Deliverable Report

Deliverable Title:

Report with the properties characterized for the different alloys used at the Trials

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V	Date	Author (Affiliation)	Actions& Approvals
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Summary

This document summarizes the main results obtained with the characterization tests carried out on different alloys, including AlSi10MnMg, AlSi8MnMg, and AlMg3, used in the HPDC trials conducted at Eurecat. The heat treatment optimized in Task 1.5 for WP1 Alloy variants cast in PMC was applied and tuned with the parts produced by HPDC. In addition, the same methodology was applied to optimize the alloy variants developed in WP2.

The mechanical properties were determined in as cast conditions, as well as after different heat treatments.

Disclaimer

Abbreviations

Abbreviation / Acronyms	Description
HPDC	High Pressure Die Casting
YS	Yield Strength
UTS	Ultimate Tensile Strength

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Table of contents

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 101003785

List of tables

List of figures

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1. Introduction and Background

In WP4 of SALEMA project, the pilots regarding HPDC will be implemented and the new HPDC alloys validated. It is a WP devoted to the assessment of the new alloys developed within the project, by manufacturing and characterizing the final properties of two HPDC demonstrators with important mechanical requirements produced in two pilot plants implemented in industrial sites.

In Task 4.2 SALEMA the alloys variants produced in Task 4.1 taking into account the research carried out in WP1 and PW2 are tested and characterized, in order to select those with better performance to be further studied and validated in the industrial demonstrators.

The present deliverable describes the results of the characterization tests carried out on the HPDC parts produced during the casting trials conducted at Eurecat:

- Selection and characterization of components produced by HPDC with a total of 12 different alloy variants: 3 variants of AlSi10MnMg0.2 alloy, 3 variants of AlSi10MnMg0.3 alloy, 4 variants of AlSi8MnMg alloy and 2 variants of AlMg3 alloy.
- Application and tunning of the heat treatment optimized in Task 1.5 in HPDC parts
- Optimization of the heat treatment of AlSi10MnMg alloy variants, by applying the same methodology used in WP1 for AlSi10MnMg alloy variants
- Mechanical and microstructural characterization of HPDC parts with different heat treatments
- Results obtained from corrosion resistance tests conducted on all alloy variants

This task of WP4 will assess the performance of a wide range of alloy variants in order to select those with better performance for final validation under full industrial.

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1.1. Objectives of task and deliverable

The main objective of this deliverable is to assess the performance of the 12 alloy variants produced by Raffmetal and supplied to Eurecat with this purpose, in order to select and have more information for their final application in the industrial demonstrators. The mechanical and corrosion properties of all 12 alloy variants has been determined following the criteria defined at the beginning of the project and collected in Deliverables 1.1 and 2.1, in order to make possible to achieve the demonstrators' requirements.

2. Characterization of AlSi10MnMg alloy variants

2.1. Experimental procedure

The characterization of the HPDC SALEMA alloys started by conducting a first set of trials with a double intention: 1) to determine the process parameters that result in the highest quality part and 2) to assess the process stability and the sensitivity of the alloy to changes in casting parameters.

In this first set of trials, several parameters were assessed, including melt temperature, melt treatment, 1st phase, 2nd phase, speed change position, and break position. The melt temperature refers to the temperature of the furnace, while the 1st and 2nd phases refer to the speed of the piston during two different instances of filling. The speed change position is the place where transition from the 1st phase to the 2nd phase occurs, while the break position refers to the position at which the piston brakes.

These parameters were tested in various combinations, and the resulting parts were evaluated through visual inspection on a 2-5 scale. For each combination, ten parts were casted and analyzed. Some of the parts with better aspect were selected for be subjected to mechanical characterization. Two tensile tests specimens were extracted from each of those parts and tested in an universal testing machine. Detailed information about the procedure followed can be found in Deliverable 4.4.

The study of different parameter combinations resulted in the identification of optimal values that formed the basis for all subsequent high-pressure die casting trials with different variants of AlSi10MnMg. The values selected for the production of the plates used for further characterization of AlSi10mnmg alloy variants were: temperature - 720ºC; 1st phase speed – 0,4 m/s; 2nd phase speed – 1,8 m/s; speed change position 295 mm; break position – 410 mm. Figure 1 illustrates the usage of these parameters during a cycle of the HPDC process.

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Figure 1 - Injection curve parameters of Buhler software

For each variant of the alloy AlSi10MnMg the target number of produced parts defined was 150. Ten additional parts were produced on each test, at the beginning to stabilize the die temperature and some related parameters such as clamping force. These ten extra parts were then sent to scrap. The furnace temperature was controlled every ten parts to make adjustments, if needed. The melt preparation included the addition of fluxes to promote the slag cleaning effect and 20 minutes of nitrogen degassing through graphite porous lance.

2.2. Production of the AlSi10MnMg0.3 and AlSi10MnMg0.2 alloy variants

To produce the AlSi10MnMg0.3 and AlSi10MnMg0.2 alloy, three different chemical compositions were utilized for each, denoted as Variant 1, 2, and 3 and Variant 4, 5 and 6, respectively. Their chemical composition is presented in Table 1, please refer to Deliverable 4.3.

Table 1 - Chemical composition of the 6 AlSi10MnMg variants selected for further development within HPDC process

AlSi10MnMg0.3	***Si***	***Fe***	***Cu***	***Mn***	***Mg***	***Cr***	***Ni***	***Zn***	***Pb***	***Sn***	***Ti***
Variant 1	9-11,5	<0,2	<0,03	0,45-0,65	0,25-0,35	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 2	9-11,5	<0,2	0,05-0,1	0,45-0,65	0,25-0,35	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 3	9-11,5	<0,2	0,05-0,1	0,45-0,65	0,25-0,35	<0,03	<0,03	0,1-0,15	<0,03	<0,03	0,05-0,15
AlSi10MnMg0.2											
Variant 4	9-11,5	<0,2	<0,03	0,45-0,65	0,15-0,25	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 5	9-11,5	0,2-0,3	<0,03	0,45-0,65	0,15-0,25	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 6	9-11,5	<0,2	0,05-0,1	0,6-0,8	0,15-0,25	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15

A melt treatment consisting in a coupled treatment of cleaning salts and nitrogen bubbling through a graphite porous lance was applied to each of the variants before starting the casting of the parts. In order to assess the quality of the melt a reduced pressure test was applied. The results of the density index for each variant are presented in Table 2. Overall, the results are considered particularly good. For more detailed information on reduced pressure tests and how to calculate the density index, refer to Deliverable 3.5.

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Table 2 - Density index results

Alloy	Density index (%)
Variant 1	2,00
Variant 2	3,51
Variant 3	2,62
Variant 4	3,30
Variant 5	3,30
Variant 6	1,63

Overall, the trials were successful for each variant. However, some process-related stoppages occurred due to occasional die sticking and part extraction failure. These stoppages resulted in a decrease in die temperature, leading to lower quality parts. Nonetheless, such events occurred sporadically and only impacted one or two consecutive parts at a time. However, such stoppages will be visible in the statistical analysis of part quality for each variant. This is because, in general, when the process runs without any issues, the parts have a high level of quality (grade 5).

2.3. Inventory of the plates produced

The quality report for each variant is presented in Table 3. Most of the produced parts exhibit the highest level of quality, followed by a small number of parts with minor surface defects, and a very small number of parts with low quality. A quality value between 1 and 5 were given to all produced parts, following the criteria described in Deliverable 4.4.

Table 3 - Quality report

Variant	1	2	3	4	5	6
Count (5)	124	132	87	147	133	135
Count (4)	25	16	22	2	8	15
Count (2-3)	1	2	0	1	1	0

2.4. Heat treatment optimization

Different heat treatments can be applied to Al castings to increase their mechanical properties through precipitation hardening, such as T4, T5, T6 or T7. Among these, the most commonly used for HPDC components of AlSi10MnMg alloys are T5 and T6. Figure 2 shows the mechanical properties that can be expected from the different heat treatments for AlSi10MnMg alloys, in order to meet desirable mechanical properties of demonstrator (shock tower and frontal frame) T6 heat treatment of AlSiMnMg alloys include solutioning of as cast component at higher temperature (~ 470-520 °C) for several hours to dissolve β-Mg2Si phase into Al matrix, which later forms to form fine nanoscale β-Mg2Si precipitates during ageing process which are highly coherent in α-Al matrix and ultimately provide higher precipitation strengthening. However considering the peculiarities of HPDC process, when thin and complex shape are produced, a solid solution treatment at higher temperature for longer period of time is usually avoided for die cast parts to minimize the risk of part distortion and surface blistering caused by the air entrapment in the castings and a T5 (direct artificial ageing) heat treatment could be applied alternatively. Therefore, the selection of solution time and temperature plays critical role. These heat treatment solutions can lead to different amount, size, distribution of the reinforcement phase Mg2Si, which practically means that different combinations of elongation, YS and UTS can be achieved by the treated alloy.

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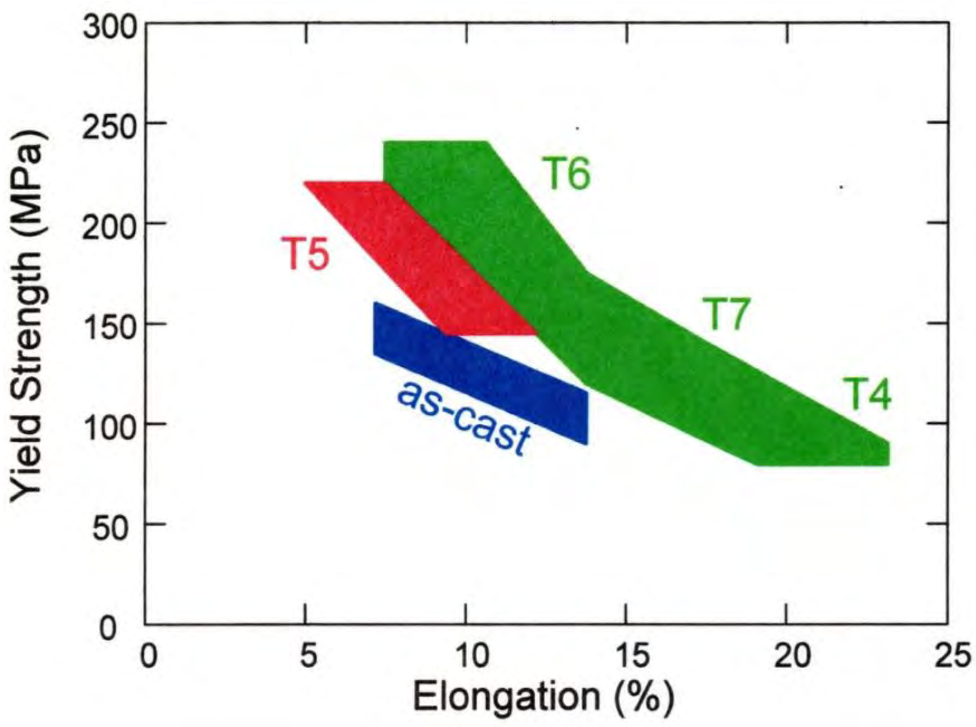

Figure 2 - Range of properties available in structural die castings, as a function of heat treatment

The heat treatment optimization conducted in Deliverable 1.5 for AlSi10MnMg alloy was revised with actual HPDC components. Variant 6 (Table 1) was selected for T5 and T6 heat treatment optimization experimental campaign at different times and temperatures to check for its effect on mechanical properties. Figure 3 shows the procedure followed for the heat treatment optimization.

