

Heat Treatment and Precipitation in A356 Aluminum Alloy

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A thesis submitted to the Faculty of Graduate Studies and Research
in partial fulfillment of the requirements of the degree of
Doctor of Philosophy

September 2003

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2003/4/28

ABSTRACT

Al-7%Si-0.3Mg (A356) foundry alloy is strontium modified and heat treated in order to obtain optimum mechanical properties. To date, the role that strontium modification plays in the precipitation process during the heat treatment is not well understood.

The objective of the present work is to obtain a better understanding of the effect that strontium modification and heat treatment parameters exert on the properties of heat-treated Al-7%Si-0.3Mg alloy. The investigation consisted of a systematic measurement of the electrical conductivity and matrix microhardness of heat-treated coupons under different process conditions. Tensile properties were also measured and a transmission electron microscopy study of critical samples was done.

The results obtained in this work indicate that the heat treatment of this alloy can be optimized. Solution heat treatment and artificial aging can be reduced and natural aging manipulated. The use of a continuous heating artificial aging is proposed.

During the heat treatment of the A356 alloy under certain conditions not only does the precipitation of the Mg_2Si occur, but also that of silicon. It was found that strontium modification does not influence the precipitation kinetics of the Mg_2Si , but it does retard the precipitation of the silicon out of the matrix.

RÉSUMÉ

L'alliage de fonderie Al-7%Si-0.3Mg (A356) est modifié par l'addition de strontium et traité thermiquement afin d'obtenir des propriétés mécaniques optimales. Jusqu'alors, le rôle de l'addition de strontium dans la cinétique de précipitation pendant le traitement thermique n'était pas bien compris.

Le but de cette étude est d'obtenir une meilleure compréhension de l'effet qu'exercent l'addition de strontium et le traitement thermique sur les propriétés de l'alliage Al-7%Si-0.3Mg traité thermiquement. Les recherches ont consisté en la mesure systématique de la conductivité électrique et de la microdureté de la matrice, d'échantillons ayant subi un traitement thermique, sous différentes conditions. Les propriétés de traction ont été aussi mesurées et une étude des échantillons considérés comme critiques a été réalisée par microscopie électronique à transmission.

Les résultats obtenus dans cette étude indiquent que le traitement thermique de cet alliage peut être amélioré. Le traitement thermique de mise en solution et le vieillissement artificiel peuvent être réduits. De plus, le vieillissement naturel peut être ajusté. L'utilisation d'un traitement thermique continu de vieillissement artificiel est proposé ici.

Durant le traitement thermique de l'alliage A356, on observe la précipitation de Mg_2Si mais aussi, celle du Si, sous certaines conditions. On a trouvé que l'addition de strontium n'influence pas la cinétique de précipitation mais il retarde la précipitation du Si en dehors de la matrice.

ACKNOWLEDGMENTS

Firstly, I would like to acknowledge the National Council of Science and Technology of Mexico (CONACYT) and the University of Nuevo Leon (UANL) for their support during all my graduate studies. I will always be indebted to these institutions. Also, I would like to thank the Natural Science and Engineering Research Council of Canada (NSERC) for providing additional support for this research.

I want to thank and express my sincere gratitude to my supervisor, Prof. John E. Gruzleski, for his guidance, support, encouragement, patience and helpful suggestions throughout this work and also for setting a high standard for my future with his professionalism.

Many thanks and gratitude to Dr. Florence Paray and Dr. Daryoush Emadi for their friendship and continuous help and suggestions during the realization of this research.

Many thanks to Peter Newcombe, Luc Millette and Val Gerstman from the Materials Technology Laboratories-CANMET for their help.

My gratitude to all the personnel and professors of the Department of Mining, Metals and Materials Engineering for their help in one way or another. Special thanks to Robert, Helen, Edwin, Monique, Ray, Carol, Barbara, Norma, Purnima, Genny, Slavak, Robert, Nelson, Bob, Prof. Mucciardi, Prof. Gauvin, Prof. Drew, Prof. Vali and Dr. K. Sears. Sincere thanks to all my friends Mathieu (Cochon-Td'G-Cd'Q), Pui Kei, Angela, Raul Sedas, José R, Noriko, Abdel, Neivi, Gregg (A's), Lisa, Chunhui, Khaled, Mehrdad, Mamoun, Mathumati, Xin (Chou Gou), Vladimir, Daryl, Faramarz, Luis, Ahmad, Farzad, Ozgur, Ramona, Fabian,

Pierre-Louis, Zina, Rocco, Lihong, Cinziana, Lan, Joel, Aurelie, Miguel, David L., Polo, Toño, Tomás, José L., Carlos, Ena, Moon and the rest of graduate students for their friendship and good times we have enjoyed at McGill.

The city of Montreal for being such an enjoyable place for studying and enjoying life and baseball.

I would also like to thank Prof. Jesús Moreno and Prof. Rafael Colás for all their guidance.

Finally, I would like to acknowledge all my family, especially my wife and parents for all they have done for me.

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Chapter 1

INTRODUCTION

The Al-7%Si-0.3Mg alloy is widely used for the casting of high strength components in automotive, aerospace and military applications, because it offers a combination of high achievable strength, castability, pressure tightness and corrosion resistance in both the sand cast and permanent mould condition. However, this alloy is rarely used in the as-cast condition, without any pre-treatment, because it exhibits relatively poor mechanical properties due to the presence of eutectic silicon in the form of coarse acicular plates which act as internal stress raisers under an applied load.

Chemical treatment and heat treatment are the two main processes used to improve the properties of this alloy. The chemical treatment, also known as modification, is a liquid state process that is achieved by the addition of small quantities of sodium or strontium to the melt. It results in a change of the morphology of the eutectic silicon phase from coarse acicular to fine fibrous, which results in an improvement of the mechanical properties. Regarding the heat treatment, this is a process used to obtain a desired combination of mechanical properties (strength and ductility). The most common is the T6, which consists of a solution heat treatment, water quenching and artificial aging. The solution heat treatment, generally, is carried out at a temperature slightly under the eutectic temperature at about 540°C for 4 to 8 hours. This treatment dissolves hardening elements (Mg and Si) in the Al matrix, homogenizes the casting and spheroidizes the eutectic silicon. After the solution treatment, castings must be quenched from this high temperature in order to avoid the precipitation of dissolved elements and to create a supersaturated solid solution at room temperature, an essential condition for further aging. Natural aging takes place

during the delay time at room temperature between quenching and artificial aging. The influence of natural aging on the properties of heat-treated castings is not yet well understood, but it appears to have a detrimental effect. Artificial aging is the step where the hardening elements are precipitated in order to improve the strength of the aluminum matrix. This process is carried out at relatively low temperatures (155°C – 200°C).

Nowadays, the effect of Na or Sr is well understood on the modification of the eutectic silicon in Al-7%Si-0.3Mg alloys. However, the effect that chemical modification may have on the precipitation of Mg_2Si during heat treatment is not clear.

Some research work has been done to date in order to investigate the effect of strontium modification on the heat treatment of Al-Si-Mg alloys. A study of the electrical conductivity of synthetic Al-Si-Mg alloys, with and without strontium, found that during solution treatment strontium somehow affects Mg_2Si dissolution into the aluminum matrix. This effect seems to be more pronounced at shorter solution treatment times than at longer ones where the effect diminishes. It was also found that the total conductivity change during T6 is smaller in strontium containing alloys than in alloys which do not contain strontium. This observation may be evidence of the strontium effect on the precipitation of Mg_2Si during aging [1]. Another research work observed a decrease in the yield strength in artificially aged A356 strontium modified alloy, which again may indicate an effect of the strontium on the precipitation of the Mg_2Si [2]. However, another study that involved the study of heat-treated A356 strontium modified alloy, did not find evidence of any effect of strontium on the precipitation of Mg_2Si [3].

The aim of the present research work is to study the influence that strontium modification and heat treatment parameters have on the properties of heat-treated Al-7%Si-0.3Mg alloy.

The particular objectives of this study are:

- To study the effect of the heat treatment process variables (solution heat treatment time, quenching temperature, natural aging time and artificial time and temperature) on the properties of unmodified and modified Al-7%Si-0.3Mg alloys.
- To study the strontium modification effect on the properties of heat-treated A356 alloy.
- To study the Mg_2Si precipitation process under different heat treatment conditions.
- The optimization of the heat treatment by a more detailed understanding of the precipitation process.

Chapter 2

LITERATURE REVIEW

2.1 Aluminum-Silicon Foundry Alloys

The industrial importance of aluminum alloys containing silicon as the major alloying element results from their excellent castability, good corrosion resistance, and the fact that they can be machined and welded [4]. As shown in Figure 2.1, the Al-Si binary system forms a simple eutectic at 577°C and 12.6% Si. It can also be seen that aluminum can dissolve a maximum of 1.62% Si into solid solution. Depending on the amount of silicon, the Al-Si casting alloys are divided into three groups [5]: hypoeutectic alloys with Si contents between 5% and 10%, eutectic alloys with 11%-13% Si, and hypereutectic alloys, commonly with a Si content between 14% and 20%. Generally, hypoeutectic and near-eutectic Al-Si alloys are used when good castability and corrosion resistance are required, moreover, small additions of Mg and Cu are added in order to enhance their response to heat treatment. The increase in properties from heat treatment, results in an extensive use of these alloys in a variety of applications in the automotive, aerospace and military industry. However, the presence of large, brittle, acicular flakes and plates of eutectic silicon leads to a substantial reduction of strength and elongation of the castings. Modification of the eutectic silicon by small additions of Na or Sr results in a noticeable improvement in strength, and a significant increase in elongation [6-8].

Hypereutectic Al-Si alloys, find uses when excellent wear resistance and low thermal expansion is required. An example of this is in the manufacturing of engine blocks, pistons and cylinder liners [9]. However, the presence of hard primary silicon in the structure makes machining difficult and expensive. It has

been found that small additions of phosphorous (~0.01%) produce finer and more evenly distributed primary silicon crystals, which results in an improved machinability, wear resistance and mechanical properties [10,11].

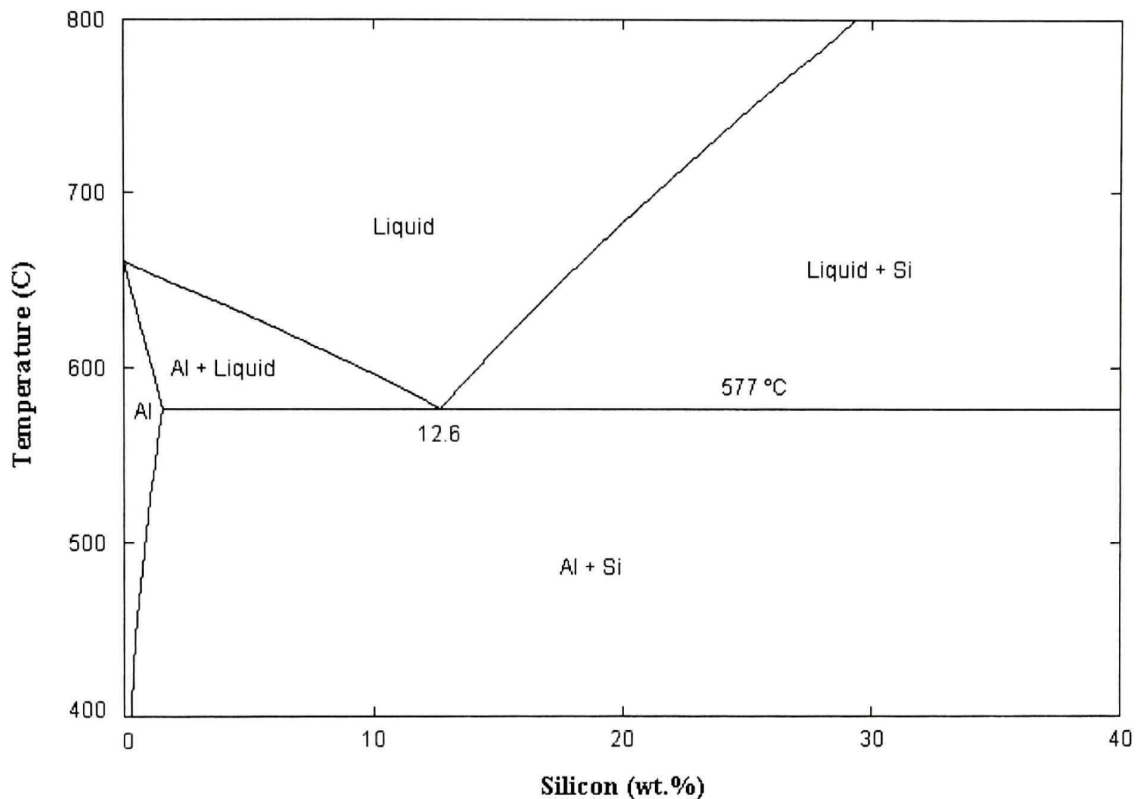


Figure 2.1. Equilibrium binary Al-Si phase diagram

2.2 Modification of Eutectic Silicon

It is well known that the eutectic silicon phase in unmodified Al-Si foundry alloys occurs in the form of coarse platelets. Modification can be defined as the transformation from coarse platelets to a fibrous, seaweed like form. Eutectic silicon modification is associated with enhanced properties of Al-Si castings [4]. Modification of Al-Si alloys can be achieved thermally by increasing the solidification cooling rate, or during the solution heat treatment, or chemically by small additions of certain elements of group IA, IIA and lanthanides prior to

pouring [12-16]. Figure 2.2 shows the typical microstructure of an A356 alloy unmodified and modified.

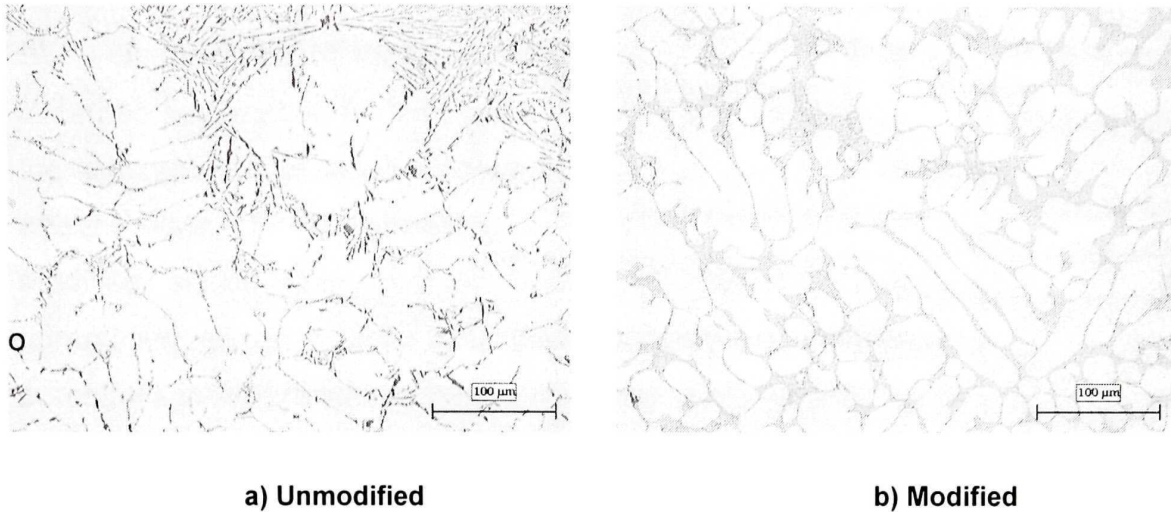


Figure 2.2. Typical as-cast microstructure of A356 aluminum alloy

There have been many theories postulated to explain the mechanism by which modifiers affect the silicon morphology. These theories can be classified in two main groups [17], the theories based on nucleation mechanisms and the theories based on growth mechanisms.

Regarding the theories that involve nucleation mechanisms, Crosley and Mandolfo [18] proposed that the nucleation of the eutectic silicon in the Al-Si alloys is controlled by aluminum phosphide (AlP) due to their very similar crystal structures, leading to the proposal that AlP is likely an effective nucleant. In commercial Al-Si alloys, the phosphorus content is high enough to provide an abundance of AlP particles in the melt. This leads to the nucleation of the eutectic silicon at small undercoolings ($<2^{\circ}\text{C}$) and the formation of coarse acicular silicon flakes. When a modifier is added, it neutralizes the phosphorus by reacting with it. Consequently the silicon nucleation by AlP is eliminated [18-20], leading to an increase in the undercooling for nucleation of silicon by aluminum and resulting in fibrous structures. The main assumption of this theory is that the Si phase is discontinuous and its formation depends on the repeated nucleation of silicon.

This has been proved to be incorrect as the silicon phase is continuous in both unmodified and modified alloys [20]

In the case of the theories that involve growth mechanisms, the most accepted explanation is that proposed by Lu and Hellawell [21] that is based on the assumption that modifiers retard the growth rate of silicon by a twin plane re-entrant edge (TPRE) poisoning mechanism [21-24]. It has been found that modified silicon fibers contain orders of magnitude more twins than do unmodified silicon phases [21], thus, considering imperfections (twins) as a potential site for branching, silicon will bend and twist during growth and will form a fibrous rather than a plate morphology. Unmodified structures do not exhibit this phenomenon due to their relative crystallographic perfection, and can do little but to form in coarse acicular fashion [4].

2.2.1 Strontium as a Modifier

Strontium is usually added to the melt in the form of Al-Sr master alloys such as Al-10 wt% Sr. Pure strontium is rarely used as a modifier due to its high reactivity with air and water vapor and the formation of a tenacious oxide layer that can completely prevent dissolution unless it is mechanically removed [4,25]. The Al-Sr master alloys have different dissolution characteristics with the optimum treatment temperatures ranging from 680°C to 750°C in A356 aluminum alloys and dissolution times about 5 to 15 min [25].

Higher recovery (>90%) and lower rate of loss are the major advantages of strontium over sodium. However, it is reported that the effect of strontium is weaker than that of sodium, especially at low freezing rates. Larger strontium additions are required to yield the same effect as sodium [4,22]. The melt treatment process itself is simple and can be done by plunging the modifier held in a perforated cup or bell below the surface of the melt.

2.3 Heat Treatment of Al-Si-Mg Alloys

Depending on the composition of the alloy and the casting process, as-cast aluminum is susceptible to dimensional growth and low strength, toughness and corrosion resistance [26]. In order to solve these problems, castings can be thermally processed under different heating and cooling cycles to improve their properties.

In the case of Al-7%Si-0.3Mg alloy, the most common heat treatment is the T6, which consists of a solution heat treatment, water quenching and artificial aging. The properties achieved with this heat treatment will depend on the combination of the different parameters involved in the process.

2.3.1 Solution Heat Treatment

To take advantage of the precipitation hardening reaction, it is first necessary to produce a supersaturated solid solution. The process by which this is accomplished is called solution heat treatment. This process basically consists of soaking the alloy at a temperature sufficiently high to dissolve the hardening elements (Mg and Si) into the α -Al in order to create a nearly homogeneous solid solution [27]. Figure 2.3 shows the pseudo-binary phase diagram of Al-Mg₂Si where the increase of solubility of Mg₂Si in α -Al with increasing temperature is seen.

Many organizations and authors recommend a solution heat treatment temperature slightly below the eutectic temperature at about 540°C [27-31], although, other authors have successfully used both lower temperatures (500°C) and higher temperatures (up to 560°C) [32-35]. However, lower solution heat treatment temperatures will require a very long treatment time (up to 17 hours) to achieve the degree of spheroidization that can be achieved at 540°C, while

higher temperatures can result in the melting of Fe- and Cu-containing particles and quenching distortion problems, depending on the casting geometry.

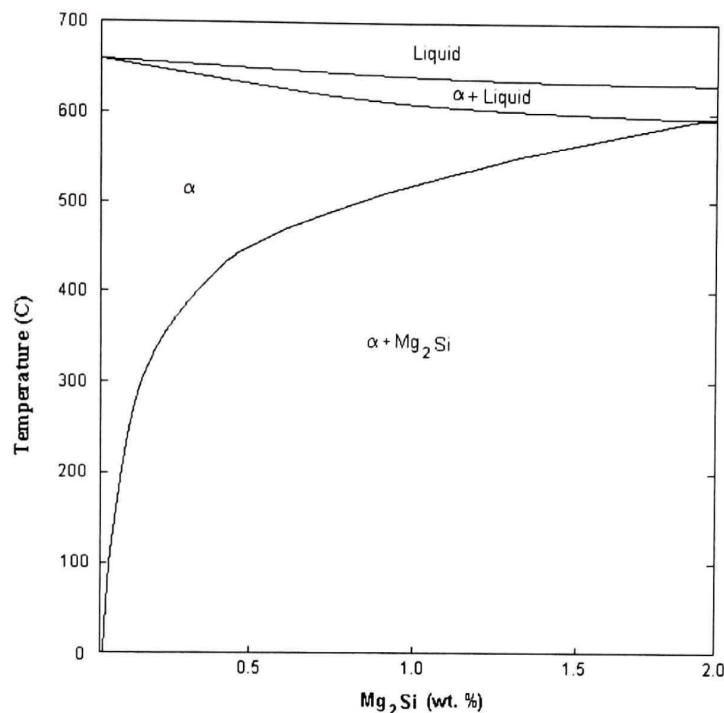


Figure 2.3 Pseudo-binary Al-Mg₂Si phase diagram

During the solution heat treatment several metallurgical processes take place: the dissolution of the hardening elements (Mg and Si) into α -Al, the homogenization of the casting, and the spheroidization and coarsening of the morphology of the eutectic silicon.

2.3.1.1 Dissolution of the Magnesium and Silicon into the Aluminum Matrix

Dissolution is a diffusion-controlled process in which the soluble phases and precipitates formed during solidification are dissolved into the aluminum matrix. According to standard practices [27-29] solution heat treatment temperatures for the A356 aluminum alloys are around 540°C for times from 4 to 12 hours. At this temperature the solid solubility of silicon and Mg₂Si in the Al matrix are 1.5 wt. pct. [36] and 1.4 wt. pct. [37] respectively. Therefore, dissolution of these elements will occur during solution treatment. It has been

found that the dissolution of the Mg_2Si in the A356 alloy occurs within 15 minutes at 540°C [38,39] meaning that the typical solution heat treatment times will put silicon in excess of that needed to form stoichiometric Mg_2Si .

Several researchers, such as Kaczorowski [40], Kovács et al. [41], and Ceresara et al. [42], have stated that excess silicon above that required to form stoichiometric Mg_2Si increases the strength of AlSiMg alloys. According to Kaczorowski [40], this can be explained by:

- 1 A greater driving force for precipitation related to an increased deviation from equilibrium.
- 2 An increase in the number of vacancy-solute clusters and a shortened diffusion path to the clusters.
- 3 An increase in cluster stability, leading to a better dispersion of precipitates.

2.3.1.2 Homogenization

During the solution heat treatment, homogenization of the as-cast structure takes place and segregation of the alloying elements is minimized. Parameters such as solutionizing temperature, and solidification cooling rate (i.e. finer or coarser microstructure) of the casting will play a very important role in the time required to achieve complete homogenization. Some constituent phases, such as iron-rich intermetallics, contain insoluble elements and are changed little by solution treatment [26].

