

Role of Ni and Zr in Preserving the Strength of 354 Aluminum Alloy at High Temperature

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Keywords: 354 aluminum alloy, Al-Si alloy, high temperature, alloying elements, heat-treatment, fracture, nickel, zirconium, Al₃Ni and Al₃Zr.

Abstract

The aim of numerous research projects has always been to achieve a high output, in conjunction with outstanding efficiency of the designated alloys, making them applicable at higher temperature. The present work investigates the effects of minor additions of Nickel and Zirconium to hypoeutectic 354 alloy containing 9%Si, 1.85%Cu, 0.5%Mg, 0.2%Ti, and 0.015%Sr. Ni and Zr were added to these alloys in amounts of 0.2% and 0.4% each, individually or in combination, to investigate their effect on the high temperature mechanical properties of the alloys studied. The alloys were cast in the form of tensile test bars (using an ASTM: B108 mold). The bars were solution heat treated for 8 hrs at 505°C, quenched in warm water, followed by aging at 190°C for 2 hrs. The aged bars were tested at 155°C and 300°C after preheating at these temperatures for times up to 100 hrs.

Introduction

The development of diesel and direct fuel injection gasoline engines with high specific powers have resulted in a big performance impact on piston materials due to increased combustion pressure and piston temperatures.[1] A reduction in the weight of key engine components together with higher service temperatures seems to allow for more efficient operation than hitherto. For these reasons, the aim of the research projects has always been to achieve a high output, in conjunction with outstanding efficiency of the designated alloys, making them applicable at higher temperatures.[2] Improved strength at elevated temperatures has been a continuing goal in aluminum alloy development for more than three decades, [3, 4] specifically in relation to preserving the mechanical properties of the cast alloys used in different components at temperatures of 200°C to 300°C. Most of the Al-Si cast alloys can operate effectively at temperatures of no higher than about 230°C, which may be attributed to the fact that the precipitates formed during thermal treatment coarsen or dissolve rapidly and then transform into more stable phases. This recommendation is made in view of the fact that, for certain applications such as those pertaining to the automotive industry, these alloys must operate over a wide range of temperatures and stress conditions where temperatures may go even higher than 230°C. [1, 5] The task of improving the mechanical properties in an aluminum alloy at high temperatures should include the knowledge of several factors which are related principally to a decrease in the strength of the metal with increasing temperature. This behavior is linked to the change or deterioration in the principal metallurgical parameters of the specific cast aluminum alloy during its performance at higher temperatures.[2]

Aluminum alloys are considerably more economical than the existing high-temperature aerospace alloys such as the Ni- and Ti-based alloys. In order to enhance their strength, one approach is to use low-cost particulate reinforcements to increase the strength of Al-Si alloys.[1] The use of silicon as the major alloying element in aluminum offers excellent properties such as castability, good weldability, good thermal conductivity, excellent corrosion resistance and good retention of physical and mechanical properties at elevated temperatures. The addition of silicon is essential in order to improve the fluidity of the molten aluminum so as to enhance the alloy castability. 354 alloys show a greater response to heat treatment as a result of the presence of both Mg and Cu. These alloys display excellent strength and hardness values although at some sacrifice of ductility and corrosion resistance. Such alloys are then used for a wide range of applications, including the following non-exhaustive list: engine cooling fans, crankcases, high speed rotating parts (e.g. fans and impellers), structural aerospace components, air compressor pistons, fuel pumps, compressor cases, timing gears, rocker arms, machine parts, and so forth.[5, 6]

The addition of transition elements such as Ni and Cu is considered to be an effective way to improve the hightemperature strength of cast Al-Si alloys in as-cast and solutiontreated conditions through the formation of stable aluminides.[7] Copper and magnesium as alloving elements are often added to improve alloy strength at room temperature as well as at higher temperatures. At temperatures over 190°C, the major alloy strengthening phases which include the $\theta'(Al_2Cu)$ and S'(Al₂CuMg) phases will tend to become unstable, coarsen rapidly, and then dissolve, leading to the production of an alloy which has an undesirable microstructure for high temperature applications. Apart from these phases, when a casting is produced, there are other microstructural features which contribute to strength, ductility, and durability; these include secondary dendrite arm spacing, grain size, and Si particle characteristics.[6, 8, 9] The mechanical properties of most Al-Si cast alloys are not severely affected by temperatures of up to 100°C, particularly those related to the tension test. Castings are used in the underaged condition, prolonged exposure to temperatures of the order of 100°C leads to additional precipitation hardening. As a result, the strength increases, and the ductility decreases slightly. At temperatures higher than 150°C, increasing temperatures and exposure times lead to an appreciable reduction in strength and an increase in ductility.[2, 101

