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Effects of rapid heating on aging characteristics of T6 tempered Al–Si–Mg alloys using a fluidized bed

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Abstract Effects of rapid heat transfer using a fluidized bed on the heat-treating response of Al–Si–Mg alloys (both unmodified and Sr modified) were investigated. Heating rate in fluidized bed (FB) is an order of magnitude greater than in conventional air convective furnaces (CF). On aging using FB, it was observed that the nucleation rate of Mg_2Si particles was greater than in CF. Thermal analyses show an endothermic reaction during aging in CF. No such transformation was observed during aging in FB. The endothermic transformation could be due to the dissolution of GP zones or metastable phase(s). The total heat treatment time for T6 temper was reduced to less than 2 h using FB.

Introduction

Increasing demands for lightweight metals as structural components in the automotive and aerospace industries have led to the extensive use of Al–Si–Mg based foundry alloys. One advantage of using lightweight materials is to increase the payload capacity. Al–Si–Mg alloys are candidate materials for such applications due to their good castability characteristics [1]. The addition of Mg makes the alloy heat treatable [1–6], which allows one to tailor mechanical properties by selecting a suitable temper. The T6 temper is a widely accepted heat treatment process to increase strength in aluminum alloys. The T6 temper is comprised of solution heat treatment, quenching and then artificially aging. The solution heat treatment increases ultimate tensile strength and ductility, while aging increases yield strength at the expense of ductility. In the case of the T4 temper (solution heat treatment followed by quenching), the increase in strength is through the solute solution strengthening mechanism—Mg and Si atoms retained in the post quenched state, and the increase in ductility is due to the spherodization of eutectic Si particles. While the increase in yield strength on aging after T4 temper is through precipitation strengthening of Al matrix by Mg_2Si particles. The resultant mechanical properties of the heat-treated alloy depend on its chemical composition.

In Al–Si–Mg alloys, it has been widely reported that Mg and Si go into solution during solution heat treatment and are retained in the Al matrix on quenching [1–2, 6, 10–12]. On subsequent aging, Mg and Si in the Al matrix combine to form Mg_2Si precipitates through various stages [4–5]:

$$\text{Solute Cluster} \rightarrow \text{Guinner Preston Zone} \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta \text{ precipitate } (Mg_2Si)$$

Various factors that affect the nucleation and coarsening rate of Mg_2Si particles are alloy composition [6], incubation period between solution heat treatment and aging, temperature, isothermal time, and heating rate. It has been reported [6] that the number density of Mg_2Si particles in Sr modified alloy is greater than that of unmodified alloy. The Sr retards the growth of Mg_2Si particles and consequently results in finer particles as compared to that in an unmodified alloy. The growth is retarded due to the decrease in diffusivity of Si in the Al matrix of Sr modified

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alloy [6]. Heat treatment of Al–Si–Mg alloys has been studied extensively [1–6]. The nucleation and coarsening rate of Mg_2Si particles during aging increases with an increase in aging temperature [10] because diffusivity increases with temperature. However, the effect of rapid heating on aging characteristics has not been closely examined. Heating rate plays a crucial role in increasing the kinetics of metallurgical processes; the effect of rapid heating through a fluidized bed heat treatment system (henceforth it will be referred to as FB) on aging characteristics of Al–Si–Mg (D357) alloy is the focus of this study. The effects of solution heat treatment using FB on the microstructure and tensile properties are given in [12].

Alloy D357 is widely used in aerospace applications; in conventional practice, the alloy is heat treated for about 20 h using a conventional circulating air convective heat-treating furnace (henceforth referred to as CF). There is a major thrust to reduce the total heat treatment time and thereby increase productivity. With the advent of FB, the net heat treatment time can be reduced by an order of magnitude.

The effect of FB processing i.e. aging after solution heat treatment, on the resultant microstructure and mechanical properties of unmodified and Sr modified D357 alloys were investigated. Tensile properties are correlated to microstructural changes, primarily to the nucleation rate of Mg_2Si particles during the aging treatment. For comparative purposes, alloys were also heat-treated using CF, and the resultant microstructure and tensile properties were examined.

