National University of Science and Technology
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PhD Thesis

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Author: Eng. Andrei Valeriu

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Influence of thermomechanical processing conditions on microstructural and mechanical characteristics of 7075 alloy

Drd. Ing. Valeriu ANDREI

Abstract

In this thesis, different experimental programs were developed with the aim of designing suitable thermomechanical processing routes to correlate the effects induced by thermomechanical processing on the microstructure and mechanical properties of aluminum alloy 7075, to obtain a suitable combination of mechanical strength properties and resilience. The developed experimental programs show a high level of complexity. The processing routes were based on the following main parameters: the deformation temperature of the 7075 alloy in the range of 225°C to 475°C, the solution quenching treatment temperature in the range of 450°C to 500°C with solution quenching treatment time of 10 minutes, artificial aging treatment temperature in the range of 100°C to 150°C with artificial aging treatment time of 12 hours. The microstructure of alloy 7075 is made up of the following phases/compounds: : α-Al, phase - η (MgZn2); phase - S (Al2CuMg); phase - T (Al2Mg3Zn3); phase - θ (Al2Cu3) and intermetallic compounds of the Al-Mn-Cr-Fe type: Al6(Fe,Mn), Al5Si2(Fe,Mn), Al3(Fe,Mn,Cr); The mechanical properties obtained by tensile testing (yield strength, ultimate tensile strength and elongation at break) and Charpy impact testing (absorbed energy and elasticity) are suffering changes depending on the applied thermomechanical processing route to the 7075 alloy.

Keywords: aluminum alloy 7075; thermomechanical processing; phases/compounds; mechanical properties.

Table of Contents

List of abbreviations and symbols

CFC	Cube with centered faces
ASTM	American Society for Testing and Materials
PTM	Thermomechanical processing
PTMI	Intermediate thermomechanical processing
PTMF	Final thermomechanical processing
HR	Hot rolling
ST	Solution treatment
AT	Ageing treatment
AR	As received
SEM	Scanning electron microscopy
EDS	Energy dispersive spectroscopy
XRD	X-ray diffraction
OM	Optical microscopy
α-Al	Basic phase alpha - aluminum
η	Eta phase ($MgZn_2$)
θ	Teta phase (Al_2Cu_3)
S	S phase (Al_2CuMg)
T	T phase ($Al_2Mg_3Zn_3$)

Introduction

With the rapid development of technology in all branches of industry, it is necessary to know and study as fully as possible the properties of the metals used in the construction of mechanical, electrical and electronic machines and apparatus, and installations for the production, transformation and transport of electrical energy, in order to use them with great efficiency.

One of the most widely used metals, because of its special properties, is aluminium. It can be said that no branch of modern industry can develop without the use of aluminium and its alloys in the form of cut, forged or cast ingots, sheet, strip, wire, foil, profiles, etc. This also explains the rapid development of aluminium production worldwide. To increase strength, aluminium is alloyed with various alloying elements, with 7075 alloy being chosen for the research topic. The choice of the research topic was made due to the development and use of 7075 alloy in various fields of activity such as aeronautics and military. Alloy 7075 is an aluminium alloy with zinc as the main alloying element. It contains very good mechanical characteristics and ductility, high strength, toughness and good fatigue strength.

Due to the applicative nature of the PhD thesis, a major role in the PhD thesis is played by thermomechanical processing at different temperatures for alloy 7075.

In order to achieve the objectives of the PhD thesis, a complex research programme has been carried out, comprising 3 stages of mechanical processing (plastic deformation by hot rolling) and thermal processing (solution treatment and artificial ageing treatment).

The structure of the thesis consists of three parts. The first part contains the overview and introduction, the metallurgical view of aluminium and its alloys, their properties and use, an overview of mechanical and thermal processing operations.

The second part contains the objectives of the thesis, sample processing methodology, advanced characterisation and research concepts used.

The third part covers microstructural evolution during thermomechanical processing, evolution of mechanical properties, processing and general conclusions, personal contributions, recommendations and future research directions.

The thesis concludes with a list of references, appendices and list of publications/dissemination of results.

Part I: Presentation of the PhD topic

Chapter 1. Influence of thermomechanical processing conditions on microstructural and mechanical characteristics of 7075 alloy

1.1. Obtaining aluminium

Aluminium is one of the most common metals in the Earth's crust (7.45%) and ranks third after oxygen and silicon. It exists in compound form in nature due to its high chemical activity.

Aluminium produced by electrolysis has a 99.5% to 99.8% aluminium content, contains both metallic and non-metallic impurities, but can be purified by various methods.

Depending on the method of production and the chemical composition, aluminium can be classified as:

- technical purity aluminium containing 0.15 - 1% impurities. It is intended for processing by rolling at high temperatures (hot rolling) or low temperatures (cold rolling) and in foundries. It is also used to produce aluminium powders and various aluminium alloys;
- high purity aluminium, containing 0.005 - 0.05% impurities. It is used in the production of special chemical devices, electrical capacitors, etc.;
- extra-pure aluminium, which contains no more than 0.0001% impurities, is obtained by zonal melting, is used for scientific research, in nuclear technology, in semiconductor technology.

Aluminium is easily alloyed with elements such as bromine, chromium, iron, manganese, nickel, titanium, zirconium and forms phases that may or may not dissolve in the aluminium matrix, leading to increases in strength and hardness as well as increasing the hot stability of alloys [1,3-5].

1.2. Physical, physico-chemical and mechanical properties of aluminium

The physical, physico-chemical and mechanical properties of various aluminium products can be influenced by impurities. The most common impurities in aluminium are iron and silicon.

Iron cannot dissolve in aluminium and forms 7% Al_3Fe (1.7% Fe) and eutectic Al-Al_3Fe. The iron impurity in aluminium at the microscopic level consists of polyhedral grains of aluminium and acylform Al3Fe precipitates. The simultaneous presence of iron and silicon produces two new phases, α-phase (Fe_3SiAl_3) and β-phase ($FeSiAl_5$), which do not occur in binary alloys. These compounds located at the boundaries of the aluminium grains reduce the plasticity of aluminium [6].

1.3. Classification of aluminium alloys

Aluminum is associated with the III-th group of the periodic table of elements, contains one stable isotope ^{27}Al and five unstable isotopes (^{24}Al, ^{25}Al, ^{26}Al, ^{28}Al) with half-reduction times ranging from 2.10 seconds to 394 seconds. Aluminium is defined by high malleability, low toughness, high specific electrical conductivity, high thermal conductivity and very high corrosion resistance in the environment and organic acids. Aluminium has a density of only 2.7 g/cm3, about one third of that of steel (7.83 g/cm^3).

The main characteristics of aluminium are shown in Table 1.2.

***Table 1.1**. Characteristics of aluminium [18].*

Property	**Values**
Atomic number	Z=13
Atomic mass	A=26.98 at/g
Crystal lattice	CFC
Lattice parameter (at 20ºC)	a= 4.04Å
Melting temperature	660ºC
Boiling point	2518ºC
Density	2.7 g/cm³
Thermal conductivity	237W

Aluminium alloys processed by remelting are divided into three groups:

- deformable alloys without solid phase transformations;
- deformable alloys with solid phase transformations, suitable for structural hardening by solution annealing and ageing heat treatments;
- casting alloys with eutectic transformation on solidification and partial solid-state transformation, some of which are suitable for hardening by quenching and ageing heat treatments [25].

Aluminium alloys are classified as: heat treatable and non-heat treatable deformable alloys, heat treatable and non-heat treatable cast alloys.