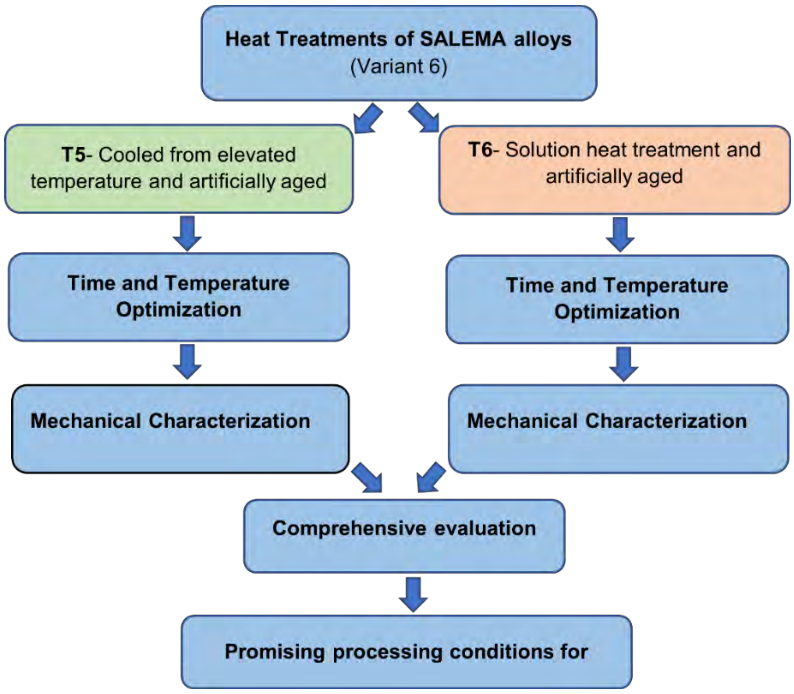

Figure 3 - Organization of work for heat treatment optimization of AlSi10MnMg alloys

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To check for strengthening effect in selected alloy, firstly it was determined the material hardness as function of time and temperature at different conditions, obtaining the hardness curves. To do this, samples were extracted from casted component. Figure 4 shows the sample selection procedure for determining peak ageing hardness.

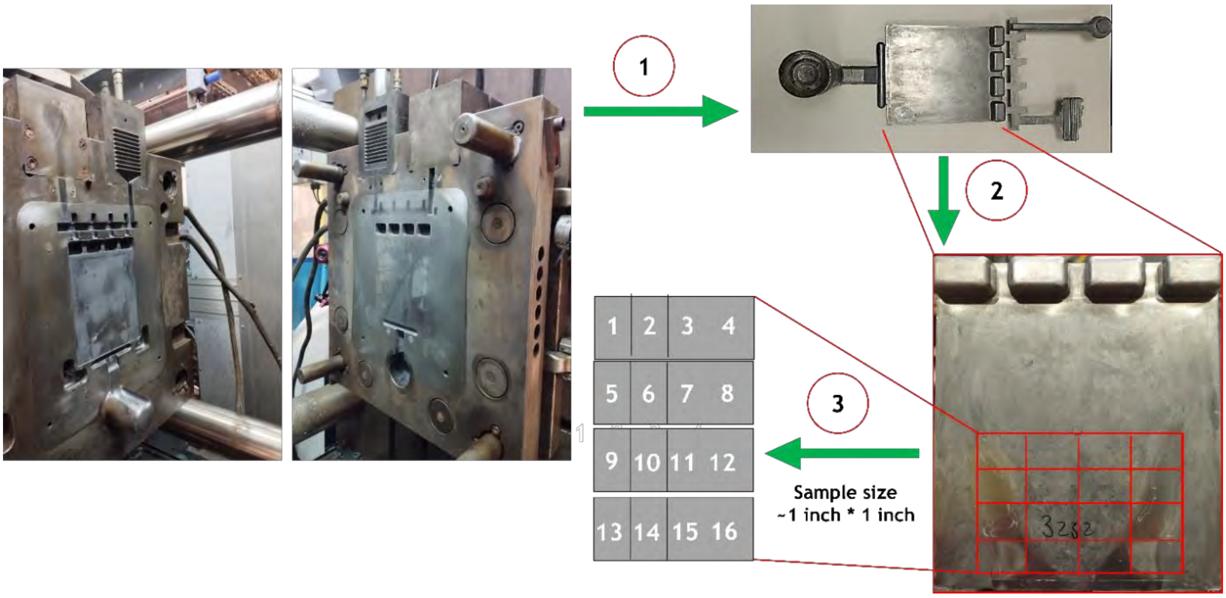

Figure 4 - Sample extraction procedure for hardness evaluation

2.4.1. Investigated T5 conditions

T5 heat treatment corresponds to an artificial ageing conducted in furnace at moderate temperature followed by air cooling. Table 4 shows investigated T5 conditions at various temperatures and ageing times. Multiple hardness reading were measured for each condition. The average value is reported in Table 5 and the hardness evolution with time for the different ageing temperatures is plotted in Figure 5.

Table 4 - Investigated T5 heat treatment conditions for peak hardness

Time	Ageing Temperature			
	190°C	210°C	230°C	245°C
0.25				*
0.5	*	*	*	*
0.75		*	*	*
1	*	*	*	*
2	*	*	*	
4	*	*	*	
8	*			

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Table 5 - Green highlighted values shows peak ageing hardness for T5 conditions

Time	Ageing Temperature			
	190°C	210°C	230°C	245°C
0.25	-	-	-	87.73
0.5	83.73	89.57	95.73	93.87
0.75	-	95.57	91.23	89.53
1	87.33	91.73	87.97	84.37
2	91.43	85.20	87.23	-
4	91.97	87.47	87.13	-
8	88.03	-	-	-

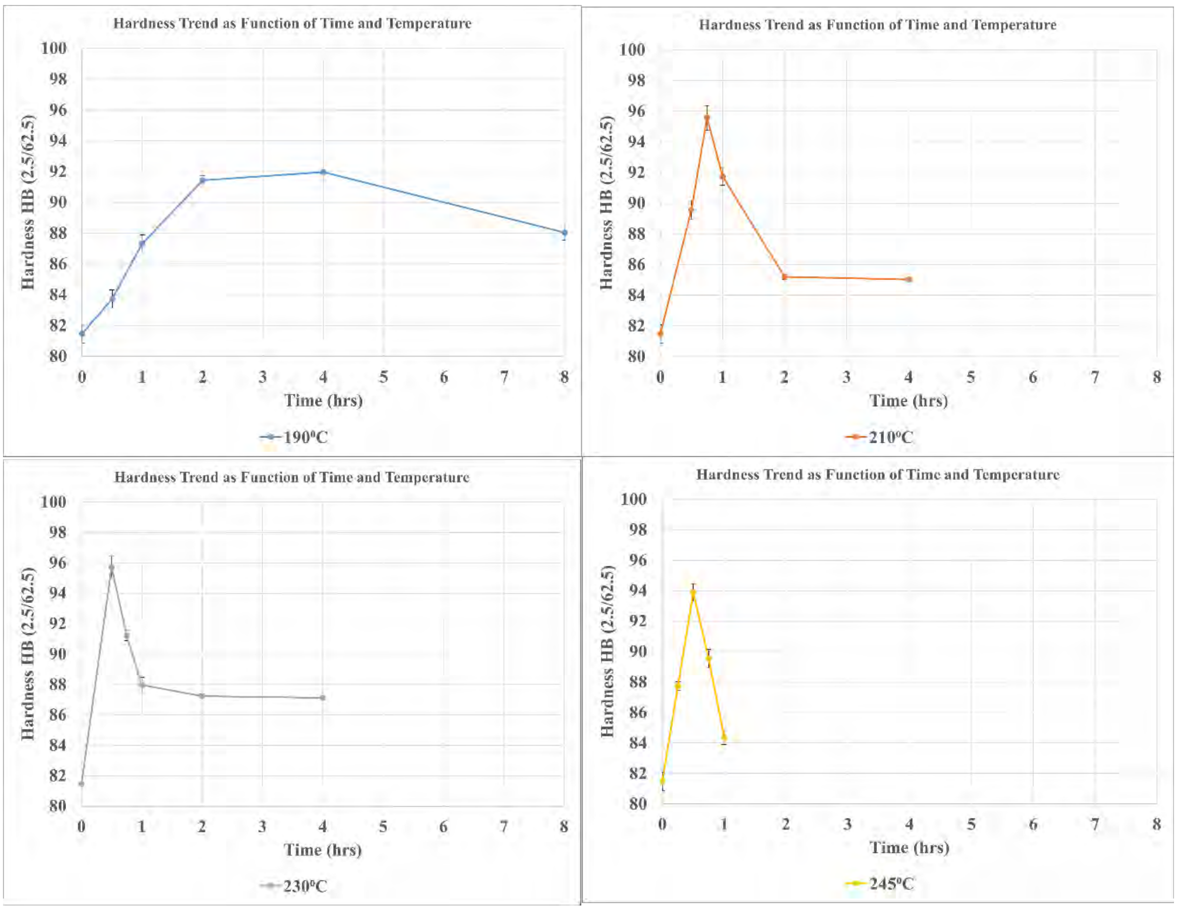

Figure 5 - Hardness trends as function of time and temperature for investigated T5 conditions

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2.4.2. Investigated T6 heat treatment

T6 heat treatment is composed by a solution treatment of the as cast component at high temperature for a given time and then rapidly quenched in quenching medium. After quenching an artificial ageing (as in T5) follows. The final colling down normally takes place naturally in open atmosphere. Among quenching medium, water quenching is the most readily available and most common used for wrought and cast aluminum alloys. Typically, quenching aluminum in water is conducted at either room or elevated temperatures (20-80°C). Water quenching has many advantages, including being readily available and inexpensive. Quenching into water at < 50-60°C often produces non-uniform quenching. This non-uniformity manifests itself as spotty hardness, distortion, and cracking. This non-uniformity is caused by relatively unstable vapor blanket formation. Therefore, followed by solution treatment specimens were quenched in 60°C water to prevent distortions caused by thermal shock.

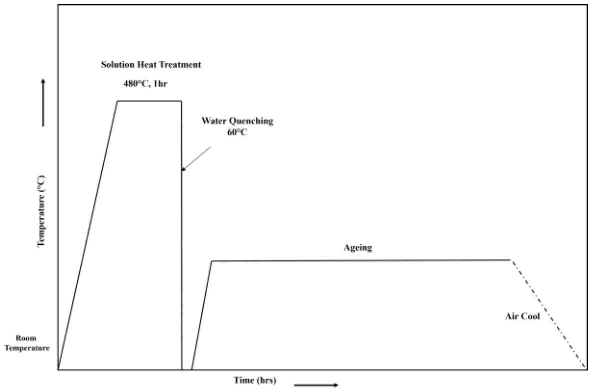

Figure 6 - T6 heat treatment cycle for Variant 6

After solution treating, sample was analyzed for surface blister and to check whether good spheroidization of Al-Si eutectic silicon was attained. Figure 7 shows a photography of one specimen after solution treatment. Very rare and small size of surface blisters were observed along with negligible distortion. Figure 8 shows optical microscopy of specimen in as cast and after solution treatment. It can be clearly observed that spheroidization of Al-Si was found enough and hence 1 hour of solutioning time at 480°C was chosen as solution treatment.

Figure 7 - Surface blisters after solution treatment

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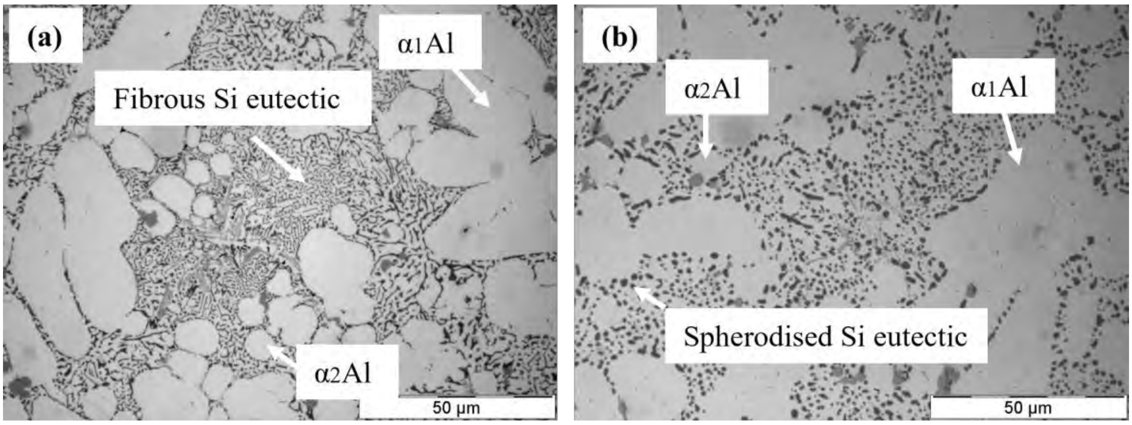

Figure 8 - (a) As cast microstructure consist of fine and fibrous Si eutectic phase (b) after solution treatment, fine fibrous eutectic Si transform into spheroidal Si

Followed by solution treatment, specimen were subjected to ageing treatment at different conditions to check for the peak ageing hardness. Table 6 present all investigated T6 heat treatment conditions. In Table 7 are presented the average values of the hardness measurements obtained for each heat treatment condition and in Figure 9 is represented the hardness evolution with ageing time for each ageing temperature.

