Effect of Heat Treatment Parameters on Distribution and Volume Fraction of Mg₂Si in the Structural Al 6063 Alloy

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Abstract: Aluminum Profiles –Production by extrusion from aluminum billets require a certain degree of homogeneity because of the segregated second phase particles, which are available in the as cast structure of the billets. Worldwide practice suggests a long homogenizing process at elevated temperatures in order to achieve such condition. This study showed that profiles extruded from homogenized and unhomogenized billets did have the same mechanical properties and even better metallurgical features, for the interest of unhomogenized one, such as second phase distribution and amount of second phase dissolved in solution. As a result, the whole homogenizing process could be eliminated without sacrificing any of mechanical properties

Key words: Homogenizing process, volume fraction, heat treatment parameters, aluminum billets

INTRODUCTION

Aluminum Alloys of the 6xxx (Al-Mg-Si system) series are the most widely used for the production of extruded sections. More than 80 percent of the aluminum alloys employed worldwide in the manufacturing of extruded sections belong to the 6xxx series^[1]. In these alloys, Mg and Si combine to form the chemical compound Mg₂Si (magnesium-silicide), the primary hardening phase^[2]. The strength of the alloy is dependent on the role of magnesium-silicide precipitates^[3-5]. Meanwhile, extrudability is strongly influenced by the amount of Mg and Si in solid solution and the size and distribution of Mg₂Si precipitate particles^[6] Since the grain boundaries have regions that are relative open, hence ,some segregation is expected (both on micro and macro scale) of the undissolved particles since at these regions the strain energy is minimum^[7]. Most of the added alloying elements to aluminum alloys are less soluble in the solid phase than in liquid, then a gap between the liquidus and solidus will exist leading to the segregation of these alloying elements. This segregation is reduced by heating the ingots for an extended period close to the solidus temperature to homogenize the structure^[8]. As the size of the cast product increases the chemical composition throughout the thickness will considerably vary, (macrosegregation). This type of segregation is not much affected by heating but by controlling the manufacturing parameters of the ingot, for example in the Direct-Chill casting process where decreasing the ingot thickness, lowering the casting speed and maximizing the superheating temperature will greatly reduce this sort of segregation.

The strength and hardness of some metallic alloys may be enhanced by the formation of extremely small uniformly dispersed particles of a second phase within the matrix. This must be accomplished by appropriate heat treatments. The general requirement for precipitation strengthening of supersaturated solid solutions involves the formation of finely dispersed precipitates during aging heat treatments. For more complex ternary and quaternary systems, solution treatments are modified according to the effect of new elements on the solid solubility and/or the eutectic melting points of the basic binary system. The aluminum - magnesium - silicon alloys would be soaked at a temperature in excess to 500°C but below the solidus of 595 °C to avoid incipient melting, however, because some alloy constituents may form complex eutectics that melt at temperatures below equilibrium eutectic temperature, the upper limit for solution treatment of aluminum-magnesium-silicon alloys is in the range of 515 to 540 °C. At 540 °C, about 0.6% Mg can be retained in solution^[8,9], hence under this condition, these alloys respond strongly to precipitation heat treatment. This practice is widely used in producing thin extruded shapes of alloys 6061, 6063, 6463 and 7005. Upon precipitation heat-treating after quenching at the extrusion press, these alloys develop strengths nearly equal to those obtained by adding a separate solution heat-treating operation^[8,9].

The micro segregation of silicon and magnesium is not severe in the aluminum-silicon-magnesium casting alloys and hence it takes only a short time to homogenize the alloy and to put the Mg_2Si into solution^[8,9]. Hot working also helps in breaking up the coarse as-cast structure of the ingot.

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Large cast products need more prolonged treatment than wrought material. In the latter, hot working is more effective in producing a homogeneous material^[10]. Manufacturing process carried at the industry, from which the billets are taken, starts with an as cast billet of AA 6063 produced by horizontal Direct-Chill (DC) casting system. The billets are then stacked and inserted to the homogenizing furnace to be homogenized at 580 °C for 10 hours (4 hours heating and 6 hours soaking time). Then these billets are cooled by forced air using large electrical fans to produce supersaturated solution. After that; they are transferred to the induction-heating furnace to be heated up to 520 °C. The hot billets are then inserted into the container of the extrusion unit where it will be extruded to the required shape. The extruded profiles are then stacked properly for the final step of aging where they will be inserted into an oven and aged artificially aged at 200°C for 8 hours. The resulting profiles will have optimum mechanical properties accepted commercially.