***Table 1.2.** Classification of aluminium-based alloys[16].*

	Class	**Series**	**Principal alloying elements**
DEFORMABLE	Heat treatable	2xxx	Cu, (Mg)
		6xxx	Mg, Si
		7xxx	Zn, Mg, (Cu)
	Non heat treatable	1xxx	Al
		3xxx	Mn, (Mg)
		5xxx	Mg
CAS		2xxx	Cu

	Heat treatable	3xxx	Si, Cu/Mg
		7xxx	Zn, (Mg)
	Non heat treatable	1xxx	Al
		4xxx	Si
		5xxx	Mg

1.4. Structural characteristics of aluminium alloys

The modification of structural properties in rolling and thermal processes takes into account the properties presented below:

a) Microstructure - the size, configuration and orientation of grains relative to the rolling direction;

b) Substructure - the internal structure of deformed grains, as distinguished from recrystallized grains;

c) Texture - the crystallographic orientation of the grains.

Table 1.4 contains several phases corresponding to 7xxx series aluminium alloys according to the Thermo-Calc database [7, 30, 41].

Table 1.3. *Common phase names corresponding to 7xxx series aluminium alloys [30].*

Phase name	Common Name and Description
$Al_{15}Si_2M_4$	A cubic precipitate, which originates from the Al-Mn-Si ternary system, aka τ9, $Al_{15}Mn_3Si_2$, $Al_{16}Mn_4Si_3$ or $Al_{15}Mn_4Si_2$. Mn can be substituted by Fe, as well as Cr and Mo. The phase observed in aluminum alloys is also designated as α.
$Al_{13}Fe_4$	An iron aluminide, which often forms as a primary phase during casting, aka Al_3Fe.
Al_2Cu – C16	The so-called θ-Al_2Cu phase that forms in many Cu-containing aluminum alloys.
Al_2Cu_OMEGA	Ω-Al_2Cu, a metastable precipitate and the coherent version of the θ phase
θ' (theta_prime)	A semi-coherent precipitate with a stoichiometry of Al_2Cu in α-(Al), i.e. the GPI zones.
THETA_DPRIME	Coherent metastable precipitates in α-(Al), also referred to as GPII zones. It has a stoichiometry close to Al_3Cu.
BETA_DPRIME	Metastable precipitate β'' related to Mg_2Si that forms in Al-Mg-Si based alloys. It may contain Al atoms ($Al_2Mg_5Si_4$) or be Alfree (Mg_5Si_6).
β' (beta_prime)	Metastable precipitate β' related to Mg_2Si, aka Mg_9Si_5/Mg1.8Si
U1_Al_2MgSi_2	An Al-containing pre-β Al-Mg-Si metastable precipitate, U1_Al_2MgSi_2
U2_$Al_4Mg_4Si_4$	An Al-containing pre-β Al-Mg-Si metastable precipitate, U2_$Al_4Mg_4Si_4$
$Al_{18}Fe_2Mg_7Si_{10}$	A quaternary phase, aka $Al_8FeMg_3Si_6$, Q, PHI and H_PHASE

Al_6Mn	A common Al-Mn compound that forms in Mn-containing aluminum alloys. Mn could be substituted by Cu and Fe, especially to a larger extent by the latter.
$Al_{28}Cu_4Mn_7$	An Al-Cu-Mn intermetallic phase that forms in aluminum alloys
Q_AlCuMgSi	A stable Al-Cu-Mg-Si quaternary phase, aka Q, $Al_5Cu_2Mg_8Si_6$, $Al_3Cu_2Mg_9Si_7$ and $Al_4Cu_2Mg_8Si_7$
QPRIME	The coherent / semi-coherent version of Q_ALCUMGSI
Mg_2Si – C1	Mg2Si, which forms in Mg- and Si-containing aluminum alloys
$Al_9Fe_2Si_2$	A common Al-Fe-Si ternary phase in aluminum alloys, aka τ6, Al5FeSi, β-AlFeSi
Al_8Fe_2Si	A common Al-Fe-Si ternary phase in aluminum alloys, aka τ5, α-AlFeSi
Al_7Cu_2Fe	An Al-Cu-Fe ternary compound that may form in some aluminum alloys
DIAMOND_A4	Si, as well as C and Ge
C14 - LAVES	A common stable precipitate in 7000 series aluminum alloys, aka. the η (MgZn2) phase, eta and the M phase. This phase includes all MgZn2-type phases.
η' (eta-prime)	The metastable η' phase, which is related to the η-$MgZn_2$ phase.
T	A stable phase in Al-Mg-Zn, Al-Cu-Mg and Al-Cu-Mg-Zn. It is modeled as $(Al,Cu,Zn)_{49}Mg_{32}$ and is often designated as $Al_2Mg_3Zn_3$ in aluminum alloys.
T’	The metastable form of T phase, T
S	The S phase, Al_2CuMg
S’	The metastable S’ phase, precursor to the S phase
Q_AlCuMgSi	The Al-Cu-Mg-Si quaternary phase, i.e. the Q Phase, aka $Al_5Cu_2Mg_8Si_6$, $Al_3Cu_2Mg_9Si_7$ and $Al_4Cu_2Mg_8Si_7$
Q_$Al_7Cu_3Mg_6$	An Al-Cu-Mg ternary phase, aka, $Al_7Cu_3Mg_6$ and the Q phase

In the quaternary alloy system where aluminium together with zinc, magnesium and copper form the alloy called zicral, the $MgZn_2$ phase improves the hardness of the material, the hardening phase Al_2Cu gives good plasticity but low temperature resistance. Iron and silicon decrease plasticity, manganese neutralises the unwanted influence of iron, increasing hardness and decreasing corrosion resistance.

1.5. Thermal processing of aluminium-based alloys

Heat treatment is a process that involves different levels of heating and cooling to alter the physical properties of metals.

Depending on the characteristics pursued, heat treatments are classified into:

- annealings that aim to restore the structure and physical-chemical properties of the material;
- solution annealing to obtain a supersaturated solid solution;
- ageing applied after solution quenching in order to restore the material to equilibrium with structural hardening (precipitation hardening) [44].

Heat treatments applied to aluminium-based alloys have two purposes:

- restoration of plasticity for subsequent plastic deformation;
- hardening of alloys to obtain maximum or optimum mechanical characteristics and association with other characteristics such as creep resistance, high temperature, dimensional stability, fatigue strength, or physicochemical properties (corrosion resistance, under load, appearance after anodizing) [45].

1.6. Mechanical processing of aluminium-based alloys

There are several types of mechanical processing of aluminium-based alloys, the most important of which are: milling, plating, hot rolling and cold rolling. For each type of mechanical processing there is a specific technology depending on the form of the raw material (ingot, slab, bar), the type of alloy and the requirements of the intermediate or final customer.

1.7. Thermomechanical processing of aluminium-based alloys

Thermomechanical processing (TMP) of aluminium alloys has been relatively little researched and applied in our country, despite the fact that it requires more practical use than conventional processes.

The benefits provided by structural properties can be exploited in two types:

- the use of adhesive coatings at the expense of mechanical ones, which leads to reduced internal stress concentration, increased fatigueability, simplification of the rolling process itself and, finally, improved crack resistance;
- properties obtained by metallurgical techniques.

1.8. Use of aluminium-based alloys

From 1940 onwards, alloys of the 7075 type began to be introduced into aircraft construction, with a mechanical strength almost double that of the alloys previously used.

From the 1950s onwards, most aircraft manufacturers stopped using 7075 alloys and only used 2xxx series alloys for critical structures. Alloy 7075 has been improved by improving the knowledge of the protective treatment. Since the 1970s, alloy 7075 has been gradually reintroduced [111].

1.9. Alloy 7075

Alloy 7075 is an aluminium alloy with zinc as the main alloying element. It contains very good mechanical characteristics and ductility, high strength, toughness and good fatigue strength. It is more susceptible to embrittlement than many other aluminium alloys due to micro-segregation, but has significantly better corrosion resistance than Al-Cu alloys.

