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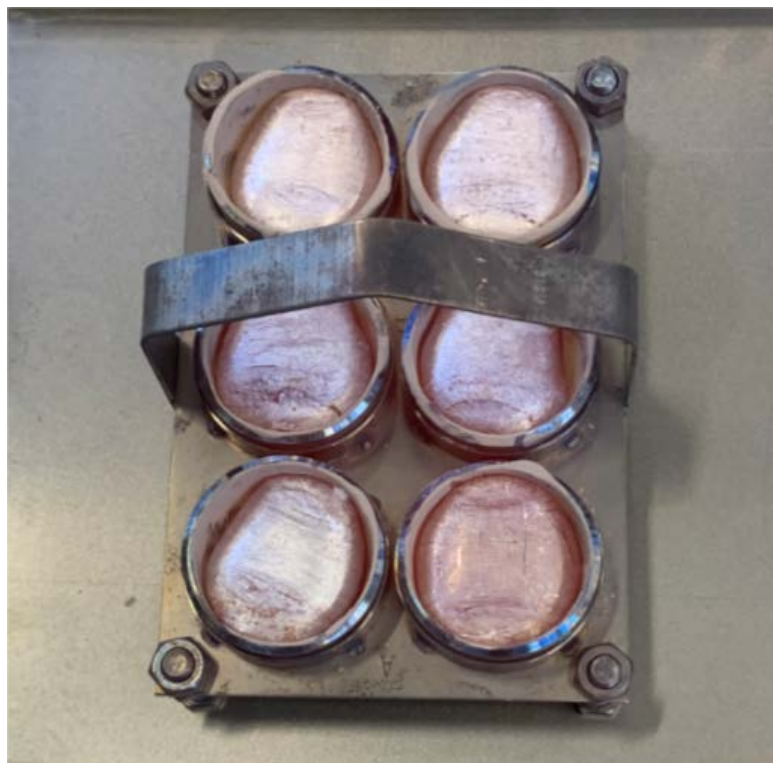
# Report

## Oxidation of Al-Mg alloys in small scale experiments

Tests at Linde

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# Report

## Oxidation of Al-Mg alloys in small scale experiments

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**ABSTRACT****Abstract heading**

While a significant amount of work has been done on the oxidation of Al-Mg alloys a limited portion of the work has been done under a true furnace atmosphere created by an air-fuel or oxyfuel burner. A series of oxidation tests were carried out in Lindes's test furnace using an air-fuel burner. Al samples weighing approximately 65 grams with between 0 and 5 % Mg were placed in a steel crucible and further placed in an air-fuel fired box furnace for 0.16, 0.5, 1, 2, 3 or 5 hours before being removed to cool. The samples were analysed afterwards by scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS). Analysis showed that all samples had a MgAl<sub>2</sub>O<sub>4</sub> layer on the surface and that at higher Mg contents internal oxidation occurred resulting in the formation of MgO or MgAl<sub>2</sub>O<sub>4</sub> clusters in the melt. This would indicate that under the oxidation conditions in an air fired furnace a significant portion of the oxidation may occur inside the melt leading to an increase in oxide inclusions.

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## 1 Introduction

For a long time, aluminium alloys with an increased Mg-content have had a problem with oxidation. In October 2018 and January and September 2019, small scale experiments at Linde in Stockholm were done to try to figure out and understand the interrelationships occurring during the oxidation of Al-Mg alloys. Atmosphere, Be-content, Mg-content and the geometry of the crucibles have been different factors that were altered to better understand oxidation of Al-Mg alloys. In this report some theory will be explained, the experiments will be described, and the results will be discussed. Some of the samples were further analysed in SEM to look into the oxide layer as well as looking at the Mg-gradient in the samples. This was done to hopefully get a better understanding of the mechanisms behind the oxidation in the alloys.

## 2 Theory/Background

In the aluminium industry Mg is a common alloying element. By adding Mg, the strength properties improve in the material amongst other things. A significant problem however with these alloys is the substantial oxidation. Generally, this happens by Mg forming MgO which again forms the spinel structured  $MgAl_2O_4$  which is a non-desired phase. To lower the oxidation level, it has proven effective to add Be in the ppm level. This lowers the oxidation significantly. Atmosphere also shows an effect and  $CO_2$  especially proves itself to lower the oxidation rate in the melt [1]. The small-scale experiments that was performed at Linde in 2018 and 2019 were done specially to observe how the difference in Mg and Be-content and type of atmosphere would influence oxidation. The background is to get a better understanding of this topic and to better understand the mechanisms that slow/inhibit oxidation in the melt.

## 3 Method

It was as previously mentioned performed experiments at Linde in Stockholm in October 2018 and again in January and September 2019. It was done over a timespan of four days where one of these days was done the experiments with the small crucibles. In 2018 one had crucibles with varying Mg content, but in 2019 you had samples with both varying Mg and Be content. Only these experiments with small crucibles will be discussed further in this report, whereas the tests in the large crucible (10 kg) are discussed elsewhere. The experiments were done with small crucibles of two different kinds. The first one was a tall ceramic crucible. This crucible will from now on be referred to as Ceram C. The second one was made from stainless steel. This crucible will from now on be referred to as Steel C. See pictures below.

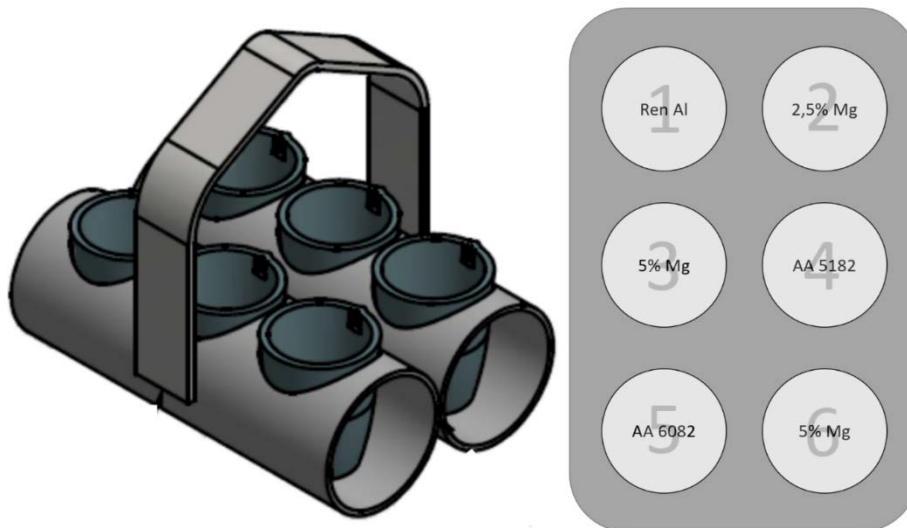


**Figure 3-1:** Left: Ceram C, the crucible used in 2018 as well as in some of the experiments in 2019. Right: Steel C, the crucible used in the other experiments in 2019.

### 3.1 Setup/preparations

In 2018 only Ceram C was used. It was prepared 6x6 samples so that one could be able to observe different alloys and look at the same alloys at different times in melted form. Sets of six samples were placed in six

different sample holders which can be observed in Figure 3-2 (left). How the alloys were distributed is illustrated in Figure 3-2 (right).



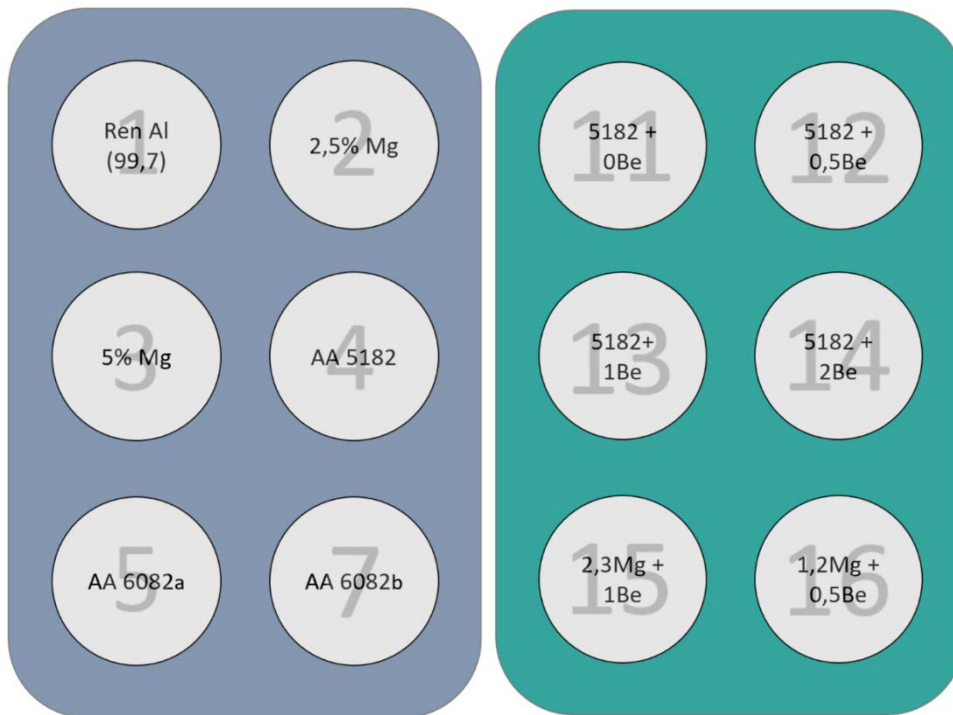
**Figure 3-2: Left: Sample holder with six crucibles, Right: Setup sample holder October 2018.**

To make the alloys with the correct content a Mg-block with the right weight was melted with pure aluminium (99,7%). The composition of these self-made alloys was confirmed with later examination of samples at Gränges. The different melts were poured and then solidified with the shape of the ceramic crucible see Figure 3-3. The 5182 and 6082 alloys were delivered as an industry standard from Alcoa and Hydro respectively.



**Figure 3-3: How the samples were pre-casted.**

In 2019 the setup was similar in many ways. The difference was that both Ceram C and Steel C were used. In the first experiment the 21<sup>st</sup> January Ceram C were used with the samples that contained Be, but the Steel C were used with the alloys that had varying Mg content. In the later experiments Steel C was also being used with the alloys containing Be. With both Ceram C and Steel C, it was used sample holders with six alloys in each. The setup can be observed in Figure 3-4 below.



**Figure 3-4: Setup 2019 samples without Be (left) and with Be (right).**

The different sample holders were signed A, B, C etc. for the samples without Be and I, II, III etc. for the sample holders with Be. The difference between the AA 6082a and the AA 6082b is that AA 6082a(old) contained 0,75% Mg, but AA 6082b(new) contained 0,85% Mg.

Regarding the Be containing alloys there was used a different procedure of how to get the right composition. In contrast to the alloys not containing Be which were made in 2018 the Be-alloys were made by mixing three different alloys with known composition delivered from Alcoa. The three being 5182 with 2ppm Be, 5182 with 0ppm Be, and pure Al (99,7). The six alloys in the sample holder with Be, is therefore a result of a calculated mixing of these alloys.

## 3.2 Execution

### 3.2.1 2018

The experiment including small crucibles was performed 26<sup>th</sup> of October, at Linde's site in Stockholm. The oven was preheated and the six sample holders with six crucibles each were placed inside the oven.



**Figure 3-5: Left: The samples being put into the oven. Right: The samples in the oven while gas being burned.**

The different sample holders were placed in the oven at the same time and taken out at different points in time. These points in time after melting were 10min, 30min, 1h, 2h, 3h and 5h. Pictures of a removed sample holder and the remaining samples in the furnace can be seen in Figure 3-6.



**Figure 3-6: Left: First sample holder out of the oven, 10 min. Right: The five remaining samples in the oven.**

The process of taking out each sample holder was repeated until the last one was taken out after 5 hours. The experiment was performed along with people from Hydro, Gränges, Alcoa and Linde.





**Figure 3-7: Left: Sample holder 3 being taken out after 1 hour. Right: The team that performed the experiment.**

### 3.2.2 2019 – 21<sup>st</sup> of January

The small-scale experiments in 2019 were performed in the same lab as in 2018. As in 2018 it was performed experiments over a timespan over five days, from 21-25<sup>th</sup> of January. The experiment involving different Al-Mg alloys held in air-fuel, to observe the oxidation over time, was performed 21<sup>st</sup> of January. This experiment consisted of more sample holders and two different crucibles unlike the 2018 experiments as mentioned previously. The reason for this was the interest of looking into the effect of Be in addition to the alloys that were investigated in 2018. The steel crucibles were, as the name says, made of steel which made it necessary to apply a coating to avoid dissolution and other unwanted reactions. The coating that was used was Velvacat ST 802 which is an alcohol-based coating from ASK chemicals. It was dried by putting the crucibles in the oven at about 730 degrees for 30 minutes and then cooled beforehand.



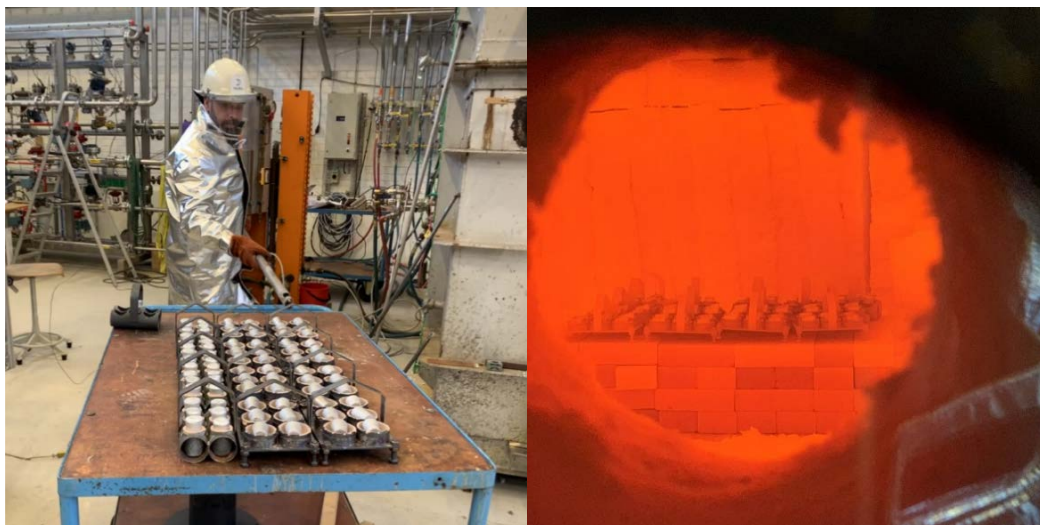
**Figure 3-8: Left: The crucibles being set into the oven for drying, Right: The crucibles with dried coating.**

After the coating was finished the experiment was executed the same way as in 2018. One of the notable differences was an increased amount of sample holders as the alloys were looked at over an increased timespan. The samples were tested at 10min, 30 min, 1h, 2h, 3h, 4h, 5h and 6h, which led to two more measurements in

time compared to 2018(4h and 6h). The precast alloys that were being melted were all in the shape of Ceram C. All of these were weighted beforehand so that one could check the weight increase afterwards. These precast samples were put into both Ceram C and Steel C (see Figure 3-9, left).



**Figure 3-9: Left: Setup with precast alloys. Note that the precast alloys in the steel crucibles have the shape of the ceramic crucible. Right: Weighing the precast samples before the experiment.**



**Figure 3-10: Left: Samples being brought into the oven. Right: Samples in the oven.**

After the samples had been in the oven the different sample holders were taken out at the right time in the same way as in 2018.



**Figure 3-11: Left: A sample holder straight after being taken out of the oven. Right: Sample holders out after 2 hours.**

### 3.2.3 2019- Further small-scale experiments after 21<sup>st</sup> January

In 2019 the main experiment with small crucibles was done Monday 21<sup>st</sup> of January, however in addition to this side experiments the other days with small crucibles were done. The reason for this was the interest in seeing the effect different atmospheres had on different alloys regarding oxidation. These tests were done as side experiments the 22<sup>nd</sup> 23<sup>rd</sup> 24<sup>th</sup> and 25<sup>th</sup> as the experiments with big crucibles were the main experiments these days. The alloys used were the Be-containing alloys that were used the 21<sup>st</sup>. They were placed in the sample holder in the same arrangement as done the 21<sup>st</sup>.

