Effect of thermomechanical treatment of Al-Zn-Mg-Cu with minor amount Sc and Zr on the mechanical properties

Azam Beigi Kheradmand (✉ Kheradmand@iaushk.ac.ir)

Islamic Azad University Shahrekord Branch

Shamseddin Mirdamadi

Department of Materials Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran

Zahra Lalegani

Young Researchers and Elite Club, Shahrekord Branch, Islamic Azad University, Shahrekord, Iran

Bejan Hamawandi (✉ bejan@kth.se)

KTH Royal Institute of Technology

Research Article

Keywords: Scandium, thermomechanical treatment, microstructure, 7000 series alloy

Posted Date: August 6th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-761139/v2>

License: © ⓘ This work is licensed under a Creative Commons Attribution 4.0 International License. [Read Full License](#)

Version of Record: A version of this preprint was published at Materials on January 13th, 2022. See the published version at <https://doi.org/10.3390/ma15020589>.

Abstract

In this study, mechanical and microstructural properties of Al-Zn-Mg-Cu-Zr cast alloy with 0.1% Sc under homogeneous, dissolution, and T6 and thermomechanical treatments with the aim of increasing the volume fraction of MgZn_2 and $\text{Al}_3(\text{Sc,Zr})$ reinforcing sediments were examined by hardness, microscopic examinations and tensile tests and software analysis. The results showed that firstly, the hardness results are well proportional to the results of tensile properties of alloys and secondly, the strength of the alloy with thermomechanical treatments compared to T6 treatments increased from 492 MPa to 620 MPa and the elongation increased from 8% to 17% and 100 % upgraded. Microstructural investigation and fracture cross section showed that $\text{Al}_3(\text{Sc,Zr})$ nanoparticles were evenly distributed among MgZn_2 particles and the alloy fracture was of semi-ductile type and in the fracture section nanoparticles less than 10 nm were observed at the end of the dimples. Also, the volume fraction of nanoparticles in the whole microstructure of thermomechanical treatment samples was much higher than that of T6 heat treated samples, so that the percentage of $\text{Al}_3(\text{Sc,Zr})$ sediments from less than 1% in T6 operation to 8.28% in quench-controlled thermomechanical operation (with 50% deformation) has arrived. QI index in thermomechanical treatment samples is 19% higher than T6 samples, so that this index has increased from 641 in T6 operation to 760 in samples under thermomechanical treatment due to sediment morphology, volume fraction of sediments, their uniform distribution in the matrix, and nano sized sediments in samples under thermomechanical treatment.

Introduction

Aluminum alloys are widely used in various industries. The addition of rare earth elements to aluminum alloys has been proven to have optimal effects [1-4]. Among aluminum alloy elements, scandium is one of the most effective elements for aluminum reinforcement. Al-Sc alloys have excellent mechanical properties at ambient temperature and at high temperatures due to the presence of hard and densely structured Al_3Sc particles [5-7]. Al_3Sc sediments remain coherent up to high temperatures with the aluminum matrix [8]. It has been shown that Al_3Sc nanoparticles are formed in aging or thermomechanical treatments [9, 10]. Thermomechanical treatment is a metallurgical process contains the combination of a plastic or mechanical deformation process such as rolling, forging, or pressurized processes and a thermal process such as heat treatment, quenching in water, heating and cooling at different speeds in a single process [11]. The microstructure (sediments or grain structure) and general properties of the alloys (mechanical properties or corrosion resistance) are affected by the heat and mechanical treatment performed on the alloy [12]. Among aluminum alloys, Al-Zn-Mg-Cu alloys along with scandium have utility in various industries. 7000 series alloys (Al-Zn-Mg-Cu) are widely used in applications such as structural components, automotive, and aerospace industries [13-15]. In recent years, researchers have studied the properties and microstructure of Al-Zn-Mg-Cu alloys under various treatments due to their high strength-to-weight ratio, relatively low price, good machinability, electrical conductivity, good corrosion resistance and excellent weldability [16-19]. Due to the high stacking fault energy of Al-Zn-Mg-Cu alloys, the hyper fine-grained structures are difficult to obtain by the conventional rolling process. For example, Al-Zn-Mg-Cu alloys treated by conventional thermomechanical processes have a grain size of more than 30 microns. Interestingly, secondary phase particles in Mg-Cu-Al-Zn alloys have been shown to play a role in grain modification during thermomechanical processing [20-25]. Therefore, the combination of alloying and the addition of elements such as scandium, and plastic deformation and heat treatment to control these sediments or particles during thermomechanical processing can dramatically affect the microstructure evolution of Al-Zn-Mg-Cu alloys to modify grains and improve their properties. Adding elements such as scandium to this group of alloys and combining the resulting properties with the properties obtained from thermomechanical treatments creates Al_3Sc particles. Al_3Sc particles act as grain modifiers in the solidification process of aluminum alloys, reinforcing sediments by fixing dislocations, and dispersed particles controlling the grain structure [6, 26]. Considering the high price of scandium, it is better to add other alloying elements to the aluminum alloy along with this element, to improve the mechanical properties and reduce the cost. The combination of scandium and zirconium leads to increased strength and reduced production costs of aluminum alloys. Because zirconium is another effective and well-known alloying element that reduces the average grain size and improves the tensile strength by forming the Al_3Zr particles [27].

Due to the fact that 7075 alloy is used in advanced industries [28, 29], and considering that the addition of scandium and zirconium to this alloy improves the mechanical and microstructural properties [30], in the present study scandium and zirconium was added to the 7075 series alloy and three-stage thermomechanical treatment was performed; and by examining the hardness, tensile strength, ductility, and QI index, these properties were compared with the properties obtained from T6 heat treatment.

Experimental Procedure

2.1. Instrument

A resistive furnace with programmability and accuracy of ± 3 degrees was used for heat treatment, and Brinell hardness tester according to ASTM E10 standard was used for hardness tests. Tensile test was performed according to ASTM E8 standard and using small samples with a Hounsfield instrument with H25KS model. Microstructure analysis was performed using a field emission scanning electron microscopy (FESEM) and high-resolution transmission electron microscopy (HRTEM), and tensile properties analysis was performed using the parameters of tensile strength, yield strength and elongation.

