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## Change in the oxide structure of Al-12Si melts in the presence of Sr

### *Al-12Si alaşımlarında yüzey oksit yapısının Sr varlığında değişimi*

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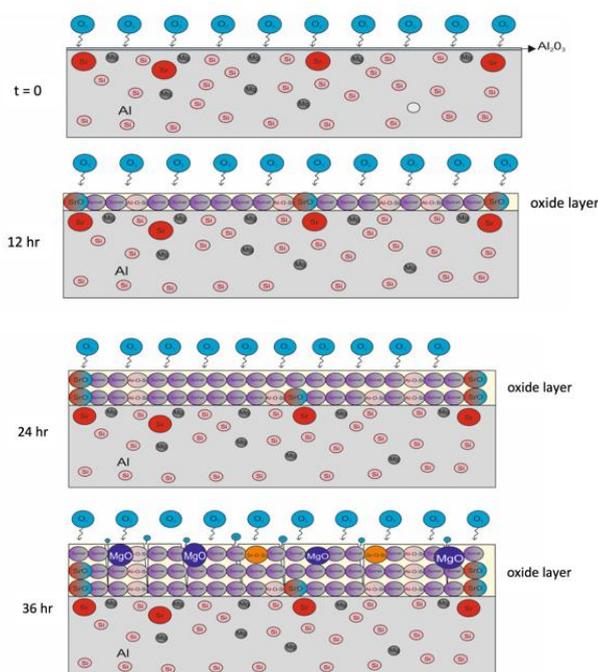
# Change in the Oxide Structure of Al-12Si Melts in the Presence of Sr

## Highlights

- ❖ Dross formation during casting of aluminum alloys is important
- ❖ Oxidation of aluminum changes with alloying elements such as Mg and Sr
- ❖ Sr significantly alters oxide formation
- ❖ Higher the duration of holding time, higher the SrO formation on the surface of the melt

## Graphical Abstract

Effect of Sr on the oxidation of Al-12Si alloy



**Figure.** Schematically illustration of the oxidation process by duration in presence of Sr in Al alloys

## Aim

Characterise the formation of dross structure on the surface of aluminum melts.

## Design & Methodology

Different ratios of Sr was added to Al12Si and holding time of melt was changed where the structure was analysed by XPS method.

## Originality

The oxidation of melt surface in Sr-containing Al alloys has been an important point but not characterised in detail by XPS. This work aims to add a comment to this gap in literature.

## Findings

Sr significantly alters the oxide structure of Al-Si melts.

## Conclusion

Complex SrO containing oxides are formed on the surface of Al melts during casting operation.

## Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

# Change in the Oxide Structure of Al-12Si Melts in the Presence of Sr

## Research Article

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## ABSTRACT

Bifilm defects are the surface entrained double oxide films that are introduced into melts during turbulence and disturbance to the surface of the melt. These defects lead to porosity formation if they are not cleaned from the melt. Thus, the characterisation of the dross that is formed on the surface of the melt becomes a key parameter. In this work, Sr containing Al12Si alloy was used. For the modification process, 100, 200 and 300 ppm Sr was added to the alloy. The oxidation tests were carried out in liquid state at 700, 750 and 800 °C for holding durations of 12, 24, 36 and 48 hours. The structure was characterised by optical microscopy, Scanning Electron Microscopy, X-Ray Diffraction and X-ray photoelectron spectroscopy techniques. It was found that after 12 hours of holding, spinel oxides were formed on the surface mainly SrO.Al<sub>2</sub>O<sub>3</sub> and MgO.Al<sub>2</sub>O<sub>3</sub>.