Fracture is essentially transgranular and interdendritic in nature up to about 200°C. The fracture and debonding of silicon particles is a major aspect of damage evolution in these allovs.[11] Such fracturing, or debonding, together with the formation and growth of voids around the particles, and the subsequent interlinkage of the voids, all lead to crack propagation in the interdendritic regions. Mechanical properties resulting from exposure to high temperatures may be analyzed on the basis of the two basic categories of fracture observed in cast Al-Si alloys: (i) fractures occurring at temperatures lower than 0.3 Tm (where Tm is the melting temperature of the alloy), and (ii) fractures occurring at temperatures higher than 0.3 Tm.[10] The fracture behavior of aluminum-silicon alloys has been studied by numerous investigators, and it has been shown that fracture occurs in three stages: (i) initiation of cracks in the Si particles; (ii) growth of these cracks into cavities, and (iii) rupture of the aluminum matrix separating the cavities.[9, 10, 12]

The use of nickel and zirconium as additions to increase high temperature strength in aluminum alloys is based on the production of precipitates in the form of Al₃N and Al₃Zr, respectively; such particles are better able to support overaging at higher temperatures than the precipitates normally present in cast aluminum alloys, i.e. Al2Cu and Mg2Si. The degree of strengthening is related to particle distribution in the ductile aluminum matrix.[2] Zirconium is used in a wide selection of aluminum alloys in order to control the microstructure and mechanical properties.[13] Minor additions of zirconium are often made to high strength wrought aluminum alloys for the purpose of regulating grain structure and inhibiting recrystallization during heat treatment, which occurs as a result of the obstructive action of coherent particles with dislocations.[14] According to the literature,[15] it would be necessary to obtain a microstructure containing thermally stable and coarsening-resistant dispersoids in order to improve mechanical properties at high temperatures in an aluminum alloy. Proof stress, tensile strength, and the elongation to failure are all improved by the addition of Zr to cast aluminum alloys.

Zirconium has the smallest diffusion flux in aluminum of all the transition metals, [16] and its addition to aluminum base alloys results in the formation of the Al₃Zr phase, which precipitates out during the initial solution heat treatment in the form of metastable $L1_2$ Al₃Zr particles. Aluminum acts as a solid solution hardener in nickel. Lattice expansion associated with atomic diameter oversize can be related to the hardening observed in nickel base alloys using additions of Al.

The presence of such coherent precipitates as Al₃Zr or Al₃Ni, which are essentially inert in the matrix, affects the strength of the material directly because they act as hard pinning points in the matrix inhibiting dislocation motion. The principal advantage of the precipitates formed is their stability at elevated temperatures: because of the low solubility of the finely dispersed second phase particles, these alloys can resist overaging and growth, thereby preserving their mechanical properties to a greater extent. The capacity of nickel for improving the strength of wrought aluminum alloys at high temperatures has already been established; nickel is thus used in combination with copper to enhance high temperature properties.[17] Binary aluminum-nickel alloys are no longer in use, but nickel is added to aluminum-copper and aluminumsilicon alloys to improve both hardness and strength parameters at elevated temperatures as well as to reduce the coefficient of thermal expansion.[18]

Experimental Procedures

Alloys and Materials

The codes and the nominal level of Ni and Zr added to the base 354 alloy to produce the alloys studied are shown in Table 1, while Table 2 lists the actual chemical composition of each alloy. The 354 base alloy, coded A, was cut into smaller pieces, then dried and melted in a 120 kg capacity SiC crucible, using an electrical resistance furnace in which the melting temperature was maintained at $750 \pm 5^{\circ}$ C.

