Accelerated Corrosion Analysis of AlSi10Mg Alloy Manufactured by Selective Laser Melting (SLM)

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The Selective Laser Melting (SLM) technology uses metal powders as building material which is melted and welded together using a high-power laser in order to obtain quick configuration of complex parts, most often for testing them. Another advantage of this method is the fact that allows obtaining any 3D geometry of the parts, even parts that cannot be processed through conventional manufacturing procedures. In this work were performed a number of tests for accelerated corrosion of AlSi10Mg alloy specimens in order to determine their mean life in the conditions of their use in a high salinity environment. For specimens, optical analysis was used the SEM microscope which has the advantage of obtaining an enlarged image of the investigated objects without processing. Following these analyses, it has been determined the mass loss of specimens due to corrosion.

Keywords: additive manufacturing, reliability, degradation analysis, accelerated corrosion test, mean life

Initially known as Rapid Prototyping technology, Additive Manufacturing (AM) of metal finished parts is recognized as an interesting alternative to other conventional or unconventional processes, thanks to its capability to produce complex shapes and integrated parts of a high strength-to-weight ratio [1, 2].

According to standard ISO/ASTM 52900:2015, Additive Manufacturing is defined as *process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies*. The basic principle of this innovative technology is that a geometric model, initially generated using three-dimensional Computer Aided Design system, can be manufactured directly without the need of process planning [3, 4].

The development of AM processes such as Selective Laser Sintering, Selective Laser Melting, Electron Beam Melting and Laser Engineered Net Shaping enabled to build parts by using metallic materials, metal matrix composites and ceramic materials [5-7]. Additive manufactured parts are now used in aerospace [8], automotive, medical fields and also in sustainable energy for building the components of small size and complex geometries [3, 4, 9-11].

Because of growing interest in sustainable energy, solar power has received a lot of attention over the past several years. In the solar power field the photovoltaic micro-power plants have a promising future because they can be used to produce both electric and thermal power for domestic use. In this case, it is need to reduce the dimensions of the components used in such systems.

Consequently, in this field of energy there is the need and have been used complex components such as the heat exchangers and regenerators (and not only) of small size and very complex shape [10-12], that can ensure an increased area of exchange between the working fluid, thereby increasing the efficiency of heat exchange and, therefore, overall performance of the system. These components require very complicated geometries that can be realized only by some innovative manufacturing processes such as Selective Laser Melting (SLM).

Selective Laser Melting (SLM) is one of the powder based additive manufacturing technologies and it is also the most rapidly growing technique in Rapid Prototyping. This is due in most part to the possibility of creating metal parts with complex shape and intrinsic engineered characteristics. In order to manufacture a component with SLM technology the CAD model is sliced into layers. The component is built-up layer-by-layer by melting the powder layer locally with a laser beam. Components manufactured with SLM offer a high geometrical flexibility and accuracy without almost any loss of material. These leads, on the one hand, to resource savings and the other hand to eco-design optimization [4, 9, 13].

The selective laser melting process is a cycling process composed of the following steps:

-adding of powder;

-laser exposure;

-lowering building plate by the value of one layer thickness;

It is well known that for obtaining components by SLM technology is used a narrow range of materials. The most popular for medical and aerospace applications is the titanium alloy Ti-6Al-4V. Other metals used in SLM are cobalt-chrome, stainless steels, tool steels, aluminium alloys such as AlSi12Mg, AlSi10Mg, gold alloys, nickel-based super alloys such as Inconel 625 and 718, and TiAl alloys [3, 4, 14].

One materials' group of great interests to those who develop the SLM process are the aluminium alloys, which are widely used in modern manufacturing [15]. SLM of

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aluminium can offer new opportunities in applications that require internal structures and cavities, like heat sinks and lightweight structures by producing parts with various structures and high freedom in geometrical complexity [10, 11, 14, 15]. In SLM can be used certain types of aluminium powders [2, 3, 13, 15] such as A6061, AlSi12, AlSi12Mg and AlSi10Mg. These are widely studied materials in SLM of aluminium.

