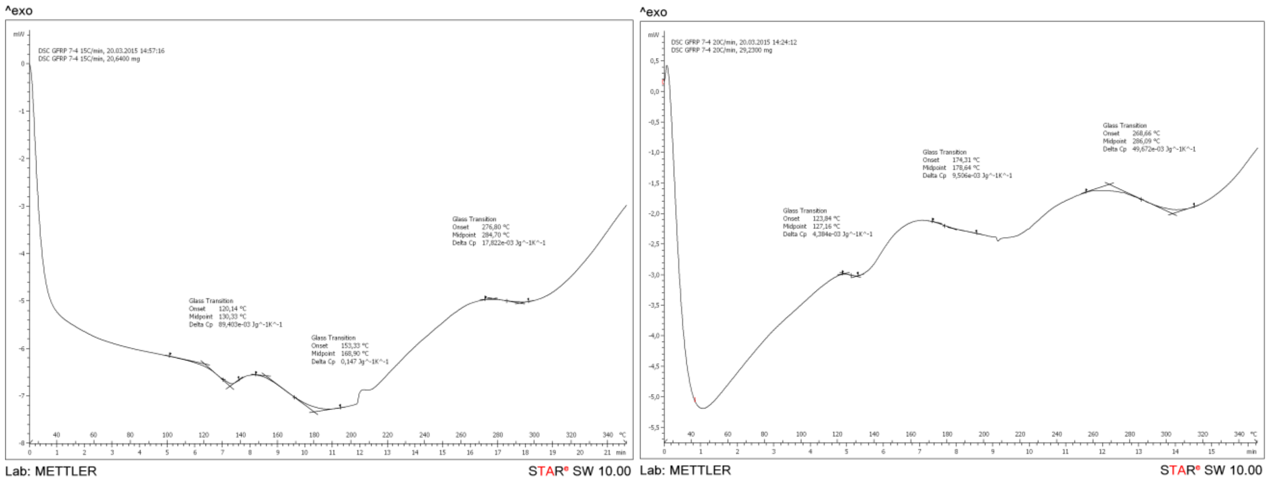
Figure 24. DSC results on filament wound pipe – **GFRP 4 - 4**