The composition of this alloy includes the following elements in the following ranges: 5.6-6.1% zinc, 2.1-2.5% magnesium, 1.2-1.6% copper and less than half a percent of silicon, iron, manganese, titanium, chromium and other metals [118].

The figure below shows the Curiosity rover's 7075 aluminum alloy wheel before it was attached to the rover and sent to Mars, labeled with all its component parts [125].

Figure 1.1. *Curiosity aluminium alloy wheel 7075-T7351 [125].*

Part II: Research objectives, methods and concepts used

Chapter 2. Research objectives, methods and concepts

2.1. Research objectives

The objectives of the thesis are classified as follows:

- to have a better understanding of how to properly process 7075 alloy using different thermomechanical processing parameters and to quantify the induced effects on the alloy microstructure and its mechanical behaviour;
- optimise the final microstructural and mechanical characteristics of 7075 alloy;
- use various investigation techniques such as optical and electron microscopy for microstructural properties and tensile and impact testing for mechanical characteristics.

2.2. Structure of the PhD thesis

The structure of the thesis consists of three parts. The first part contains the overview and introduction, the metallurgical view of aluminium and its alloys, their properties and use, an overview of mechanical and thermal processing operations.

The second part contains the objectives of the thesis, sample processing methodology, advanced characterisation and research concepts used.

The third part covers microstructural evolution during thermomechanical processing, evolution of mechanical properties, processing and general conclusions, personal contributions, recommendations and future research directions.

The thesis concludes with a list of references, appendices and list of publications/dissemination of results.

2.3. folosite Research methods and concepts used

In order to achieve the objectives of the PhD thesis, a complex research programme was carried out, comprising mechanical processing (plastic deformation by rolling) and thermal processing (solution hardening and ageing) stages. Figure 2.1 shows the scheme of the experimental programme used to investigate the influence of thermomechanical processing conditions on the microstructural and mechanical characteristics of alloy 7075.

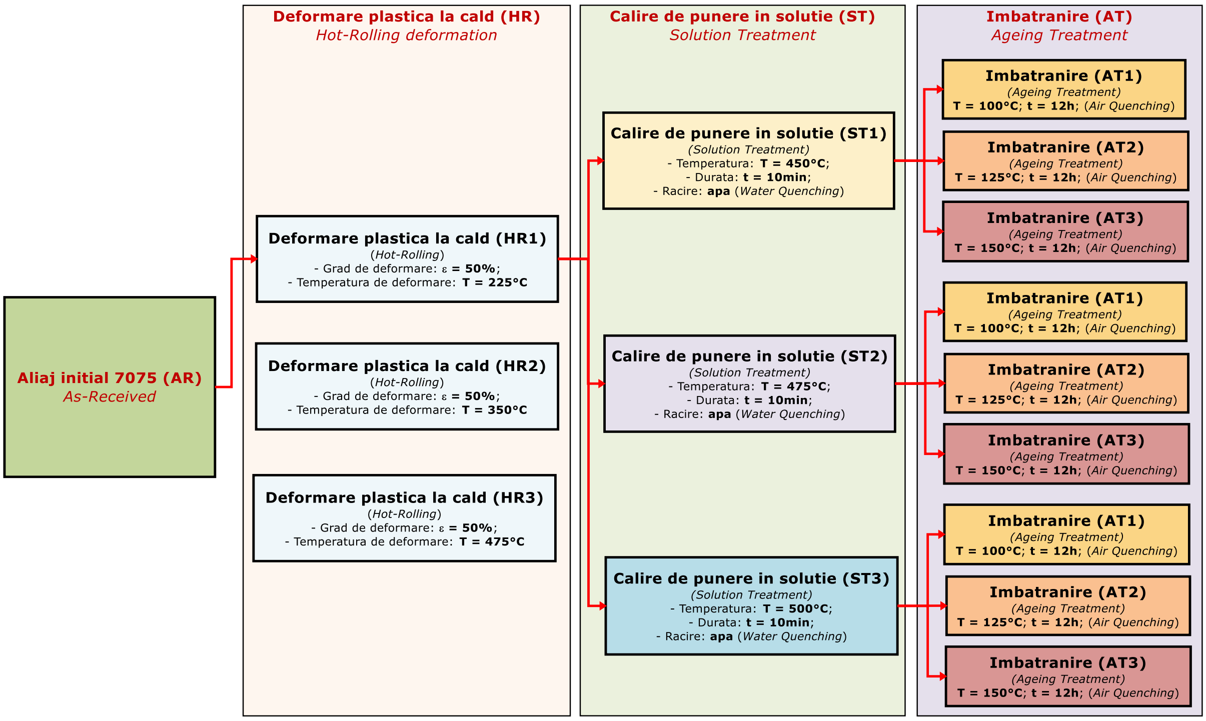


Figure 2.1. *Scheme of the experimental program used to highlight the influence of thermomechanical processing conditions on the microstructural and mechanical characteristics of alloy 7075.*

Hot plastic deformation (HR)

A number of 3 structural states (HR1, HR2, HR3) resulted from hot plastic deformation. For each structural state, 12 samples were heated and hot plastic deformed. The holding time in the heating oven for hot plastic deformation was 2.5 min/mm (t = 30 min).

Solution treatment (ST) and ageing treatment (AT)

The solution treatment and ageing treatment resulted in 9 structural states and 27 structural states, respectively.

The variation parameter of the solution treatment is the treatment temperature, which was located at 450°C (structural condition - ST1), 475°C (structural condition - ST2) and 500°C (structural condition - ST3), the treatment time in all cases being t = 10min.

The parameter of variation of the ageing treatment is the treatment temperature, which was located at 100°C (structural condition - AT1), 125°C (structural condition - AT2) and 150°C (structural condition - AT3), the treatment duration in all cases being t = 12.

Part III: Results and conclusions

Chapter 3. Microstructural and mechanical characterisation of 7075 alloy in initial state

3.1. Microstructural characterisation of 7075 alloy in initial state

The as-received (AR) 7075 alloy has been fully microstructurally characterized. Microstructural characterisation was carried out using the following investigation techniques: X-ray diffraction (XRD), optical microscopy (OM) and scanning electron microscopy (SEM).

The chemical composition of the 7075 alloy used is shown in Table 3.1. It can be seen that it is mainly alloyed with Zn (5.57%gr), Mg (2.38%gr) and Cu (1.38%gr). Also, in the chemical composition of alloy 7075, Fe (0.25%gr), Cr (0.19%gr), Si (0.16%), etc. are found in smaller quantities.

Table 3.1. *Chemical composition of alloy 7075 in AR structural state.*

Zn, %gr	Mg, %gr	Cu, %gr	Fe, %gr	Cr, %gr	Si, %gr	Mn, %gr	Ti, %gr	V, %gr	Al, %gr
5,57	2,38	1,38	0,25	0,19	0,16	0,095	0,029	0,01	rest

XRD analysis of 7075 alloy in its initial state (AR) (Figure 3. 1), showed that the following phases and compounds are present in its microstructure: α-Al, phase - η ($MgZn_2$); phase - S (Al_2CuMg); phase - T ($Al_2Mg_3Zn_3$); phase - θ (Al_2Cu_3) and Al-Mn-Cr-Fe intermetallic compounds: $Al_6(Fe,Mn)$, $Al_5Si_2(Fe,Mn)$, $Al_3(Fe,Mn,Cr)$, etc. XRD analysis also shows that the majority of the phase present consists of the base phase α-Al.