Table 6 - Investigated T6 heat treatment conditions for peak hardness

Solubilization Temperature (°C)	Solubilization Time (h)	Quenching Medium	Ageing Temperature (°C)	Ageing Time (h)
480	1	Water (60°C)	190	0.5
				1
				2
				4
				6
			210	0.5
				1
				2
				4
				6
			230	0.5
				0.75
				1
				2
				4

Table 7 - Green highlighted values shows peak ageing hardness for T6 conditions

Solution treatment (480°C for1hr)			
	Ageing Temperature		
Time	190°C	210°C	230°C
0 (As Quenched)	65.7	65.7	65.7
0.5	68.87	72.13	72.5
0.75	-	-	74.2
1	71.37	77.53	73.5
2	76.77	75.3	70.23
4	87.37	75.27	68.13
6	85	74.27	-

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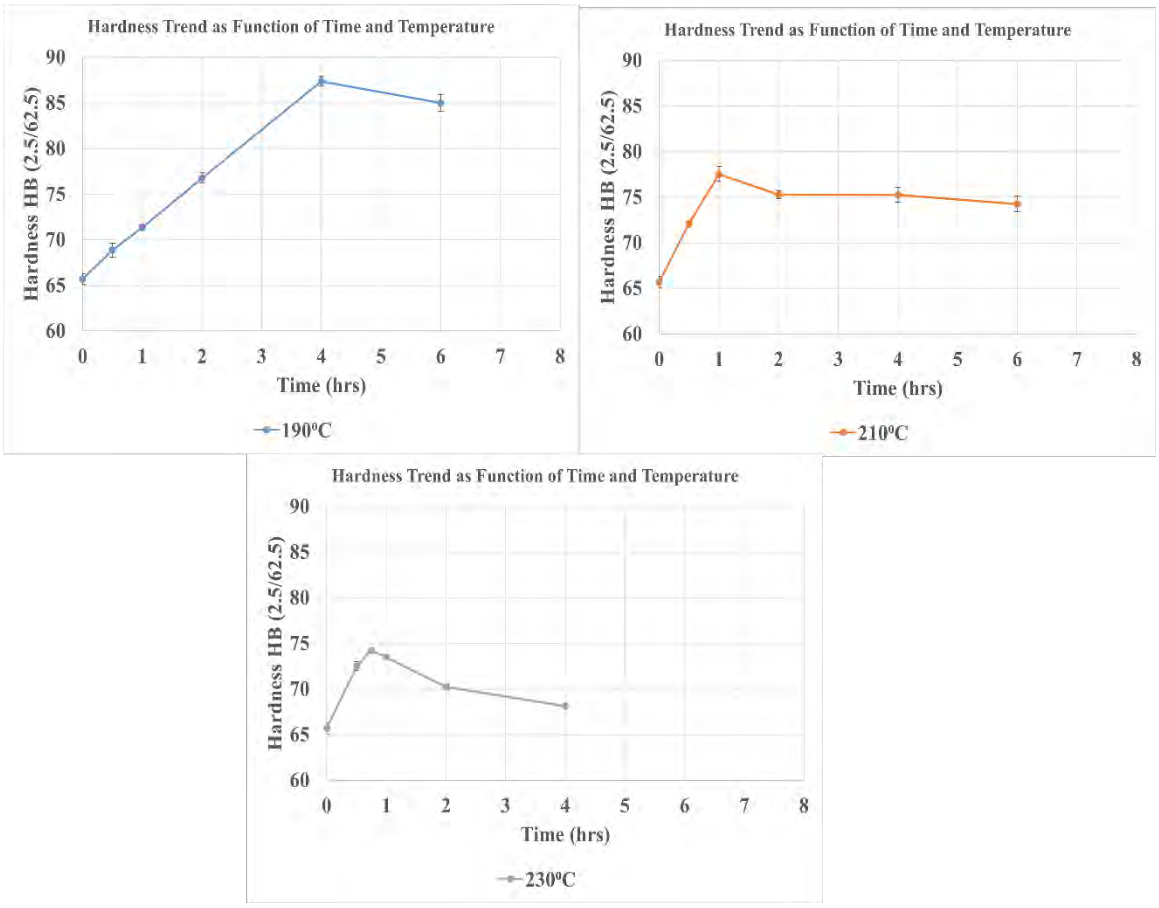

Figure 9 - Hardness trends as function of time and temperature for investigated T6 conditions

Table 8 - T6/7 number of parts for each variant and posterior mechanical testing

Heat treated (T6-T7) 1 h at 490ºC + 1 h at 230ºC						
Variant	**1**	**2**	**3**	**4**	**5**	**6**
CRF corrosion	15	15	15	15	15	15
CRF welding	10	10	10	10	10	10
Tensile tests + micro	4	4	4	4	4	4
Tensile tests 90º	-	4	-	-	4	-
Fatigue	21	-	21	-	-	21
TEF	10	-	10	-	-	10
Bake Paint	4	-	-	-	-	4
FORD 3-Point bending	5	5	5	5	5	5
FORD Riveting	8	-	8	-	-	8
FORD tensile at different strain rates	10	10	-	10	10	10
TOTAL Heat treatment (T6)	105	58	88	53	58	105

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In addition to the optimal full T6-T7 heat treatment, parts of variant 2, 4 and 5 were subjected to other T6-T7 heat treatments in addition to T4 and T5 alternative heat treatments, in order to screen the mechanical properties that could by obtained with other thermal conditions. All the conditions tested are enumerated in Deliverable 4.4.

2.5. Mechanical characterization

Tensile test specimens were extracted from parts with and without the T6-T7 heat treatment and were subjected to tensile test according to A2 section from UNE-EN ISO 6892-1. A total of 6 specimens, 2 specimens from at least 3 different components, were tested. The average results obtained for yield strength (Y.S.), ultimate tensile strength (U.T.S) and elongation for the components tested with and without heat treatment are presented in Figure 20.

It can be observed that the heat treatment is not very effective with variants with low Mg content (4, 5 and 6) and yield strength and UTS remains almost in the same level after the heat treatment. The ductility reached in both thermal conditions (as-cast and T6) is clearly below the demonstrator requirements of 8%. The gain obtained with the T6 heat treatment is smaller than expected. And, in general, the alloy variants with lower Mg level present higher ductility.

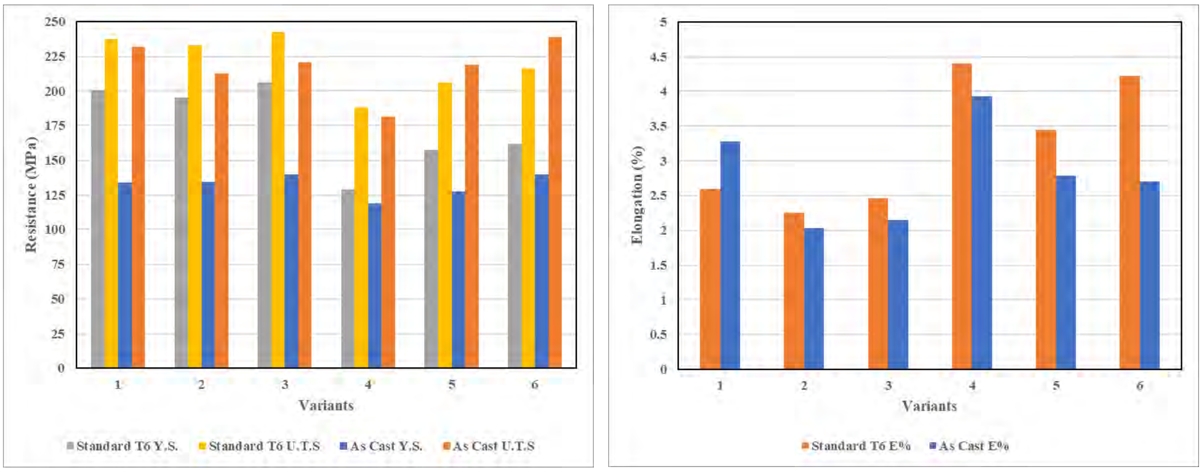

Figure 10 – Tensile tests results of the T6-T7 heat treated components from the 6 alloy variants investigated

For some of the alloy variants (2, 4 and 5) an analysis of different T5 heat treatments were conducted. The evolution of the mechanical properties for the different ageing conditions for each of the variants investigated are presented in Figure 11.

The results of the tensile tests shows that all the T5 conditions lead to an increment in both, yield strength and UTS, with a consequent decrement in ductility. The values of YS and UTS are pretty similar for all T5 conditions, however, 2h at 210ºC is associated with a larger impact on ductility loss for all 3 variants investigated. Therefore, this T5 condition, seems to be the less convenient of the 3 combinations investigated to be used in the final demonstrators.

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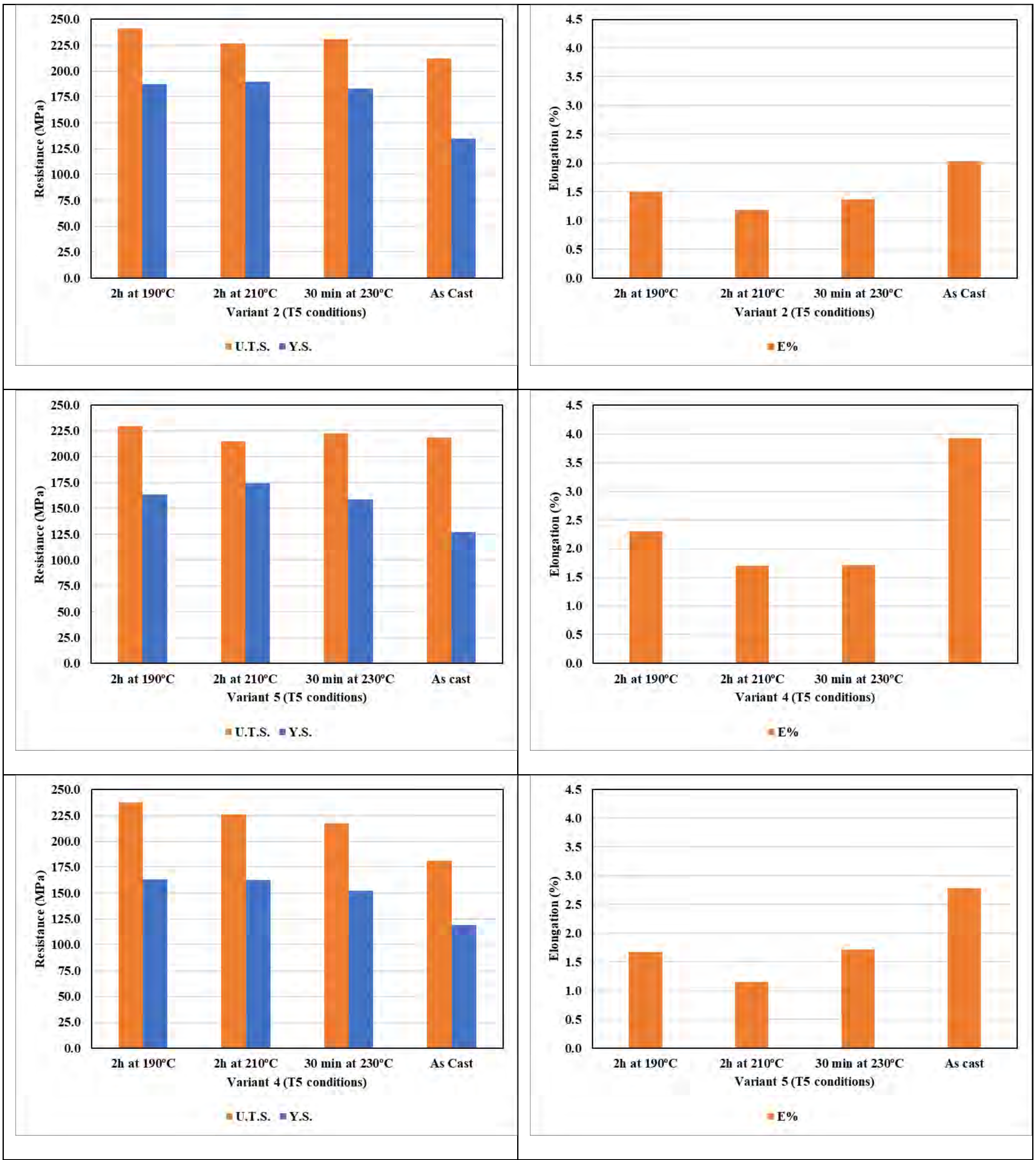

Figure 11 – Tensile tests results of T5 heat treatment for different ageing conditions for variant 2 (top charts), variant 4 (middle charts) and variant 5 (bottom charts)

2.6. X-ray and soundness analysis of parts

A total of 4 specimens from variants 1 and 6 were selected for an X-ray and destructive inspection in order to determine the soundness of the parts and the amount and type of defects present on them.