**Keywords:** Strontium modification, Al-12Si, oxide films, XPS.

## Al-12Si Alaşımlarında Yüzey Oksit Yapısının Sr Varlığında Değişimi

### ÖZ

Alüminyum alaşımlarının döküm proseslerinde yüzeyden sıvı metale karışan bifilm hataları en önemli hatalardan biri olarak değerlendirilirler. Bu yüzden, ergitme prosesleri sırasında yüzeyde oluşan oksit yapılarının karakterizasyonu çok önemlidir. Bu çalışmanın amacı Sr içeren Al12Si alaşımlarında farklı sıcaklık ve bekleme sürelerinde yüzeyde oluşan oksit yapısının karakterize edilmesidir. 100, 200 ve 300 ppm seviyelerinde Sr ilave edilen Al12Si alaşıma 700, 750 ve 800 °C'lerde 12, 24, 36 ve 48 saat boyunca bekleme sürelerinde oksit yapısındaki değişim incelenmiştir. Optik mikroskop, taramalı elektron mikroskobu, XRD ve XPS analizleri ile yüzeyde oluşan oksitler karakterize edilmiştir. 12 saat bekleme süreleri sonrasında yüzey oksit yapısının SrO.Al<sub>2</sub>O<sub>3</sub> ve MgO.Al<sub>2</sub>O<sub>3</sub> yapılarından oluşmaya başladığı tespit edilmiştir.

**Anahtar Kelimeler:** Sr modifikasyonu, Al12Si alaşımı, oksit oluşumu, XPS.

### 1. INTRODUCTION

In the last decade, it became very important to reduce weights of aircrafts and automobiles because of environmental concerns. Thus, low production costs, perfect castability, significant mechanical features, recyclability makes Al-Si alloys useful for a wide industrial usage. Especially in automobile industries, parts that are exposed to high temperatures are produced from 3xx alloys [1].

In order to achieve higher fluidity and mechanical properties, Al-Si alloys are modified with strontium. However, according to researchers in the area [2-8], there are various discussions on whether strontium generates porosity in those alloys or not. Many scientists claim that strontium modification increases the amount of oxides in the melt and these oxides results in porosity. On the other hand, some scientists claim that strontium modification

has no relation with porosity formation but it decreases surface tension of the liquid which makes it vulnerable to hydrogen diffusion [9].

Liu [10] has pointed out that in A356 alloy which is modified by strontium, AlSrO compounds are found in the porosities. He also showed that there is a direct proportion between strontium amount and porosity.

Iwahori [5] and Serratos [2] have claimed that SrO affects porosity formation however, it is possible to prevent Sr-containing oxide formation by degassing or fluxing the melt.

Garat [11] has investigated the surface oxide structure of Sr modified A356 alloy. He determined that the surface structure was no longer thin and protective (i.e. pure amorphous alumina) but had a thickness of up to 500 micrometres that contained SrO.

Emadi [12] has stated that strontium addition to the A356 alloy increases inclusion quantity. According to their thermogravimetric analyses, strontium increases

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oxidation and it exists in the form of  $\text{SrAl}_4\text{O}_7$ ,  $\text{SrAl}_2\text{O}_4$  and  $\text{Al}_2\text{Si}_2\text{Sr}$  on the surface.

Miresmaelli [9] claims that SrO formation on the surface depends on the weight of alloy and oxidation duration. In the experiment which is done by 47 gr alloy, he stated that strontium has played a significant role in oxidation and SrO has been formed on the surface. However, when 2.5 kg Al-Si alloy was modified with 100 ppm strontium, it had shown no sign of SrO on the surface; only  $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3\cdot\text{MgO}$  spinel structures were found after 300 minutes of oxidation. He explained the situation thermodynamically and said that strontium atoms can't diffuse to the surface in the given time, therefore with the increase in oxidation time SrO can be seen again on the surface. Miresmaelli [4] has claimed in another work that in short oxidation periods  $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3\cdot\text{MgO}$  spinel structures are formed and SrO is not found on the surface. However, it's indicated that  $\text{Al}_2\text{Si}_2\text{Sr}$  intermetallic is seen on the surface and it plays a vital role in porosity formation. After long periods of oxidation surface consists more than 50% of SrO spinel.

Yuen [13] has studied the thermal analysis of A356 with and without Sr modification. It was found that there was no crucial weight gain in the modified sample while in the unmodified alloy, the weight gain was high. On the other hand, Raiszadeh [13-15] has designed a test where a bubble of air was kept floating in the liquid aluminium for several hours and in-situ oxidation of aluminium by the air inside the bubble was examined. It was found that by increased duration, the oxide structure was transformed into complex Sr-containing oxides. Mehrabian [17] shown experimentally how trapped air bubble can result in breakaway oxidation and illustrated this mechanism impeccably. Ozer [18] and Bhagyanathan [19] studied the recycling efficiency of aluminum cans and showed that oxidation during remelting is the key parameter for improved melt cleanliness.

