RESEARCH PAPER

# Effect of Minor Amount of Scandium on the Recrystallization Behavior of 7000 Series Aluminium Alloys

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***Abstract:*** *In the present study, the effect of adding minor amounts of scandium and zirconium elements to the 7075 alloy on the re-crystallization behaviour of one aluminium alloy (7000 series) was investigated. For this purpose, two kinds of Al-Zn-Mg-Cu-Sc-Zr alloys with the same amount of Zr and different amount of Sc were prepared. Homogenization durations and temperatures of alloys after alloying were obtained by DSC analysis and optical microstructure observations. The results showed that the optimum homogenization temperatures for Al-Zn-Mg-Cu-0.05Sc-0.1Zr and Al-Zn-Mg-Cu-0.1Sc-0.1Zr alloys were 500 and 4900C respectively, and the optimum duration for both alloys was 12hrs. After homogenization of alloys, the re-crystallization behaviour of the alloys was investigated by Brinell hardness test. Obtained results showed that although the starting re-crystallization temperature for both alloys was similar in 2 hrs, but it was 130°C for alloys with 30% forming, and 120°C for alloys with 50%forming and recrystallization temperature for Al-Zn-Mg-Cu-0.1Sc-0.1Zr alloy was 350in 2 hrs. Despite what was expected, the hardness of Al-Zn-Mg-Cu-0.05Sc-0.1Zr was more than the hardness of Al-Zn-Mg-Cu-0.1Sc-0.1Zr alloy. This can be attributed to the presence of brittle intermetallic compound of Al3Fe.*

***Keywords:*** *Re-crystalyzation, Al-Zn-Mg-Cu-Sc-Zr, 7000 series aluminium.*

## 1. INTRODUCTION

7000 series alloys have been widely applied in aerospace and military industries due to their excellent mechanical characteristics such as high specific strength and low density [1]. Scandium (Sc) is a potential element as grain refinement, strength augment and thermal stability due to the formation of abundant amounts of $Al_3Sc$ particles [2]. According to the aluminium-rich end of the Al-Sc binary phase diagram, a eutectic reaction among the liquid, Al matrix, and $Al_3Sc$ precipitates reacts in the Al-rich region as follows: Liquid → α-Al + $Al_3Sc$ [3,4].

For eutectic reaction, the Sc content should reach about 0.55 - 0.60 wt % [2,5]. However, considering the high price of Sc, to obatain more economic aluminium alloys with outstanding mechanical properties, it is necessary to introduce other rare earth elements with lower price. Zirconium (Zr) as the effective and cheaper rare earth element, is a suitable selection for partial replacement of Sc for its grain refinement and strength augment because of the formation of $Al_3Zr$ particles. However, alloys containing Sc exhibit better grain refinement than ones containing Zr with the same content, which is due to the faster diffusion rate of Sc atoms than that of Zr atoms in melted alloys [6]. Aluminium alloys containing Sc require higher recrystallization temperature and effectively inhibit recrystallization. Scandium is the most effective recrystallization inhibitor element for aluminium alloys. The anti-recrystallization effect of scandium additive is attributed to high density (number of particles per unit volume) of dispersed $Al_3Sc$ particles. Due to the similarity of the aluminium lattice (matrix) to $Al_3Sc$ phase in terms of structure and particle size, during the decomposition of the solid solution, the $Al_3Sc$ particles precipitate in the form of stable, dense spherical solid particles (with the intermediate stage vanished). The small inter-particle space exhibits an effective anti-recrystallization impact which remains in effect as long as the particle and the matrix are coherent. Once the coherency is disturbed, rapid particle growth occurs, the inter-particle spaces enlarge, and the anti-recrystallization effect fades away [7].

Besides, higher strength and hardness can be gained in Al-Zn-Mg-Cu-Sc-Zr alloys after different heat treatments because of precipitate strengthening effect of $Al_3$ (Sc, Zr) particles [8,9].

It has been proved that, dispersed $Al_3Sc_xZr_{1-x}$ particles impose large contributions into interlocking of dislocations and minor boundaries, and tend to greatly retard post-homogenization recrystallization of 7XXX alloy series during mechanical and thermal processes [10]. Van Wu showed that, smaller, more compact spherical precipitates of the $Al_3$(Zr, Sc) are resulted from optimal homogenization conditions and largely contribute to inhibition of the recrystallization. As a consequence, volume fraction of the recrystallized structure decreases, and hence higher strength is obtained following the aging treatment in Al-Zn-Mg-Sc-Zr alloys [11].

It is obvious that, none of the aluminide phases formed with zirconium and scandium can inhibit the recrystallization, but the $Al_3$ (Zr, Sc) imposes a large inhibitory effect on the dislocation movements, as compared to the $Al_3Sc$ phase of the alloy [12]. The resultant delay in the recrystallization phenomenon and exact temperature and time at which the recrystallization comes to completion in different alloys depends on their content of scandium and/or zirconium and the processes performed on the alloy.

Weng et al. observed that the nucleation mechanism of recrystallization in Al-Mg-Mn-Sc-Zr alloys was sub grain coalescence and sub grain growth. Moreover, they indicated that the re-crystallization temperature was increased to 450°C due to the high density of $Al_3Sc$ precipitates with strongly pinning effect in dislocations and sub grain boundaries [13].

Deng et al. studied recrystallization mechanism and mechanical properties of Al-Zn-Mg-Cu-Sc-Zr alloys using the adding rare-earth elements of 0.10SC/0.10Zr and 0.25Sc/0.10Zr (wt%). They reported that the anti recrystallized effect and mechanical performances were promoted with the increasing micro alloying content of Sc [11].