2.3.1.3 Spheroidization and Coarsening of the Eutectic Silicon

The morphology of the eutectic silicon plays an important role in determining the mechanical properties of casting components. Particle size, shape and spacing are factors that characterize the silicon morphology. As discussed before, under normal casting conditions, silicon particles are present in the form of coarse platelets. However, there are several ways in which the silicon morphology can be altered: a) by the addition of small amount of chemical elements such as Na or Sr, b) by a very rapid solidification cooling rate or c) by subjecting the casting to a high temperature treatment for long periods. At elevated temperatures (during solution heat treatment) spheroidization and coarsening of the eutectic silicon take place in an effort to reduce the surface energy [9]. At the beginning of the solutionizing treatment, the acicular silicon platelets in the unmodified structure begin to break down into smaller fragments and gradually spheroidize. In the case of already semispherical modified silicon, the spheroidization takes place at an early stage. Li et al [43] reported that in modified alloys spheroidization was complete after 1 hr of solution heat treatment while in unmodified alloys coarse needles of Si were visible even after 12 hours. A schematic of the spheroidization and coarsening process is shown in Figure 2.4.

The coarsening process is also known as Ostwald ripening, the physical process that involves the growth of larger particles at the expense of smaller ones. Thus, the number of silicon particles will decrease during the solution heat treatment as their average size increases. The coarsening phenomenon involves mass transfer by the detachment of atoms from smaller particles, and their diffusion through the matrix to join onto larger particles. A concentration gradient is the driving force for this process. Smaller particles have a relatively small radius of curvature (i.e. higher concentration), on the other hand, larger particles have larger radius of curvature (i.e. lower concentration). It is this concentration difference which will cause the preferential dissolution of smaller particles [44].

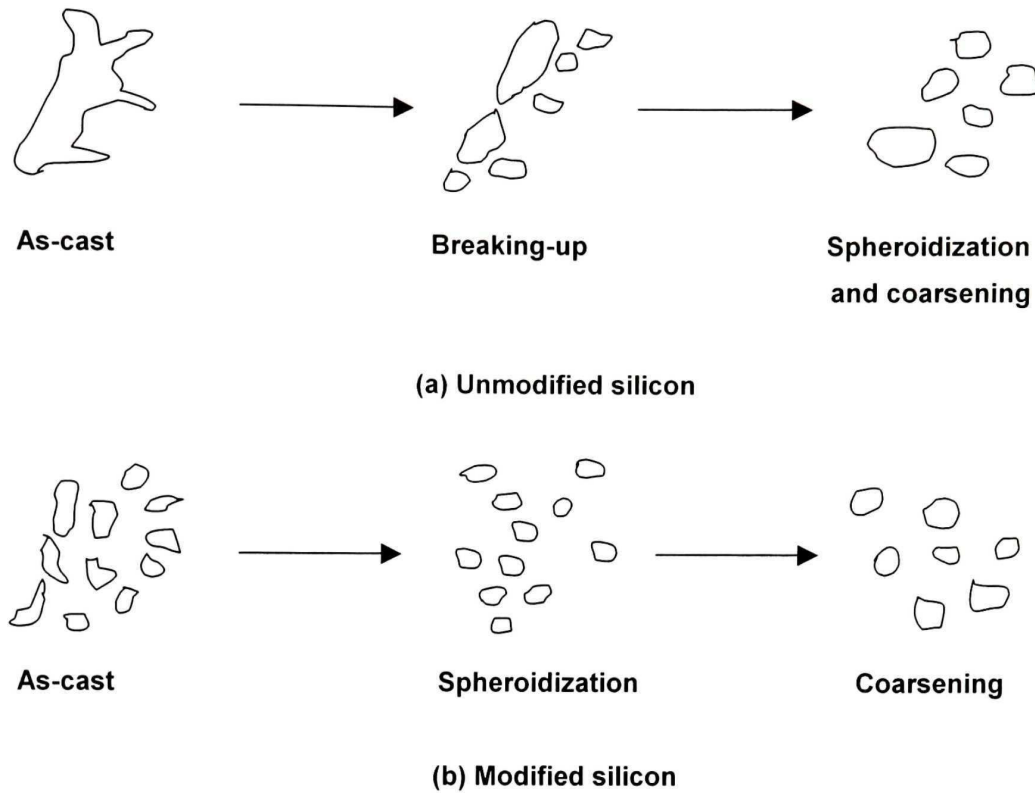


Figure 2.4 Schematic characterization of the three stages of spheroidization and coarsening of the eutectic silicon phase [9]

According to the Lifshitz, Slyozov and Wagner model (LSW) [43,44], the coarsening rate can be expressed using the following relationship:

$$r_t^3 - r_0^3 = 8D\sigma V_m^2 C_{\alpha}(\infty) t/9RT \quad (1)$$

Where:

- r_t is the average particle radius at time t ,
- r_0 is the average particle radius at time $t = 0$,
- D is the volume diffusion coefficient of the diffusing species,
- σ is the specific surface free energy of the particle/matrix interface,
- V_m is the molar volume of the precipitate,
- $C_{\alpha}(\infty)$ is the equilibrium solute content in the matrix,
- R is the gas constant,
- T is the temperature in K.

2.3.2 Quenching

Quenching is the rapid cooling process following the solution heat treatment used in order to produce a supersaturated solid solution at room temperature. Quenching allows the maximum level of solute supersaturation and quenched-in vacancies to be retained in the aluminum matrix.

A balance must be maintained between fast quenching (to retain most of the hardening elements and compounds) and minimizing residual stress and distortion in the castings being quenched. Slow quenching rates will result in precipitation during quenching, localized overaging, loss of grain boundaries corrosion resistance and in some cases, a lower response to the age hardening process [45,46]. Quenching too fast creates high thermal gradients and thermal stresses, which cause distortion [47]. The dependence of the mechanical properties on the quench rate is called quench sensitivity [48-49].

According to ASTM standard B-917 [28] during quenching it is important that cooling proceeds rapidly through the 400°C to 260°C range in order to avoid the type of premature precipitation detrimental to tensile properties and corrosion resistance. For casting alloys the quench delay should not exceed 45 s. In the case of the A356 alloy, sand casting or permanent mould, it is necessary to reduce quench delay time. ASTM standard B-597 [50] specified a maximum quench delay of 10 s.

In Al-Si-Mg alloys the effect of the quenching will depend on the quantity of Si and Mg present in the aluminum matrix [41]. For instance, Shivkumar et al [34] found, using microprobe analysis, that as much as 1.3 % Si can be retained in the solid solution of the aluminum matrix after an Al-7% Si-0.4 Mg alloy had been solution heat treated at 540°C and water quenched. This leaves around 1% of Si in excess of the amount needed to form stoichiometric Mg_2Si . A higher Mg content in the alloy will reduce the excess silicon content of the matrix. It has

been found experimentally [51] that the silicon diffuses from the solid solution towards the existing eutectic silicon. Thus, the amount of excess silicon can be reduced with lower quenching rates.

2.3.3 Preaging

Preaging can be considered as the practically unavoidable delay between solution treatment and artificial aging [52]. When the temperature during preaging is equal to room temperature the process is known as natural aging. The effect of the preaging on the properties of aluminum alloys will depend on the chemical composition, storage time and temperature.

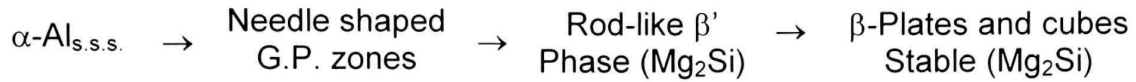
It has been found that Al-Si-Mg alloys experience a decrease in the electrical conductivity during the first hours of natural aging until reaching a plateau at approximately 3 hours [1,53]. This may be caused by the formation of coherent small atomic clusters that lead to a distortion of the crystal lattice and the generation of coherency strains that result in lower conductivity.

A very pronounced reduction of the yield strength (YS), ultimate tensile strength (UTS), and hardness of the Al-Si-Mg alloys has been reported [54] during the 3 first hours of natural aging. The decrease in strength is usually accompanied by an increase in ductility.

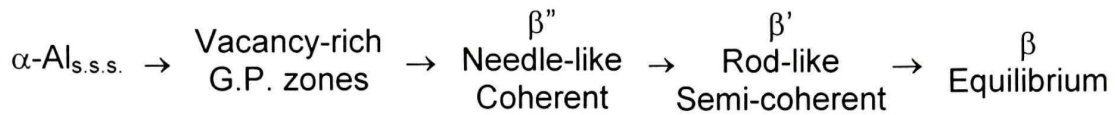
2.3.4 Precipitation Hardening

Precipitation strengthening is a process that involves a particulate dispersion of obstacles to dislocation movements; this is achieved by a second phase precipitation process. The degree of strengthening obtained will depend on the metallic system involved, the volume fraction and size of the particles, and the nature of the interaction of the particles with the dislocations [55].

In the case of Al-Si-Mg alloys the purpose of this treatment is to precipitate out of solution the hardening particles (Mg_2Si) that were dissolved during the solution heat treatment. The precipitation sequence in these alloys is as follows [56,57]:



Other authors [58] consider the precipitation sequence to be as follows:



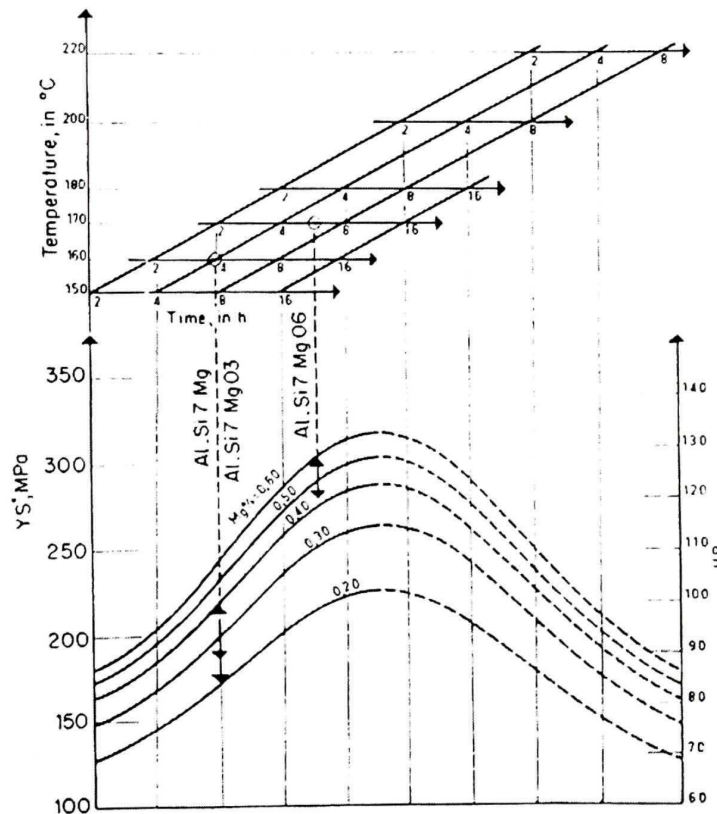
Precipitation begins with the formation of spherical Guinier Preston (G.P) zones. Soon, these zones elongate in the [100] matrix direction and assume a needle shape [59]. These needles grow with time to rods and eventually to platelets.

The aging temperature and time are the two main parameters used to control precipitation hardening in the Al-Si-Mg system. However, as discussed previously, natural aging time may also influence it. Increase of artificial aging temperature will result in improved ultimate tensile strength, yield strength and hardness, however, the elongation will be sacrificed. This effect is shown in Table 2.1 [54]. It is important to bear in mind that compromises have to be made in order to reach the desired combination of mechanical properties.

Table 2.1. Effect of aging temperature on tensile properties of sand cast and permanent mould test bars of alloy 356 [54]

Aging Temperature (°C)	Sand cast bars			Permanent mould test bars		
	UTS	YS	E	UTS	YS	E
	(MPa)	(MPa)	(%)	(MPa)	(MPa)	(%)
3 h at 155°C	295	214	6.5	303	207	14.6
3 h at 165°C	298	233	5.0	327	241	11.8
3 h at 175°C	301	247	4.0	331	262	10.6

Figure 2.5 [60] shows the influence of artificial aging time and temperature as a function of the Mg content on probable yield strength (YS) and hardness. It can be seen, for instance, that for an alloy containing 0.3 % Mg and aged at 160 °C the maximum hardness and YS is reached at approximately 20 hours. Longer aging times will result in lower strength properties due to the overaging of the Mg_2Si .



2.3.5 Precipitation Hardening Mechanisms

In order to understand the principles of precipitation hardening it is important to realize that when a mobile dislocation in a stressed metal meets a second phase particle, a balance of different forces appears as shown schematically in Figure 2.6.

Considering the force balance between the tension line (T) of the dislocation and the resistance force (F) of the particle it can be concluded that:

$$F = 2T \sin \theta \quad (2)$$

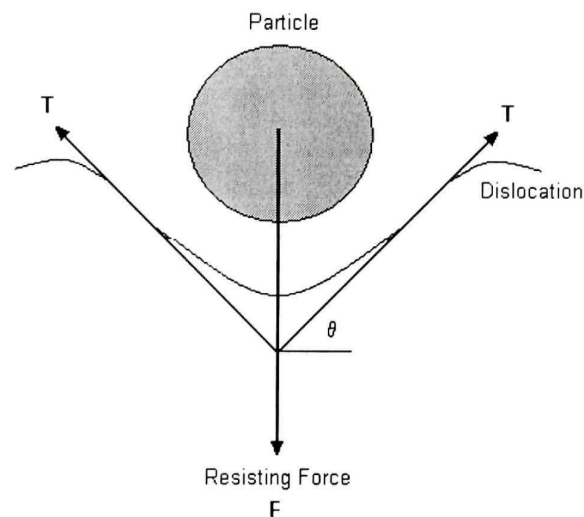


Figure 2.6 Balance of forces acting during particle resistance to dislocation movement [55]

Analyzing the force balance it can be seen that as F increases, the bowing of the dislocation increases (θ increases). If the particle is hard, such that the resistance force F is greater than $2T$, then the dislocation will bypass the particle either by Orowan looping or cross-slip, and the particle will remain unchanged. In Figure 2.7, it can be seen that the particle strength becomes irrelevant because the bypass operation depends only on the interparticle spacing. On the other hand, if the strength of the particle is such that the maximum resistance force is

attained before $\theta=90^\circ$, then the particle will be sheared and the dislocation will pass through the particle as shown in Figure 2.8.

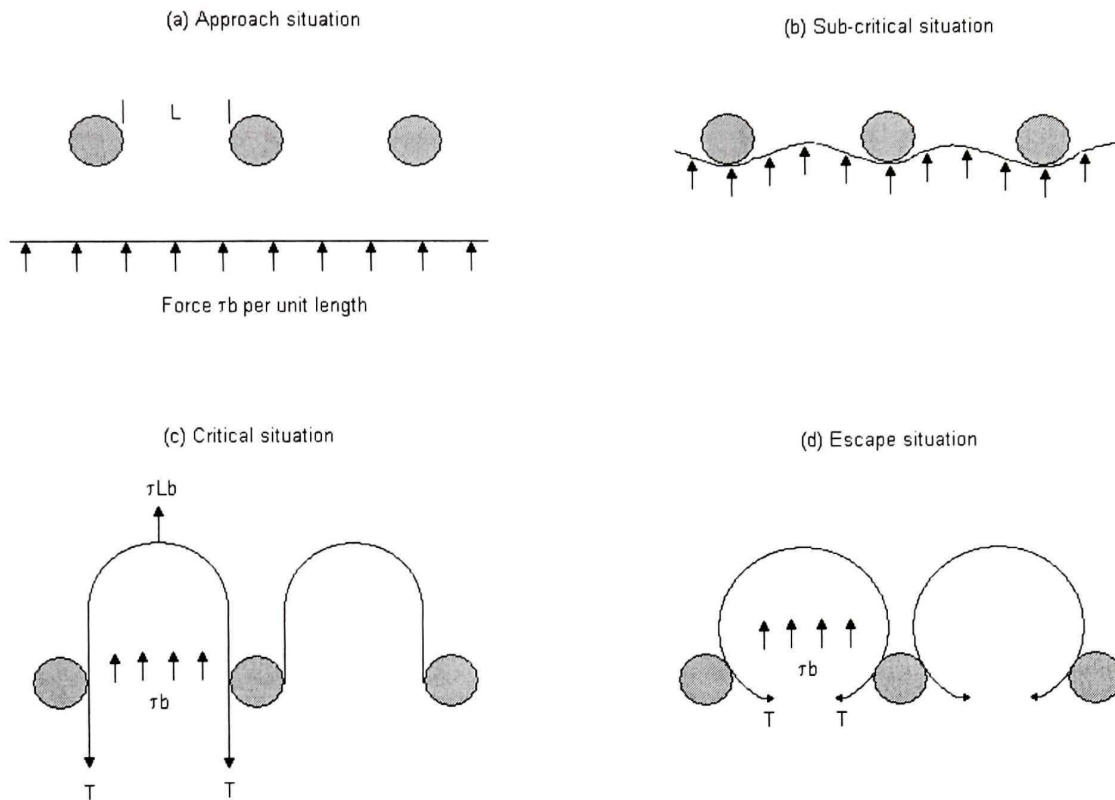


Figure 2.7. Dislocation meets hard undeformable second phase particles: dislocation release at higher stresses may occur by Orowan looping or cross-slip

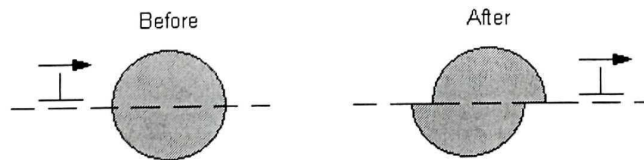


Figure 2.8. Dislocation motion may continue through second phase particles (cutting)

2.3.6 Electrical Conductivity and Heat Treatment

It has been shown that electrical conductivity [61-63] or resistivity techniques [64,65] can be employed as a non-destructive method to monitor the microstructure, modification process and heat treatment of Al-Si-Mg alloys in which the eutectic silicon undergoes changes in form and size. Moreover, electrical conductivity measurements have been successfully used in the evaluation of natural and artificial aging during heat treatment [1,53].

It has been also found that strontium modified Al-Si and Al-Si-Mg alloys exhibit a larger electrical conductivity [1,62,63]. This is related to the differences in the eutectic silicon shape, since the electrons flow more easily through the eutectic regions as shown in Figure 2.9.

In the case of solution heat treatment, electrical conductivity measurements can be related to the changes in silicon morphology as shown by Mulazimoglu [9]. He also found that precipitation of Mg_2Si during artificial aging results in higher electrical conductivity. This can be explained by the precipitation of Mg_2Si and the consequent improvement of the electron flow in the aluminum matrix.

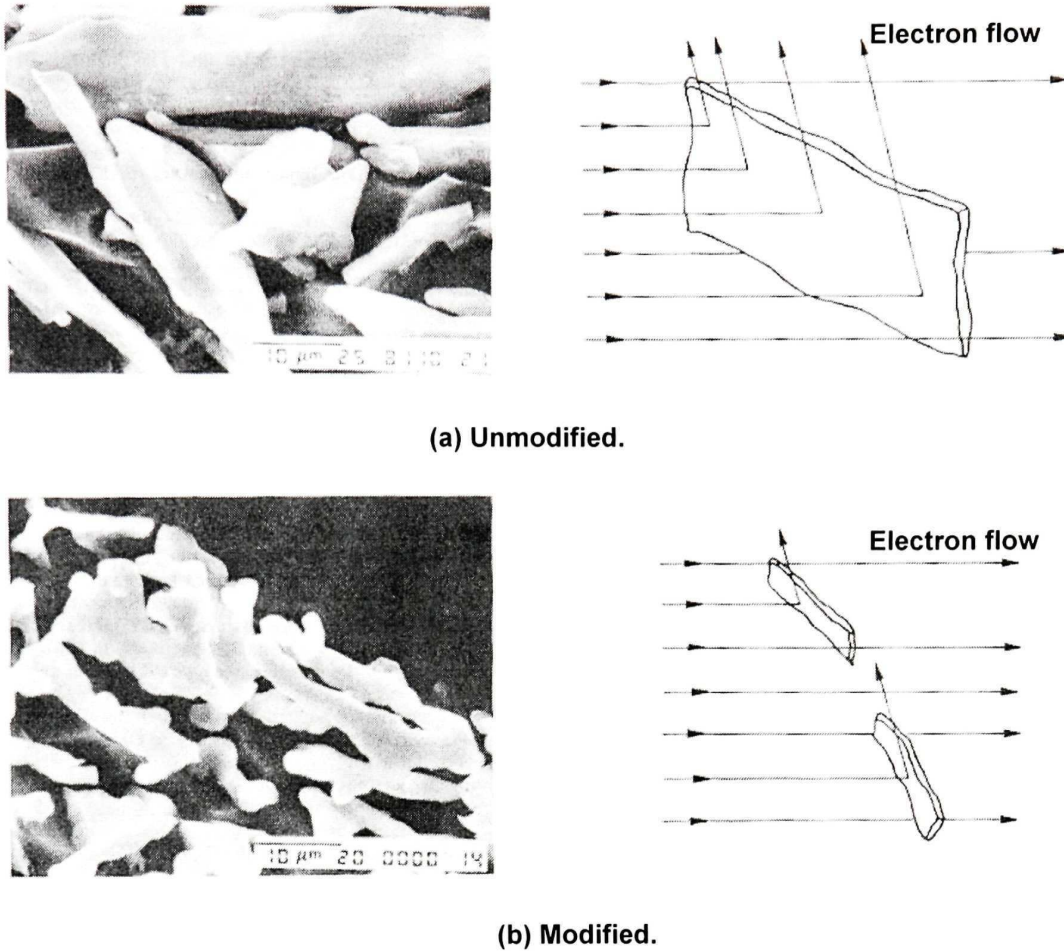


Figure 2.9. SEM micrographs of deep-etched Al-Si eutectic alloys and the schematic representation of the electron collision with the eutectic silicon phase [62]

2.3.7 Study of Mg_2Si Precipitation Using Transmission Electron Microscopy

Many authors have studied the precipitation of the Mg_2Si in wrought alloys by transmission electron microscopy (TEM) [52,66-75], but little research work involving TEM in Al-Si-Mg casting alloys has been done to date [49,76,77].

TEM research provides information about the crystal structure, size, shape and distribution of the precipitates that are necessary parameters to understand

precipitation hardening. One of the disadvantages of TEM studies is that the volume that can be analyzed per sample is small, around $10^{-20} - 10^{-18} \text{ m}^3$ [75].

A TEM study by Jacobs [67] on the morphology and crystal structure of the metastable Mg_2Si precipitates formed during aging of an Al-1.2% Mg_2Si revealed that the first precipitates formed were needle-shaped G.P. zones, aligned along $\langle 100 \rangle$ directions of the matrix, which were coherent with the matrix along their major axes. He also found that as aging continues, these zones become rod-shaped precipitates with a hexagonal unit cell [$a=0.705 \pm 0.005 \text{ nm}$; $c=0.405 \pm 0.001 \text{ nm}$]. Finally, the rods transform into platelets of the equilibrium phase (Mg_2Si) which have the same crystal structure as bulk magnesium silicide (Mg_2Si), that is, a face-centered cubic structure with $a=0.639 \text{ nm}$.