Most of the experiments were set at 4 hours, but on the 24<sup>th</sup> they were held for 8 hours. In addition to these experiments it was performed an experiment with the alloys 6082 and 5182. This was performed Wednesday the 23<sup>rd</sup> in the afternoon. Six samples of 5182 and six with 6082 were placed for four hours in the oven. They were later used as nucleation seeds in big crucibles. However, the weight was measured before and after oxidation in all the samples. An overview over what was done regarding small crucibles from the 22<sup>nd</sup> – 25<sup>th</sup> can be seen below.

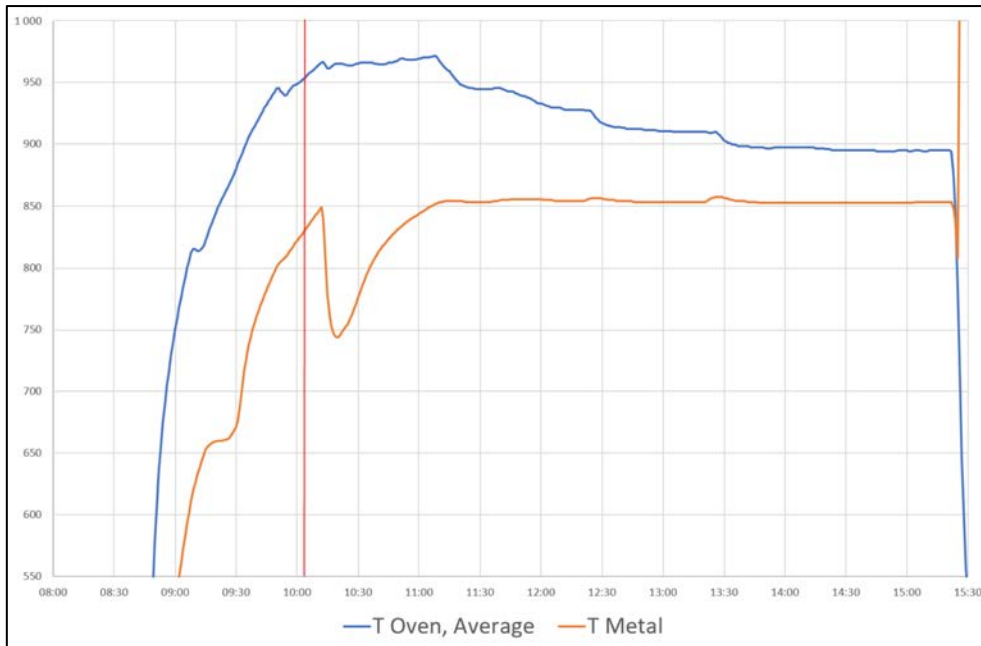
**Table 1: Experiments done 22<sup>nd</sup> -25<sup>th</sup> of January 2019.**

<b>Tuesday 22<sup>nd</sup></b>	<b>Wednesday 23<sup>rd</sup></b>	<b>Thursday 24<sup>th</sup></b>	<b>Friday 25<sup>th</sup></b>
<b>Morning: (Oxy fuel)</b> - Be-tests with Steel crucibles, 4 hours <b>Afternoon: (Oxy fuel + air leakage)</b> - Be tests with Steel crucibles, 4 hours	<b>Morning: (Air fuel)</b> - Be-tests with Steel and Ceramic crucible, 4 hours <b>Afternoon: (Air fuel + air leakage)</b> - Be tests with Steel crucible, 4 hours - 5182 and 6082 tests for nucleation, ceramic crucible, 4 hours	<b>All day: (Oxy fuel + air leakage)</b> - Be tests with steel crucibles, 8 hours - Al-Mg tests with ceramic crucibles, 8 hours	<b>All day: (Oxy fuel + extra H2O)</b> - No data collected

### 3.3 Oxidation conditions

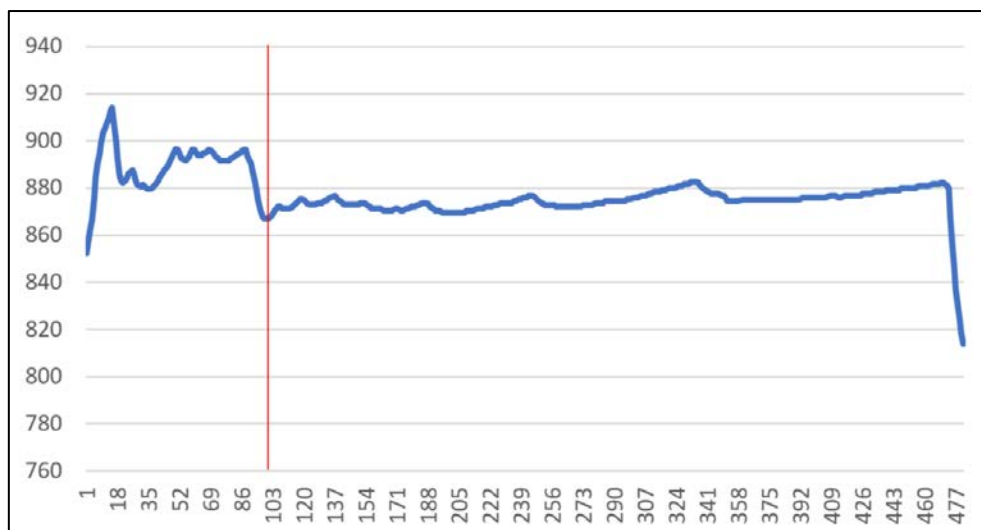
It was attempted to have as identical conditions as possible regarding the two experiments including small crucibles in 2018 and 2019. Parameters such as temperature and atmosphere were the most important conditions that had to be similar.

The temperature in the two experiments was attempted to be as similar as possible. The temperature in the oven was between 840°C and 970°C. In 2018 the metal temperature was also measured, something that wasn't done in 2019. (See Figure 3-12)



**Figure 3-12: Temperature 2018. The red line indicates the point in time when melting occurred. X-axis shows time and Y-axis shows temperature (°C).**

In 2019 only the temperature in the oven was measured as no thermocouple was put into any of the samples. The temperature in the experiment can be observed in Figure 3-13.



**Figure 3-13: Temperature 2019, oven. The red line indicates the point in time when melting occurred. X-axis shows time(min) and Y-axis shows temperature (°C).**

The atmosphere was also attempted to be as similar as possible. Unfortunately, two different types of gases were used the two different years. In 2018 the gas used consisted of 97,8 % propane, and 2,2% butane to get the desired atmosphere. In 2019 the gas used consisted of 92,62% methane, 6,45% ethane, 0,67% propane, 0,12 % butane and 0,14% nitrogen.



**Figure 3-14: Atmosphere 2018, the red line indicates the point in time when the samples melted.**

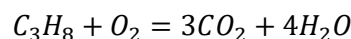
The atmosphere in 2018 consisted of about 2% O and 12 % CO<sub>2</sub> while in the atmosphere in 2019 consisted of about 2% O and 10% CO<sub>2</sub> (dry basis).

**Table 2: Atmosphere 2018 (Number 8).**

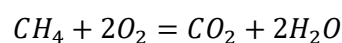
Nr	Datum	Leg.	Metall-temp	Bränsle	Brännare	Extra	Oxygen torr	CO2 torr
1	22.10.2018	5182	830	propan	oxy-fuel		3,7	87,4
2	23.10.2018	5182	860	propan	luft		1,1	12,6
3	23.10.2018	5182	860	propan	luft	läckluft	11,3	6,65
4	24.10.2018	5182	860	propan	oxy-fuel		5,5	79,4
5	24.10.2018	6082	870	propan	luft		1,1	12,9
6	25.10.2018	6082	870	propan	luft	läckluft	11,2	7,0
7	25.10.2018	6082	870	CH4+H2	oxy-fuel	1/4.6	6,6	83,5
8	26.10.2018	5 st	850	propan	luft		2,2	12,4

This data neglects the water vapour in the atmosphere and counts the actual atmosphere minus water vapour as 100% of the atmosphere. If one assumes pure propane and pure methane in the two experiments one gets the reaction equations:

2018:



2019:

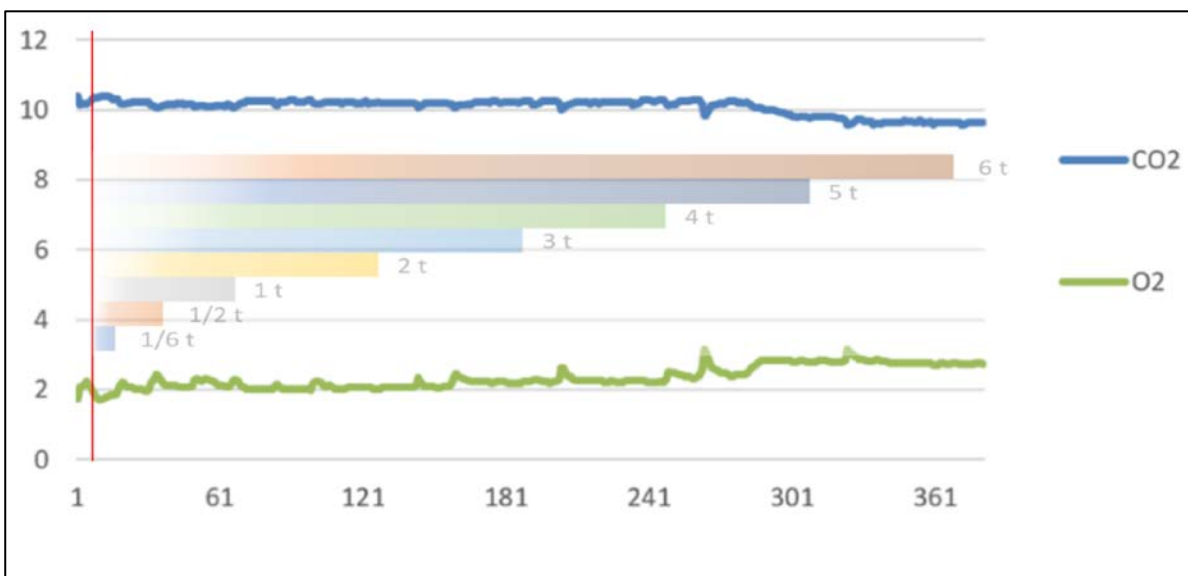


As one could observe water vapour should not be ignored as it is a substantial part of the atmosphere and is produced in a substantial amount by burning the gases. As one also could observe the ratio between water vapour and CO<sub>2</sub> is not the same in the two equations. More water vapour is produced by burning methane compared to propane which is something worth noticing as it could have influenced the two experiments. The correct atmosphere which doesn't ignore water vapour for 2018 be calculated by the equation:

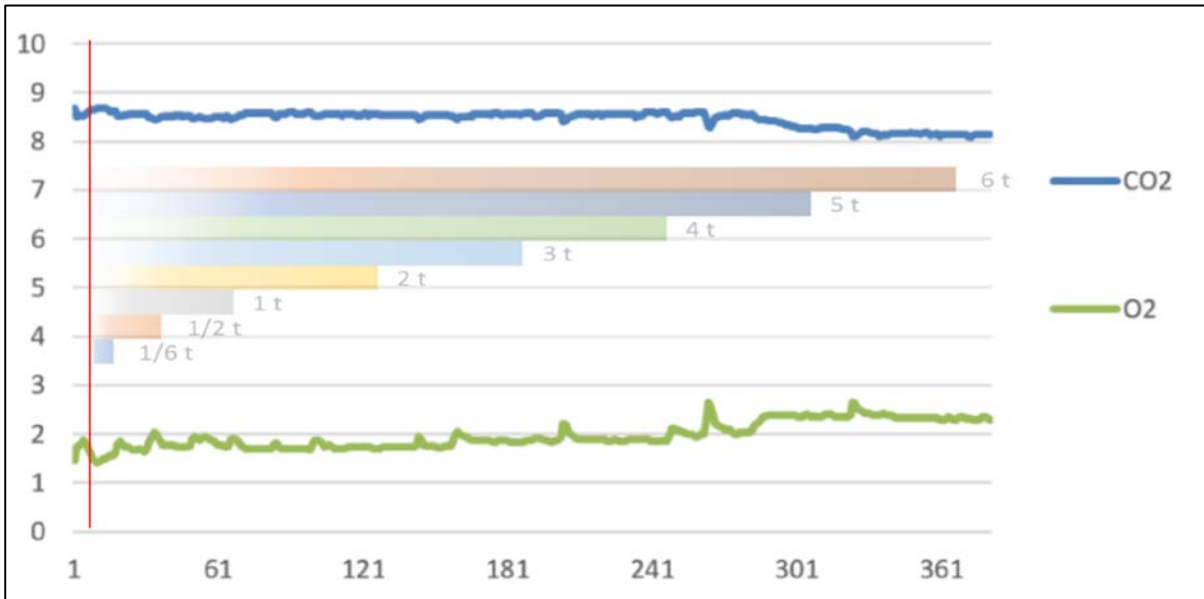
$$\frac{\%x_{dry}}{100\% + \frac{4}{3}12,4\%} = \frac{\%x_{wet}}{100\%}$$

Were %x<sub>dry</sub> is the percentage of one of the gases compared to the dry atmosphere excluding water vapour and %x<sub>wet</sub> is the percentage of one of the same gas but compared to wet atmosphere not excluding water vapour gas. This gives %CO<sub>2</sub> = 10,6% and %O<sub>2</sub> = 1,9% including water vapor.

From 2019 the atmosphere was plotted in the timespan one was interested in. Both dry and wet data were plotted (Figure 3-15 and Figure 3-16). In this experiment the gas with methane was used.



**Figure 3-15: Atmosphere 2019, dry data (excludes water vapour). X-axis shows time (min) and the Y-axis shows percentage of the total atmosphere.**



**Figure 3-16: Atmosphere 2019, wet data (includes water vapour). X-axis shows time(min) and the Y-axis shows percentage of the total atmosphere.**

Wet measurements are the plotted data that include water vapour. The red lines indicate the point in time for melting of the metal in the crucibles.

As one could observe, the atmosphere in the two years are not that different. The CO<sub>2</sub> concentration deviates a bit as it was about 10,6 % in 2018 but between 8 and 9 % in 2019.

When data from the two experiments are compared to each other it is assumed that the differences in conditions are negligible and that the data is comparable. However, in later experiments this is something that could and maybe should be controlled better so that the data compared would be more trustworthy.