a) heating rate 5°C/min

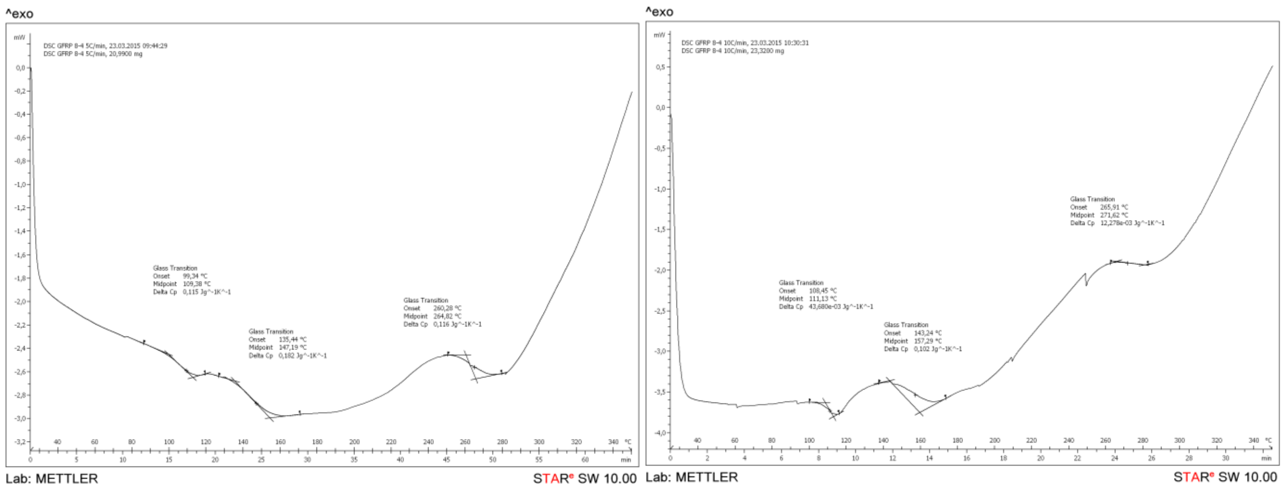


b) heating rate 10°C/min

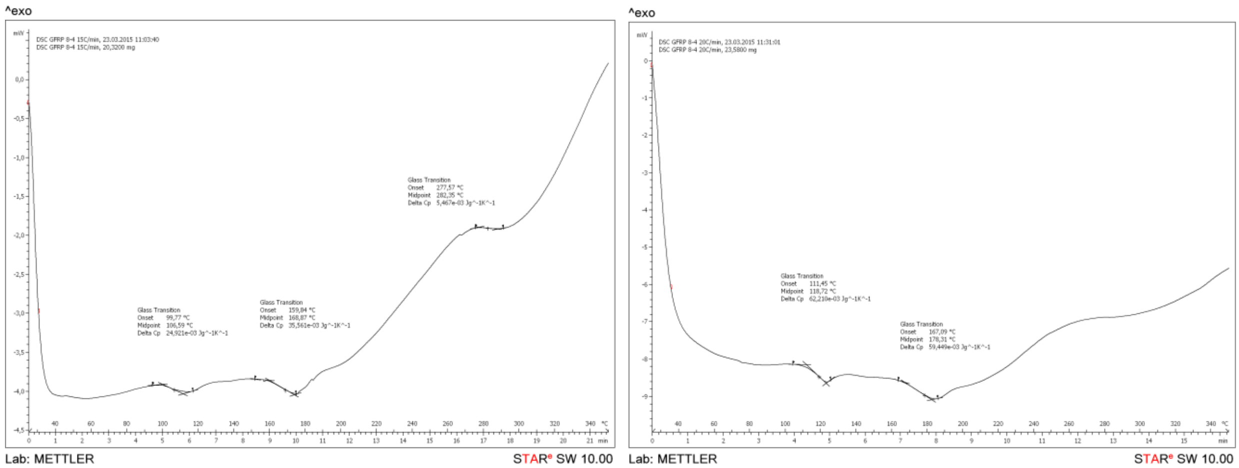


a) heating rate 15°C/min b) heating rate 20°C/min

Figure 25. DSC results on filament wound pipe – **GFRP 7 – 4**

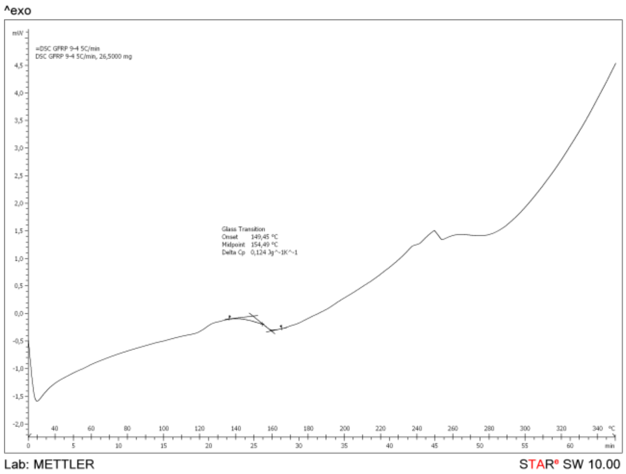


a) heating rate 5°C/min b) heating rate 10°C/min

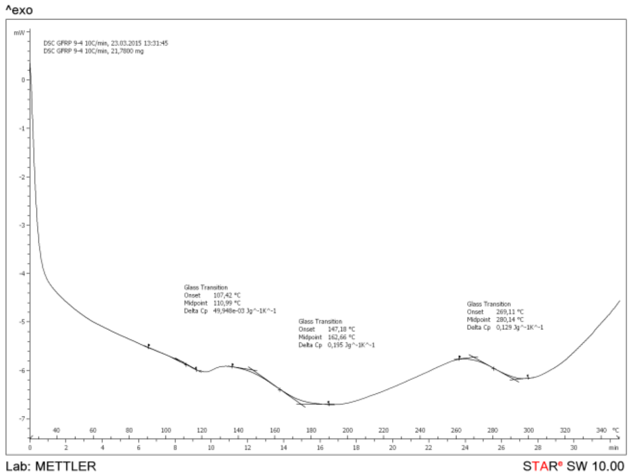


a) heating rate 15°C/min b) heating rate 20°C/min

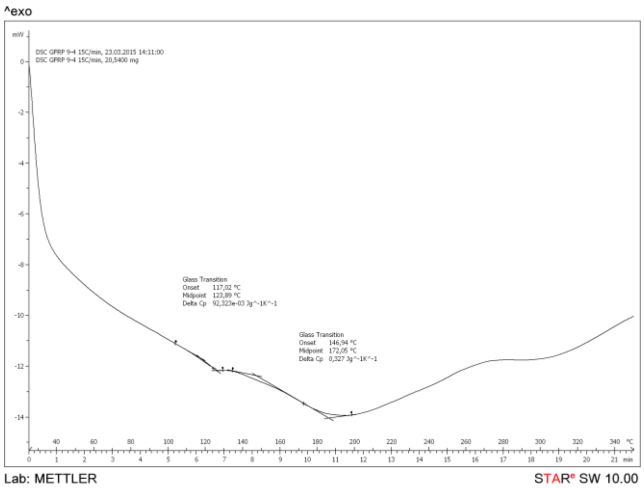
Figure 26. DSC results on filament wound pipe – **GFRP 8 – 4**



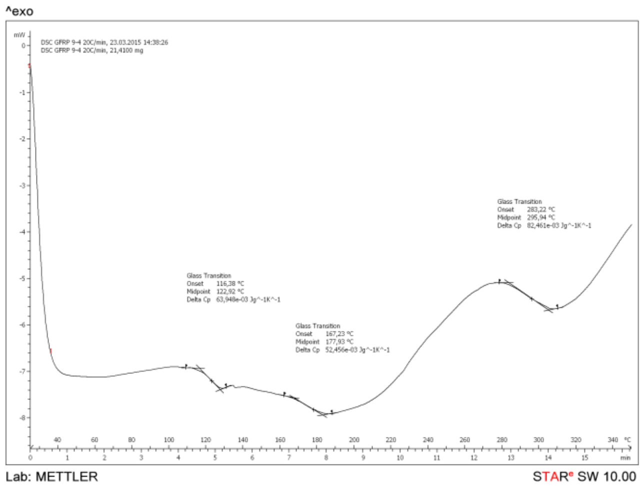
a) heating rate 5°C/min



b) heating rate 10°C/min



a) heating rate 15°C/min



b) heating rate 20°C/min

Figure 27. DSC results on filament wound pipe – **GFRP 9 – 4**

Table 5. DSC analysis data obtained for GFRP 3-4, 4-4, 7-4, 8-4 and 9-4 by heating rate of 5,10,15 and 20 °C/min

Heating rate of **GFRP 3-4**								
Glass Transition	**5°C/min**		**10°C/min**		**15°C/min**		**20°C/min**	
Onset (°C)	131,18	266,15	149,69	275,19	158,34	283,04	87,77	165,55
Midpoint (°C)	147,73	270,91	162,95	281,86	168,92	286,06	94,14	178,77