Matsuda et al. [73] showed that the morphology of the G.P zones of an Al-1.6% Mg_2Si is that of a fine plate of a monolayer in thickness, 2.5 nm in width, and less than 30 nm in length. However, other authors [74,78,79] consider the G.P zones as spherical clusters with unknown structure. Moreover, they consider the needle shaped precipitates as the β'' precipitates, which possess a monoclinic structure. Lynch et al. [80], also determined a monoclinic structure for β'' ($a = 0.30 \text{ nm}$, $b = 0.33 \text{ nm}$, $c = 0.40 \text{ nm}$, $\alpha = \beta = 90 \text{ deg}$, and $\gamma = 78 \text{ deg}$). All researchers agree that hardening of the AlMgSi alloys is due to the precipitation of needle-like G.P zones or β'' .

Andersen [69], found that the larger the density and volume percent β'' the larger the yield strength. This conclusion appears to be independent of the size of the precipitates in a range of 46 to 70 nm.

Chapter 3

EXPERIMENTAL

3.1 General

The experimental procedure used in this work was designed to investigate the effect of strontium modification and process variables such as: solution heat treatment time, quenching temperature, natural aging time and artificial aging time and temperature on the properties and precipitation of the Mg_2Si during the heat treatment of Al-7%Si-0.3Mg alloys.

The experimental work needed to complete this research work can be divided into the following main areas:

- Fabrication and heat treatment of the samples.
- Electrical conductivity and microhardness measurements.
- Transmission electron microscopy.
- Measurement of tensile properties.

3.2. Sample Fabrication

Cast bars used to obtain the coupons, which were used for further heat treatment and evaluation, and tensile test bars for the determination of the mechanical properties were produced. The manner in which they were produced is described in the following sections.

3.2.1 Materials

The specimens were all made from commercial A356 alloy. This alloy is widely used in the automotive, aerospace and military industry for its high achievable strength, castability, pressure tightness, low weight and the fact that it can be machined and welded [1]. The chemical composition of the unmodified A356 alloy used in this work is given in table 3.1.

Table 3.1 Chemical composition of A356 alloy	
Elements	Wt%
Si	7.20
Mg	0.35
Fe	0.13
Cu	0.02
Mn	0.007
Zn	0.00
Ti	0.15
Al	Balance

Strontium was introduced into the melt in the form of a master alloy of Al-10%Sr. The amount of master alloy added to the melt was the quantity necessary to achieve ~250 ppm of strontium in the cast bars. Pre-modified A356 alloy containing 150 ppm of strontium was used to produce the tensile test bars used to evaluate the effect of natural aging.

3.2.2 Melting and Modification

Two different melts were prepared. The first one was prepared by melting down unmodified A356 alloy in an induction furnace at 725°C. Then, the molten metal was transferred to an electric resistance furnace preheated to 735°C. The molten metal was then degassed with ultra high purity argon using a silicon

carbide (SiC) lance. The quantity of hydrogen before, after degassing and during pouring was measured using the Alscan method and was also evaluated with the reduced pressure test (RPT). The quantity of hydrogen and quality of the visual appearance of reduced pressure test samples of the degassed unmodified alloy are seen in Table 3.2 and Figure 3.1.

Table 3.2 Hydrogen content measured with Alscan before (1), during (2-3) and after degassing (4) and during casting (5) of unmodified A356 alloy

Sample#	ml/100g Al (STP)
1 (Before degassing)	0.209
2 (During degassing)	0.181
3 (During degassing)	0.125
4 (After degassing)	0.095
5 (During casting)	0.093

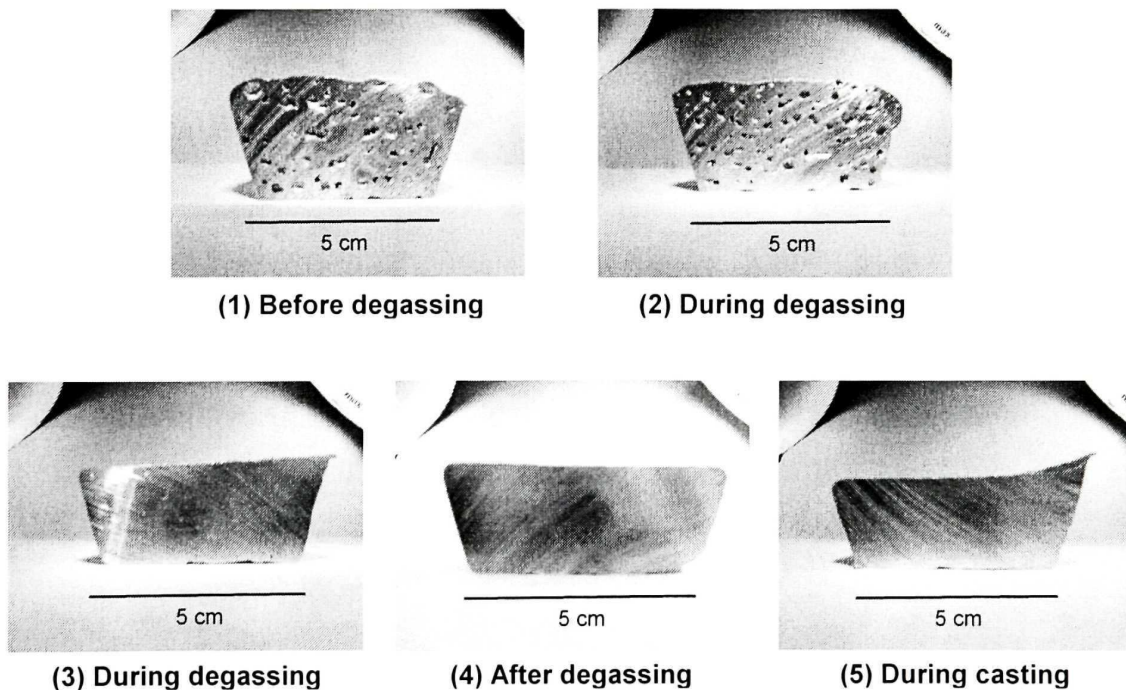


Figure 3.1. RPT samples taken before (1), during (2-3), after degassing (4) and during casting (5) of unmodified A356 alloy

Once casting of the unmodified metal was completed Al-10%Sr master alloy was added to the melt. Table 3.3 shows the chemical composition of the modified melt after modification and at the end of pouring. As with the unmodified metal, the quantification of hydrogen by Alscan and RPT was carried out. The results are given in table 3.4 and Figure 3.2 respectively.

Table 3.3 Chemical composition of the A356 alloy after modification and at the end of pouring

Elements	After modification	End of pouring
	Wt. %	Wt. %
Si	7.17	7.17
Mg	0.35	0.33
Fe	0.13	0.13
Cu	0.02	0.02
Mn	0.007	0.007
Zn	0.00	0.00
Ti	0.15	0.15
Sr	0.027	0.025
Al	Balance	Balance

Table 3.4 Hydrogen content measured with Alscan before (1) and after degassing (2) and during casting (3-4) of modified A356 alloy

Sample#	ml/100g Al (STP)
1 (Before degassing)	0.097
2 (After degassing)	0.093
3 (During casting)	0.097
4 (During casting)	0.108

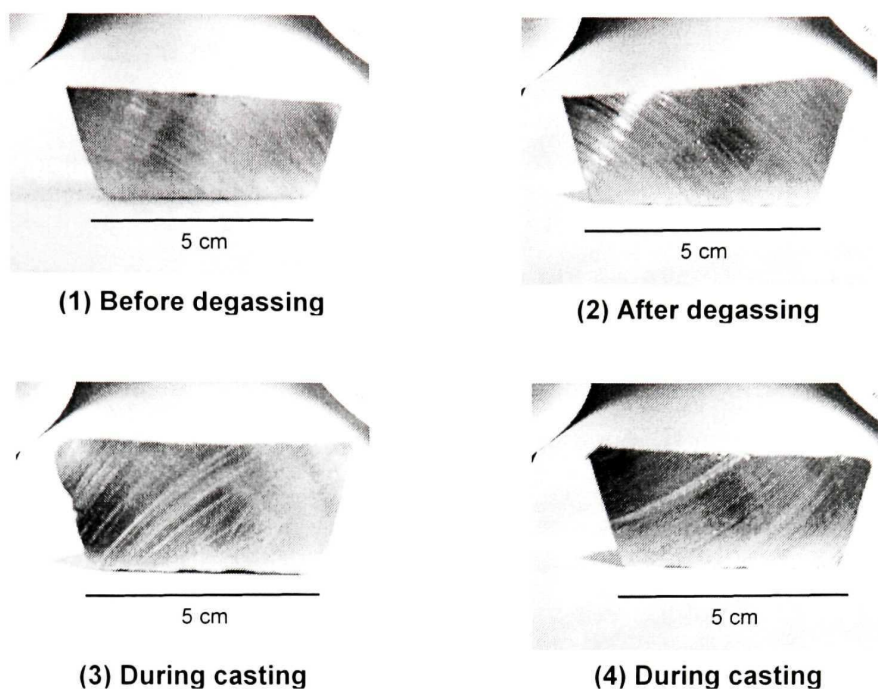


Figure 3.2 RPT samples taken before (1) and after degassing (2) and during casting (3-4) of modified A356 alloy.

The second melt was prepared in an industrial scale gas-fired furnace and was then degassed with high purity argon. Pre-modified alloy of the chemical composition given in Table 3.5 was used.

Table 3.5 Chemical composition of pre-modified A356 alloy

Elements	Wt%
Si	7.28
Mg	0.33
Fe	0.098
Cu	0.008
Mn	0.001
Zn	0.00
Ti	0.107
Sr	0.015
Al	Balance

3.2.3 Sample Production

Cylindrical bars (2.54 cm diameter X 20 cm length) were prepared by pouring metal from the first melt at 715 °C into a gray-iron mould pre-heated to 200°C. The mould was pre-heated in order to obtain microstructures similar to those obtained by the permanent mould process. The bars were then sectioned along their length into 9 mm thick coupons and evaluated in order to choose the most suitable and homogeneous areas for further work. It was found that 10 cm of the central portion of the bar were the most homogeneous; this is shown in Figure 3.3. The average dendrite arm spacing, microhardness and electrical conductivity of 15 samples chosen randomly from the central section of different bars are given in Table 3.6.

VHN= 69.2 ±2.6 DAS= 22.7±1.9 µm VHN= 67.4 ±1.89 2.4 DAS= 21.8±1.4 µm VHN= 66.7 ±2.6 DAS= 20.2±2 µm VHN= 65.1 ±1.9 DAS= 19.9±1.89 µm	20	32.2 % IACS	VHN= 68.6 ±3.1 DAS= 22.8±1.84 µm VHN= 66.1 ±2.6 DAS= 22.3±2.5 µm VHN= 67.9 ±1.9 DAS= 21.2±2.1 µm VHN= 64.3 ±2.4 DAS= 19.9±2.07 µm	20	34.3 % IACS
	19			19	
	18	32.8 % IACS		18	34.4 % IACS
	17			17	
	16	33.0 % IACS		16	34.6 % IACS
	15			15	
	14	33.2 % IACS		14	34.9 % IACS
	13			13	
	12	33.3 % IACS		12	35.2 % IACS
	11			11	
	10	33.4 % IACS		10	35.2 % IACS
	9			9	
	8	33.2 % IACS		8	35.4 % IACS
	7			7	
	6	33.5 % IACS		6	35.4 % IACS
	5			5	
	4	33.6 % IACS		4	35.6 % IACS
	3			3	
	2	34.6 % IACS		2	35.8 % IACS
	1	34.9 % IACS		1	36.1 % IACS
	Unmodified			Modified	

Figure 3.3 Typical values of microhardness, dendrite arm spacing and electrical conductivity found in the bars used in this research work

All the samples used further in this research work were selected from the central section of the bars, and the measurement of the electrical conductivity was always used as a quality control parameter considering only the samples presenting conductivities values of 33.2 ± 0.2 %IACS and 35.2 ± 0.2 %IACS for the unmodified and modified samples respectively.

Table 3.6. Average parameters of coupons used for heat treatment

Parameter	Unmodified	Modified
Dendrite arm spacing (μm)	20.8 ± 0.32	20.0 ± 0.43
Electrical conductivity (%IACS)	33.4 ± 0.12	35.3 ± 0.10
Microhardness ($\text{g}/(\mu\text{m}^2)$)	65.9 ± 0.7	67.8 ± 1.4

The tensile test bars used for experiments at high artificial aging temperature were produced in a permanent mould according to ASTM standard B109, using the alloy from the second melt. The pouring temperature was 715°C . The temperature of the mold in the sprue and gauge length areas was monitored and controlled as shown in Figure 3.4 in order to obtain good metal feeding and a controlled microstructure. The second set of tensile test bars was cast with material from the melt prepared in an industrial furnace using a permanent mould according to ASTM B108.

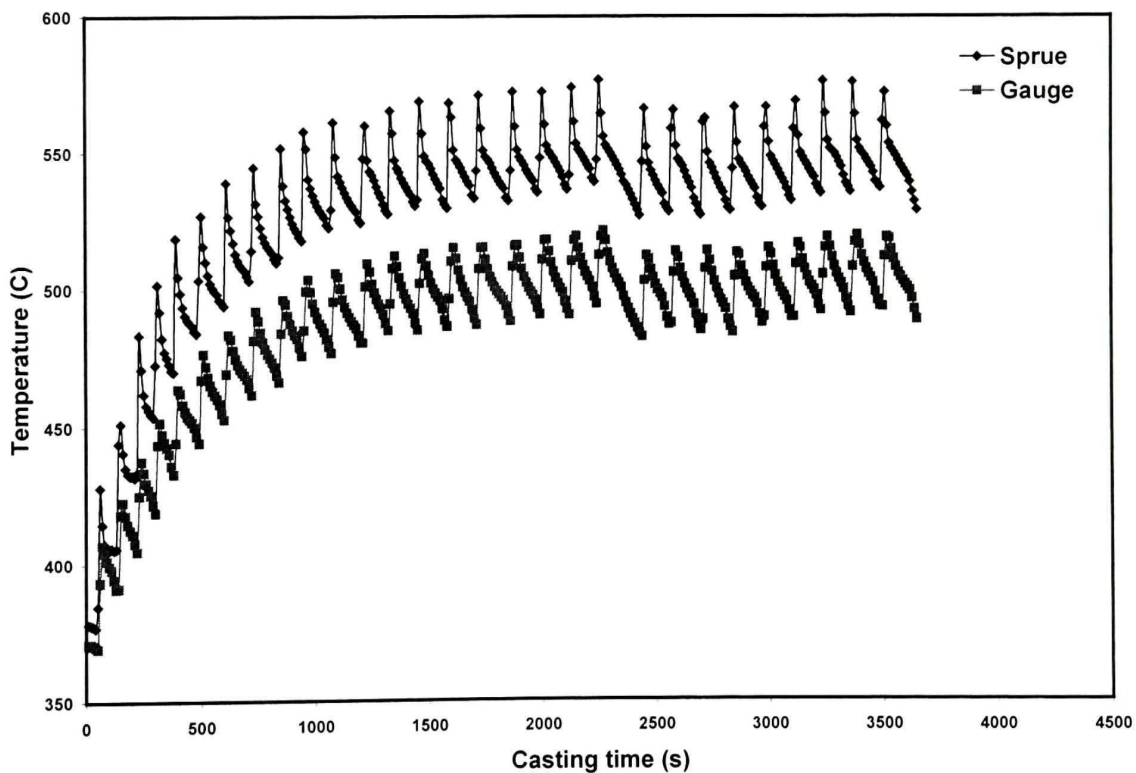


Figure 3.4 Temperature of the mold in the sprue and gauge length regions during casting of tensile test bars

3.3 Heat Treatment

It is a common practice to heat-treat the A356 alloy in order to improve its mechanical properties. In this research work, the effect of the different heat treatment parameters such as solution heat treatment time, quenching temperatures, natural aging time, artificial aging time and temperature and strontium modification on the properties and Mg_2Si precipitation of A356 were evaluated. All the heat treatments of the unmodified and modified coupons were carried out in an electric resistance furnace (Lindberg Blue M BF 51732). The calibration of the furnace temperature was carried out with 3 thermocouples at different places in the furnace, and at different temperatures. The results obtained from the calibration indicate a maximum variation of $\pm 2^\circ C$ at any of the treatment temperatures used in the zone where the heat treatments took place.

For each given condition, except those in which the solutionizing time and quenching temperature were the variables, all the samples were solution heat treated and quenched at the same time leaving only the other conditions, such as natural and artificial aging time and temperature, as variables. At least three coupons were heat treated in each condition.

The temperature selected to carry out all the solution heat treatments was $540^\circ C$ while the artificial aging temperatures used were $155^\circ C$, $170^\circ C$ and $236^\circ C$. ASTM standards [28] recommend for the A356 alloy a solution heat treatment of $540^\circ C$ for 4 to 12 hours. Other organizations and authors [27,29-33] consider a temperature of $540^\circ C$ and a time of 4 hours as the optimum. In this research work a solution heat treatment time of 4 hours was mainly used to evaluate the Mg_2Si precipitation. Evaluations at 2 and 6 hours of solutionizing time were also done. It is very important to mention that the evaluation of the properties and Mg_2Si precipitation in this work is focused on the T6 condition (solution heat treated, quenched and artificially aged).

Quenching was performed by immersion of the samples in water with a quench delay no longer than 6 seconds. The water bath used was a modified domestic hot-water tank. The temperature, depending on the quenching condition, was set and controlled to 22°C, 50°C or 70°C. In the case of the 0°C quenching, ice was added to the water until the temperature was reached and had stabilized. To evaluate the effect of natural aging on the properties and precipitation of the A356 alloy, some coupons were left resting 6, 12 and 20 hours at room temperature. The temperatures used for artificial aging of the coupons were 155°C, 170°C and 236°C, as mentioned above, for times of 3, 6, and 9 hours in the case of the first two temperatures, and for 0, 0.4, 0.8, 1.5, 3, 6 and 9 hours in the case of the last. In all the heat treatment stages, the soaking time did not begin to be counted until the furnace reached the desired temperature. A summary of the heat treatment schedules of the coupons is given in Table 3.7.

For the tensile test bars only a few heat treatment experiments, considered as critical were done. A summary of those heat treatments is shown in Table 3.8. All the heat treatments were performed in a convection oven because of its larger capacity and better temperature control.

Table 3.7. Heat treatment schedules of the coupons*

Cycle	Solution treatment		Quenching		Artificial aging	
	Temperature (°C)	Time (h)	temperature (°C)	aging (h)	Temperature (°C)	Time (h)
A	540	2	22	0	155	3,6,9
B	540	4	22	0	155	3,6,9
C	540	6	22	0	155	3,6,9
D	540	4	0	0	155	6
E	540	4	22	0	155	6
F	540	4	50	0	155	6

Table 3.7. Heat treatment schedules of the coupons* (Continuation)

Cycle	Solution treatment		Quenching temperature (°C)	Natural aging (h)	Artificial aging	
	Temperature	Time			Temperature	Time
	(°C)	(h)			(°C)	(h)
G	540	4	70	0	155	6
H	540	4	Forced air	0	155	6
I	540	4	22	0	155	6
J	540	4	22	6	155	6
K	540	4	22	12	155	6
L	540	4	22	20	155	6
M	540	4	22	0	170	6
N	540	4	22	6	170	6
O	540	4	22	12	170	6
P	540	4	22	20	170	6
Q	540	4	22	0	185	6
R	540	4	22	6	185	6
S	540	4	22	12	185	6
T	540	4	22	20	185	6
U	540	4	22	0	155	3,6,9
V	540	4	22	0	170	3,6,9
W	540	4	22	0	236	0,0.4,0.8, 1.5,3,6,9

* Unmodified and modified

Table 3.8. Heat treatment schedules for the tensile test bars

Cycle	Solution treatment		Quenching temperature (°C)	Natural aging (h)	Artificial aging	
	Temperature	Time			Temperature	Time
	(°C)	(h)			(°C)	(h)
A*	540	4	22	0	155	6
B*	540	4	22	6	155	6
C*	540	4	22	12	155	6
D*	540	4	22	20	155	6
E*	540	4	22	0	170	6
F*	540	4	22	6	170	6
G*	540	4	22	12	170	6
H*	540	4	22	20	170	6
I**	540	4	22	18.75	236	0.16
J**	540	4	22	18.75	236	0.32
K**	540	4	22	18.75	236	0.50

*Modified

** Unmodified and modified

3.4 Electrical Conductivity Measurements

Electrical conductivity measurements at room temperature of the coupons from the as-cast through to the final T6 condition were performed using a K.J Law Model 4900C electrical conductivity meter. This instrument measures the conductivity by an eddy-current technique and displays it as a percentage of the International Annealed Copper Standard (% IACS); with 100% IACS defined to be $1.7241 \mu\Omega\text{cm}$ at 20°C [81]. Moreover, this instrument features temperature compensation to adjust the reading to a standard lab condition of 20°C and an effective constant penetration depth of 1.27 mm [82]. According to ASTM standard E 1004-91 [83], the accuracy of the results obtained from this test can be affected by the following parameters:

- Quality or degree of coupling between the probe coil and the metal,
- Electromagnetic field penetration through thin test objects,
- Discontinuities or inhomogeneities in the metal near the position of the probe coil,
- Variations in the temperature of the probe coil, test object, and standards,
- Surface conditions of the test object,
- Test object geometry.

To avoid these interferences in the electrical conductivity measurements, the following actions were taken:

- The area of the coupons to be examined was ground with SiC paper #120, #320, #400 and #600 to obtain a flat smooth surface,
- The thickness of the coupons was at least the same as the standards,
- Coupons showing visible imperfections were rejected,
- It was ensured that the standards, instruments and coupons were at the same temperature before any measurements were taken,
- The instrument was calibrated against three known aluminum standards and compensated for temperature,
- Touching of the standards and coupons during the measurements was avoided,
- The edge effect was avoided by always taking the measurements at the center of the coupons,
- The calibration of the instrument was verified at the end of the testing of each sample.

The accuracy of the instrument was verified against standards. The variations are seen in Table 3.9 and Figure 3.5. The results obtained indicate that the error of the electrical conductivity measurements performed in this research work is small.