In this study, the effect of adding minor amounts of scandium and zirconium elements to the 7075 alloy on the recrystallization behaviour of an aluminium alloy (7000 series) was investigated.

## 2.EXPERIMENTAL PROCEDURE

### 2.1. Material Preparation

For casting of alloys, at first, a model (containing 4 vertical moulds with the dimensions of 200 mm×40 mm×40 mm) was designed and constructed cast iron mould and after casting, the mould was put under turning and milling. The schematic of the mould layout is shown in the Fig. 1.

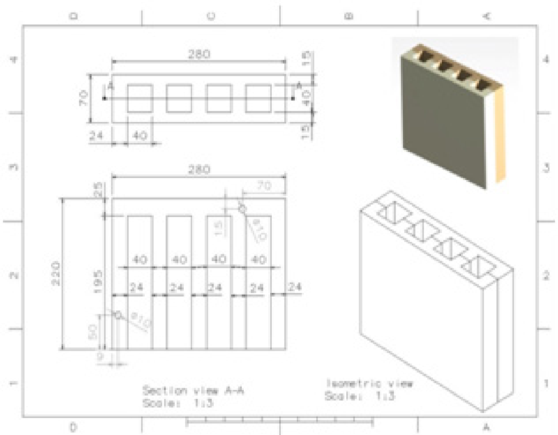


**Fig. 1.** Schematic of the mould.

### 2.2. Melting and Casting

7075 aluminium alloy was melted at 750°C. The melting operation was carried out under argon gas in a resistive furnace. Since the Sc in the master alloy was in the form of $Al_2Sc$ and the melting temperature of this phase is 800°C, the latter temperature was selected as super heating temperature, so, when the furnace temperature reached 800°C, Al-2Sc and Al-15Zr master alloys were added to the melt and then, the precipitations in the master alloys were allowed to be dissolved for 30 min. In order to homogenize the melt, the mixture was mixed by using a graphite lancer containing argon. At the end of melting, to adjusting the amount of zinc and magnesium, Al-Zn and Al-Mg master alloys were added to the melt. The ingots were cast in the cast iron mould. Two kinds of aluminum alloys with same Zr amount (0.08%) and different Sc amount (0.05% and 0.1%) were prepared by the explained method.

**Table 1.** Quantumetric analysis results 7000series+0.05Sc.

| Al | Zn | Mg | Cu | Ti | Zr | V | Fe | Pb | Sn | Si | Mn | Cr | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Balanced | 5.29 | 2.47 | 1.9 | 0.126 | 0.079 | 0.0091 | 0.5 | 0.045 | 0.009 | 0.235 | 0.119 | 0.0954 | 0.0138 |

**Table 2.** Quantumetric analysis results 7000series+0.1Sc.

| Al | Zn | Mg | Cu | Ti | Zr | V | Fe | Pb | Sn | Si | Mn | Cr | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Balanced | 6.14 | 3.32 | 1.19 | 0.07 | 0.082 | 0.008 | 0.24 | 0.0025 | 0.009 | 0.153 | 0.0934 | 0.172 | 0.014 |

The amount of scandium was analyzed by the ICP method. From now on, the Al-Zn-Mg-Cu-0.08Zr-0.05Sc and Al-Zn-Mg-Cu-0.08Zr-0.1Sc alloys are called 7000 series+0.05Sc and 7000 series+0.1Sc, respectively.

The results of the performed quantum analysis on these alloys are presented in Tables 1 and 2.

### 2.3. Preparation of Basic Test Samples

After casting and conducting ICP and quantumetric tests, casting ingots were cut to 100 mm × 20 mm × 6 mm by wire cut and then polished.

## 3. RESULTS AND DISCUSSION

### 3.1. DSC Analysis

In order to study the transformations temperatures in the casting alloys and the temperature for homogenization heat treatment, the samples were subjected to DSC analysis before and after homogenization. For analysis, the specimens were carefully weighted and placed on a 40-micron aluminium standard crucible. At first, the crucible cap was pierced with a needle and then placed on the crucible and tightened. The crucible was placed inside the machine and is heated in the temperature range of 0 to 550$^0$C at an appropriate heating rate under the atmospheric atmosphere.

In order to determine the heating rate of the samples, at first, 10°C/min was selected and a sample of 7075 alloy was analyzed. Since the DSC analysis did not show complete transformations temperatures at 10°C/min heating, the sample was subjected to DSC analysis at a heating rate of 5°C/min. The result of the DSC analysis at the lower rate is presented in Fig. 2.

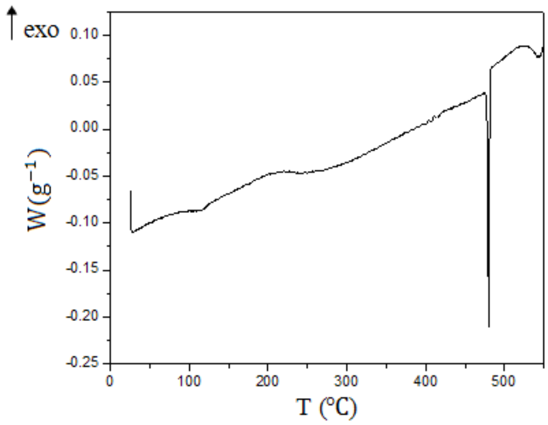


**Fig.2.** The result of the DSC analysis on a sample of casting 7075 alloy (heating rate:5°C / min).

The result of the analysis at 5$^0$C/min showed more transformations points in the analyzed sample, so that the 7000 series + 0.1%Sc and the 7000 series + 0.05%Sc alloys were subjected to the DSC analysis with the same heating rate of 5$^0$C/min. The results of the DSC analysis of the 7000 series + 0.05% Sc and 7000 series + 0.1% Sc casting alloy specimens are shown in Figs 3 and 4, respectively.

