

Experimental researches on the corrosion behavior and microstructural aspects of heat treated Al-Zn-Mg-Cu alloys

Maria-Cristiana Enescu, Ileana-Nicoleta Popescu, Raluca Zamfir, Alina Molagic, Vasile Bratu

Abstract—The material for experiments was an aluminum alloys series 7xxx obtained by thermo-mechanical processing. We studied the influence of different artificial aging parameters on mechanical characteristic and corrosion of Al-Zn-Mg-Cu-alloy. The Al alloys was solution treated at $475 \pm 5^\circ\text{C}$ for 60 minutes, quenched in water $40\text{--}60^\circ\text{C}$ and artificial aging at 120, 150, 180, 210°C at different time of aging (60, respectively 300, 540, 720 minutes). After heat treatment the obtained alloys were corroded in solution of 53g NaCl + 1000 ml distilled water and boiled in these solutions for 24 hours. After corrosion test, samples were prepared for optical microstructural analyzing and mechanical tested. We observed that the best corrosion behavior has the sample aged at 180°C /12 hours, when coalescence occurs and the weakest corrosion behavior has the sample aged at 180°C /1 hour (better behavior in comparison with the sample aged at 210°C). At 150°C the maximum hardness is higher, while the decreases of hardness in time are lower and corrosion behavior is better for all samples.

Keywords—7xxx aluminum alloys, Artificial ageing, Micro-structure analysis, General corrosion

I. INTRODUCTION

THE wide range use of aluminum and aluminum alloys in the transportation industry (aircraft, automotive industry, etc.) is based on superior mechanical characteristics of these alloys, low specific weight and corrosion resistance. Superior mechanical characteristics and optimal chemical characteristics of aluminum alloys are obtained due to the possible application of structural hardening treatments and compositional modifications. The 7xxx series alloys are very strong heat treatable alloys and they can be strengthened through heat treatment (precipitation hardening) based on the combination of zinc (mostly between 4–6 wt %) and magnesium (range 1–3 wt %) [1,2]. The high strength Al-Zn-Mg-(Cu, Zr) system or 7xxx series alloys is a highly complex one and are greatly determined by the main phases in the alloys, i.e. Guiner-Preston zones, η' , η , T, S, Mg_2Si and Fe rich intermetallic phases [3-6]. In commercial 7xxx alloys, only η (MgZn_2 ; $(\text{Al}, \text{Zn})_2\text{Mg}$), T ($\text{Mg}_3\text{Zn}_3\text{Al}_2$; $\text{Al}_6\text{Mg}_{11}\text{Zn}_{11}$) and S (Al_2CuMg) phases could appear in his structure [5-8].

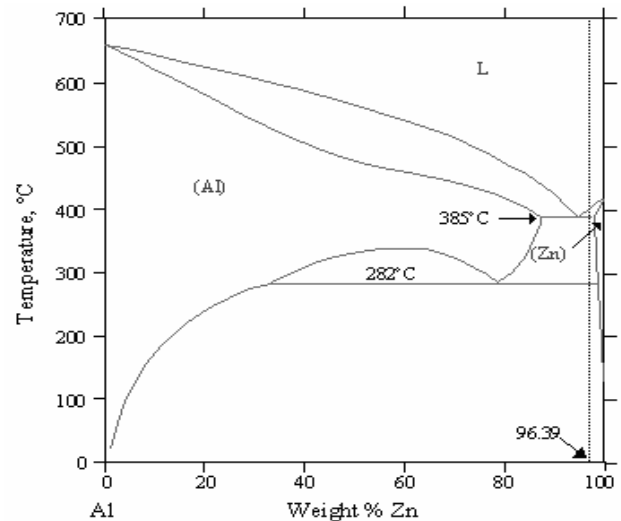


Fig 1 The Al-Zn equilibrium phase diagram [9]

According to the binary equilibrium diagram of Al-Zn (Fig.1) [9], because of existence of solvus solubility, at temperature decreasing, decrease the solubility of solid solution.

The decreased solubility of solid solution leads to its saturation and the material becomes thermodynamically unstable and therefore will tend to decompose into two new phases. Since the material is put into solution and then rapidly cooled (quenching) at a temperature below the solvus curve, the alloy matrix remains in the metastable state of supersaturated solid solution. At reheating of material at moderate temperatures, below the solvus line, at ageing, from solid solution occurs precipitation of a metastable phase in alloy matrix. The phases which appear in matrix at the precipitation process in 7xxx series alloys deduced from X-ray and electron microscope investigations consisted in supersolid solid solution \rightarrow spherical Guiner Preston (GP) Zones \rightarrow Internally ordered GP zones \rightarrow intermediate M (MgZn_2) phase \rightarrow Stable T ($\text{Al}_2\text{Mg}_3\text{Zn}_3$) phase [10]. Heat treatment process parameters such as aging temperature and aging time operate differently according to different conditions. Technical purity aluminum, Al-Mg alloys, Al-Mg-Si alloys with Mg_2Si as intergranular precipitate and other aluminum alloys where is not observed or there are small potential differences between the matrix and intergranular precipitates, are generally not susceptible to severe intergranular corrosion [11]-[15].

On the other hand, the aluminum alloys in which intergranular precipitates are nobler than matrix (the CuAl_2 precipitates from Al-Cu alloys) or less noble than the matrix (the Mg_2Al_3 precipitate in Al-Mg alloys, and the MgZn_2 precipitate in Al-Zn-Mg alloy system) may be susceptible to intergranular corrosion. The susceptibility degree to intergranular attack depends on the nature, size, quantity, intergranular precipitates distribution and therefore of heat treatment applied to these alloys [16]-[21].