Table 3.9 Variations of the measurements on standards of electrical conductivity

Standard Value	Average Measured	Standard Deviation	% IACS deviated from standards
29.9	29.76	± 0.05	-0.14
44.9	44.98	± 0.06	+0.08
45.3	45.37	± 0.08	+0.07

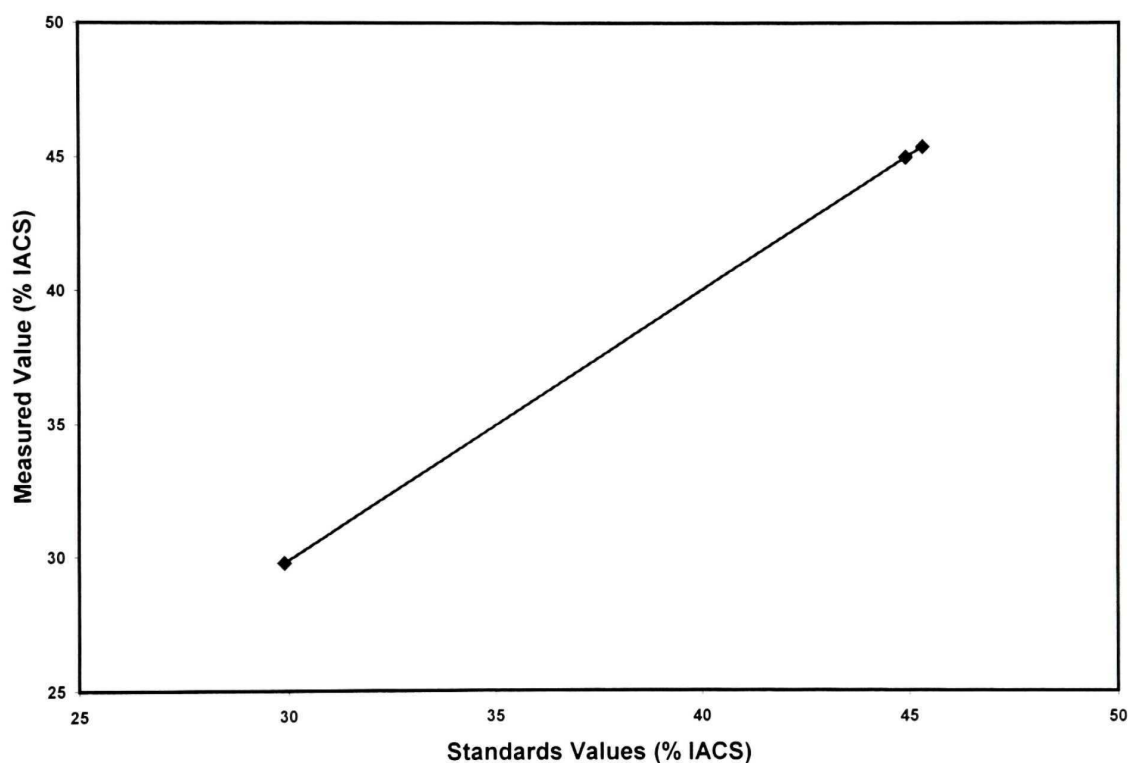


Figure 3.5 Calibration curve of the electrical conductivity meter

In the case of the tensile test bars used to analyze the effect of natural aging a Foerster Sigmatest conductivity measurement instrument was used. The examined area was prepared in the same way as the coupons. The readings of the electrical conductivity measurements between the two instruments were compared and no significant differences were found.

3.5 Microhardness

Microhardness measurements with a Buehler Microhardness tester Model 1600 using a load of 10 g were done to evaluate the changes in the aluminum matrix due to the heat treatment. The diamond indenter used was a Vickers indenter in the shape of a square based pyramid with an angle of 136° between faces.

In order to obtain the best possible results it was necessary to have mirror-like polished coupons. This was obtained with a typical metallographic polishing that consisted of the following:

- Mounting the sample using cold mounting resin (Technovit 40040),
- Grinding with SiC paper #120, #320, #400 and #600,
- Polishing with 5µm and 0.3µm alumina,
- Final polishing with 0.05µm colloidal silica,
- No chemical etching was performed.

Another important consideration was to obtain symmetrical microhardness indentations. This is related to the inclination of the sample with respect to the indenter. For this reason, the mounted coupons were placed in a universal clamp and leveling device when tested. At least ten indentations were performed per coupon. The size of the indentation was measured with a micrometer integrated to a microscope at 400X. The value of the indentation was then transformed to a microhardness value using the following formula [84]:

$$HV = 1.854 \times 10^3 (F/d^2) \quad (1)$$

Where:

F = Load (g),

d = Indentation average length (µm).

The microhardness values reported in this work are the average values of three coupons at the given condition. A typical microhardness indentation is shown in Figure 3.6.

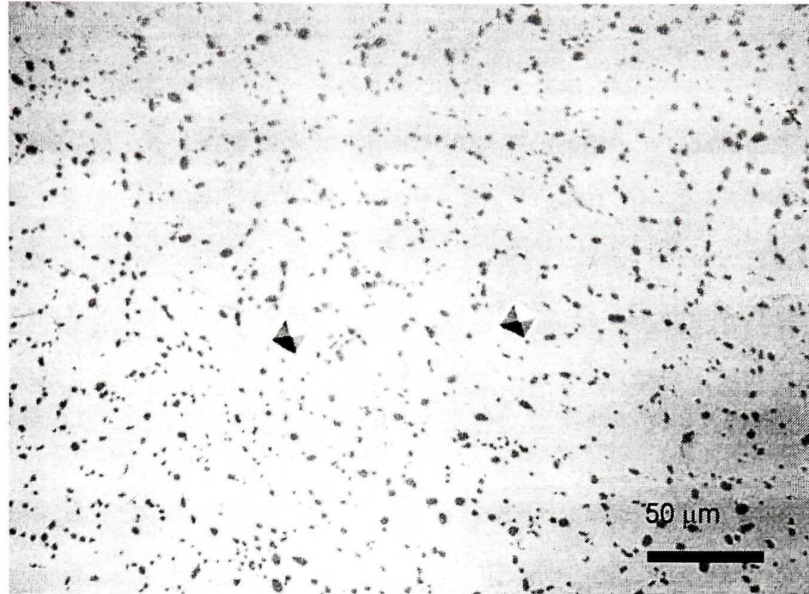


Figure 3.6 Typical microhardness indentation

3.6 TEM Sample Preparation and Observation

Thin foils were prepared from the heat-treated coupons at conditions that were considered of critical importance for the information they could provide to this research work. A summary of the heat treatment conditions of the samples chosen for TEM examination is given in Table 3.10.

The thin foils were prepared by electropolishing using a Struers Tenupol 3 machine, a solution of 15% nitric acid and 85% methanol at -35°C with a current of $\sim 0.15\text{A}$. Figure 3.7 summarizes all the steps taken for their preparation.

Samples were studied in a fully computerized TEM Jeol JEM-2011 at an acceleration voltage of 200KeV. The images were then acquired with a Gatan DualView 300W 1.3K X 1K CCD camera attached to the microscope.

EDS and line scan were made in a Philips CM20 FEG (TEM/STEM) with an Oxford system and INCA software. Dead time of 30%, acquisition time of 50 seconds, built in K factors and process factor of 5 were used.

Table 3.10 Heat treatment condition of the coupons used for TEM examination

Cycle	Solution treatment		Quenching temperature (°C)	Natural aging (h)	Artificial aging	
	Temperature (°C)	Time (h)			Temperature (°C)	Time (h)
A	540	2	22	0	155	6
B	540	4	22	0	155	6
C	540	6	22	0	155	6
D	540	4	0	0	155	6
E*	540	4	0	0	155	6
F	540	4	22	0	155	6
G	540	4	70	0	155	6
H	540	4	Forced air	0	155	6
I	540	4	22	0	155	6
J	540	4	22	12	155	6
K	540	4	22	20	155	6
L	540	4	22	0	170	6
M	540	4	22	12	170	6
N	540	4	22	20	170	6
O	540	4	22	0	236	0
P*	540	4	22	0	236	0
Q	540	4	22	0	236	1.5
R	540	4	22	0	236	6

*Unmodified alloy

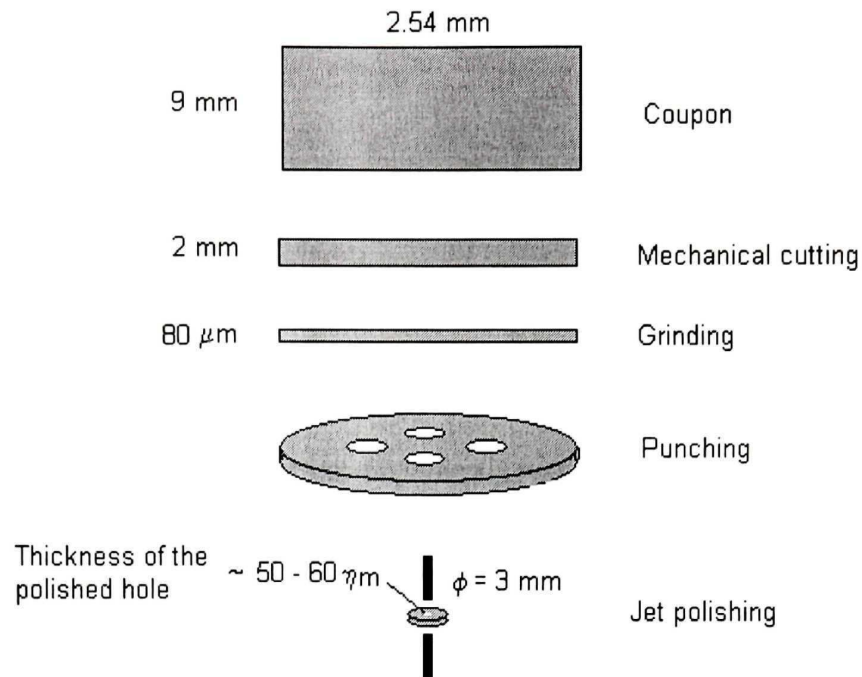


Figure 3.7 Sample preparation of thin foils for TEM

3.7 Tensile Properties Testing

The tensile properties of samples produced at certain heat treatment conditions considered as critical, were evaluated in order to obtain information related to the precipitation of Mg_2Si . In the case of evaluation of the natural aging effect on the properties tests were performed on four samples, whereas, the evaluation of properties at a high artificial temperature aging temperature (236°C) was made on three samples.

All the tests were performed at 23°C using an Instron 8500 automated testing system at a rate of 4 points/second and a crosshead speed of 4 mm/min. Yield strength was calculated at an offset strain of 0.2% while elongation was calculated at rupture with measurements from an extensometer. Typical strain-stress curves are shown in the appendix.

Chapter 4

RESULTS AND DISCUSSION

4.1 General

As described in the introduction, the main objective of this work was to study the influence that strontium modification and heat treatment parameters might have on the properties of heat-treated Al-7%Si-0.3Mg alloy. Therefore, the investigation was divided into four areas that involve the detailed analysis of the following:

- A study of the effect of solution heat treatment times of 2, 4 and 6 hours on the electrical conductivity, microhardness and Mg_2Si precipitation of unmodified and modified coupons artificially aged at 155°C,
- A study of the effect of different quenching conditions on the electrical conductivity, microhardness and Mg_2Si precipitation of unmodified and modified coupons artificially aged at 155°C,
- A study of the effect of different natural aging times on the electrical conductivity, microhardness, Mg_2Si precipitation and tensile properties of unmodified and modified coupons and tensile test bars artificially aged for 6 hours at 155°C and 170°C,
- A study of the effect of different artificial aging times and temperatures on the electrical conductivity, microhardness, Mg_2Si precipitation and tensile properties of unmodified and modified coupons.

The results obtained in these studies are presented and discussed in this chapter.

4.2. Solution Heat Treatment

The effect of different solution heat treatment times on the microstructure of unmodified coupons is shown in Figure 4.1. It can be seen that in the as-cast state the eutectic silicon is present in the form of coarse acicular particles. When the coupon is subjected to 2 hours of solution treatment it is observed that the silicon needles break up into smaller particles. As the treatment continues for 4 and 6 hours the particles begin to spheroidize and to coarsen.

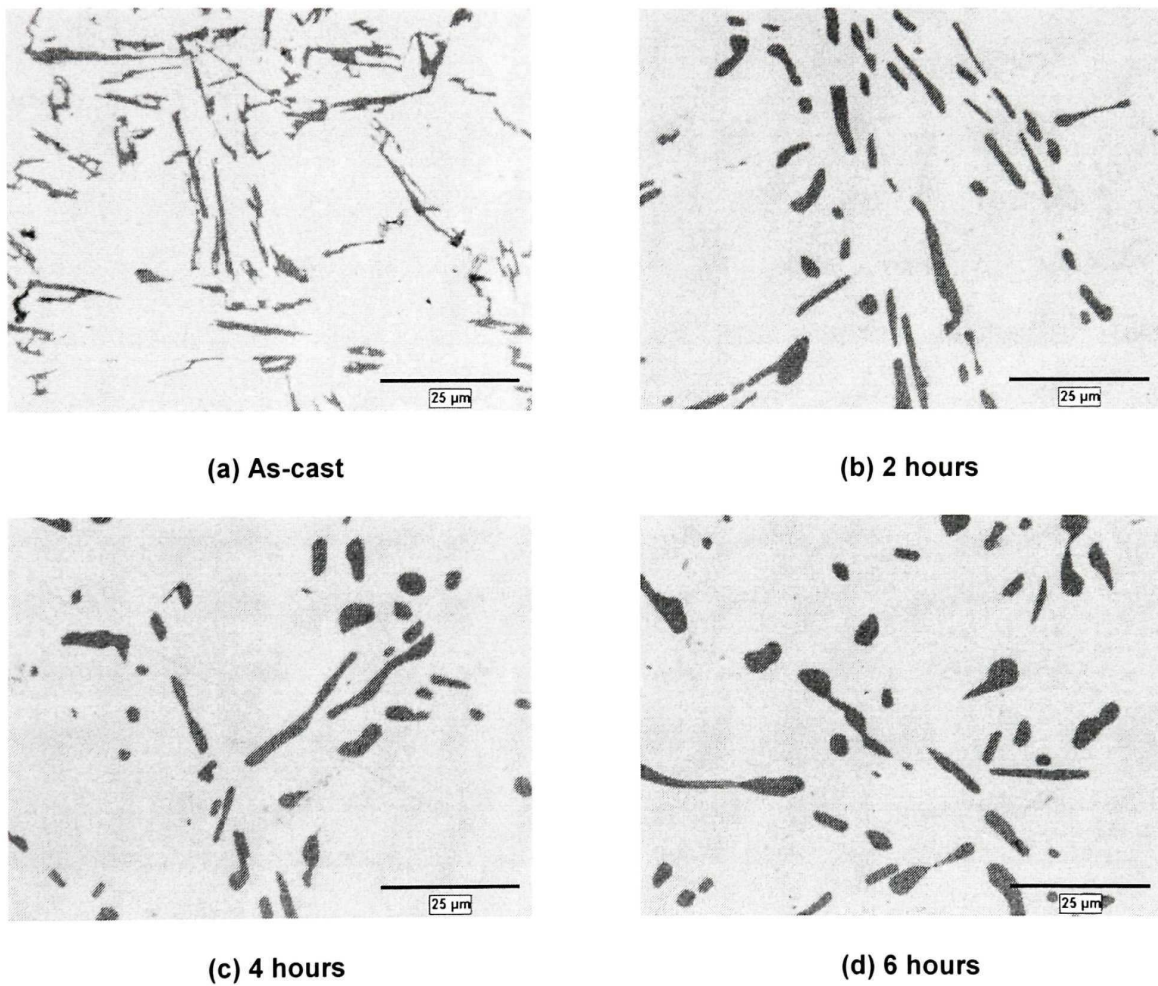


Figure 4.1. Microstructural changes occurring in the unmodified A356 alloy upon solution heat treatment at 540°C

In the case of the modified microstructure, the effect of different solution heat treatment times is shown in Figure 4.2. In this Figure it is seen that the as-cast silicon is present in the form of fine fibrous particles. After 2 hours of treatment, it is observed that a large quantity of the silicon has spheroidized and the coarsening process has begun. As the treatment continues and reaches 4 hours the silicon has completely spheroidized and at 6 hours only further coarsening takes place.

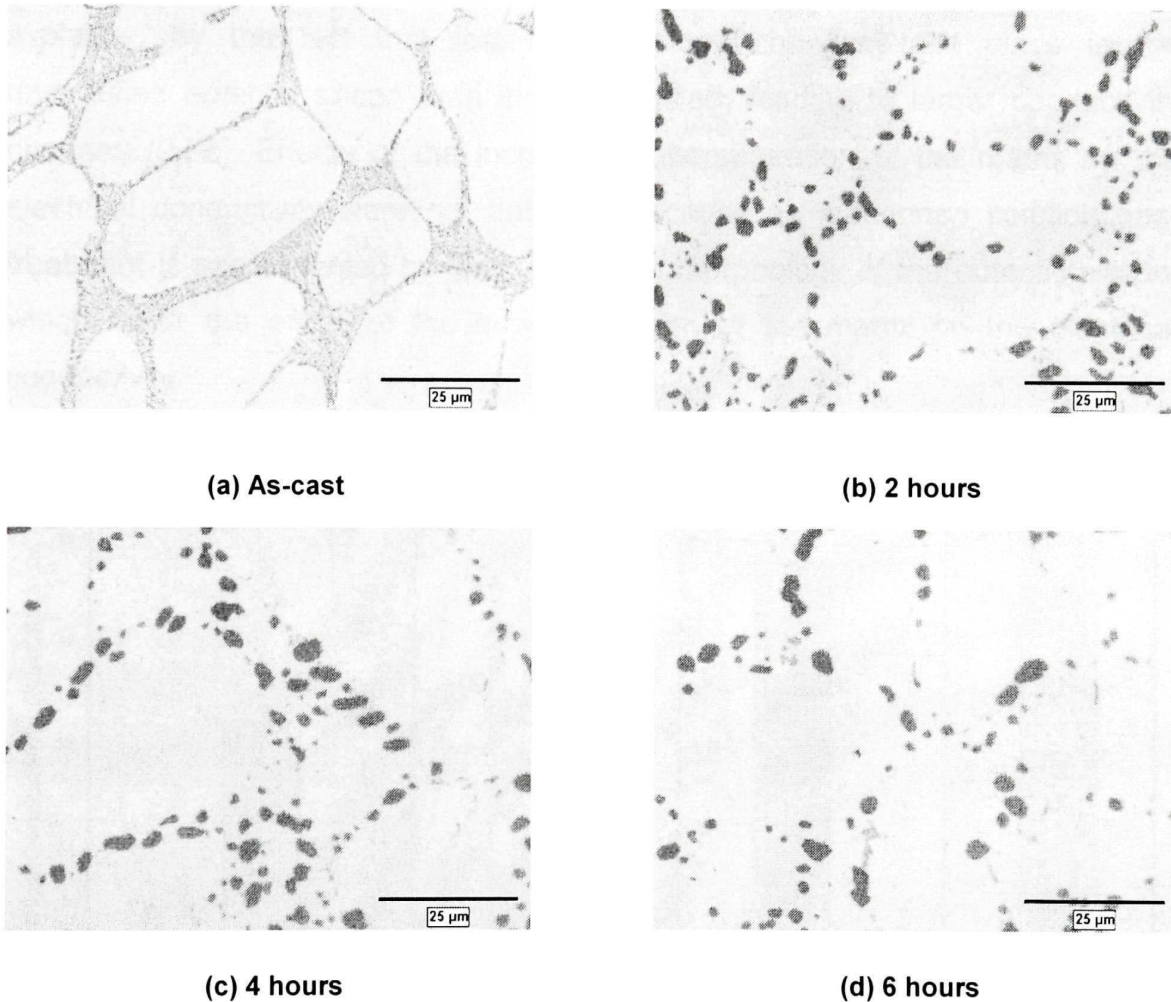


Figure 4.2. Microstructural changes occurring in the modified A356 alloy upon solution treatment at 540°C

The behaviour of the electrical conductivity of unmodified and modified coupons as a function of different solution heat treatment times is shown in

Figure 4.3. It is observed that the modified alloy exhibits a higher electrical conductivity in the as-cast condition than the unmodified alloy. This behaviour has been explained by the fact that the electrons flow more easily through the finer eutectic silicon in the modified alloy than in the coarse acicular silicon present in the unmodified alloys [62]. In Figure 4.3, it is also noted that the change in the electrical conductivity of the unmodified alloy with solution heat treatment is larger than in the modified one, with both alloys having almost the same value after two hours of solution heat treatment time. This has been explained by the fact that larger morphology changes take place in the unmodified eutectic silicon than in the modified, leading to larger conductivity changes [9,62]. Effects of the increased supersaturation of the matrix on the electrical conductivity were not detected because as mentioned solution heat treatment is accompanied by changes in the morphology of the eutectic silicon, which mask the effect of the supersaturation of the matrix on the electrical conductivity.

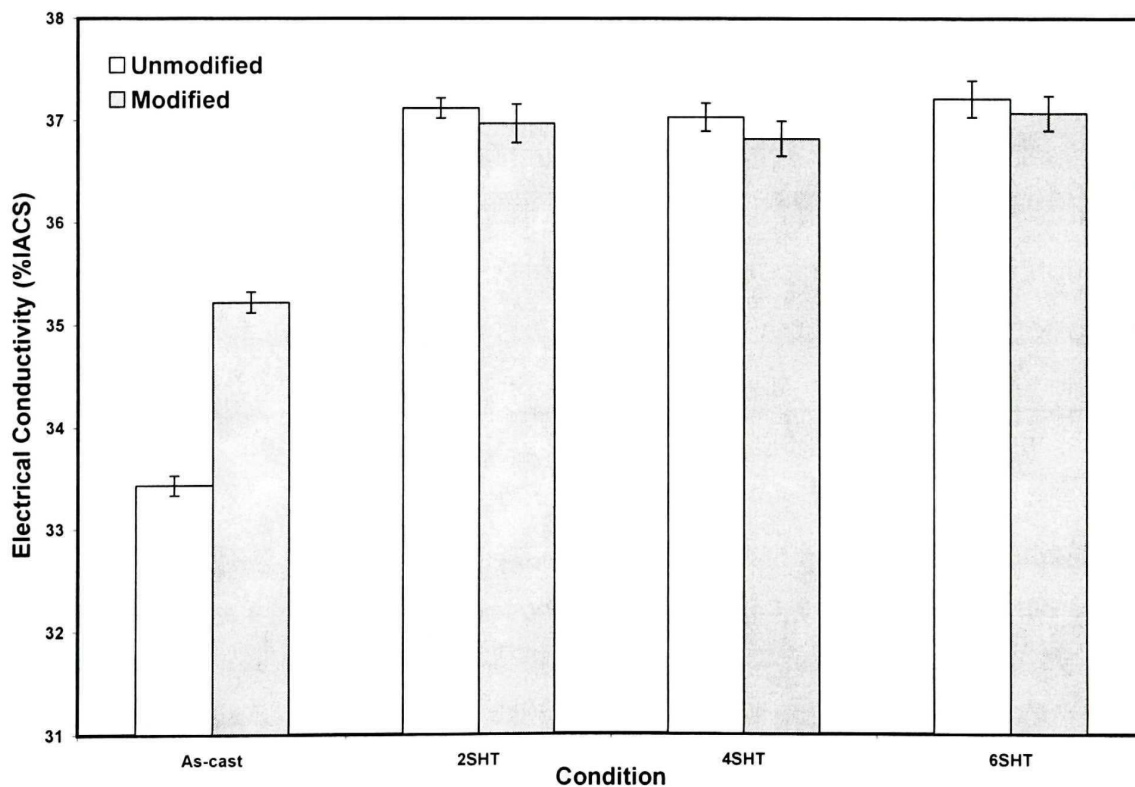


Figure 4.3. Electrical conductivity at T4 condition of unmodified and modified A356 coupons upon solution treatment at 540°C. The standard deviations are shown

The influence of different solution heat treatment times on the matrix microhardness of coupons artificially aged for 3, 6 and 9 hours at 155°C is presented in Figure 4.4. It is observed that the coupons solution heat-treated for 2 hours exhibit larger microhardness values in the T6 condition than those treated for 4 and 6 hours. On the other hand, no significant effect of the strontium modification is observed on the microhardness. The improved microhardness could only be explained if small vacancy clusters are formed as solution heat treatment proceeds leading to a reduction in the number of possible nuclei for further precipitation.