Experimental procedure

Alloy composition

Alloy D357 was procured from Hitchcock Industries, MN, USA in the form of cast plates. The chemical composition of the alloy is given in Table 1. The chemical composition was measured using spectrographic technique and is given in weight percentage. In addition to the basic alloying elements (Si, and Mg), the alloy also contains Fe as an impurity element. It is well know that the presence of Fe as an impurity element in most Al–Si–Mg based foundry alloys has a deleterious effect on ductility [8]. Be is added to counter this by spherodizing the Fe rich intermetallics [13]. The alloy also contains Ti as an alloying element, and its intermetallic (Al_3Ti) is a well-known grain refiner [9]. The heat treatment characteristics of both unmodified and Sr modified D357 alloys were studied.

Table 1 Chemical composition of alloys

Alloy	Si	Mg	Fe	Sr	Ti	Be	Al
D357	6.9	0.56	0.06	–	0.18	0.05	Balance
D357(Sr)	7.1	0.56	0.07	0.015	0.18	0.05	Balance

Heat treatment

Cast bars of alloy D357 were solution heat treated, quenched in water, and aged. The size of cast bars was 15 mm × 15 mm × 100 mm. A laboratory type fluidized bed [7] was used for solution heat treating and aging. Staurolite sand ($FeAl_5Si_2O_{12}OH$) was used as the fluidized bed material; particle size was in the range of 80–120 μm. The sand particles were heated to the desired temperature by means of a series of heating elements placed beneath the bed. The temperature variation in the bed was found to be within 1–2 °C of the set temperature. For purposes of comparison, parts were also heat treated in the CF. Cast bars were solution heat-treated and aged using FB and CF at different temperatures and for various time intervals. The solution heat treatment was carried out at 543 °C using FB. Through thermal analysis during solidification of the alloy, we determined that the solidus temperature was 546 °C. The solution heat treatment temperature chosen for this study was 3°C below the solidus temperature to avoid incipient melting. The heat treatment schedule for aging after solution heat treatment is shown in Table 2. Prior to aging, cast bars were solution heat treated to optimum condition of 30, and 60 min, respectively for Sr modified and unmodified D357 alloys and quenched in water at 25 °C. These times were selected based on our previous study [12], where we reported that the above-mentioned solution heat treatment times were optimum for these respective alloys. The time delay during quenching was less than 10 s. Aging was carried out at two different temperatures 160 °C, and 190 °C. The incubation period between the solution heat treatment and aging was 48 h. It has been reported [11] that after quenching, natural aging begins immediately with the formation of clusters in A356 alloy, which reduces the strength though elongation improves. Artificial aging at 170 °C subsequent to natural

Table 2 Heat treatment schedule for D357 alloy T6 treatment*

Processing	Aging Time (minutes)				
FB	30	60	90	120	
CF		60		120	360

*Note: Cast plates were solution heat treated for 60 min (unmodified alloy), and 30 min (Sr modified alloy) at 543 °C followed by quenching in water at room temperature prior to aging. Times given are the isothermal holding times. Aging was carried out at 160°C and 190 °C.

aging for 12 h decreases tensile strength [11]. After 20 h of natural aging, there is a general recovery of the tensile strength. However, artificial aging at 185 °C reduces both detrimental effects of natural aging, and the time necessary to recover the lost strength during natural aging. Extended natural aging of A356 alloy for more than 20 h showed no significant influence on the precipitation rate of Mg_2Si particles.

Thermal analysis

Phase transformations of alloy D357 that occur during aging were studied using thermal analysis (TA) methods. The TA work was carried out by analyzing the first derivative of the temperature–time profile monitored during aging. A phase or physical transformation is accompanied by a release (exothermic) or absorption (endothermic) of thermal energy, which is detected by superimposing the first derivative (dT/dt) curve on the heating profile (i.e. Temperature (T) versus Time (t)). An exothermic curve will result in a sudden increase of dT/dt value, whereas an endothermic event results in a sudden decrease of dT/dt value at the point of transformation. Temperature measurements were carried out using DASY Lab view software coupled with a data acquisition system at an acquisition rate of 10 data per second.