Unfortunately the additions of Zn and Mg decrease the corrosion resistance. The existence of micro oxide film defects, inhomogeneity, potential differences between intermetallic particles and aluminum matrix and galvanic couples conduct to localized chemical attack of aluminum alloys. The susceptibility of Al-Zn-Mg-(Cu) alloy to intergranular corrosion increase also when the alloy is exposed to solutions containing chloride ions [12]-[15], [23].

II. PROBLEM FORMULATION

The research consisted in intercrystalline corrosion and hardness tests in correlation with microstructure analysis of heat treated Al-Zn-Mg-Cu samples alloy, in order to obtain the best corrosion resistance and high mechanical characteristics.

A. Materials and Experimental Procedure

When The material selected for investigations in this research was rolled 7xxx series aluminium alloys; the chemical composition of investigated alloy was determined according to ASTM standard methods. The Al-Zn-Mg-Cu samples was heat treated in the following conditions: solution treated at $475 \pm 5^\circ \text{C}$ for 60 minutes, quenched in warm water ($40-60^\circ \text{C}$) and then artificial ageing at different temperatures ($120, 150, 180, 210^\circ \text{C}$) and different times (60, respectively 300, 540, 720 minutes).

Solution treatment was made into laboratory room furnace and artificial ageing was made into furnace ITM 50 with automatically adjustment of temperatures. After heat treatment were performed mechanical tests to determine Brinell hardness. For the hardness tests we used a Hardness Tester with 2.5 mm diameter ball indenter and load 62.5 Kgf. The corrosion was made in solution of 53g NaCl + 1000 ml distilled water and boiled in these solutions for 24 hours.

After intergranular corrosion test, samples were prepared for microstructural analyzing in the following conditions: were grinded on metallographic paper up to 600 grit, polished with slurry of Al_2O_3 (1 μm granulation) and then etching with Vilella's Reagent. (45 ml Glycerol, 15 ml Nitric acid and 30 ml Hydrochloric acid) according with specific standardization [22], [23].

To investigate the general corrosion resistance was used an electrochemical method: the electrochemical corrosion was made in the same type of solution like previous one (53g NaCl + 1000 ml distilled water), using a potentiostatic device type PAR (Princeton, Applied, Research) model 173. For the

electrochemical corrosion tests we have linked the cell work to the potentiostatic device and we measured the stationary potential and the current density after a stabilization time of 2-3 minutes. The device for electrochemical corrosion consisted between three electrode systems: the working electrode, which are the aluminum alloy sample, the auxiliary electrode (or counter electrode) and saturated calomel electrode as reference electrode. The working electrode was a cylinder of Al-Zn-Mg-Cu alloy with following dimensions: 10 mm in diameter and 20 mm in length. The working electrode area was 1 cm^2 (the remaining surface of the work-piece being coated with a resin). And the auxiliary electrode was platinum plate, placed in a separate compartment and protected by glass from the rest of the cell to prevent contamination of electrolyte by electrolysis products.

III. PROBLEM SOLUTIONS

The chemical composition of the Al-Zn-Mg-Cu alloy used in the present study is presented in Table I.

Table I

Element	Zn	Mg	Cu	Fe	Si	Ti	Al
(wt%)	5	1.5	1.24	0.29	0.03	0.02	Balance

Thus, solution treatment was made at $475 \pm 5^\circ \text{C}$ for 60 minutes, quenched in warm water ($40-60^\circ \text{C}$) followed by aging. At heating of supersaturated solid solution obtained by quenching of precipitated hardening alloy, the metastable state of the alloy tends to move into more stable states by removing from solution of alloying element in excess, as precipitate.

The phase nature and structural characteristics of the precipitate that are formed depends on temperature and heating duration. The most important properties changes of the alloy occur in the early stages of aging (at low temperatures and short durations of heating) when the under microscopic precipitates are formed. The dependence of hardness on different condition of aging (temperature and times) is shown in Fig 2.

Aging curves show that the hardening effect occurs during the formation of coherent precipitates (Guinier-Preston zones, intermediate metastable precipitates), but the hardening effect decrease with the appearance of semi-coherent precipitates (intermediate precipitates), and incoherent precipitates (equilibrium precipitates). The coherent precipitates, GP zones considered that pre-precipitates and coherent intermediate precipitates (with lamellar shapes) have relatively uniform germination, and are linked by coherent interference matrix. Intermediate, semicoherent precipitates are formed by heterogeneous germinations on some defects of matrix, for example on screw dislocations. The equilibrium precipitates heterogeneous germinate on grain boundaries of matrix and the precipitate-matrix interface is incoherent. The microstructures of heat treated Al-Zn-Mg alloys are presented in Figures 3.

