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Effect of Sub-Zero Treatment on Tensile Properties of Aluminum 2024

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A B S T R A C T
Challenge of change of alloy properties during service life of extra safety parts was always considered by industrialists and consequently researchers. Fuselage and wings of airplane repeatedly in ascent and descent is affected respectively by cooling to -55 0C and heating to room temperature. Hence, the effect of above temperature changes on tensile properties is unknown. In this study in laboratory, the situation of airplane body is tried to simulate and then the changes of microstructure and consequently the changes of tensile properties and hardness after holding for 10 and 4 hours were studied respectively in temperature -600C and -1960C. Results showed that by using of sub-zero treatment, hardness is without change but tensileproperties are increased to control sample and when the cooling rate was low (≈1 degree per minute), more improvement will be achieved in tensile properties

Keywords: Population density of particles, Liquid nitrogen, Sub-Zero treatment Toughness

1. Introduction

Challenge of change of alloy properties during service life of extra safety parts was always considered by industrialists and consequently researchers because after controlling the quality of extra safety parts, if the change of alloy properties is along with drop, it is resulted in premature failure ordestruction suddenly [1]. Fuselage of passenger airplanes is aluminum alloy 7075 or aluminum alloy 2024. Both alloys are precipitation hardening and after precipitation hardening, their yield strength will be increased up to four times. In this situation, very low density of noted alloys causes increasing specific strength of these alloys, but these relatively cheap alloys will be unrivaled for public applications in air-space [2]. The studies on aluminum alloys showed that holding in cooling and then heating causes change in microstructure and at last change of properties. For example Nayan et al [3-5] reported the improvement of tensile properties of aluminum alloy 2195 after the holding at very low temperatures (-2530 C). Also Li et al [6] reported the improvement of strength of aluminum alloy of group 5000 after holding at the temperature of -1960 C. On the other hand, if the cooling rate will be very low to sub-zero temperature (less than 10C per minute), the results of improving fatigue strength and increasing tensile strength of steels are appeared [7] while high cooling rate (more than 30 C per minute) shows the results of decreasing tensile strength and dropping fatigue strength [8]. The reason of improving above properties is attributed to the precipitation of new and nano-size carbide particles in the matrix of steel and also decreasing residual stresses in microstructure of steel (at low cooling rate) while decreasing above properties is attributed to the formation of micro cracks (at high cooling rate). Hence after airplane taking off, very low temperature (about -550 C) in the time of flight and then room temperature in descend causes change microstructure of airplane body alloy that is along with changing physical and mechanical properties of the alloy. So in this study the main purpose is answer to this question that how is the effect of cooling and heating respectively during flight and rest in hangar on tensile properties and the hardness of aluminum alloy 2024? Many studies were performed on using low temperatures for the improvement of formation process of aluminum alloys of group 1000 [9], 2000 [10], 5000 [11, 12], 6000 [13], and 7000 [14]. While airplane body is repeatedly affected in ascend and descend respectively by cooling to -550 C and heating to room temperature. Hence the effect of above temperature changes is unknown on hardness and tensile properties. In this study in laboratory, the situation of airplane body of aluminum alloy

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2024 is tried to simulate and then the changes of microstructure and consequently the changes of tensile properties and the hardness of aluminum alloy 2024 which is cooled with different rates, after holding for 10 and 4 hours will be studied at sub-zero temperatures of -600C and -1960C, respectively.

2. Materials and Methods

In this study, 3 set of samples used for studying microstructure, hardness and tensile properties (Figure 1). Chemical composition of aluminum alloy 2024 used in this study listed in Table 1.(Figure 1). Chemical composition of aluminum alloy 2024 used in this study listed in Table 1.

Figure 1: Dimension of tensile samples

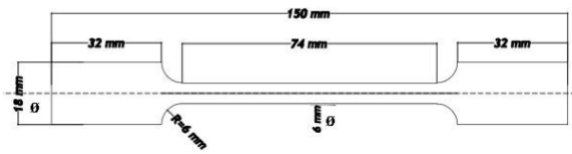


Figure 1: Dimension of tensile samples

Table 1: Chemical composition of aluminum alloy 2024

Element	Zn	Fe	Mg	B	V	Pb	Cu	Si	Mn	Al
Wt%	0.12	0.28	1.4	0.003	0.02	0.02	4.4	0.14	0.43	Balance

Element Zn Fe Mg B V Pb Cu Si Mn Al Wt% 0.12 0.28 1.4 0.003 0.02 0.02 4.4 0.14 0.43 Balance