In the literature, it is known that the production of mechanical parts made of metal powders may imply the presence of residual stress and porosity which also have a significant influence on the static and dynamic mechanical properties of the material. The presence and distribution of residual porosity in materials produced by SLM as well as the specific morphology strongly influences the properties of the components subjected to a cyclic loading.

Therefore, for producing and efficient use of the components made by SLM technology in sustainable energy systems, and not only, are necessary experimental investigations on residual porosity, microstructure, thermal properties, behaviour to different environment factors and mechanical properties.

Buchbinder showed in [14, 16] that AlSi10Mg is an appropriate material for SLM by producing components of nearly 100% density. Also, he showed that AlSi10Mg components have a wide range of mechanical properties depending on the solidification process. Thijs in [13] investigated that the AlSi10Mg components manufactured by SLM have an extremely fine microstructure and hence a high hardness. Prashanth [17] observed an extremely fine cellular structure for Al-12Si alloy processed by SLM. Such a microstructure leads to yield and tensile strengths which are four and two times higher than the corresponding values of the respectively cast material. Kempen observed in [18, 19] higher mechanical properties of AlSi10Mg components or at least comparable to the cast AlSi10Mg material. Brandl [20] investigated the microstructure of samples manufactured by SLM using anAlSi10Mg powder alloy. The samples were fabricated for high cycle fatigue and machined afterwards [2]. In [2, 21] were explored the feasibility of introducing high-strength aluminium alloys for industrial applications, focusing mainly on the production of custom powder with different particle sizes and different distributions of elementary components. Louvis in [15] clarifies the importance of oxide films in the SLM processing of aluminium and its alloys, and identify methods for increasing part density without the use of excessive laser powers. Wong in [11] fabricated heat sinks using 6061 aluminium powder and the paper concentrated upon the properties of the part produced. Aluminium 6061 is used with the process to illustrate the improvement in heat transfer provided by higher conductivity feedstock materials. In [12] is showed the innovative approach of manufacturing filigree and highly complex components by AM. They proved that AM gives superior properties to the component compared to conventional manufacturing methods.

Although in the literature there are studies on the microstructure, mechanical properties of aluminium alloy components obtained by SLM technology little information have been published on their behaviour in different external environments.

It is well known that, especially the materials used in manufacturing components for sustainable energy devices are exposed to various external factors that depend on the place where they are working. These factors can be air humidity, salted water or salted atmosphere, sun exposure, temperature etc. To find the optimum material with optimum properties for building different parts for sustainable energy systems and, also, their behaviour in such environments are very important for increasing the product reliability.

In this paper is investigated the behavior and of the lifetime of AlSi10Mg specimens, manufactured by SLM technology, using corrosive accelerated testing. To obtain in short time relevant information about the behavior of AlSi10Mg specimens it is important to accelerate the degradation process. This goal can be reached by using proper equipment, which is able to simulate in a short time the normal action of corrosive factors during months or years, coupled with professional software programs for Accelerated Degradation Testing (ADT).

Experimental part

SLM setup and materials

Selective Laser Melting (SLM) is a three axes manufacturing process used to produce complex shape parts, starting from the 3D model, saved as *.stl file. During the manufacturing process, a very fine powder of metal is applied in successive layers and melted, layer by layer, with a laser beam. The principle of the method consists in sectioning the piece with a beam of parallel planes and its construction layer by layer on a SLM machine as follows: the first layer of the physical model is created by melting the metal powder along the section geometry. The model is then lowered by the thickness of the next layer, and the process is repeated until completion of the model. In accordance with the manufacturing speed and the surface quality, the layer thickness can be set between 20 - 75 μm.

The system uses atomized metal powders to produce dense metal parts of aluminium alloys, titanium, stainless steel, cobalt-chromium or tool steel but the number of compatible materials increases year by year.