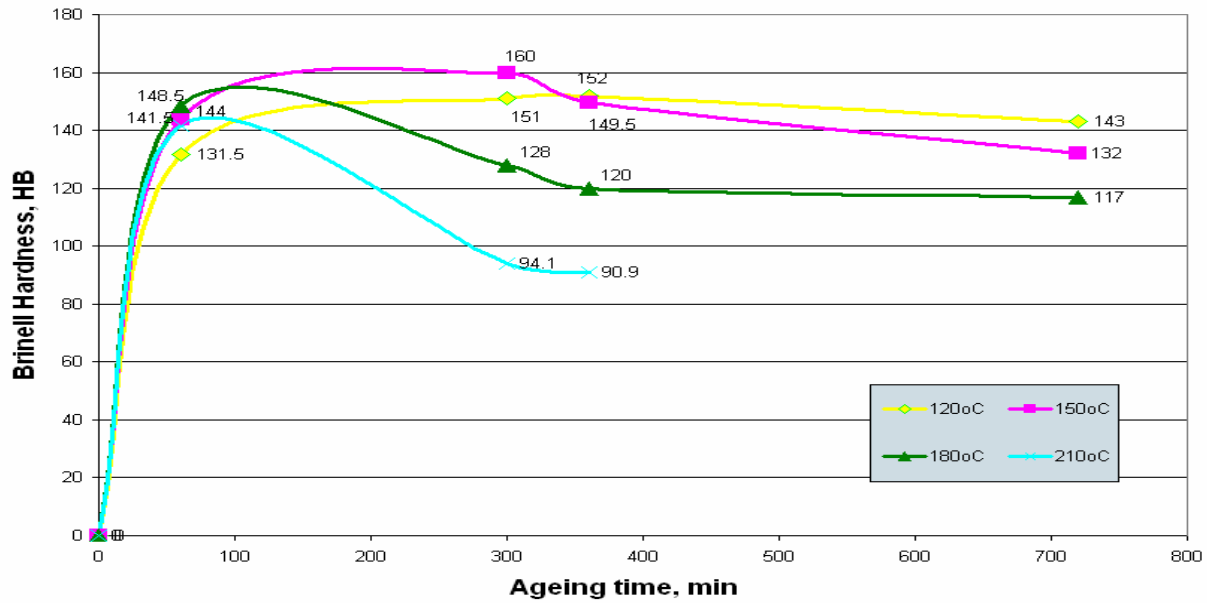


Fig. 2 The Brinell Hardness variation depending on different temperatures (120-210°C), and different times of aging (60-720 min)

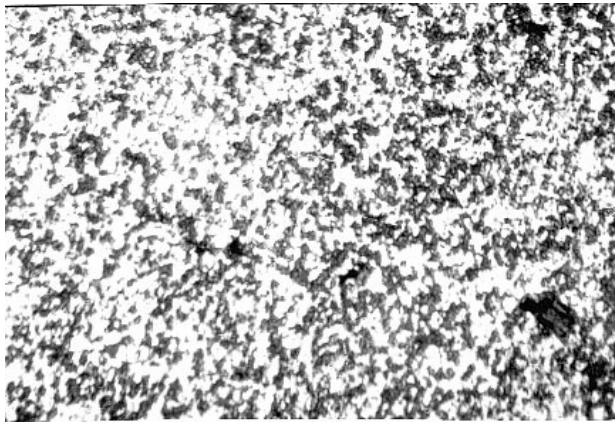


Fig. 3a)

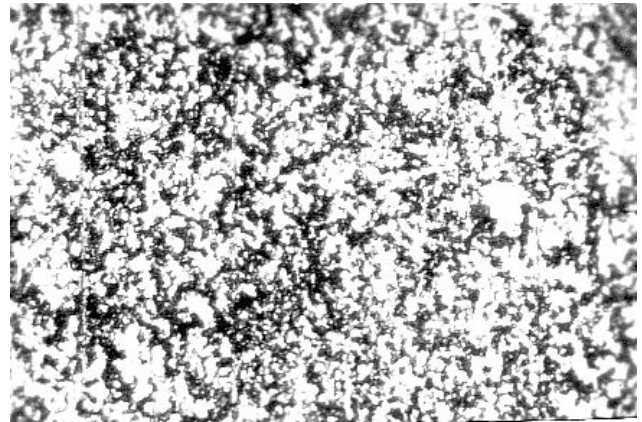


Fig. 3b)

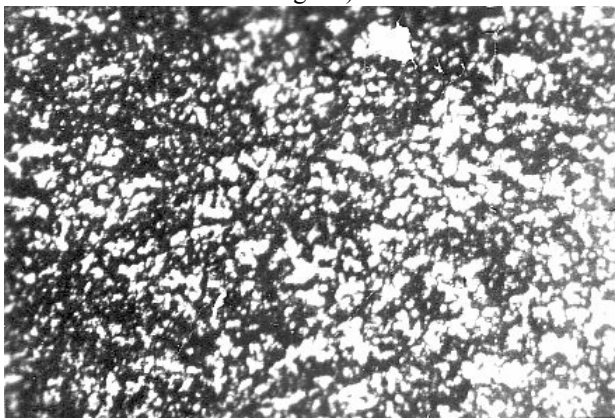


Fig. 3c)

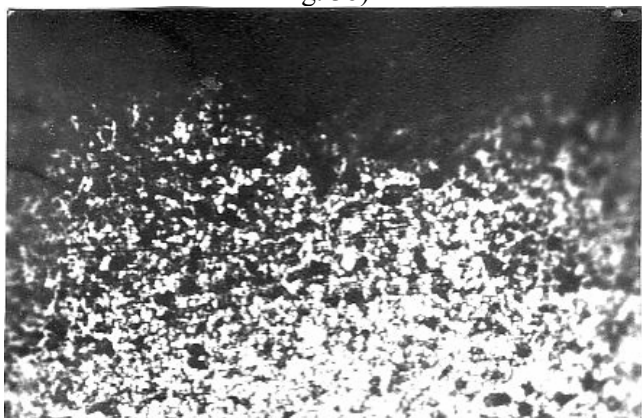


Fig. 3d)

Fig. 3 The microstructures of heat treated Al-Zn-Mg alloys solution treated at $475 \pm 5^\circ \text{C}$ for 60 minutes, quenched in warm water ($40\text{-}60^\circ \text{C}$) and then artificial ageing at 210°C and different ageing duration (a, b) 60 min, (c, d) 300min, x 200 magnification

At analysis of microstructures in correlation of Brinell Hardness variation, show that as temperature and the duration of aging increases, we reached the maximum

hardness (fine precipitates occurred), and after that, the hardness decrease as a result of appearance of coarse precipitates.