For the study of microstructure, field emission (gun) scanning electron microscopy (FESEM) with commercial name of TESCAN MIRA3 and also scanning transmission electron microscopy (STEM) with commercial name TITAN with the power of 300 KV equipped with energy dispersive spectroscopy (EDS) system was used. GOTECH 7100L machine was used for measuring tensile properties and diamond pyramid indenter was used with the force of 10 kg and the time 10 s for Vickers hardness test. Valid laboratory centers (confirmed by Iran standard institute) were used for accuracy and precision of results.For the study of microstructure, field emission (gun) scanning electron microscopy (FESEM) with commercial name of TESCAN MIRA3 and also scanning transmission electron microscopy (STEM) with commercial name TITAN with the power of 300 KV equipped with energy dispersive spectroscopy (EDS) system was used. GOTECH 7100L machine was used for measuring tensile properties and diamond pyramid indenter was used with the force of 10 kg and the time 10 s for Vickers hardness test. Valid laboratory centers (confirmed by Iran standard institute) were used for

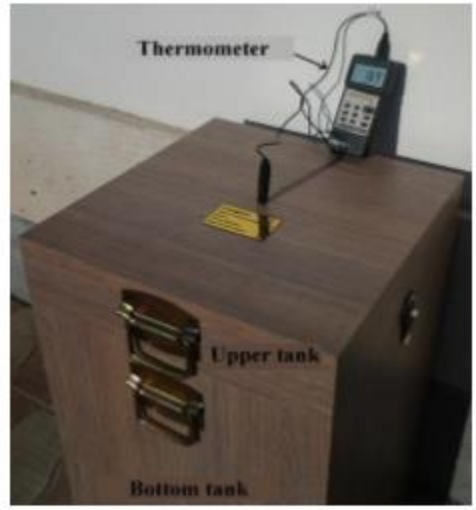
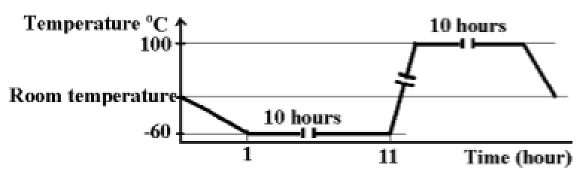


Figure 2: Sub-zero treatment equipment

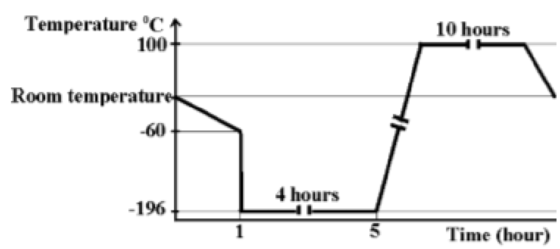
The last stage is tempering. For performing tempering, the only sub-zero samples (group 1 and group 2) were placed in the temperature of 1000 C (boiling temperature of water during cleaning procedure of airplane) for 10 hours.

Finally, all tensile samples (control, group 1 and group 2) were paid by grinding and then polishing.

Sub-zero treatment cycle and flowchart of research method are explained in Figure 3 and Figure 4, respectively.



(a)Group 1 sub-zero treatment cycle at -60oC



(b)Group 2 sub-zero treatment cycle at -196oC (pure liquid nitrogen)

Figure 3: Sub-zero treatment cycle (a) Group 1 sub-zero treatment cycle at -60oC (b) Group 2 sub-zero treatment cycle at -196oC (pure liquid nitrogen)

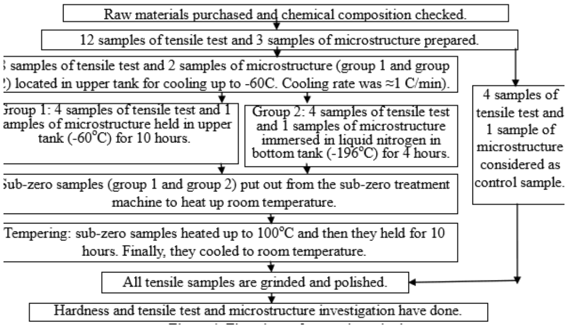


Figure 4: Flowchart of research method

3. Results and Discussion

Chemical composition of particles in control, group 1 and group 2 samples determined by EDS. They are showed in Figure 5, Figure 6 and Figure 7, respectively.