In engineering applications, instead of using any of the parameters of tensile strength and elongation to failure alone, it is better to derive a more acceptable parameter from the combination of tensile strength and elongation. Therefore, in this study, the quality index (QI) was used [31-33]. QI obtains from the UTS semi-log plot in terms of elongation to failure from the following equation [34]:

$$QI = UTS(MPa) + 150 \log(\%EI) \quad (1)$$

Considering that the purpose of this study was to increase the volume fraction of sediments and their uniform distribution using thermomechanical treatments, MIP software was used to calculate the volume fraction of sediments created in thermomechanical treatments and the results are compared with the volume fraction of sediments from T6 treatment.

2.2. Preparation of samples

Alloy samples were prepared by alloying and casting. For this purpose, alloy 7075 was first melted in a resistive furnace under argon gas and then Al-2Sc and Al-15Zr alloys were added to it and the initial Al_3Sc and Al_3Zr particles were allowed to dissolve, then before casting and for balancing the amount of zinc and magnesium, Al-Zn and Al-Mg alloys were used, and finally, the melt was mixed with a graphite lancer and under argon gas. Then the casting was performed in a vertical cast iron mold. The amount of scandium and zirconium in the alloy was analyzed by ICP method and other elements by quantometer analysis. Table 1 shows the chemical analysis of the alloy.

Table 1: Chemical composition of Al-Zn-Mg-Cu-0.1%Zr-0.1%Sc

Al	Zn	Mg	Cu	Ti	Mn	Cr	Ni	Fe	Be	Pb	Sn	Zr	Si	Sc
balanced	6.14	3.32	1.20	0.07	0.093	0.17	0.009	0.24	0.0007	0.0025	0.0093	0.1	0.153	0.1

After alloying and casting, some specimens with the dimension of 6mm $\times$ 100mm $\times$ 20mm were cut from the center of the ingots. Then the resulting specimens were machined and polished to remove contamination and surface scratches. Based on DSC analyzes and metallographic studies, the temperature and time of homogenization of the specimens were obtained. The homogenization temperature and time for the alloy was 500°C and 18 hours, respectively. After homogenizing the specimens, the thickness of the specimens was reduced from 6 mm to 4 mm by hot rolling process at 500°C. Secondary annealing was then performed for 6 hours and the dissolution temperature of the specimens was obtained based on electrical resistance test and hardness test. Dissolution of specimens was performed in two ways: normal and controlled quenching. After dissolution, the thickness of the specimens decreased from 4 mm to 2.8 mm (30% rolling) and 2.0 mm (50% rolling). The cross-section reduction process was performed by rolling at 100°C so that before each rolling pass, the specimens were kept at 100°C for 3 minutes and then rolled. After reaching the desired thickness, the specimens were exposed to 100, 120, 130, 140, 150, 200, 250, 300, 350, 400, 450, and 500°C for 2 hours to investigate the effect of temperature on hardness, and to investigate the effect of time, the specimens were subjected to ageing treatments at a constant temperature of 120°C for 10 minutes to 24 hours. In order to obtain accuracy in hardness measurement, three specimens were tested at each temperature and time. The microstructure of thermomechanical specimens was investigated by metallographic, FESEM, EDS, and TEM analysis. Aged specimens were subjected to tensile testing (according to ASTM E8 standard) at the peak time and temperature of age hardening and the mechanical properties of the specimens were obtained. QI index was calculated and finally, while investigating the fracture

surfaces of the specimens with FESEM and EDS analysis, sediment distribution and size of dimples were analyzed using MPI software. The specifications of the specimens and the performed thermomechanical treatments are presented in Table 2.

Table 2: Specifications of specimens and the performed thermomechanical treatments

Alloy code	Alloy composition	Thermomechanical treatment
A	Al-Zn-Mg-Cu-0.1%Zr-0.1%Sc	Homogenizing+ Rolling+ Secondary annealing+ Dissolution+ 30% rolling at 100°C+ Ageing
B	Al-Zn-Mg-Cu-0.1%Zr-0.1%Sc	Homogenizing+ Rolling+ Secondary annealing+ Dissolution+ 50% rolling at 100°C+ Ageing
C	Al-Zn-Mg-Cu-0.1%Zr-0.1%Sc	Homogenizing+ Rolling+ Secondary annealing+ Modified dissolution+ 30% rolling at 100°C+ Ageing
D	Al-Zn-Mg-Cu-0.1%Zr-0.1%Sc	Homogenizing+ Rolling+ Secondary annealing+ Modified dissolution+ 50% rolling at 100°C+ Ageing

Results And Discussion

3.1. Effect of ageing temperature on hardness

Figure 1 shows the effect of ageing temperature on the hardness of alloys in thermomechanical treatments with modified quench and conventional dissolution. As can be seen, the peak hardness temperature for alloys with 30% and 50% rolling are 130°C and 120°C, respectively. It is also observed that the hardness of alloy A is higher than the hardness of alloy C. In fact, the hardness of specimens with controlled quench thermomechanical treatment is higher than the hardness of specimens with conventional dissolution treatment.

According to figure 1a, it can be seen that in thermomechanical treatments with conventional dissolution, the hardness also increases with increasing the deformation rate. Comparison of the results of thermomechanical treatments with conventional dissolution with thermomechanical treatments with controlled quenching (figure 1b) shows that the peak hardness in thermomechanical treatments with controlled quenching has increased compared to the peak hardness in thermomechanical treatments with conventional dissolution. So that these values for 50% and 30% deformation in thermomechanical treatments with conventional dissolution are 195 and 160 Brinell, respectively, and in thermomechanical treatments with controlled quenching are 220 and 185 Brinell, respectively. On the other hand, it can be seen that in both treatments, the peak hardness temperature for the alloys with 50% and 30% rolling are 120°C and 130°C, respectively. Also in both curves (figures 1a and b) three areas can be observed. The first area is associated with hardness increasing. The second zone is the over ageing zone, where the density of growing sediments appears to be greater than the density of nucleating sediments. This growth of sediments leads to a decrease in hardness. It may be said that in the aged state, on the one hand, the amount of solid solution is insignificant and on the other hand, the sediments in the matrix begin to thicken. This makes it easier for the dislocations to move, and thus reduces the hardness. In the third area, the hardness is almost constant.