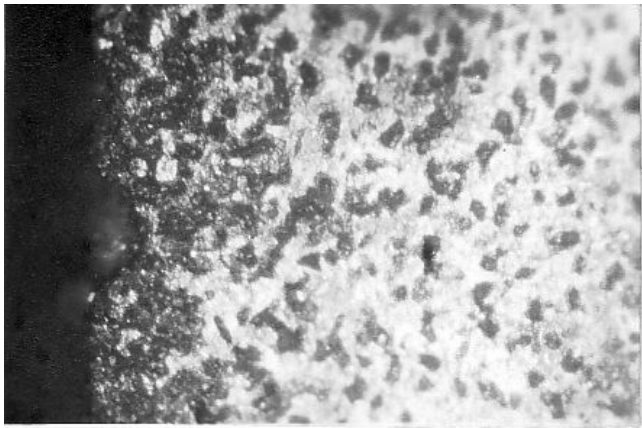


Fig. 3e)

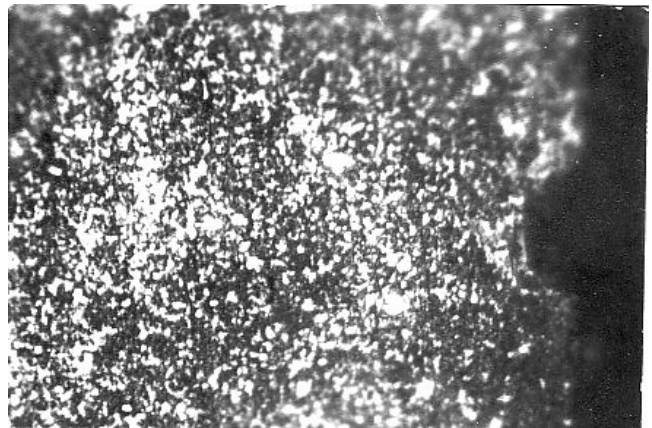


Fig. 3 f)

Fig. 3 The microstructures of heat treated Al-Zn-Mg alloys solution treated at $475\pm 5^\circ\text{C}$ for 60 minutes, quenched in warm water ($40\text{-}60^\circ\text{C}$) and then artificial ageing at 210°C and different ageing duration (e, f) 540 min ; x 200 magnification

This phenomenon is explained by the development process of coalescence on downward curve of the variation hardness diagram. According with Fig. 1 we observe that as the aging temperature increases, decreases the time of aging which the material presents maximum values of hardness.

For artificial aging has been shown that there is a critical temperature below which precipitates in the matrix uniformly germinate. Also, at this temperature, the germination of grain occurs not homogenous at grain boundaries on dislocations and other imperfections. At low aging temperatures, short maintaining aging durations and at high super-saturation, respectively, result that decomposition occurs by forming of Guinier-Preston zones. In terms of such no-uniform precipitation, the corrosion rate of structural hardening alloys is small, instead at higher aging temperatures, when precipitation is not uniform, the corrosion rate increases. At higher temperatures and longer maintenance during aging, the coalescence precipitation phenomena are favored and consequently are favored also the decreases of tensile strength and corrosion rate. In general, precipitation hardened alloys are more susceptible to intercrystalline corrosion when are aging to peak hardness and less susceptible at overheat conditions. In Fig 4 are shown the polarization curves (the current density I [mA] as function of electrode potential E [V]) for Al-4Cu-1,5 Mg -Mn solution treated at $475\pm 5^\circ\text{C}$ for 60 minutes, quenched in warm water ($40\text{-}60^\circ\text{C}$) and then artificial ageing at 60-720 minutes.

The polarization curves were made under a constant aging temperature and different duration of aging (Fig. 3 a-e) and also for different aging temperatures ($120, 150, 180$ and 210°C) and at 300 minutes maintaining time of aging (Fig. 4). Analyzing the structures from Figures 2 we notice that there is a corrosion layer with separation of cavities forming material and corrosive. It is observed that with increasing duration of aging, the corrosion resistance decreases.

IV. DISCUSSION

We can see also that the required hardness and the stability in time of this hardness are ensured at $150^\circ\text{C}/300$ minutes artificial aging. Analysis of microstructures presented in Fig. 2 (d, e and f) in correlation of Brinell hardness variation, show that as temperature and the duration of aging increases, we reached the maximum hardness (fine precipitates occurred), and after that, the hardness decrease as a result of appearance of coarse precipitates .

This phenomenon is explained by the development process of coalescence on downward curve of the variation hardness diagram. Aging curves show that the effect of hardening occurs during the formation of coherent precipitates (Guinier Preston zone), but that it decreases the occurrence of incoherent precipitates.