Alloy code	Nominal composition (wt%)
G1	354 Alloy (containing 200 ppm Sr + 0.25%Ti)
G2	354 Alloy + 0.4%Ni
G3	354 Alloy + 0.4%Zr
G4	354 Alloy + 0.2%Ni + 0.2%Zr
C5	354 Allow + 0.49 Ni + 0.49 Zr

Table 1. Nominal composition of the alloys prepared for the present study

Table 2. Chemical composition of the alloys used in the present work

Alloy	Element (wt%)										
	Si	Fe	Cu	Mn	Mg	Ti	Sr	Ni	Zr	Al	
G1	9.40	0.08	1.85	0.01	0.49	0.22	0.015	-	-	87.6	
G2	9.20	0.08	1.84	0.01	0.49	0.22	0.015	0.46	-	87.8	
G3	9.10	0.07	1.83	0.00	0.45	0.21	0.015	-	0.40	87.8	
G4	9.00	0.08	1.85	0.00	0.45	0.21	0.013	0.20	0.20	87.8	
G5	9.10	0.08	1.86	0.00	0.46	0.22	0.012	0.40	0.40	87.6	

All alloys were grain refined and modified using Al-5%Ti-1%B and Al-10%Sr master alloys, respectively, to obtain levels of 0.25% Ti and 200 ppm Sr in the melt. The melts were degassed for ~15-20 min with a rotary graphite impeller rotating at ~130 rpm, using pure dry argon. Following this, the melt was carefully skimmed to remove oxide layers from the surface, and then carefully poured into an ASTM B-108 permanent mold preheated at 460°C, to prepare test bars for tensile testing. Each casting provided two tensile bars, each with a gauge length of 70

mm and a cross-sectional diameter of 12.7 mm, as shown in Figure 1. Samplings for chemical analysis were also taken at the beginning, middle, and end of the casting process to ascertain the exact chemical composition of each batch of test bars produced per alloy. For the alloys G2, G3, G4 and G5, the master alloys were cut into smaller pieces, dried and then added to the base alloy melt. The additions were made just before degassing to ensure homogeneous mixing of the additives together with the degassing.



Figure 1. Dimensions of the tensile test bars used in the present study.

Heat Treatment

Heat treatment of the test bars used for tensile testing involved solution heat treating them at 505°C for 8 h. The solution heat-treated samples were quenched in warm water at 60°C followed by artificial aging for a duration of 2 hrs at 190°C. After aging, the test bars were allowed to cool naturally at room temperature at 25°C. All heat treatments were carried out in a Lindberg Blue M electric resistance furnace.

Tensile Testing

All of the samples, whether as-cast, solution heat-treated, or at high temperature, were tested to the point of fracture using an Instron Universal mechanical testing machine at a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$. The yield strength (YS) was calculated according to the standard 0.2% offset strain, and the fracture elongation (El) was calculated as the percent elongation (%El) over the 25.4 mm gauge length. The ultimate tensile strength (UTS) was obtained from the data acquisition system of the Universal machine. In order to reach and stabilize the intended test temperature during the tests, at the time that the samples were mounted in the tensile machine, the furnace was already pre-set at the required temperature; the samples then were kept mounted in the furnace of the tensile testing machine for 30 minutes before starting the test. The test bars was tested as the as-cast and heat-treated conditions, and at 155°C and 300°C (high temperature) for times of 10, 40, and 100 hrs at each temperature. Five samples were used for each alloy/heat treatment/testing condition. The average %El, YS, and UTS values obtained from the five samples tested per condition were considered as the values representing that specific condition.

Metallography

Metallographic examination was carried out using an optical microscope linked to a Clemex image analysis system, for which polished samples were prepared following standard procedures to examine the fracture path and the components of microstructure involved in the crack formation occurring on and below the surface of metallographic samples. For metallographic characterization, three samples of G1, G4, and G5 alloys, ascast, heat-treated and at high temperature 155°C and 300°C

conditions, were sectioned off from test bars corresponding to each alloy. Jeol 840A scanning electron microscopy (SEM) equipped with an energy dispersive x-ray system (EDS) and a Hitachi S-4700 field-emission scanning electron microscopy (FE-SEM) were used to examine fracture surface for these alloys.

Results and Discussion

This section presents the results obtained from the tensile testing and microstructure examination. The results are discussed so as to explain the effects of the Ni and Zr alloying additions (viz., G1, G2 and G5 alloys) used on the high temperature performance of 354 alloys.