3.2. Effect of ageing time on hardness

Figure 2 shows the effect of ageing time on the hardness of the specimens. As can be seen, both curves fluctuate with increasing time without a constant trend, but the maximum hardness in thermomechanical treatments with controlled quenching is higher than that with conventional dissolution. As mentioned, the hardness of specimens with controlled quenching thermomechanical treatments is higher than the hardness of specimens with conventional dissolution treatments. It can be related to the method of dissolution treatments and its effect on the number of sediments. In controlled quenching, dissolution and quenching treatments increase the diffusion coefficient and activate energetic centers for sediment nucleation. Therefore, in this case, the number of sediment nucleation sites increases. On the other hand, thermomechanical treatments produce many high-energy centers that require the least energy for nucleation, which is a good source for sedimentation, and the number of sediments increases and their distribution becomes uniform.

According to figure 2, it can be seen that in the first hour of ageing, the hardness fluctuates due to the interaction of dislocations and sediments. This fluctuation can be due to the effects of ageing and partial recrystallization. Hardness fluctuates until the effects of ageing predominate and the hardness of the alloy increases due to the increase in the number of sediments. Henceforth, the hardness fluctuation will be low and eventually the hardness of the specimens will remain almost constant and the hardness of the specimens with 50% deformation will be more than the specimens with 30% deformation. It is also observed that at times of more than 16 hours, the hardness is almost constant due to the thermally stable $\text{Al}_3(\text{Sc}, \text{Zr})$ sediments. $\text{Al}_3(\text{Sc}, \text{Zr})$ phase is coherent with aluminum matrix [35], and these coherent particles have very good thermal stability due to their low solubility and very low diffusion coefficient in aluminum matrix [36].

Comparing the maximum hardness obtained in T6 hardening treatment with thermomechanical treatment, it is observed that the maximum hardness obtained in thermomechanical treatment is higher than the T6 hardening treatment. This increase in hardness is related to the deformation performed before the ageing treatment. During deformation, the interaction of two sets of processes controls the behavior of the material: material softening processes including partial recovery and recrystallization; hardening processes including the increasing of dislocations density and the formation of sediments. The main variables affecting these processes are the deformation temperature, the method of dissolution treatment, and the applied deformation rate. Under the same conditions of dissolution treatment, the deformation before ageing increases the diffusion coefficient; and as the deformation rate increases, the density of the dislocations increases. These dislocations are region for heterogeneous nucleation as well as rapid diffusion pathways. Therefore, due to the increase in diffusion power, the high-energy centers for nucleation are increased and by rolling at 100°C, the effects of dislocations are recovered, and during ageing at 120°C, the volume fraction of sediments increases.

3.3. Microstructural investigation

In metallographic studies, the size and shape of the grains (coaxial or elongated) are considered. The microstructure of the alloy containing 0.1% scandium after thermomechanical treatments with conventional dissolution (50% rolling) is shown in figure 3. Figure 3a shows elongated grains in the rolling direction and intermetallic particles. Figure 3b shows that few numbers of grains have recrystallized.

The microstructure of the alloy containing 0.1% scandium after thermomechanical treatments with controlled quenching and different deformation is shown in figure 4. In figures 4a and c, elongated grains in the direction of rolling and black particles are seen at some grain boundaries that were insoluble during the dissolution treatment. In figures 4b and d, few numbers of coaxial grains can be seen.

In order to study more precisely the effect of thermomechanical treatments and also the effect of deformation rate and dissolution method on alloy microstructure, the alloy microstructure in 50% and 30% deformations and also in two methods of conventional dissolution and controlled quenching treatments were investigated. The purpose of this study was to determine the grain size and volume fraction of sediments generated during the process. Figure 5 shows the grain size in the alloy after thermomechanical treatments with 50% rolling in two states of controlled quenching and conventional dissolution. The average grain length in figure 5a is 25 μm and in figure 5b is 19 μm . This is in accordance with the metallographic study of these specimens (figures 3 and 4).

The distribution of sediments in the alloy under thermomechanical treatments with conventional dissolution and controlled quenching and 50% deformation is shown in figure 6. Figure 6a shows white spherical sediments with an average size of 100 nm and figure 6b shows white spherical sediments with an average size of 186 nm. In both images (figures 6a and b), two types of sediments can be seen. Also, there are a number of disk-shaped sediments that have been created by the accumulation of spherical sediments together.

The distribution of sediments in the sample under thermomechanical treatments with conventional dissolution and 30% deformation is shown in figure 7. In this figure, two types of sediments can be seen, some of them are spherical and white, and most of them are light gray, which are distributed in the aluminum matrix. The average size of spherical sediments is 90 nm.

The distribution of MgZn_2 and $\text{Al}_3(\text{Sc}, \text{Zr})$ sediments in the matrix in the specimen under thermomechanical treatments with conventional dissolution and 30% deformation along with the element distribution map is given in figure 8. Figure 8a, taken at 50000x magnification, shows two types of sediment: type B sediments are spherical and white, and type A sediments are light gray

which are seen in the shape of disk from the accumulation of sediments together. In figure 8b, type B sediments are indicated by orange arrows. Figure 8c shows the elements distribution map. As can be seen, zinc, magnesium, scandium and zirconium elements are distributed in the matrix. The distribution of zinc and magnesium elements are observed in whole microstructure, and the distribution of scandium and zirconium elements are observed in some parts of microstructures.

The elemental analysis of the phases is given in Table 3 (according to figure 8).

According to this table, phase A is rich in zinc and magnesium and the ratio of zinc to magnesium is 2: 1. Phase B is rich in scandium and zirconium, and the composition of scandium and zirconium is higher than the chemical composition of the primary alloy.

Table 3: EDS analysis of points A and B in figure 8

Phase	Chemical composition (%at.)					
	Al	Sc	Zr	Mg	Zn	Ti
A	93.54	-	-	1.55	3.12	-
B	95.91	0.38	1.52	-	1.96	0.23

The distribution of sediments in the alloy containing 0.1% Sc under thermomechanical treatments with controlled quench dissolution conditions and 30% deformation is shown in figure 9.