ΔCp ($Jg^{-1}K^{-1}$)	0,371	0,104	0,178	0,025236	0,028541	0,023067	0,042795	0,149

Heating rate of **GFRP 4-4**									
Glass Transition	**5°C/min**			**10°C/min**			**15°C/min**			**20°C/ min**
Onset (°C)	112,08	140,30	248,59	91,67	146,47	268,28	109,69	156,53	272,29	84,18
Midpoint (°C)	107,39	158,70	256,49	104,08	155,82	274,03	115,47	166,91	282,33	93,82
ΔCp ($Jg^{-1}K^{-1}$)	0,067	0,034	0,058	0,112	0,057	0,039	0,073	0,034	0,03	0,148

Heating rate of **GFRP 7-4**											
Glass Transition	**5°C/min**		**10°C/min**			**15°C/min**			**20°C/min**		
Onset (°C)	137,89	263,44	109,77	148,87	267,16	120,14	153,33	276,80	123,84	174,31	268,66
Midpoint (°C)	146,48	269,73	115,94	156,69	276,32	130,33	169,90	284,70	127,16	178,64	296,09
ΔCp (Jg^{-1}K^{-1})			0,033	0,15	0,037	0,089	0,147	0,018	0,044	0,095	0,050

Heating rate of **GFRP 8-4**											
Glass Transition	**5°C/min**			**10°C/min**			**15°C/min**			**20°C/min**	
Onset (°C)	99,34	135,44	260,28	108,45	143,24	265,91	99,77	159,84	277,57	111,48	167,09
Midpoint (°C)	109,38	147,19	264,82	111,13	157,29	271,62	106,59	168,87	282,35	118,72	178,31
ΔCp (Jg^{-1}K^{-1})	0,115	0,182	0,116	0,044	0,102	0,012	0,025	0,035	0,055	0,062	0,060