Experimental: Aluminum 6063 alloy billet ~175mm in diameter at two conditions namely, those were directly taken from the DC casting mold and the industrially homogenized one. The chemical composition of the alloy, in wt%, is 0.412Si, 0.182Fe, 0.0030Cu, 0.015Mn, 0.468Mg, 0.011Zn, 0.0008Pb, 0.017Ti, 0.0023 Cr and balance Al. Aluminum 6063 alloy extruded profiles, at two conditions; as extruded (from both homogenized and unhomogenized billets) and artificially aged was used. Metallographic etchant (M10) especially designed for 6063 alloy^[11] was used to reveal the Mg₂Si precipitates. The billets were cut into 2X 2X 20 cm strips. These strips, at different conditions, were machined to the required dimensions for the tensile test according to the ASTM (E8) standard. Three temperatures were suggested to work at, namely 580 °C (analogous to that originally used by the industry), 550 °C and 520 °C. Different soaking periods were adopted at each temperature, namely, 2, 3, 4, 5 and 6 h. Muffle furnace was used to homogenize the strips as well as the meallographic specimens. 10 strips were used for each run. 15 min was given as an allowance time to reach the homogenizing temperature. Vickers microhardness measurements for the treated samples (as cast and extruded) were taken. Microstructure of samples, before or after extrusion, was examined by optical microscope. According to the ASTM standard E562, point-counting method was used to determine the average volume fraction of the second phase, in the etched specimens, at various conditions.

RESULTS AND DISCUSSION

Figures (1-3) show the Stress-Strain relationships of three groups of aluminum 6063 samples solution at different temperatures (520, 550 and 580°C) and for various holding times i.e., 2, 3, 4, 5 and 6 h



Fig. 1:Stress - Strain diagram of Aluminum strips homogenized at 520 °C for 2, 3, 4, 5 and 6 hrs



Fig. 2: Stress - Strain diagram of Aluminum strips homogenized at 550 °C for 2, 3, 4, 5 and 6 hrs



Fig. 3:Stress - Strain diagram of Aluminum strips homogenized at 580 °C for 2, 3, 4, 5 and 6 hrs

respectively. When Mg₂Si particles dissolve in the matrix the tensile and yield stress values will be effected after cooling. Since these dissolved particles upon cooling will form strain fields acting as obstacles for dislocation motion. But it was clear that either holding time or holding temperature had small effect on the mechanical properties and that generally higher temperature gave higher tensile strength; which means that homogenizing at any mentioned temperature and for any holding time will create a stronger billet compared to ~150 MPa tensile strength of the as-cast billet. Such increase in strength is, of course, not favored for the extrusion purposes,

Figure 4 shows the Stress-Strain curves for extruded profiles. These profiles were taken from a fully homogenized billet at 580 °C for 6h and from ascast (unhomogenized) billet at two conditions (aged and un -aged). After aging at 200 °C for 8 h, fine



Fig. 4: Stress-strain curves of extruded aluminum (6063) profiles from Homogenized billet at 580oC for 6 hours and forms as-cast (Unhomogenized) billet. Aged at 200°C for 8 hours



Fig. 5: Vickers micro-hardness of Extruded (Aged and Un-Aged) profiles from fully homogenized and As-Cast billets



Fig. 6 Vickers micro-hardness versus holding time of aluminum strips soaked at 520, 550 and 580 °C at 2, 3, 4,5 and 6 hour soaking



Fig. 7: Volume fraction of the second phase (Mg2Si)Vs. soaking time of aluminum strips, soaked at 528, 558 and 588°C for 2, 3, 4, 5 and 6 hrs

precipitates of second phase particles (Mg₂Si) formed and gave rise to increase the tensile properties of the alloy. It is seen here that the values of tensile and yield stresses of both profiles, from fully homogenized and from as-cast billets are very close to each other, in both cases, before and after aging. This means that hot extrusion has produced homogenous structures with homogeneous properties regardless the condition of the starting billet. This is also clear in the average Vickers's microhardness values, Fig. 5, for extruded profiles, at thie same conditions mentioned in Fig. 4.

Figure 6 shows the variation of average Vickers's microhardness value for solution treated samples with soaking time at various treating temperatures (520,550 and 580 °C). Hardness is also affected by the amount of dissolved second phase particles and consequently, as time and/or temperature increases the hardness is expected to increase as well, but as is seen in this figure; holding time from 2 to 5 h at all temperatures didn't almost affect the hardness. This is because at these high temperatures and due to complex eutectics formed; a local melting occurred and so the treatment involved continuous melting and solidification that is why hardness didn't change. Also increasing the temperature from 520 to 550 °C didn't affect hardness for the same reason. At 580 °C more particles were dissolved and upon cooling, although part of them remained in solution, resulting in some increase in hardness compared to 520 or 550 °C treatment temperatures. Continuous melting and hence solidification through 520 and 550 °C treatments helped to homogenize the structure. Hence, when enough homogeneity was reached, energy was consumed in dissolving more solute atoms rather than homogenization, this caused the sudden increase in hardness is noticed at 6h Fig. 6. However high hardness is not favored for extrusion process as is known.