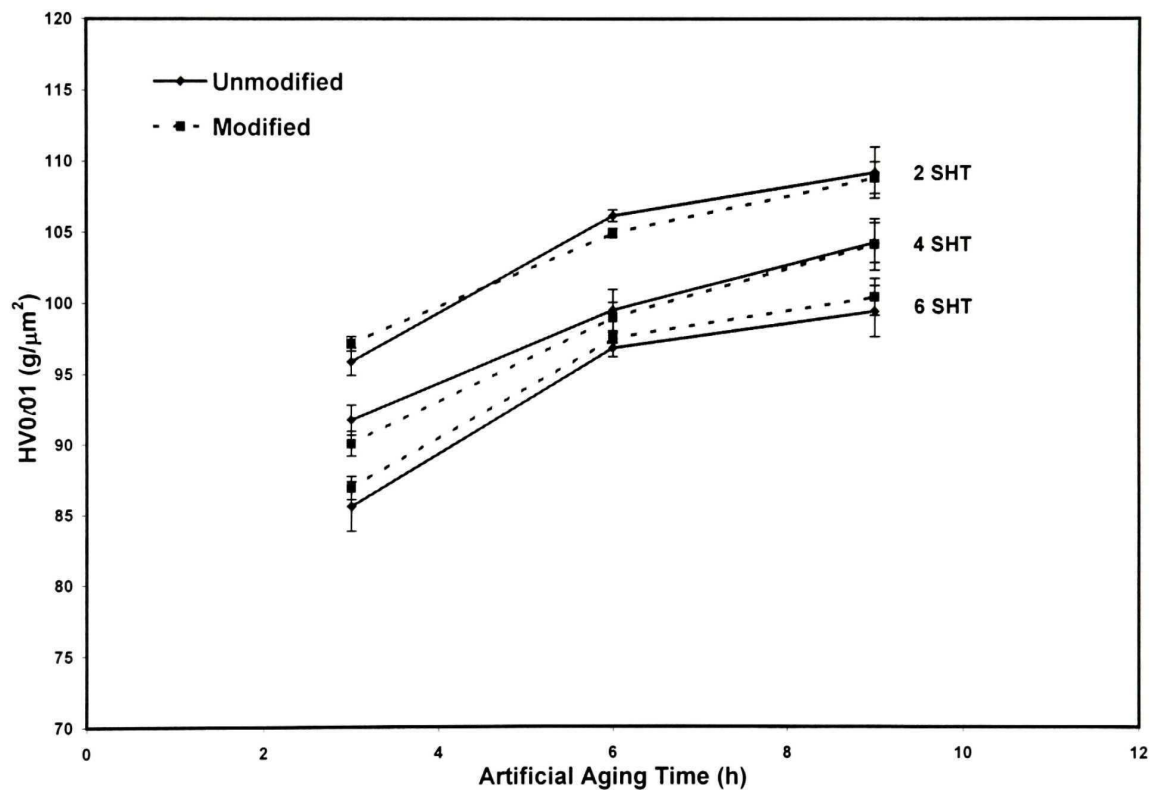


Figure 4.4. Matrix microhardness of unmodified and modified A356 coupons solution heat-treated for 2, 4 and 6 hours at 540°C and artificially aged for 3, 6 and 9 hours at 155°C

Figure 4.5 shows typical TEM micrographs of the β'' -Mg₂Si present in the modified coupons solution heat treated for 2, 4 and 6 hours and then artificially aged for 6 hours at 155°C. Table 4.1 gives the analysis of the average length and

diameter of at least 350 β'' - Mg_2Si precipitates in total per condition. It was noted that the average length of the precipitates reaches a minimum with a solution heat treatment of 6 hours. On the other hand, the average diameter seems to reach a maximum with 4 hours of solution heat treatment. However, in both cases the standard deviation does not allow a clear conclusion to be reached.

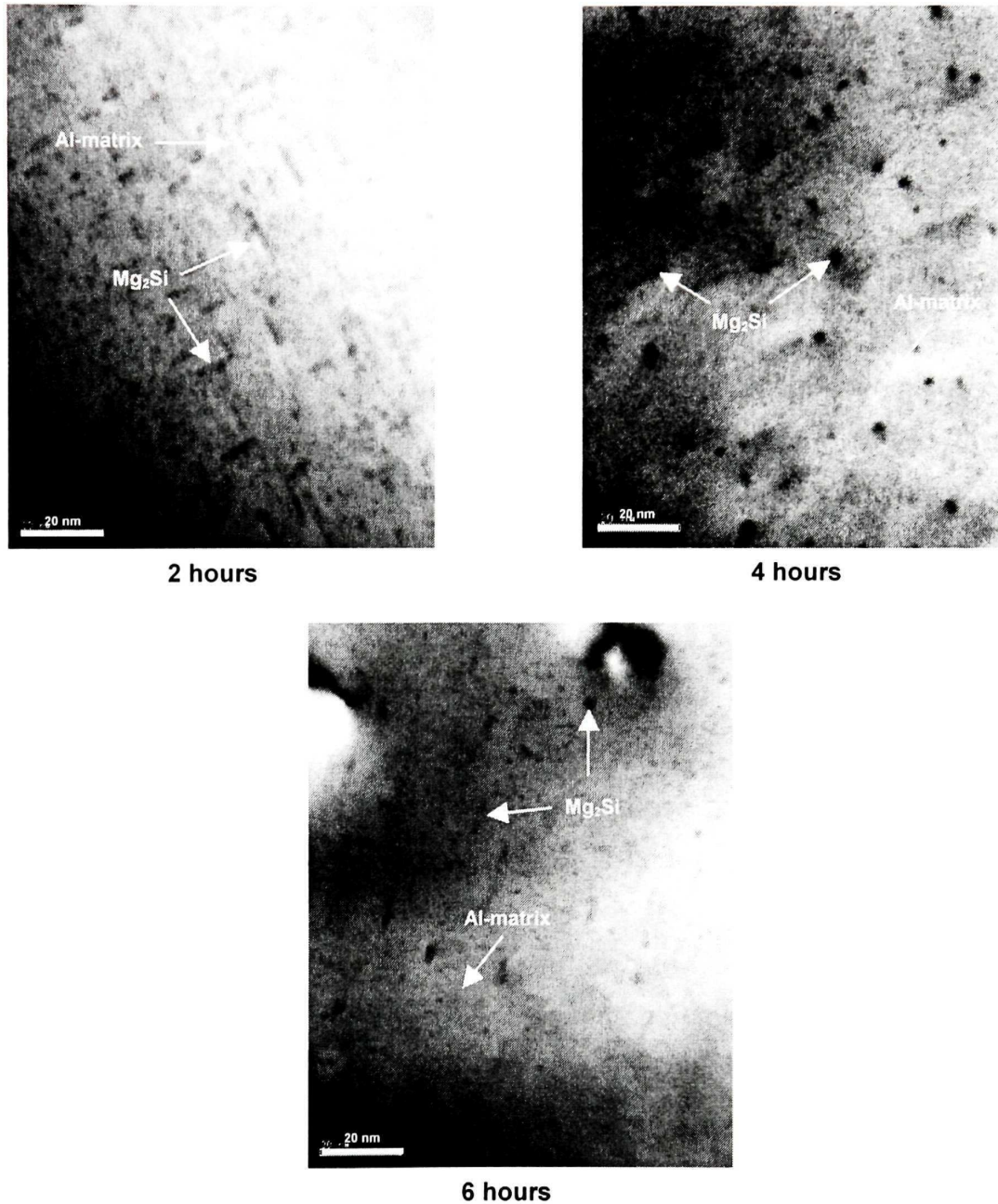


Figure 4.5. Transmission electron micrographs of modified coupons solution heat-treated for 2, 4 and 6 hours at 540°C and artificially aged 6 hours at 155°C

Table 4.1. Average length and diameter of the precipitates solution heat-treated 2, 4 and 6 hours and artificially aged 6 hours at 155°C

Solution heat-treatment time	Average length	Average diameter
(h)	(nm)	(nm)
2	10.5±1.4	2.1±0.5
4	10.4±3.2	2.7±0.7
6	8.8±1.6	2.1±0.6

Figures 4.6 and 4.7 show histograms of the precipitate length and diameter distribution of the β'' -Mg₂Si as a function of solution heat treatment time. In the case of the length distribution, it is clearly seen that the most normal distribution is obtained with samples solution heat-treated 2 hours. It is also seen that the distribution at 4 hours becomes wider due to the presence of larger precipitates. On the other hand, at 6 hours the distribution shifts to the left indicating the presence of smaller precipitates. A similar behaviour is observed in the distribution of precipitate diameter.

The electron diffraction patterns of the coupons at the different conditions are shown in Figure 4.8. In these images, it is possible to observe that no extra diffractions spots appear indicating that the contribution of the precipitates to the diffraction spots is small. If characterization of these precipitates were needed micro or nano diffraction should be used. It is known, that the most common sources of extra spots in diffraction patterns are second-phase particles, twins, martensite plates, etc. These spots may be used to check the crystal structure and lattice parameter of the phases and its orientation relationship with the matrix [85]. Figure 4.9 shows an example of an electron diffraction pattern presenting extra spots. Measurements of the lattice parameter resulted in values of 4.16±0.14 Å, 4.01±0.02 Å and 4.12±0.10 Å for 2, 4 and 6 hours of solution treatment respectively. These values are close to that of pure aluminum (4.049 Å). It is believed that the observed differences can be attributed to measurement uncertainties rather than metallurgical effects.

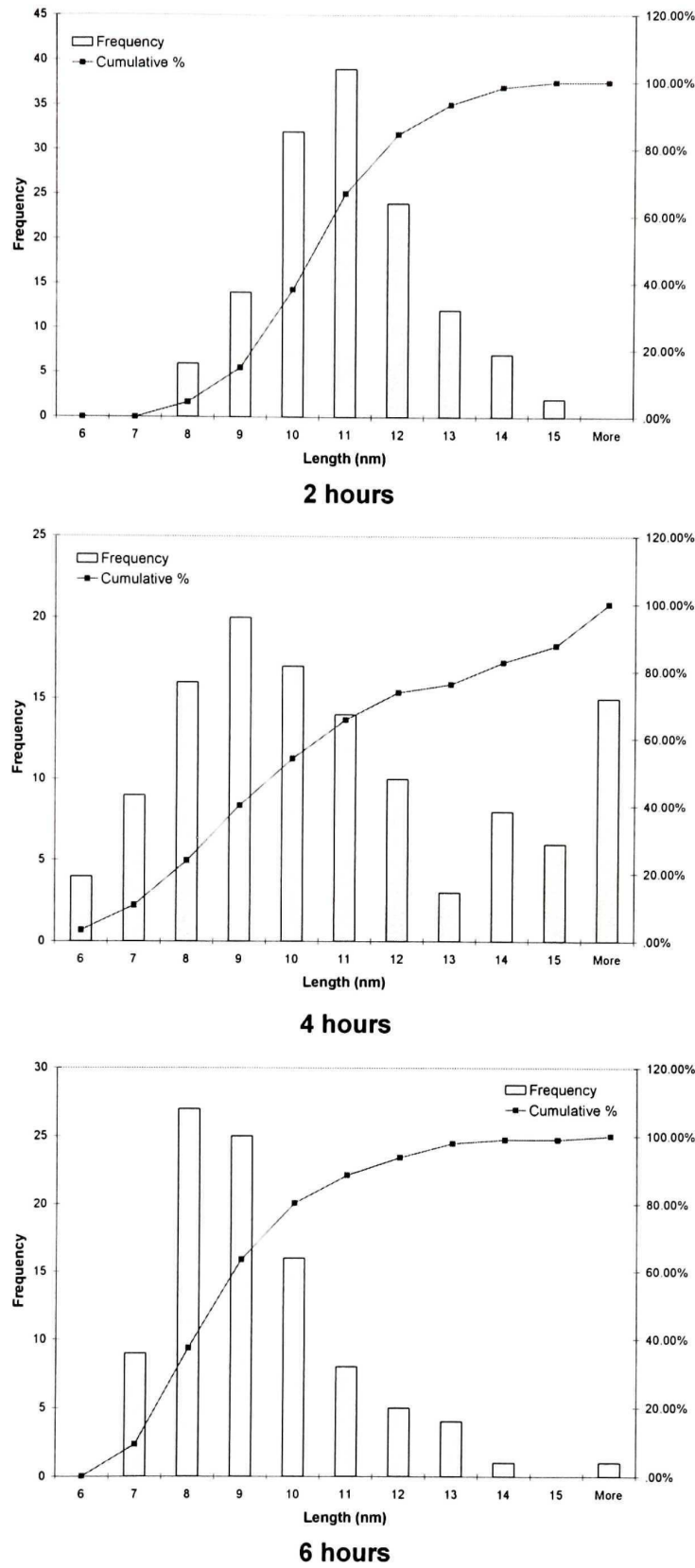


Figure 4.6. β'' - Mg_2Si length distribution in coupons solution heat-treated for 2, 4 and 6 hours at 540°C and artificially aged 6 hours at 155°C

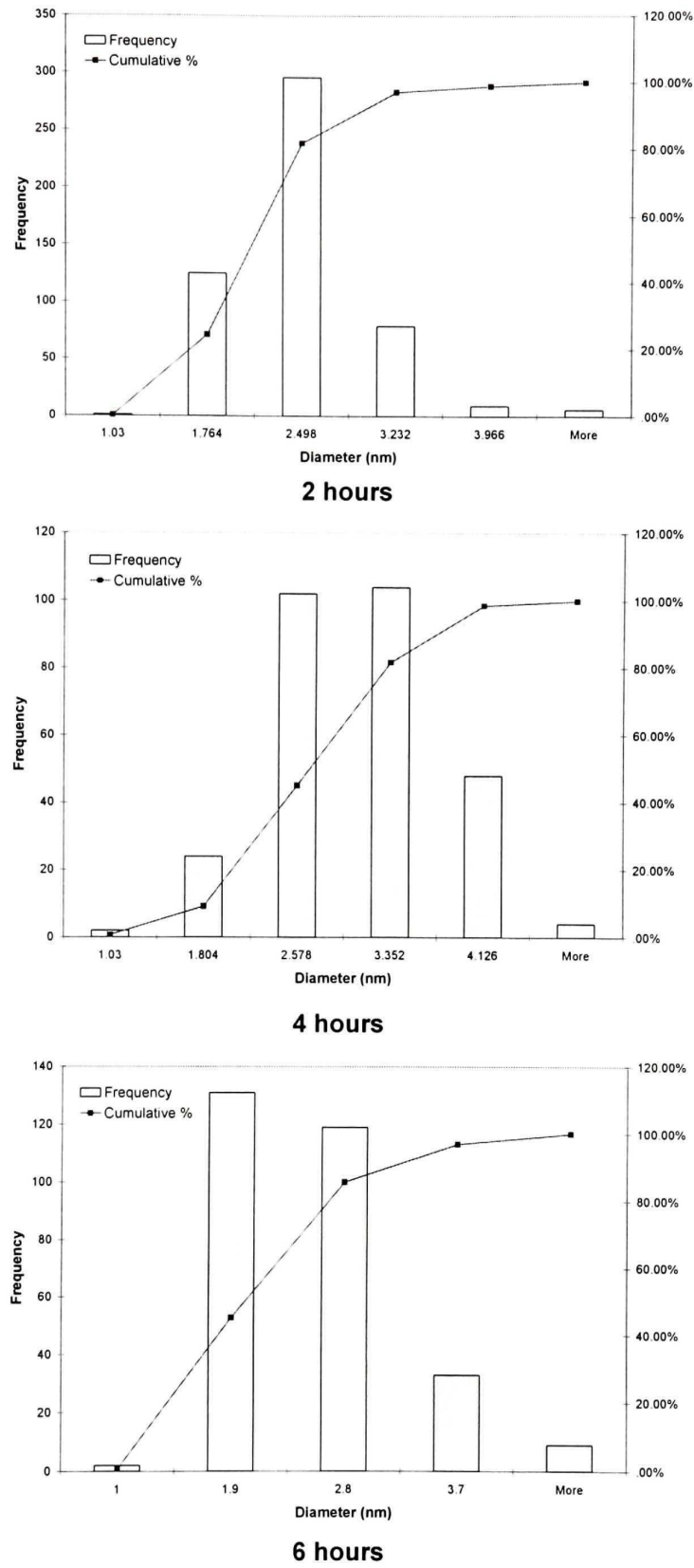


Figure 4.7. β'' - Mg_2Si diameter distribution in coupons solution heat-treated for 2, 4 and 6 hours at 540°C and artificially aged 6 hours at 155°C

The differences that can be seen between the electron diffraction patterns for different solution heat treatment times arise from the zone axis (B). In the case of solution heat treatment times of 2 and 4 hours the zone axis is $B=[011]$ and for 6 hours $B=[013]$.

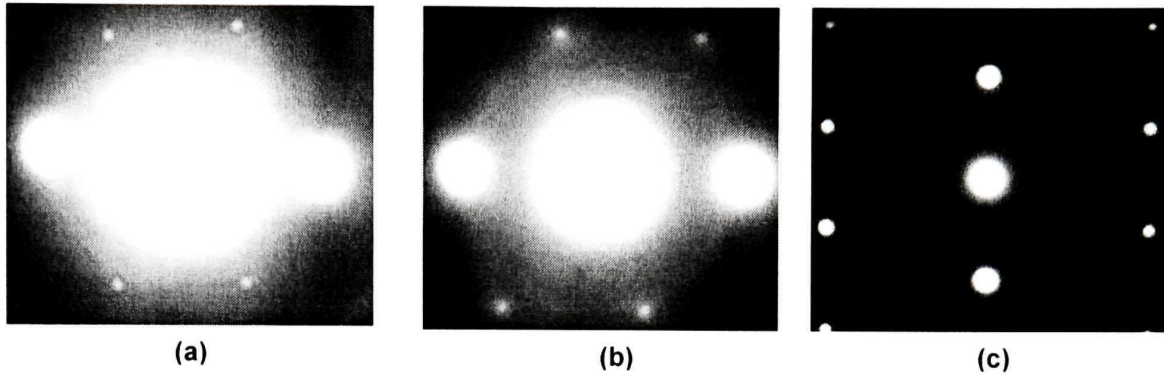


Figure 4.8. Electron diffraction patterns of modified coupons solution heat-treated for: (a) 2 hours, (b) 4 hours and (c) 6 hours at 540°C and artificially aged 6 hours at 155°C

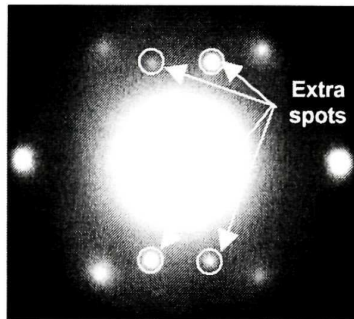


Figure 4.9. Electron diffraction pattern of a modified coupon solution heat-treated for 4 hours and artificially aged 6 hours at 236°C presenting extra diffraction spots

4.3 Quenching

The effect of different quenching temperatures on the electrical conductivity of unmodified and modified coupons solution heat-treated 4 hours and then artificially aged 6 hours at 155°C is shown in Figure 4.10. In this figure, it is possible to see that the electrical conductivity does not show any significant differences between the unmodified and modified coupons quenched at 0°C, 22°C, 50°C and 70°C. The coupons quenched with compressed air at room temperature showed higher conductivity values due to a reduction in the quenching rate [86] that results in decreased supersaturation in the solid solution. This decrease increases the electron flow through the matrix.

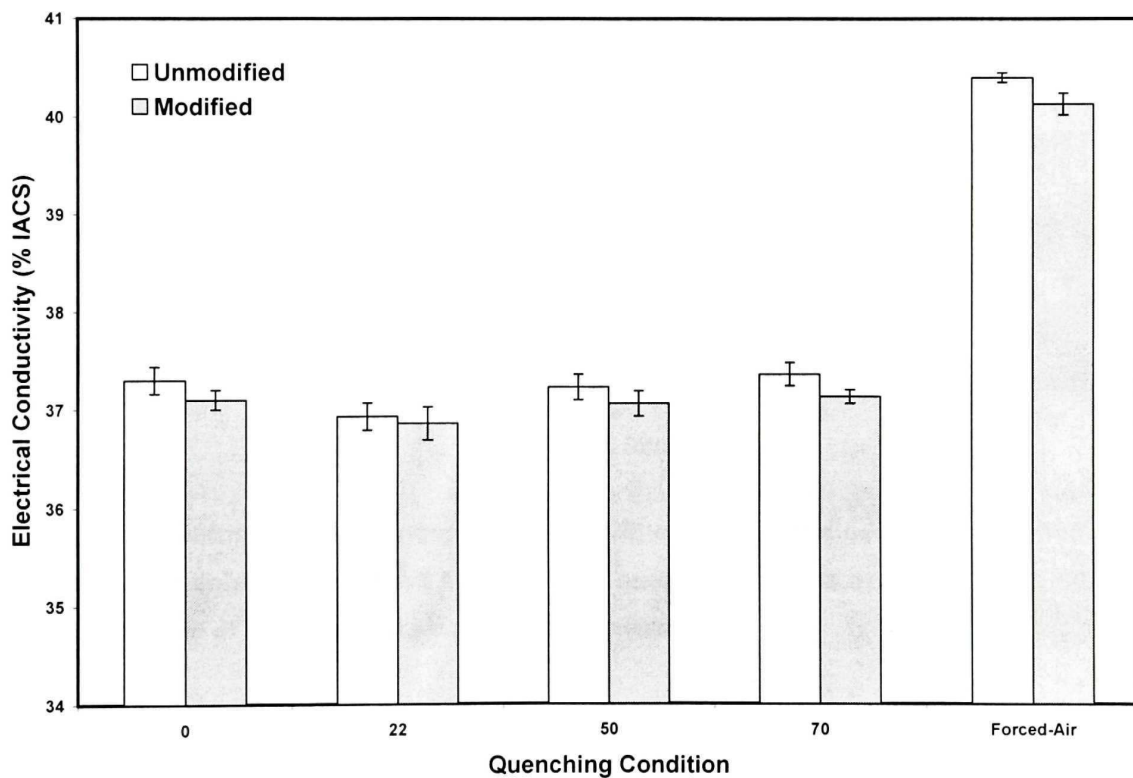


Figure 4.10. Electrical conductivity at T6 condition of unmodified and modified A356 coupons upon solution treatment at 540°C and quenching temperatures of 0°C, 22°C, 50°C, 70°C and compressed air at room temperature

When the microhardness is used to characterize the effect of quenching condition (see Figure 4.11), coupons quenched with water at 0°C and 22°C and compressed air at room temperature yield similar values. Moreover, it is seen that coupons quenched at 50°C and 70°C show slightly improved microhardness values (~4%). However, the size of the standard deviation makes that difference almost insignificant.