Heating rate of **GFRP 9-4**									
Glass Transition	**5°C/min**	**10°C/min**			**15°C/min**		**20°C/min**		
Onset (°C)	149,45	107,42	147,18	269,11	117,02	146,94	116,38	167,23	283,22
Midpoint (°C)	154,49	110,99	162,66	280,14	123,89	172,05	122,92	177,93	295,94
ΔCp (Jg^{-1}K^{-1})	0,124	0,050	0,195	0,129	0,092	0,327	0,064	0,052	0,082

Based on the DSC scans for different configurations of filament wound pipes at different heating rate and from the DSC analysis data shown on Table 5, it can be observed that curves have two or three exothermic peaks. In all composites the first peak which indicates on the first glass transition temperature (Tg) is observed at a temperature range from 90°C to 120 °C, whereas the second peak glass transition temperature (Tg) have been increased to temperature range of 148°C - 179°C and the third peak glass transition temperature (Tg) from 256°C to 297 °C. Also, it can be noticed that the glass transition temperature (Tg) increases with increasing of the heating rate. The DSC scans give the heat capacity of the samples which is also required for the characterization of the glass transition process for polymer composites. The cross linking reaction between the resin and fibers in the filament wound pipes produced with lower and upper level of velocity of winding results in minor changes on the glass transition temperature. Obviously, the velocity of winding does not influence on the glass transition characteristics. The values for Tg of the analyzed composites are similar for all configuration of filament wound pipes which indicate that the degree of crosslinking is already reached in all composites. The values of the heat capacity of the composites prove that a high degree of crosslinking is already reached in all composites.

The thermal stability of filament wound pipes (glass fiber reinforced plastic – GFRP) composite samples was measured and about 20 mg of each sample was heated from 25°C to 1000°C at heating rate of 20°C/min under argon/air flow. Thermogravimetric (TGA) curves for the composite samples are shown in Figures 28 - 32, whereas TGA results are summarized in Table 6.