The material used for the tested parts is AlSi10Mg, having the following chemical composition: Al 85.7-86.6 Si 9.0 - 10.0 Fe 2.0 Ni 0.5 Mg 0.40 - 0.6 Zn 0.50 Mn 0.35 Ti 0.15 Sn 0.15 Cu 0.03. AlSi10Mg is a widely-used alloy, because has a high corrosion resistance and low density, compared with other alloys. It has also a good hardenability, a good dynamic and static resistance and a very good thermal conductivity. The tested parts (fig. 1) are obtained on a Selective Laser Melting machine, SLM250HL [22] by using the following parameters:

- Laser power: 200 W;
- Focal point: 100 μm;
- Layer thickness: 50 μm;

a b c d

Fig.1. The tested specimens of aluminum
a, b – the specimens, c – the specimen after 100 hours' immersion in a 5% NaCl saline solution, d – the specimen after 200 hours' immersion in a10% NaCl saline solution

- Layer thickness: 50 μm;
- Building speed: 20 cm^3 / h
- Platform temperature: 200° C
- Inert gas, Argon 4.6 bar, 2 L/min
- Laser optics cooled by water and pressured air.

Design of accelerated degradation testing

The accelerated degradation test (ADT) was used, in this paper, to estimate the lifetime of AlSi10Mg specimens manufactured through SLM technology. When the specimen's life, during normal conditions of use, is much higher than the maximum time, allowed for testing, test engineers choose to increase the levels of stress at higher levels than the normal use.

The accelerated experiments have been performed on specimens manufacture by SLM technology of a number of 8 specimens of AlSi10Mg materials using as main stresses: temperature, humidity and UV radiation and was realized using ACS-Sunrise climatic chamber. After that, the specimens were exposed at the accelerated experiments in a saline environment. The accelerated degradation testing was carried out using the chamber with saline environment [23].

In order to increase the accuracy of reliability estimates in accelerated degradation testing problem, a carefully designed ADT plan is required. The objective of ADT plan is to minimize the uncertainty of the life and reliability extrapolations to normal use conditions by determining the optimum stress levels to be used in the test as well as the optimum allocation of test products at each level.

The case study was directed at the accelerated testing in a saline environment of a number of 8 specimens from aluminium alloy (AlSi10Mg) manufactured by SLM technology. The accelerated testing was carried out using the salt spray test chamber [24].

Each of the specimens made of aluminium alloy (AlSi10Mg) manufactured by SLM technology was subjected to salt spray tests. For the execution of the accelerated testing plan it was necessary to establish the following parameters:

-The acceleration model: Inverse Power Law;

-The number of products subjected to accelerated testing: 8 specimens;

-Distribution: Weibull;

-The accelerated stress regime: accelerated testing regime using NaCl - sodium chloride of 5% and 10% concentration;

- Stress levels: 2 levels of testing 5% NaCl and 10% NaCl; the number of tested specimens corresponding to the level of acceleration: 4 and 4 specimens.

The ALTA 7 software enables further degradation analysis by using the following degradation models: linear, exponential, power, logarithmic, Gompertz and Lloyd-Lipow. Within this paper has been used the *power* model having the following equation:

$$y = b \cdot x^a \quad (1)$$

where: *y* is the degradation measurement, *x* is the inspection time and *a* and *b* are model parameters.

Results and discussions

Accelerated degradation analysis

The degradation analysis represents an important data analysis technique used for determining the information regarding the degradation of a quality or performance feature, which is associated with the reliability of the specimens manufactured through a SLM technology. The degradation analysis is useful for the tests performed on specimens with a very high reliability and lifetime, where it is not possible the products failure testing within normal conditions of use.

In this paper, the salt deposits of eight AlSi10Mg specimens, exposed to a salt spray test (SST), were carefully analysed and measured. The AlSi10Mg specimens were inspected on every 50 h for measuring the salt deposits dimensions. Failure is defined when a salt deposition has the area of 1 mm^2 or greater. In the table 1 are presented the accelerated degradation data for the 8 specimens of AlSi10Mg after the salt spray test.