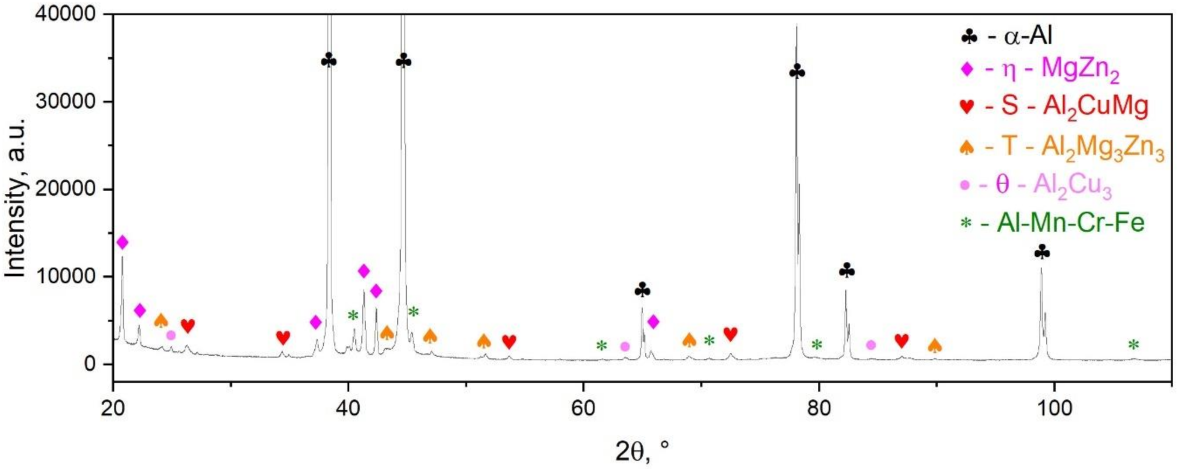
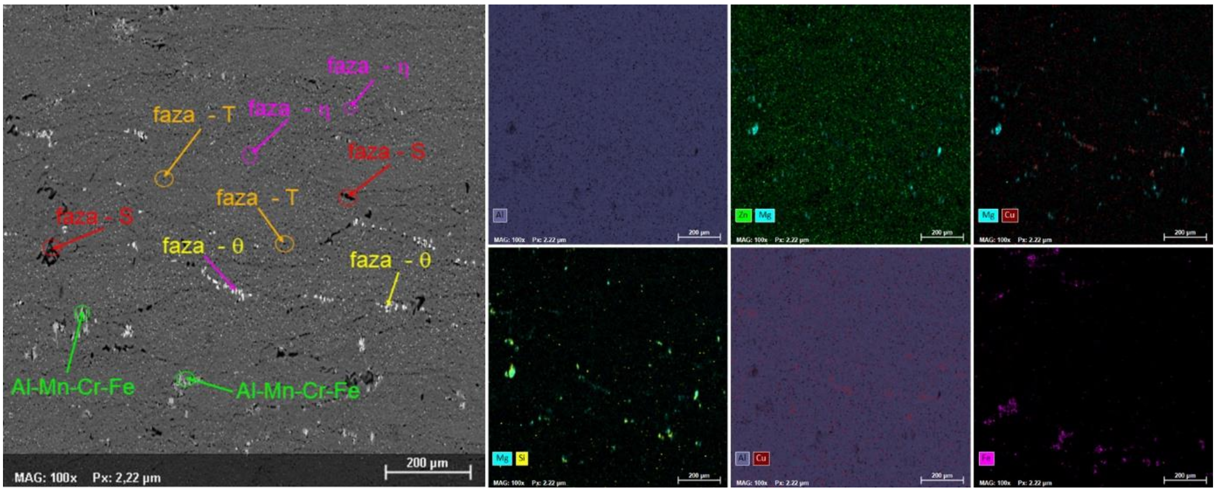


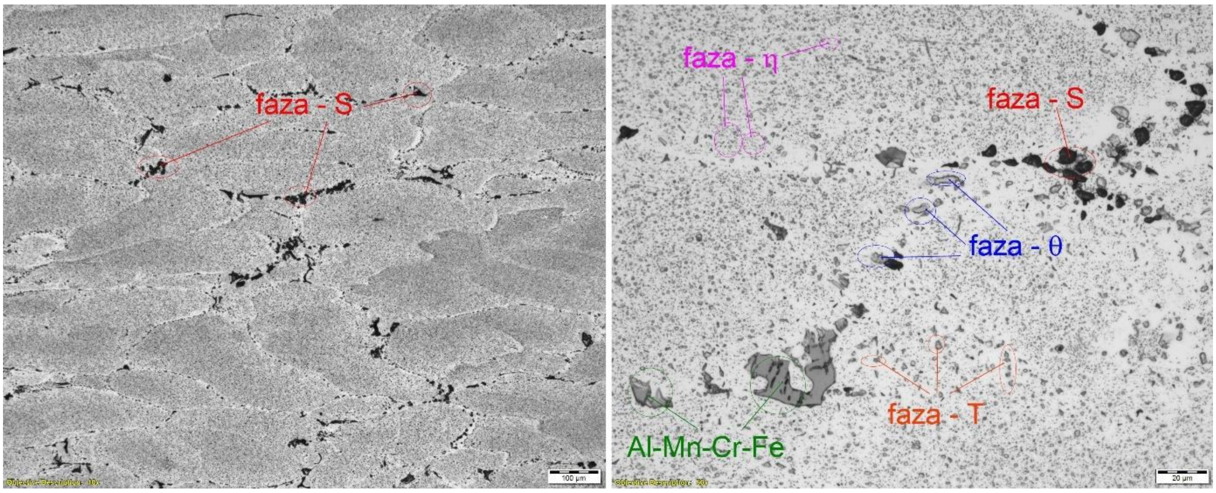
Figure 3.1. *XRD spectrum of 7075 alloy in initial state (AR)*

SEM electron microscopy analysis of the microstructure of the 7075 alloy in its initial state (AR) (Figure 3.2) confirms the observations from XRD analysis, showing that a number of secondary phases and compounds are dispersed in the base mass (α-Al phase), as follows: η-phase ($MgZn_2$); S-phase (Al_2CuMg); T-phase ($Al_2Mg_3Zn_3$); θ-phase (Al_2Cu_3) and intermetallic compounds of the Al-Mn-Cr-Fe type. It is also observed that the η ($MgZn_2$) and T ($Al_2Mg_3Zn_3$) secondary phases exhibit spheroidal morphology and submicron size, while the S (Al_2CuMg), θ (Al_2Cu) and Al-Mn-Cr-Fe type intermetallic compounds phases exhibit spheroidal/elongated morphology and micron size.



a) ***b)***

Figure 3.2. *SEM image of the microstructure of alloy 7075 in its initial state (AR) - a ; dispersion of the main alloying elements in the base mass – b.*



a) ***b)***

Figure 3.3. *Optical microscopy image of the microstructure of alloy 7075 in its initial state (AR); a: X100 ; b: X500.*

Analysis of the microstructure of 7075 alloy in its initial state (AR) using optical microscopy (Figure 3.3) confirms the observations from XRD and SEM electron microscopy. Again, it is observed that in the base mass grains (α-Al phase) η (MgZn2) and T ($Al_2Mg_3Zn_3$) phases are present dispersed, while at the grain boundary S (Al_2CuMg), θ (Al_2Cu_3) phases and Al-Mn-Cr-Fe intermetallic compounds are present. Optical microscopy also confirms that the secondary η ($MgZn_2$) and T ($Al_2Mg_3Zn_3$) phases exhibit spheroidal morphology and submicron size, while the S (Al_2CuMg), θ (Al_2Cu) and Al-Mn-Cr-Fe intermetallic compounds exhibit chunky/elongated morphology and micron size.