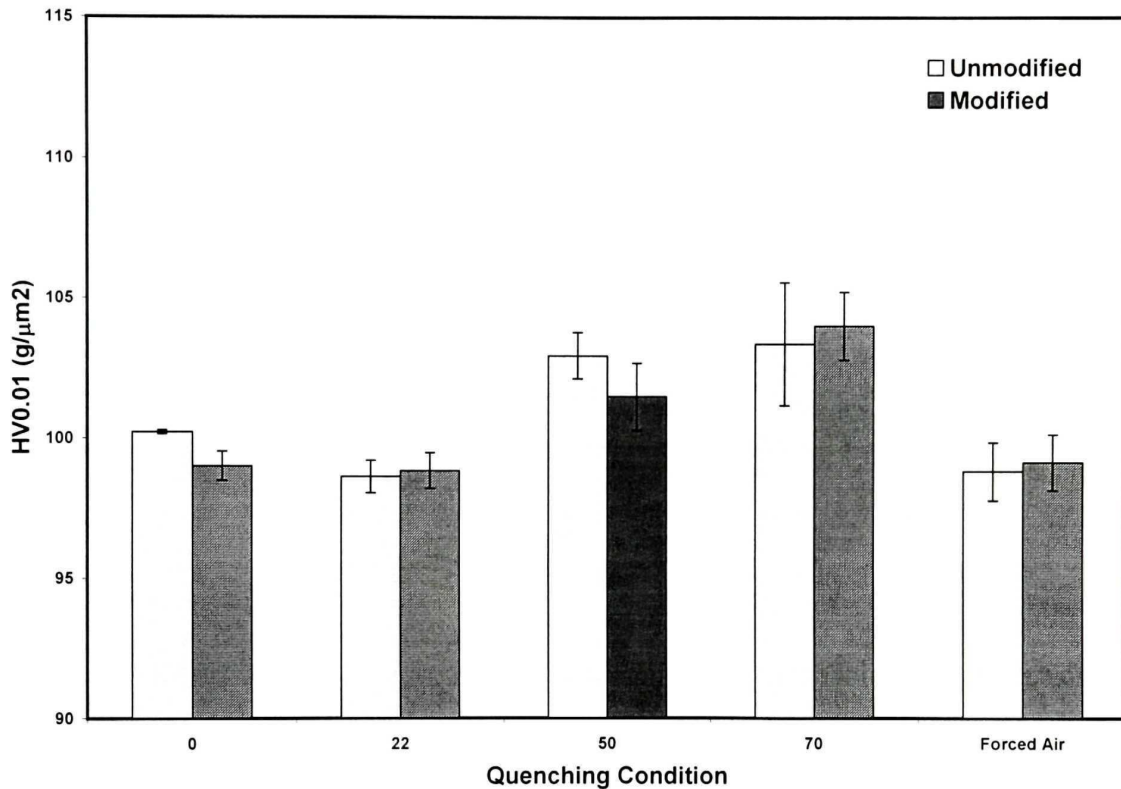


Figure 4.11. Matrix microhardness at T6 condition of unmodified and modified A356 coupons upon solution treatment at 540°C and quenching temperatures of 0, 22, 50, 70°C and with the use of compressed air at room temperature

It has been stated that a rapid quenching ensures that all Mg and Si are retained in solid solution, and that the highest strength attainable is obtained with fast quenching rates [49,59]. However, as shown in Figure 4.12, Tsukuda et al [87] have reported that the reduction of the quenching rate (increase of the quenching media temperature) does not affect the YS and only minimally influences the UTS. Such behaviour would be expected in view of the consistency of the microhardness shown in Figure 4.11. The decrease in the

elongation could be related to the more uniform size distribution of the precipitates formed when quenching occurs at lower cooling rates.

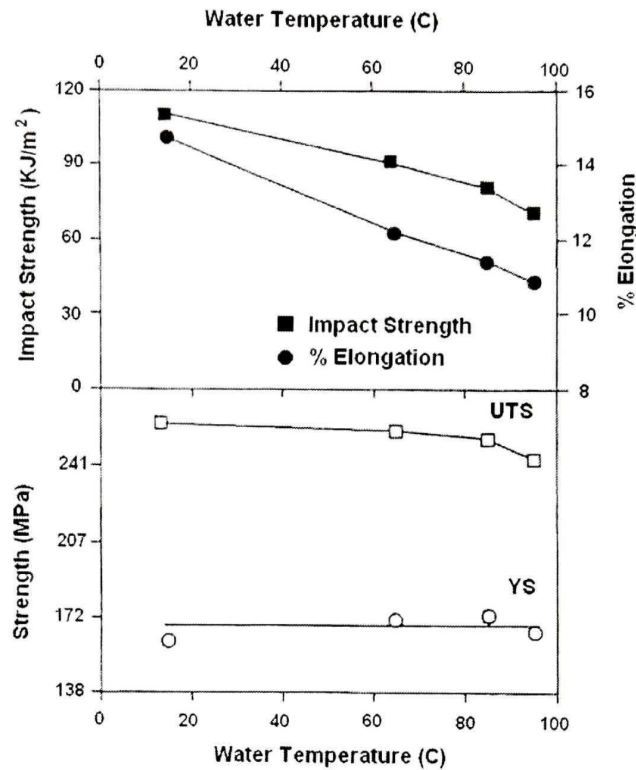
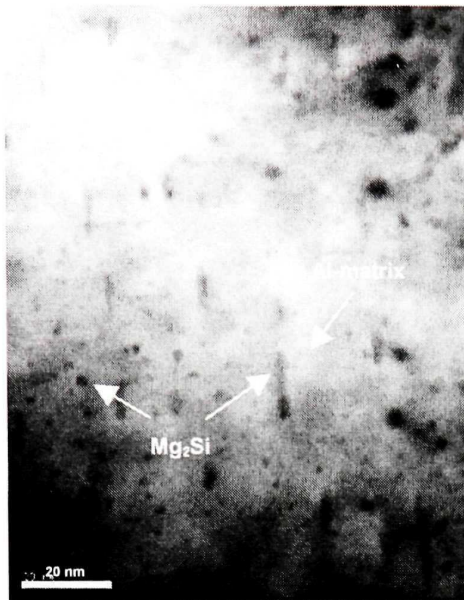
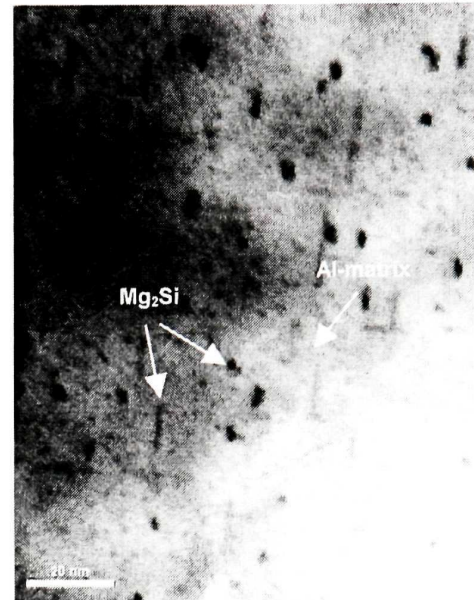


Figure 4.12. Effect of water temperature at quench on the mechanical properties of A356 alloy [87]

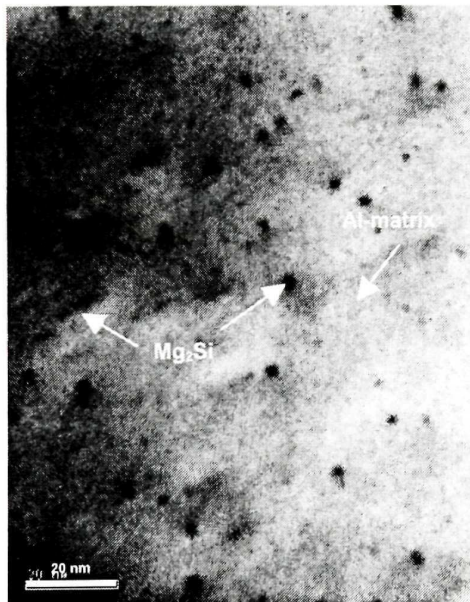
Figures 4.13 and 4.14 show TEM micrographs of the coupons quenched at different conditions. The analysis of the precipitate length and diameter measured from several of these micrographs is presented in Table 4.2. From this analysis, it is possible to see that there are no significant size differences in the precipitates between the samples quenched at different conditions.



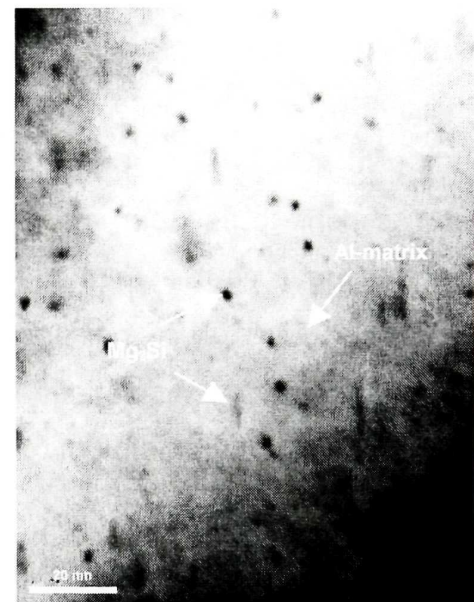
0°C (unmodified)



0°C (modified)



22°C (modified)



70°C (modified)

Figure 4.13. Transmission electron micrographs of coupons solution heat-treated for 4 hours at 540°C, quenched at different conditions and artificially aged for 6 hours at 155°C

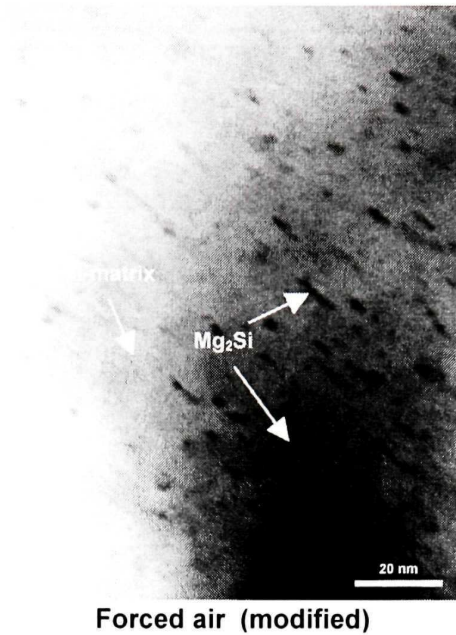


Figure 4.14. Transmission electron micrographs of coupons solution heat-treated for 4 hours at 540°C, quenched with compressed air and artificially aged for 6 hours at 155°C

Figures 4.15 to 4.18 show histograms representing the β'' -Mg₂Si precipitate length and diameter distribution. In the case of the length, the distribution is wider with higher quenching rates (0°C and 22°C). On the other hand, as the quenching media temperature increases (reduced quenching rate) the distribution becomes narrower. In the case of compressed air quenching it is possible to see a similar behaviour as the quenching at 70°C is seen. This behaviour indicates the presence of more homogeneous and smaller precipitates. A very similar behaviour is observed with respect to the diameter distributions.

Table 4.2. Average length and diameter of the precipitates solution heat-treated 4 hours, quenched at 0°C, 22°C, 50°C, 70°C and compressed air and artificially aged 6 hours at 155°C

Quenching Condition	Average length (nm)	Average diameter (nm)
0°C (unmodified)	10.9±2	2.3±0.6
0°C (modified)	11.2±1.7	2.27±0.4
22°C (modified)	10.4±3.2	2.74±0.7
70°C (modified)	10.1±1.1	2.2±0.4
Compressed air (modified)	10.0±1.6	2.1±0.4

Figure 4.19 shows the different electron diffraction patterns obtained from the different quenching conditions. In these images (as in the ones shown in Figure 4.8) it is possible to observe that no extra diffraction spots appear. As mentioned previously it does not mean that precipitation has not taken place.

The electron diffraction patterns indicate that lattice parameters vary from 4.04 ± 0.03 Å for the unmodified coupon quenched at 0°C, 4.08 ± 0.08 Å and 4.01 ± 0.02 Å, 4.02 ± 0.02 Å, 4.01 ± 0.002 Å for the modified coupons quenched at 0°C, 22°C, 70°C and compressed air respectively. As was the case with the solution heat treatment the differences between the calculated lattice parameter and that for pure aluminum appear to be from measurement uncertainties only.

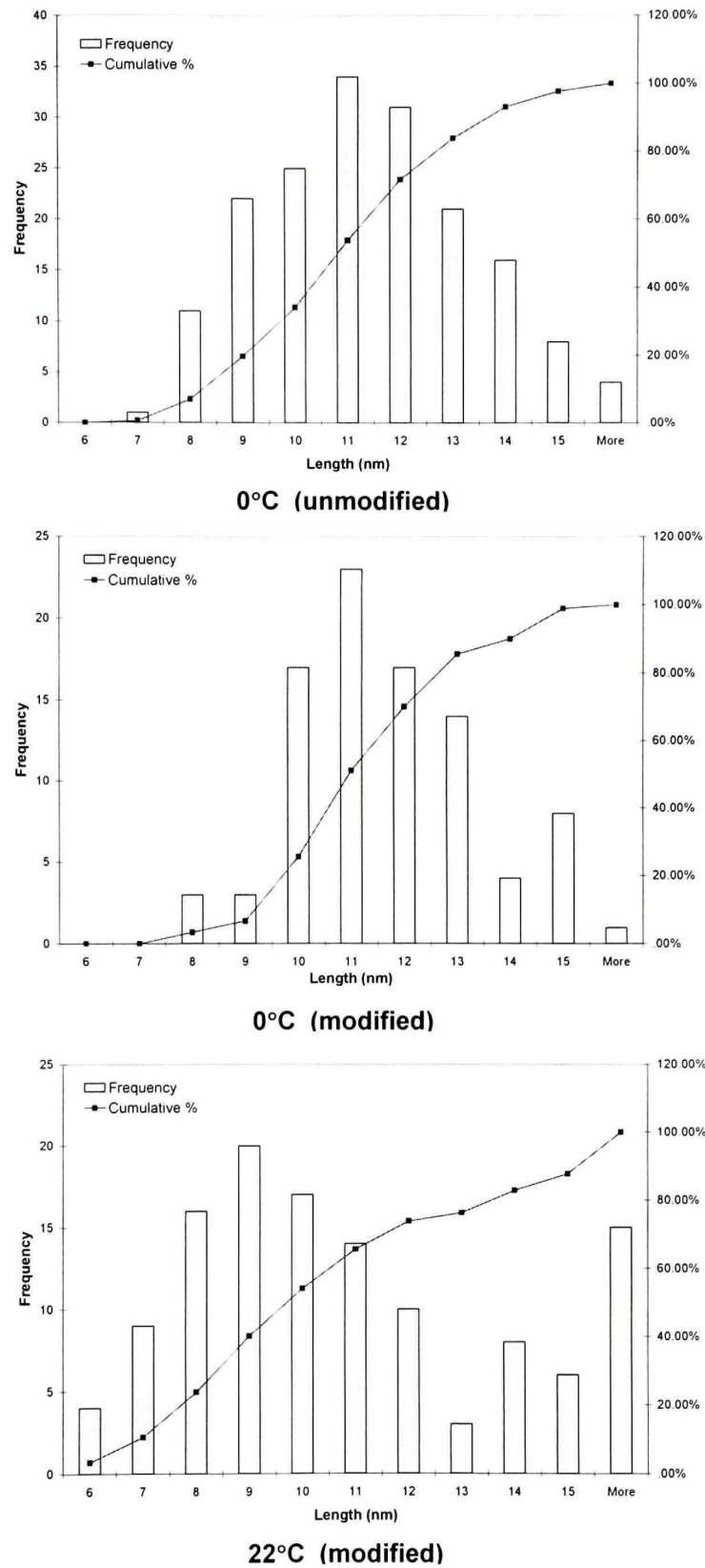


Figure 4.15. β'' - Mg_2Si length distribution in coupons solution heat-treated for 4 hours at 540°C quenched at different conditions and artificially aged 6 hours at 155°C

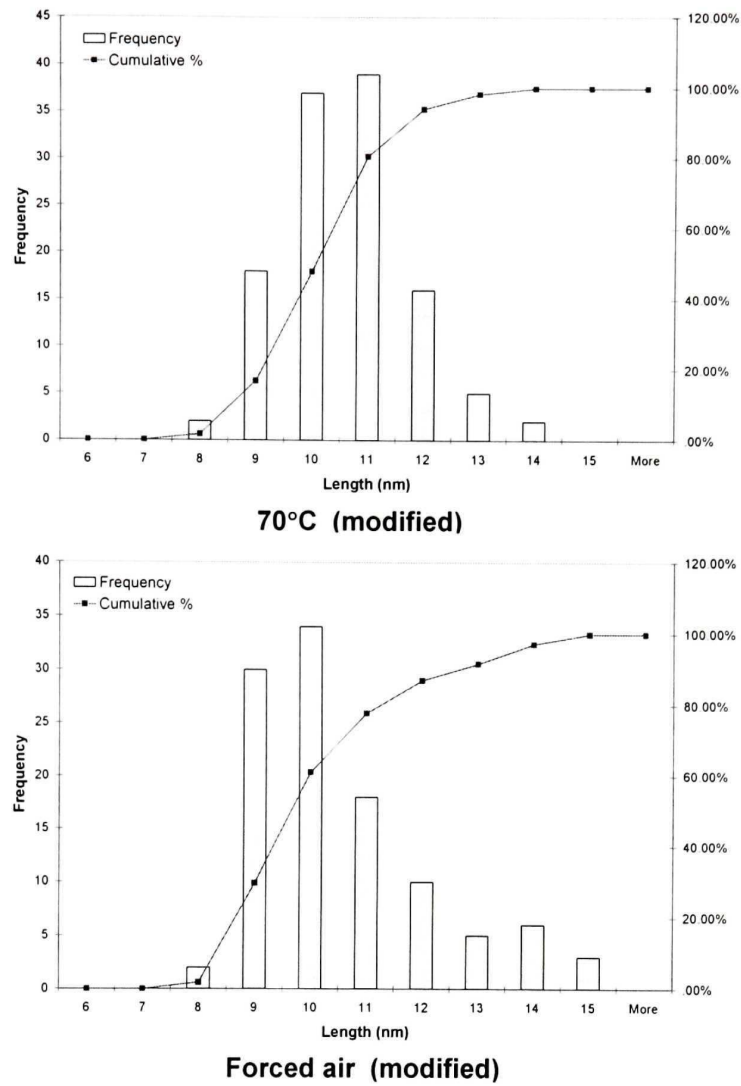


Figure 4.16. β'' - Mg_2Si length distribution in coupons solution heat-treated for 4 hours at 540°C quenched at different conditions and artificially aged 6 hours at 155°C

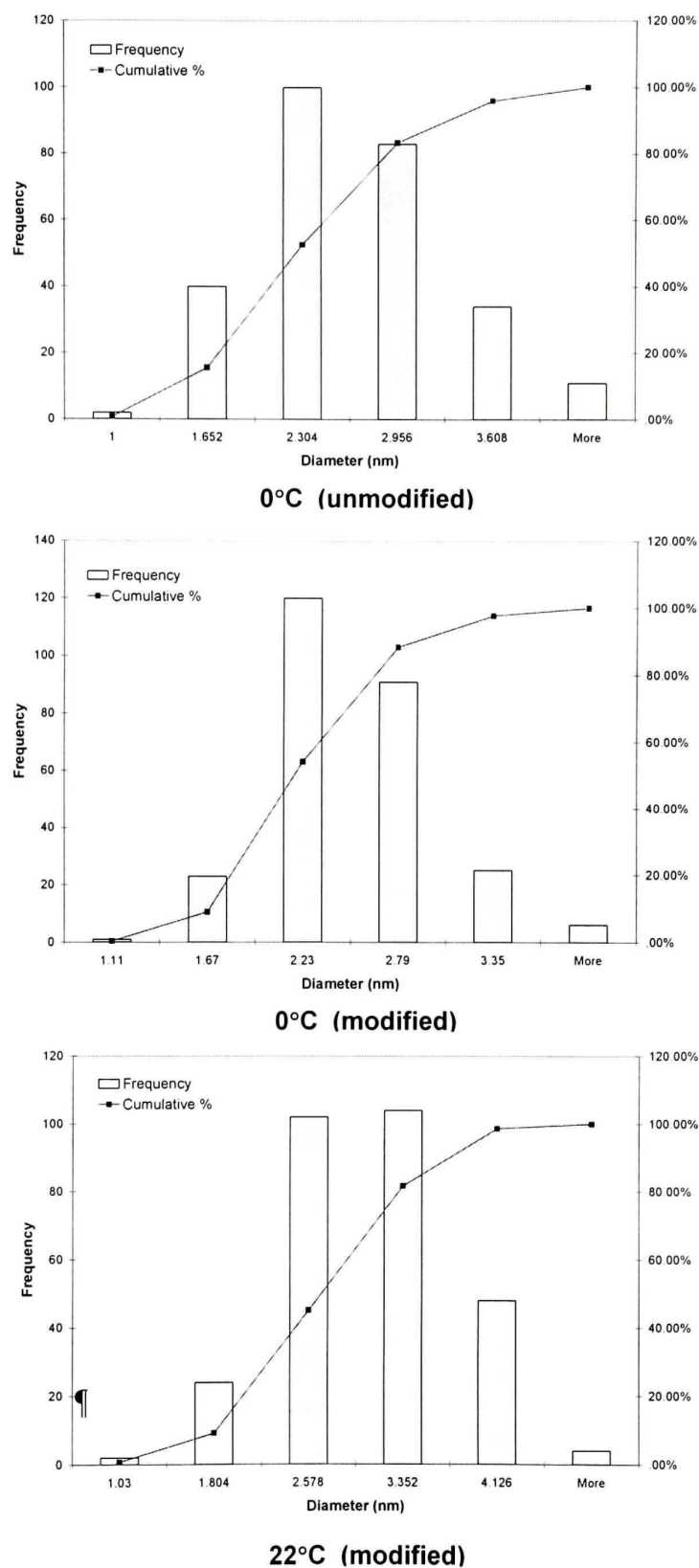


Figure 4.17. β'' - Mg_2Si diameter distribution in coupons solution heat-treated for 4 hours at 540°C quenched at different conditions and artificially aged 6 hours at 155°C