In control sample, it is observed that dark particles including A, B, C rich in elements are light towards copper while bright particles are included into D particle rich in elements towards such as lead. Al2Cu compound including 33.3 atomic percent of copper and the rest aluminum while A, B, C particles are rich particles of copper between 9 and 23 atomic percent. Therefore, these particles are transfer compounds Al2Cu. Based on phase diagram of aluminum-lead (Figure 8), since lead and aluminum do not make any compounds or solutions, the bright particle D is pure lead, but present

Raw materials purchased and chemical composition checked. 12 samples of tensile test and 3 samples of microstructure prepared. 4 samples of tensile test and 1 sample of microstructure considered as control sample.

8 samples of tensile test and 2 samples of microstructure (group 1 and group 2) located in upper tank for cooling up to -60C. Cooling rate was ≈1 C/min). Group 1: 4 samples of tensile test and 1 samples of microstructure held in upper tank (-60oC) for 10 hours. Group 2: 4 samples of tensile test and 1 samples of microstructure immersed in liquid nitrogen in bottom tank (-196oC) for 4 hours. Hardness and tensile test and microstructure investigation have done.

Tempering: sub-zero samples heated up to 100oC and then they held for 10 hours. Finally, they cooled to room temperature. All tensile samples are grinded and polished. Sub-zero samples (group 1 and group 2) put out from the sub-zero treatment machine to heat up room temperature. aluminum in its EDS is derived from the matrix and it is not related to the D particle.

In group 1, it is observed that particles are included into A, B, C and D particles, rich in elements, relatively light such as copper and magnesium (especially for particle A). The compound Al2Cu is included in 33.3 atomic percent of copper and the rest aluminum while A, B, C, D particles, rich in copper are between 6 and 26 atomic percent. Therefore, these particles are transfer compounds of Al2Cu. In particles B, C, D there is iron impurity up to 5 atomic percent while particle A has no impurity and it is included in about 55 atomic percent of aluminum and 26 atomic percent of copper and 18 atomic percent of magnesium, so chemical composition of particle A is as Al2CuMg1.5 (general form is AlCuxMgySiz). In group 2, it is observed that particles are included into A, B, C, D particles, rich in elements, relatively light such as copper and magnesium (especially for particle A). The compound Al2Cu is included in 33.3 atomic percent of copper and the rest aluminum while A, B, C, D particles, rich in copper are between 5.5 and 22 atomic percent. Therefore, these particles are transfer compounds of Al2Cu. In particles B, C, D there is iron impurity up to 6 atomic percent while particle A almost has no impurity and it is included in about 58.5 atomic percent of aluminum and 22 atomic percent of copper and 19 atomic percent of magnesium, so chemical composition of particle A is as Al3CuMg (general form is AlCuxMgySiz). Result of average of grain size, population density of particles, hardness and tensile test are listed in Table 2.

Sample	Grain size	Population density of particles	Hardness	Elongation	Yield strength	Tensile strength
Control	3.8 µm±3%	5547 mm^{-2}±5%	128 HV±3%	4.0 %±10%	366 MPa±5%	437 MPa±3%
Group 1	3.3 µm±3%	6934 mm^{-2}±5%	126 HV±3%	5.5 %±10%	390 MPa±5%	462 MPa±3%
Group 2	3.3 µm±10%	7858 mm^{-2}±5%	127 HV±3%	5.0 %±10%	398 MPa±5%	463 MPa±3%

Table 2: Average results of grain size, population density of particles, hardness, elongation, yield strength and tensile strength for all samples Tensile strength Yield strength Elongation Hardness Population density of particles Grain size Sample 437 MPa+3% 366 MPa +5% 4.0 % +10% 128 HV +3% 5547 mm -2+5% 3.8 µm +3% Control 462 MPa+3% 390 MPa +5% 5.5 % +10% 126 HV +3% 6934 mm -2+5% 3.3 µm +3% Group 1 463 MPa+3% 398 MPa +5% 5.0 % +10% 127 HV +3% 7858 mm -2+5% 3.3 µm +10% Group 2