3.2. Mechanical characterisation of 7075 alloy in initial state

The as-received (***AR***) 7075 alloy has been fully mechanically characterized. The mechanical characterisation was carried out using tensile testing and Charpy impact testing. For statistical relevance all mechanical tests were performed in duplicate. Figure 3.4 shows the appearance of the resulting typical stress-strain curves. It can be seen that the 7075 alloy in its initial state (***AR***) shows high ductility, with elongation at break having a higher value (18-20)%. It is also observed that the maximum strength limit exceeds the value/threshold of 250MPa.

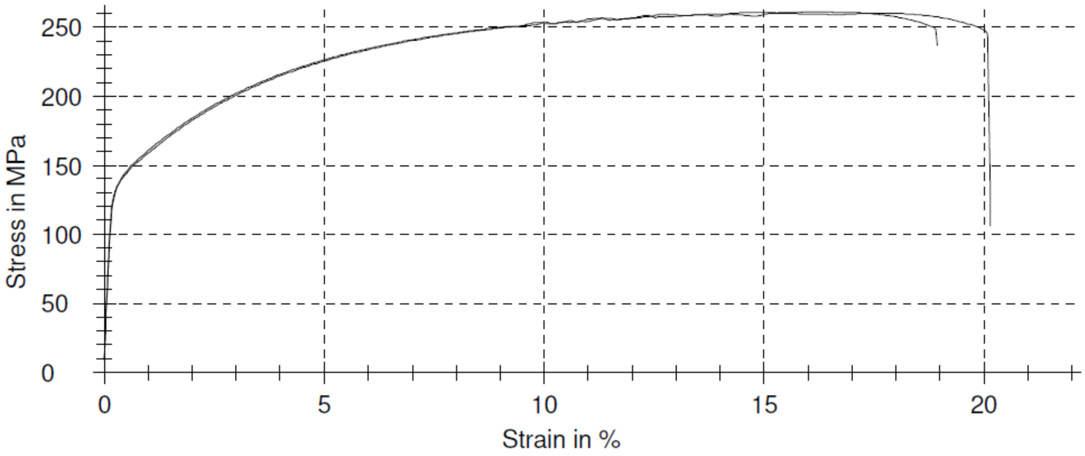


Figure 3.4. *Typical stress-strain curves for 7075 alloy in initial state.*

Table 3.2. *Mechanical characteristics for alloy 7075 in AR structural state.*

Samples number	**Mechanical properties**				
	Tensile testing			***Resilience***	
	Ultimate tensile strenght, Rm [MPa]	*Yield strenght, $Rp_{0.2}$ [MPa]*	*Elongation, A_{50} [%]*	*Absorbed energy, E [J]*	*KCV, El [j/cm^2]*
0.1	261	145	17	6,53	16
0.2	261	144	17,5	6,66	16
Average value:	***261***	***144,5***	***17,25***	***6,59***	***16***

Table 3.2 shows the mechanical characteristics determined from the analysis of the tensile (stress-strain) curves and the impact tests. It can be seen that for both tests (tensile and resilience) the values of the mechanical characteristics obtained are very close/approximately identical, which shows/confirms that the 7075 alloy in its initial state (***AR***) shows a homogeneous microstructure throughout the base mass.

Chapter 4. Influence of hot plastic deformation temperature on microstructural and mechanical characteristics of 7075 alloy

In order to study the influence of hot plastic deformation temperature on the microstructural and mechanical characteristics of 7075 alloy, the first stage of experiments was carried out:

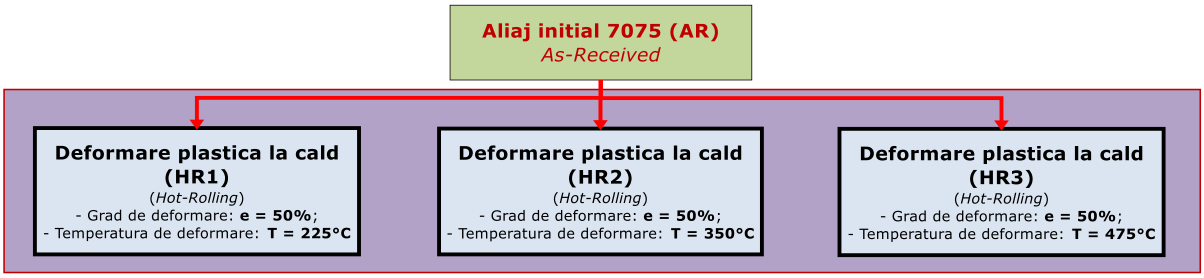


Figure 4.1. *Thermomechanical processing scheme (first stage) applied to alloy 7075 to determine the influence of hot plastic deformation temperature on microstructural and mechanical characteristics.*

The main parameter that has been taken into account in the design of the applied thermo-mechanical machining routes is the recrystallization temperature of the 7075 alloy, which is close to 400°C, with mechanical machining experiments being carried out at temperatures below, near and above the recrystallization temperature.

The first experimental step (see Fig. 4.1) consists of mechanical processing (by hot plastic deformation - HR) of the as-received (***AR***) 7075 alloy in a wide temperature range, from 225°C - well below the recrystallization temperature (~400°C) to 475°C - well above the recrystallization temperature (~400°C).

In all cases, hot rolling was carried out by rolling with a total degree of deformation of 50%, with hot rolling temperature (***HR***) as a parameter of variation, this being at 225°C (structural condition - ***HR1***), 350°C (structural condition - ***HR2***) and 475°C (structural condition - ***HR3***).

Since the holding time at hot prerolling temperatures can drastically influence the microstructure of the alloy, in order to have a basis for comparison, it was chosen to apply the same hot holding time for all deformation temperatures (225°C - ***HR1***, 350°C - ***HR2*** and 475°C - ***HR3***) of 2.5 min/mm sample thickness (t = 30 min).

4.3. Conclusion

For the first stage of the thermomechanical processing scheme applied in the present study in order to investigate the developments in the microstructure and mechanical properties of 7075 alloy, the following conclusions were found:

- During the hot plastic deformation process, carried out at different temperatures in the range 225°C - 475°C, different deformations of the texture of the initial grain colonies occurred along the rolling direction, increasing with increasing rolling temperature;
- The analysis of the optical microstructure of the 7075 alloy in the hot plastically deformed state (***HR***) confirms the observations resulting from the XRD analysis and the SEM electron microscopy, the following secondary phases being observed in the grains of the base mass (the α-Al phase): phase - η (MgZn2); phase - S (Al2CuMg); phase - T (Al2Mg3Zn3); phase - θ (Al2Cu3) and intermetallic compounds of the Al-Mn-Cr-Fe type;
- As the hot plastic deformation temperature increases, the phases and compounds in the base mass of the 7075 alloy change their shape, size and dispersion;
- The crystallographic parameters of the α-Al base phase related to the hot plastically deformed 7075 alloy at different temperatures evolved with the increase of the rolling temperature by increasing the α-Al crystal lattice parameter, increasing the average size of the coherent α -Al crystalline domain and decreasing the degree of residual deformation of the elementary cell α-Al;
- After analyzing the stress-strain curves, it is found that the 7075 alloy in the hot rolling (***HR***) state has a high ductility, the elongation at break having a high value (15-18)%, it is also observed that the maximum strength limit exceeds the value/threshold of 300Mpa;
- The mechanical properties of alloy 7075 in hot rolling state (***HR***) have evolved as follows:
    - the most advantageous value obtained for mechanical tensile strength was for sample ***HR3*** (hot rolling: T = 475°C; ε = 50%) with a value of RM = 344 MPa, 24.13% higher than the value of sample ***HR2*** (hot rolling: T = 350°C; ε = 50%) and against the value of sample ***AR*** (initial state); mechanical strength properties increase with increasing rolling temperature;
    - the most advantageous value obtained at the yield point was for sample ***HR1*** (hot rolling: T = 225°C; ε = 50%) with a value of Rp0.2 = 249 MPa, 17% higher than the value of the sample ***HR2*** (hot rolling: T = 350°C; ε = 50%) and 41.97% higher

than sample AR (initial state); with the increase of the rolling temperature, the properties of the yield point decrease;