Regarding the experiments with varying atmosphere in 2019 the conditions were as following:

**Table 3: Conditions in the samples from 22<sup>nd</sup> – 25<sup>th</sup> including the conditions for the 4 hours sample the 21<sup>st</sup>.**

Day	Atmosphere/burner	CO <sub>2</sub> wet	H <sub>2</sub> O wet	O <sub>2</sub> wet	N <sub>2</sub> wet	Metal T	Time
21 <sup>st</sup>	Air fuel	8,5	16,3	1,8	73,4	-	4
22 <sup>nd</sup> morning	Oxy fuel	31,2	60	1,4	7,3	836	4
22 <sup>nd</sup> afternoon	Oxy fuel+ air leakage	26,5	51,2	4,2	18,2	855	4
23 <sup>rd</sup> morning	Air-fuel	8,8	16,9	1,5	72,8	841	4
23 <sup>rd</sup> afternoon	Air-fuel + air leakage	5	9,5	10,2	75,2	841	4
24 <sup>th</sup>	Oxy fuel+ air leakage	26,6	51,2	4,2	17,9	857	8
25 <sup>th</sup>	Oxy fuel + extra H <sub>2</sub> O	10,6	86,3	1,4	1,7	850	4

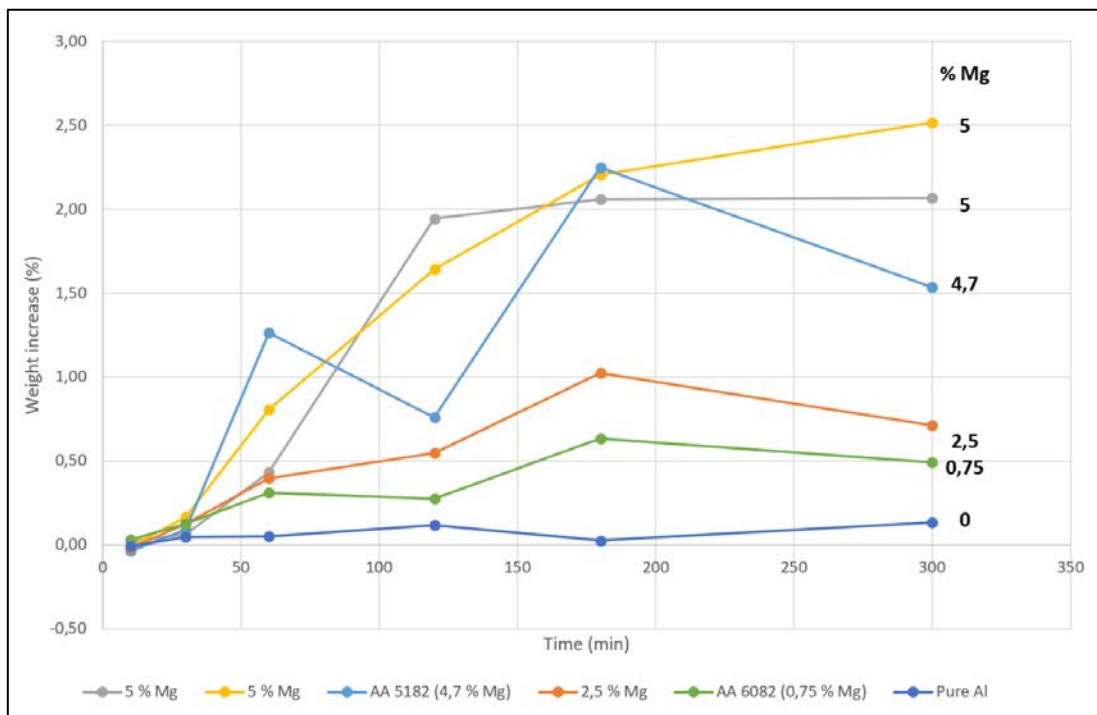
The metal T was not measured the 21<sup>st</sup>, but since the oven temperature was at 874(Avg., 4 hours) one can assume it was about the same metal temperature as the experiments the last four days.

## 4 Results

Overall the experiments performed the two years have given interesting results. It can be observed that the Mg content, Be-content, atmosphere and surface area all have an effect on oxidation.

### 4.1 2018

In 2018 the weight increase of the different samples showed a clear correlation between the Mg-content and the weight increase after oxidation. However, the results from the AA 5182 measurements vary significantly.

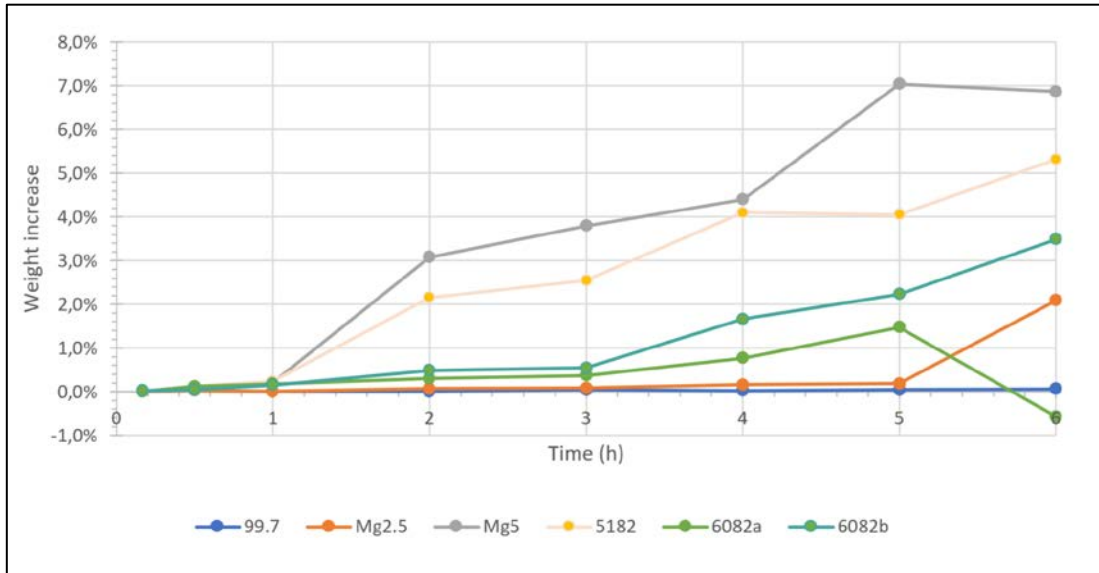


**Figure 4-1: Weight gain over time due to oxidation, small scale samples, different alloys with different Mg-content, Air Fuel, 26.10.2018.**

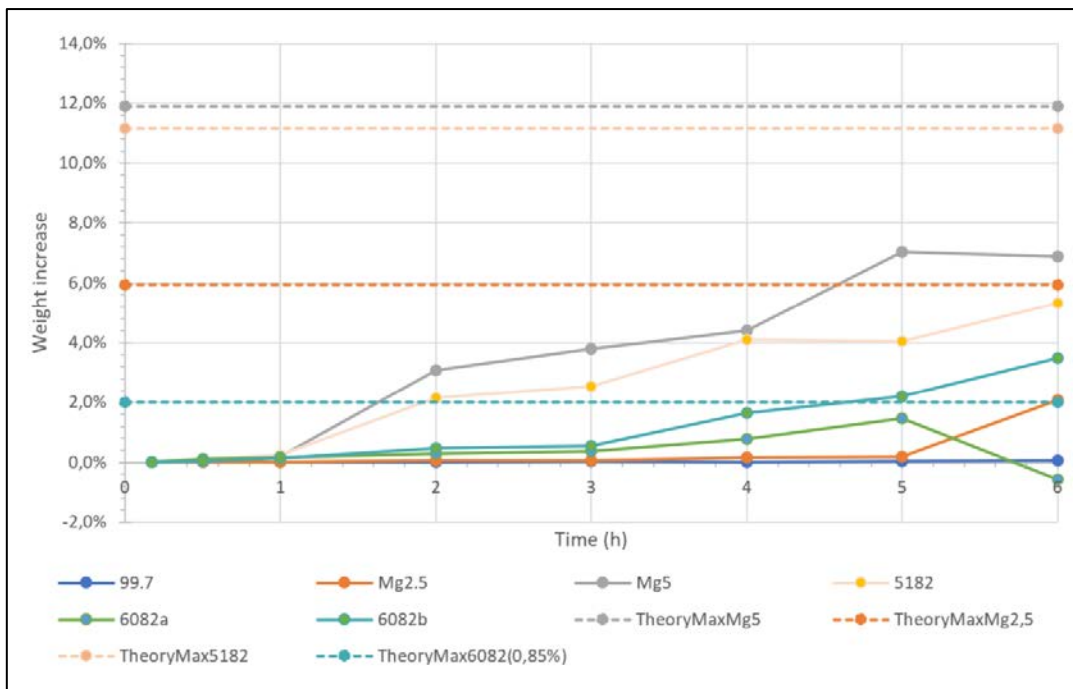


**4.2 2019**

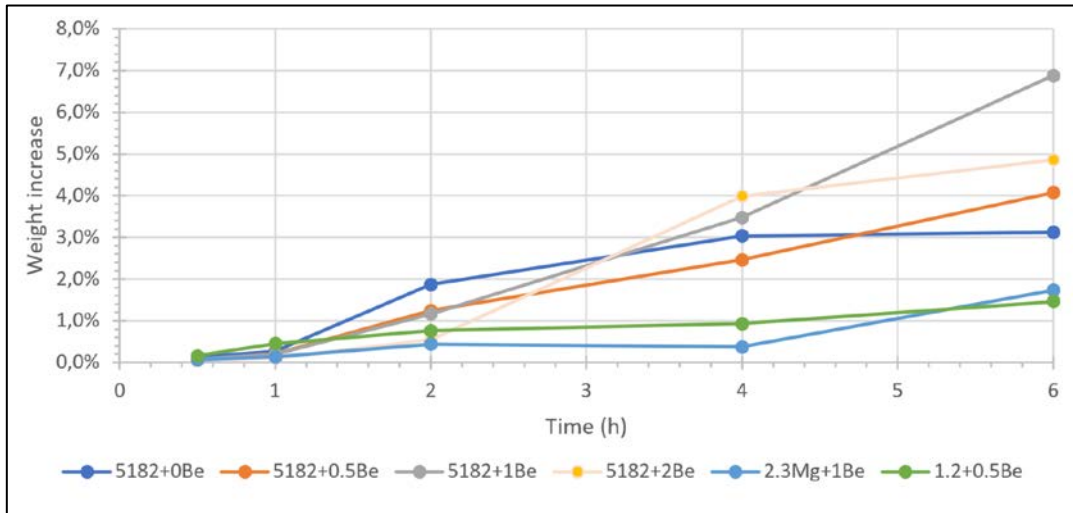
21<sup>st</sup> of Jan



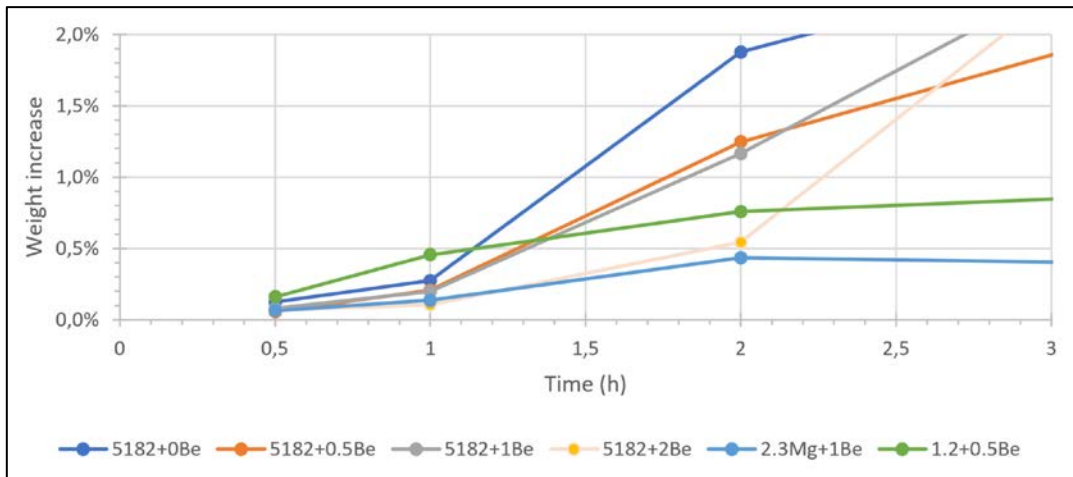
**Figure 4-2: Weight gain due to oxidation, small scale samples, Al-Mg alloys (No Be), Air Fuel, 21.01.2019.**



**Figure 4-3: Measured weight gain compared to theoretical weight gain given formation of only MgAl<sub>2</sub>O<sub>4</sub>, Air Fuel, Al-Mg samples, 21.01.2019.**



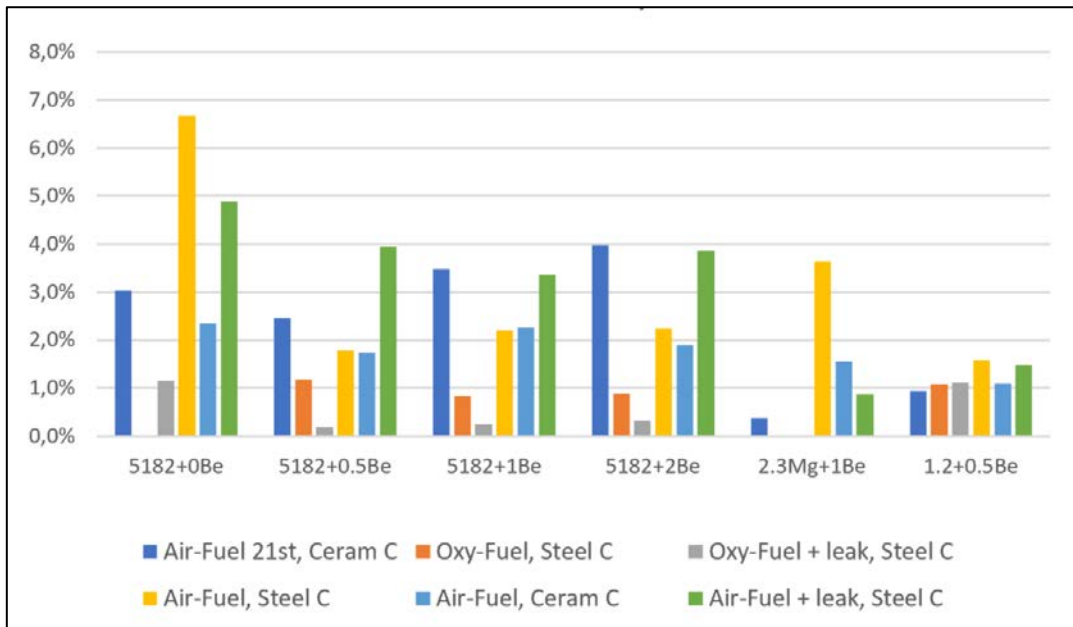
**Figure 4-4: Weight gain over time, samples with varying Be-content, Air Fuel, 21.01.2019.**



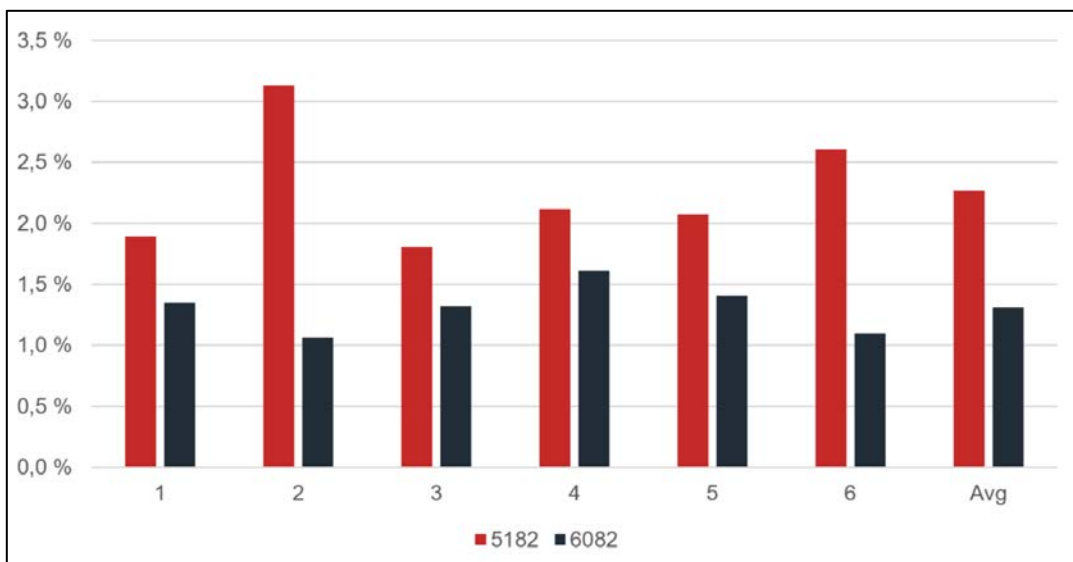
**Figure 4-5: Weight gain Be-samples, first three hours, Air Fuel, 21.01.2019.**

22<sup>nd</sup> -25<sup>th</sup> of Jan

The data gathered from the experiments regarding varying atmosphere resulted in Figure 4-6. The reason for some of the data being left out is because it either some metal got stuck in the crucible splashed out making that data point invalid.