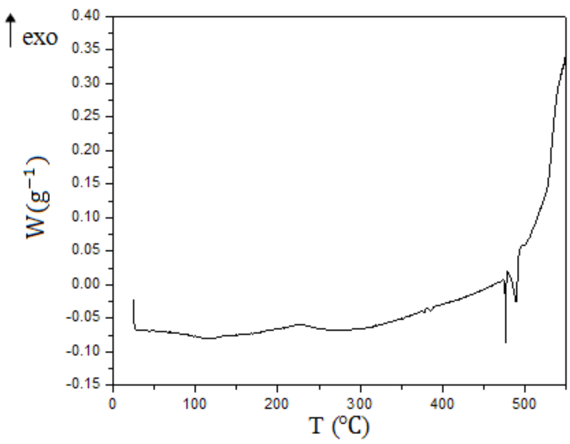


**Fig.3.** DSC 7000 series+0.1% SC casting alloy with (heating rate: 5$^0$C/min)





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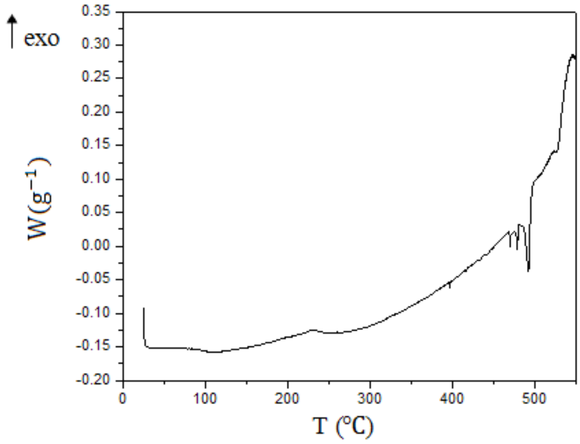


**Fig.4.** DSC 7000 series+0.05% SC casting alloy with heating rate:5$^0$C/min

Figs. 4 and 5 show that the final endothermic temperature in the 7000 series+0.1%Sc and 7000 series +0.05%Scare respectively 482°C and 486.21°C. According to the results, the temperature of the homogenization of this alloy should be above this temperature.

**3.2. Determining the Temperature and Time of Homogenization**

To determine the homogenization time and temperature, the casting specimens were homogenized at about 500°C in a heat treatment furnace (Nabertherm furnace) for 3, 5, 10 and 12 hrs and then underwent optical microscopy. The chlorine etch solution used for etching the samples for 20 – 30 seconds. The metallographic results of the casting samples and the metallographic images of the homogenized samples are shown in Figs. 5 .and 6, respectively

Base on the microscopic observation, to ensure complete omission of dendritic structure, homogenization process was continued for 12 hrs.

For exact determining of homogenization temperature, specimens were subjected to different temperatures in respect of DSC diagrams (470, 480, 490 and 500°C).

Based on microscopic observation, optimum temperatures for homogenization of 7000 se-