- the most advantageous elongation value obtained was for sample ***HR3*** (hot rolling: T = 475°C; ε = 50%), with a value of A50 = 16%, 59.37% higher than sample ***HR1*** (hot rolling: T = 225°C; ε = 50%) and 7.8% lower than ***AR*** sample (initial state); the increased elongation values for the mentioned samples are due to the ductility of the material, which is good in the mechanically processed state with the increase of the rolling temperature;

  by comparison with the reference sample ***AR***-17% it can be claimed that the material shows a very good ductility in a mechanically unprocessed state;
- the most advantageous value obtained for the absorbed energy was for sample ***HR3*** (hot rolling: T = 475°C; ε = 50%) with a value of E = 6.5 J, 9.23% higher than the value of sample ***HR1*** (hot rolling: T = 225°C; ε = 50%) and 1.38% lower than the ***AR*** sample value (initial state);
- the most advantageous value obtained for elasticity was for sample ***HR3*** (hot rolling: T = 475°C; ε = 50%) with a value of El = 17 J/cm2, 11.76% higher than the value of the sample ***HR1*** (hot rolling: T = 225°C; ε = 50%) and 5.88% higher than the value of sample ***AR*** (initial state); increasing the rolling temperature improves the resilience properties;

Chapter 5. The influence of the solution treatment temperature on the microstructural and mechanical characteristics of the 7075 alloy

To study the influence of the temperature of the solution quenching heat treatment on the microstructural and mechanical characteristics of the 7075 alloy, the second experimental stage was carried out:

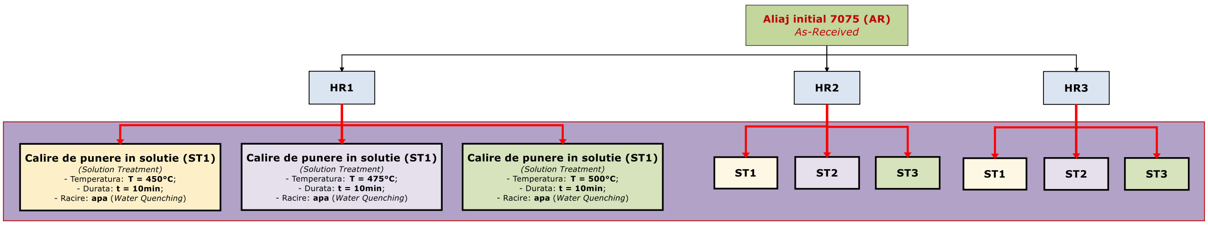


Figure 5.1. *Thermomechanical processing scheme (second experimental stage) applied to alloy 7075 to determine the influence of solution quenching heat treatment temperature on microstructural and mechanical characteristics.*

The second experimental stage (see Fig. 5.1) consists of thermal processing experiments developed at temperatures below, close to and above the eutectic temperature – solidus line (~ 477°C), because alloy 7075 possesses a wide range of temperatures for the region between the solidus line (~477°C) and the liquidus line (~652°C), which allows it to be processed in a semi-solid state.

Considering that thermal processing can influence the microstructure of the alloy, in order to have a basis for comparison, it was chosen to apply solution treatment after mechanical processing (hot plastic deformation), at temperatures of 450°C (state structural state – ***ST1***), 475°C (structural state – ***ST2***) and 500°C (structural state – ***ST3***), the treatment duration being in all cases t = 10min.

5.4. Conclusions

For the second stage of the thermomechanical processing scheme applied in the present study in order to investigate the evolutions in the microstructure and mechanical properties of alloy 7075, the following conclusions were found:

- After the solution treatment, with the temperature increases from 450°C (***ST1***) to 500°C (***ST3***), the phases have a smaller and more spherical size; as the treatment temperature increases, the soluble phases dissolve into the solid solution.
- The high degree of prior deformation of the alloy gives a comminuted structure and, as a result, the rate of dissolution of secondary precipitates at the tempering temperature is higher.
- The analysis of the optical microstructure of alloy 7075 in the hot plastically deformed state (***HR***) and solution hardened (***ST***) confirms the observations resulting from the XRD analysis, being also observed in this case the following secondary phases in the grains of the base mass (phase α- Al): phase - η (MgZn2); phase - S (Al2CuMg); phase - T (Al2Mg3Zn3); phase - θ (Al2Cu3) and intermetallic compounds of the Al-Mn-Cr-Fe type;
- The crystallographic parameters of the α-Al base phase related to hot plastically deformed and solution quenched 7075 alloy at different temperatures evolved with increasing rolling and quenching temperature by increasing the α-Al crystal lattice parameter, increasing the average size of of the coherent crystalline domain α -Al and the decrease of the degree of residual deformation of the elementary cell α -Al;
- After analyzing the stress-strain curves, it is found that alloy 7075 in the hot rolling state (***HR***) and hardened (solution treatment - ***ST***) state has a high ductility, the elongation at break having a high value (7-15%), it is also observed that the maximum strength limit exceeds the value/threshold of 300Mpa;
- The mechanical properties of the 7075 alloy in the hot rolling (***HR***) and hardened (solution treatment – ***ST***) state evolved as follows:
    - a most advantageous value was obtained for the mechanical tensile strength, in sample ***HR3-ST3*** (hot rolling: T = 475°C; ε = 50% and solution quenching T = 500°C) with a value of RM = 337 MPa, 1.78% higher than the value of sample ***HR3-ST2*** (hot rolling: T = 475°C; ε = 50% and solution quenching T = 475°C) and 2.23% lower than the value of sample ***HR3*** (hot rolling: T = 475°C; ε = 50%); it is observed that the results of samples ***HR3-ST1, HR3-ST2, HR3-ST3*** have higher

values than the other tested samples, which results that with the increase of the rolling and tempering temperature, the mechanical resistance values increase;

  - the most advantageous value was obtained at the yield point, in sample ***HR1-ST3*** (hot rolling: T = 225°C; ε = 50% and solution quenching T = 500°C) with a value of Rp0. 2 = 259 MPa, 9.28% higher than the value of sample ***HR1-ST1*** (hot rolling: T = 225°C; ε = 50% and solution quenching T = 450°C) and 3.67 % higher than the value of sample ***HR1*** (hot rolling: T = 225°C; ε = 50%); it is observed that the results of samples ***HR1-ST1, HR1-ST2, HR1-ST3*** have higher values than the other tested samples, which results that rolling at a lower temperature and quenching at a high temperature influence the increase of properties at the yield point;
  - the most advantageous value was obtained for elongation, in sample ***HR3-ST3*** (hot rolling: T = 475°C; ε = 50% and solution treatment T = 500°C) with a value of A50 = 15% , 24.67% higher than the value of sample ***HR3-ST1*** (hot rolling: T = 475°C; ε = 50% and solution treatment T = 450°C) and 6.67% lower than value of sample ***HR3*** (hot rolling: T = 475°C; ε = 50%); increasing the thermomechanical processing temperature positively influences the elongation properties, the material becomes more ductile;
  - o the most advantageous values were obtained for absorbed energy and elasticity, for sample ***HR3-ST2*** (hot rolling: T = 475°C; ε = 50% and solution treatment T = 475°C) with a value of E = 11 J and El = 26 J/cm2 , 36.79% higher in E (energy absorbed at break) compared to the value of sample ***HR3-ST3*** (hot rolling: T = 475°C; ε = 50% and solution quenching T = 500°C) and 38.68% higher than the value of sample HR3 (hot rolling: T = 475°C; ε = 50%) and for El (elasticity) by 36.53 % higher than the value of sample ***HR3-ST3*** (hot rolling: T = 475°C; ε = 50% and solution quenching T = 500°C) and 34.61% higher than the value of sample ***HR3*** (rolling hot: T = 475°C; ε = 50%); with the increase of the rolling and tempering temperature, the resilience values increase; exceeding the eutectic during the solution quenching treatment considerably decreases the resilience properties of the material;

- With the increase in hot rolling temperature and solution treatment, the mechanical test results have improved considerably.