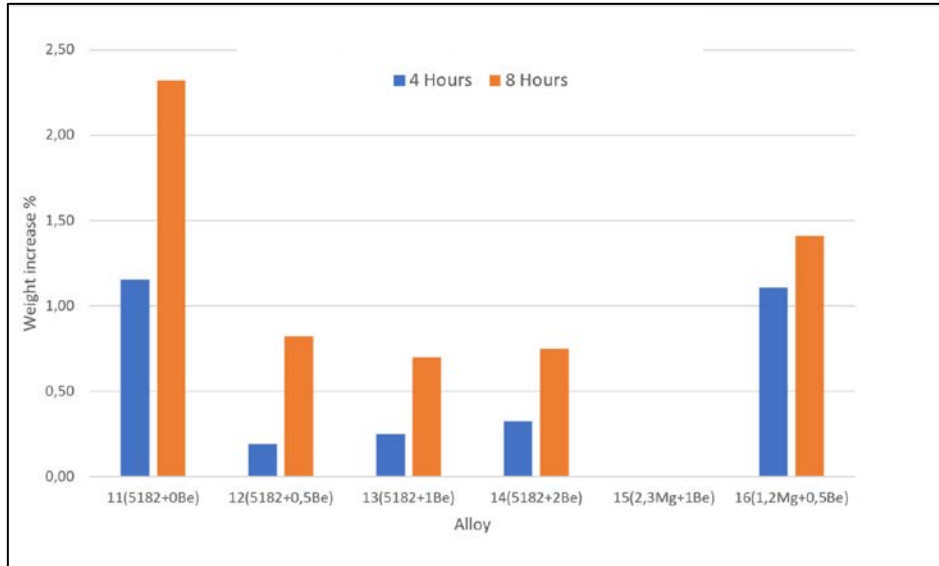


**Figure 4-6: Weight gain six different alloys with varying Be-contents, after 4 hours with different atmospheres, 2019. The X-axis shows the different alloys and the Y-axis is the weight gain due to oxidation.**



**Figure 4-7: Weight increase, 5182 vs 6082, Air Fuel 4 hours, Ceram C, 23.01.2019 samples used for Seeds in large scale tests. The X-axis is the six different samples of each alloy and the Y-axis is weight gain of each sample due to oxidation.**

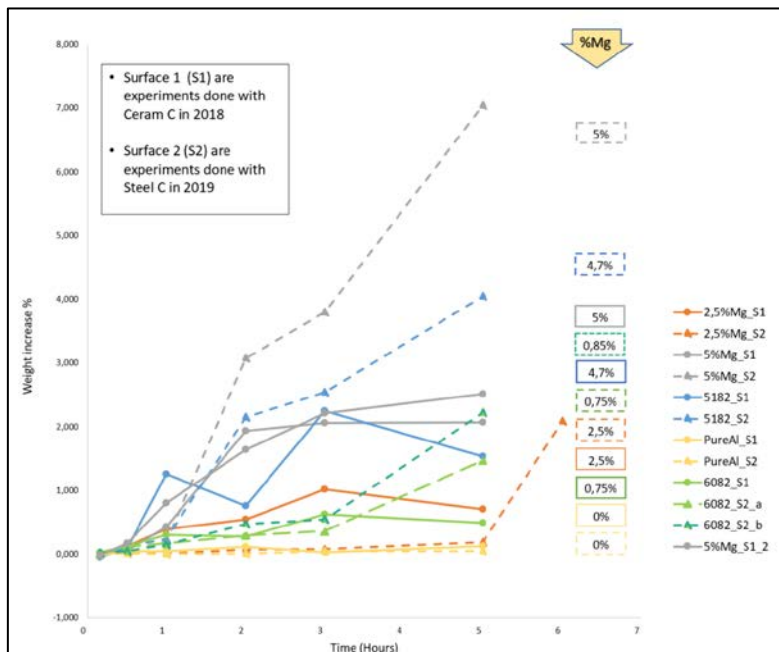
The reason for the data from alloy 15 (Figure 4-8) is missing is because of spilling of metal in the 8-hour experiment and due to dissolution in the crucible in the 4-hour experiment. To see which alloy is which see Figure 3-4.



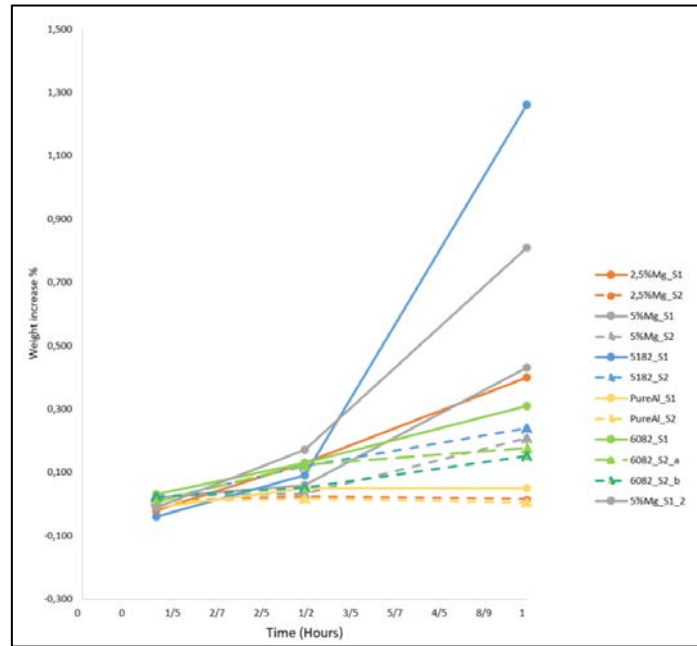
**Figure 4-8: Be samples, 4 hours vs 8 hours, Oxy fuel + leakage, 2019.**

### 4.3 Surface area

As the two crucibles were different in shape and surface area it was investigated if and how much of an effect these changes would have on the oxidation. Comparing the results between the two crucibles could be seen in Figure 4-9 and Figure 4-10.

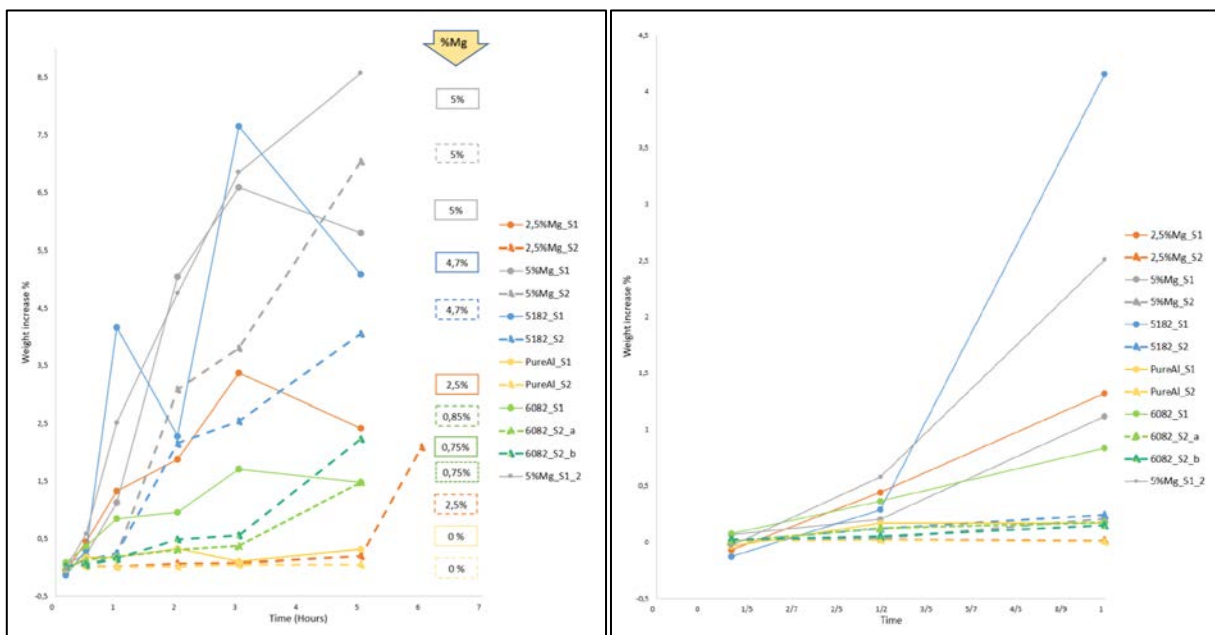


**Figure 4-9: Weight gain Al-Mg samples (No Be) 2018 vs 2019. Dotted lines indicate 2019 samples and the continuous lines indicates the samples from 2018. The sample surface area in 2019 was bigger compared to 2018**



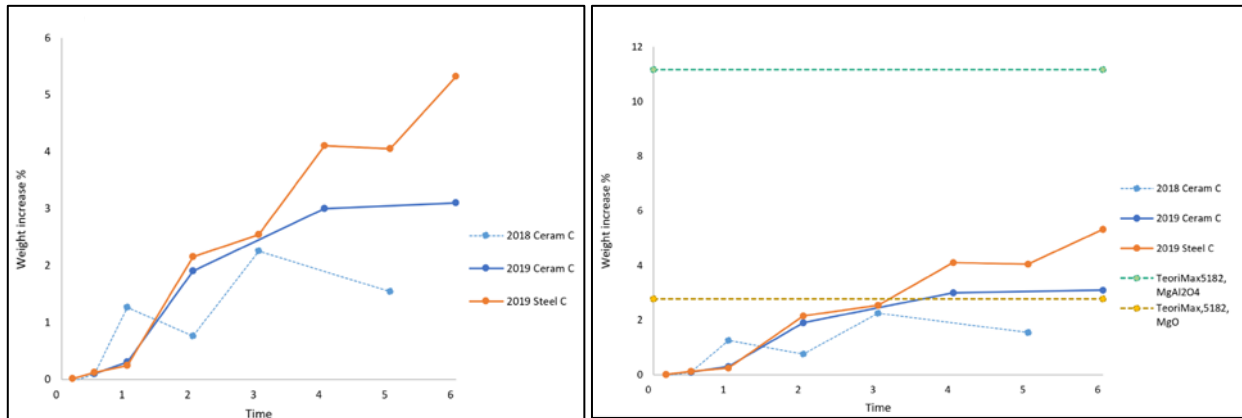
**Figure 4-10: 2018 vs 2019, weight increase, first hour.**

The difference in surface areas between the two crucibles were measured. The surface areas were found to be 6.61 cm<sup>2</sup> for Ceram C and 22.90 cm<sup>2</sup> for steel C. This gave a S2/S1 ratio of 3,47. However, the metal in the Ceram C from 2018 often went over the crucible edge which meant a larger surface area. With the crucible edge S1 was increased to 12,57 cm<sup>2</sup>. This gave a ratio of 1,82. As the factor could vary between 1,82 and 3,47 it was necessary with visual observation and measurements of the surface areas of the different samples. The factor was commonly between 2,7 and 3,4. By taking into account the surface factor in each sample and then multiplying the S1 with the correct factor Figure 4-11 was made:



**Figure 4-11: Left: Weight increase 2018 vs 2019 with surface factor taken into account. Right: Weight increase 2018 vs 2019 including surface factor first hour.**

As 5182 was used with both Ceramic and Steel crucible in 2019 it was investigated the difference in oxidation between these two samples as they were held at the same conditions. Graphs with theoretical weight increase is also shown in Figure 4-12.



**Figure 4-12: Left: Weight increase two 5182-samples done in 2019 in the same conditions with one of them in Ceram C and the other in Steel C (dotted line is data from 2018). Right: Weight increase 5182, 2019 including theoretical weigh increase given complete oxidation to both MgO and MgAl<sub>2</sub>O<sub>4</sub>**

#### 4.4 Discussion

The results clearly show that the oxidation rate is dependent on the Mg-content. Figure 4-1 and Figure 4-2 shows that Mg content certainly have an effect on oxidation as higher Mg-content gives a higher weight gain. Figure 4-7 also shows that 5182 (4,7%Mg) experience a significantly higher weight gain than 6062 (0,85%Mg). This figure also shows the variety in weight gain within the same alloy type.

When looking at figures involving the high Mg content samples >2,5% (Figure 4-1, Figure 4-2, Figure 4-12) it appears that breakaway oxidation happens relatively quick approximately between 1 and 2 hours. For the other samples there is no obvious point in time for were this happens, which means that breakaway hasn't occurred yet, or won't occur at all, or that the data itself isn't accurate enough to determine breakaway time. When comparing the 2018 and 2019 experiments one could observe that the surface area affects the oxidation. This could also be shown in Figure 4-12 which compares two of the same alloys in 2019 done at the same atmosphere for 6 hours, which shows that the samples in the Steel C experience more oxidation. One interesting result when comparing the experiments done in 2018 and 2019 is when looking at the first hour of oxidation. Even though the Steel C samples experienced more weight gain overall, the samples in the Ceram C samples experienced more oxidation the first hour as can be seen in Figure 4-10 and Figure 4-11(right). The cause of this is unknown, but the setup with Ceram C shape samples in the Steel C crucibles could have influenced this.

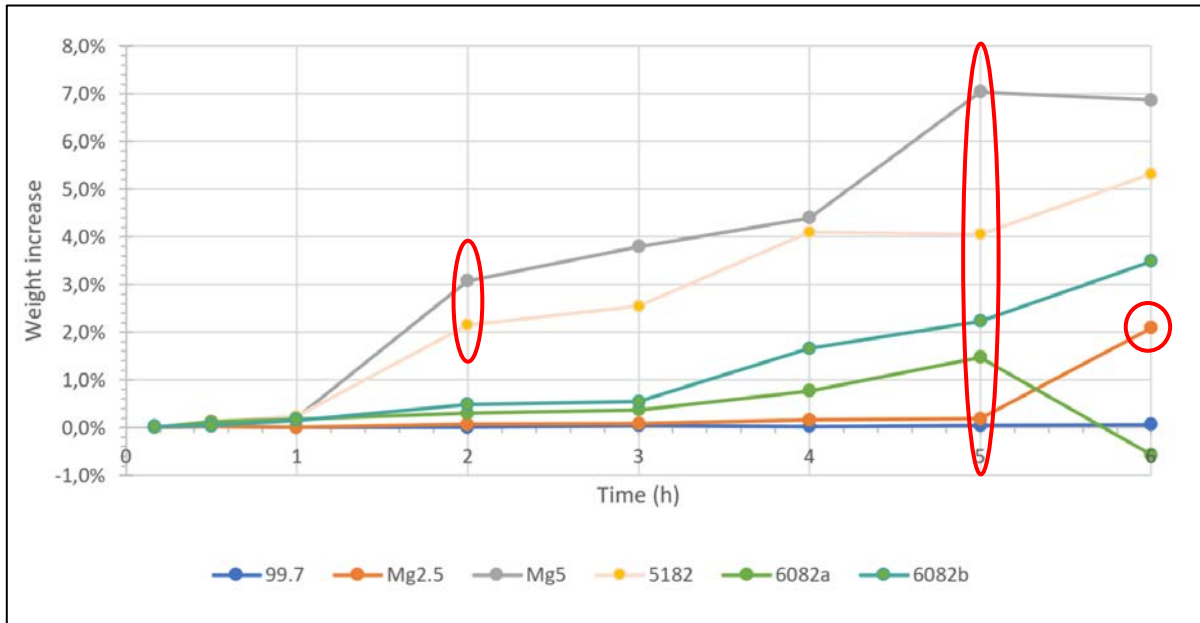
From the experiments regarding atmosphere the atmosphere that stands out is the oxy fuel type atmospheres as these atmospheres show the least amount of weight gain as could be seen in Figure 4-6. Another interesting result worth mentioning is how the atmosphere doesn't seem to have a large effect on the sample containing 1,2 % Mg and 0,5 ppm Be as shown in Figure 4-6. This could be either because the Be-effect dominates or simply because of the low Mg-content. A combination of the two is also a possibility.

The 4-hour vs 8-hour chart in Figure 4-8 shows clearly that the 8-hour samples experience more weight gain which means that the oxidation reactions happens over a timespan of at least over 4 hours, but probably more.

## 5 Material characterization

### 5.1 Samples, method

Some of the samples from the experiment the 21<sup>st</sup> of January 2019 were further analysed in scanning electron microscope (SEM). These samples were from the steel crucible. The samples chosen can be seen in Figure 5-1 and Table 4.