Tensile Test Results - As-Cast and Heat-Treated

Figures 2 and 3 compare the tensile properties of the alloys in the as-cast and heat-treated conditions with those obtained at high temperatures of 155°C and 300°C. The selected solution heat treatment temperature may appear to be over-conservative, but it was deemed necessary to avoid the risk of any incipient melting of the copper phases in the alloy samples, thereby avoiding the possibility of deteriorating the mechanical properties to a great extent. Increased strength after the treatment is normally to be observed in similar 354 alloys which use a solution heat treatment temperature of 525°C over a 2-3 hours period, in conjunction with analogous aging treatments [19]. The strength values for the as-cast alloy samples show a difference of 60 MPa between G1 (340.24) and G5 (277.85) allovs, respectively. After T6 heat treatment, the UTS, YS and El% values immediately increase. This augmentation in the mechanical properties is related to the changes occurring in the form of the silicon particles which become more rounded, decreasing their aspect ratio and density. The changes in the morphology of the Si particles during solution heat treatment are reflected principally in the increase in the ductility values rather than in that of the strength. The Si particles in the as-cast alloys act as stress concentrators since they are harder than the matrix; serving as stress concentration sites; such particles tend to promote crack propagation during load application thereby decreasing the ductility of the alloy. After solution heat treatment, tensile results for alloy G4 (containing 0.2%Ni +

0.2%Zr) show an increase in UTS, YS and %El of 121 MPa, 165 MPa, and 0.10%, respectively. The G1 and G5 alloys display a slight increase in UTS of approximately 40MPa and 25 MPa after heat treatment (T6), respectively. Alloy G4 is the only alloy which shows a large increase in strength in comparison with base alloy G1. In fact, alloy G4 (with 0.2%Ni + 0.2%Zr) displays greater strength than alloy G3, which contains 0.4%Zr, or even alloy G5, which contains 0.4%Ni + 0.4%Zr.

Tensile Test Results at High Temperature 155°C

Tensile data for tests carried out at 155°C are shown in Figure 2. The figure clearly reveals that the G2 to G5 alloys do not show any significant change in UTS and YS at 155°C for holding times of 10, 40 and 100 hours at temperature used in this study. The limited amount of research work which has been carried out on the casting of aluminum alloys with zirconium additions shows that a reduction in the grain size as well as an increase in the strength of alloy G3 with respect to the G1 base alloy is to be expected. [14, 15, 16] While it was found that the addition of 0.4%Zr did indeed reduce the grain size, no increase in the resistance of alloy G3 was observed. Therefore, at this temperature, and for the three holding times investigated, it may be said that the strength and ductility values exhibited by the five alloys lie approximately in the range of ~300 MPa UTS, ~290 MPa YS, with a %El of ~1.5%, respectively.

Tensile Test Results at High Temperature 300°C

Figure 3 shows the results obtained for tensile tests carried out at 300°C for times up to 100 hrs. As will be shown in the next section, the morphology of the intermetallic phases observed in the structure has a harmful influence on the mechanical properties. The addition of 0.4%Ni + 0.4%Zr was not sufficient to resist softening at 300°C, so that the strength values decreased to 52MPa UTS, and 45MPa YS, while the ductility increased to 22%. As was observed in the work of Knipling [20] on Al-Zr and Al-Zr-Ti alloys, Zr-rich nano-particles precipitate during solidification forming coherent precipitates which are resistant to coarsening at temperatures in order of 275°C-425°C. Such Zrrich particles would thus improve the properties of alloy G4 at 300°C compared to the other alloys. It is possible that alloy G5 in T6 condition did not perform the same properties than as-cast condition did. This opposite behavior can be explained by the fact that alloys in the T6 condition have been submitted before to a solution heat treatment at 505°C. Such temperature would change the coherency of the Zr-rich precipitates formed during the solidification of alloy G4. Moreover, at 300°C for the times proposed and tested, the UTS at 10 hrs started to decrease, reaching 53 MPa at 100 hrs in almost all alloys. The YS exhibited a similar behavior. The most significant results obtained at 300°C during 100 hrs were the high ductility values obtained at this temperature, giving percent elongation values



Figure 2. Tensile test results obtained for alloys G1, G2, G3, G4 and G5 tested at room temperature and at high temperature 155°C, after holding times of 10, 40 and 100 hrs: a) UTS; b) YS; and c) %El.