Chapter 6. Influence of aging treatment temperature on microstructural and mechanical characteristics of alloy 7075

To study the influence of the aging heat treatment temperature on the microstructural and mechanical characteristics of the 7075 alloy, the third experimental stage was carried out:

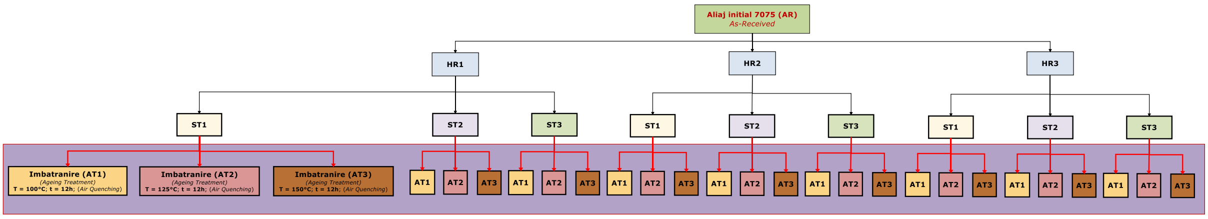


Figure 6.1. *Thermomechanical processing scheme (third stage) applied to alloy 7075 to determine the influence of aging treatment temperature on microstructural and mechanical characteristics.*

The third experimental stage (see Fig. 6.1) consists of thermal processing experiments developed at temperatures below, close to and above the germination temperature (T6 state – 120°C), because the 7075 alloy comprises a reduced range of temperatures for the region between below-aging (<100°C) and over-aging (>150°C). Given that thermal processing can influence the microstructure of the alloy, in order to have a basis for comparison, it was chosen to apply artificial aging thermal treatment after mechanical processing (hot plastic deformation) and solution treatment, at the temperatures of 100°C (structural state – ***AT1***), 125°C (structural state – ***AT2***) and 150°C (structural state – ***AT3***), the treatment duration being in all cases t = 12h.

6.10. Conclusions

For the third stage of the thermomechanical processing scheme applied in the present study in order to investigate the evolutions in the microstructure and mechanical properties of alloy 7075, the following conclusions were found:

- After artificial aging treatment, as the temperature increases from 100°C (***AT1***) to 150°C (***AT3***), the size and dispersion of the phases/compounds varies.
- The analysis optical microstructure of alloy 7075 in the aged state (ageing treatment - ***AT***) confirms the observations resulting from the XRD analysis of the other two previous experimental stages, the following secondary phases in the grains of the base mass being observed in this case as well (the α-Al phase): phase - η ($MgZn_2$); phase - S (Al_2CuMg); phase - T ($Al_2Mg_3Zn_3$); phase - θ (Al_2Cu_3) and intermetallic compounds of the Al-Mn-Cr-Fe type;

- After analyzing the stress-strain curves, it is found that alloy 7075 in hot rolling (***HR***), quenched (solution treatment - ***ST***) and aging treatment state (**AT**) has high ductility, elongation having a high value (7-15%), also it is observed that the maximum strength limit exceeds the value/threshold of 350 Mpa;
- The mechanical properties of alloy 7075 in hot rolling (***HR***), quneched (solution treatment - ***ST***) and aging treatment (***AT***) evolved as follows:
    - a most advantageous value was obtained for the mechanical tensile strength, for sample ***HR1-ST2-AT1*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 475°C and artificial aging T =100°C) with a value of RM = 377 MPa, 31.03% higher than the value of sample ***HR1-ST2-AT3*** (hot rolling: T = 225°C; ε = 50%, solution quenching T = 475°C and artificial aging T=150°C) and 15.78% higher than the value of sample ***HR1-ST2*** (hot rolling: T = 225°C; ε = 50%, solution quenching T = 475°C); it is observed that the most advantageous results (with values over 300 Mpa) are found in the samples where lamination was carried out at temperatures of 225°C and 450°C and artificial aging at a temperature of 100°C, which results that with the decrease in temperature of aging increase the mechanical resistance values.
    - the most advantageous value was obtained at the yield point, in sample ***HR1-ST3-AT1*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 500°C and artificial aging at 100 °C) with a value of $Rp_{0.2}$ = 260 Mpa, 23.07% higher than the value of sample ***HR1-ST3-AT3*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 500°C and artificial aging at 150°C) and 0.58% higher than the value of sample ***HR1-ST3*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 500 °C); it is observed that the most advantageous results (with values above 240 MPa) are found in the samples where the rolling was realised at the same temperature (225°C) and artificial aging at the temperature of 100°C, which results that with the decrease of the rolling temperature and aging, increase the values at the yield point; thermomechanical processing doubled the values obtained at the yield point compared to the values obtained in the initial sample (***AR***);
    - the most advantageous value was obtained for elongation, for sample ***HR3-ST3-AT1*** (hot rolling: T = 475°C; ε = 50%, solution quenching T = 500°C and artificial aging at 100°C) with a value of A50 = 15%, 20% higher than the value of sample ***HR3-ST3-AT2*** (hot rolling: T = 475°C; ε = 50% , solution quenching T = 500°C and artificial aging at 125°C) and identical value to sample ***HR3-ST3*** (hot rolling: T = 475°C; ε = 50%, solution treatment T = 500°C); with the increase the rolling

temperature and the decrease the aging temperature, the elongation values of the material increase, making it more ductile;

- with regard the resilience results, the most advantageous values were obtained for absorbed energy and elasticity, for sample ***HR3-ST2-AT3*** (hot rolling: T = 475°C; ε = 50%, solution quenching T = 475°C and artificial aging at 150°C) with a value of E = 11 J and El = 29 J/cm2, with 23.89% higher to E (absorbed energy) than the value of sample ***HR3-ST2-AT1*** (hot rolling: T = 475°C; ε = 50%, solution treatment T = 475°C and artificial aging at 100°C) and 6.19% higher than the value of sample ***HR3-ST2*** (rolling hot: T = 475°C; ε = 50%, solution treatment T = 475°C) and for El (elasticity) 24.14% higher than the value of sample HR3-***ST2-AT1*** (hot rolling : T = 475°C; ε = 50%, solution treatment T = 475°C and artificial aging at 100°C) and 10.34% higher than the value of sample ***HR3-ST2*** (hot rolling: T = 475°C; ε = 50%, solution treatment T = 475°C); the conclusion is that with the increase in rolling temperature, tempering and aging, the resilience values increase;

Chapter 7. General conclusions and personal contributions

7.1. General conclusions

The thesis is centrally concerned with the investigation of how thermomechanical processing parameters influence the microstructure of the alloy, thus showing the mechanical properties of the 7075 alloy. As a key influencing parameter in the thermomechanical processing route, the thesis considers temperature, being a key parameter both in mechanical processing through hot deformation, as well as in heat treatment through solution placing and aging treatments.