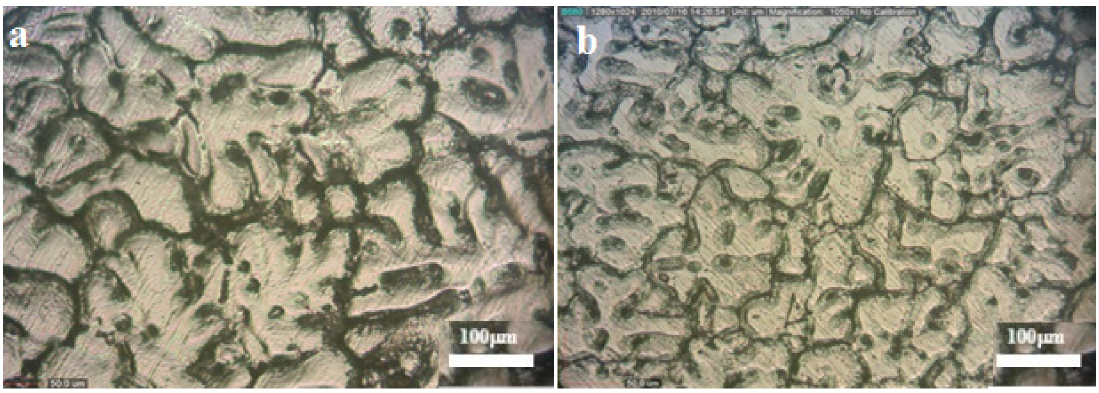


**Fig.5.** The metallographic images of the casting samples , a) 7000 series+0.1Sc, and b) 7000 series+0.05Sc

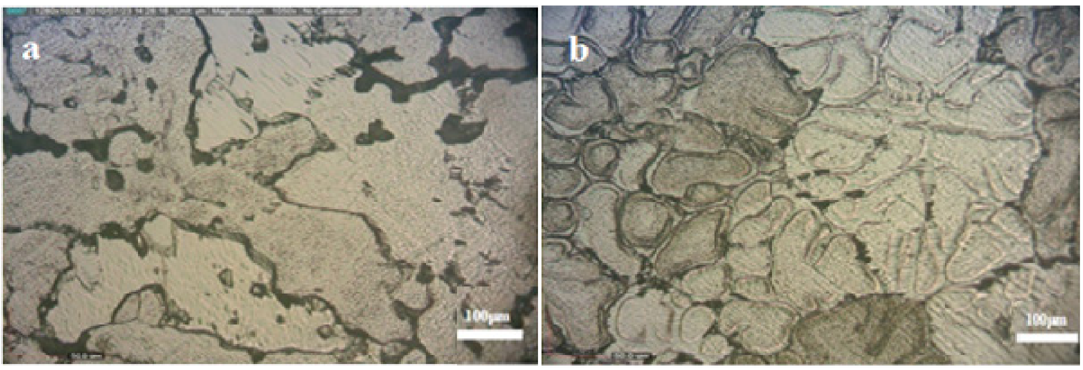


**Fig.6.** Metallographic images of homogenised alloys, a) 7000 series+0.1Sc homogenised temperature and time is 500°C and 12hrs, and b) 7000 series+0.05Sc homogenised temperature and time is 490°C and 12hrs.





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ries+0.1Sc and 7000 series+0.05Sc were found to be 500°C and 490°C, respectively.

### 3.3. Annealing Behaviour of the Alloy

To investigate annealing of the alloy specimens, the samples with dimensions of 50mm×10mm×6mm were subjected to secondary annealing and cold rolled at 30 and 50%. Then, the specimens were cut to a length of 10mm and exposed to temperatures of 150, 200, 250, 300, 350, 400, and 450 for 2 h inside a furnace. Finally, the samples were cooled in the furnace, and subjected to hardness measurement. The results of hardness .measurement are shown in Figs. 7 and 8

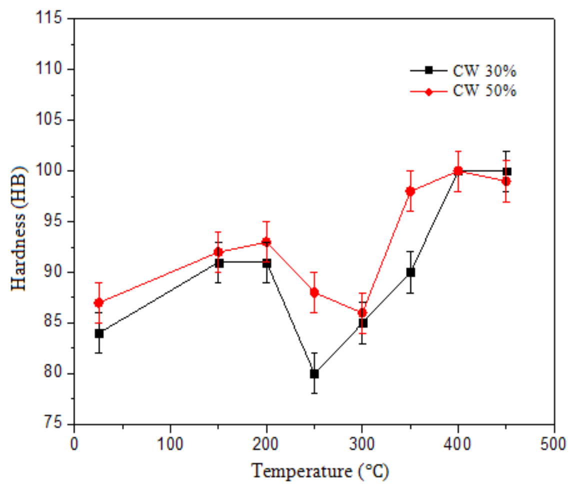


**Fig. 7.** Effect of temperature on the hardness of Al-Mg-Zn+0.05%Sc.

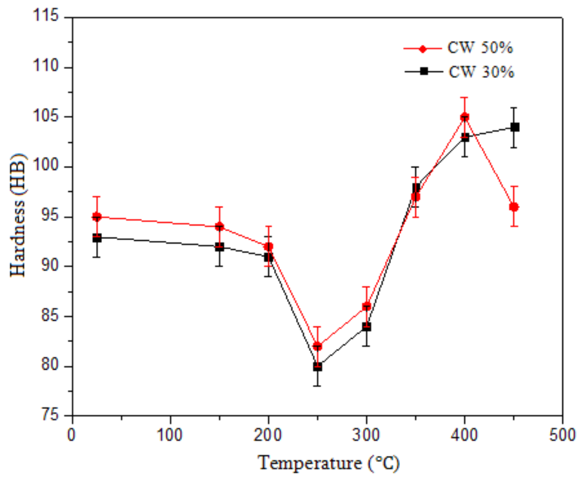


**Fig. 8.** Effect of temperature on the hardness of Al-Mg-Zn+0.1%Sc.

As shown, the hardness curves could be divided into three regions. The first region is temperature recovery stage with a decrement in hardness that occurs between 150 and 250. In this region, dislocations density made by cold work will be reduced; so, the hardness is reduced, too. Since the low diffusion coefficient of Sc in Al [14] and its high thermal stability, the formation of $Al_3$ (Sc,Zr) precipitate starts in high temperature. Therefore, the second region is $Al_3$ (Sc,Zr) precipitate stage with an enhancement in hardness. After 400, the hardness value decreases due to many of recrystallized grains and their growth.

To determination of recrystallization temperature, the samples were cooled in air after exposure to temperatures of 120, 130, 150, 200, 250, 300, 400, 450, and 500°C for 2 h.

### 3.4. Recrystallization Behaviour of the Alloys

After homogenization of the samples with a thickness of 6 mm according to thermal analysis and microscopic images, they were cold rolled to a thickness of 5 mm.

Prior to further forming, the samples were subjected to secondary annealing. The 7000series+0.05Sc and 7000series+0.1Sc samples were annealed for 6hr at 490 and 500°C, respectively and formed to 30% and 50% by cold rolling. To determine recrystallization temperature, the samples were cut to a length of 10mm and placed in a furnace at temperatures of 120, 130, 150, 200, 250, 300, 400, 450, and 500 for 2hrs. Then, they were air-cooled and subjected to Brinell hardness test. The results of re-crystallization temperature are shown in Figs. 9 and 10.