Figure 3. Tensile test results obtained for alloys G1, G2, G3, G4 and G5 tested at room temperature and at high temperature 300°C, after holding times of 10, 40 and 100 hrs: a) UTS; b) YS; and c) %El.

ranging between 22 and 30% for the five alloys studied, as shown in Figure 3c).

Microstructural Examination

The parameters controlling the microstructure of Al-Si casting alloys include alloy composition, melt treatment, solidification conditions, and the heat treatment applied to the casting. In the present case, the microstructural examination will focus on the fracture behavior, to understand the tensile test results in light of the microstructural features observed in samples prepared from tested bars of G1, G2 and G5 alloys, using longitudinal sections obtained below the fracture surface of the test bars, as well as on the fracture surfaces themselves. The former would provide determining the fracture mechanism, whereas the latter would shed light on the phase responsible for the fracture and the nature of the fracture surface.

Fracture Behavior

Figures 4 to 6 show optical micrographs of the longitudinal sections below the fractures surface of G1, G2 and G5 alloy samples, corresponding to various conditions (as-cast, T6, and 300° C/100 hrs. Prominent features observed are the white α -Al dendrites and the grey Al-Si eutectic phase in the interdendritic regions. It will be observed from these micrographs that the

formation of microcracks begins with the craking of the brittle eutectic Si particle. The copper phase was observed mostly as small pockets of the blocklike Al₂Cu; the relatively high percentage of magnesium, and the strontium content, all led to an increase in the tendency of this phase to segregate to the limits of the α -Al dendrites. The microstructures in Figures 4 to 6 also revealed an elongation of the dendrites in the direction of the loading axis.

The interaction of Zr and Ni with different elements to form Zr-Ni-Al-Cu, Zr-Ni-Al, and Zr-Ti-Si phases has been reported by several researchers studying these systems.[19, 20, 21, 22] Figure 4a) shows the microstructure beneath the fracture surface of the alloy G1 sample in the T6 heat-treated condition (at 50x); Figure 4b) shows the corresponding microstructure of the G1 sample tested at high temperature 300°C/100 hrs. Likewise, Figures 5a) and 5b) show the microstructures beneath the surface fracture of alloy G2 samples corresponding to the same T6 and 300°C/100 hrs conditions, while Figure 6 depicts those corresponding to alloy G5.

Several studies [19, 25, 26, 27] mention that the spheroidization process of the eutectic silicon throughout heat treatment takes place in two stages: fragmentation or dissolution of the eutectic Si branches and the spheroidization of the separated branches. Controlling the microstructure during solidification is, therefore, very important. The nucleation and growth of the Al-Si eutectic

in hypoeutectic Al-Si alloys been shown to influence the formation of defects such as shrinkage and microporosity. These defects have an important effect on the ultimate mechanical properties of the casting alloys. Solution temperature is the most relevant parameter that can influence the kinetics of Si morphology transformation during the course of solution heat treatment. Normally, the fracture path is found to be mostly transgranular, and the appearance of the fracture surface is more



flat. The fracture of the α -Al dendritic network is ductile but in general depends on the morphology of eutectic Si and the quantity of brittle intermetallic phases present. The fracture surfaces of the heat-treated alloy samples and those tested at high temperature showed that coarse and deep dimples are the major feature of the fracture surface compared to the shallow dimples observed in the samples corresponding to the as-cast condition.



Figure 4. Optical micrographs at 50x showing the fracture surface of alloy G1 samples: a) T6 condition, and b) tested at 300°C/100h.



Crack propagation Eutectic Si a-Al b)

Figure 5. Optical micrographs at 50x showing the fracture surface of alloy G2 samples: a) T6 condition, and b) tested at 300°C/100h.



Figure 6. Optical micrographs at 50x showing the fracture surface of alloy G5 samples: a) T6 condition, and b) tested at 300°C/100h.