Mohanty [20] stated that SrO is not stable in the aluminum alloy. In his work, synthetic SrO particles were degraded to Sr and after that it was shown that elementary Sr was transformed into  $\text{AlSiSr}$ .

With regard to the discussions in this field, in order to address these issues, a series of oxidation tests with various Sr content in Al12Si alloy was carried out by using XPS analysis as characterisation tool.

## 2. MATERIALS and METHOD

Al-12Si eutectic alloy has been used in experimental studies. The chemical composition of the alloy is given in Table 1.

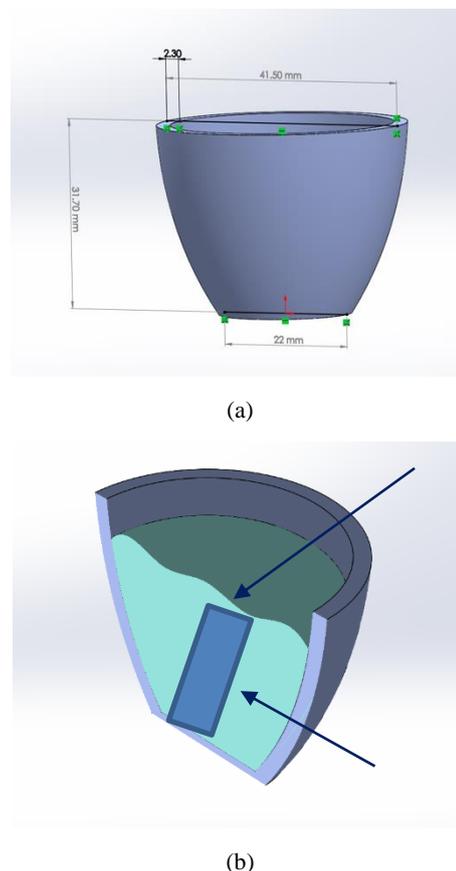
Sr was added to the melt as AlSr10 master alloy, aiming to reach the levels of 100, 200 and 300 ppm Sr. Experiments were carried out in a resistance furnace at 700, 750 and 800 °C temperatures for 12, 24, 36, 48 hours. The samples were placed in alumina crucibles.

After the samples were left to solidify, they were cut in half vertically, grinded and polished with 1-micron alumina. The cross section of the samples was investigated by optical microscopy and SEM (Scanning Electron Microscopy) for microstructural analysis and the surfaces were subjected to XPS (X-ray Photoelectron Spectroscopy) analysis. The test matrix consisted of 36 samples in total. In order to eliminate the display of all sample images, only selected ones were presented in this work.

**Table 1.** Chemical composition of the alloy

Al	Si	Mg	Fe	Cu	Mn	Zn
87.05	11.96	0.06	0.46	0.09	0.29	0.024

The samples were placed in a crucible where the dimension is given in Fig 1 with the location of the sample examination points shown with arrows.



**Figure 1.** (a) crucible used in oxidation studies, (b) sample locations used in examinations

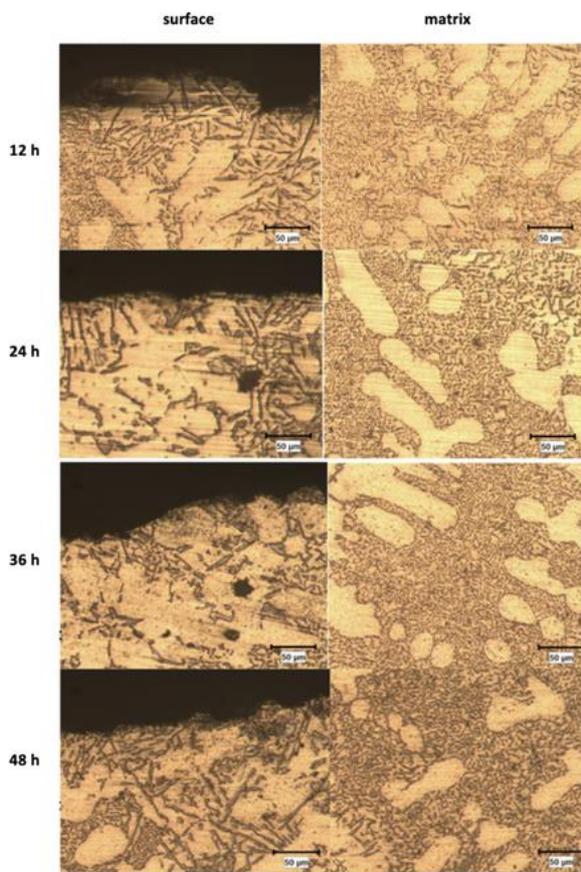
## 3. RESULTS AND DISCUSSION

The optical emission spectroscopy analysis of samples are given in Table 2 which shows the levels of Sr achieved in the alloy.