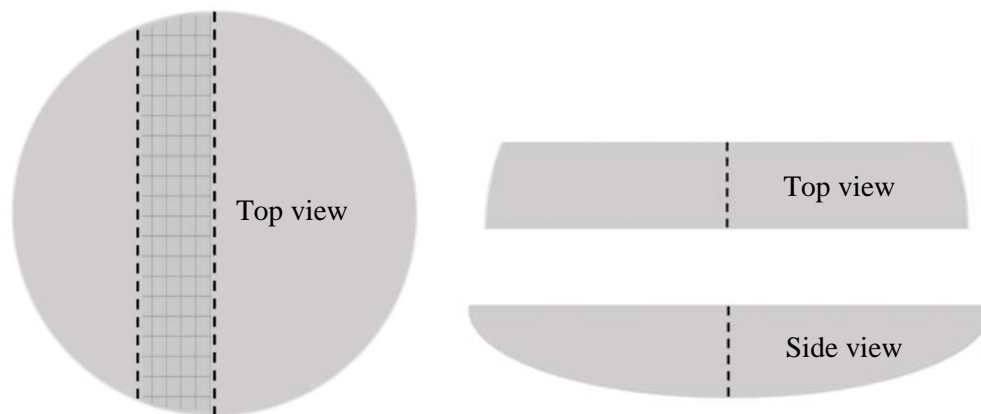


**Figure 5-1: The samples chosen to be looked further into. The samples chosen are indicated by the red circles.**

**Table 4: The samples chosen and their specifications.**

Name	Alloy/Mg-content	Hours in oven, Air Fuel
G1	Pure Al/ 0% Mg	5 Hours
G2	2,5% Mg	5 Hours
G3	5% Mg	5 Hours
G4	5182/ 4,7% Mg	5 Hours
G5	6082a / 0,75% Mg	5 Hours
G7	6082b / 0,85% Mg	5 Hours
H2	2,5% Mg	6 Hours
D3	5% Mg	2 Hours
D4	5182/ 4,7% Mg	2 Hours

These were chosen as it was assumed that the Steel C samples were easier to cut and would be a good start compared to the Be-containing samples. The samples were cut vertically on the middle and a little further out to get the right thickness for the microscope. Due to the samples being too big for the SEM after this step it was necessary to cut them into two pieces each.



**Figure 5-2: Left: How each sample were cut. The marked area corresponds to the part that were further analysed. Right: How the sample had to be cut once more (seen from above and from the side) to be small enough for the SEM and epoxy casting.**

After the preparation of the samples were finished (embedding in epoxy, polishing, drying, carbon coating) the samples were investigated in the SEM. The main focus of the investigation was the oxide layer and the different mechanisms in the formation of the oxide layer of the sample. How Mg was distributed in the material was also a focus point.

The SEM used in all the imaging was a LVFESEM Zeiss Supra and the EDS-detector was a EDAX Octane Pro. The acceleration voltage used in all the images was 10 kV. The EDS was generally used to identify the different phases, and see how the elements were distributed in the sample. The phases identified of interest was Al-bulk, spinel( $\text{MgAl}_2\text{O}_4$ ) and MgO. They were generally identified by using point scans and using the atomic percentage of each element and compare to the stoichiometric equivalent percentage. For example 50% Mg and 50% in at% would indicate MgO. Equivalently 57% O, 29% Al and 14% Mg would indicate spinel phase.

## 5.2 Results

Pictures were taken of all the different samples. Both mapping and imaging was done on each sample, but only the most interesting and important pictures are included in this report.

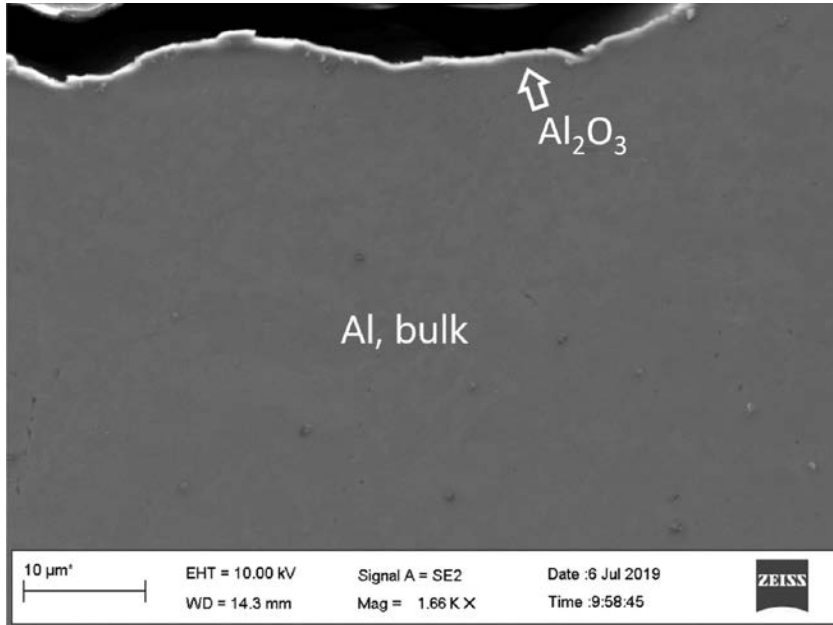
### General

In general, a uniform oxide layer was observed. The oxide layer was found to be the spinel phase in all the samples containing Mg, and the uniform oxide layer was always observed to be at the top closest to the surface. The spinel layer was found to be somewhat inhomogeneous. Small chunks of Al-metal were often “trapped” inside the layer. In addition to these phases two more phases were observed in some of the samples. MgO was found underneath the spinel layer very unevenly distributed. As they could seem like cauliflower this phase is from now on referred to as MgO-cauliflower phase. This phase is similar to the cauliflower like oxide growths seen by Steglich [2] and Cerwinski [3]. Spinel was also observed underneath the uniform spinel layer, and this spinel seemed to form due to different conditions and mechanisms compared to the uniform spinel layer. Due to this, these two are referred to differently. They are distinguished into spinel(L) and spinel(C).



**G1: Pure Al/99,7% for 5h**

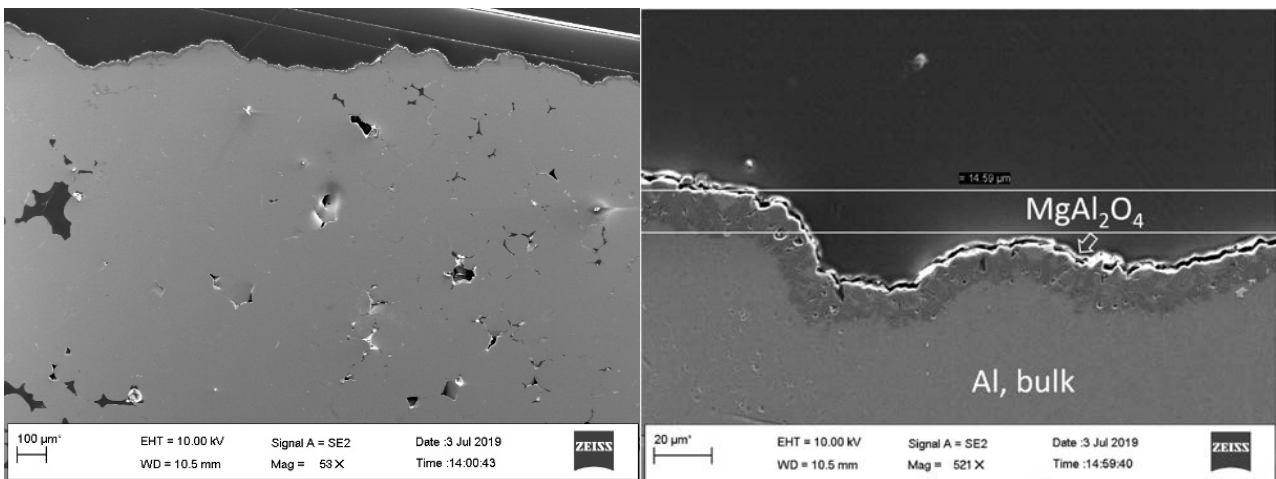
For the Mg-free sample a uniform alumina layer measuring  $< 1\mu\text{m}$  thick was found on the top surface. The sample and the alumina layer can be seen in Figure 5-3.



**Figure 5-3:** The pure aluminium sample showing only an  $\text{Al}_2\text{O}_3$  oxide layer on the surface.

**G2: 2,5%Mg for 5h**

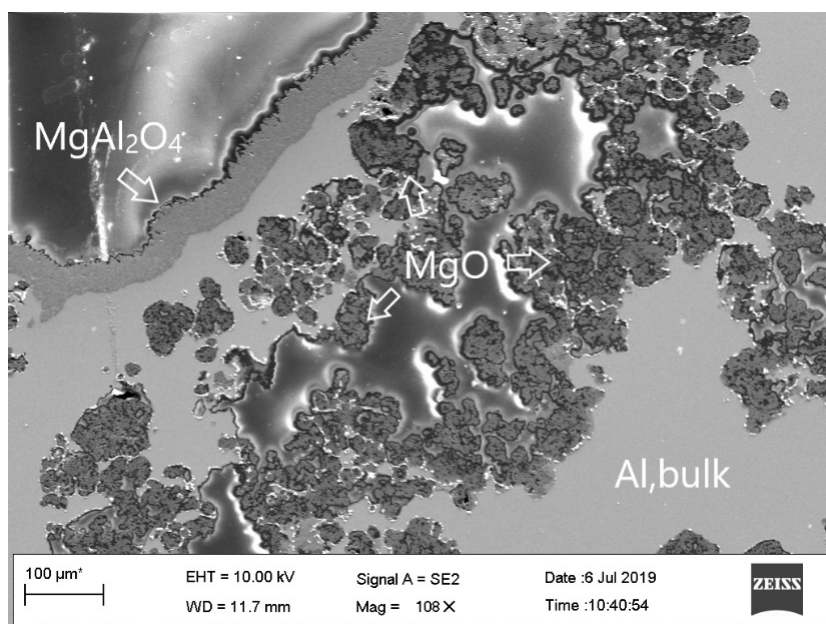
For the sample containing 2,5% Mg held for 5 hours, an oxide layer with uniform thickness was observed at the surface. The oxide was examined and found to be the spinel phase  $\text{MgAl}_2\text{O}_4$  (Figure 5-4, right). This was shown by using the EDS as explained previously. Except from this layer no other oxidation phases were observed. An overview of the sample can be seen in Figure 5-4 (left).



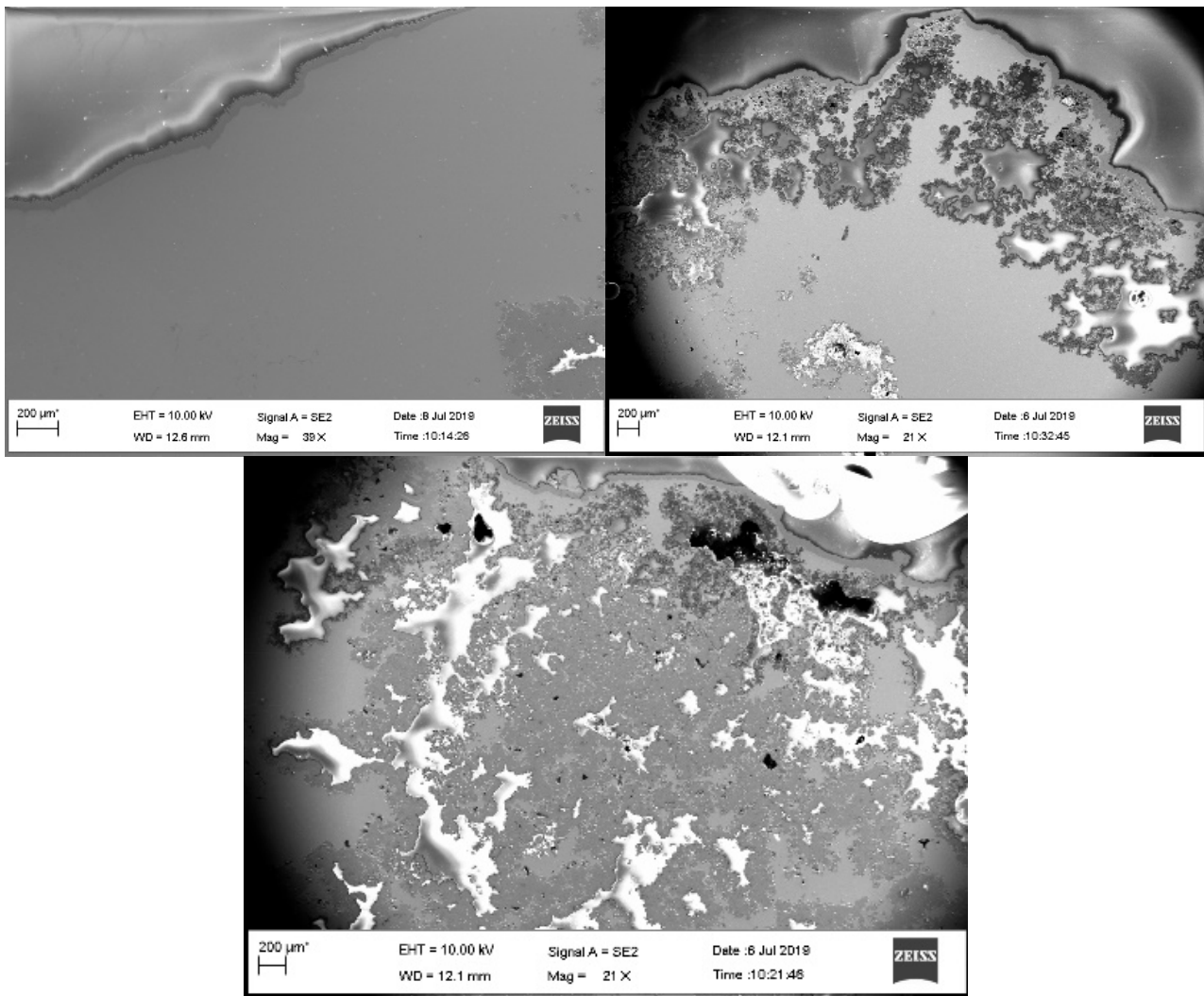
**Figure 5-4:** Left: Overview of the sample. Right: Close up at the surface where the spinel layer can be observed.

### G3: 5% Mg for 5h

The sample containing 5% Mg held for 5 hours gave some interesting results. In this sample both spinel  $MgAl_2O_4$  and  $MgO$  were observed. The dense layer at the surface was the spinel phase, and the  $MgO$  phase was found underneath this layer relatively close to the surface as seen in Figure 5-5. This phase was observed to be granular cauliflower like as the phase wasn't evenly distributed, but in large clusters. Large pores and holes were often found next to the phase. In addition to these observations the spinel phase was also found underneath the evenly distributed spinel layer as well in approximately the same interval as the  $MgO$  phase was found (Figure 5-6, bottom). It should be noted that many places in the sample only the spinel layer was found and nothing more underneath except bulk Aluminium. This build upon the fact that the  $MgO$  and spinel phases underneath the spinel layer wasn't evenly distributed, but in clusters randomly distributed close to the surface. This variation of how the sample looked underneath the spinel layer can be seen in Figure 5-6. Point scans done in the aluminium bulk close to the oxide phases gave a Mg-content around 2-3%.