Microstructural observations

Microstructural characterization of the as-cast and heat-treated alloy were carried out using scanning electron microscopy (SEM). Standard metallographic techniques were used for sample preparation. Samples for SEM were prepared by electro-polishing after grinding on the samples with SiC paper (4000 grit size). The electro-polishing was performed at 30 V for 20 s using a freshly prepared electrolyte, whose composition was 60% Ethyl alcohol, 20% Perchloric acid and 20% Ethylene glycol by volume. Electro-polished samples were then etched with concentric nitric acid for 5 s and immediately rinsed in running water. Nitric acid etchant is used to observe Mg_2Si precipitates in aged samples under SEM.

Evaluation of mechanical properties

The ultimate tensile strength, yield strength, and elongation of as-cast and heat-treated test bars were measured. Tensile specimens were machined from the as-cast and heat-treated alloys. Tensile bars were machined as per ASTM standard specification #B557 with 1 inch gauge length. Tensile properties were measured at a 0.1 inch/min extension rate. Merlin software was used for data acquisition and control during testing. At least five tests were conducted for each heat treat condition and the average value is reported. During tensile tests, a few samples fractured prematurely from the neck region due to the presence of macro defects—inclusions or porosity that resulted in poor data; these samples were excluded from the data.

Results and discussion

Microstructure

Prior to aging, the unmodified and Sr modified cast bars were solution heat treated at 543°C for 60 min and 30 min, respectively using FB, and subsequently aged at 160 °C and 190 °C in both FB and CF. Solution treatment times were selected based on results reported in an extensive study on solution heat treatment of Al based cast alloys using FB [11]. The effect of aging at 190 °C and 160 °C using either FB or CF on precipitation of the Mg_2Si phase can be seen in Figs. 1a–c, and 2a–b, respectively; the isothermal holding time was 60 min. The Mg_2Si particles were detected by EDX analysis; apart from the presence of Mg and Si in the ratio of 2:1, the EDX spectrum also consists of Al peak, which was picked up from the background matrix. Analyses of the aged microstructures reveal uniformly distributed spherical Mg_2Si particles in the matrix. The uniform distribution of Mg_2Si particles in the Al matrix is evidence that through solution heat treatment at 543 °C, the microsegregation of Si and Mg were significantly reduced in FB when solution heat-treated for 60 min (for unmodified alloy) and 30 min (for Sr modified alloy), respectively.

The nucleation rate of Mg_2Si particles in the Al matrix during aging is greater in the Sr modified alloy compared to the unmodified alloy. The size of Mg_2Si particles increases with increasing aging time and temperature; this is because coarsening rate increases with an increase in aging temperatures; Both spherical and needle shaped Mg_2Si particles are observed on aging using the FB at 160 °C and 190 °C for one hour. After an hour of aging treatment in the FB at 190 °C, the size range of spherical shaped particles are 20–60 nm for the Sr modified alloy and 40–90 nm for the unmodified alloy. The size of Mg_2Si particles is smaller in Sr modified alloy as compared to the unmodified alloy because of the low coarsening rate in the modified alloy; Sr reduces the diffusivity of Si in the matrix [6]. In general, after one hour of aging at 190 °C, the number and size of needle shaped Mg_2Si particles in Sr modified alloy are greater than those in the unmodified alloy. A large number of needles indicate that Ostwald ripening is the favorable mode for coarsening in the Sr modified alloy. In contrast, a high number of spherical Mg_2Si particles as observed in the unmodified alloy indicating that coalescence is the favorable mode of