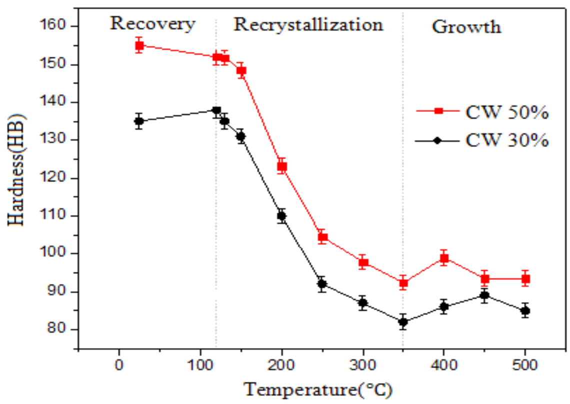


**Fig. 9.** The effect of annealing temperature on the hardness of 7000series +0.1%SC.





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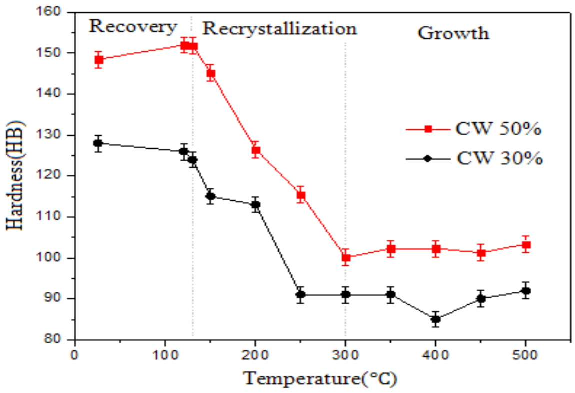


**Fig. 10.** The effect of annealing temperature on the hardness of 7000series +0.05%SC.

According to Fig. 9, the the recrystallization begins at 120 but as can be seen in Fig. 11(a), a small number of grains have recrystallized ("A" region in Fig. 11(a)), and recrystallization is not observed in the other regions. Also, it is declared that in the elongated grains, the precipitates have preferred orientation along the rolling direction ("B" region in Fig. 11(a)). "C" region shows grains that are completely elongated and not recrystallized. According to Fig. 12(a), in case of cold worked (but not annealed), all grains are elongated along the direction of rolling. FESEM images (Fig. 12(b)) indicate no recrystallized grains and a very small fraction of co-

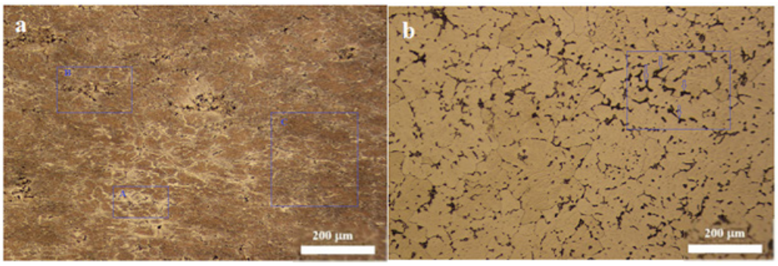


**Fig. 11.** Metallographic images of annealed alloy at 120,
a) recrystallized and not recrystallized grains regions, b) preferred orientation of precipitates along the rolling direction.

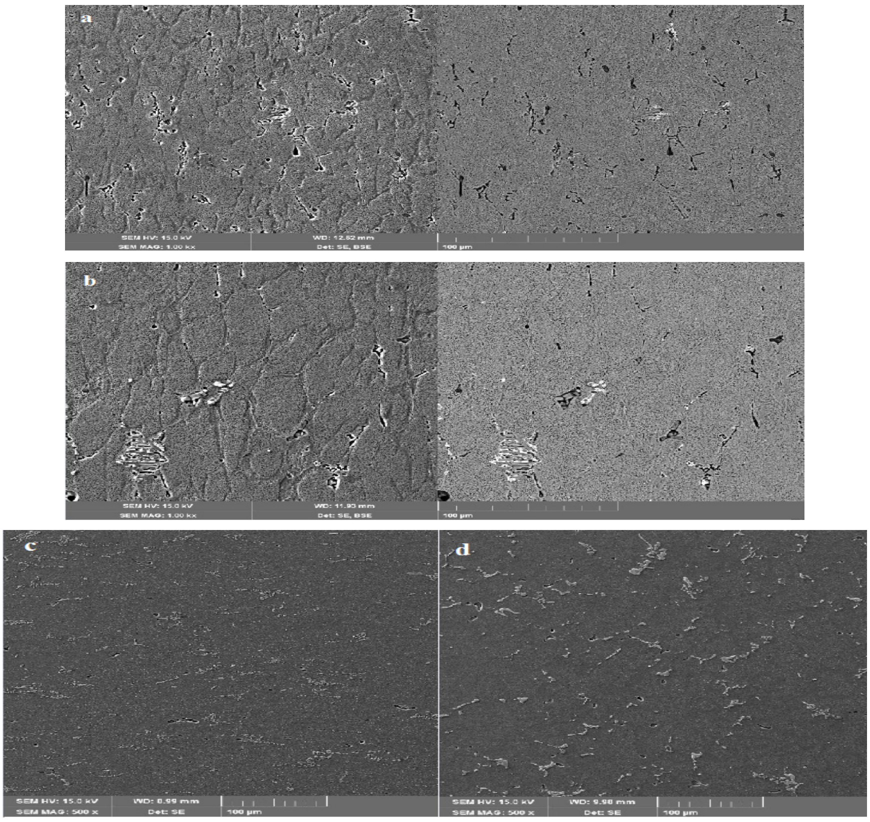


**Fig. 12.** FESEM images of cold worked alloy (not annealed), a) preferred orientation of grains along the rolling direction, b) small fraction of coaxial grains, c) second phases the direction of rolling, d) orientation of particles in case of homogenization.




