A Novel Solution Heat Treatment of 7075-Type Alloy

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Abstract

The design of critical aerospace alloys is primarily built on optimizing strength and ductility, both of which can be enhanced by controlling the alloying element additions as well as heat treatment conditions. The 7075 alloy is one such aerospace alloy. The main objective of this study was to optimize the 7075 strength. Several experimental alloys were prepared and tensile test bars were cast using an ASTM B-108 type permanent metallic mold. The as-cast samples were then solution heattreated at 470°C for times up to 48 hrs. The solution heat-treated bars were also aged in order to improve the allov strength through precipitation hardening. Line scans for Mg, Cu and Zn were obtained from the various heat-treated alloy samples using an electron probe microanalyzer equipped with EDX and WDS facilities. Peaks corresponding to the Mg, Cu and Zn concentrations in the as-cast samples disappeared after solution treatment, reflecting optimized homogeneity structures. The newly developed versions of the 7075 alloy displayed UTS of ~1 GPa.

Introduction

Cast aluminum The design of aerospace aluminum alloys is mainly rooted in optimizing the strength and ductility, both of which can be enhanced by controlling the added alloying elements as well as the heat treatment conditions. The objective of this study was to optimize the strength of a 7075 (Al-Zn-Mg-Cu) alloy, whose tensile properties exceed 500 MPa, depending on the production procedures and heat treatment techniques applied. Moreover, these alloys have other favorable properties such as resistance to stress-corrosion cracking and fracture toughness. The 7075 alloy also demonstrates a high response to age hardening.¹⁻³ It was reported that the T73 treatment with aging at a higher temperature reduces tensile strength by 15%. Using a combination of T6 (120°C/24hs) and T73 tempers was recommended to resist stress corrosion cracking; also, increasing the Cu content compensates for the overaging effect in strength reduction.^{2,4} Thermomechanical treatments, i.e. combinations of aging and deformation, and RRA (retrogression aging) are other procedures used to optimize the 7000 alloy series tensile properties. The RRA sequence after solution heat treatment and quenching in cold water is: i) T6 aging, 120°C/24h, ii) short time heating, 200-250°C/5-10 min, followed by cold water quenching and iii) T6 re-aging, 120°C/24h.5,6

At room temperature, high strength age hardenable 7XXX alloys are difficult to process by plastic deformation processes. The 7075 alloy develops an ultrafine structure and higher strength when it has been processed cryorolling (CR) temperature compared to at room temperature rolling (RTR). The higher strain (more than 3.4) sample produced ultrafine grained in case of RTR, but the yield stress was still low due to the effective suppression of dynamic recovery and accumulation of higher dislocation density during low temperature rolling. In the case of CR samples, the hardness, yield strength and tensile strength were increased from 107 to 194, 155 to 559 and 368 to 602 MPa, respectively. It was concluded that cryorolling is an ideal process to deform the 7075 alloys up to a high rolling stain (3.4) and the RTR limits the deformability of these alloys due to deformation of GP zones.⁷

Many studies⁸⁻¹⁴ focused on the effect of heat treatment on 7075 alloys. The solutionizing and aging temperatures are the main factors controlling the relative alloying elements which precipitate in the grain boundaries; the solutionizing temperature is the main influence on grain boundary segregation. The precipitates on the grain boundaries consisted of large amounts of Mg, Si and Al along with a lesser amount of Zn and Cu. The Zn-rich precipitates were predominant in heat treatments during low solution annealing (394°C) while the Fe-rich precipitates were predominant in the case of high solution temperature (482-527°C).⁸ Precipitates with high interfacial energies tend to precipitate at grain boundaries, resulting in embrittlement. Low interfacial energy means easy nucleation, a uniform precipitate distribution and resistance to coarsening at elevated temperatures.⁹

The microstructure of commercial 7075 alloy in the peak-aged (T651) temper contains predominantly the η transition phase before formation of the stable n-MgZn₂ phase. Some of these transition phase particles are heterogeneously nucleated on dislocation lines. In the T7 microstructure, the overall particle concentration is high, consisting mainly of coarse transition phase particles η_1 , η_2 and η_4 beside small amount of fine particles of the last three phases. Increases in the hardness of the 7075 alloy is believed to arise mainly from of the fine dispersion of small ή particles.¹⁰ Retrogression aging treatments for 7075 alloy sheets at various temperatures (180, 200 and 220°C) and times (2-80 min) showed that an increase in treatment temperature decreased both the hardness and strength, while increasing the impact toughness. This can be explained by the dissolution of phases in the matrix during RRA and enlargement of segregation during subsequent aging. The decrease in the

hardness in the first stage of RRA is explained by the partial dissolution of the GP zones while the subsequent increase in hardness refers to the formation and growth of the η -phase to a specific size of stable η particles.¹¹