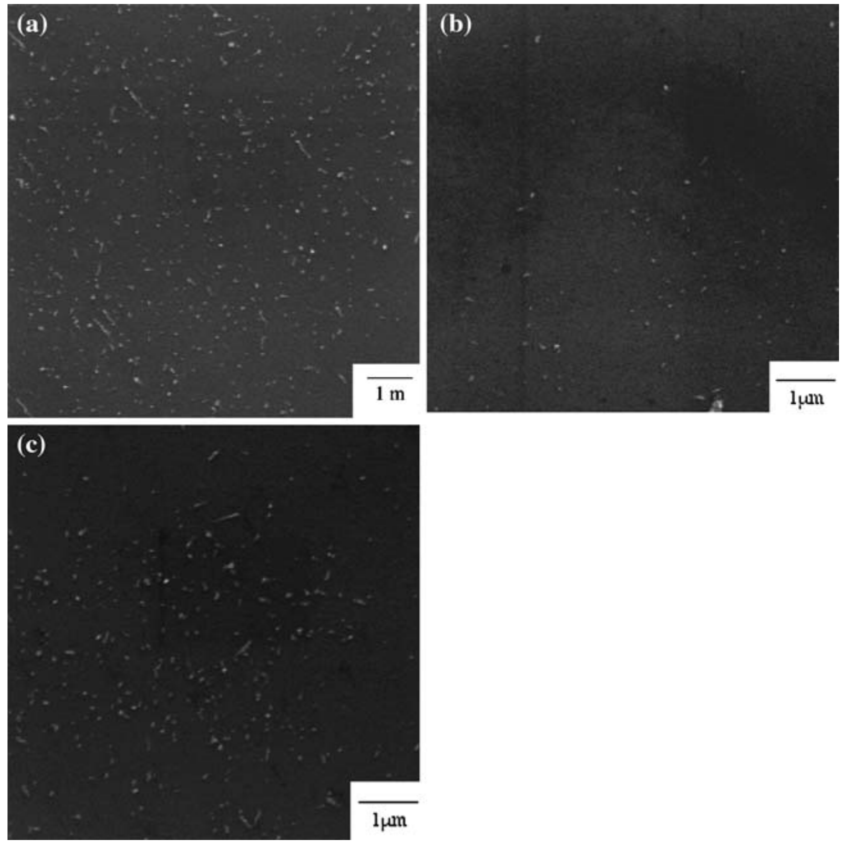

Fig. 1 Micrographs of aged D357 alloy at 190 °C for 60 min: (**a**) aged in FB-modified alloy, (**b**) aged in CF-modified alloy, and (**c**) aged in FB-unmodified alloy. These micrographs were taken from the center of the dendritic region

coarsening. In sum, coarsening in Sr modified alloys is dominated via Ostwald ripening; whereas in unmodified alloys coarsening mainly occurs through the coalescence of small spherical particles.

In general, the nucleation rate of Mg_2Si is greater while aging using the FB as compared to the CF. Within one hour of aging at 160 °C using the FB, Mg_2Si particles precipitate (Fig. 2a) in the Al matrix. In contrast, no Mg_2Si particles are observed in samples aged using the CF for one hour (Fig. 2b).

From these observations, it is apparent that the heating rate plays a dominant role in the microstructural evolution during FB heat treatment. The effect of ramp-up time or heating rate on phase transformation during heat treatment was studied through thermal analyses.

Thermal analysis

Figures 3a, and b show the heating profiles and their first derivative for D357 alloy, during aging using CF, and FB, respectively. Before the aging treatment, alloys were solution heat treated at 534 °C for 6 h and 30 min using CF, and FB, respectively followed by quenching in water. The heating profile shows that it takes about 60 min to attain the aging temperature in CF. In contrast, in FB, it takes about 6 min when the bed is in a fluidizing state. Aging in CF results in an endothermic transformation. The transformation in unmodified alloy starts at 80 °C and ends at 120 °C. The presence of Sr has negligible effect on the start and end temperatures of the endothermic reaction. However, no such transformations are observed (Fig 3b) during aging in FB. It may be noted that precipitation is an exothermic transformation. The endothermic transformation is most likely due to the dissolution of GP zones, clusters or some metastable phase(s) such as β'' or β'. Since aging in FB has resulted in a greater precipitation rate as compared to those in CF, it can be deduced that the endothermic transformation prior to precipitation using CF is the major cause for its relatively slow aging kinetics. However, further in situ work is needed to validate the thermal analysis results. The rapid aging kinetics in D357 alloys (both unmodified and Sr modified) heat treated using FB has a significant effect on the resultant mechanical properties.