**Table 2:** Sr content of the samples

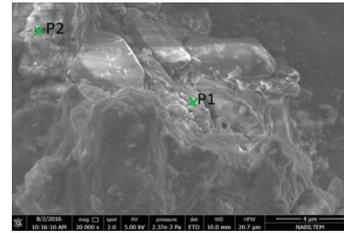
ppm	Al	Si	Mg	Fe	Cu	Mn	Zn	Sr
100	87,65	11,24	0,075	0,507	0,10	0,370	0,026	<b>0,0133</b>
200	86,96	12,00	0,068	0,471	0,09	0,295	0,023	<b>0,0220</b>
300	87,33	11,64	0,065	0,469	0,09	0,300	0,023	<b>0,0302</b>

An example of microstructural change occurred on the samples is given in Figure 2. On the left, the unmodified Si structure can be clearly seen on the surface of the oxidized samples. On the right, fully modified Si eutectic is seen which is taken from the bulk of the sample.



**Figure 2.** Micrographs of 200 ppm Sr modified Al-12Si alloy at 750 °C

As can be seen in Figure 2, Si near the surface became long and coarse compare to the matrix of the samples. SEM image of surface oxide of 200 ppm Sr-Modified Al-12Si alloy after oxidation for 12 h at 750 °C is shown in Fig. 3a. EDS analysis of P1 and P2 are given in Fig 3a and b. It is determined from EDS analysis of the related points that surface oxide includes CaO (P1) and AlSrO (P2) as white particles respectively.



(a)

EDAX ZAF Quantification (Standardless)  
Element Normalized  
SEC Table : Default

Element	Wt %	Element	Wt %
O K	9.41	O K	43.93
MgK	0.69	MgK	3.87
AlK	8.21	AlK	30.04
SiK	1.01	SiK	3.48
SrL	1.43	SrL	16.08
CaK	79.25	CaK	2.60
Total	100.00	Total	100.00

(b)

(c)

**Figure 3:** (a) SEM images (b) EDS result from P1 (c) EDS result from P2 of 200 ppm Sr alloy melted at 750 °C

In Figure 4(a), significant amount of featherlike/fibrous shapes are determined as SrO containing Al<sub>2</sub>O<sub>3</sub>.MgO.SrO complex oxide structures from EDS analysis taken from P1.

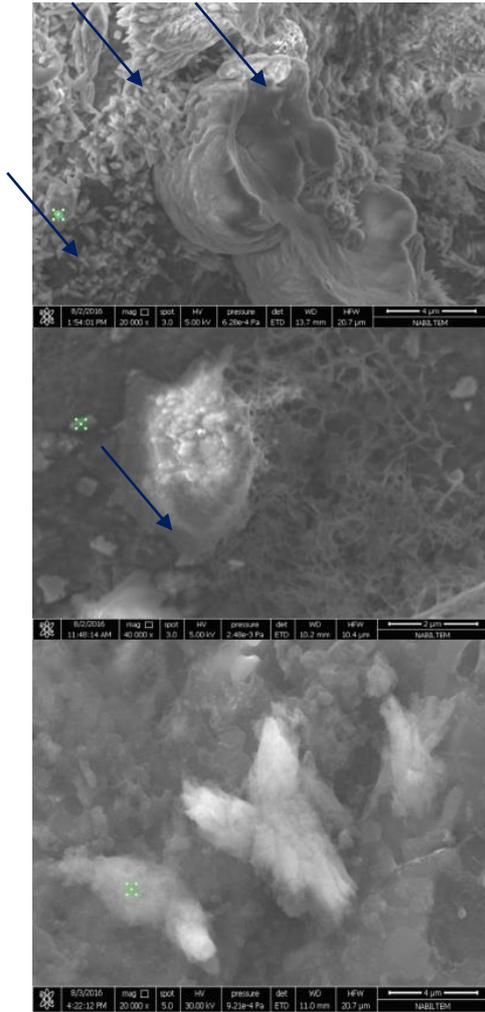


EDAX ZAF Quantification (Standardless)  
Element Normalized  
SEC Table : Default