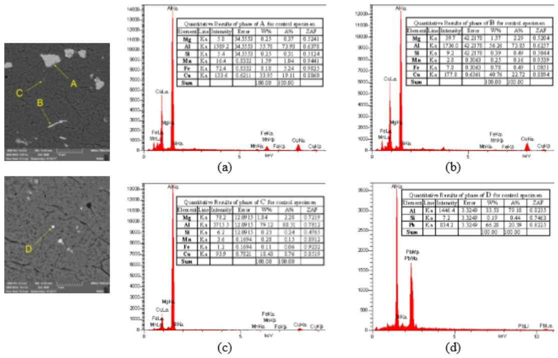


Figure 5: EDS of phases for control sample

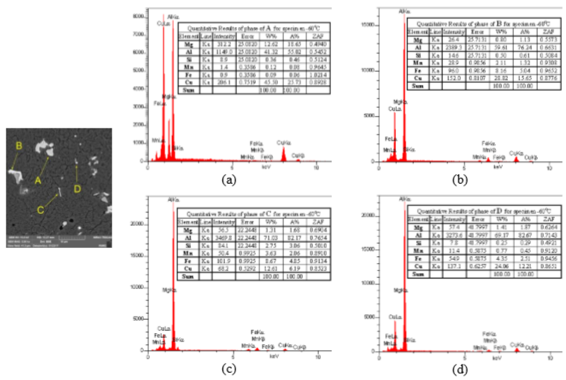


Figure 6: EDS of phases for group 1

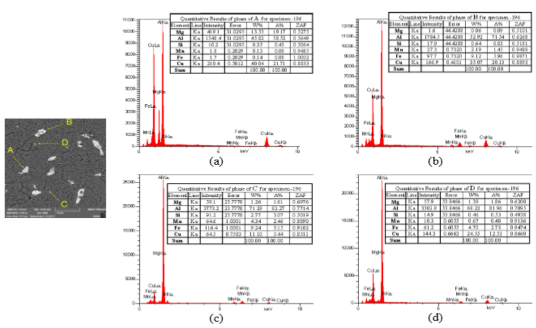


Fig 7EDS of phases for group 2

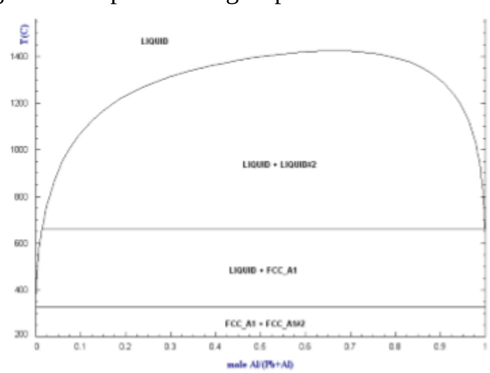
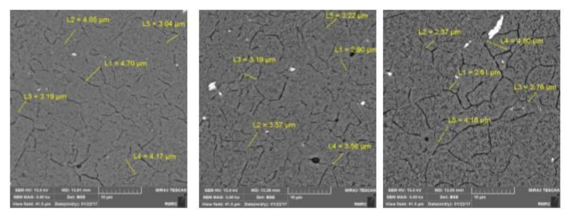


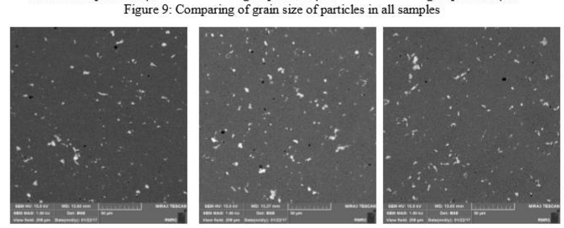
Figure 8: Phase diagram of Al-Pb

According to Table 2, in this study with performing sub-zero treatment at temperature -60oC (group 1) and -196oC (group 2), the elongation, yield strength and tensile strength towards control sample were increased 1.5 and 1 percent, 24 and 32 MPa and 25 and 26 MPa, respectively. Present results are consistent with the results of Nayan et al [3]. They found that temperature -1960C increases the elongation, yield strength and tensile strength of aluminum alloy 2195 2 percent, 45 MPa and 79 MPa towardstemperature of room, respectively and also temperature -2530C increases the elongation, yield strength and tensile strength of aluminum alloy 2195 2 percent, 93 MPa and 139 MPa towards temperature of room, respectively. Transmission electron microscopy (STM) studies showed that the formation of new plate-shaped precipitate Al2CuLi which are precipitated on {111} with the same structure of aluminum is the factor of this improvement. The images of grain size and population density of particles were showed in Figure 9 and Figure 10, respectively.