Radiographic inspection was performed according to EN ISO 12681 standard, with the aim of detecting the presence of defects (air entrapment, shrinkage cavities) and defining the "quality mapping" for each casting.

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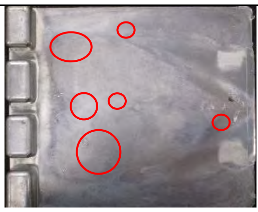
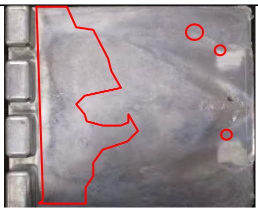
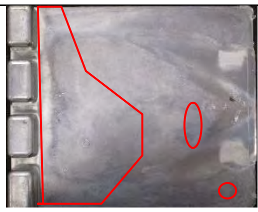
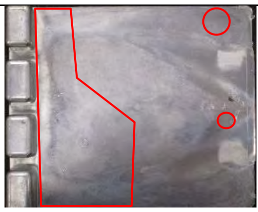
Results achieved are summarized in Table 9Table 12, in which the critical (in terms of defects concentration) areas are identified by red borders. The red, white or green bars on the side of photos identify, respectively, critical, average or good castings in view of further mechanical testing.

Table 9 – Images of the parts depicting the zones with presence of defects detected with X-ray

Variant #1 as Cast				
	1417 – low amount of porosity, with small size	1435 – medium amount of porosity, clustered in various regions of the casting	1482 – low amount of porosity, with small size	1521 – low amount of porosity, with small size
Variant #1 T7				
	1470 – high amount of porosity, with banded distribution	1483 – medium amount of porosity, in some cases clustered, in most cases dispersed	1484 – medium amount of porosity, mainly clustered	1507 – medium amount of porosity, in some cases clustered, in most cases dispersed
Variant #6 as Cast				
	1560 – low amount of single porosity, with small size	1576 – low amount of single porosity, with small size	1577 – low amount of single porosity, with small size	1578 – low amount of single porosity, with small size
Variant #6 T7				
	1610 – medium amount of porosity, in some cases clustered, in most cases dispersed	1644 – medium amount of porosity, in some cases clustered, in most cases dispersed	1650 – medium amount of porosity, in some cases clustered, in most cases dispersed	1655 – high amount of porosity, in some cases clustered, in most cases dispersed

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The results of radiographic inspections allowed the selection of areas from which cutting specimens for tensile testing. These specimens were taken both vertically and horizontally from castings (Figure 12 and Figure 13). The results of the tensile tests conducted on the extracted specimens are presented in Table 10.

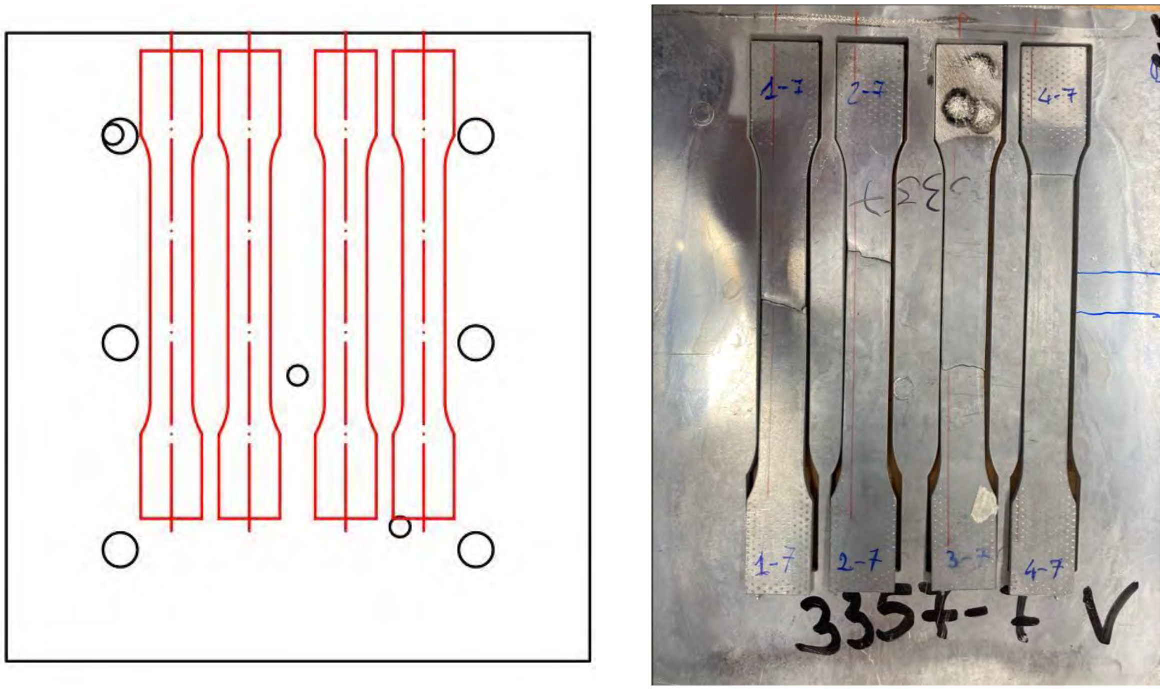

Figure 12 – Examples of specimens taken vertically from the castings

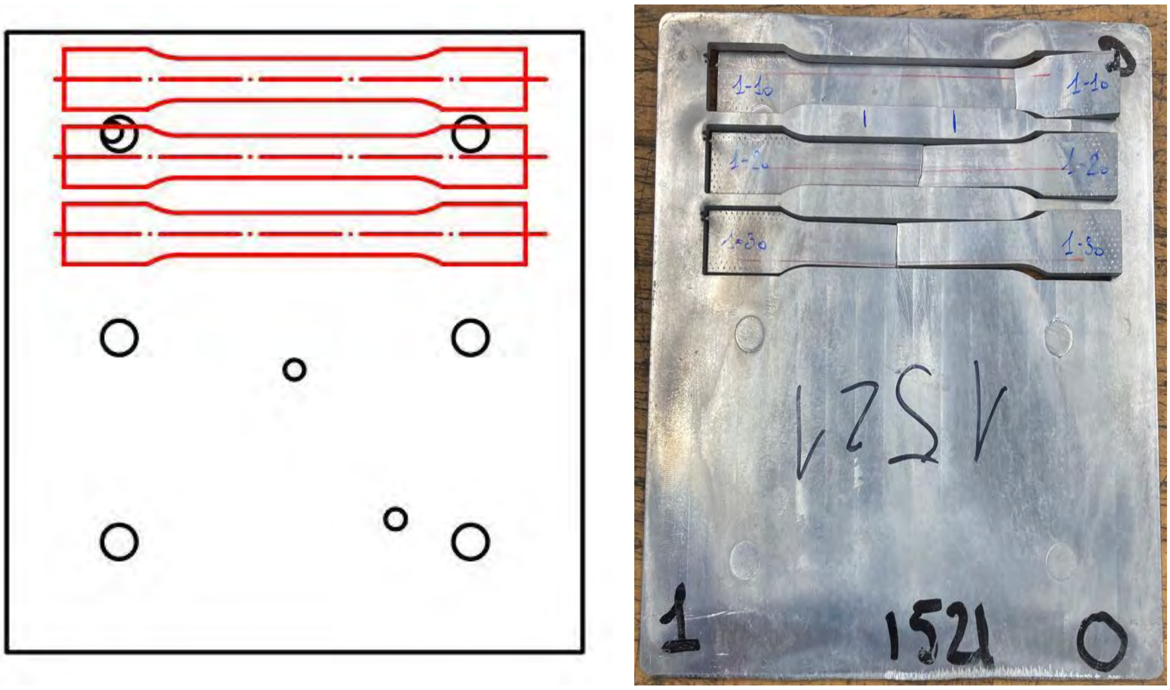

Figure 13 – Examples of specimens taken horizontally from the castings

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Table 10 – Tensile properties obtained for different variants, orientations and thermal conditions

Variant	YS (MPa)	UTS (MPa)	Elongation (%)
#1, as cast, vertical	135 ± 4.0	239 ± 21.5	2,90 ± 1.25
#1, as cast, horizontal	138 ± 2.5	224 ± 18.6	2,02 ± 0.70
#1, T7, vertical	200 ± 2.5	242 ± 4.2	2,05 ± 0.50
#1, T7, horizontal	197 ± 1.0	211 ± 11.1	0,66 ± 0.40
#6, as cast, vertical	139 ± 2.9	213 ± 25.5	1,90 ± 0.10
#6, as cast, horizontal	137 ± 1.5	214 ± 15.4	1,57 ± 0.50
#6, T7, horizontal	164 ± 2.0	201 ± 10.5	1,63 ± 0.90

Some variability have been observed in mechanical behavior, mainly due to some defects, as shown in Figure 14 (fracture surfaces for variant #1, with relevant presence of inclusions) and in Figure 15 (defects detected by metallographic examinations).

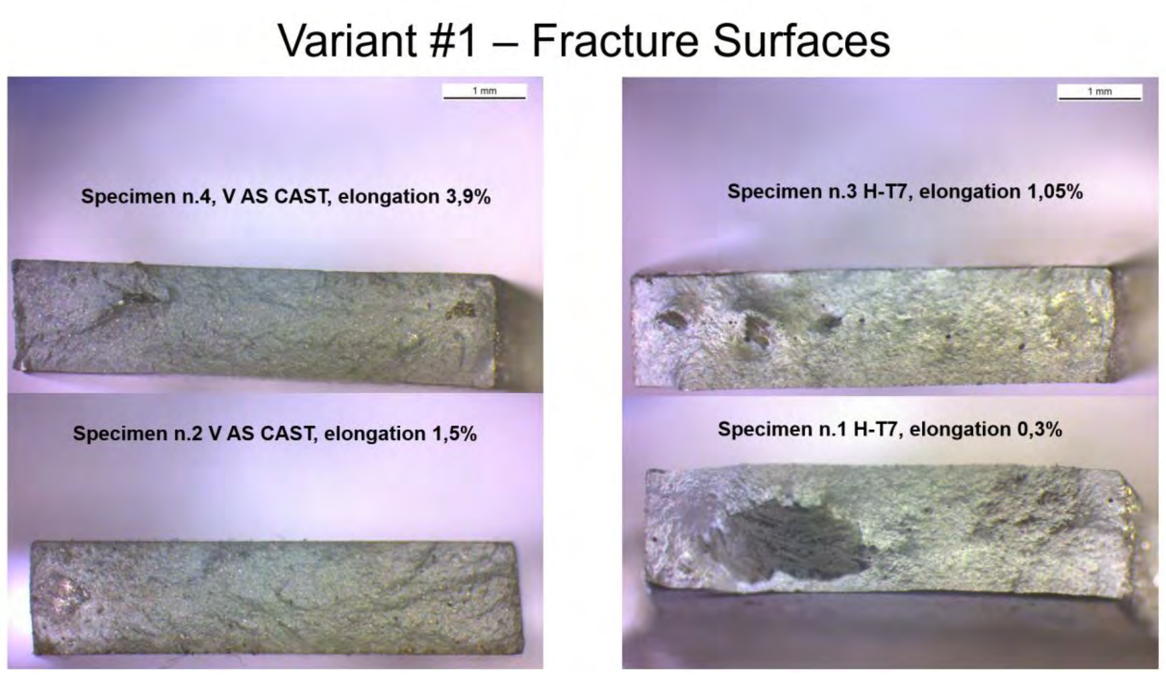

Figure 14 – Fractographic examination of variant 1 specimens

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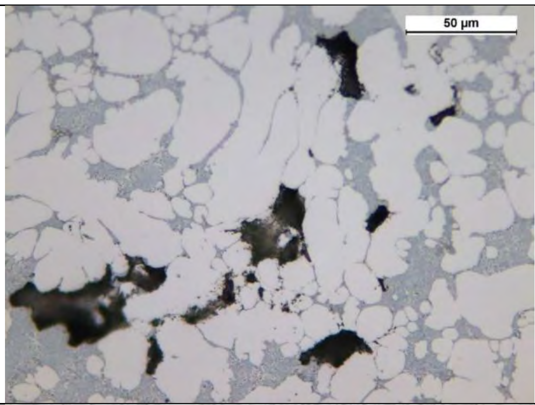
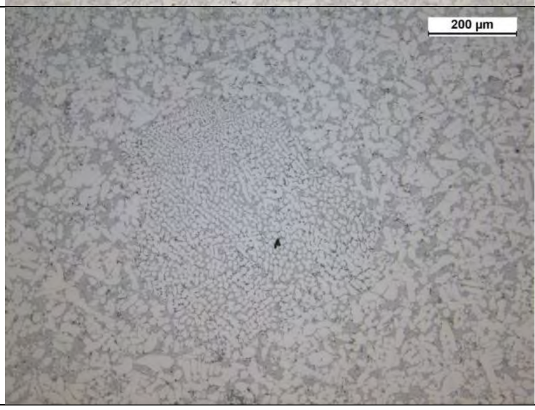
Porosity	
Cold joint	
Cold flakes	