Element	Wt %
O K	41.30
MgK	5.28
AlK	31.00
SiK	5.75
SrL	14.22
CaK	2.45
Total	100.00

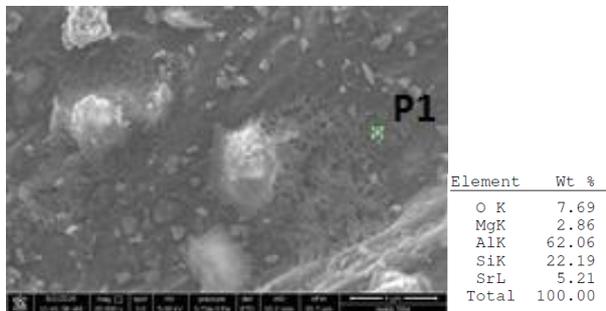
**Figure 4:** (a) SEM image (b) EDS result obtain from Point 1 of 200 ppm Sr modified oxidized at 750 °C for 24 h

SEM images of different compositions of Sr containing samples which exposed to various temperatures of oxidation are given in Fig 5. White particles seen on Fig 4a and Fig 5b are identified as AlSrO compound from EDS analysis. In Fig 5(c) Its determined that structures including SrO are exist in bigger pellets.



**Figure 5:** (a) SEM images of surface oxide after 48 h at 750 °C (b) 200 ppm Sr (c) 300ppm Sr

Miresmaeili [4] claimed that  $Al_2O_3.MgO.SrO$  complex structures may be present on the surface oxide. Additionally, it was mentioned that Si can be found in surface oxide layer as  $Al_2Si_2Sr$  and  $SiO_2$ . Determination of  $SiO_2$  from EDS analysis of P1 (Fig 6) together with  $Al_2O_3.MgO.SrO$  in the surface oxide shows similarities with Miresmaeili’s work.



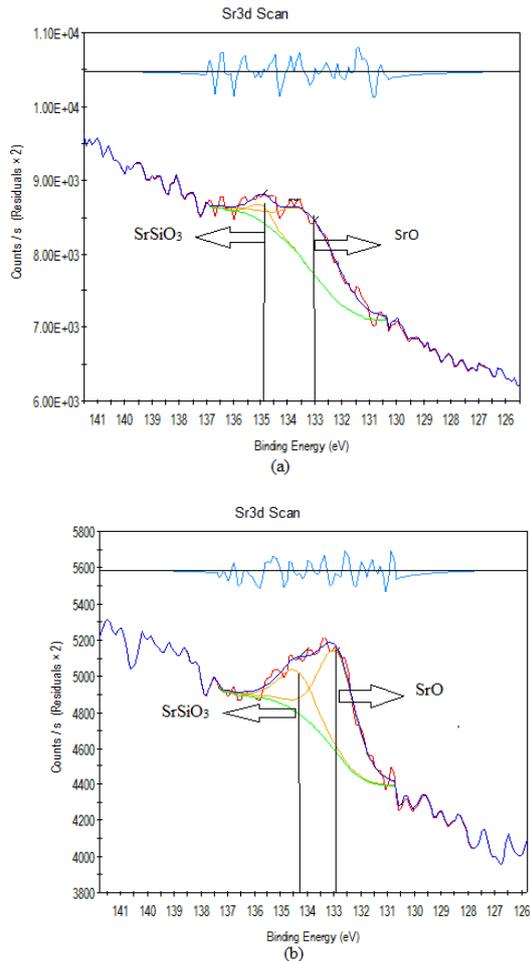
**Figure 6:** SEM images of surface oxide of 200 ppm Sr-Modified Al-12Si alloy melt after 48h oxidation at 750 °C