Figure 9a shows two types of sediment A and B with different contrast. Sediment A is found slightly at the grain boundaries and mostly inside the grain. Sediments A are light white and sediments B are light gray. Figure 9b shows the distribution of A sediments and is shown with orange arrows. EDS analysis of these phases is shown in Table 4. Figure 9c shows the distribution map of scandium and zirconium elements in the microstructure. The densities of scandium and zirconium are higher in some parts of the microstructure. According to Table 4, phase A is rich in scandium and zirconium and the percentage of scandium and zirconium elements in this phase is much higher than the initial chemical composition of the alloy. Phase B is also rich in zinc and magnesium and the ratio of zinc to magnesium is 2: 1.

Table 4: EDS analysis of points A and B in figure 9

Phase	Chemical composition (%at.)						
	Al	Sc	Zr	Mg	Zn	Ti	Si
A	95.65	1.48	2.59	-	1.49	0.1	0.18
B	94.86	-	-	3.22	1.92	-	-

Sediment distribution in the specimen under thermomechanical treatment with controlled quenching and 50% deformation is shown in figure 10. Figure 10a shows two types of sediment with different contrast. The type D sediments are spherical and white and the type C sediments are light gray. Figure 10b shows the distribution of type D sediments with green arrows and the distribution of type C sediments with yellow arrows. In figure 10c, the element distribution maps of aluminum and scandium are shown and it is observed that the density of scandium element is higher in some parts of the matrix. Figure 10d shows the element distribution map of zinc and magnesium and shows that they are distributed in the whole matrix.

The elemental analysis of the phases of figure 10 is presented in Table 5. According to the table 5, C sediments are rich in magnesium and zinc and the ratio of zinc to magnesium in this phase is 2: 1. D sediments are rich in scandium and zirconium, and the amount of scandium and zirconium in this phase is several times higher than that of scandium and zirconium in the chemical composition of the primary alloy.

Table 5: EDS analysis of points C and D in figure 10

Phase	Chemical composition (%at.)					
	Al	Sc	Zr	Mg	Zn	Si
C	95.29	-	-	2.92	1.49	0.3
D	88.97	2.82	6.09	2.48	-	-

According to FESEM images and EDS analysis of sediments, $\text{Al}_3(\text{Sc,Zr})$ and MgZn_2 sediments are observed, these sediments are uniformly distributed in the microstructure. Tao and Romestch have also shown that $\text{Al}_3(\text{Sc,Zr})$ particles are formed in thermomechanical treatments [37, 38]. Total microscopic observations on the distribution of sediments in two alloy specimens containing 0.1% Sc, under thermomechanical treatment with 50% rolling at 100°C, in two modes of controlled quenching and conventional dissolution show that the number of $\text{Al}_3(\text{Sc,Zr})$ sediments in the specimen under thermomechanical operation with controlled quenching is more than the specimen undergoing thermomechanical treatment with conventional heat treatment. It seems that increasing the diffusion rate due to controlled quenching increases the nucleation sites of the sediment and thus increases the number of sediments.

It is also observed that under the same ageing conditions (50% rolling at 100°C and ageing at 120°C for 12 hours), the grain size in the alloy under thermomechanical treatments with controlled quenching is less than the grain size in the alloy under thermomechanical treatments with conventional dissolution. The presence of more $\text{Al}_3(\text{Sc,Zr})$ particles in the alloy and their inhibitory effect on grain growth due to controlled quenching, have optimized the grain size. Since the number of sediment particles in the alloy under controlled quenching thermomechanical treatments is higher than the alloy under conventional dissolution thermomechanical treatments, the grain structure is modified. Minor amounts of scandium and zirconium improve the grain structure and alloy strength and restrict the grain [39].

It can be said that by using thermomechanical treatments, sediment nucleation conditions are facilitated and more nucleation sites lead to uniform distribution of sediments throughout the structure. As the FESEM images (figures 8 and 9) show, $\text{Al}_3(\text{Sc,Zr})$ and MgZn_2 sediments are uniformly distributed throughout the structure (instead of aggregation at the grain boundaries), which increases the strength of the structure and does not reduce ductility. These sediments are dynamic sediments created during thermomechanical treatments. In general, dynamic sedimentation is related to the high density of point defects and the high recovery rate during rolling [40]. Sediment distribution during thermomechanical and age hardening treatments creates effective sites for trapping and accumulation of dislocations around sediments. Therefore, during the tensile strain of aged specimens, the specimens will elongate before fracture (more ductility). Therefore, the combined effect of improved grain structure and precipitation hardening can be considered as the reason for increasing the strength of the alloy [41]. In the EDS analyzes (Tables 3 to 5), elements of silicon and titanium are observed. Zhao et al. [42] reported that the presence of impurities such as silicon, copper and titanium in the diffusion area around $\text{Al}_3(\text{Sc,Zr})$ particles increases the heterogeneous nucleation capacity of this phase. The volume fraction of sediments during thermomechanical treatments (conventional dissolution and controlled quenching) is analyzed by MIP software and is shown in figure 11. According to this analysis, the volume fraction of MgZn_2 and $\text{Al}_3(\text{Sc,Zr})$ sediments in the specimen under thermomechanical treatment with controlled quenching and 30% rolling was 33.91% and 6.66%, respectively, and in the specimen with 30% rolling and conventional dissolution was 32.58% and 4.94%, respectively, and in the specimen with controlled quenching and 50% rolling was 30.43% and 8.21%, respectively.

The results of sediment distribution analysis with MIP software performed in specimens undergoing T6 treatment were compared with the distribution of sediments in specimens under thermomechanical treatment and are shown as a comparison table in Table 6. It can be seen that firstly, in thermomechanical treatments, the distribution of $\text{Al}_3(\text{Sc,Zr})$ and MgZn_2 sediments has become more uniform than T6 heat treatment and also the volume fraction of sediments has increased. The volume percentage of $\text{Al}_3(\text{Sc,Zr})$ has increased from 0.38% in T6 treatment to 8.2% in thermomechanical treatment with controlled quenching (50% deformation) and

the volume percentage of MgZn_2 from 7.67% to 33%. Secondly, with increasing the deformation, the volume fraction of sediments in the microstructure has increased. As mentioned, the increase in sediment nucleation sites and the less energy required for nucleation due to thermomechanical treatments is the reason for the increase in sediment in the microstructure and their uniform distribution.