**Figure 5-5: The surface of the sample with evenly distributed spinel layer with  $MgO$  cauliflower phase underneath.**

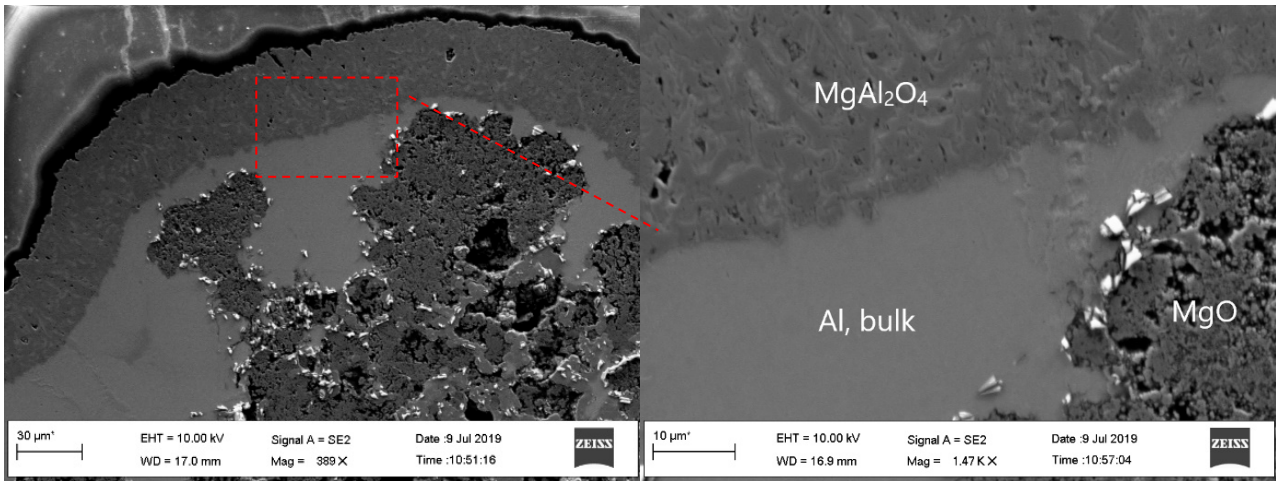


**Figure 5-6: Left: Mostly Al-metal underneath the spinel layer. Right: MgO formation under the spinel layer. Bottom: Substantial spinel(C) formation.**

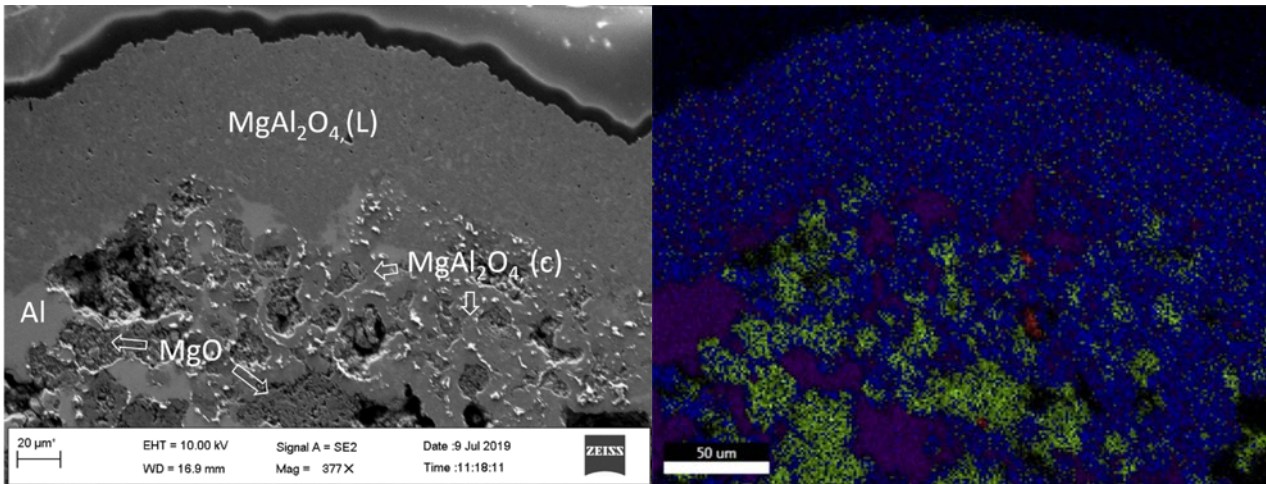
#### **G4: 5182/4,7%Mg for 5h**

The alloy 5182(4,7% Mg) held at 5 hours also gave interesting results, with many similarities to sample G3 (5% Mg), as seen by comparing Figure 5-5 and Figure 5-7. In Figure 5-8 it can be observed spinel phase surrounding the MgO cauliflower phase. This observation may indicate that this spinel(C) phase has grown on the MgO and that the MgO is starting to transform. This transformation from MgO cauliflower phase to spinel(C) phase could be the reason why some places it is observed MgO cauliflower phase underneath the spinel layer and at other points spinel randomly distributed underneath the spinel oxide layer. It should be noted that spinel(C) by itself without any MgO nearby was mainly observed in G3. In G4 the spinel(C) was usually found next to MgO. This could be explained by the MgO to spinel(C) transformation was fully finished some places in the G3 sample. The spinel(L) and the spinel(C) phase could be distinguished as it seems that the spinel(C) phase is formed at a much later stage in the oxidation process. Even though the spinel(C) seems to form around the MgO it is mainly occurring around the MgO closest to the surface in G4.

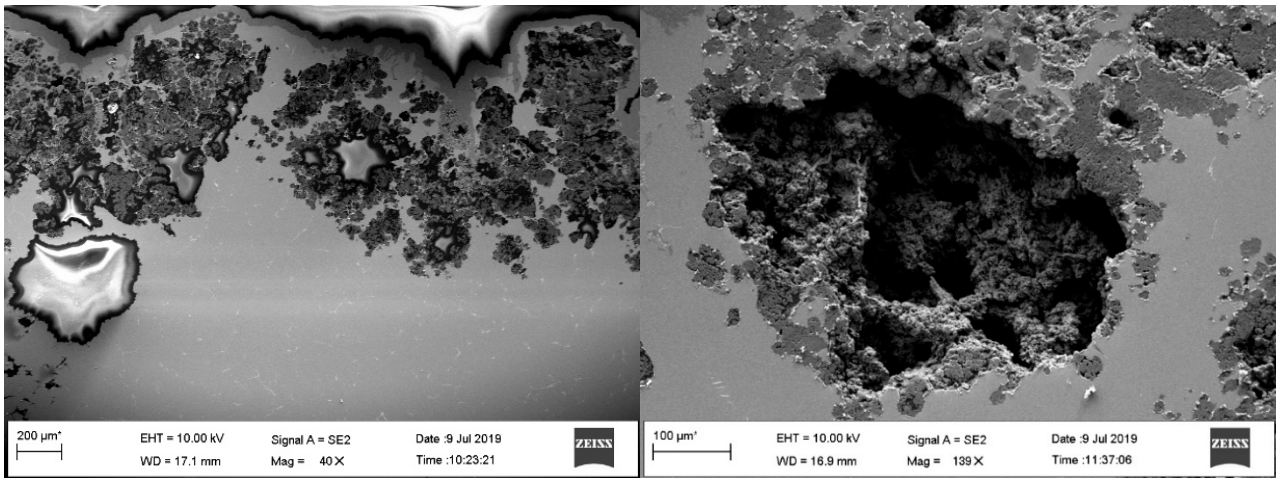
It is worth mentioning is that even though G3 and G4 were very similar it seemed like more spinel(C) was formed in G3. It also seemed like, from the observations made, that more MgO cauliflower phase was found in G4. Additional images on MgO cauliflower-phase can be seen in Figure 5-9.



**Figure 5-7: Left: Image of the spinel layer with MgO cauliflower phase formed underneath. Right: Zoomed in picture of the interfaces between the phases.**



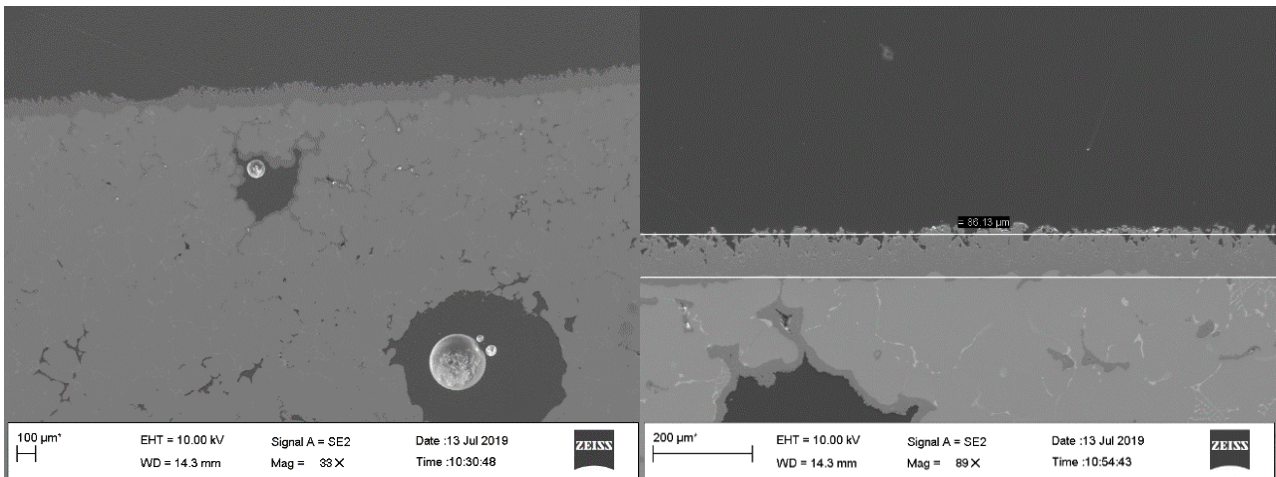
**Figure 5-8: Left: Picture showing the different phases present. The different spinel phases are the spinel(L) which is the oxide layer and the spinel (c) which is the spinel growing around the MgO-phase. Right: EDS mapping of the image, Blue = O, Green = Mg, Purple = Al.**



**Figure 5-9:** Left: Cauliflower MgO formation with large pores. Right: One large pore with MgO clusters around it, arguably formed by MgO formation.

**G5: 6082 (0,75% Mg) for 5h**

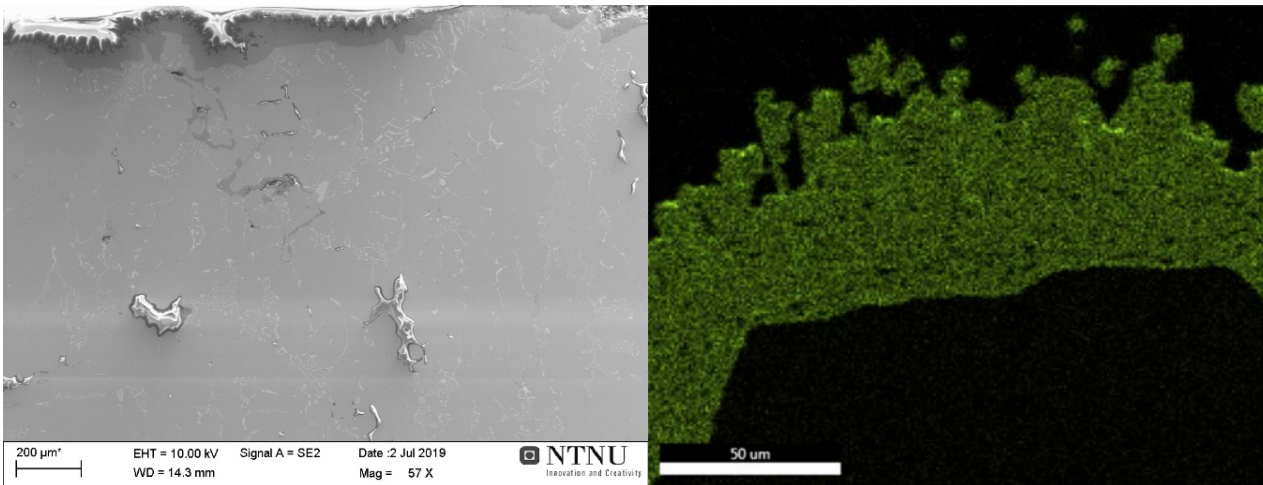
In the 6082 containing 0,75% Mg held for 5 hours the most interesting observation found was that the spinel layer was very thick considered that it only contained 0,75% Mg in comparison to the other samples(see Figure 5-10). The layer was mostly between 60 and 80 µm thick. No MgO-cauliflower formation or spinel(C) was found.



**Figure 5-10:** Left: Overview of the sample close to the surface. Right: A closer look into the oxide layer with measurement of the layers thickness.

**G7: 0,85% Mg for 5h**

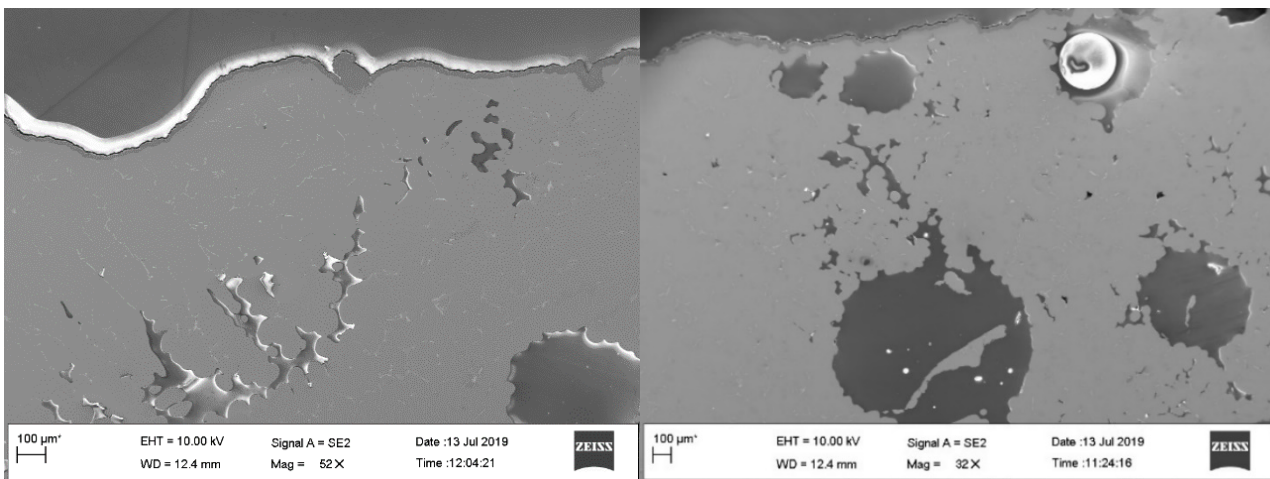
In the 6082-sample containing 0,85% the observations made was in many ways very similar to G5. Even though the oxide layer measured was thick regarding the low Mg-content it wasn't as thick as the layer found in G5. It generally was measured to be between 30-45 µm thick. As in G5 no spinel(C) or MgO were found. An overview of the sample and EDS mapping on the surface can be seen in Figure 5-11.



**Figure 5-11: Left: Overview of the G7-sample close to the surface. Right: EDS mapping of the spinel oxide layer showing Mg-distribution (green = Mg).**

**H2: 2,5% Mg for 6h**

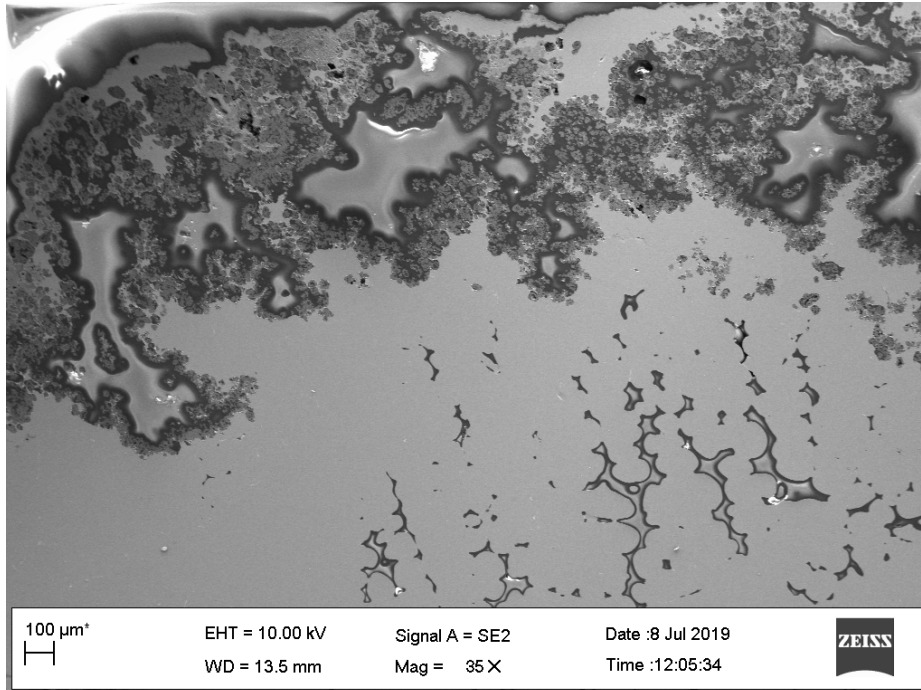
For the sample containing 2,5% Mg that was held for 6 hours no MgO cauliflower formation was observed (Figure 5-12). A spinel oxide layer was formed on this sample as well, but it was relatively thin compared to other samples such as G3 and G5. It was observed to be between 16 and 32  $\mu\text{m}$  in the majority of the sample. To try to find out where the Mg in the sample was located multiple point scans were taken at different depths from the surface. The results showed that the majority of Mg still was in the bulk, but it also indicated that the bulk had an increased Mg content at around 3,4% the first few millimeters from the oxide layer. The sudden increase in weight that was measured in the weighing doesn't seem to correlate with the observed sample. The oxide layer itself had increased a significant amount from G2, but this is not enough to justify the measured increase.