Intercrystalline corrosion is the electrochemical corrosion which taking place at the boundary secondary phase. The intercrystalline corrosion is explained as follows: by reducing of solid solution between the secondary phase and solid solution appears a potential difference and forming a galvanic micro-cell.

After analysis of polarization curves we can observed first: an active process, in which, corrosion current intensity increases over a small range of potential. After this zone, metallic material enter in a passivation zone, zone characterized of a small change in current intensity within a high potential.

In the passive corrosion zone the process takes place more slowly than in the active zone. From Fig. 4 we observed that with increasing aging time, the modification process from passive to active one is carried out at current density becoming much smaller.

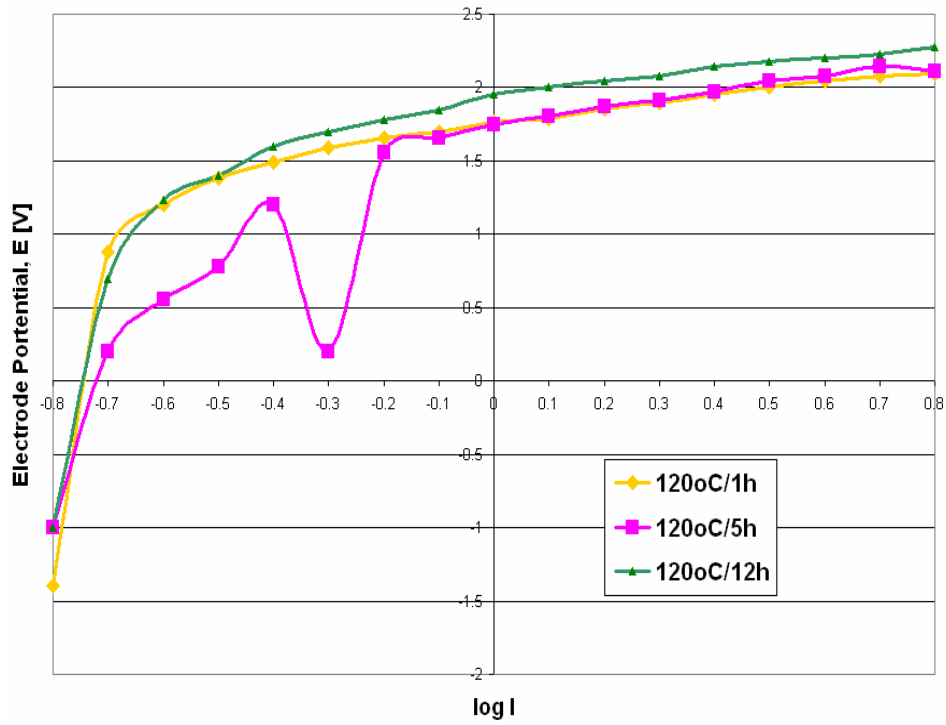


Fig. 4a)

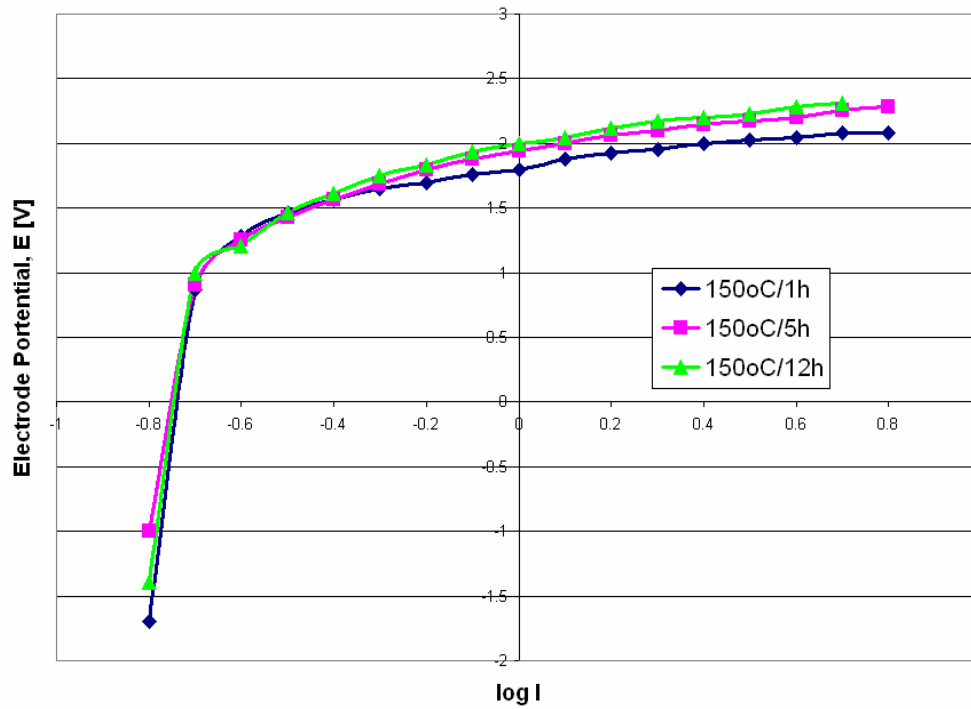


Fig. 4b)

Fig. 4 Polarization curves for Al-Zn-Mg-Cu solution treated at $475 \pm 5^\circ\text{C}$ for 60 minutes, and then artificial ageing at (a) 120°C ; (b) 150°C ;