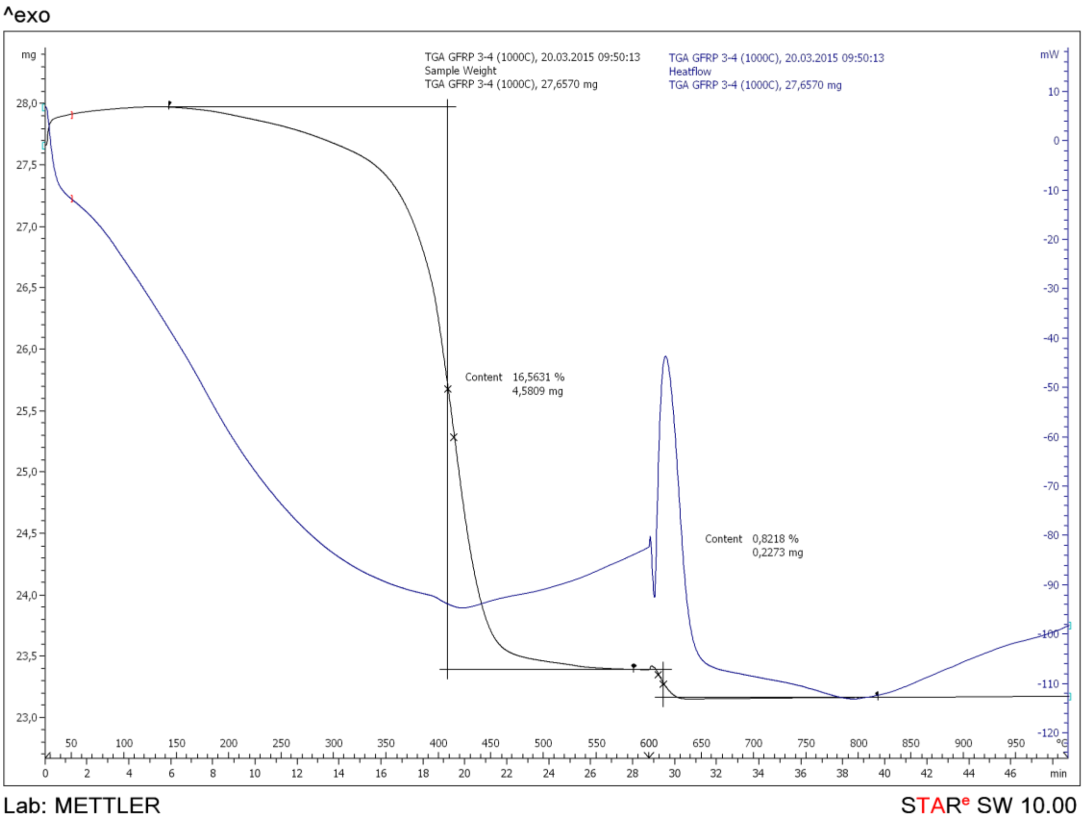


Figure 28. Thermogravimetric curve on filament wound pipe – **GFRP 3 – 4**: weight loss (mg) versus temperature

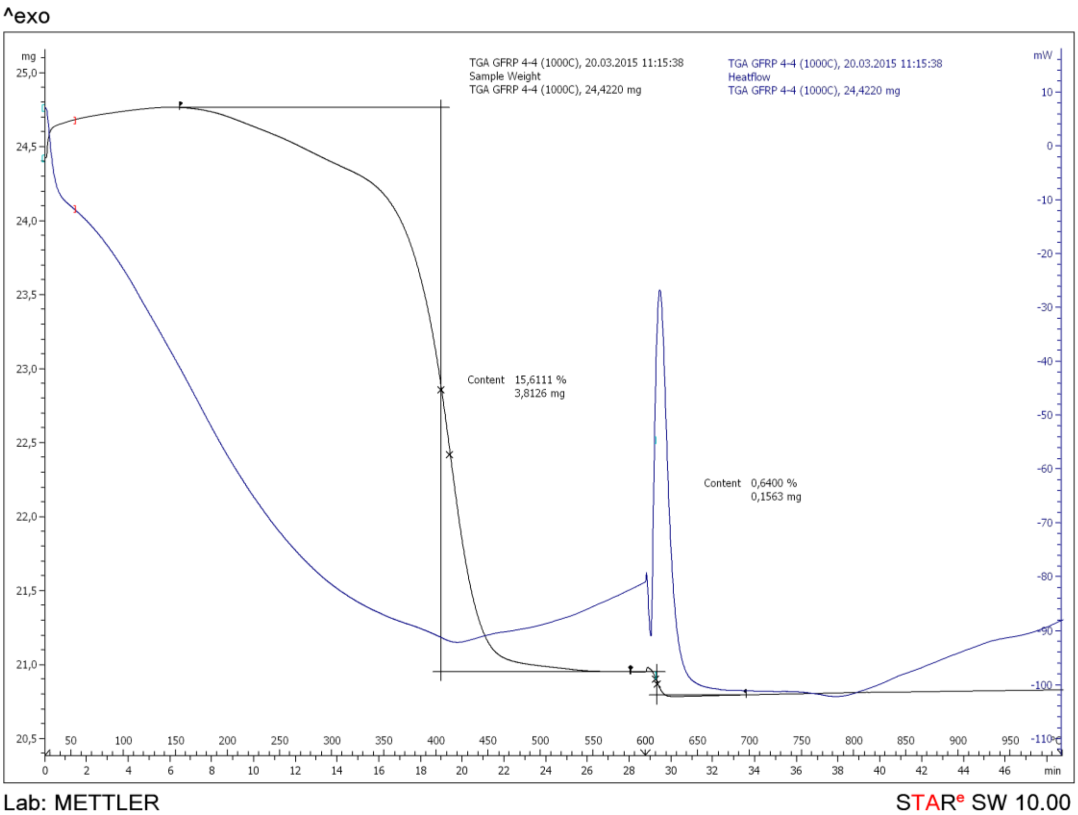


Figure 29. Thermogravimetric curve on filament wound pipe – **GFRP 4 – 4**: weight loss (mg) versus temperature