**Table 3:** Binding energy values of 200 ppm Sr modified oxidised at 750 °C

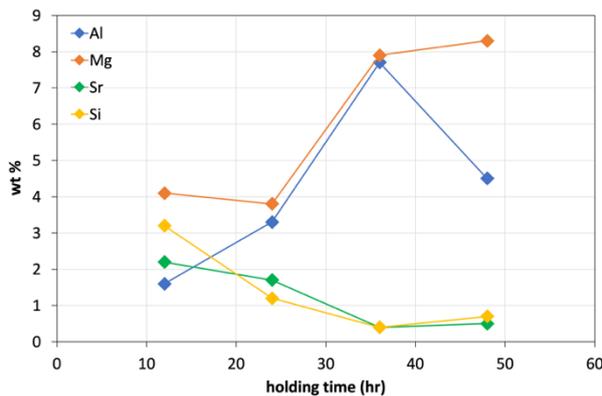
	12 h	24 h	36 h	48 h
Al2p	74,31	74,32	74,4	73,57
		75,72	73,59	74,69
		72,12		74,15
Mg1s	1303,99	1303,76	1303,54	1303,85
				1303,08
O1s	531,5	531,55	531,7	531,31
			532,76	532,27
	532,57		530,15	530,34
			530,88	
Si2p	101,85(+2)	101,98(+3)	101,57(+2)	101,9(+3)
	102,60(+3)			
	101,08(+2)			
Sr3d	133,32	133,33	134,93	132,9
	135,1	135	133,06	134,38

In Table 3, the binding energy of surface oxide of samples produced by 200 ppm Sr which were held for different hours in 750 °C is given.

Sr3d binding energies of 12 and 24 hour oxidised samples are 133,32-135,1 eV and 133,33-135 eV, respectively. This value corresponds to SrO [21]. The BE values of samples which were oxidised for 36 and 48 hours reveal a shift in Sr3d which are 134,93-133,06 eV and 132,9-134,38 eV respectively. The BE values of 102-102.6 eV, 134.2-134.6 eV, 530.7-531.6 eV show that Sr can be in the form of  $SrSiO_3$  which was also reported by Kirsch [22], El Kazzi [23] and Spreitzer [24]. These findings indicate that SrO is stable up to 24 hours; and after longer holding periods, it transforms into  $SrO.SiO_2$  spinel oxide as seen in Figure 7. Eguskiza [25] had found that after 5 hours of holding A356 in liquid state had caused up to 40% of Sr loss from the matrix which had resulted in formation of SrO on the surface. A similar finding was reported by Zhang [26] where it was claimed that 150 minutes was good enough for A356 holding at 750°C for the fading of Sr.



**Figure 7:** XPS diagrams of Sr3d for the 200 pm modified Al12Si oxidized at 750 °C for (a) 38 and (b) 48 hours.



**Figure 8:** Deconvolution of XPS results showing the weight % of oxides

According to SEM-EDS and XPS results, it can be seen that the surface oxide consists of Si and thereby SiO<sub>2</sub> is formed. There are limited number of researches in the literature that reveals the presence of SiO<sub>2</sub>. For example, Miresmailli [9] claims that SiO<sub>2</sub> can be present on the surface based on the EDS analysis due to Si and O peaks. Dennis [27] reported that unidentified weak peaks were

present in XRD analysis together with Al<sub>2</sub>O<sub>3</sub> and MgO peaks. The surface of the oxide structure is so rough and uneven. Also, the thickness of the layer is so thin, therefore these peaks were identified as SrSiO<sub>3</sub> and SrAl<sub>4</sub>O<sub>7</sub> but without certainty. On the other hand, according to the XPS analysis of this work, this uncertainty can be unravelled. In order to observe SiO<sub>2</sub>, Si has to be in the form of Si<sup>+4</sup> where Si2p and O1s reveals 103 and 533 eV. Kirsch [22] and El Kazzi [23] reported that metallic Si has to have 99.5 eV for Si2p. Based on the values found in Table 2, after 12 hours of oxidation, Si2p has 101,85eV, 102,6eV and 101,08eV for Si<sup>+2</sup>, Si<sup>+3</sup> and Si<sup>+2</sup>, respectively. Similarly, after 24 hours, Si2p shows 101,98 eV which corresponds to Si<sup>+3</sup> concluding a non-stoichiometric structure [28]. Thus, up to 24 hours, Si cannot be in the form of SiO<sub>2</sub>. For the oxidation of the alloy above 36 hours, it can be concluded that together with SrO, Si has non-stoichiometric Si<sub>x</sub>O<sub>y</sub> structure which forms a spinel oxide as SrSiO<sub>3</sub>.