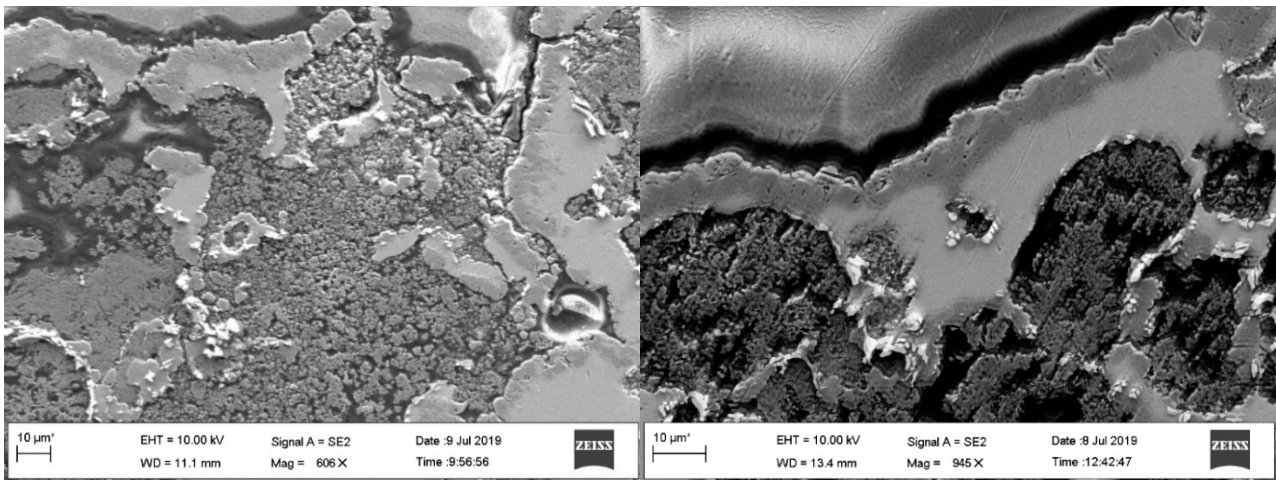
**Figure 5-12: Left: Overview of the H2 sample. Right: Another overview of the sample**

**D3: 5% Mg for 2h**

The 5 % Mg sample held for 2 hours showed that the MgO cauliflower formation already had been formed at this point in time (Figure 5-13). The spinel oxide layer was relatively thin (Figure 5-14), and no substantial spinel(C)-phase was observed even though some was observed which could indicate that the MgO to spinel reaction had started to occur some places in the sample already after 2 hours.



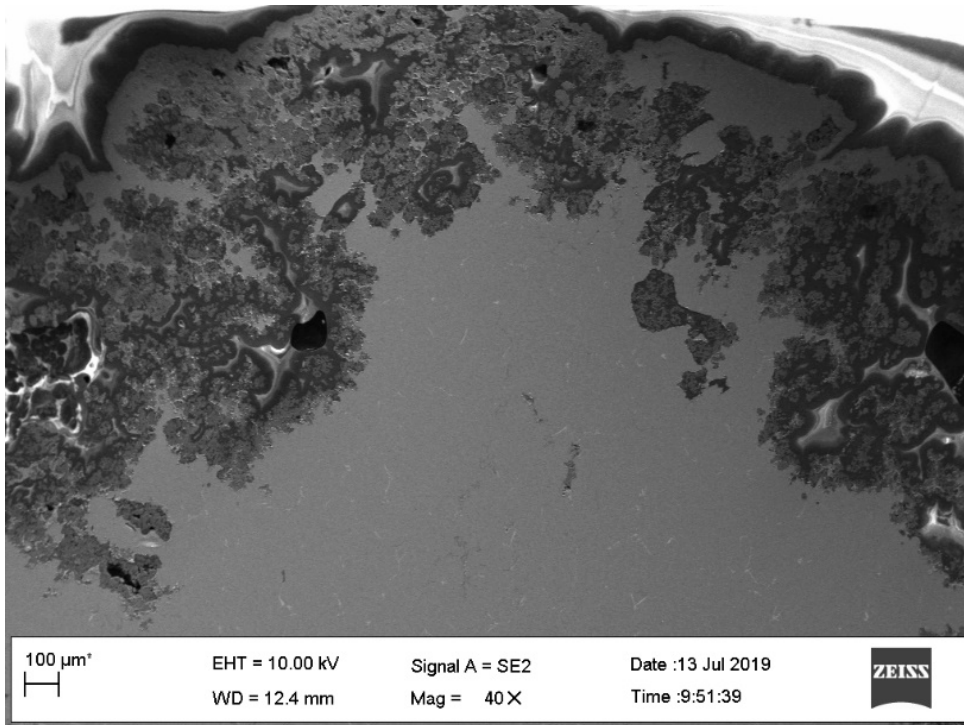
**Figure 5-13: Overview showing MgO-cauliflower formation close to the surface**



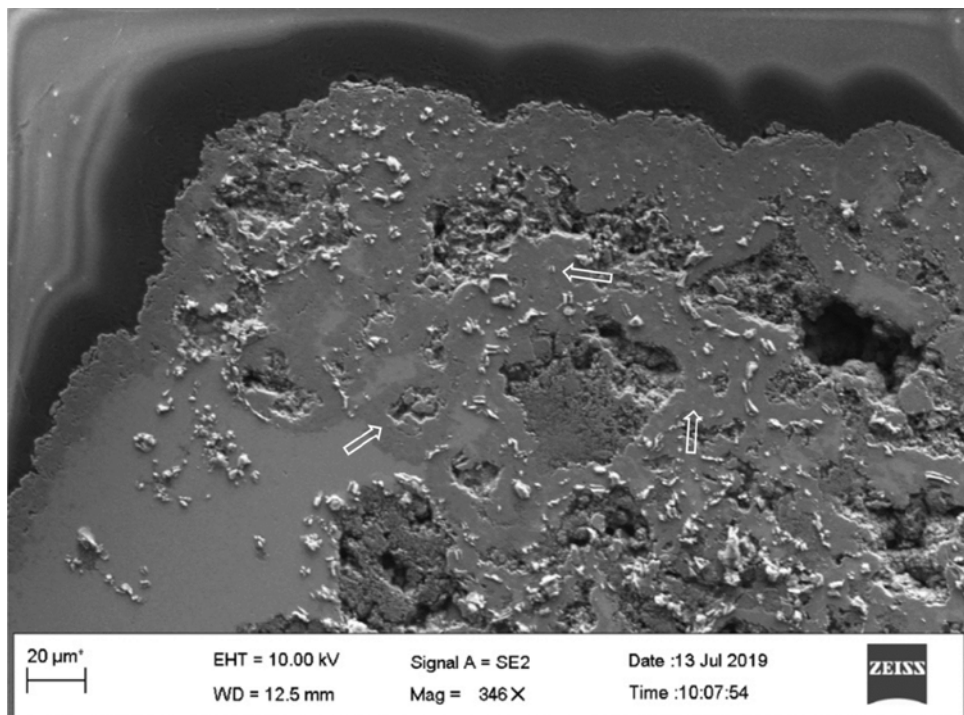
**Figure 5-14: Closer look near the surface, with spinel layer and MgO-cauliflower underneath.**

**D4: 5182/4,7%Mg for 2h**

The 5182-sample held for 2 hours was similar to the 5% held for 2 hours. MgO-cauliflower was observed near the surface across the whole sample and the spinel layer was very thin (about 8 μm). Some spinel(C) was observed as well, but very little compared to the same samples held for 5 hours. It should also be noted that the spinel(C) that was observed was observed next to the MgO cauliflower phase closest to the surface. This can be seen in Figure 5-16 and Figure 5-17 were Figure 5-16 is right next to the surface and Figure 5-17 is a little bit further down from the surface. An overview of the sample can be observed in Figure 5-15.

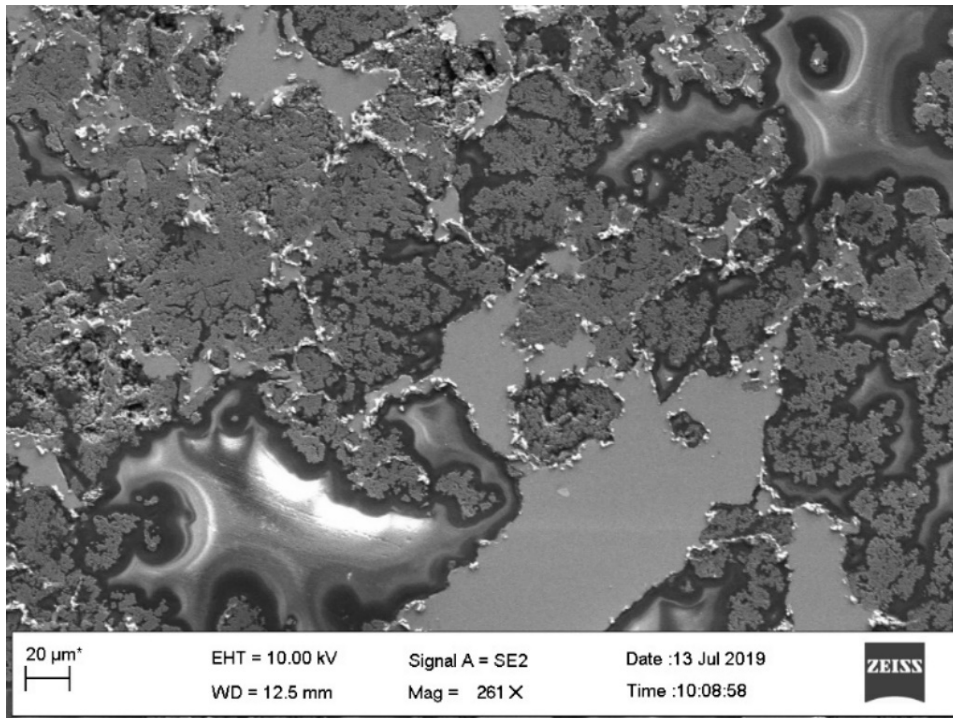


**Figure 5-15: Overview of the sample close to the surface. The MgO cauliflower phase is observed under the spinel layer at the surface.**



**Figure 5-16: MgO cauliflower phase close to the surface with spinel(C) starting to form (see arrows for spinel).**





**Figure 5-17: MgO cauliflower phase further down, about 500 μm from surface with no spinel(C) observed**

**Spinel layer thickness**

In all the samples the spinel oxide layer thickness was measured and is given in Table 5. Figure 5-4 (right) shows how it was done in each sample. In addition to this multiple point to point measures was done in a few of the samples for more data.

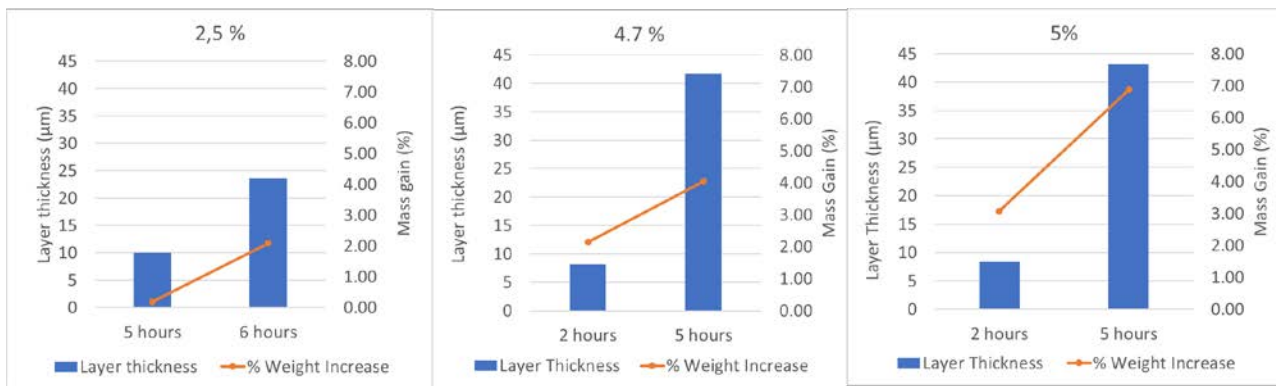
It should be noted that these measurements are taken from only a few images in the sample, so the data found might not be 100% representative in all the samples.

**Table 5 Measured thickness of the uniform spinel layer including average, median and standard deviation of the data gathered. The dimmed standard deviations are values based on few measurements.**

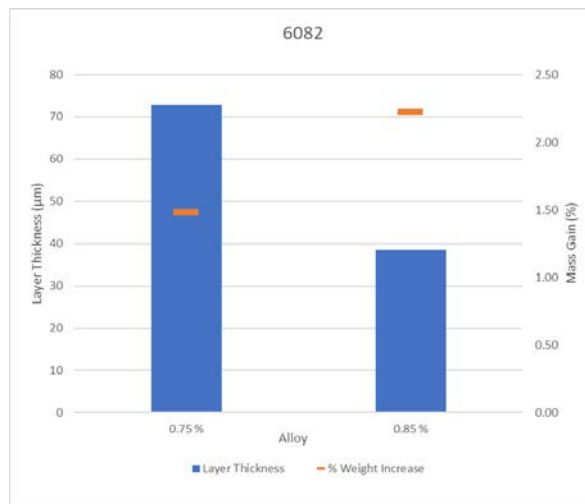
Sample name	Mg-content	Time in oven	MgAl <sub>2</sub> O <sub>4</sub> -layer average thickness, μm	Standard deviation	Mass Gain %
G1	0 %	5 hours	-	-	0.05
G5	0,75 %	5 hours	72,9	6,6	1.48
G7	0,85 %	5 hours	38,5	7,8	2.22
G2	2,5 %	5 hours	10,0	3,3	0.19
H2	2,5%	6 hours	23,6	5,7	2.09
D4	4,7 %	2 hours	8,2	2,1	2.15
D3	5 %	2 hours	8,3	0,4	3.08
G4	4,7 %	5 hours	41,7	14,1	4.05
G3	5 %	5 hours	43,2	8,6	7.04

Plotting the measured  $MgAl_2O_4$  layer thickness and % mass gain together gives Figure 5-18 and Figure 5-19. Here it can be seen that the 2.5 % Mg alloy and the high Mg alloys show a different behaviour as for the 2.5 % alloy a doubling of the oxide layer gives 10 fold increase in the mass gain. Whereas, the 4.7 and 5 % alloys show that an 8 fold increase in layer thickness only gives a doubling of the mass gain. This would seem to confirm that result that the oxidation mechanism for the 2.5 % alloy was different than the 4.7 and 5 % alloy. It is interesting to note that while the 4.7 and 5 % alloy had a similar  $MgAl_2O_4$  layer thickness, but the 5 % alloy had nearly double the oxidation mass gain at both 2 and 5 hours.

For the 6082 alloy only one time was investigated here it could be seen that the  $MgAl_2O_4$  layer and mass gain do not correlate as the thicker layer has a lower mass gain after 5 hours. This would indicate that there is a significant variation in the oxide layer of the samples with a lower Mg content.



**Figure 5-18 Spinel layer thickness versus percent mass gain**



**Figure 5-19 Spinel layer thickness versus % weight increase for the 2 6082 alloys investigated.**

### 5.3 Discussion

The results obtained are in many ways different compared to previous work on similar subjects. The most interesting results differentiating from previous work could be the spinel layer and where the  $MgO$ -cauliflower phase formed. Previous work from such as Cochran [4] has concluded that the oxide layer formed consisted of  $MgO$  before breakaway. From the observations gathered in this work it shows clearly that the oxide layer is  $MgAl_2O_4$ -spinel even at short times. As the earliest sample looked into was after 2 hours it can be a possibility that a  $MgO$  layer existed before this and formed into spinel between 0 and 2 hours. The other thing mentioned was where the  $MgO$  cauliflower phase formed. In previous work from Smith [5], the  $MgO$  phase

have been found to form on top of the layer and therefore reaching the surface. In this work the MgO cauliflower phase was exclusively found to be located underneath the spinel layer. This is a very interesting observation and it could indicate that O-diffusion occurs faster than the Mg-diffusion outwards resulting in internal oxidation.