Figure 15 – Examples of defects detected by metallography

2.7. Corrosion resistance

2.7.1. Methodology

For the evaluation of the corrosion behaviour of the SALEMA alloys and the possible differences between the variants, a corrosion test has been performed: ASTM B368. The test was performed on all the six AlSi10MnMg variants under examination on the as-cast and the T6 conditions.

ASTM B368 was performed on the specimens that were previously subjected to cataphoresis (thickness 12 µm ±2, baking condition 20 min 175°C). After the superficial treatment, a scratch was done on the protective layer for exposing the aluminium substrate to the corrosion test. Then the specimens were placed in the corrosion chamber for the Copper-Accelerated Acetic Acid-Salt Spray (Fog) testing (CASS test). The duration of the CASS test is 144h. At the end of the test, a standard tape is applied on the scratch and pulled for assessing the width of the removed material. This test is done for the evaluation of the material behaviour under the cataphoresis layer.

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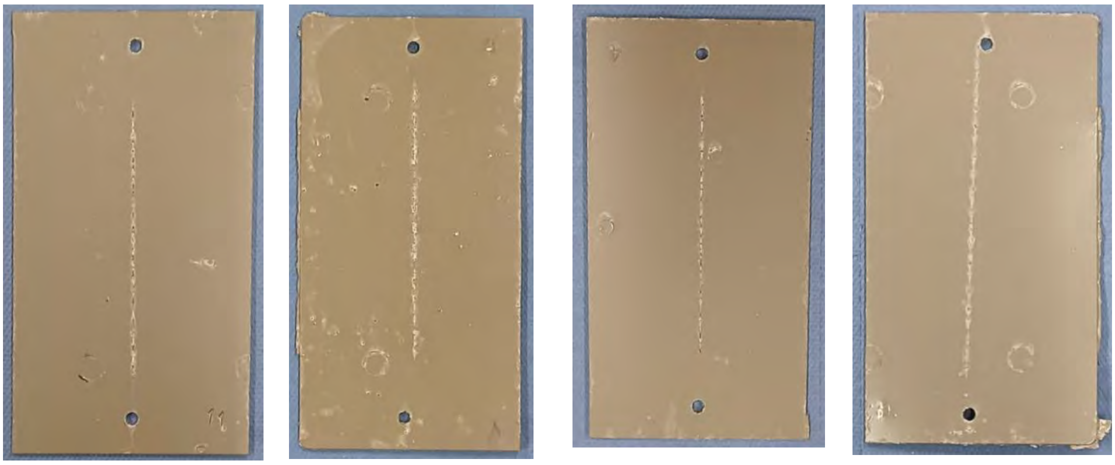
Test parameters are summarized in Table 11.

Table 11 - Main parameters used in the corrosion tests

Test	Corrosion	
Dimension	100x200 mm	
Standard	ASTM G85 A3	ASTM B368
Surface state	No coating	Cataphoresis

2.7.2. Results

The specimens (Figure 16) assessed following the ASTM B368 standard showed similar corrosion behaviour and no differences before and after the heat treatment. Regardless to the variant and the heat treatment, all the specimens were compliant to the requirements listed in the standard. In particular, the groove width left after pulling away the tape was minor than 1,5 mm and so they passed the test.



As Cast T6 As Cast T6

Variant 2 (AlSi10MnMg0.3) Variant 4 (AlSi10MnMg0.2)

Figure 16 - Corrosion results test ASTM B368

2.8. Alternative tests with NADIA reference die

The mechanical properties obtained with the plates cast at Eurecat were confronted with values from tensile specimens directly casted by HPDC with the final shape. These specimens were obtained with a die property of Univ. of Padova and design and constructed in another European project (NADIA) at the facilities of one of the NADIA project partners (SAEN).

The confrontation was done only for Variant 6. The casting parameters used were defined by SAEN, and fixed according to their previous experience with this die for AlSi10MnMg alloy. In Figure 17 is presented a picture of some of the parts produced during the casting trial.

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Figure 17 – Parts cast with NADIA die Hardness trends as function of time and temperature for investigated T6 conditions

The castings have 2 flat and 1 round tensile tests specimen. One of the flat and the round specimens were tested for each casting conditions. The chemical composition was measured in the first and the last of the shots taken for this investigation and are presented in Table 12. It can be observed that, with the exception of Mn, which is just slightly below the defined limits, all the elements are within the designated composition range.

Table 12 – Chemical composition measured in the tensile test specimens and Variant 6 composition range

	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Pb	Sr	Ti
Part 23	9,53	0,28	0,057	0,586	0,215	0,023	0,007	0,03	0,01	0,013	0,086
Part 31	9,89	0,292	0,06	0,596	0,227	0,022	0,007	0,03	0,01	0,013	0,084
Variant 6	9-11,5	0,2-0,3	0,05-0,1	0,6-0,8	0,15-0,25	0-0,03	0-0,03	0-0,07	0-0,03	0,01-0,02	0,05-0,15

The results obtained in the tensile tests for the flats specimens are presented in Table 13 and for the round specimens in Table 14. The mechanical properties obtained with both type of specimens are similar, with the exception of elongation, which is significantly higher for the round specimens. Nevertheless, in both cases, the values of elongation obtained are much better than for the flat plates produced at Eurecat.

Table 13 – Values obtained with the flat specimens tested

Part num.	*Widness mm*	*Thickness mm*	*L_0 mm*	*E GPa*	*$R_{p0.2}$ MPa*	*R_m MPa*	*A_t %*
23	9,9	3	32	77	133	280	5,6
26	9,9	3	32	72	137	284	5,8
27	9,9	3	32	73	136	285	6,6
28	9,9	3	32	73	135	284	6,5
31	9,9	3	32	76	134	286	6,9
			Average	74,2	135	283,8	6,3
			Stad. Dev.	2,2	1,6	2,3	0,6

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Table 14 – Values obtained with the round specimens tested

Part num.	Widness mm	Thickness mm	L_0 mm	E GPa	$R_{p0.2}$ MPa	R_m MPa
23	9.9	32	73	135	287	8.6
26	9.9	32	74	135	289	8.9
27	9.9	32	72	135	286	8.7
28	9.9	32	69	135	286	8.4
31	9.9	32	75	133	280	7.2
		Average	72.6	134.6	285.6	8.4
		Stad. Dev.	2.3	0.9	3.4	0.7

3. Characterization of AlSi8MnMg alloy variants

3.1. Characterization of casting plates produced at Eurecat

As well as for AlSi10MnMg alloy variants, before producing the plates to be used for the exhaustive characterization of the different alloy properties, a preliminary test was carried out to define the best casting parameters for this new alloy family.

Table 15 – Chemical composition of the 4 AlSi8MnMg variants selected for further development within HPDC process

AlSi8MnMg0.3	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Pb	Sn	Ti
Variant 7	7,5-8	<0,2	<0,03	0,6-0,7	0,15-0,25	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 8	7,5-8	<0,2	<0,03	0,6-0,7	0,25-0,35	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 9	8,5-9	<0,2	0,2-0,3	0,6-0,7	0,15-0,25	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15
Variant 10	8,5-9	<0,2	0,2-0,3	0,6-0,7	0,25-0,35	<0,03	<0,03	<0,07	<0,03	<0,03	0,05-0,15

As well as for AlSi10MnMg alloy a screening with pseudo-optimum casting parameters was performed, in order to adapt the optimal casting parameters to the new alloy family. The lower amount of Si present in AlSi8MgMn alloys than AlSi10MnMg alloys decreases alloy flowability and, therefore, optimal casting parameters could slightly differ.

The casting parameters used for AlSi10MnMg alloys were used as reference and a lower and upper value was also tested (Table 16). 10 parts were cast for each defined combination of casting parameters and the cast were visually inspected, giving a value from 1 to 5 according to their aspect.

Table 16 – Level of the different casting parameters investigated for AlSi8MnMg alloy variants

Casting parameter	Melt temp	1st phase speed	2nd phase speed	Vel. Change point	Break position
Lower value		0,35 m/s	1,5 m/s	290 mm	400 mm
Ref. value	720ºC	0,40 m/s	1,8 m/s	295 mm	410 mm
Upper value	740ºC	0,45 m/s	2,1 m/s	300 mm	420 mm

The values of visual quality given to each single part were used to determine the combination of casting parameters more suitable for obtaining good quality castings. In Figure 18 are presented charts showing the evolution of the casting quality with the different process parameters.

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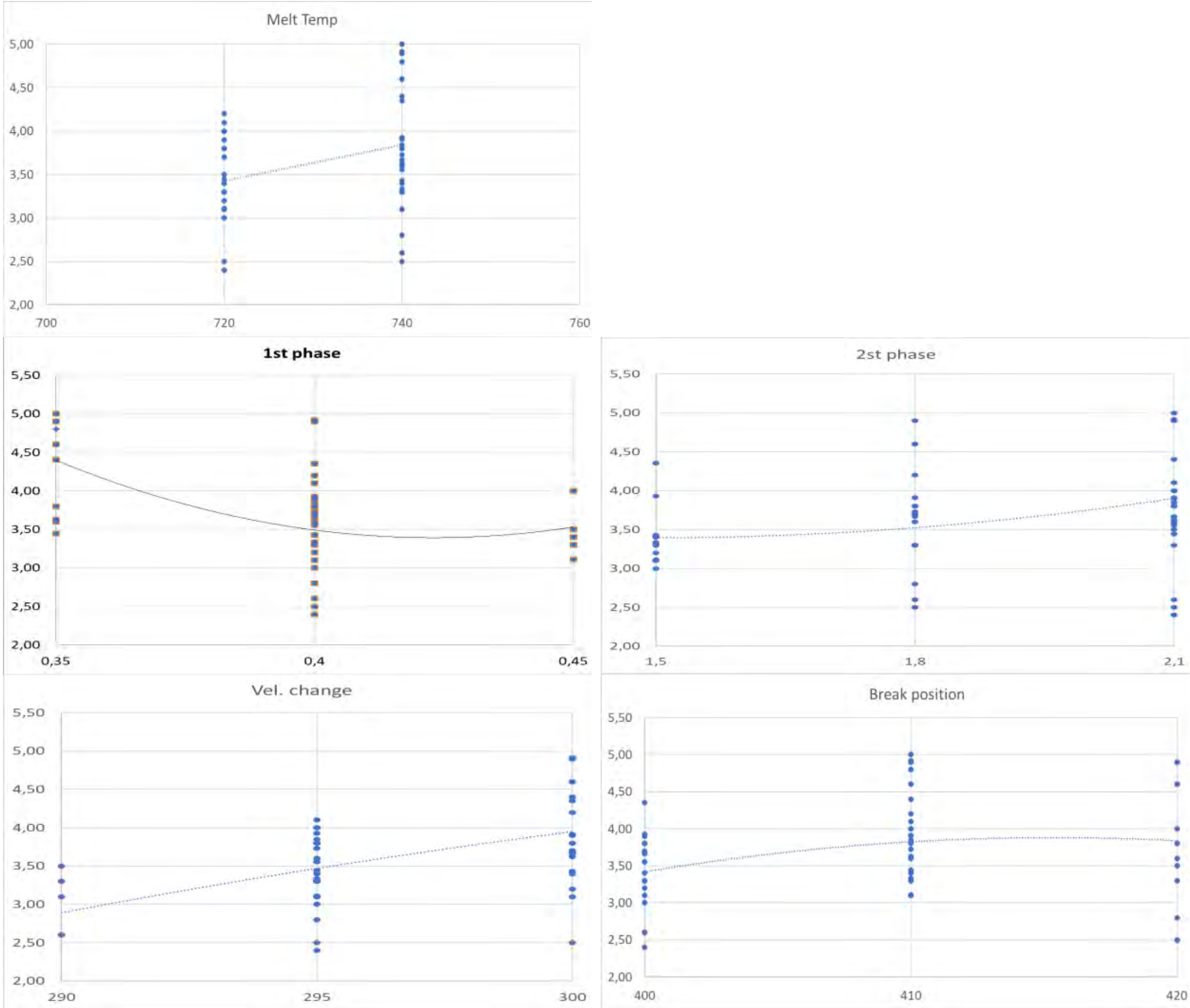