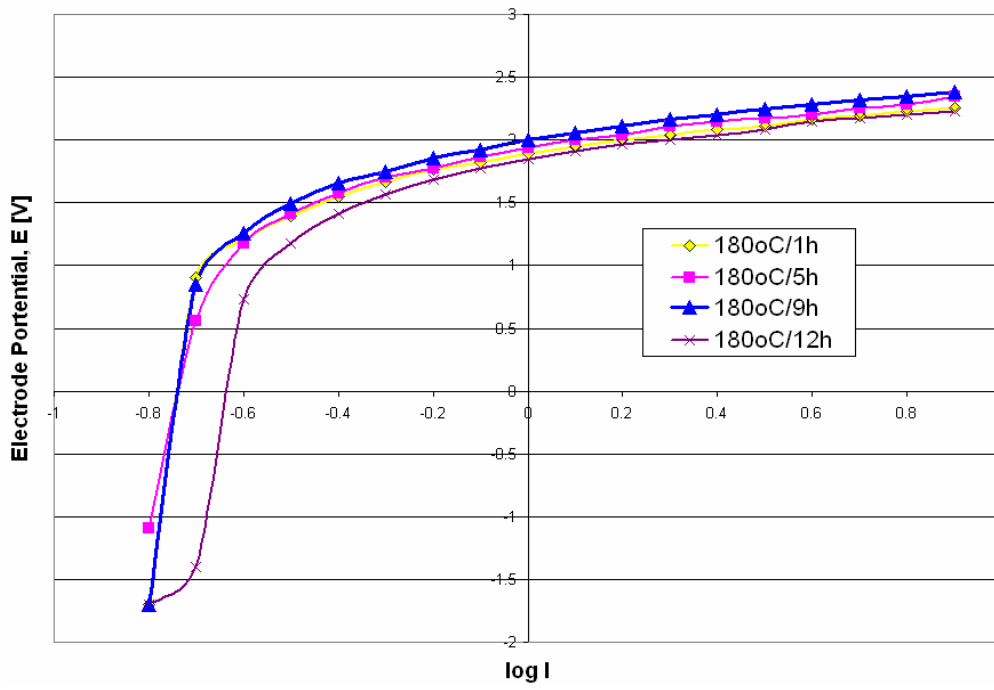


Fig.4 c)

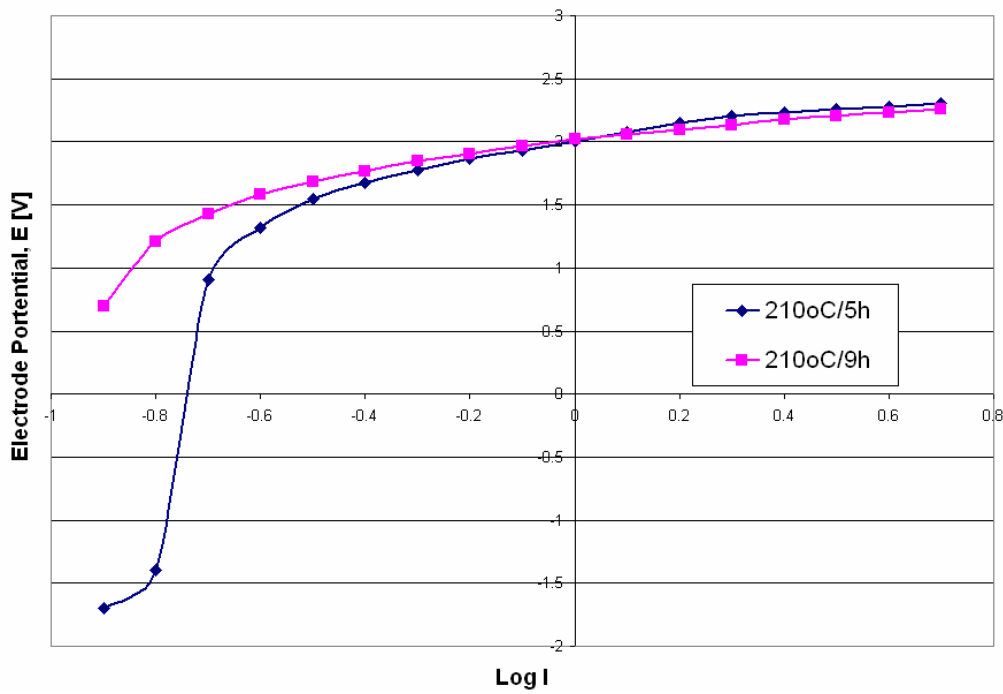


Fig. 4 d)

Fig. 4 Polarization curves for Al-Zn-Mg-Cu solution treated at $475 \pm 5^\circ\text{C}$ for 60 minutes, and then artificial ageing at (c) 170°C (d) 210°C and different duration of aging (between 60-720 min)