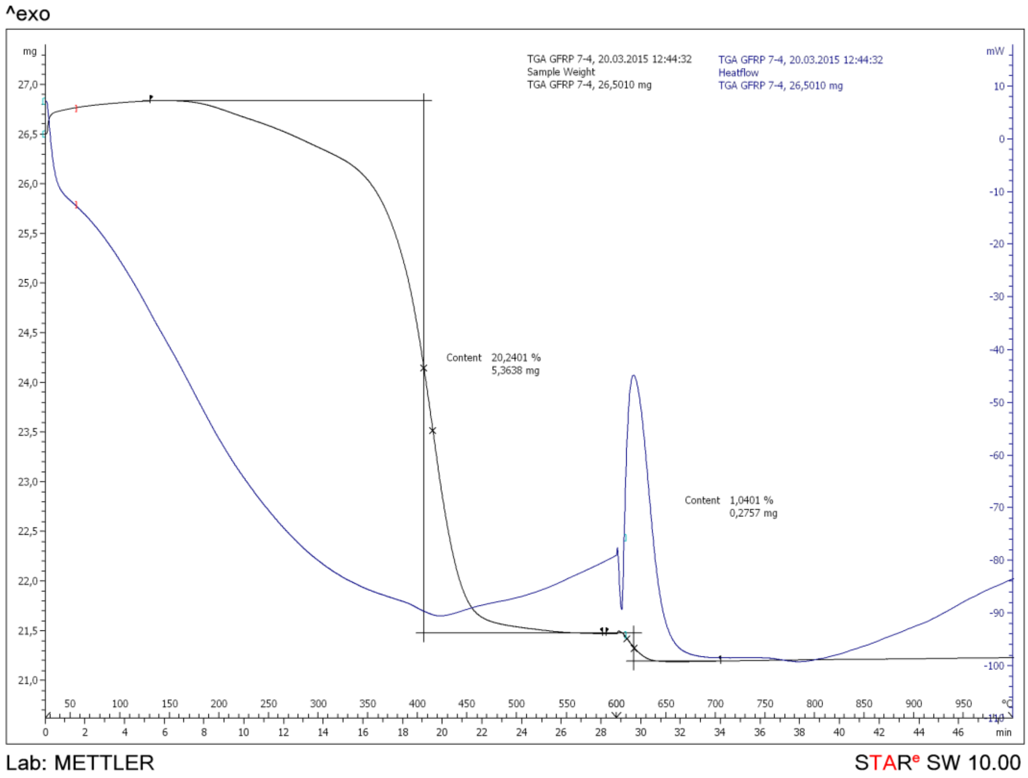


Figure 30. Thermogravimetric curve on filament wound pipe – **GFRP 7 – 4**: weight loss (mg) versus temperature