Table 6: Comparison of volume fraction of $\text{Al}_3(\text{Sc,Zr})$ and MgZn_2 sediments in alloys containing 0.1% Sc in specimens under different treatment conditions, using MIP software analysis

Treatment	Volume fraction of MgZn_2 (%)	Volume fraction of $\text{Al}_3(\text{Sc,Zr})$ (%)
Age hardening with controlled quenching	17.91	1.96
Age hardening with conventional dissolution	2.76	0.38
Thermomechanical with controlled quenching and 30% rolling	33.91	6.66
Thermomechanical with controlled quenching and 50% rolling	30.43	8.2
Thermomechanical with conventional dissolution and 30% rolling	32.58	4.94

In order to more accurate study of microstructure in the specimens under thermomechanical treatment, the specimen with the highest volume fraction of sediments (specimen under thermomechanical treatment with 50% deformation and controlled quenching) was selected and its microstructure was investigated by TEM. The TEM micrograph of this specimen is shown in figure 12. According to figure 12, there are two types of sediments in the microstructure with rod and spherical morphology. Some of spherical particles are shown with orange arrows and rod-shaped particles are shown with yellow arrows.

EDS analysis of particles with rod and spherical morphology is shown in figure 13. According to the given EDS analysis, the spherical particles in the microstructure are of $\text{Al}_3(\text{Sc,Zr})$ sediments type and the rod particles are of MgZn_2 type. Also, the diffracted pattern in figure 12 is related to an area of the aluminum matrix that is predominantly with spherical $\text{Al}_3(\text{Sc,Zr})$ sediments and is taken in the region axis (011). Superlattice diffractions such as (100) in the selected area diffraction patterns (SADP) confirm the existence of these sediments [41].

3.4. Investigation of mechanical properties of the alloys

The results of the tensile test of the specimens (UTS, QI, and elongation) are presented in and Table 7. As can be seen in both T6 and thermomechanical treatments, the QI values in the controlled quenching methods are higher than that in the conventional dissolution methods. Also, the QI obtained from thermomechanical treatment is more than T6 heat treatment. The higher QI in thermomechanical treatments is due to the increase in the volume fraction of $\text{Al}_3(\text{Sc,Zr})$ sediments, their spherical morphology and their uniform distribution throughout the structure, which leads to a simultaneous increase in tensile strength and elongation to fracture, resulting in increased QI.

Table 7: Tensile properties of alloys

Type of treatment	(MPa)	(MPa)	%el	QI (MPa)
Thermomechanical with controlled quenching and 30% rolling	514	560	16	745
Thermomechanical treatment with controlled quenching and 50% rolling	552	579	14	750.92
Thermomechanical treatment with conventional dissolution and 30% rolling	599	611	10	750
Thermomechanical treatment with conventional dissolution and 50% rolling	595	612	9.5	760
Age hardening with controlled quenching	587	600.47	10	740
Conventional T6	472.15	498.7	8	641

The results show that the tensile strength of the alloy containing 0.1%Sc with thermomechanical and ageing treatments has increased by 25% compared to the alloy under T6 treatment, so that the tensile strength of the alloy has increased from 490 MPa to 620 MPa. This is while the rate of elongation has increased. However, ductility is expected to decrease with increasing strength. While with thermomechanical treatments, in addition to increasing strength, ductility has also increased compared to T6 treatments. It can also be seen that with controlled quenching thermomechanical treatments, the ductility increased to 16% while the tensile strength decreased by only 10% compared to thermomechanical treatments with conventional dissolution. In fact, there is 10% decrease in strength and 50% increase in ductility in thermomechanical treatments with modified quenching compared to thermomechanical treatments with conventional dissolution. This change in tensile strength and ductility relative to alloy with T6 treatment is 20% and 70%, respectively. In the thermomechanical treatment, after the dissolution, rolling was performed at 100°C temperature in successive passes. Since this temperature is the recovery temperature of the alloy, so after each rolling pass, remaining at 100°C helps to recover the effects of dislocations and increases ductility, and on the other hand, the energy required for sediment nucleation is provided and the number of sediment nucleation sites in the matrix increases. FESEM images show that a large number of sediments are uniformly distributed in the matrix, so the main reason for increasing the strength of the specimens is strengthening with sediments. These sediments prevent dislocation movements. In fact, the size and distribution of sediments and discontinuities of sediments have a great effect on improving the mechanical properties of Al-Zn-Mg-Cu alloy [43]. Simultaneous acquisition of strength and ductility in aluminum alloys is difficult [20]. Improved ductility in specimens can be due to the reduction of dislocations during age hardening treatments, and the presence of nano-sized particles in the aged specimens. Because the density of sediments is high, more dislocations accumulate around the sediments, causing a long-term tensile strain and increasing ductility [41].

Sediment distribution affects hardness, tensile strength and failure mechanism. When the sediments are small and well distributed, the strength and hardness are greater than when the sediments are large. Therefore, in order to achieve high hardness and strength, heat treatment should be done in such a way that fine sediments are created with uniform distribution in the structure [44]. In the present study, $\text{Al}_3(\text{Sc,Zr})$ sediments are small sediments with uniform distribution among MgZn_2 sediments. The morphology and distribution of these phases play an essential role in the performance of the alloy [23]. Small spherical homogeneous particles improve plasticity. These particles and phases participate in strengthening processes [24]. Therefore, the interaction between dislocations and sediments increases the mechanical properties obtained in thermomechanical treatments [36]. It has been shown that thermomechanical treatments greatly affect the mechanical properties and microstructure of aluminum alloys [25]. Therefore, by performing thermomechanical treatments, tensile strength and elongation can be increased simultaneously [26].

3.5. Investigation of fracture cross-section

Fracture is often the end point of deformation processes. Therefore, fracture surfaces can indicate the deformation processes to which matter has been exposed [27]. The mechanical properties of materials directly depend on their fracture mechanism, especially on the processes of crack growth and microscopic fracture. Therefore, qualitative fracture study has a fundamental role in materials research. FESEM has been used for this purpose. FESEM images of the fracture surfaces of the specimens under thermomechanical treatment, on which the tensile test was performed are shown in figure 14.