Table 1

THE DEGRADATION DATA OF AlSi10Mg SPECIMENS, MANUFACTURED THROUGH SLM TECHNOLOGY

Inspection Time [h]	Degradation [mm^2]	Sodium chloride [NaCl %]	Unit ID
100	5	5	1
150	7	5	1
200	8	5	1
250	10	5	1
100	3	5	2
150	5	5	2
200	8.5	5	2
250	11	5	2
100	2	5	3
150	4	5	3
200	4.5	5	3
250	6	5	3
100	4	5	4
150	5	5	4
200	8	5	4
250	12	5	4
100	6	10	5
150	8	10	5
200	11	10	5
250	12.5	10	5
100	7.5	10	6
150	8	10	6
200	10.5	10	6
250	13	10	6
100	9	10	7
150	12	10	7
200	12.5	10	7
250	13	10	7
100	7	10	8
150	9.5	10	8
200	12.5	10	8
250	14	10	8

The experimental data, obtained from the specimen's degradation analysis, were computed using the ALTA7 software program. In figure 2 were plotted the degradation analysis results of AlSi10Mg specimens. The purple line indicates the critical level of degradation, which in this case is given by a salt deposit area of 1 mm.

Through the Power degradation model, the degradation data were extrapolated to failure times that correspond to accelerated reliability tests. For the AlSi10Mg specimens estimated with a high reliability, the life duration and reliability indicators determination, under normal conditions of use, requires a longer testing time. For this reason, it has been decided to use the accelerated testing techniques. These tests are performed at more intense regimens compared to the nominal usage regime, aiming to magnify the degradation of the AlSi10Mg specimens. The economical aim of these tests is to shorten the time period

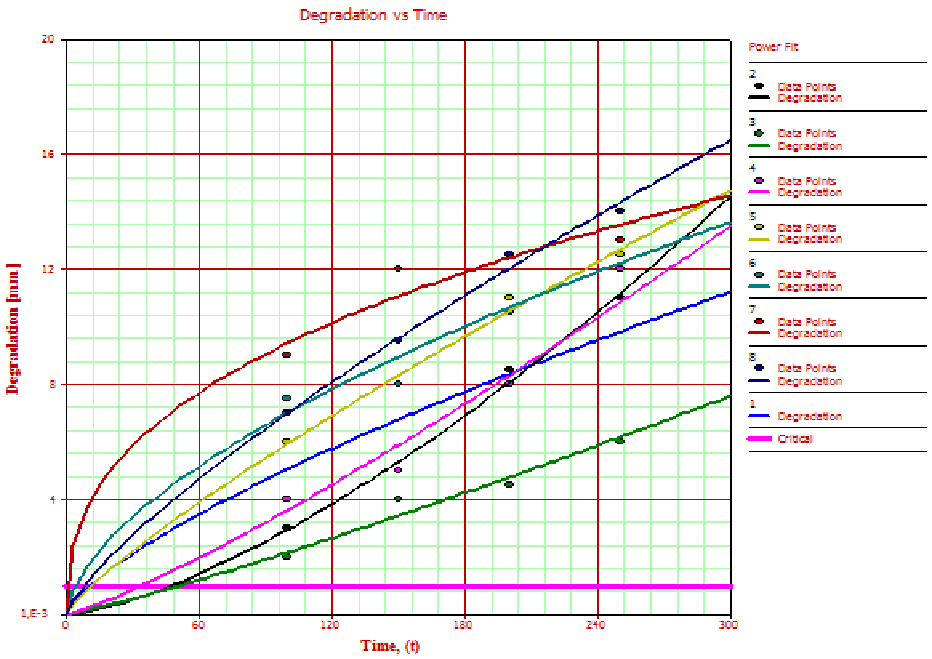


Fig. 2. The degradation obtained from the accelerated tests in accordance with the inspection time of the specimens