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axial grains. In Fig. 11(b), arrow marks show the preferred oriented precipitates along the rolling direction and at grain boundaries.

FESEM images (Fig. 12(c)) also show second phases along the direction of rolling where it can be seen that, in case of homogenization, particles do not have any preferred orientation (Fig. 12(d)).

The reduction of hardness after 120 can be attributed to the increase in system internal energy due to cold working which increases the crystal defects and voids. When temperature reaches to 120, the energy level reduces leading to dislocation density reduction and system's total energy drop and consequently, results in hardness reduction. Presence of $Al_3$(Sc,Zr) nanoparticles is the main reason for recrystallization lack at 120, which is shown in Fig. 13.

The effects of $Al_3$(Sc,Zr) particles on the recrystallization behavior can be caused by two factors including the function of particles as nucleation sites for recrystallization in Al alloy, and their considerable pinning effect on grain boundaries. Depending on particle size, shape, and volume fraction, they can suppress recrystallization [15]. These coherent $Al_3$ (Sc,Zr) particles [16] in Al matrix are distributed in subgrain interiors and on sub grain boundaries. They lock the dislocations and prevent recrystallization. Dispersoid $Al_3$ (Sc,Zr) particles have an excellent thermal stability. To investigate the effect of time on recrystallization behavior, annealing process was carried out at constant temperature of 120 for 12hrs. Fig. 14(a) shows FESEM images of the samples. As shown, increasing annealing time results in more recrystallized and coaxial grains, but there are still no recrystallized grains.

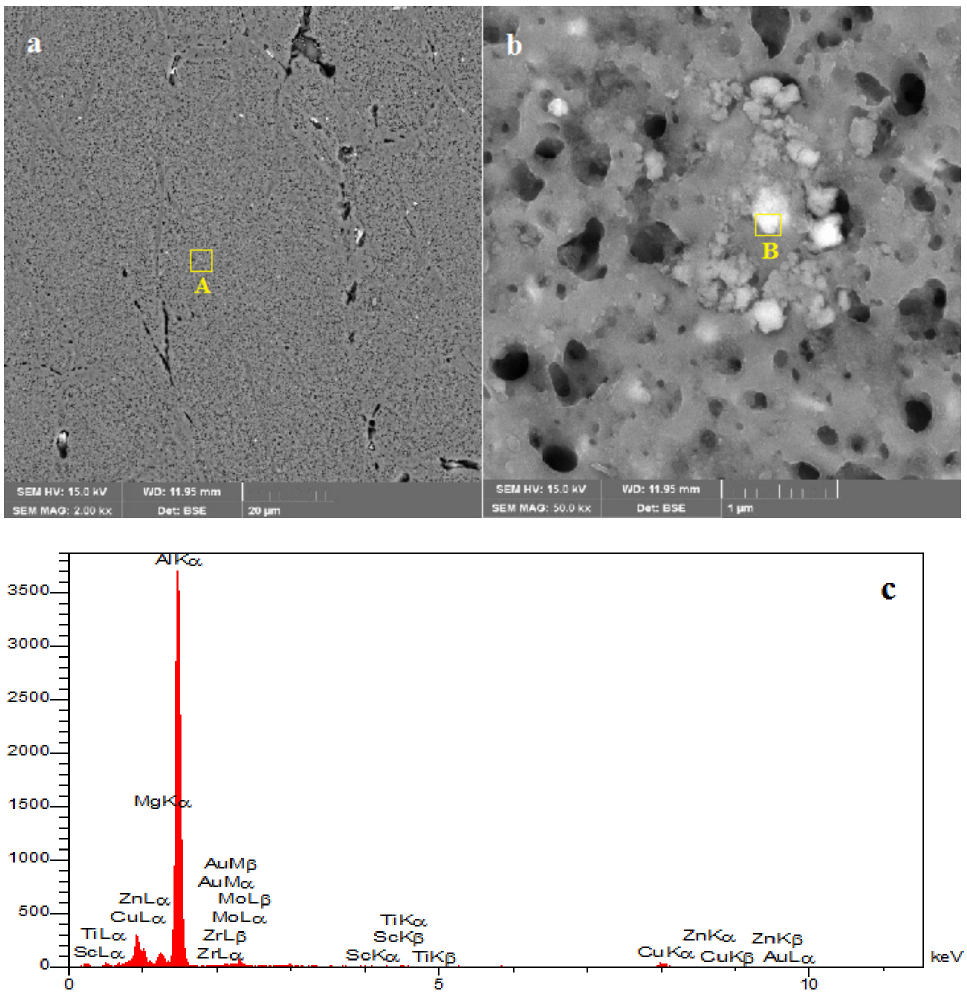


**Fig. 13.** FESEM of annealed alloy(7000series+0.1Sc+0.0.08Zr) at 120, a) presence of $Al_3$ (Sc,Zr) nanoparticles, b) magnification of "A" zone in image a, c) EDS analysis of "B" zone in image b.