The microstructure of the grain boundary particles which depend on the aging process is the main parameter controlling the 7075 alloy mechanical properties. The high strength of this alloy in the RRA temper is considered to arise from both the presence of many fine $\hat{\eta}$ particles, which are probably coherent, and of the high overall concentration of particles in this structure.¹²

The double aging (DA) of 7075 alloy has positive effect on hardness, yield and ultimate tensile strengths. Moreover, double aging to peak hardness results in a significantly reduced processing time from 48 to 2 hours, which can lead to reduced energy and cost. Thermomechanical double aging causes further acceleration of precipitation, reducing the total heat treatment time to 80 minutes, with an increase in both hardness and strength, but a decrease in the ductility relative to single aging.¹³

During a study of secondary aging, it was concluded that the T614 temper produces tensile properties close to or greater than those for the T6 condition; the fracture toughness enhances as well. The T814 and T914 tempers are effective in utilizing secondary precipitation, while the T816 and T916 produce improved mechanical properties.¹⁴

The main objective of this study was adjusting the composition and heat treatment technique of the 7075-type alloys to obtain an UTS of \sim 1 GPa.

Experimental Procedures

The as-received Various 7075 alloy compositions were used in the current study. Samplings for chemical analysis were taken from each alloy. Table 1 shows the average chemical composition of both the base metal and the base alloy investigated.

| Alloy Code / | Si | Fe | Cu | Mn | Cr | Ni | Zn | Mg | Ti | Zr | Al |
|-------------------|-------|-------|---------|---------|---------|-------|-------|---------|-------|---------|-------|
| Alloying Elements | | | | | | | | _ | | | |
| Base Metal | 0.050 | 0.090 | < 0.001 | < 0.001 | < 0.001 | 0.006 | 0.002 | < 0.001 | 0.005 | < 0.001 | 99.83 |
| (Pure aluminum) | | | | | | | | | | | |
| A (Base Alloy) | 0.172 | 0.384 | 1.980 | 0.330 | 0.303 | 0.022 | 6.420 | 2.260 | 0.016 | 0.211 | Bal. |

Table 1. Average chemical composition (wt %) of the base metal and the base alloy

Measured Mg, Zn, Si, Cu, Fe and other additions were made to the melt. Alloying elements were added in the form of master alloys or pure metals to obtain the predetermined level/levels of each. Prior to casting, the molten metal was degassed for 15 min using pure, dry argon to remove the hydrogen and inclusions. Alloy castings were produced using an ASTM B108 permanent mold which had been preheated to 450°C. Tensile test bars, in standard specifications, were cut from the castings.

The test bars were prepared for each alloy composition and divided into different sets according to both the recommended traditional heat treatment and the proposed new heat treatment conditions. Six sets of the base alloy were conventionally heattreated, where all of them were solution heat-treated at 470°C/8h, then quenched in 65°C warm water. One set was kept in the solution heat-treated condition, while the other five sets were followed by different aging conditions: the first set had single aging at 120°C/24h, the second set had single aging at 280°C/8h, the third set had double aging at 120°C/24h, followed by 180°C/8h, the fourth set had retrogression and re-aging at 180°C/8h, followed by 120°C/24h, and, finally, the last set had retrogression and re-aging at 280°C/8h, followed by 120°C/24h, respectively. Two sets of the new alloy were heat-treated by means of homogenization and a proposed new aging process. For each heat treatment, ten test bars were used.

Tensile testing was carried out for the heat-treated test bars at room temperature using an MTS Servohydraulic mechanical testing machine working at a strain rate of 1.0×10^{-4} /s. The elongation of the test specimens was measured using a strain gauge extensioneter attached to the specimen during the tension test. A data acquisition system was attached to the MTS machine to provide the results of the tensile test. For each sample tested a stress-strain curve was used to illustrate the mechanical behavior of each specimen under the loads applied. The tensile test results obtained from testing a specific specimen present the data pertaining to elongation to fracture, yield strength at 0.2% offset strain and ultimate tensile strength. Ten tensile test bars for each composition were tested in the heat-treated conditions. For the new alloy, the test bars were stretched by the same MTS tensile testing machine.