The volume fraction of sediments at the fracture surface and the average size and the percentage of dimples at the fracture surface, which determine the type of fracture, are analyzed by using MIP software and their comparison results are presented in figure 15.

At the fracture surface of the samples, a large number of fine dimples are observed. The form of fracture indicates the interconnection of micro-cavities whose formation, growth and their interconnection is the dominant phenomenon in the fracture mechanism [28]. The size of dimples in the specimen with less deformation (30%) is smaller than the specimen with more deformation. Fracture surfaces have dimples that are characteristic of ductile fracture. Many micro cracks start from these large dimples. The formation of fine $\text{Al}_3(\text{Sc,Zr})$ sediments in aged alloys can create suitable areas for crack nucleation. The fracture surface of the specimens shows that the predominant fracture phenomenon is semi-ductile fracture and some brittle fracture is also seen in the fracture surfaces.

As can be seen in the fracture surface images of the specimens, a large number of nanometer-sized $\text{Al}_3(\text{Sc,Zr})$ particles are present on the fracture surface. The presence of these nano sediments at the fracture surface may indicate that the probable mechanism of fracture in these specimens is the Orowan mechanism. Other research has shown that the mechanism of Al_3Sc nanoparticle sediment strengthening is the Orowan mechanism (dislocation bypassing behavior) [45]. The joining of dimples together during tensile deformation causes the formation of large cracks and eventually the fracture. As the strain increases, the new cavities become larger and the failure process progresses as these cavities join together. Joining cavities is an internal necking mechanism that often occurs at low to moderate stress levels [46]. In specimen with a higher percentage of deformation, the shape and size of the dimples become larger and a more compact dimple structure is created at the fracture surface. Nucleation and joining of cavities and dimples may lead to the growth of cracks along the grain boundaries.

Conclusion

Thermomechanical treatments consisted of three stages of dissolution, multi-pass rolling process at 100°C recovery temperature, and hardening at 120°C temperature for 16 hours. The age hardening temperature in this treatment for specimens with 50% and 30% deformation was obtained 120°C and 130°C, respectively. Tensile and yield strength increased 110% in specimens with thermomechanical treatment compared to annealed specimens. While elongation has not changed much. In T6 age hardening treatment, tensile / yield strength and elongation compared to the annealed specimen increased by 50% and decreased by 40%, respectively. While tensile / yield strength and elongation in thermomechanical treatment increased by 25% and 50% compared to T6 treatment, respectively. Simultaneous increase in tensile / yield strength and elongation in these specimens is due to the uniform distribution of $\text{Al}_3(\text{Sc,Zr})$ nanoparticles throughout the microstructure and among MgZn_2 particles. Investigation of the fracture surfaces showed that $\text{Al}_3(\text{Sc,Zr})$ nanoparticles were distributed at the fracture surface and the fracture surface consisted of small dimples indicating the semi-ductile fracture of the alloy, and the possible fracture mechanism is the Orowan mechanism.

Declarations

Funding:

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Conflict of Interest:

The authors declare that they have no conflict of interest.

References

- [1] Y. Deng, Z. Yang, and G. Zhang, "Nanostructure characteristics of $\text{Al}_3\text{Sc}_{1-x}\text{Zr}_x$ nanoparticles and their effects on mechanical property and SCC behavior of Al-Zn-Mg alloys", *Materials*, vol. 13, p. 1909, 2020.
- [2] B. Li, H. Wang, J. Jie, and Z. Wei, "Effects of yttrium and heat treatment on the microstructure and tensile properties of Al-7.5Si-0.5Mg alloy," *Mater. Des.*, vol. 32, no. 3, pp. 1617–1622, 2011.