Table 2
THE DEPENDENCE BETWEEN FAILURE TIMES AND THE MAIN RELIABILITY INDICATORS FOR AlSi10Mg SPECIMENS

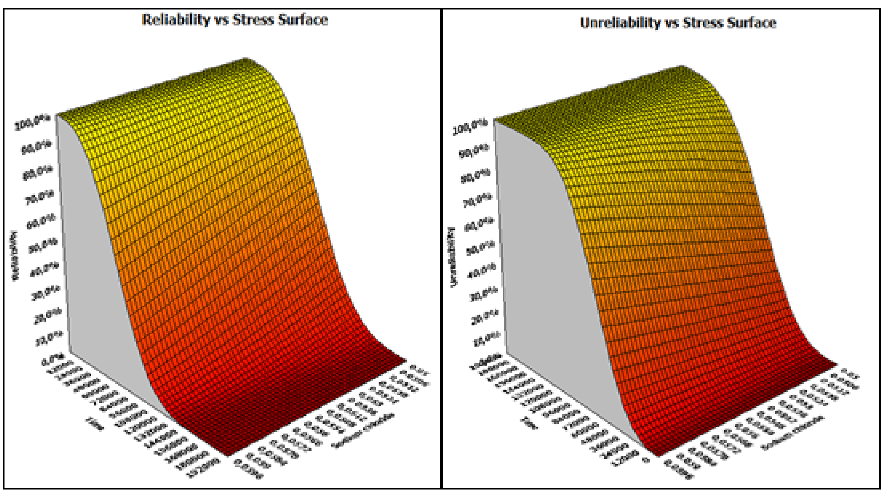
Time to failure [h]	Reliability	Unreliability	Probability density function	Failure rate
25618	0.917	0.083	0.0000018	0.0084797
59468	0.798	0.202	0.0000077	0.0384816
88699	0.679	0.321	0.0000109	0.0838695
88786	0.559	0,441	0.0000109	0.0840343
100921	0.44	0.56	0.0000106	0.1087693
108977	0.32	0.68	0.0000098	0.1272086
139245	0.201	0.799	0.0000049	0.2117118
140272	0.083	0.917	0.0000048	0.2150201

and the testing related costs, while maintaining the same modes, failure mechanisms and structures defects. Within the accelerated tests is accepted the hypothesis that the progress speed of processes increases when the stress conditions increase. Within this work, the sodium chloride concentration has been intensified to 5 and 10% and were determined the main reliability indicators (reliability function, non-reliability function, failure rate, probability density of the running time) for a nominal usage regime of the specimens (0.05% NaCl concentration) in an environment similar to the sea or ocean saltwater atmosphere. For data analysis of accelerated reliability tests have been used the Inverse Power Law model and the Weibull distribution. Using the accelerated data statistical processing, the main reliability indicators were determined (table 2) for times of failure, in normal use conditions, corresponding to the 8 specimens of AlSi10Mg.

Using the calculated values (the number of hours in normal testing conditions) the three-dimensional reliability function (fig. 3.a) and the three-dimensional unreliability function 3D (fig. 3.b) were plotted.

A probability density function three-dimensional plot (fig. 4.a) represents the relative frequency of failures as a function of time and stress. The failure rate three-dimensional plot (fig. 4.b) expresses the number of failures per time unit at a given moment, taking into account the number of specimens that are operating at that time according to the stress.

The main purpose of accelerated reliability testing is to determine the mean life in normal use conditions. Using