Figure 18 – Evolution of the visual quality of the parts with the different level of process parameters

It was observed that, with the general quality of the parts improved when the melting temperature was increased from 720ºC to 740ºC. The lower fluidity of AlSi8MnMg, had to be compensated by a temperature increment. Also a faster 2nd phase lead to better final quality of the parts. Therefore, the 2nd phase was also increased from the 1,8 m/s to the 2,1 m/s. Other parameters that show that could improve the quality of the parts, respect the parameters used in AlSi10MnMg alloy were the 1st phase speed, which was decreased to 0,35 m/s from the 0,4 m/s and the point where the speed is changed from 1st to 2nd phase, which was delayed to 300 mm instead of the previous 290 mm. The only parameter remaining at the same level was the break position, that was kept at 410 mm.

A minimum of 150 parts where casted from each alloy variant with the parameters that were related with a better part quality: 740ºC of melt temperature, 0,35 m/s of 1st phase speed, 2,1 m/s of 2nd phase speed, 300 mm of velocity changing point and a break position of 410 mm. Most of those parts were qualified with a 4 or 5 in the visual inspection checking.

3.1.1. X-ray analysis

Four plates of each alloy variant were selected for X-ray inspection following the same procedure described previously for AlSi10MnMg alloy variants. The results are summarized in Table 17, in which the critical (in terms of defects concentration) areas are identified by red borders. The red, white or

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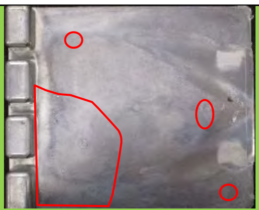
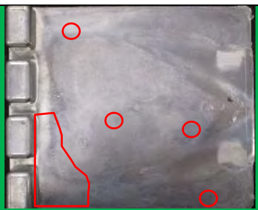
green bars on the side of the photos identity, respectively, critical, average or good castings in view of part selection for mechanical testing.

Table 17 – Images of the parts depicting the zones with presence of defects detected with X-ray

Variant #7 as Cast				
	3318 – high amount of porosity, in some cases clustered, in most cases dispersed, with bigger size	3327 – Diffused porosity, in some cases clustered, in most cases dispersed	3354 – Diffused porosity, in some cases clustered, in most cases dispersed	3357 – high amount of porosity, in some cases clustered, in most cases dispersed, with bigger size
Variant #8 as Cast				
	3173 – high amount of porosity, mainly clustered	3175 – high amount of porosity both clustered (bands) and dispersed, with bigger size	3180 – high amount of porosity, mainly clustered	3190 – high amount of porosity both clustered (bands) and dispersed, with bigger size
Variant #9 as Cast				
	2895 – medium amount of porosity, both clustered and dispersed	2897 – medium amount of porosity, both clustered and dispersed	2986 – medium amount of porosity, both clustered and dispersed	2987 – medium amount of porosity, both clustered and dispersed
Variant #10 as Cast				
	3391 – medium amount of porosity, clustered in various regions	3392 – low amount of porosity, clustered in various regions	3428 – high amount of porosity, clustered in various regions	3430 – medium amount of porosity, clustered in various regions

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3.1.2. Tensile tests

From some of the plates produced at Eurecat tensile specimens were machined and tested following A2 section from UNE-EN ISO 6892-1 and the same procedure described in Section 2.5. The average values obtained in the tests for the different alloy variants are presented in Table 18.

Table 18 – Average values obtained in the tensile tests for the different alloy variants

Variant	YS (MPa)	UTS (MPa)	elongation (%)
#7, as cast, vertical	122 ± 4,4	236 ± 10,9	4,6 ± 1,4
#8, as cast, vertical	127 ± 3,2	227 ± 27,8	3,1 ± 2,2
#9, as cast, vertical	113 ± 4,2	224 ± 6,6	3,1 ± 0,5
#10, as cast, vertical	123 ± 2,7	241 ± 9,9	3,8 ± 0,8

In addition, and in order to confront the obtained values with an independent testing site, some of the plates subjected to X-ray inspection at Univ. of Padova, were also mechanically characterized. The specimens were machined in the vertical direction, following the description presented in Section 2.6 (Figure 12). The average values obtained for each alloy variant are shown in Table 19 – Average values obtained in the tensile tests for the different alloy variantsTable 19.

Table 19 – Average values obtained in the tensile tests for the different alloy variants

Variant	YS (MPa)	UTS (MPa)	elongation (%)
#7, as cast, vertical	117 ± 4,7	240 ± 7,6	5,9 ± 1,1
#8, as cast, vertical	132 ± 0,8	231 ± 10,4	2,9 ± 0,5
#9, as cast, vertical	131 ± 5,3	238 ± 16,2	2,5 ± 0,6
#10, as cast, vertical	146 ± 2,6	239 ± 21,2	2,3 ± 0,8

It can be observed that the values of yield strength obtained in both testing facilities are similar to those reported in Deliverable 2.4 for specimens cast with the NADIA die. However, UTS and, specially, elongation is much lower than the values obtained with the NADIA parts. This decrement is, presumably, due to the higher presence of defects present in the plates.

3.1.3. Cross sections of tensile tests specimens

In order to better evaluate the variability of the castings and have more information about the reason of the pour ductility a cross-sections of some of the broken specimens, taken very close to fracture surface. The images of those cross-sections taken with an optical microscope are presented in Figure 19. In general, it can be observed pores and, in some cases, a considerable amount of porosity. Porosity is one of the most common defects present in HPDC and have a severe impact on the ductility of aluminium alloys.

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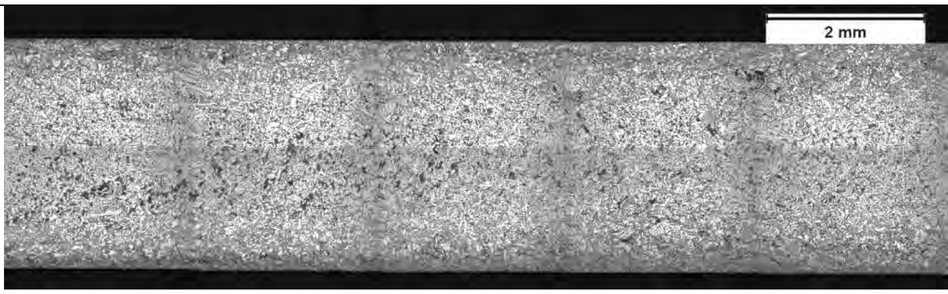
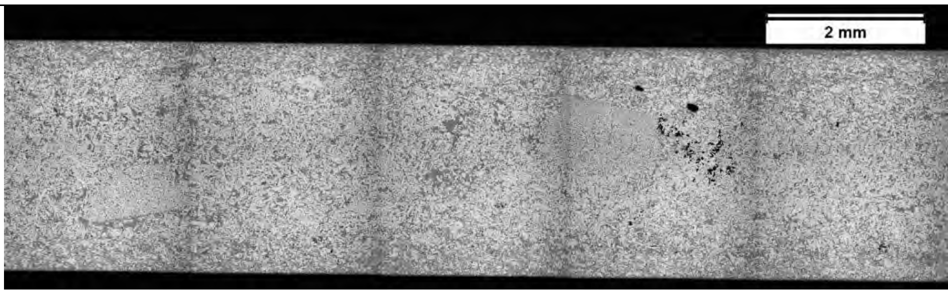
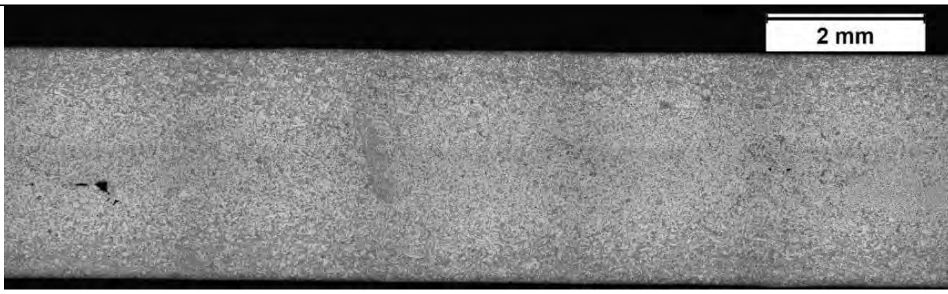
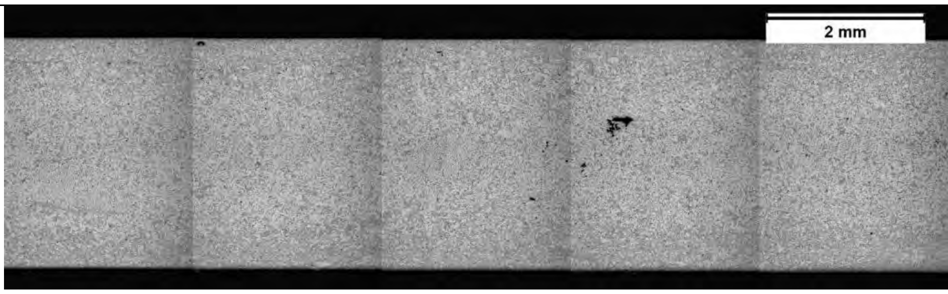
Variant	
#7, as cast, vertical	
#8, as cast, vertical	
#9, as cast, vertical	
#10, as cast, vertical	

Figure 19 – Cross-sections of tensile specimens

3.2. Characterization of parts produced with NADIA reference die

As already mentioned and detailed in Deliverable 2.4, 4.3 and 4.4, the same AlSi8MnMg alloy variants had been already tested with the NADIA die used to confront results obtained with variant 6 in section 2.8. The chemical composition of the 4 variants is the same presented in Table 15. Some of the parts produced were subjected to different T5 and T6 conditions to get the required mechanical properties requested by the frontal frame and shock tower. After optimization of the heat treatment, the obtained mechanical properties for both T5 and T6 conditions are presented in Table 20 and Table 21, respectively.