The topography of the fracture surface is commonly examined using scanning electron microscopy (SEM), where the samples may be without almost any special preparation. However, it is important that the specimens should be examined immediately after failure because of the very fast superficial oxidation of Alalloys. The fracture surfaces of the alloys studied were examined using SEM to find the features responsible for crack initiation. Fractographs of samples corresponding to G1, G2 and G5 alloys in the as-cast and T6 heat-treated conditions, and those tested at 300°C/100 hrs and EDS spectra corresponding to the constituents observed in the latter samples are provided in Figures 7 to 9.

Compared to the as-cast condition, it was noted that the silicon platelets in the T6-treated samples were fragmented into smaller platelets with spherical edges, implying that at the 505°C solution heat treatment temperature, the spheroidized particles grew progressively larger viz., they coarsened. The fracture was transcrystalline ductile in nature, exhibiting fine dimple morphology. In addition, other casting defects such as microporosities, oxide inclusions and shrinkage, could also be responsible for the fracture since the size of these defects can be larger than the size of the particles present in the microstructure. Figure 7 displays backscattered images of G1 base alloy samples in the a) as-cast, and b) T6 heat-treated conditions, and c) in the sample tested 300°C/100 hrs. The AlFeCuMg phase was detected in the high temperature sample, as was confirmed by the EDS spectrum shown in d), obtained from the AlFeCuMg phase particles observed in c).

Similarly, Figure 8 shows the corresponding backscattered images of G2 alloy samples for the a) as-cast, b) T6 heat-treated, c) high temperature tested conditions, where the EDS spectrum in d) detected the presence of Al₃CuNi, and (Al,Si)₃(Ti,Zr) phases. The (Al,Si)₃(Ti,Zr) phase, present in the microstructures of alloys containing Zr, is a needle-like Zr-rich phase. As mentioned previously, the copper phase was seen mainly as small pockets of the block-like Al₂Cu, attributed to the strontium and high percentage of magnesium present in the alloy, which cause an increase in the segregation of this phase at the limits of the α -Al dendrites.

In the case of G5 alloy, the backscattered images shown in Figure 9 for the same three conditions reveal the presence of AlCuMgNi phase in the high temperature 300°C/100 hrs sample, as was detected by the EDS spectrum shown in d).



Figure 7. Backscattered images of G1 alloy samples: a) as-cast, b) T6-treated, c) tested at 300°C; d) EDS spectrum corresponding to the AlSiCuMg phase observed in c).



Figure 8. Backscattered images of G2 alloy samples: a) as-cast, b) T6-treated, c) tested at 300°C/100h; and d) the EDS spectrum after T6 heat-treated corresponding to the AlCuNi phase observed in b).





Figure 9. Backscattered images of G4 alloy samples: a) as-cast, b) T6-treated, c) tested at 300°C/100h; d) EDS spectrum corresponding to the AlCuMgNi phase observed in c).

Conclusions

From an analysis of the tensile test data and microstructural observations presented here, the following conclusions may be drawn.

- The addition of 0.4%Ni + 0.4%Zr (Alloy G) is not sufficient to resist softening at 300°C. (cf. UTS: 52MPa, YS: 45MPa, %El: 22% with UTS: 359 MPa YS: 343, %El: 1.23 after T6 treatment).
- 2. An increase in UTS, YS and %El of 121 MPa, 165 MPa and 0.10%, respectively is observed after T6 heat treatment of G4 alloy (containing 0.2% Ni and 0.2% Zr).
- 3. The addition of 0.4%Ni to alloy 354 leads to a decrease in the tensile properties compared to the base alloy G1. This decrease may be attributed to a nickel-copper reaction which could interfere with the formation of the Al₂Cu, strengthening precipitates.
- 4. Addition of Zr and Zr+Ni increase the high temperature tensile properties, in particular, those of alloy G4, (containing 0.2%Ni and 0.2% Zr).
- 5. The needle-like phase (Al,Si)₃(Zr,Ti) is the main feature observed in the microstructures of G3 and G5 alloy samples which containing 0.4% Zr.
- The ductility (%El) of the alloys studied increases on going from the as-cast to the T6 heat-treated samples, to those tested at 155°C and 300°C, respectively.
- 7. The microstructure beneath the fracture surface showed elongation of the dendrites in the direction of the loading axis.
- 8. The fracture surfaces of heat treated samples and those tested at high temperature showed that coarse and deep dimples are the major feature of the fracture surface compared to the shallow dimples observed in the as-cast condition.

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