The images taken also shows a clear distinction between the samples containing  $> 2,5\%$  Mg (4,7% and 5%) and the low Mg samples. The MgO doesn't seem to form in the samples with Mg-content  $\leq 2,5\%$  because its thermodynamically impossible, something previously discussed by Surla [6]. From the results it also seems that the MgO formation is important regarding breakaway oxidation as it leads to what seems like a very high weight increase rate.

When comparing the results from the SEM to the weight measurements they seem to correlate quite well. 2,5% was measured to have very low weight gain overall and from the images taken this makes sense as the layer was very thin and no MgO or spinel(C) had formed. The G5 and G7 weight increase could be explained by their thick oxide layer, but as G5 had the thicker spinel layer and was measured to have less weight gain than G7 the data doesn't seem to correlate perfectly with the images taken. This could be due to the fact that only a small portion of the total oxide layer was examined in the SEM.

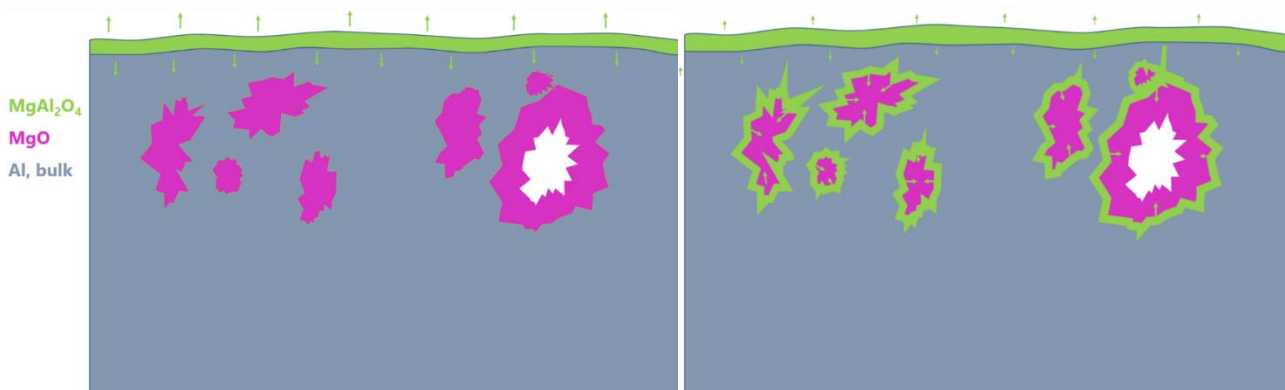
Overall the G5 sample had a spinel layer that was thicker compared to all the other samples. It could be that the mechanism behind the formation of the spinel layer was promoted by the low Mg-content and that's why it formed.

In many ways it seems like the lower Mg-content leads to more spinel-layer formation and that as long as you have Mg content under a specific level no MgO will form. This could explain why 2,5% Mg experienced the least amount of oxidation overall at least to a certain point. Even though this could explain the thicker layer in G5 and G7 it still doesn't explain the thickness of the samples containing  $> 2,5\%$  Mg, but it could seem like other mechanisms also play a significant role in this regime

The sudden increase in the high content Mg samples between 1 and 2 hours that was assumed to be breakaway oxidation might as well be the formation of MgO-cauliflower phase. This is very likely the case as it was observed in the 2-hour samples. The MgO cauliflower phase' uneven distribution could be the thing causing the unpredictable surfaces seen macroscopically in the samples.

The spinel-layer formation doesn't seem like the biggest problem at all, as it forms in all the Mg containing samples and it doesn't lead to such a massive weight gain as the MgO phase seem to do. The layer grows in what seems to be linear in respect to time and is uniformly distributed on the surface. From the samples investigated the growth itself doesn't seem to be quick unlike the MgO growth which seem very quick and disruptive. The amount of oxidation the MgO cauliflower contributes to, especially when it transforms into spinel(C) is significantly higher than the spinel layer. This builds upon the assumption that it is the formation of MgO is the breakaway oxidation.

The mechanism involving the transition from MgO-cauliflower to spinel(C) is also a thing worth discussing. As observed the spinel(L) and spinel(C) didn't seem to grow at the same time. In the samples where MgO formed spinel was found to grow "around" it in some places in the samples often near the MgO closest to the surface. A proposed mechanism of how it forms can be seen in Figure 5-20.



**Figure 5-20: Proposed mechanism for formation of spinel(C). Left: Only MgO and spinel(L). Right: Spinel(C) starts to form around the MgO and grows inwards.**

Regarding the Mg-gradient close to the surface it is very sudden in all the samples as the oxide-layer-metal interface is very distinct. The Mg-content in the bulk seem to be less than original close to the oxide-phases as Mg-has reacted to form the oxide phases. Even though significant oxidation occurred in some of the samples none of the samples had complete Mg consumption as Mg was present in the Al-bulk

## 6 Future Work

Regarding this subject there is certainly future work that could be interesting looking further into. The Be-samples wasn't looked further into as time didn't allow it. This could be worth investigating to see further into the Be-mechanisms preventing oxidation.

Looking further into samples with Mg-content close to the 2,5% could be very interesting looking further into to try to figure out what the limit is for MgO formation if there is any. Looking at these samples in a longer timespan (8+ hours) could be interesting to see if MgO forms or not

Looking at multiple samples of the same alloy over a wide timespan could also be worth going further into to further understand the mechanisms and how the reaction happens over time.

Try to figure out how the spinel layer forms in the early stages of oxidation could be interesting if the spinel layer forms directly or if any MgO layer existed in the early phases. Investigating samples between 0-2 hours could therefore be worth doing. A closer look into the growth of the spinel layer, if it is linear or exponential in respect to time is also something that could be interesting

In future experiments regarding this topic there are a few things worth doing different to get more representative data:

- The crucibles must be made of the same material, at least if comparing the oxidation between the samples done in each of them is of interest. The crucibles being made of two different materials (steel and ceramic material) made the data less trustworthy.
- The atmosphere needs to be more similar if comparing data. In these experiments to different gases was burned which led to different water vapour content which make the data gathered from the experiments less comparable. The gas burned need to be the exact same.
- Having multiple samples done in the same conditions and not one could be worth doing as the data in for example Figure 4-7 showed that there are quite significant variations in samples exposed in the exact same conditions due to what seems like random events.

## 7 September experiments 2019

An additional set of small-scale tests were carried out in September 2019. These tests had two objectives. First was a preliminary investigation into the effects of salt additions on the oxidation rate. Second, was the effects of atmosphere on the oxidation of different alloys.

### 7.1 Atmospheres

The test conditions for the September 2019 experiments are given in Table 6.

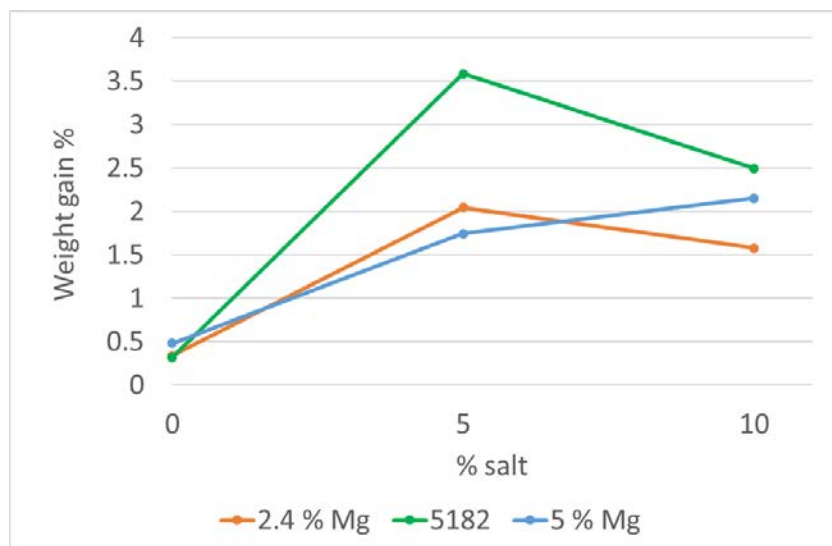
**Table 6: experimental conditions for trial in September 2019**

	Tuesday 17 <sup>th</sup>	Wednesday 18 <sup>th</sup>	Thursday 19 <sup>th</sup>	Friday 20 <sup>th</sup>
<b>Alloys</b>	2.4Mg, 5 Mg, 5182	2.4Mg, 5 Mg, 5182	2.4Mg, 5 Mg, 5182	2.4Mg, 5 Mg, 5182
<b>Atmosphere</b>	10CO <sub>2</sub> , 40H <sub>2</sub> O, 10 O <sub>2</sub> , 40N <sub>2</sub>	10CO <sub>2</sub> , 10H <sub>2</sub> O, 10 O <sub>2</sub> , 70N <sub>2</sub>	16CO <sub>2</sub> , 14H <sub>2</sub> O, 2 O <sub>2</sub> , 68N <sub>2</sub>	38CO <sub>2</sub> , 11H <sub>2</sub> O, 10 O <sub>2</sub> , 40N <sub>2</sub>
<b>Salt</b>	0, 5, 10 %	0, 5, 10 %	None	None

The salt used for the two tests involving salt was A412-2 from Pyrotek. This salt contains 10% Na<sub>3</sub>AlF<sub>6</sub>, 45% KCl and 45% NaCl. The samples used were held in the oven for either 1 or 4 hours.

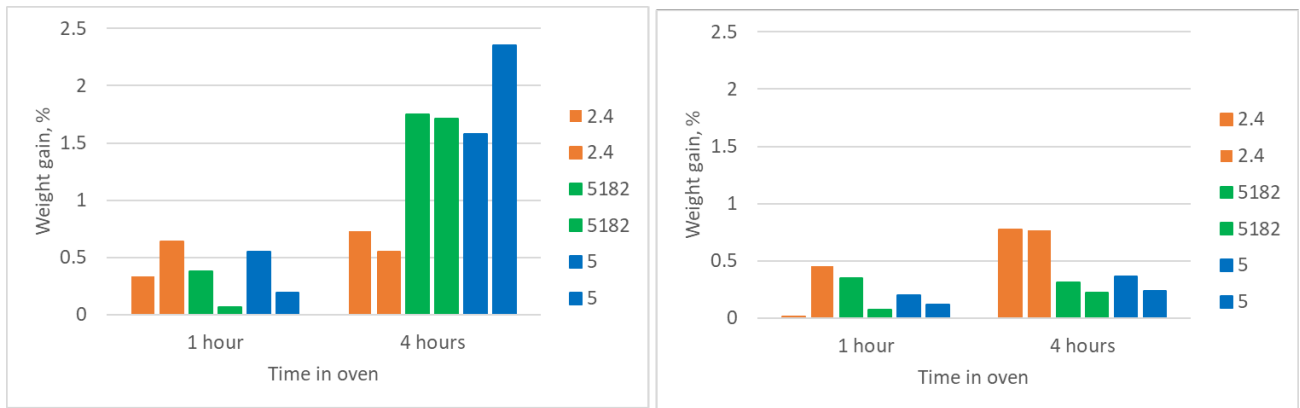
### 7.2 Results

The addition of salt resulted in a notable change in the surface appearance of the samples. Further addition of between 5 and 10 % salt to the samples increased % mass gain due to oxidation as shown in Figure 7-2. No conclusive result on the effects of the amount of salt was found as both 5 and 10 % salt increased the oxidation compared to the salt free samples.



**Figure 7-1: Effects of salt additions on the oxidation mass gain of alloys with 2.4-5 % Mg.**

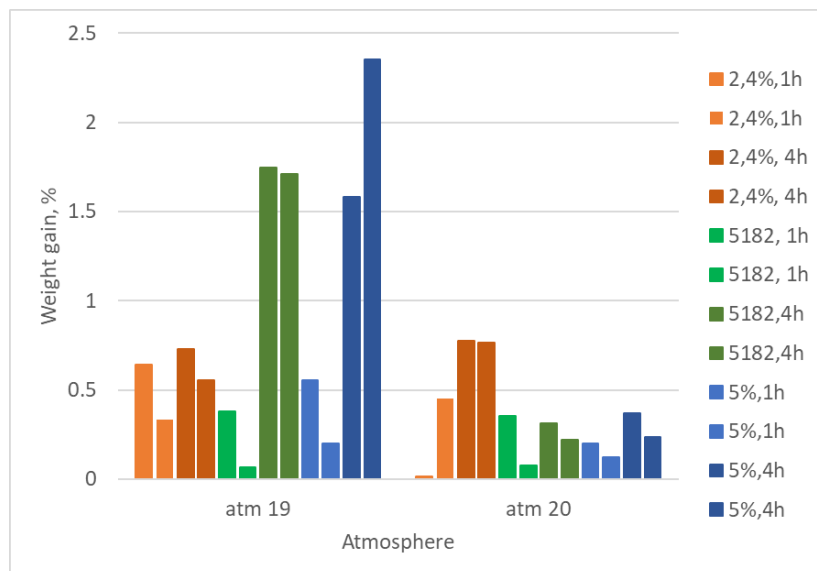
In the salt free samples, the results regarding varying Mg-content was gathered. The results from the 19<sup>th</sup> of September showed a clear connection between Mg-content and oxidation as the higher Mg content samples showed more oxidation as one might expect. However, the results regarding the same thing gotten from the 20<sup>th</sup> of September turned out to be very different. The alloy with 2,4% Mg showed more oxidation than the samples with 4,7% and 5% Mg (see Figure 7-2). It should be noted that the 2,4% samples oxidized almost the same in both experiments. It was the higher Mg-content samples that showed a big difference.



**Figure 7-2: (left) Samples done the 19<sup>th</sup> of September, (right) Samples done the 20<sup>th</sup> of September.**

It was further looked at the difference between the two atmospheres used on the 19<sup>th</sup> and 20<sup>th</sup> of September. A comparison between all samples can be observed in Figure 49.

One of the notable differences between the atmosphere used the 19<sup>th</sup> and 20<sup>th</sup> of September is the CO<sub>2</sub>-content as can be seen in Table 7.



**Figure 7-3: Comparison between the two atmospheres used 19<sup>th</sup> and 20<sup>th</sup> of September regarding oxidation.**

### 7.3 Discussion

Regarding the experiments done in September 2019 the results show that the amount of oxidation changes a lot between the experiments done the 19<sup>th</sup> and 20<sup>th</sup> of September. As the atmospheres that was used were different these days, the probable explanation for this is that the atmospheres played a significant role in the

oxidation-reaction of the samples. The difference between the two atmospheres seemed to affect the higher Mg-samples, but the 2,4% seemed to be almost the same as it oxidized almost the same amount in both cases. An explanation for this could be that the dominating oxidation-mechanism in the oxidation in the 2,4% Mg samples is different from the 4,7% and the 5% Mg samples. This build up upon what was observed in SEM. The atmosphere could therefore be inhibiting the dominating mechanism for oxidation in the 4,7% and 5% Mg samples, but not in the samples with a lower Mg content. Indicating that the reaction of the atmosphere with the Mg is important to inhibit the oxidation.

By comparing the two atmospheres and the results, it could indicate that it is the higher CO<sub>2</sub>-content that inhibits the formation of cauliflower like oxide-phases in the high Mg-samples. However, the formation of the uniform spinel-layer layer was unaffected. This is more speculation and is definitely something that should be investigated further. This should be done by characterisation tools such as SEM or light microscopy.

Regarding salt content, the results showed that salt didn't inhibit oxidation at all, rather the opposite. It should be noted that the salt samples didn't have a lot of data. The dataset was not complete so one can't draw a certain conclusion apart from the one mentioned above.

#### 7.4 Future work

From these experiments there is definitely one thing that should be looked further in to. This is further investigation on samples done with different atmosphere. Looking into the higher Mg-content samples done in different atmospheres is something that should be done in the future. Cauliflower formation especially could be interesting to look at.

When talking about the salt experiments, something that could be done is a better overall experiment with more data and less change in variables so one could see if there is a connection with higher certainty.

## 8 References

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