(a)FESEM image of grain size of control sample is 3.8 µm (b)FESEM image of grain size of group1 is 3.3 µm c)FESEM image of grain size of group2 is 3.3 µm

Figure 9: Comparing of grain size of particles in all samples

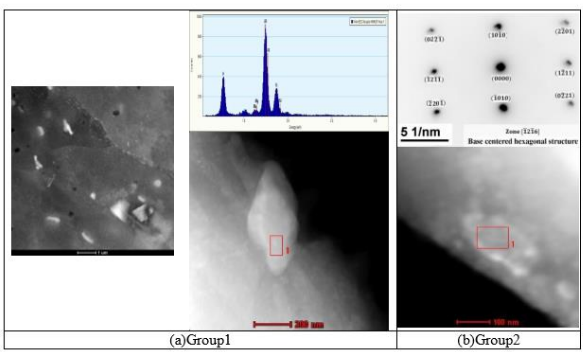


(a) FESEM image of population density of control sample is 5547 mm^{-2} (b) FESEM image of population density of group 1 is 6934 mm^{-2} (c) FESEM image of population density of group 2 is 7858 mm^{-2}

Figure 10: Comparing of population density of particles in all samples

In present study, only yield strength of the sample of group 1 (-600C) has trivial increasing towards the sample of group 2 (-1960C) while the elongation is 0.5 percent of declining and tensile strength is nearly the same. Also in the study of Nayan [3] when sub-zero temperature reaches from -1960 C to -2530C, the elongation is without change but yield strength and tensile strength show increasing 48 MPa and 60 MPa, respectively. The reason of this difference is in the cooling rate. Based on Figure 3b, in the present study, the sample of group 2 from -600C to -

1960C was cooled suddenly that can be the origin of the formation of micro cracks while in the study of Nayan et al [3] there is no point to the cooling rate but it is supposed that the cooling rate was so slow (less than 1 degree per minute). Based on Table 2, increasing tensile properties of sub-zero samples (group 1 and group 2) towards control sample is attributed to decrease the size of grain (from 3.8 micrometer to 3.3 micrometer) and on the other hand to increase population density of particles (from 5547 to 7858 particles per each square millimeter). Above result is consistent with results of Li et al [6]. They found that sub-zero treatment at the temperature of -1960C to the time of 48 hours refines the grain size of aluminum-zinc-magnesium-copper alloy and nano particles formed up to 40 nanometer in microstructure that its result is the improvement in strength. Based on Figure 10 and Figure 11, new particles (made by the sub-zero treatment) are transfer precipitate of AlCuxMgySiz with the structure of base centered hexagonal and they are nano-size and sub-micron size (less than one micrometer). Therefore, yield strength increases more and tensile strength increases less and hardness had no significant change.



(b)Group2 (a)Group1 Figure 11: Image of STEM of nano AlCuxMgySiz particles (a)Group1 (b)Group2

Therefore, from the viewpoint of temperature changes, ascent and descend of airplane had no negative effect on tensile properties of airplane body so that it causes the improvement of tensile properties of airplane body, especially if the time of peaking will be long. General figures of parameters illustrated in Figure 12 for friendly users and comparisons of parameters. They are obtained from Table 2.

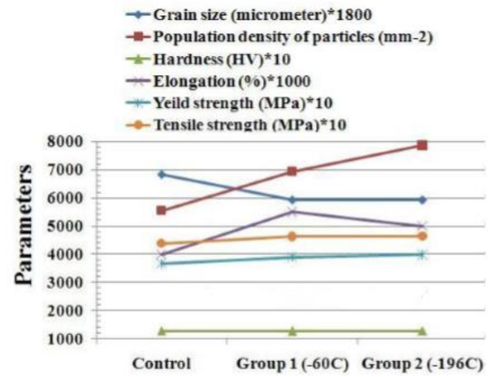


Figure 12: Parameters versus specimens obtained from Table 2

3. Conclusions

In this study, the effect of sub-zero treatment on tensile properties of aluminum alloy 2024 which has application in constructing the body of passenger airplanes, is compared and investigated at two temperatures -600C and -1960C. Results showed that by performing sub-zero treatment at the temperature -600C (group1) and -1960C (group2), the elongation, yield strength and tensile strength towards control sample were increased 1.5 and 1 percent, 24 MPa and 32 MPa and 25 MPa and 26 MPa, respectively. On the other hand, yield strength of the sample of group 1 to the sample of group 2 has increasing (8 MPa) while the elongation had 0.5 percent decreasing and tensile strength is nearly the same. Hence tensile properties towards control sample is increased but in the low cooling rate (≈1 degree per minute), more improvement is achieved in tensile properties.

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