- [3] B. Li, H. Wang, J. Jie, and Z. Wei, "Microstructure evolution and modification mechanism of the ytterbium modified Al-7.5%Si-0.45%Mg alloys," *J. Alloys Compd.*, vol. 509, no. 7, pp. 3387–3392, 2011.
- [4] T. V Atamanenko, D. G. Eskin, L. Zhang, and L. Katgerman, "Criteria of Grain Refinement Induced by Ultrasonic Melt Treatment of Aluminum Alloys Containing Zr and Ti," 2010.
- [5] E. A. Marquis and D. N. Seidman, "Nanoscale structural evolution of Al₃Sc precipitates in Al(Sc) alloys," *Acta Mater.*, vol. 49, no. 11, pp. 1909–1919, 2001.
- [6] J. Royset, "Scandium in Aluminium alloys overview: Physical Metallurgy, Properties and applications," *Metall. Sci. and Technology*, vol. 25, no. 2, pp. 11–21, 2007.
- [7] T. Ponomareva, M. Ponomarev, A. Kisarev, and M. Ivanov, "Wire arc additive manufacturing of Al-Mg alloy with the addition of scandium and zirconium", *Materials*, Vol. 14, p. 3665, 2021.
- [8] S. Iwamura and Y. Miura, "Loss in coherency and coarsening behavior of Al₃Sc precipitates," *Acta Mater.*, vol. 52, no. 3, pp. 591–600, 2004.
- [9] Y. Tao, Z. Zhang, D. R. Ni, D. Wang, B. L. Xiao, and Z. Y. Ma, "Influence of welding parameter on mechanical properties and fracture behavior of friction stir welded Al-Mg-Sc joints," *Mater. Sci. Eng. A*, vol. 612, pp. 236–245, 2014.
- [10] P. A. Rometsch, H. Zhong, K. M. Nairn, T. Jarvis, and X. Wu, "Characterization of a laser-fabricated hypereutectic Al–Sc alloy bar," *Scr. Mater.*, vol. 87, pp. 13–16, 2014.
- [11] E. P. Degarmo, J T. Black, and R. a Kohser, "Materials and Process in Manufacturing," *Mater. Process Manuf.*, p. 383, 2003.
- [12] M. Vasheghani Farahani, E. Emadoddin, M. Emamy, and A. Honarbakhsh Raouf, "Effect of grain refinement on mechanical properties and sliding wear resistance of extruded Sc-free 7042 aluminum alloy," *Mater. Des.*, vol. 54, pp. 361–367, Feb. 2014.
- [13] Y. Deng *et al.*, "Corrosion behaviour and mechanism of new aerospace Al-Zn-Mg alloy friction stir welded joints and the effects of secondary Al₃Sc_xZr_{1-x} nanoparticles," *Corros. Sci.*, vol. 90, pp. 359–374, 2015.
- [14] J. Hirsch, "Recent development in aluminium for automotive applications," *Trans. Nonferrous Met. Soc. China (English Ed.*, vol. 24, no. 7, pp. 1995–2002, 2014.
- [15] P. Vijaya Kumar, G. Madhusudhan Reddy, and K. Srinivasa Rao, "Microstructure and pitting corrosion of armor grade AA7075 aluminum alloy friction stir weld nugget zone – Effect of post weld heat treatment and addition of boron carbide," *Def. Technol.*, vol. 11, no. 2, pp. 166–173, 2015.
- [16] J. Lu, Y. Song, L. Hua, K. Zheng, and D. Dai, "Thermal deformation behavior and processing maps of 7075 aluminum alloy sheet based on isothermal uniaxial tensile tests," *J. Alloys Compd.*, vol. 767, pp. 856–869, 2018.
- [17] A. Ghosh and M. Ghosh, "Microstructure and texture development of 7075 alloy during homogenisation," *Philos. Mag.*, vol. 6435, pp. 1–21, 2018.
- [18] Y. F. Mo *et al.*, "Fabrication of 7075-0.25Sc-0.15Zr Alloy with Excellent Damping and Mechanical Properties by FSP and T6 Treatment," pp. 2–7, 2018.
- [19] X. Peng, Y. Li, G. Xu, J. Huang, and Z. Yin, "Effect of Precipitate State on Mechanical Properties , Corrosion Behavior , and Microstructures of Al – Zn – Mg – Cu Alloy," *Met. Mater. Int.*, no. 0123456789, 2018.
- [20] J. Waldman, H. Sulinski, and H. Markus, "Effect of ingot processing treatments on the grain size and properties of Al alloy 7075," *Met. Trans*, vol. 5, no. 3, pp. 573–584, 1974.
- [21] J. A. Wert, N. E. Paton, C. H. Hamilton, and M. W. Mahoney, "Grain Refinement in 7075 Aluminum By Thermomechanical Processing," *Metall. Trans. A, Phys. Metall. Mater. Sci.*, vol. 12 A, no. 7, pp. 1267–1276, 1981.

- [22] J. D. Robson, D. T. Henry, and B. Davis, "Particle effects on recrystallization in magnesium-manganese alloys: Particle pinning," *Mater. Sci. Eng. A*, vol. 528, no. 12, pp. 4239–4247, 2011.
- [23] A. Gholinia, F. J. Humphreys, and P. B. Prangnell, "Production of ultra-fine grain microstructures in Al-Mg alloys by conventional rolling," *Acta Mater.*, vol. 50, no. 18, pp. 4461–4476, 2002.
- [24] E. Di Russo, M. Conserva, F. Gatto, and H. Markus, "Thermomechanical treatments on high strength Al-Zn-Mg(-Cu) alloys," *Metall. Trans.*, vol. 4, no. 4, pp. 1133–1144, 1973.
- [25] Y. Lang, H. Cui, Y. Cai, and J. Zhang, "Effect of strain-modified particles on the formation of fined grains and the properties of AA7050 alloy," *Mater. Des.*, vol. 39, pp. 220–225, 2012.
- [26] J. Røyset and N. Ryum, "Scandium in aluminium alloys," vol. 50, no. 1, pp. 19–44, 2005.
- [27] G. Li *et al.*, "Effect of Sc/Zr ratio on the microstructure and mechanical properties of new type of Al-Zn-Mg-Sc-Zr alloys," *Mater. Sci. Eng. A*, vol. 617, 2014.
- [28] J. C. Williams and E. A. Starke, "Progress in structural materials for aerospace systems11The Golden Jubilee Issue—Selected topics in Materials Science and Engineering: Past, Present and Future, edited by S. Suresh.," *Acta Mater.*, vol. 51, no. 19, pp. 5775–5799, Nov. 2003.
- [29] T. Dursun and C. Soutis, "Recent developments in advanced aircraft aluminium alloys," *Mater. Des.*, vol. 56, pp. 862–871, 2014.
- [30] K. Turba, P. Málek, and M. Cieslar, "Superplasticity in a Zr and Sc modified AA7075 aluminium alloy produced by ECAP," *Kov. Mater.*, vol. 45, no. 3, pp. 165–170, 2007.
- [31] Y. Kaushik, *A Review on use of Aluminium Alloys in Aircraft Components*, vol. 3. 2015.
- [32] M. Tiryakioglu and J. Campbell, *Quality Index for Aluminum Alloy Castings*, vol. 8. 2014.
- [33] E. Czekaj, J. Zych, Z. Kwak, and A. Garbacz-Klempka, "Quality Index of the AlSi₇Mg_{0.3} Aluminium Casting Alloy Depending on the Heat Treatment Parameters," *Arch. Foundry Eng.*, vol. 16, no. 3, pp. 25–28, 2016.
- [34] E. R. Wang, X. D. Hui, S. S. Wang, Y. F. Zhao, and G. L. Chen, "Improved mechanical properties in cast Al-Si alloys by combined alloying of Fe and Cu," *Mater. Sci. Eng. A*, vol. 527, no. 29–30, pp. 7878–7884, 2010.
- [35] A. E8-2016, "Standard Test Methods for Tension Testing of Metallic Materials."
- [36] Y. H. Zhao, X. Z. Liao, S. Cheng, E. Ma, and Y. T. Zhu, "Simultaneously increasing the ductility and strength of nanostructured alloys," *Adv. Mater.*, vol. 18, no. 17, pp. 2280–2283, 2006.
- [37] S. R. Koteswara Rao, B. Kamsala Devi, K. Sreenivasa Rao, and K. Prasad Rao, "Thermo-mechanical treatments of Sc- and Mg-modified Al-Cu alloy welds," *Int. J. Adv. Manuf. Technol.*, vol. 45, no. 1–2, pp. 16–24, 2009.
- [38] V. G. Davydov, T. D. Rostova, V. V. Zakharov, Y. A. Filatov, and V. I. Yelagin, "Scientific principles of making an alloying addition of scandium to aluminium alloys," vol. 280, pp. 30–36, 2000.
- [39] G. Peng, K. Chen, S. Chen, and H. Fang, "Evolution of the second phase particles during the heating-up process of solution treatment of Al-Zn-Mg-Cu alloy," *Mater. Sci. Eng. A*, vol. 641, pp. 237–241, 2015.
- [40] T. Liu, C. nian He, G. Li, X. Meng, C. sheng Shi, and N. qin Zhao, "Microstructural evolution in Al-Zn-Mg-Cu-Sc-Zr alloys during short-time homogenization," *Int. J. Miner. Metall. Mater.*, vol. 22, no. 5, pp. 516–523, 2015.
- [41] Z. Zhao, J. Cui, J. Dong, and B. Zhang, "Effect of low-frequency magnetic field on microstructures and macrosegregation of horizontal direct chill casting 7075 aluminum alloy," *J. Mater. Process. Technol.*, vol. 182, no. 1, pp. 185–190, 2007.