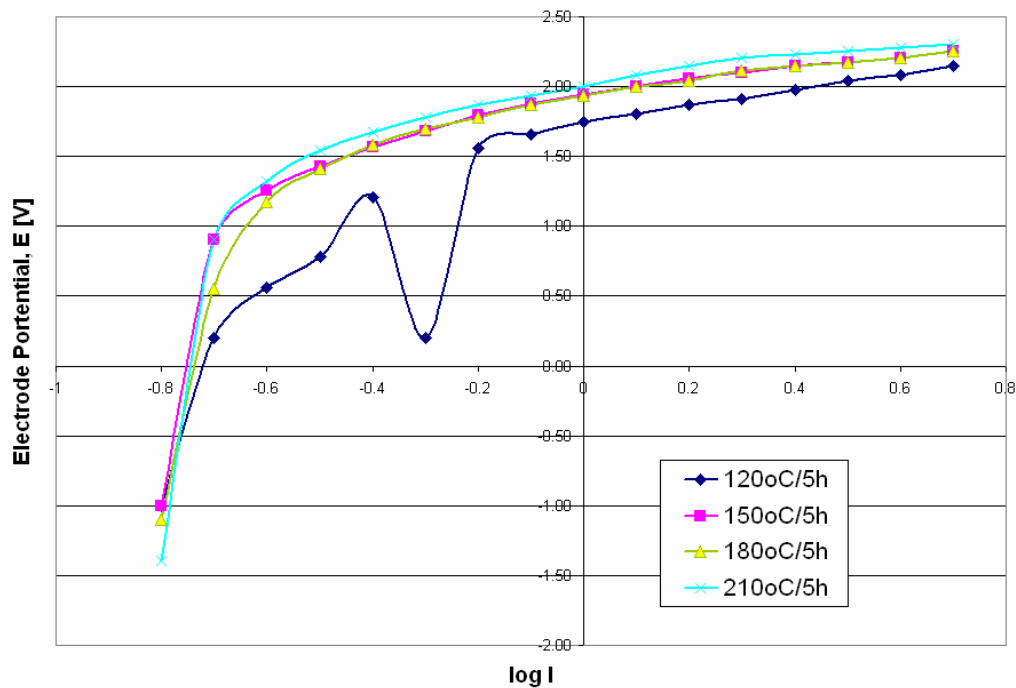


Fig. 5 Polarization curves for Al-Zn-Mg-Cu heat treated at different ageing temperature and maintained at same aging time (300 minutes)

V. CONCLUSIONS

At 210°C, in terms of mechanical properties, the alloys quickly reach maximum hardness, but decrease occurs rapidly, which means that these characteristics change it during operation. In terms of corrosion, at 210°C, the best behavior has 9 hours aged sample, but it does not have good mechanical characteristics.

The best corrosion behavior has the sample aged at 180°C /12 hours, when coalescence occurs and the weakest corrosion behavior has the sample aged at 180°C /1 hour (better behavior in comparison with the sample aged at 210°C). At 150°C the maximum hardness is higher, while the decreases of hardness in time are lower and corrosion behavior is better for all samples.

At 120°C, the peak hardness is lower than at 150°C, if it is kept almost constant in time, the coalescence phenomena do not occur, instead, the corrosion behavior is weak, especially for the sample which was aged 5 hours (maintaining duration) which has the worst corrosion behavior.

This sample had also sudden variations of anodic current, which indicate the occurrence of localized corrosion phenomena - phenomena associated with formation of Pitts or some crevices). At 120°C aging temperature when coherent precipitates are formed, the corrosion behavior is weak.

For samples aged at 210°C, when occurs the precipitation coalescence phenomena, it improves corrosion behavior in detrimental of mechanical characteristics.

At 150°C aging temperature, from mechanical characteristics point of view, the hardness of samples aged at 180°C and 210°C have similar values, but instead at 180°C we observed that a sudden decrease of hardness over time.

The corrosion behavior at 180°C is better at long maintaining time, but the mechanical properties (low hardness) are weaker. In these circumstances I consider that aging performed at 150°C with duration of time between 1 to 5 hours it is which one that provides the best corrosion behavior.

Following experimental data obtained, it is noted that in terms of microhardness the best behavior is at 150°C during 300 minutes, followed by the values of hardness obtained at ageing parameters of 120°C to 300 minutes.

In terms of corrosion we observe that the Al-Zn-Mg-Cu alloys sample tested at artificial ageing at 120°C with to 300 minutes, the obtained results are not so good.

For aging temperature of 180°C and 210°C we are found that maximum hardness is obtained at lower aging times. From corrosion point of view we observed that the behaviors of all analyzed samples are similarly.

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Maria Cristiana Enescu was borne in Balta Sarata (Teleorman County, Romania), February, 1st, 1967. She graduated in Chemistry field in 1995 at University of Bucharest, Romania, Faculty Chemistry. Between September 1995 and August 1998 she was chemistry teacher at Dambovita School Inspectorate. Between September 1998 she was teacher in Materials Science at the University Valahia of Targoviste, Faculty of Material Science, Mecatronics and Robotics, "Valahia" University from Targoviste (RO). In 2002 she made a specialization stage in France for 4 month at Rouen University, Science Faculty, Department of Physic, at LECAP Laboratory where studied and characterized some polymers, under scientific coordination of Prof. dr. Jean-Marc Saiter. From 2010 she received her Ph.D. in Material Science and Engineering field at University "Politehnica" of Bucharest, Romania, with a Thesis concerning "Studies and research on structure and properties of new composite materials" under the guidance of Prof. Dr. Eng. Simona Zamfir (the scientific coordinator of her thesis). Lecturer Dr. Ch. Maria Cristiana Enescu has published in Proceedings of Conferences and Journals about 27 scientific research papers and presented 1

Poster and Oral Communications, 5 research projects (as collaborator and responsible of project) and 3 books.

Lecturer Dr. Ch. Maria Cristiana Enescu is Member of Chemistry Society from Romania, SChR (Febr. 2008), she is also Member of Robotic Society from Romania, SSR, partner of International Federation of Robotics, <http://srr.ssai.valahia.ro> (Febr. 2007).