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Table 20 – Optimal mechanical properties obtained after T5 heat treatment for the different AlSi8MnMg alloy variants

T5				
	Alloy 7	Alloy 8	Alloy 9	Alloy 10
Heat Treatment Conditions	Ageing Temp=190°C Time=4h	Ageing Temp=190°C Time=4h	Ageing Temp=190°C Time=4h	Ageing Temp=190°C Time=4h
Yield Strength 0.2% (MPa)	179	205	199	219
Ultimate Tensile Strength (MPa)	289	306	315	331
Elongation (%)	6.8	6.5	5.9	5.3

Table 21 – Optimal mechanical properties obtained after T6 heat treatment for the different AlSi8MnMg alloy variants

T6				
	Alloy 7	Alloy 8	Alloy 9	Alloy 10
Heat Treatment Conditions	Sol. treatment Temp=510°C Sol. treatment Time=0.5 Ageing Temp=190°C Time=2h	Sol. treatment Temp=510°C Sol. treatment Time=0.5 Ageing Temp=210°C Time=1h	Sol. treatment Temp=510°C Sol. treatment Time=0.5 Ageing Temp=190°C Time=2h	Sol. treatment Temp=490°C Sol. treatment Time=1h Ageing Temp=230°C Time=0.5h
Yield Strength 0.2% (MPa)	158	205	178	182
Ultimate Tensile Strength (MPa)	238	254	258	251
Elongation (%)	12.9	10.9	10.4	11.0

3.2.1. Microstructural observation

A microstructural observation was conducted on samples extracted from gauge length of flat tensile test bar cast on the NADIA die in the as-cast, T5 and T6 thermal conditions. The microstructure was examined using optical microscope (OM). The cross section of the metallographic specimens was firstly grinded with silicon carbide impregnated emery papers from 200 to 2000 grit mesh size and finally polished with 3µm and 1µm alumina suspension paste respectively. After polishing samples were ultrasonically cleaned to remove any spots or dust particles present on specimens. Then samples were etched by solution of 0.5 ml HF: 99.5 ml distilled H2O for 10 seconds. After etching, samples were washed under running water and dried with air.

Figure 20 shows optical micrographs of as cast AlSi8MnMg alloys processed by HPDC at low and high magnification respectively. Porosity is a common defect observed in the HPDC process due to the air entrapment in the filling process and the shrinkage during solidification. Porosity is considered one of the main limitations affecting ductility of aluminium alloys. In all alloys several pores with various dimensions were observed, as can be observed in Figure 20, minimum porosity for AlSi8MnMg alloy was observed in Variant 6.

The as cast microstructure of all alloys consists of two types of α-Al primary phases i.e. α1-Al & α2-Al, continuous network of eutectic silicon phase and multiple intermetallic phases. The α1-Al is a primary phase also known as externally solidified crystals (ESC), which is solidified in the shot sleeve or formed during the molten melt transfer from holding furnace to shot sleeve. The α2-Al is a primary phase which is solidified in die cavity. The α2-Al phase, with globular shape, nucleated in die cavity is much finer than α1-Al phase because of high cooling rate. The coarse α1-Al entered into die cavity along with

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molten metal that forms the rest of the microstructure. The eutectic Si phases start precipitation once α2-Al finishes. β-Mg2Si phase was also identified distributed among Al-Si eutectic region. The distribution of other intermetallic compounds was found relatively uniform in the microstructure. In AlSiMnMg alloys two types of iron rich intermetallic compounds were observed; α-AlSiMnFe and βAlFeSi. The α-AlSiMnFe was found in coarse and fine form with different morphologies like, flower like, star like, blocky and polyhedral. Similar like α-Al phase, coarse α-AlSiMnFe was formed in shot sleeve solidification range while fine α-AlSiMnFe in die cavity in the pro-eutectic stage. The fine α-AlSiMnFe showing equiaxial morphology can benefit the strength, especially when the particle size was less than 1 µm. The β-AlFeSi, with needle like structure, is rarely observed distributed among the eutectic region. After T5 heat treatment, no significant changes in microstructure was observed, all the phases observed in as cast microstructural analysis was found in all T5 heat treated. However, significant change in microstructure of T6 heat treated samples were observed for all alloys. The fine fibrous eutectic silicon observed in as cast microstructure was transformed into spheroidal form during solution treatment. The β-Mg2Si phase were rarely observed after solution treatment which refers to effective solution heat treatment i.e., well dissolving of β intermetallic phase into α-Al matrix. The wider distribution of α-Al phase was found when compared to cast condition, indicating growth of α-Al phase. The coarsening to these α-Al phases occur during solution treatment due to high temperature diffusion and merging of neighbouring α-Al grains. The needle like β-AlFeSi was not observed as they transform into small fragments during solution treatment. The α-AlSiMnFe was found highly stable as no significant changes in their morphologies were observed in both T5 and T6 heat treated conditions.

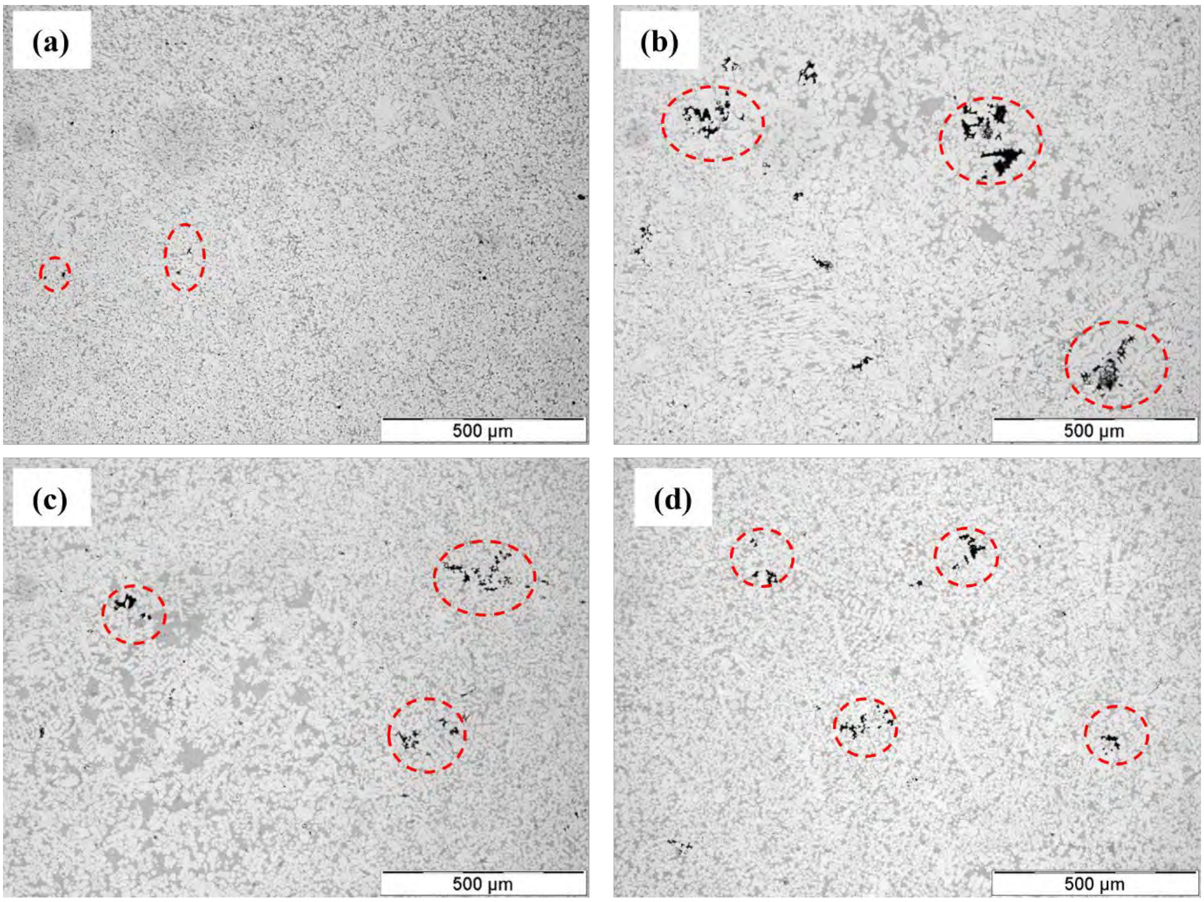

Figure 20 – Porosities in as cast (a) Alloy 7, (b) Alloy 8, (c) Alloy 9 and (d) Alloy 10

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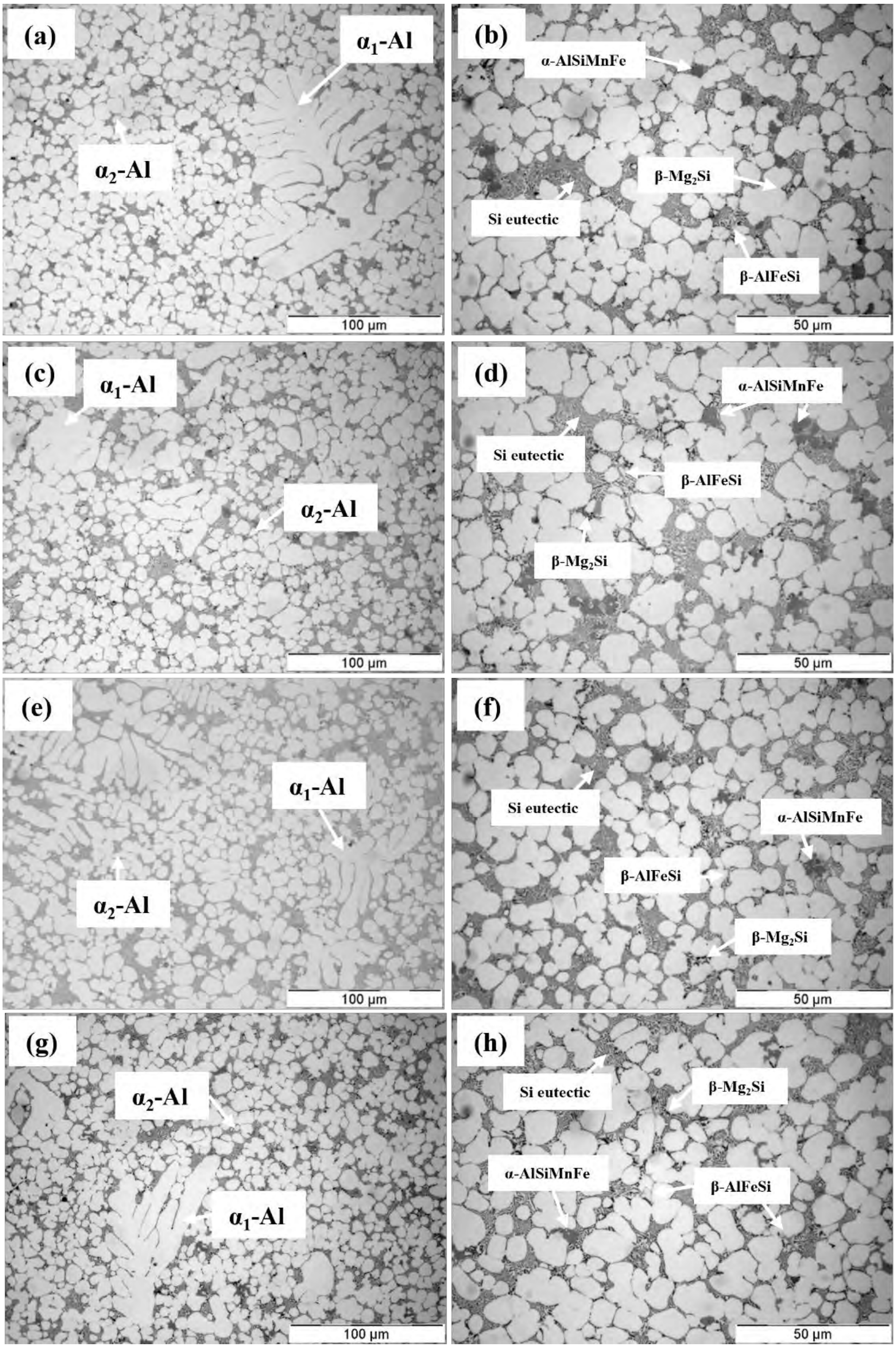

Figure 21 – Low magnification optical micrographs of as cast (a) Alloy 7, (c) Alloy 8, (e) Alloy 9 and (g) Alloy 10 showing primary and secondary α-Al ; high magnification optical micrographs of as cast (b) Alloy 7, (d) Alloy 8, (f) Alloy 9 and (h) Alloy 10 showing various phases present