From each of these samples prepared for metallographic characterization, two samples, one as-cast and one solution heat-treated, were sectioned off to study each alloy condition. The microstructures of the polished sample surfaces were examined using an optical microscope linked to a Clemex image analysis system and were examined using the JEOL 840A scanning electron microscope equipped with an energy dispersive x-ray system (EDS). Line scans for Mg, Zn and Cu were obtained from the various heat-treated alloy samples using an electron probe microanalyzer equipped with EDX and WDS facilities.

Results and Discussion

Several authors have studied the effects of alloying elements in addition to heat treatments.¹⁵⁻¹⁹ The Ag-free T6-tempered (121°C/24h) 7075 alloys exhibit low strengths that are attributed to a relatively coarse dispersion of the hardening precipitates while the Ag-containing alloys developed higher strengths. When Cr and Mn are added to the Ag-containing alloys, they resulted in low strengths as with the Ag-free alloys, due to quench sensitivity of particles containing either Cr or Mn. On the other hand, adding the last two elements to Ag-free alloys resulted in higher strengths. Adding 0.3 to 0.4% Ag did not increase the strength of single and double-aging tempered alloys in both T6 and T7 treatments (121, 157, 162 and 177°C), whereas the SCC resistance was improved. Alloys containing Cr, particularly those also containing Zr and Mn, were more

resistant and increased in strength, as a result of the reduced sensitivity to quenching rate. Higher strengths were obtained by increasing Cu content in alloys containing Mn and Zr in the case of double aging treatments. Zinc levels in Cr-containing alloys increased both the stress-corrosion resistance and quench sensitivity with little strength advantage.^{15,16}

The direct chill (DC) cast 7XXX series, modified with Zr and Sc, led to the following conclusions: i) Sc additions produced an equiaxed grain morphology, grain-refined microstructure, where 0.18%Sc reduced the grain size to 120 μ m, and inhibited the formation of twinned columnar grains and solidification cracks, ii) higher Sc levels such as 0.38% and 0.48% formed the brittle primary Al₃(Sc, Zr) phase, consequently leading to a deterioration of mechanical properties, iii) homogenization and T6 treatments developed both the strength and ductility of alloys containing Sc, where the yield strength obtained was 490-590

MPa along with 15% elongation at room temperature, but at the liquid nitrogen temperature (-196°C to -210°C), this was 610 MPa and 10%, respectively, with a UTS of 720 MPa.¹⁷ The Sc-modified 7075 alloy presented the highest strength (640 MPa) and significantly increased the SCC resistance. Adding 0.2% Sc resulted in the Al₃(Sc_{1-x}Zr_x) phase which refined the microstructure. The addition of Ce has little strengthening effect.¹⁸ The presence of 2% of Al-5Ti-1B master alloy to Al-12Zn-3Mg-2.5Cu alloy reduced its grain size from 480µm to 40 µm; the hardness of both Ti-refined and T6-tempered was significantly increased.¹⁹

In the present work, the effects of alloying elements and heat treatment conditions applied are explained and confirmed by the tensile properties values summarized in Table 2.

| Table 2. | Tensile | properties | values t | for allo | vs/conditions | s studied |
|----------|---------|------------|----------|----------|---------------|-----------|
| | | P | | | | |

| Alloy Code and Condition / Tensile Properties | UTS (MPa) | El (%) |
|---|-----------|--------|
| A / Solution heat treatment (8h @ 470°C) | 429 | 4.63 |
| A / Single aging (24h @ 120°C) | 580 | 1.01 |
| A / Single aging (8h @ 280°C) | 386 | 4.16 |
| A / Double aging (24h @ 120°C + 8h @ 180°C) | 525 | 0.92 |
| A / Retrogression and re-aging (8h @ 180°C + 24h @ 120°C) | 496 | 1.02 |
| A / Retrogression and re-aging (8h @ 280°C + 24h @ 120°C) | 312 | 3.5 |
| New Alloy / Homogenization and stretching | 597 | 3.62 |
| New Alloy / Proposed new aging process | 980 | 1.13 |

It is seen that the maximum UTS attained was 580 MPa for single aging samples of the base alloy at $120^{\circ}C/24h$, which is normally higher than the solution heat-treated alloy (429 MPa), while the minimum UTS reached was 312 MPa for retrogression and re-aging samples at 280°C/8h, followed by $120^{\circ}C/24h$ as a result of over-aging effect, dissolution of phases in the matrix during retrogression treatment and segregation of some particles at the grain boundaries. The best aging condition was, as recommended, single aging at $120^{\circ}C/24h$. Compared to the famous 7075 alloy, the base alloy in current study, the new alloy after homogenization and stretching resulted in a UTS of 597 MPa (~600 MPa) while, after the proposed aging process, the UTS was 980 MPa (~1 GPa) which renders this alloy, in the proposed heat treatment technique, as a super strong alloy. The comparison between the two alloys (i.e. base and new), are

shown and confirmed by the stress-strain diagrams in Figure 1, for both the solution heat-treated and aging conditions.