Figure 7 shows the volume fraction variation of the precipitated particles in the solution treated sample at three treating temperatures (520, 550 & 580 °C) for different soaking time (2, 3, 4, 5 & 6 h). It is seen that no clear trend can be drawn from 550 & 580°C curves, continuous melting and solidification phenomenon of the second phase particles is also evident here; that is why no specific trend can be seen. The 520 °C treatment showed a decreasing trend, which means that dissolution of the second phase was a little bit more effective but still within a small range. This can be considered as an evident of less incipient melting has occurred at this temperature.

While Figure 8 shows the volume fraction of the second phase in extruded profiles (un-aged and aged) from both fully homogenized at 580 °C for 6 h and ascast billets. Homogenization resulted in more dissolved amount of particles in the extruded un-aged profiles compared to those extruded from as-cast billet, but after aging at 200 °C for 8 h the volume fraction dropped markedly for the profiles extruded from as-cast billet



Fig. 8: Volume fraction of the second phase (Mg2Si) for extruded profiles from aluminum billet homogenized at 580°C for 6 h and from As-Cast (Unhomogenized) billet axed at 288°C







Mg,Si precipitates in aluminum 6063 profile Extruded from a fully homogenized billet at 580oC for 6 hour soaking at that temperature; no aging was performed to the profile 200x

(c)

6063 profile Extruded from a fully homogenized billet at 580oC for 6 hour soaking at that temperature; the profile was then aged at 200oC for 8 hours.200x.

- (d)
- Fig. 9: Photomicrographs of Extruded profiles from As-Cast & Fully homogenized billets (in both un-aged & aged conditions).

compared to those extruded from fully homogenized billet. This may due to the lattice defects produced during hot extrusion process, the extrusion also helped (with its high plastic deformation and temperature) in dissolving more particles. This means that hot extrusion helped breaking down the coarse structure of the as cast to produce a more homogenous structure especially after aging (Fig. 9). However, the hardening effects due to the aging (Fig. 5) may be attributed to the very fine particles, which are more effective, unresolved by optical microscope.



Fig. 10: Photomicrographs of As-Cast & treated Aluminum strips.

Figure 10 shows the second phase precipitates (Mg₂Si) in the as-cast condition & that fully homogenized at 580 °C for 6 h (a &b), it is seen here that there is some difference in the volume fraction due to the effect at high treating temperature. Plate (c) shows the structure of homogenized strips at 520 °C for only 2 h soaking time compared to the structure i of homogenized sample at 580 °C for 2h as soaking time (plated d). At 520 °C temperature, it is seen that finer distribution was achieved and better particle compared to severe segregation for the 580 °C treatment due to incipient melting. This means that the 580 °C treatments deteriorated the structure^[12] and eventually during long soaking time (up to 6 h) the structure brought back into almost similar to the starting (ascast) structure.

It could be conclude that more uniform distribution of the Mg₂Si particles with more or less regular size was achieved in the profiles extruded from as-cast billets before and after aging compared to poor distribution and irregular shape and size of the particles with some particles depleted areas in the fully homogenized sample (580 °C for 6h). This means that extruded profiles from as-cast billets will have better microstructure than corresponding profiles extruded from homogenized billets before and after aging. Alternatively homogenization at lower the temperature and for shorter time i.e. 520 °C for 2 h could be adopted since it gives betters microstructure than that homogenized at 580°C for the same period (2h).

CONCLUSION

Profiles extruded from homogenized billets and from as-cast billets were of about the same hardness and strength. Homogeneity and volume fraction of the second phase particles was even better for those extruded from as-cast billets, this implies that the elimination of the homogenizing process will not affect the mechanical properties of the final product and it might be even better. Thermal treatment only, 6063 billets was not enough for aluminum to achieve acceptable homogeneity while thermo mechanical treatment was more efficient (hot extrusion). If homogenizing is required then it should be done at temperatures not exceeding 520 °C and with a soaking time not more than 5 h and hence 2 h is recommended. As a consequence, it is recommended to have a strong water-spray or forced cold air stream to quench the profiles as they emerge from the extruding die orifice to get higher strength products after final aging process.

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