Mechanical properties

Figures 4a–c and 5a–c show the variation of ultimate tensile strength (UTS), yield strength (YS), and elongation, respectively with aging time and temperature of unmodified,

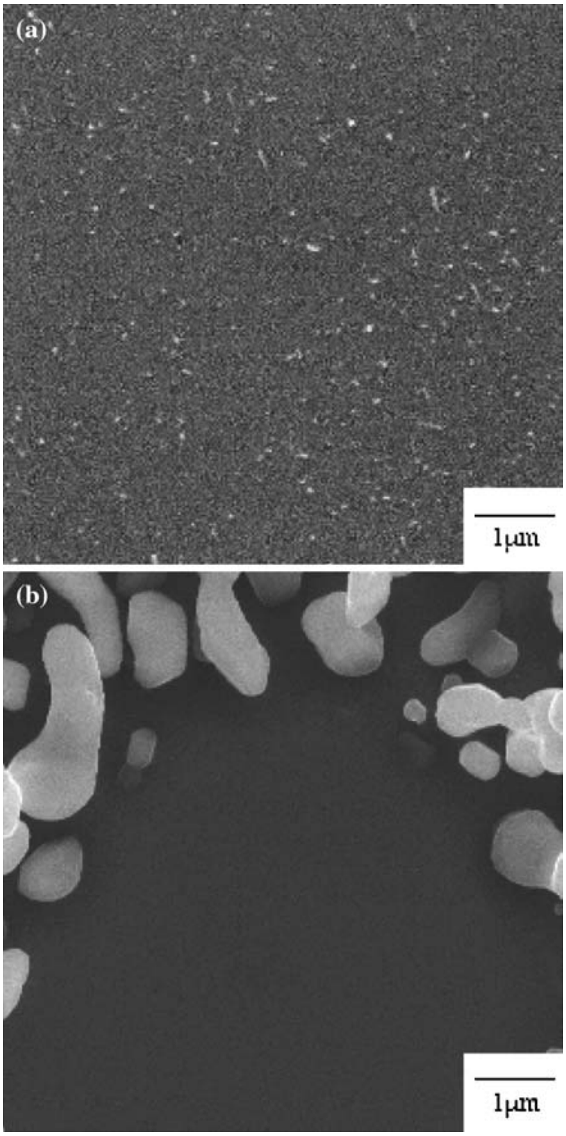

Fig. 2 Effect of aging at 160 °C for 60 min using (a) FB (center of the dendritic region) and (b) CF