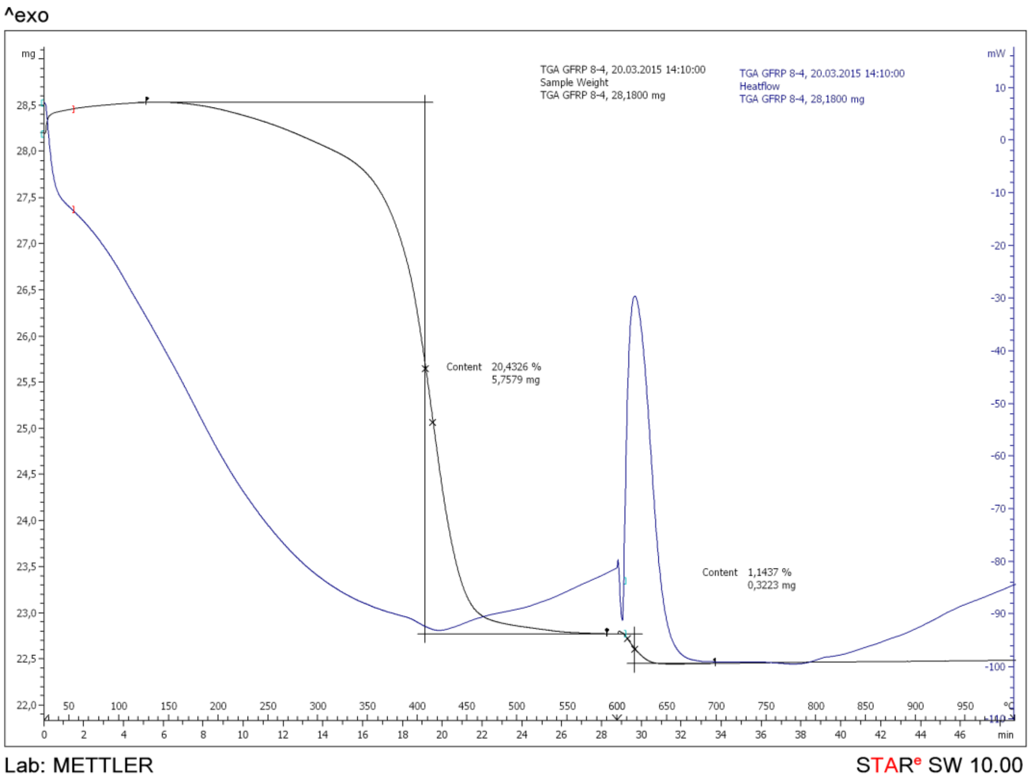


Figure 31. Thermogravimetric curve on filament wound pipe – **GFRP 8 – 4**: weight loss (mg) versus temperature

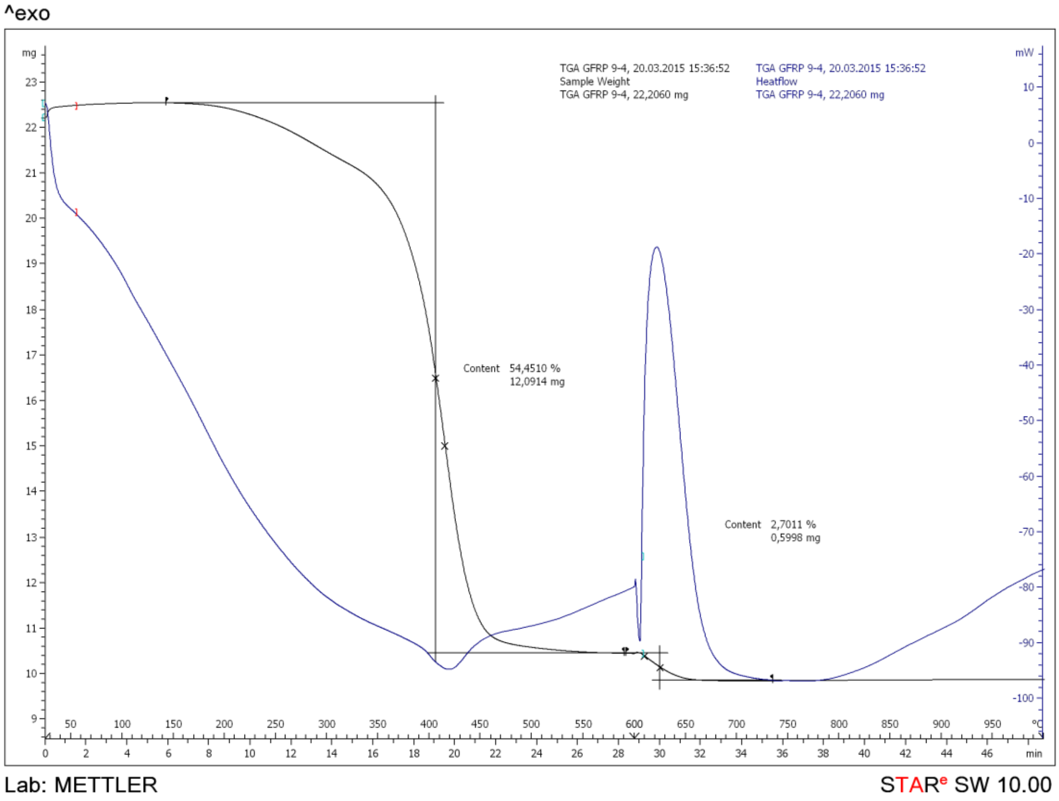


Figure 32. Thermogravimetric curve on filament wound pipe – **GFRP 9 – 4**: weight loss (mg) versus temperature

Table 6. Thermal stability of filament wound pipes based on glass fibers and epoxy resin

Temperature range (°C)	Weight loss (%)				
	GFRP 3-4	**GFRP 4-4**	**GFRP 7-4**	**GFRP 8-4**	**GFRP 9-4**
150 - 600	16,5631	15,6111	20,2401	20,4326	54,4510
600 - 1000	0,8218	0,6400	1,0401	1,1437	2,7011

As it can be observed, thermal degradation of all composites indicate two stages weight loss process. The first stage, occurring in the temperature range from 150°C to 600°C, is correlated to the degradation of the epoxy resin and for all composites is about 20% mass. These data are in accordance with the finds for the thermal stability of the epoxy resin. For the second stage, which is occurring in the temperature range from 600°C to 1000°C, a small shoulder can be noticed which corresponds to the thermal degradation of glass fibers. Since the degradation process occurs in two steps, it can be explained by the degradation phenomena associated with the different composite components. For the sample GFRP 9-4 there is some deviation which can be explained with the possibilities that in the tested sample there is a huge quantity resin and a very small quantity of fibers. So, the weight loss of about 50% is due to the weight loss of the resin.