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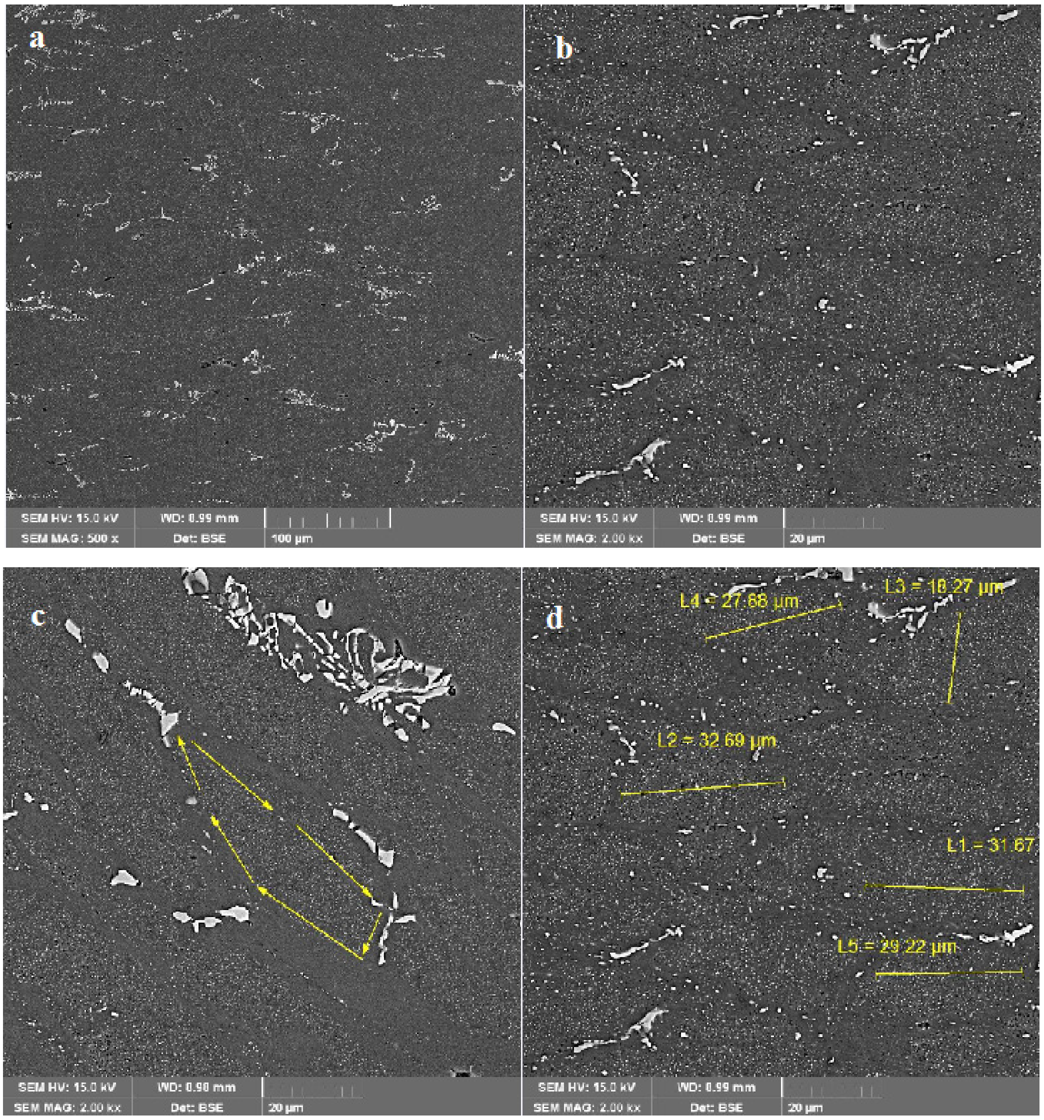


**Fig. 14.** a) FESEM images of annealed alloy(7000series+0.1Sc+0.1Zr) at 120 for 12hours, b) FESEM images of annealed alloy at 350 for 2hours, c) grain length at 120/12hours, d) grain length at 350/2hours.

To assess the effect of temperature on recrystallization behavior, annealing process was carried out at temperature of 350 and constant time of 2hrs and microstructural investigation was conducted by FESEM. According to the hardness curves of Fig. 9, the temperature of 350 is the final temperature of recrystallization region.

According to Fig. 14(b), the grains are coaxial and elongated grains are partially observed. Fig. 14(c, d) shows results of the elongated grains at different times and temperatures. As shown in Fig. 14(c, d), the grains at 120/12hrs are longer than that for 350/2hrs. This observation shows that the effect of temperature on recrystallization is more than time. Therefore, the recrytallization temperature of Al-Zn-Mg-Cu-0.1Sc-0.1Zr is 350. Deng et al [11] reported that, as a result of annealing at 350, there was a large cube band which retains from the initial cube grain and different crystal orientations under different boundary angles show cube orientation of subgrains which promote nucleation.

The third region of the graph in Fig. 9 is grain growth region. In this region, the hardness remains almost constant with increasing temperature. The start of growth temperature is approximately 350.

The effect of Sc content on recrystallization behavior was investigated in the alloys with the



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same content of Zr and different Sc. Recrystallization diagrams of the alloys are shown in Fig. 10 . As can be seen, this curve also fallows the usual form of recrystallization diagrams and has three regions of recovery, recrystallization, and grain growth.

Fig. 15 shows the effect of Sc content on hardness and recrystallization behavior of  alloy.