The following key general conclusions result from the thesis:

- The microstructure of alloy 7075 is constituted of the following phases/compounds: α-Al, phase - η ($MgZn_2$); phase - S (Al_2CuMg); phase - T ($Al_2Mg_3Zn_3$); phase - θ (Al_2Cu_3) and intermetallic compounds of the Al-Mn-Cr-Fe type: $Al_6(Fe,Mn)$, $Al_5Si_2(Fe,Mn)$, $Al_3(Fe,Mn,Cr)$;
- Increasing the solution treatment temperature from 450°C (***ST1***) to 500°C (***ST3***) leads to the dissolution of various secondary phases/compounds, influencing the mechanical behavior; the weight fraction of the constituent phases/compounds plays a very important role in the mechanical behavior;
- Increasing the aging treatment temperature, from 100°C (***A1***) to 150°C (***A3***), leads to the precipitation/consolidation of various secondary phases/compounds, it also influences the mechanical behavior; the weight fraction of the constituent phases/compounds plays a very important role in the mechanical behavior;
- Suitable combination of high strength and high ductility properties can be obtained by combining solution treatment with aging treatment (solution temperature and temperature and duration of aging treatment should be carefully chosen):
  - in general, the aging treatment leads to a small decrease in strength properties and a small increase in ductility;
  - in general, when strength properties are desired: the solution treatment temperature should be increased and the aging treatment should be decreased;
  - in general, when hardness properties are desired: the solution treatment temperature must be increased and the aging treatment must be increased.
- The phase transformations and the mechanisms that produce precipitation hardening of heat-hardenable aluminum-based alloys are the basis of their applicability in high-

responsibility work areas such as that of flight equipment. The three decisive heat treatment steps are as follows:

  - heating to put the soluble precipitates into solution;
  - rapid cooling to maintain the solubilized elements in solid solution;
  - reheating for a uniform reprecipitation with hardening effect.

- From the cycle of applied thermal treatments (quenching for placing in solution and aging), the most extensive transformations take place during aging. They consist in the decomposition by diffusion of the supersaturated solid solution obtained during quenching. The formation of Guiner-Preston zones and transitional precipitates until the equilibrium precipitate is obtained represents a sequence of transformations each characterized by particular mechanical properties. Optical microscopy studies regarding the structure can only be applied from the moment when the precipitates are no longer coherent with the matrix. For the extremely effective situation is electron microscopy.
- The choice of the optimal aging temperature is a particularity of each alloy, exceeding the respective temperature, leading to significant decreases in the mechanical resistance properties.
- The intervening hardening mechanisms, hardening by internal stresses (Mott-Nabaro), chemical hardening or dispersion hardening are decisive in the correct choice of working parameters.
- The mechanical properties of the 7075 alloy during the thermomechanical processing routes evolved as follows:
  - in the mechanical tensile strength test, the most advantageous value RM = 377 MPa was obtained in sample ***HR1-ST2-AT1*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 475° C and artificial aging T=100°C) by 35.81% higher than the lowest value RM = 242 MPa, obtained in sample ***HR2-ST1-A3*** (hot rolling: T = 350°C; ε = 50% , solution treatment T = 450°C and artificial aging T=150°C);
  - in the yield strength test the most advantageous value Rp0.2 = 260 MPa, was obtained in sample ***HR1-ST3-AT1*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 500 °C and artificial aging at 100°C) by 44.42% higher than the lowest value Rp0.2 = 144.5 MPa, obtained in the ***AR*** sample (initial state);
  - in the elongation test the most advantageous value A50 = 17.25%, was obtained in the ***AR*** sample (initial state) with 62.32% higher than the lowest value A50 = 6.5%, obtained in the ***HR3*** sample (lamination at hot: T = 475°C; ε = 50%,);

  - in the resilience test (absorbed energy and elasticity) the most advantageous values E = 11 J and El = 29 J/cm^2 were obtained for sample ***HR3-ST2-AT1*** (hot rolling: T = 475°C; ε = 50% , solution treatment T = 475°C and artificial aging at 100°C) by 51.82% and 55.17% respectively higher than the lowest values E = 5.3 J and El = 13 J/cm^2, obtained on sample ***HR1-ST1-AT1*** (hot rolling: T = 225°C; ε = 50%, solution treatment T = 450°C and artificial aging at 100°C).

7.2. Personal contributions

A series of original/personal contributions, from the point of view of novelty, resulting from this thesis can be presented as follows:

- Carrying out a complex literature study, focused on aluminum-based alloys, mainly alloy 7075, which belongs to the class of zinc-based alloys, to determine the most influential thermomechanical processing parameters when designing a route that combines mechanical processing and thermal applied to the 7075 alloy, aiming to obtain a suitable combination of mechanical properties.
- The development of original experimental programs considering the existing laboratory infrastructure in order to achieve the assumed objectives.
- Investigation of the effects induced by hot deformation in the case of alloy 7075 in an experimental space ranging from a temperature of 225°C to 475°C.
- Investigating the effects induced by solution treatment on the exposed microstructure and mechanical properties, in a range of treatment temperatures varying from 450°C to 500°C, with the same treatment duration of 2min/mm.
- Investigating the effects induced by an aging treatment applied after the solution treatment on the presented microstructure and mechanical properties.
- Obtaining suitable thermomechanically processed samples of alloy 7075 to be used in evaluating the effects induced by thermomechanical processing based on the developed experimental programs.
- Development of specific investigation and characterization procedures applied to thermomechanically processed 7075 alloy samples in order to obtain data on microstructure and mechanical properties focused on OM, SEM, XRD and tensile tests.

7.3. Future research directions

Future directions for further research in the field of thermomechanical processing and characterization of alloy 7075 can be summarized as follows:

- In addition to conventional examination methods such as optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD), other advanced investigative techniques may be involved, such as: scanning electron microscopy transmission (TEM), electron backscatter diffraction (EBSD) and other descriptive-analytical methods to accurately study the microstructural constituents of the alloy (phase morphology, crystallography, etc.), secondary phase precipitation, occurrence of deformation mechanisms (slip/twinning), dislocation propagation, etc. , about providing additional information in understanding the relationship between mechanical properties and microstructure.
- In a similar series of investigations carried out within the experimental programs, the study of the increase in heating time/solution treatment/artificial/natural aging treatment on the evolution of microstructure and mechanical properties of alloy 7075 can be extended. Also, it can be taken into account the influence of cooling conditions on quenching.
- It is possible to extend the research by modifying the mechanical processing and heat processing steps of the 7075 alloy with additional processing steps that can provide a better combination of properties, thus expanding the possible end-user applications.
- It is possible to extend the research by modifying the chemical composition of the 7075 alloy with additional alloying elements/content, which may provide better insight into different possible end-user applications.

List of published scientific papers

(in the field of doctoral thesis)

A – Papers published in ISI listed / indexed journals

- first author:

1. ***Valeriu ANDREI,*** Irina Varvara BALKAN, Marian TURCIN, *Hot-rolling deformation behaviour of 7075 aluminium alloy*, UPB Bulletin: Series B Chemistry and Materials Science, Vol 85, Iss. 4, 2023.

2. ***Valeriu ANDREI,*** Marian TURCIN, Nicolae SERBAN, Irina Varvara BALKAN, *Simultaneous influence of rolling temperature and solubilization quenching conditions on structure and properties of a ZICRAL type aluminum alloy (class 7075),* UPB Bulletin: Series B Chemistry and Materials Science, Vol 85, Iss. 4, 2023.

B – Public support in the ROMAT 2022 international conference

3. ***Valeriu ANDREI,*** Carmen STANICA, Marian TURCU, Nicolae SERBAN, Marian TURCIN, Vasile Danut COJOCARU, *The influence of termomechanical processing parameters on the microstructural and mechanical properties of 7075 alloy,* International Conference on Materials Science and Technologies-RoMAT 2022 November 24-25, 2022, Bucharest, Romania, www.romat2022.ro.

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