Figure 2 shows the base alloy as-cast equiaxed microstructure (a, b) and the solution heat-treated one (c, d). In (b), the Al₂Cu, Mg₂Si and α -Fe phases were all dissolved, except for the Fe-rich phases, as confirmed in the high magnified (d). The homogenized microstructure is shown in (c).

Figure 3 shows a backscattered image of the base alloy as-cast microstructure in (a) and the high magnification image shown in (b), where the AlFeCuZn, AlFeSi and AlZnCuMg phases were detected. Figures (c, d and e) are the EDS spectra corresponding to the previous three phases detected in the as-cast structure.



Figure 1. Stress-strain diagrams for test bars of: (a) solution heat-treated base alloy, A, (b) new alloy after homogenization, (c) base alloy after single aging and (d) new alloy after aging.



Figure 2. Optical micrographs of the base alloy: (a) as-cast microstructure, (b) high magnification of (a), (c) solution heat-treated microstructure and (d) high magnification of (c).



Figure 3. (a) Backscattered image of the as-cast microstructure of the base alloy, (b) high magnification of (a), (c), (d) and (e) are the EDS spectra corresponding to the AlFeCuZn, AlFeSi and AlZnCuMg phases shown, respectively, in (b).

Figure 4 shows a backscattered image of the base alloy solution heat-treated microstructure in (a) and the high magnification image shown in (b), where the α -Fe and AlFeCu insoluble phases were observed. Figure (c) is the EDS spectrum corresponding to the last AlFeCu phase detected in the homogenized structure. Table 3 summarizes the identifications of the observed phases in Figures 3 and 4, which in good agreement with those reported in the literature.

For all microstructures studied, line scans were used to investigate the distribution of alloying elements before and after solution heat treatment for both the base and new alloys. Figure 5 shows the distribution of three common elements Mg, Cu and Zn in the 7075 alloys. The peaks in Figure 5(a, b and c), corresponding to the Mg, Cu, and Zn concentrations in the ascast samples, disappear after solution treatment, reflecting the optimized homogeneity of the solution-treated alloys.



Figure 4. (a) Backscattered image of heat-treated microstructure of base alloy, (b) presences of AlFeCu and α -Fe intermetallics in (a), (c) high magnification image of (b) and (d)EDS spectrum corresponding to AlFeCu phase presented in (b).

| Figure No. | | | - | Approximate Composition | | | | | |
|------------|-------|--------|--------|-------------------------|--------|--------|-------|-------|---|
| | Si | Al | Fe | Cr | Cu | Mg | Mn | Zn | |
| 3(e) | 0.140 | 26.827 | 0.168 | 0.009 | 21.078 | 34.271 | 0.040 | 17.42 | $T(Al_3Cu_2Mg_4Zn_2)$ |
| 4(b) | 0.617 | 75.332 | 16.755 | 0.968 | 2.311 | 0.049 | 3.310 | 0.554 | Al ₃ (Fe,Cu,Mn,Cr) |
| 4(b) | 3.683 | 77.508 | 11.006 | 2.485 | 0.717 | 0.079 | 3.918 | 0.578 | Al ₈ (Fe,Mn,Cr) ₂ Si/Al ₁₅ (Fe,Mn,Cr) ₃ Si ₂ |

Table3 Identification of phases observed in various figures



Figure 5. (a), (b) and (c) are the elements distribution of Mg, Cu and Zn, respectively, in both the as-cast and heat-treated cases applying line scan techniques.

Conclusions

- 1. Conventional and/or commercial heat treatment of 7075 alloy led to a maximum UTS of 580 MPa.
- 2. The use of proper additives, cold/hot deformation, homogenization and aging, as applied in this work, shows that 7075 alloys have the potential to reach UTS levels as high as 980 MPa, after aging.
- 3. Alloy ductility could be significantly improved using proper casting technology. From this study, it is expected that the modification of both Alloy compositions and casting techniques will achieve better percentage elongation values.

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