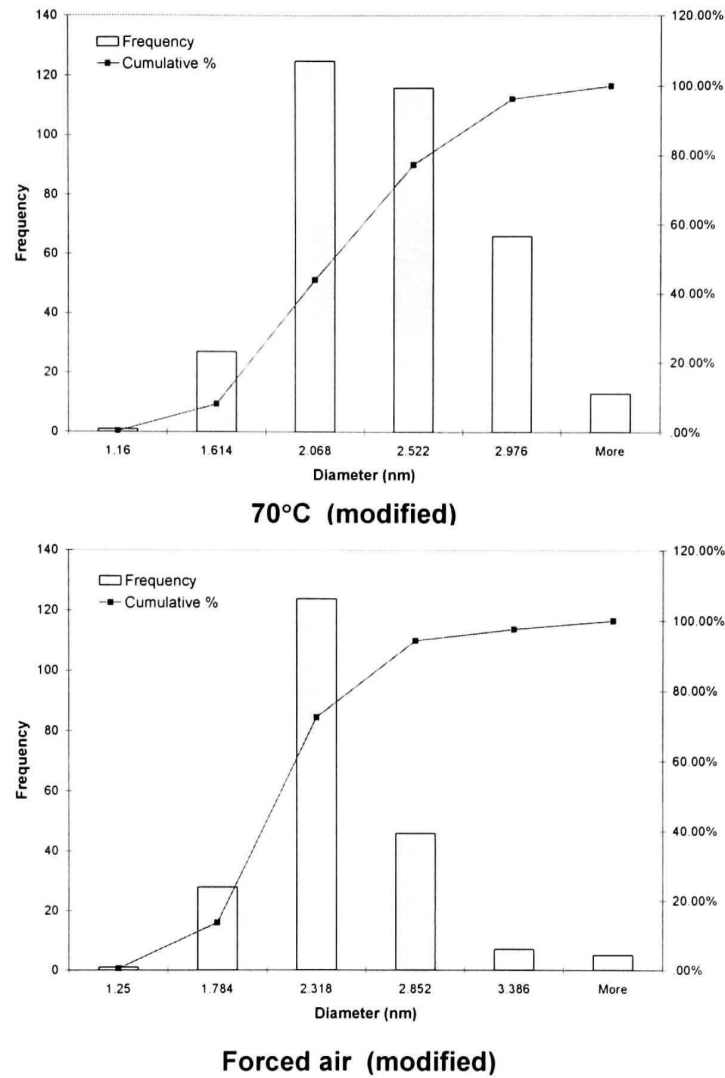


Figure 4.18. β'' - Mg_2Si diameter distribution in coupons solution heat-treated for 4 hours at 540°C quenched at different conditions and artificially aged 6 hours at 155°C

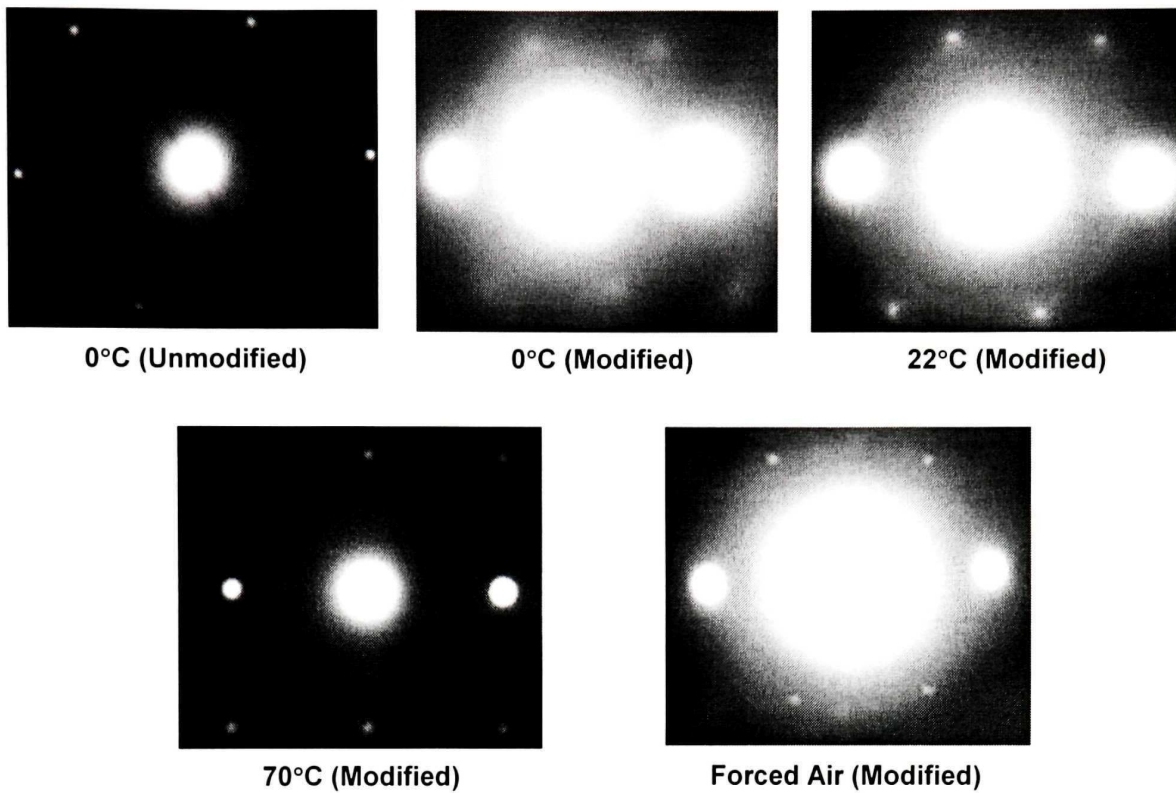


Figure 4.19. Electron diffraction patterns of coupons solution heat-treated for 4 hours at 540°C, quenched at different conditions and artificially aged for 6 hours at 155°C

4.4 Natural Aging

The effect of natural aging time on the electrical conductivity of unmodified and Sr-modified A356 coupons after quenching (T4 condition) can be seen in Figure 4.20. It is observed that the electrical conductivity decreases during the first hours of natural aging until reaching a plateau at approximate 3 hours. It can also be noted that Sr modification does not exert any significant influence on the kinetics of natural aging. Measurements on tensile test bars showed the same behaviour (Table 4.3).

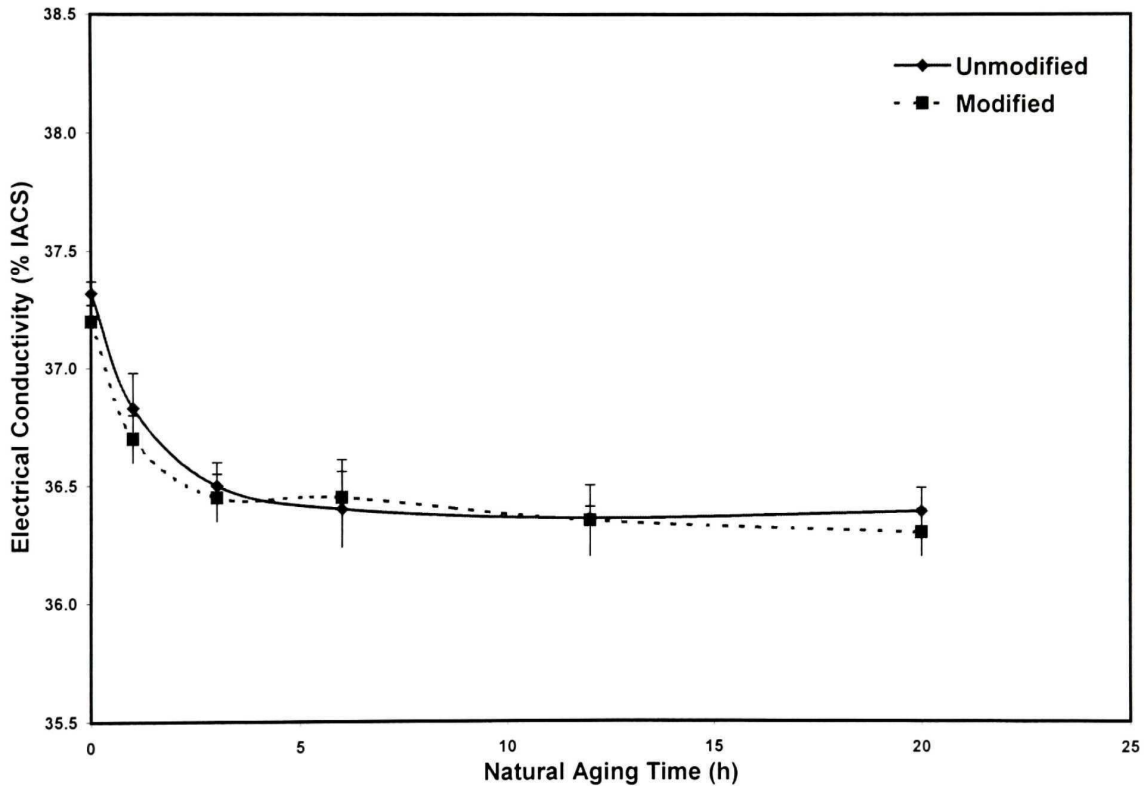


Figure 4.20. Effect of natural aging on the electrical conductivity of unmodified and modified quenched coupons (T4 condition)

Even though the electrical conductivity of supersaturated solid solution alloys generally increases when precipitation of alloying elements takes place, there are cases such as in the Al-Si-Mg alloys, in which the formation of coherent small clusters, known as Guinier-Preston zones (G.P. zones) leads to a distortion

of the crystal lattice and the generation of coherency strains that result in a lower conductivity and the loss of supersaturation (driving force) necessary for further precipitation. The plateau reached in the conductivity after three hours of natural aging indicates that a stage of slow growth of such clusters has been reached [1]. Figure 4.21 shows the electrical conductivity of unmodified coupons subjected to long natural aging time periods. A small continuous decrease in the conductivity can be observed with natural aging, which indicates that the cluster growth phenomenon is taking place.

It is known that during the solution heat treatment, the value of the electrical conductivity will increase due to the fragmentation of the eutectic silicon [62]. A slight decrease follows as solution heat-treatment continues due to the dissolution of alloying elements such as Mg and Si into the primary phase (α -Al). Coarsening of eutectic silicon does not have any detectable effect on the conductivity. The hardness of the α -Al will increase due to solute hardening of the matrix. As precipitates form, there is usually an increase in electrical conductivity as they have a smaller effect on electron scattering than do elements dissolved in solid solution. On the other hand, precipitation is generally accompanied by an increase in hardness, particularly if the precipitates are coherent with the matrix.

Table 4.3 Effect of natural aging on electrical conductivity after water quench of Sr-modified tensile bars

Condition	Electrical Conductivity (% I.A.C.S.)
As-quenched	38.7 ± 0.25
6 hours of natural aging	38.3 ± 0.20
12 hours of natural aging	38.0 ± 0.30
20 hours of natural aging	7.7 ± 0.50

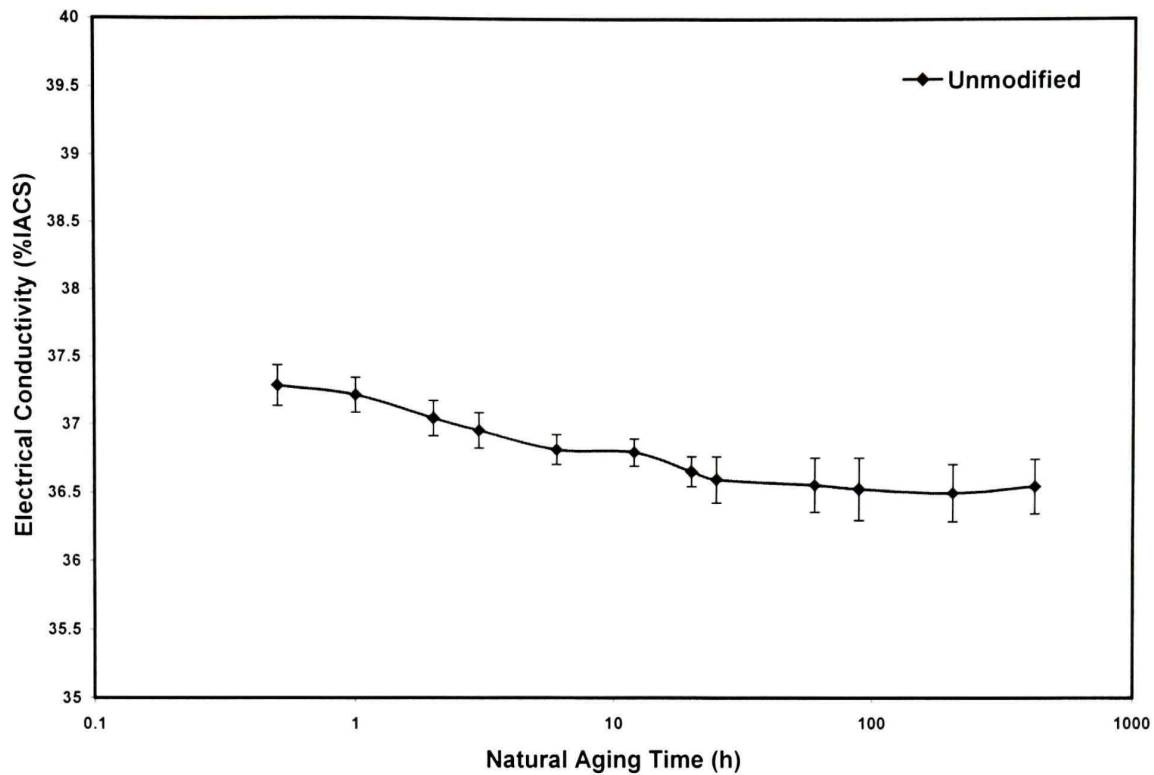


Figure 4.21. Effect of extended natural aging on the electrical conductivity of unmodified quenched coupons (T4 condition)

The influence of the natural aging on the matrix microhardness of unmodified coupons at T4 condition is shown in Figure 4.22. In this figure, a small increment in the microhardness value is observed as natural aging takes place. This behaviour indicates that the clusters or GP-zones are slowly growing.

Figure 4.23 and Table 4.4 show the effect of natural aging on the electrical conductivity of coupons and tensile test bars artificially aged 6 hours at 155°C. It is seen that no significant effects are detected. On the other hand, a detrimental effect on microhardness is seen in Figure 4.24 with natural aging times up to 20 hours. The influence of natural aging times up to 20 hours on the mechanical properties is shown in Table 4.5 where a detriment on both the yield and tensile strength is observed as natural aging increases. On the other hand, there appears to be a tendency towards an improvement in elongation with natural aging time, but the size of the standard deviation makes reaching a clear conclusion difficult even though it decreased with natural aging.

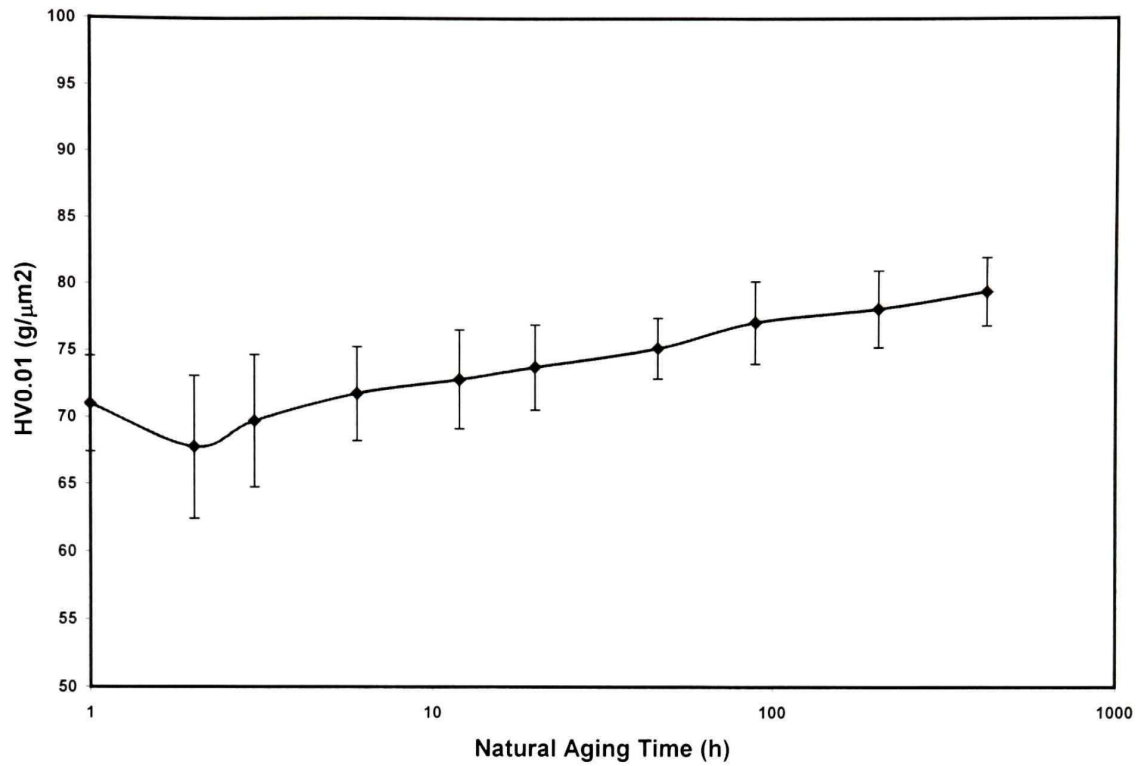


Figure 4.22. Effect of extended natural aging on the matrix microhardness of unmodified quenched coupons (T4 condition)

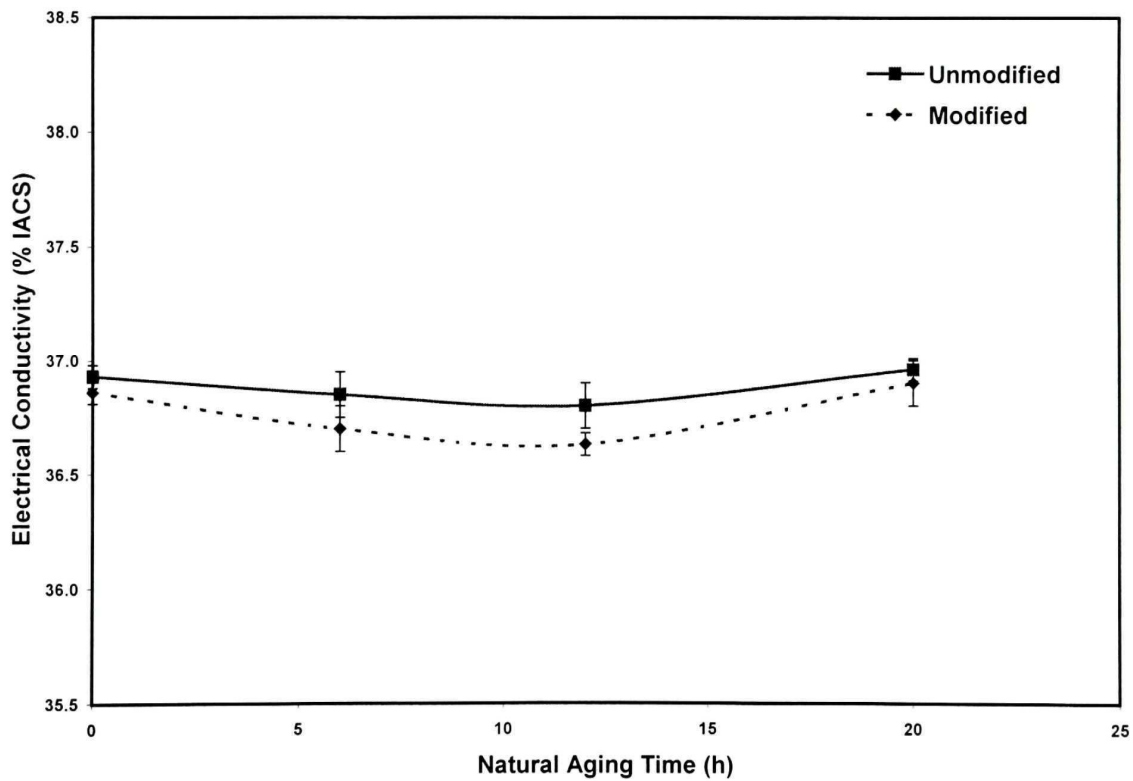


Figure 4.23. Electrical conductivity of unmodified and modified coupons artificially aged 6 hours at 155 °C

Table 4.4. Electrical conductivity of artificially aged 6 hours at 155 °C tensile test bars

Condition	Electrical Conductivity (% I.A.C.S.)
No natural aging + 6 hours at 155 °C	39 ± 0.10
6 hours of natural aging + 6 hours at 155 °C	38.7 ± 0.30
12 hours of natural aging + 6 hours at 155 °C	38.8 ± 0.40
20 hours of natural aging + 6 hours at 155 °C	38.9 ± 0.30

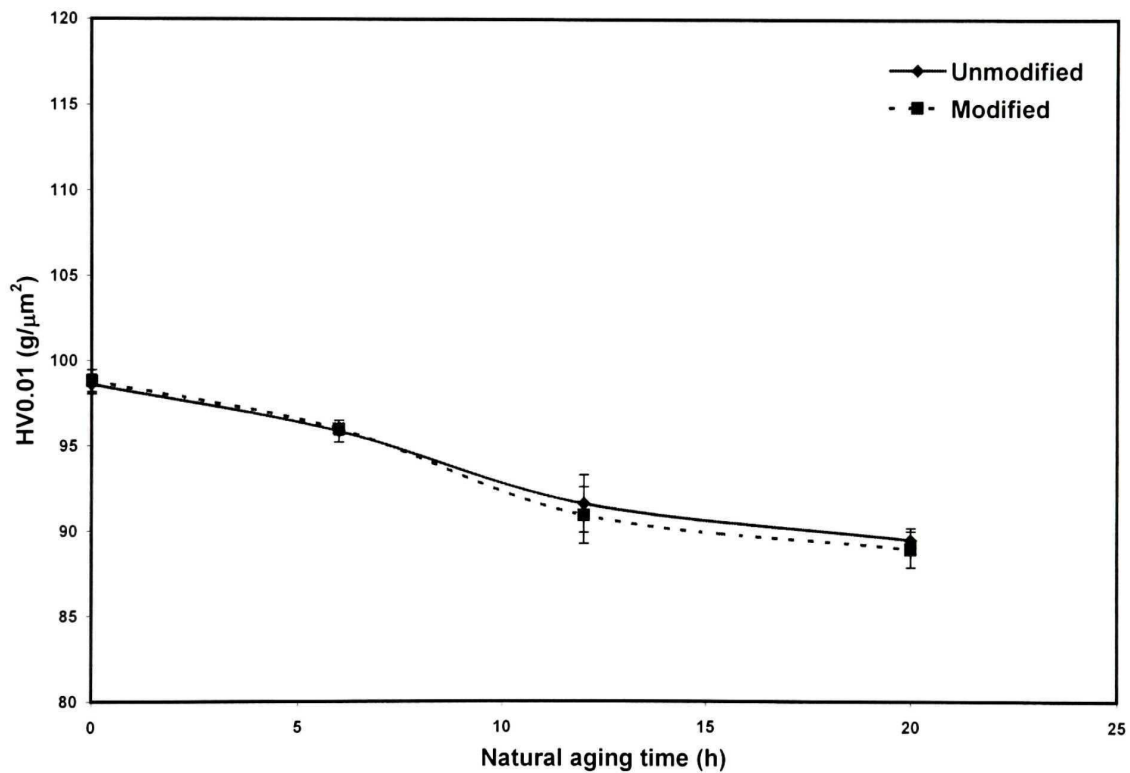


Figure 4.24. Matrix microhardness of unmodified and modified coupons artificially aged 6 hours at 155 °C

Table 4.5. Tensile properties of artificially aged 6 hours at 155 °C test bars

Condition	UTS (MPa)	YS (MPa)	Elongation (%)
No natural aging + 6 hours at 155 °C	294 ± 5.6	216.7 ± 3.2	8.05 ± 1.22
6 hours of natural aging + 6 hours at 155 °C	283 ± 3.7	193.2 ± 2	9.78 ± 1.71
12 hours of natural aging + 6 hours at 155 °C	282.9 ± 2.5	187.2 ± 6.6	10.72 ± 0.95
20 hours of natural aging + 6 hours at 155 °C	281.9 ± 1.9	184.4 ± 4.5	11.76 ± .48

Figure 4.25 shows the TEM micrographs obtained from coupons subjected to different natural aging conditions and artificially aged 6 hours at 155°C. The analysis of the average length and diameter is presented in Table 4.6. In this table, it is possible to see that both the average length and diameter of the precipitates show a tendency to decrease with natural aging time. However, in the case of the length, the magnitude of the standard deviation does not allow making a conclusion on the effect of the length of the precipitates in the microhardness and yield strength.