- [42] Y. H. Zhao, X. Z. Liao, Z. Jin, R. Z. Valiev, and Y. T. Zhu, "Microstructures and mechanical properties of ultrafine grained 7075 Al alloy processed by ECAP and their evolutions during annealing," *Acta Mater.*, vol. 52, no. 15, pp. 4589–4599, 2004.
- [43] Y. Huang and P. B. Prangnell, "Continuous frictional angular extrusion and its application in the production of ultrafine-grained sheet metals," *Scr. Mater.*, vol. 56, no. 5, pp. 333–336, Mar. 2007.
- [44] D. K. Xu, P. A. Rometsch, and N. Birbilis, "Improved solution treatment for an as-rolled Al–Zn–Mg–Cu alloy. Part I. Characterisation of constituent particles and overheating," *Mater. Sci. Eng. A*, vol. 534, pp. 234–243, 2012.
- [45] D. N. Seidman, E. A. Marquis, and D. C. Dunand, "Precipitation strengthening at ambient and elevated temperatures of heat-treatable Al (Sc) alloys," vol. 50, pp. 4021–4035, 2002.
- [46] S. Shekhar, R. Sarkar, S. K. Kar, and A. Bhattacharjee, "Effect of solution treatment and aging on microstructure and tensile properties of high strength β titanium alloy, Ti–5Al–5V–5Mo–3Cr," *Mater. Des.*, vol. 66, pp. 596–610, 2015.

Figures

Figure 1

The effect of ageing temperature on the hardness of alloys in thermomechanical treatments with: a) conventional dissolution, and b) modified quench.

Figure 2

The effect of ageing time on the hardness of alloys in thermomechanical treatments with: a) conventional dissolution, and b) modified quench.

Figure 3

Metallographic image of alloy containing 0.1% scandium under thermomechanical treatment with conventional dissolution: a) after dissolution and 50% deformation, and b) ageing at 120°C for 12 hours.

Figure 4

Metallographic image of alloy containing 0.1% scandium under thermomechanical treatment with controlled quenching: a) after dissolution and 30% deformation, b) ageing at 130°C, c) after dissolution and 50% deformation, and d) ageing at 120°C

Figure 5

Grain size in alloy containing 0.1% Sc under two thermomechanical treatments with 50% deformation: a) thermomechanical treatments with conventional dissolution, and b) thermomechanical treatments with controlled quenching at 120°C for 12 hours.

Figure 6

Sediment distribution in two alloy specimens containing 0.1% Sc under thermomechanical treatment with 50% deformation at 120°C for 12 hours: a) with controlled quenching, and b) with conventional dissolution.

Figure 7

Distribution of sediments in the specimen under thermomechanical treatments with conventional dissolution, 30% rolling, and ageing at 120°C for 12 hours.

Figure 8

Distribution of $MgZn_2$ and $Al_3(Sc,Zr)$ sediments in aluminum matrix in alloy under thermomechanical treatment with conventional dissolution and 30% rolling: a) $Al_3(Sc,Zr)$ and $MgZn_2$ sediments, b) distribution of $Al_3(Sc,Zr)$, and c) element distribution maps.

Figure 9

Distribution of MgZn₂ and Al₃(Sc,Zr) sediments in aluminum matrix in alloy containing 0.1% Sc, under thermomechanical treatment with controlled quench and 30% rolling: a, b) Distribution of MgZn₂ and Al₃(Sc,Zr) sediments, and c) element distribution maps.

Figure 10

Distribution of MgZn_2 and $\text{Al}_3(\text{Sc,Zr})$ sediments in aluminum matrix in alloy containing 0.1% Sc, under thermomechanical treatment with controlled quench and 50% rolling: a, b) Distribution of MgZn_2 and $\text{Al}_3(\text{Sc,Zr})$ sediments, c) element distribution maps of aluminum-scandium elements, and d) element distribution maps of zinc-magnesium elements.

Figure 11

Analysis of $\text{Al}_3(\text{Sc,Zr})$ and MgZn_2 sediment distribution in the microstructure and volume fraction of sediments in alloy containing 0.1%Sc under thermomechanical treatment (The yellow regions are $\text{Al}_3(\text{Sc,Zr})$ sediments and the blue regions are MgZn_2 sediments) with: a) controlled quenching and 30% rolling, b) conventional dissolution and 50% rolling, and c) controlled quenching and 50% rolling.

Figure 12

TEM micrograph of the specimen containing 0.1%Sc under thermomechanical treatments with 50% deformation and controlled quenching.

Figure 13

EDS analysis of particles in the TEM micrograph given in figure 12: a) spherical particles, and b) rod particles.

Figure 14

Dimples on the fracture surface in the specimen under thermomechanical treatment with controlled quenching and a) 30% rolling, and b) 50% rolling; c, d) nano-sediments at the end of dimples.

Figure 15

a) Size of dimples, and b) percentage of dimples at the fracture surface.