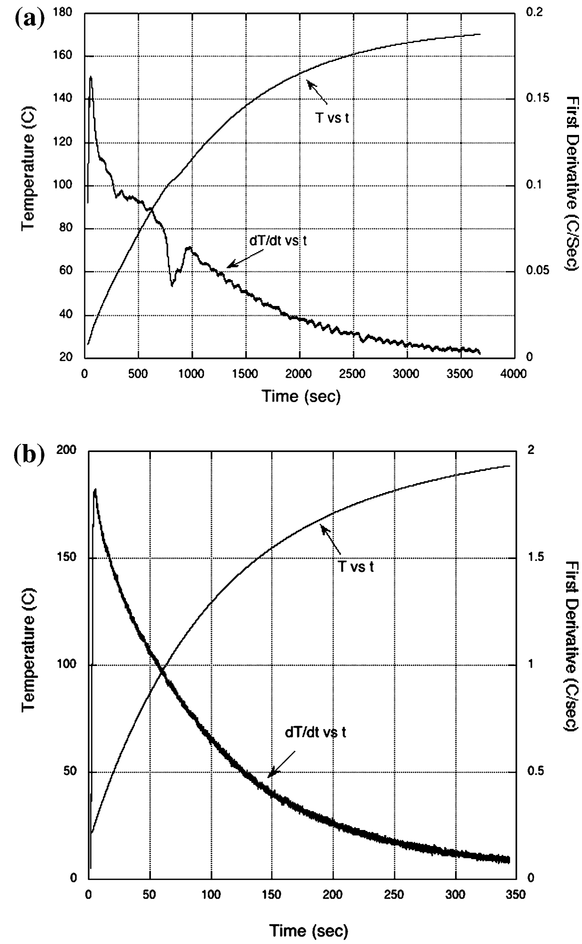

Fig. 3 (a) Thermal analysis of aging treatment of D357 alloy in the CF. (b) Thermal analysis of aging treatment of D357 alloy in fluidized bed

and Sr modified D357 alloys. Castings of Sr modified and unmodified D357 alloys were solution heat treated using FB at 543 °C for 30 min, and 60 min, respectively and were subsequently aged in both FB, and CF. As expected, both UTS and YS values increase with increasing aging time and temperature. However, elongation values decrease rapidly with increasing aging temperature and time. In general, aging of D357 alloys (both Sr modified and unmodified) in FB results in increased UTS and YS values compared to when the alloy is processed in CF. Both UTS and YS values increase significantly from the as-cast state within 30–60 min of aging time in FB, and beyond this no significant change is observed. Whereas, alloys aged in CF show much slower aging kinetics than those processed in FB. However, in contrast to the UTS and YS trend, FB aged alloys have a lower ductility (less elongation) vis-à-vis CF aged alloys.

Conclusions

- Six minutes are required to attain the aging temperature in FB (fluidized bed), compared to 60 min in CF (conventional furnace); over an order of magnitude difference exists between FB and CF aging times.
- Nucleation rate of Mg_2Si particles in FB is greater than that in CF.
- Thermal analysis shows an endothermic reaction during aging in CF. No such transformation was observed during aging of the alloy in FB.
- Nucleation rate of Mg_2Si particles is greater in the Sr modified alloy as compared to the unmodified alloy.
- Coarsening rate of Mg_2Si particles is greater in the unmodified alloy as compared to the Sr modified alloy.