After 12 hours of oxidation, BE values of Mg1s is 1303,99 eV and O1s is 531,5 eV. After 24 hours, Mg1s is around 1303,76 eV and O1s is 531,55 eV. Therefore, the observation of O1s in the BE values of 531 eVs, it can be concluded that Al<sub>2</sub>O<sub>3</sub>.MgO spinel oxide is present [29]. After 36 and 48 hours of oxidation, Mg1s values become 1303,54 eV and 1303,85 eV respectively; whereas O1s values are 530,15 eV and 530,34 eV. These values correspond to MgO [30]. Based on these findings, it can be concluded that up to 24 hours of oxidation, Al<sub>2</sub>O<sub>3</sub>.MgO is present, and after 24 hours, MgO becomes the dominant oxide structure on the surface. Jang [31] reported that for the melts containing 4-5 wt% Mg, the stable oxide was transformed into Al<sub>2</sub>O<sub>3</sub>.MgO and MgO which leads to poor melt quality. The deconvolution of XPS results (Figure 8) shows that Mg is more stable on the surface and the remaining oxide structures are present towards the main matrix.

Figure 9 shows the schematical representation of oxidation mechanism. It can be seen that at t=0 time, there is a thin layer of amorphous oxide on the surface. As the oxidation process continues, SrO begins to form on the surface. Since the density of the oxide structure is higher, the breakaway oxidation mechanism takes places and new surface is exposed to oxygen; thus, the oxide layers thickens after 24 hours. As the duration is increased to over 36 hours, Mg also starts to be oxidized, resulting in the formation of complex spinel structures are Al<sub>2</sub>O<sub>3</sub>.MgO and Al<sub>2</sub>O<sub>3</sub>.SrO as well as SrO.SiO<sub>2</sub>.

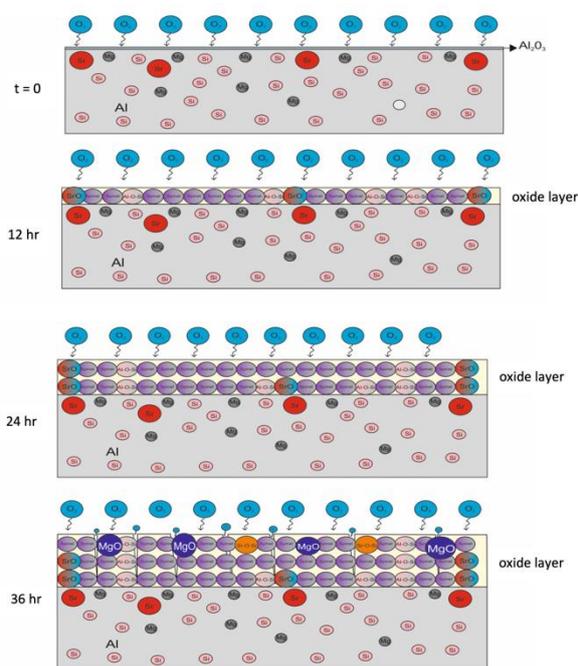
#### 4. CONCLUSION

The oxide structure of Al12Si alloy changes depending on temperature and duration.

24 hours is a critical duration when the alloy is held in the liquid state at 750 °C The aluminium oxide on the surface starts to transform into complex oxide structures.

Complex and spinel compounds like Al<sub>2</sub>O<sub>3</sub>.MgO, Al<sub>2</sub>O<sub>3</sub>.MgO.SrO, SrO.SiO<sub>2</sub>, SrO.Al<sub>2</sub>O<sub>3</sub> are found in the

surface oxides according to results of SEM-EDS and XPS analysis.



**Figure 9:** Schematically illustration of the oxidation process by duration

#### DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

#### AUTHORS' CONTRIBUTIONS

**Cem KAHRUMAN:** Supervision, methodology, investigation.

**Uğur ALEV:** Performed the experiments and analyse the results.

**Derya DISPİNAR:** Writing, review and editing, supervision.

#### CONFLICT OF INTEREST

There is no conflict of interest in this study.

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