I. N. Popescu was borne in Slatina (Olt County, Romania), August, 24th, 1969. She graduated in Materials Science field in 1994 at University "Politehnica" of Bucharest, Romania, Faculty of Materials Science and Engineering. Between October 1994 and July 1999 she was Engineer and Researcher at Metallurgical Research Institute ICEM S.A, Bucharest at Research Department for Special Alloys and Sintered Products.

In 2002 she made a specialization stage in France for 4 month at Rouen University, Science Faculty, Department of Physic, at LECAP Laboratory where studied and characterized some polymers, under scientific coordination of Prof. dr. Jean-Marc Saiter. From June 2006 she received her Ph.D. in Material Science and Engineering field at University "Politehnica" of Bucharest, Romania, with a Thesis concerning "Researches on Development of New Composite Materials with Aluminum Alloys Matrix" under the guidance of Prof. Dr. Eng. Simona Zamfir (the scientific coordinator of her thesis).

From October 1999 until February 2003 was Assistant Professor and than, until now, she has been Lecturer at Faculty of Material Science, Mecatronics and Robotics, "Valahia" University from Targoviste (RO).

She is specialized in Materials Science and Engineering, especially in Processing and Characterization of Metallic Composites (Al, Cu or Fe as a matrix). Her current research interests include processing by powder metallurgy (P/M) of aluminum based composites reinforced with ceramic particles. Lecturer Ileana Nicoleta Popescu has published in Proceedings of Conferences and Journals about 36 scientific research papers and presented 32 Poster and Oral Communications and has 17 research projects (as collaborator and responsible of project).

Lecturer Dr. Eng. Ileana Nicoleta Popescu is Membership of General Association of Romanian Engineer AGIR (www.agir.ro) since 1994, she is also Member of Chemistry Society from Romania, SChR (Febr. 2006), is Member of Robotic Society from Romania, SSR, partner of International Federation of Robotics , <http://srr.ssai.valahia.ro> (Febr. 2007) and Member of Romanian Association of Fracture Mechanics, ARMR (2008).



Raluca Ioana Zamfir was born in Bucharest, Romania, on 18th May 1972. She graduated in Metallurgical Engineering field in 1997 at the University "Politehnica" of Bucharest, Romania at Faculty of Material Science and Engineering, University "Politehnica" of Bucharest. She graduated in Masters Degrees in 1998 in the field Ferrous metal materials with special purpose, at the same university.

In 2009, she received her Ph.D. in Metallurgy (Material Science and Engineering field) from the same University "Politehnica" of Bucharest (UPB) with a thesis concerning the "Contributions to the study of structural and operational characteristics of laser surface treated austenitic stainless steels". Between 1997 -2000 she worked as engineer at the University "Politehnica" of Bucharest (Romania), Faculty of Material Science and Engineering, "Material Science and Physical Metallurgy" Department. From 2000 - to the present she was Assistant Professor and Lecturer at the same university. Lecturer Dr. Eng. Raluca Zamfir has taught many courses at the University of "Politehnica" of Bucharest (i.e. Electrochemical and corrosion protection materials, Degradation and protection materials, Unconventional heat treatments, Degradation and protection of biomaterials, In biomaterial surface engineering). Beginning with 2006 she becomes the scientific coordinator of licenes students at UPB in Physical Metallurgy, Unconventional (i.e. Laser Surface) Heat Treatments and Corrosion fields. The research activities are in the field of Surface Engineering, Unconventional Heat Treatments, Biocompatible Materials, Biocompatible Materials Corrosion and so on. Lecturer Dr.Eng. Raluca Ioana Zamfir has published many scientific research papers, and research projects. She is Member of the Romanian Society for Biomaterials.

Alina Maria Molagic was born in Bucharest, Romania May 27th 1974. She graduated in Metallurgical Engineering field in 1999 at the University "Politehnica" of Bucharest, Romania at Faculty of Material Science and Engineering, University "Politehnica" of Bucharest. She graduated in Masters Degrees in 2000 in the field "Ferrous metal materials with special purpose", at the same university. In 2010, she received her Ph.D. in Metallurgy (Material Science and Engineering field) from the same University "Politehnica" of Bucharest (UPB) with a thesis concerning the "Collection and characterization of biocompatible layers deposited on Ti-6Al-4V alloy by magnetron sputtering method under radio frequency". Dr.Eng. Alina Maria Molagic has published many scientific research papers, and research projects (some as responsible of project). She is Member of the Romanian Society for Biomaterials.

Vasile Bratu was born in Romania January 23th 1966. He graduated in Metallurgical Engineering field in 1990 at the University "Politehnica" of Bucharest, Metallurgy Faculty, Syderurgy Specialization. In 2003 he received her Ph.D Degree, in Metallurgy (Material Science and Engineering field) from the same University "Politehnica" of Bucharest (UPB) with a thesis concerning the "Research on improving the quality of continuously cast billets". Beginning 1993 to present teaching in Materials Engineering: - Mathematical modeling and simulation; Physical - Chemical Processes of Continuous Casting Billet; Optimization of solidification; and „Developing and refining steel” fields. Associate Professor Vasile Bratu has published about 50 scientific research papers, and research projects (some responsible of project). In present he is the Dean of the Materials Engineering, Mechatronic and Robotics Faculty, University "Valahia" from Targoviste.