a) Reliability function

b) Unreliability function

Fig. 3. Reliability indicators

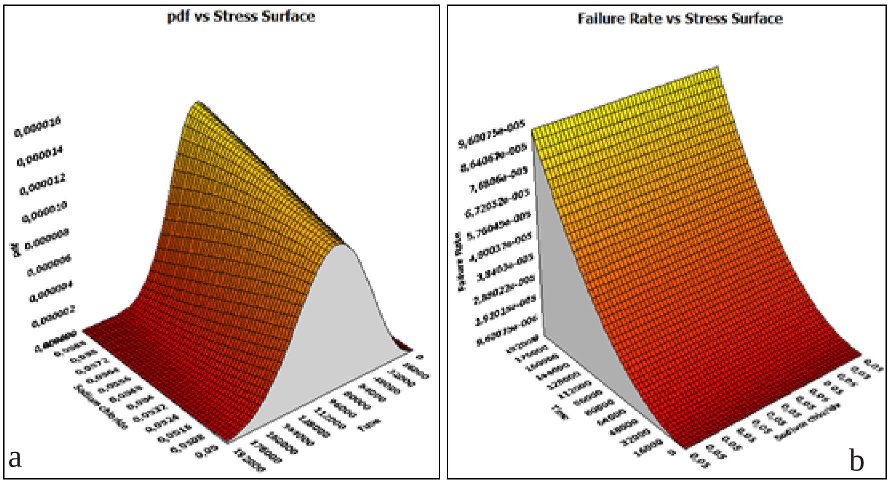


Fig. 4. Reliability indicators
a) PDF function
b) Failure rate

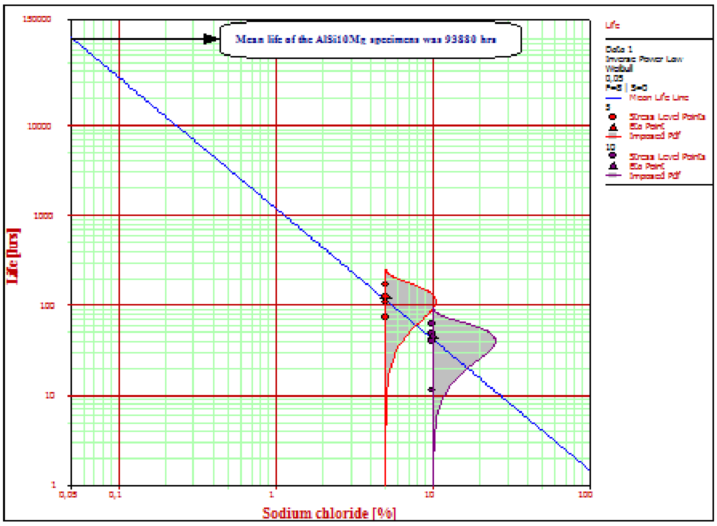


Fig. 5. The determination of the mean life of the specimens from aluminium alloy (AlSi10Mg) manufactured by SLM technology in normal use conditions

the data resulted from accelerated reliability tests we can determine the mean life of the specimens from aluminium alloy (AlSi10Mg), manufactured by SLM technology, in normal use conditions, (0.05 % NaCl concentration). In figure 5, by drawing a line through the mean number of hours to failure for the 2 levels of acceleration (5% NaCl and 10% NaCl) and marking the point of intersection of this line with the vertical line at the normal level of stress of 0.05 % NaCl, we found out the mean number of hours to failure in normal use conditions. The total number of hours to failure in normal use conditions for the specimens from aluminium alloy (AlSi10Mg) manufactured by SLM technology is 93880 h (about 10 and a half years).

The corrosion analysis of the specimens

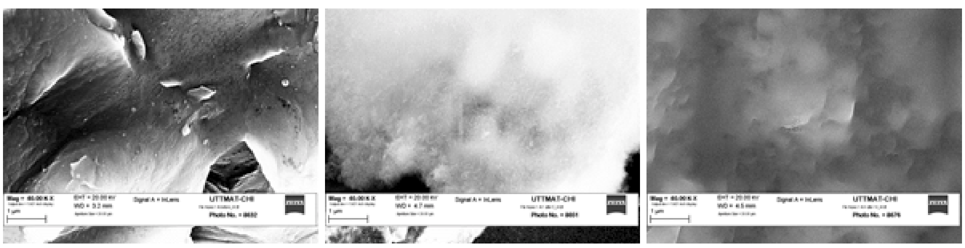
A scanning electron microscope (SEM) is used for morphological, chemical and microstructural characterizations of materials where a high-resolution field emission LEO 1525 SEM is used. The SEM is equipped with an Energy Dispersive X-ray (EDX) microanalysis which provides both qualitative and quantitative information about the chemical composition of the sample, and with an Electron Back Scattered Diffraction (EBSD) system that allows resolving local crystallographic orientations in the range of 100nm. When finishing the tests, important information about the corrosive resistances of our specimens was obtained.

The specimens tested in the chamber with saline atmosphere presents significant changes within their microstructural characteristics with attached salt particles on their structure. The following figures present the microstructural characteristics changes of the analyzed material, before and after the immersion into the chamber with saline atmosphere, for 100 h, using saline solution of NaCl (sodium chloride) of 5% concentration and for 200 h, using saline solution of NaCl of 10% concentration, at different magnifying scales (figs.6, 7, and 8).