According to the all obtained results in this investigation for mechanical and thermal characteristics, it can be concluded that from the mechanical point of view there are significant differences in the filament wound pipes with different fiber orientation.

For the tensile properties, the filament wound pipes winded with angle 90^0 had shown much better value than pipes winded with angle 10^0. But, the filament wound pipes winded with angle 45^0 had shown ten times better tensile strength from samples winded with angle 10^0 but less than samples winded with angle 90^0. From received results it can be noticed that tensile properties of composite specimens depended from winding angles in filament winding technology. Namely, the bigger winding angle lead to higher hoop tensile properties of filament-wound tubular samples. Also, it can be noticed that fiber tension and velocity of the filament winding don't influence the tensile strength of the specimens.

For the compression properties, it can be noticed that transverse compression properties of composite specimens depended from winding angles in filament winding technology. Namely, the lower winding angle lead to higher transverse compression properties of filament-wound tubular samples. Also, it can be noticed a slight influence of the fiber tension on the compression strength. That means, the higher fiber tension leads to higher value of the compression strength. The velocity of the filament winding doesn't influence on the compressive strength of the specimens.

From the thermal characterization, it can be concluded that the all filament wound pipes have good thermal stability and their complete weight loss were observed at temperature interval from 600 ^{o}C to 1000 ^{o}C. The values for the glass transition temperature (Tg) of the analyzed composites are similar for all configuration of filament wound pipes which indicate that the degree of crosslinking is already reached in all composites. The values of the heat capacity of the composites prove that a high degree of crosslinking is already reached in all composites.

The glass transition temperature (Tg) in the composites increases with the increasing of the heating rate.

The cross linking reaction between the resin and fibers in the filament wound pipes produced with lower and upper level of velocity of winding result in minor changes on the glass transition temperature. Obviously, the velocity of winding does not influence on the glass transition characteristics.

5. Future collaboration with the host institution

Preliminary conversation were carried out for the future cooperation in the frame of some European projects like EUREKA, NATO Science for Peace programme etc. The future collaboration will be in the direction of the continuing the investigation connected with FRP for construction.

6. Foreseen publications resulting or to result from the STSM

The results obtained during this STSM stay and also with the ones previously obtained will be presented in a form of mutual publications in well-known international journals e.g. Journal of Composite for Construction, Composite Part-B, Construction and Building Materials etc. Also, the results of this stay will be presented at several congresses and conferences all over the world and other national conferences.

7. Other comments

I would like to express my gratitude to the COST Action TU 1207 for the financial support of this STSM and giving me the chance to make the predicted investigation manly connected with WG1 activities.

Also, I am grateful to Dr Renata Kotynia for hosting me at Lodz University of Technology (TUL), Faculty of Civil Engineering, Architecture and Environmental Engineering and for her exceptional engagement in the course of my involving in the mechanical and thermal measurements, interpretation of the results and theoretical background. Also, many thanks to MSc. Marian Jedryka for his technician help in the mechanical measurements during this stay and many thanks to Dr. Magdalena Maciejewska from Chemical Department at Lodz University of Technology for her technician help in the thermal measurements.

8. Confirmation by the host institution of the successful execution of the STSM

I confirm that Dr VINETA SREBRENKOSKA, COST-STSM-TU1207-23020, from Faculty of Technology, University Goce Delchev, Shtip, Macedonia was hosted at Lodz University of Technology (TUL), Faculty of Civil Engineering, Architecture and Environmental Engineering for the period indicated above and that this report is an accurate account of the activities carried out by the STSM grantee.

STSM Grantee name and signature	**Date**
Vineta Srebrenkoska	**10.04.2015**
Host Institution name and signature	**Date**
Lodz University of Technology (TUL), Faculty of Civil Engineering, Architecture and Environmental Engineering, Zeromskiego 116, 90-924 Lodz, Poland Supervisor of the Applicant Associate Prof. Renata Kotynia	**10.04.2015**

http://www.tu1207.eu