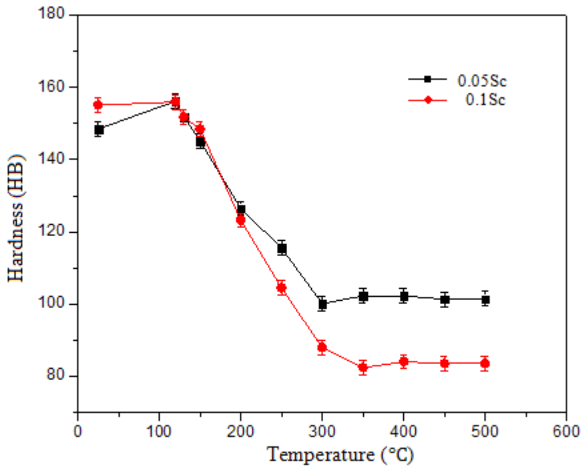


**Fig. 15.** Effect of Sc content on hardness and recrystallization behavior.

According to the results of reported in the literature [17,18], enhanced hardness is expected by increasing Sc content. But, according to Fig. 15, the hardness of 7000series+0.05Sc alloy is more than that of 7000series+0.1Sc alloy. In the alloy containing 0.05Sc, hardness is increased due to presence of brittle intermetallic compound of $Al_3Fe$. Fig. 16 shows optical microscopy images of 7000series+0.05Sc alloy. According to the Fig.16, the grains are in a coaxial state, and there are many recrystallized grains at 120. The presence of $Al_3Fe$ intermetallic compound can be observed in Fig. 16(b).

Fig. 17 shows FESEM images and EDS analysis of the alloy. According to EDS analysis of Fig. 17, "A" region shows Fe-rich phase. The amount of Fe in 7000series+0.05Sc alloy is more than that in 7000+0.1Sc alloy (Table 1 and 2). Therefore, the amount of $Al_3Fe$ intermetallic compound is higher in 7000+0.05Sc alloy and it has more effect on the hardness and mechanical properties of the alloy.

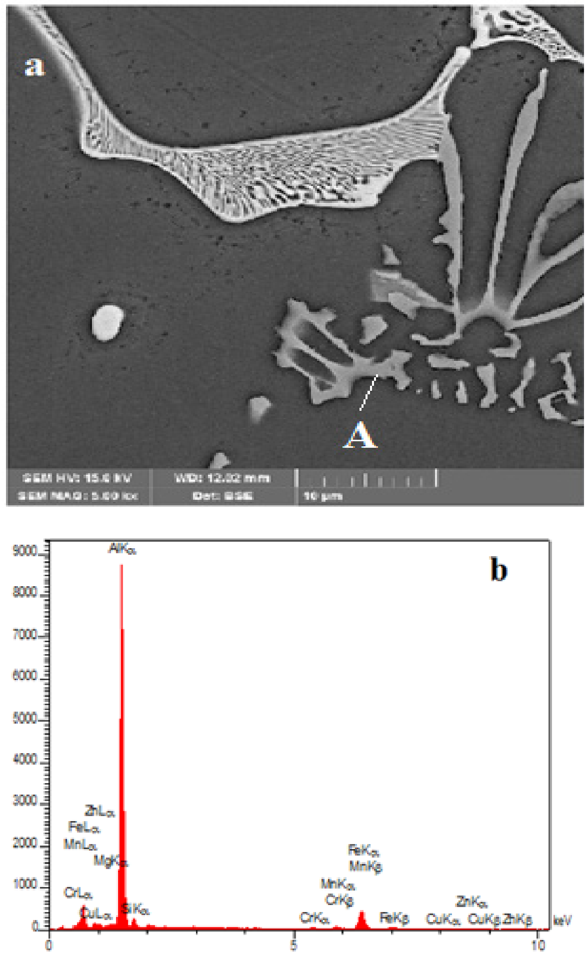


**Fig. 17.** FESEM and EDS analysis of 7000series+0.05Sc alloy at 120

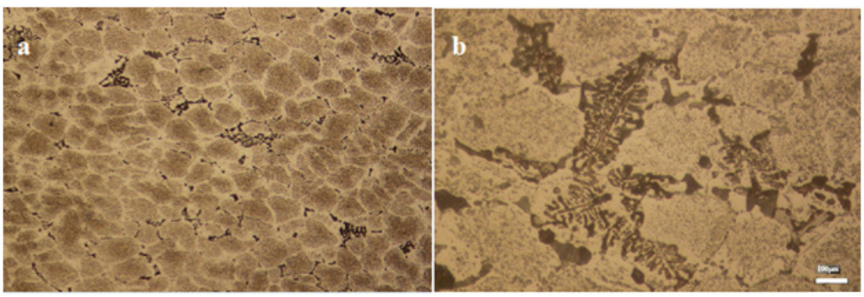


**Fig. 16.** Optical microscopy images of 7000series+0.05Sc-0.1Zr alloy at 120, a) coaxial and recrystallized grains, b) presence of $Al_3Fe$ intermetallic compound.





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## 4. CONCLUSIONS

1. According to DSC analysis and optical microscopic images, the optimum homogenization temperatures of 7000 series + 0.05Sc and 7000 series + 0.1Sc were found to be 490 and 500°C, respectively. Homogenization time for both alloys was 12 hours.
2. The recrystallization temperature for both alloys in the 50 and 30% cold rolling were 120 and 130$^0$C, respectively In these alloys, according to the low amount of scandium and high amount of cold work, its effect on the re-crystallization temperature is more than the effect of scandium amount.
3. The starting temperature of recrystallization and obtaining the highest amount of recrystallized grains for 7000series+ 0.1Sc alloys was 350.
4. The maximum hardness for the alloy with 0.1% Scandium was138 Brinell and for the alloy with 0.05% Sc was143 Brinell. In the alloy containing 0.05Sc, hardness is increased due to presence of brittle intermetallic compound of $Al_3Fe$.

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