These conclusions were elaborated after analyzing the microstructural characteristics, at a scanning electron microscope, of all the specimens, before and after passing the testing procedures.

The weight loss analysis of the specimens

Another determination performed within this paper aims to determine the weight loss of the specimens after their immersion in a saline solution, having a NaCl concentration

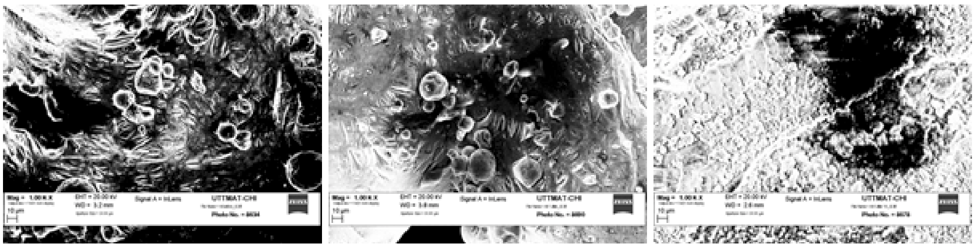


Initial structure   100 hours 5% NaCl   200 hours 10% NaCl

Fig. 6. Microstructural changes at 40000x magnifying scale

Initial structure | 100 hours 5% NaCl | 200 hours 10% NaCl

Fig.7. Microstructural changes at 10000x magnifying scale



Initial structure | 100 hours 5% NaCl | 200 hours 10% NaCl

Fig.8. Microstructural changes at 1000x magnifying scale

Days	ΔW(g)
0	0
3	0.0056
6	0.01
9	0.0152
15	0.0253
21	0.0329
27	0.043
41	0.0741
54	0.0886

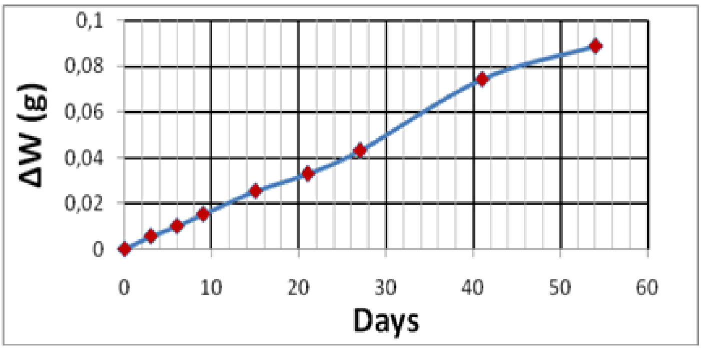


Table 3
THE WEIGHT LOSS OF THE AlSi10Mg SPECIMEN WITHIN 60 DAYS

of 10%. The experiment was performed during 54 days and consists in the extraction of specimens, on specific time intervals that are shown in table 3. After extracting the specimens are performed weight measurements in order to determine the weight loss.

The analysed specimens have the following characteristics:

Dimensions: 25 mm × 10 mm × 3 mm

Area: 7.1 cm^2

Density of AlSi10Mg alloys: 2.68g/cm^3

The weight loss of the sample before and after the corrosion test ($\Delta W = W1 - W2$), using saline solution of NaCl of 10% concentration, is indicated in table 3.

Conclusions

The specimens that have been analysed within this paper have a lattice structure and can be generated only through additive processes. For this reason, has been used the SLM technology for their production.

In only eleven days the accelerated corrosion tests are able to produce equivalent corrosion to that observed in ten and a half years in a normal outside environment (0.05% NaCl solution concentration) close to sea or ocean with saltwater. This testing time reduction, doubled by the obtained corrosion data for AlSi10Mg specimens, makes the accelerated corrosion testing techniques, a very efficient method for determining the corrosion behaviour of the materials manufactured through SLM technology.

The specimens tested in the chamber with saline atmosphere presents significant changes within their microstructural characteristics with attached salt particles on their structure. In order to verify the salt deposition and the corrosion areas on the specimens' surface the microscopic (SEM) analyses were carried out. In addition to the salt particles attachment on the specimen structure was detected also a mass loss because of the corrosion phenomena appearance.

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