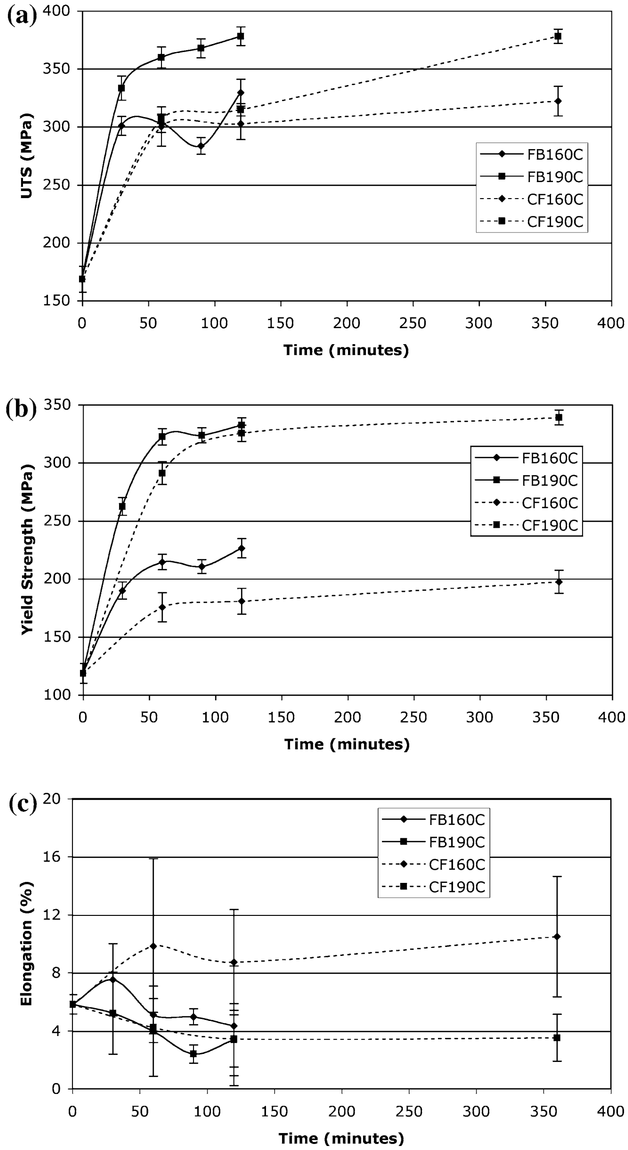

Fig. 4 Effects of T6 heat treatment (Solution HT, quenching, and aging) for unmodified D357 alloy on (**a**) Ultimate tensile strength (UTS), (**b**) Yield strength, and (**c**) Elongation

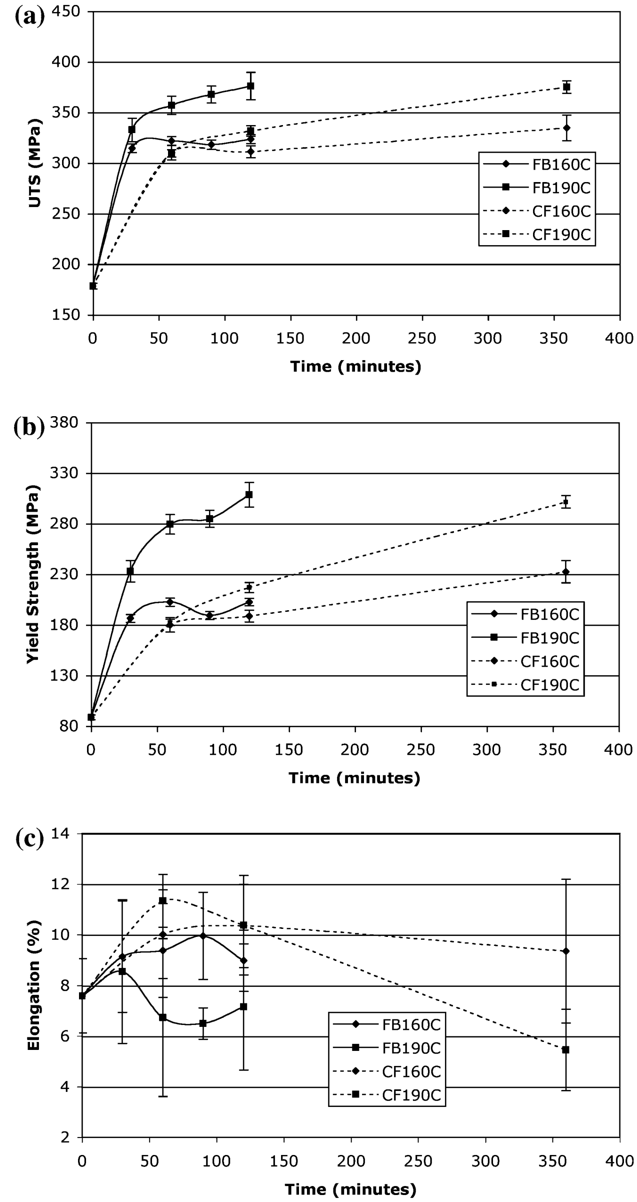

Fig. 5 Effects of T6 heat treatment (Solution HT, quenching, and aging) for Sr modified D357 alloy on (**a**) Ultimate tensile strength (UTS), (**b**) Yield strength, and (**c**) Elongation

Coalescence of smaller particles is the dominant mechanism for coarsening of Mg_2Si particles in the unmodified alloy. Whereas in the Sr modified alloy, Ostwald ripening is the dominant mechanism of coarsening.

- Optimum aging time, in FB is in the order of an hour. Total heat treatment cycle for T6 temper in Al–Si–Mg alloys is reduced to less than 2 h using FB; a significant reduction of cycle time.

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