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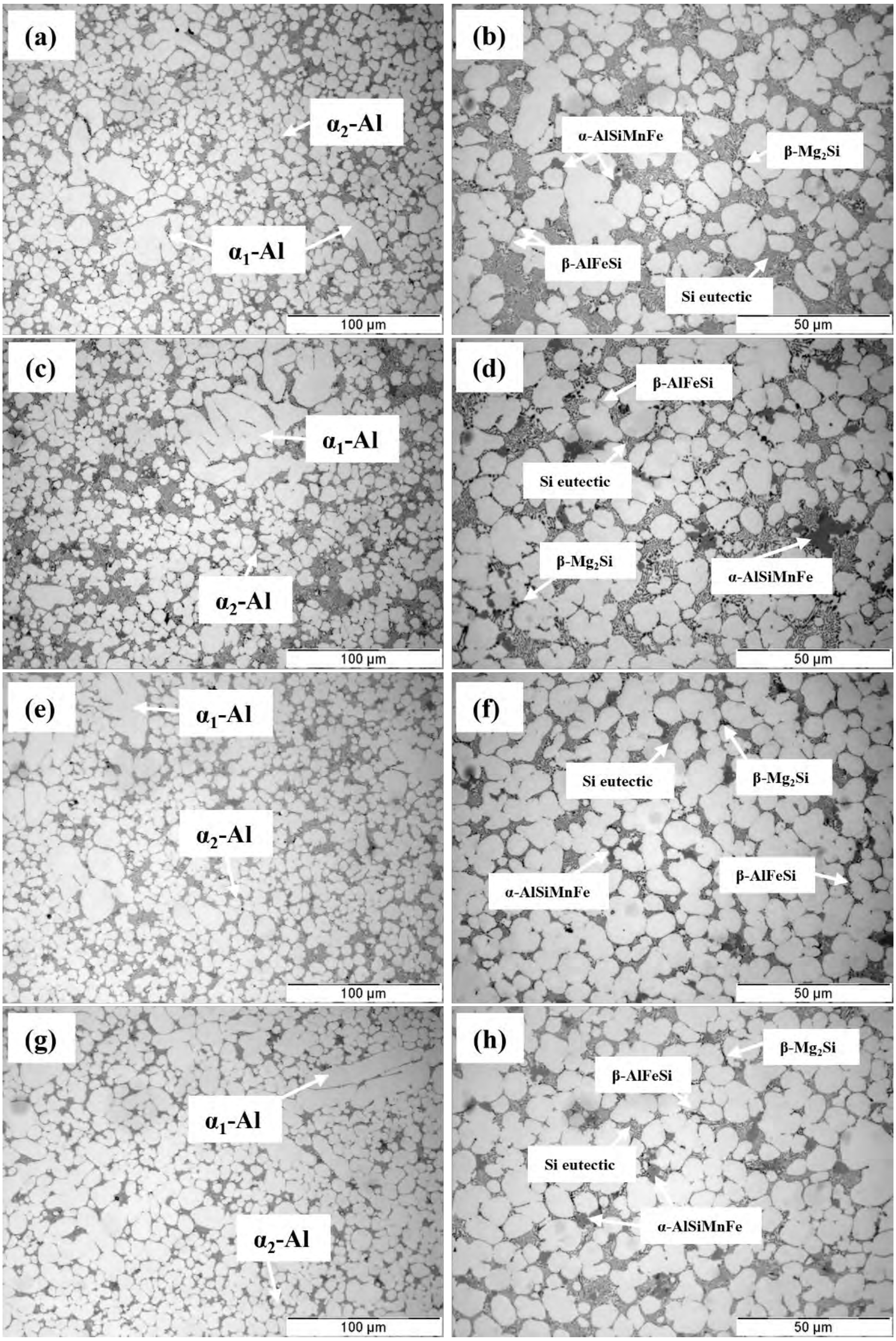

Figure 22 – Low magnification optical micrographs of T5 heat treated (a) Alloy 7, (c) Alloy 8, (e) Alloy 9 and (g) Alloy 10 showing primary and secondary α-Al ; high magnification optical micrographs of T5 heat treated (b) Alloy 7, (d) Alloy 8, (f) Alloy 9 and (h) Alloy 10 showing various phases present

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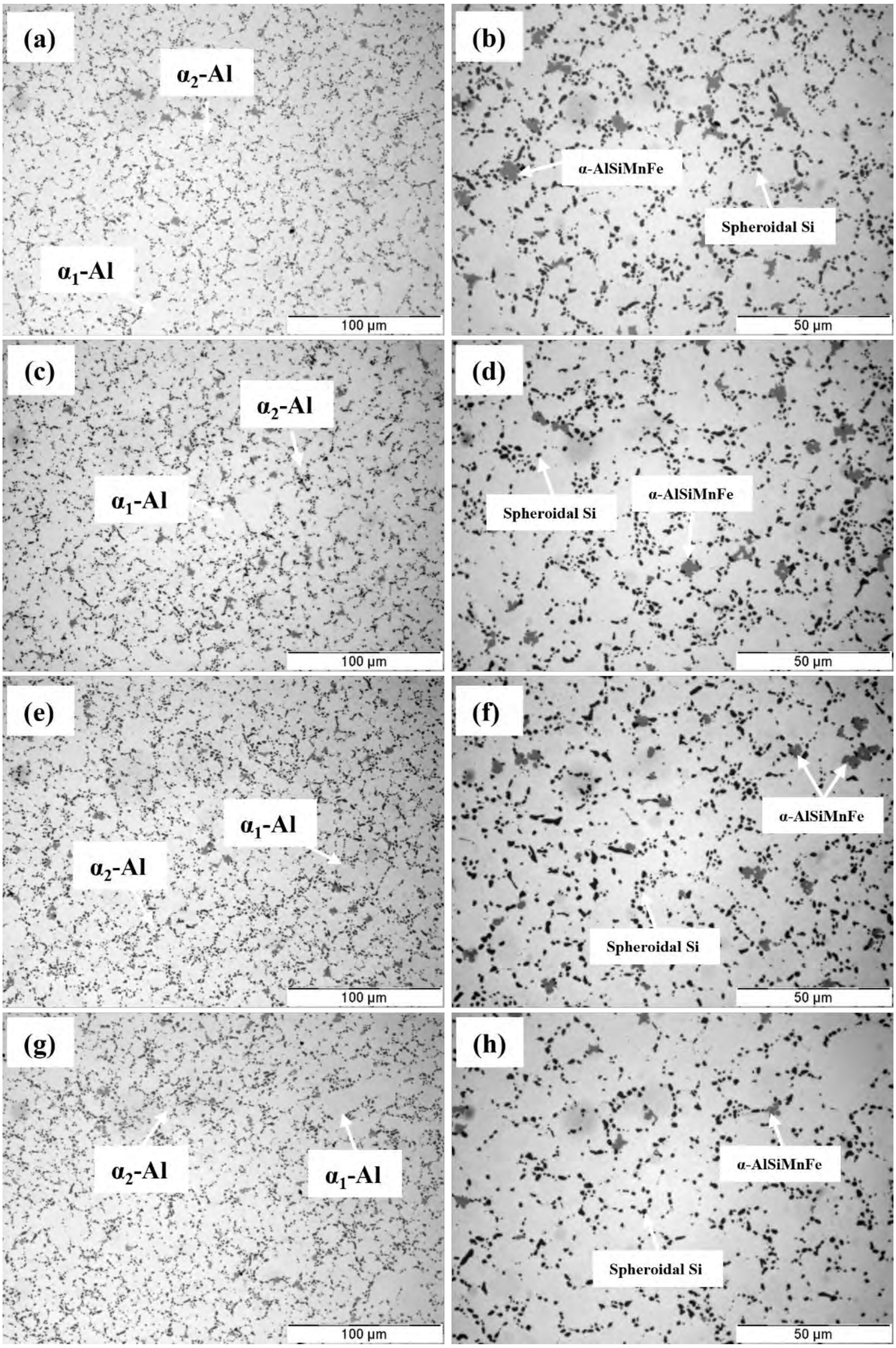

Figure 23 – Low magnification optical micrographs of T6 heat treated (a) Alloy 7, (c) Alloy 8, (e) Alloy 9 and (g) Alloy 10 showing primary and secondary α-Al ; high magnification optical micrographs of T6 heat treated (b) Alloy 7, (d) Alloy 8, (f) Alloy 9 and (h) Alloy 10 showing various phases present

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4. Characterization of AlMg3 alloy variants

Two AlMg3 alloy variants were selected for further development and to be tested at Eurecat HPDC industrial laboratory (Table 22).

Table 22 – Chemical composition of the 2 AlMg3 variants selected for further development within HPDC process

AlSi8MnMg0.3	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Pb	Sn	Ti	Co
Variant 11	0,2-0,3	<0,15	0-0,05	0,8-1,1	2,6-2,8	<0,03	<0,03	<0,08	<0,03	<0,03	<0,1	0,3-0,4
Variant 12	0,2-0,3	<0,15	0-0,05	0,9-1,2	2,1-2,3	<0,03	<0,03	<0,08	<0,03	<0,03	<0,1	0,3-0,4

AlMg3 alloy variants were found to present lower fluidity and poorer castability that all the variants previously investigated. The preliminary trials conducted with these alloy variants show that was hard to fill up the whole cavity and obtain castings without cold joints or other filling defects. Cracking of the part, during solidification, was also a commonly observed issue.

In order to get parts with the better possible quality, a casting parameter optimization was done, following the same procedure as it has been previously described for AlSi8MnMg alloy variants. For AlMg3 alloy variants the process parameters that give better results were:

- Melting temperature: 740ºC
- 1st phase speed: 0,45 m/s
- 2nd phase speed: 3 m/s
- Velocity changing point: 290 mm
- Breaking point: 420 mm

With this casting parameters a minimum of 150 parts were cast.

4.1. Tensile tests

From some of the plates produced at Eurecat tensile specimens were machined and tested following A2 section from UNE-EN ISO 6892-1 and the same procedure described in Section 2.5. The average values obtained in the tests for the different alloy variants are presented in Table 18.

Table 23 – Average values obtained in the tensile tests for the different alloy variants

Variant	YS (MPa)	UTS (MPa)	elongation (%)
#11, as cast, vertical	113 ± 6	141 ± 22	2,1 ± 0,9
#12, as cast, vertical	98 ± 15	124 ± 39	2,5 ± 1,6

The mechanical properties attained show the great difficulty observed to completely fill and cast sound parts with the geometry of 3mm flat plates. This is quite clear by comparing the mechanical properties obtained with Eurecat flat plates and the result of the tensile tests conducted on the tensile test specimens obtained directly with NADIA die (Deliverable 2.4 and 4.4).

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Table 24 – Average values obtained in the tests conducted with the tensile specimens obtained with NADIA die

Variant	YS (MPa)	UTS (MPa)	elongation (%)
#11, as cast, vertical	125 ± 6	235 ± 22	13,9 ± 0,9
#12, as cast, vertical	123 ± 0,4	234 ± 1,3	9,8 ± 1,6

With the NADIA die, it was proved that these alloys could achieve the minimum requirements of 120 MPA of YS, 180 MPa of UTS and 8% of Elongation without any kind of heat treatment. However, the trials conducted with the flat plates, suggest that these mechanical properties could be difficult to reach in a component with 3 mm thickness and a considerable extension.

5. Conclusions and Outlook

The present document summarizes the investigation conducted on the alloys developed in SALEMA and assess their performance.

- For AlSi10MnMg alloy variants:
  - Mechanical properties can be reached by HPDC, as demonstrated by the parts produced with the NADIA die
  - 3 mm thick part with a considerable extension, Eurecat casting plate, was produced, presenting diverse concentration of defects. The minimum requested mechanical properties are not meet in those parts
  - All alloy variants fulfil the corrosion requirements defined and assessed by Stellantis

- For AlSi8MnMg alloy variants:
  - Eurecat testing plate also present variable quality for this alloy family and do not meet the minimum mechanical requirements defined for the demonstrators
  - Mechanical properties obtained in direct cast tensile specimens meet easily the requirements
  - The microstructure observed is composed by the expected phases and evolve as anticipated when a solution treatment is applied

- For AlMg3 alloy variants:
  - Present lower fluidity and castability than the alloys containing Si and higher casting speeds are required in order to completely fill up the die cavity
  - Mechanical properties in the flat plates are quite poor, especially elongation, and do not meet the minimum requirements, which can be reached successfully with the tensile specimens from NADIA die

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6. Next steps

In the following months the properties defined will be conducted the characterization of the demonstrators produced with the SALEMA alloys and it would be checked if it is possible to replicate the properties obtained with the NADIA die and achieve the minimum requirements under industrial production conditions:

- Determination of tensile properties, fatigue, toughness and corrosion of Variant 4 and Variant 7 on the Frontal Frame from Endurance
- Determination of tensile properties, fatigue, toughness and corrosion of Variant 6 and Variant 12 on the Shock Tower from Fagor Ederlan

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