Regarding the precipitate length and diameter distribution, this is shown in Figures 4.26 and 4.27. In these images it is seen that with no natural aging the precipitate length distribution is wider than the one found in samples with natural aging. When the coupons are subjected to 12 hours of natural aging a clear shifting to left side of the histogram, indicating a decrease in the precipitate length is observed. As the natural aging continues (20 hours) an increase in the number of longer precipitates is detected. In the case of the precipitate diameter the behaviour is generally the same as that of the length.

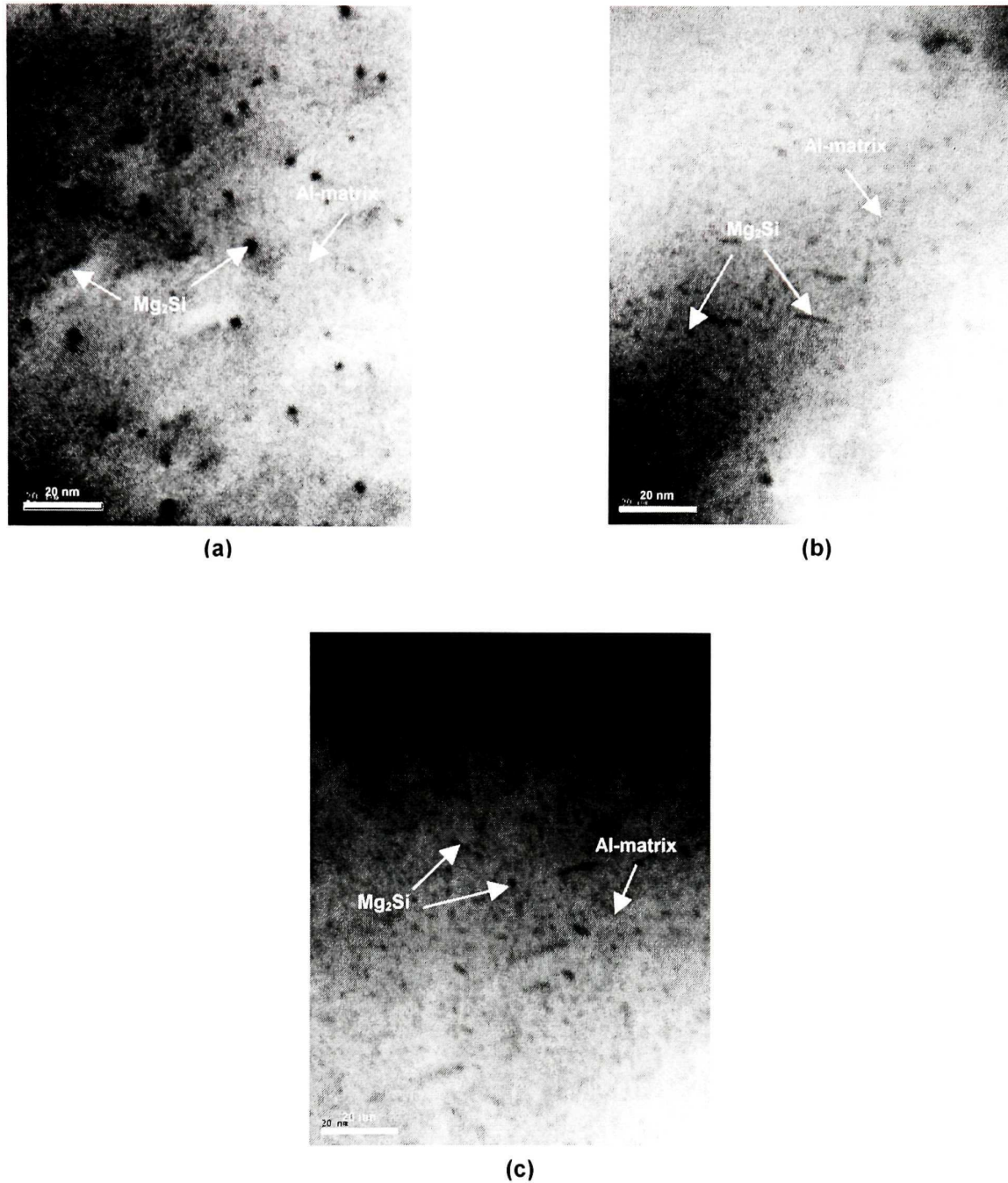
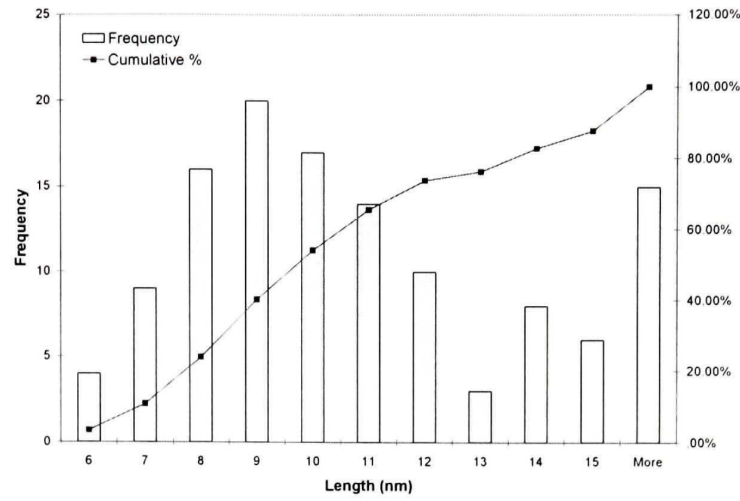


Figure 4.25. Transmission electron micrographs of modified coupons subject to (a) no natural aging, (b) 12 hours and (c) 20 hours and artificially aged 6 hours at 155°C

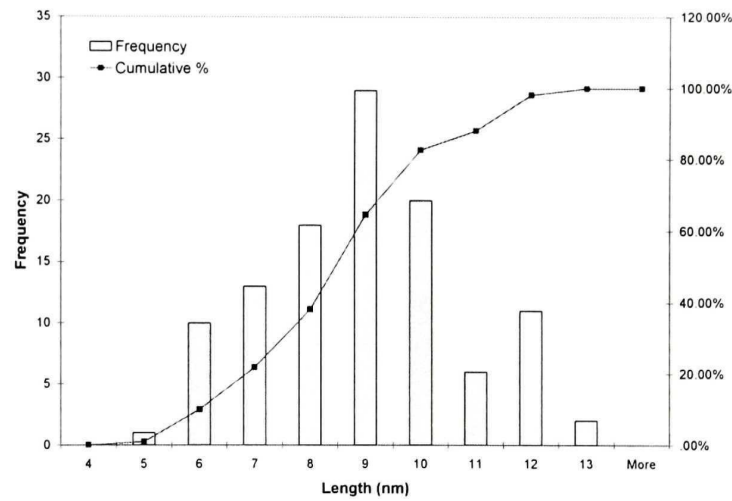
Table 4.6. Average length and diameter of the precipitates subjected to different natural aging conditions and artificially aged 6 hours at 155°C

Natural Aging Time	Average length	Average diameter
(h)	(nm)	(nm)
0	10.4±3.2	2.7±0.7
12	8.5±1.7	1.7±0.3
20	8.9±2.3	1.7±0.3

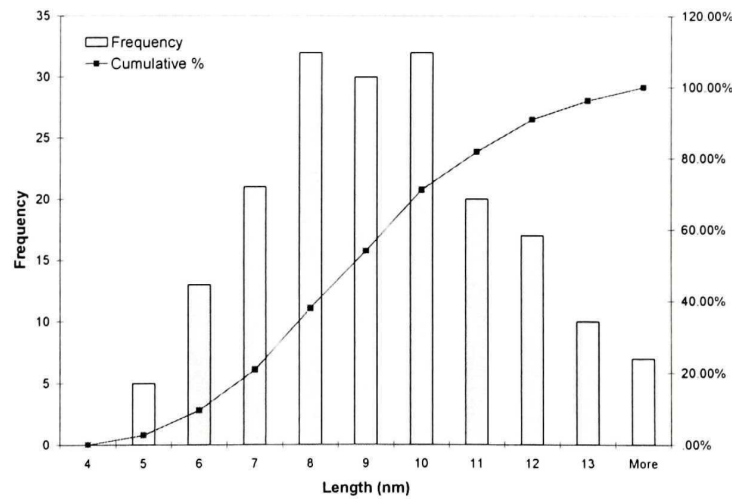
Electron diffraction patterns obtained from coupons natural aged at different conditions and artificially aged at 155°C are shown in Figure 4.25. Like in the other conditions previously evaluated, the diffraction pattern indicates coherency with the matrix. The measured values of the lattice parameter gave values of 4.01 ± 0.02 Å, 4.05 ± 0.02 Å and 4.05 ± 0.03 Å for coupons with 0, 12 and 20 hours of natural aging respectively. Again, as in the previous cases the differences between the measured values of the lattice parameter and the lattice parameter of pure aluminum are believed to come from measurement uncertainties.



No natural aging

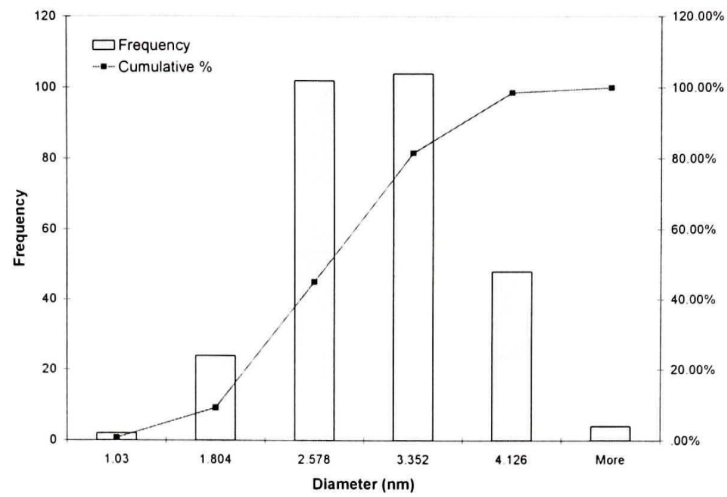


12 hours of natural aging

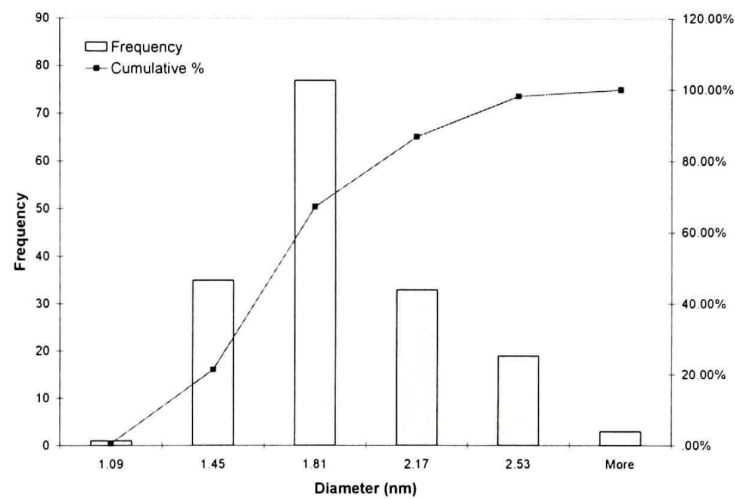


20 hours of natural aging

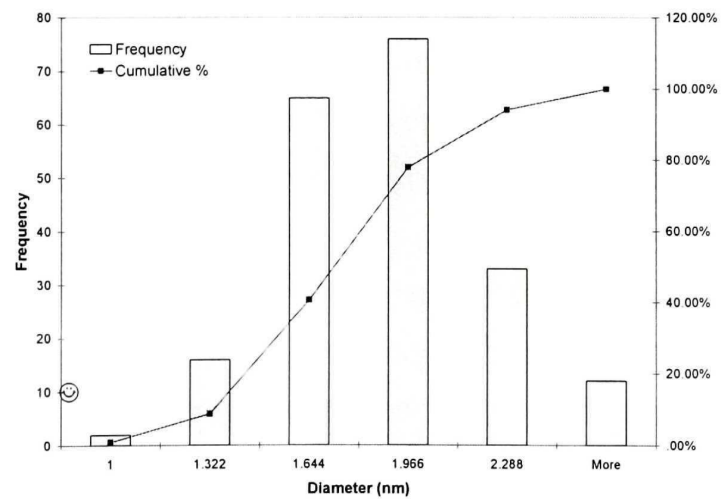
Figure 4.26. β'' - Mg_2Si length distribution in coupons subjected to different natural aging conditions and artificially aged 6 hours at 155°C



No natural aging



12 hours of natural aging



20 hours of natural aging

Figure 4.27. β'' - Mg_2Si diameter distribution in coupons subjected to different natural aging conditions and artificially aged 6 hours at 155°C

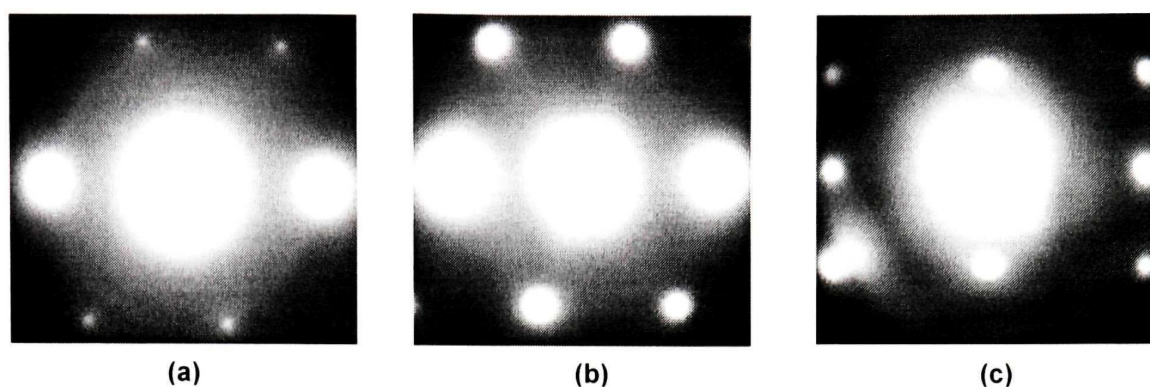


Figure 4.28. Electron diffraction patterns of modified coupons natural aged for: (a) 0 hours, (b) 12 hours and (c) 20 hours and artificially aged 6 hours at 155°C

The behaviour of the electrical conductivity as a function of natural aging time of coupons artificially aged at 170°C is shown in Figure 4.29. It is clearly seen that the electrical conductivity decreases up to 12 hours of natural aging followed by a recovery at 20 hours. The same behaviour was found with the tensile test bars, as shown in Table 4.7. In this case, the decrease of the electrical conductivity is associated with a decrease in the yield strength of the alloy as seen in Table 4.8.

The matrix microhardness of coupons subjected to different natural aging conditions and artificially aged at 170°C is given in Figure 4.30. In this figure, a decrease in the microhardness value indicating a negative effect of natural aging up to 12 hours is observed. As natural aging continues and reaches 20 hours a clear recovery in the microhardness is observed.

Tensile test data of samples heat-treated at 170°C with different natural aging times is shown in Table 4.8. The general trend is that natural aging times up to 12 hours have a detrimental effect on the yield strength in particular with a minor effect on the tensile strength accompanied by some improvement in the elongation. When natural aging is extended to 20 hours there is a general recovery in properties. As noted previously, the data is not conclusive enough to

draw definite conclusions in the case of the UTS and elongation, due to their close values and large standard deviation.

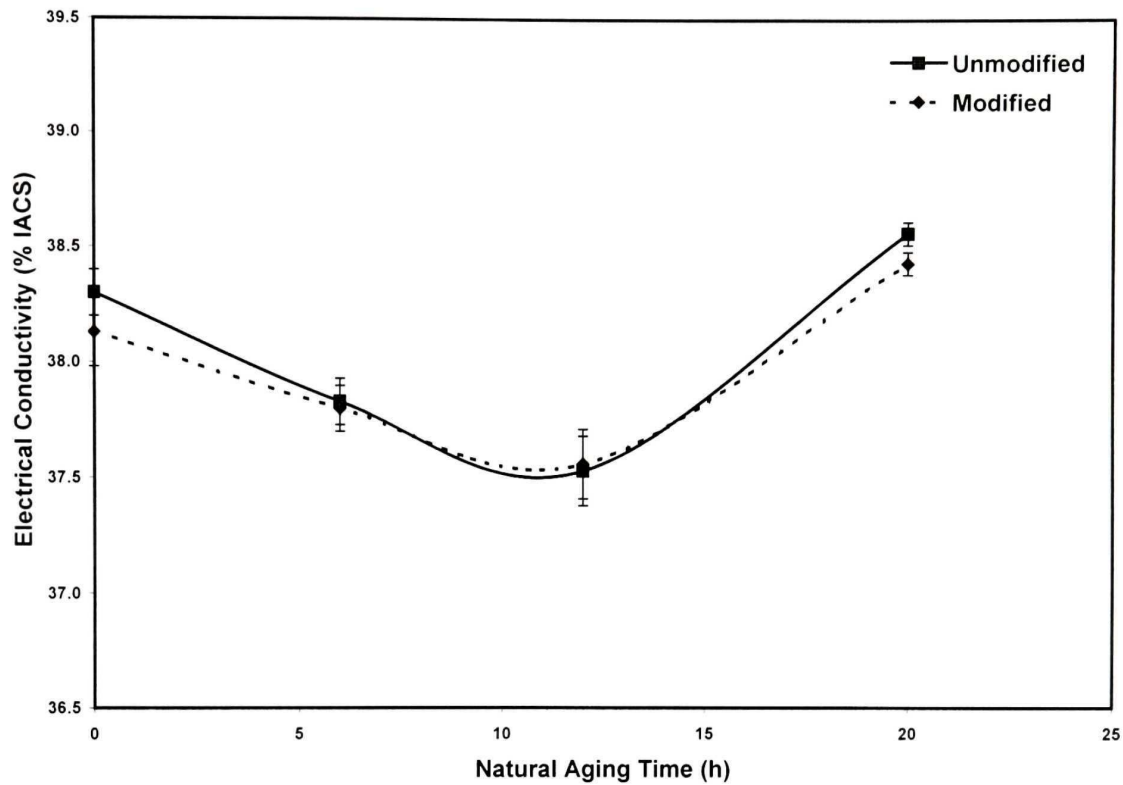


Figure 4.29. Effect of the natural aging time on the electrical conductivity of unmodified and modified coupons artificially aged 6 hours at 170 °C

Table 4.7. Electrical conductivity of artificially aged 6 hours at 170 °C tensile test bars

Condition	Electrical Conductivity (% I.A.C.S.)
No natural aging + 6 hours at 170 °C	39.5 ± 0.10
6 hours of natural aging + 6 hours at 170 °C	39.4 ± 0.20
12 hours of natural aging + 6 hours at 170 °C	39 ± 0.20
20 hours of natural aging + 6 hours at 170 °C	39.7 ± 0.10

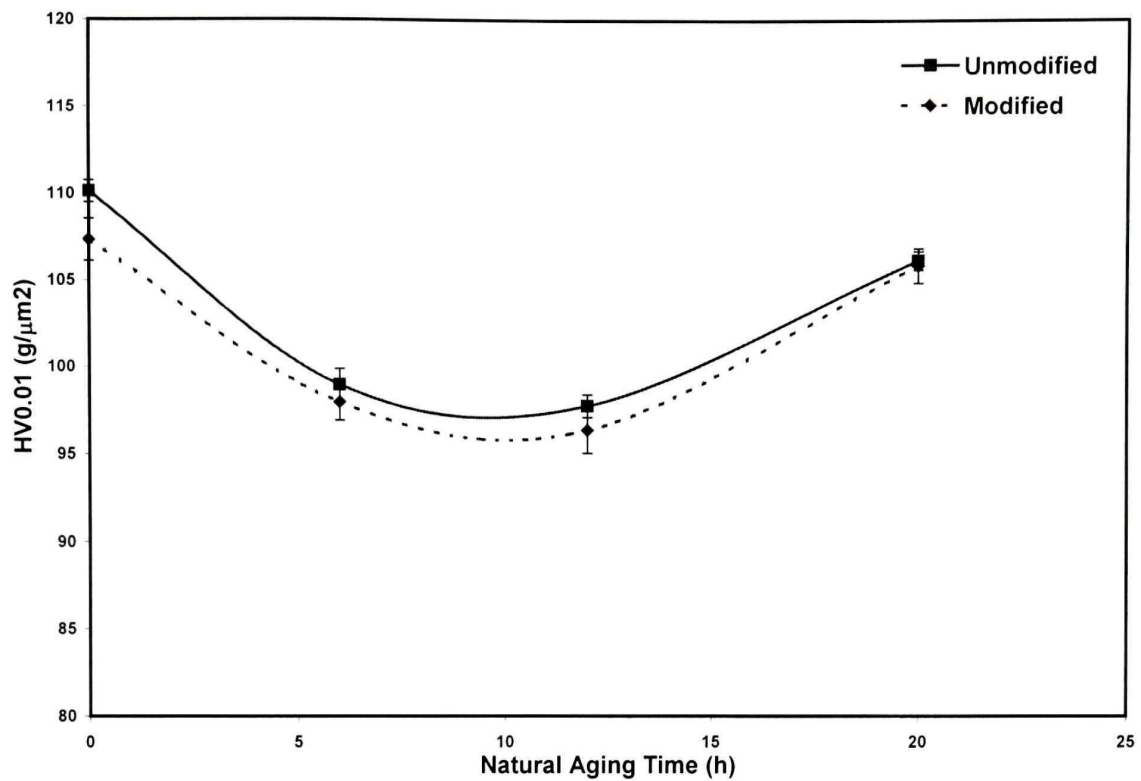


Figure 4.30. Effect of the natural aging time on the matrix microhardness of unmodified and modified coupons artificially aged 6 hours at 170°C

Condition	UTS (MPa)	YS (MPa)	Elongation (%)
No natural aging + 6 hours at 170 °C	312.1 ± 2.0	248.8 ± 4.6	7.23 ± 1.34
6 hours of natural aging + 6 hours at 170 °C	307.5 ± 3.7	243.6 ± 3.2	8.3 ± 3.2
12 hours of natural aging + 6 hours at 170 °C	299.1 ± 5	218.1 ± 3.9	9.39 ± 1.12
20 hours of natural aging + 6 hours at 170 °C	309.9 ± 1.7	248.3 ± 4.7	6.12 ± 1.35

TEM micrographs showing the Mg_2Si precipitates present in the aluminum matrix after different natural aging conditions and artificial aging of 6 hours at 170°C are shown in Figure 4.31. Precipitate length and diameter measurements on at least 15 of these pictures were done. The results are presented in Table 4.9. In these results, it is noted that as natural aging occurs both the average length and diameter tend to decrease up to 12 hours. A recovery in their size is observed at 20 hours.

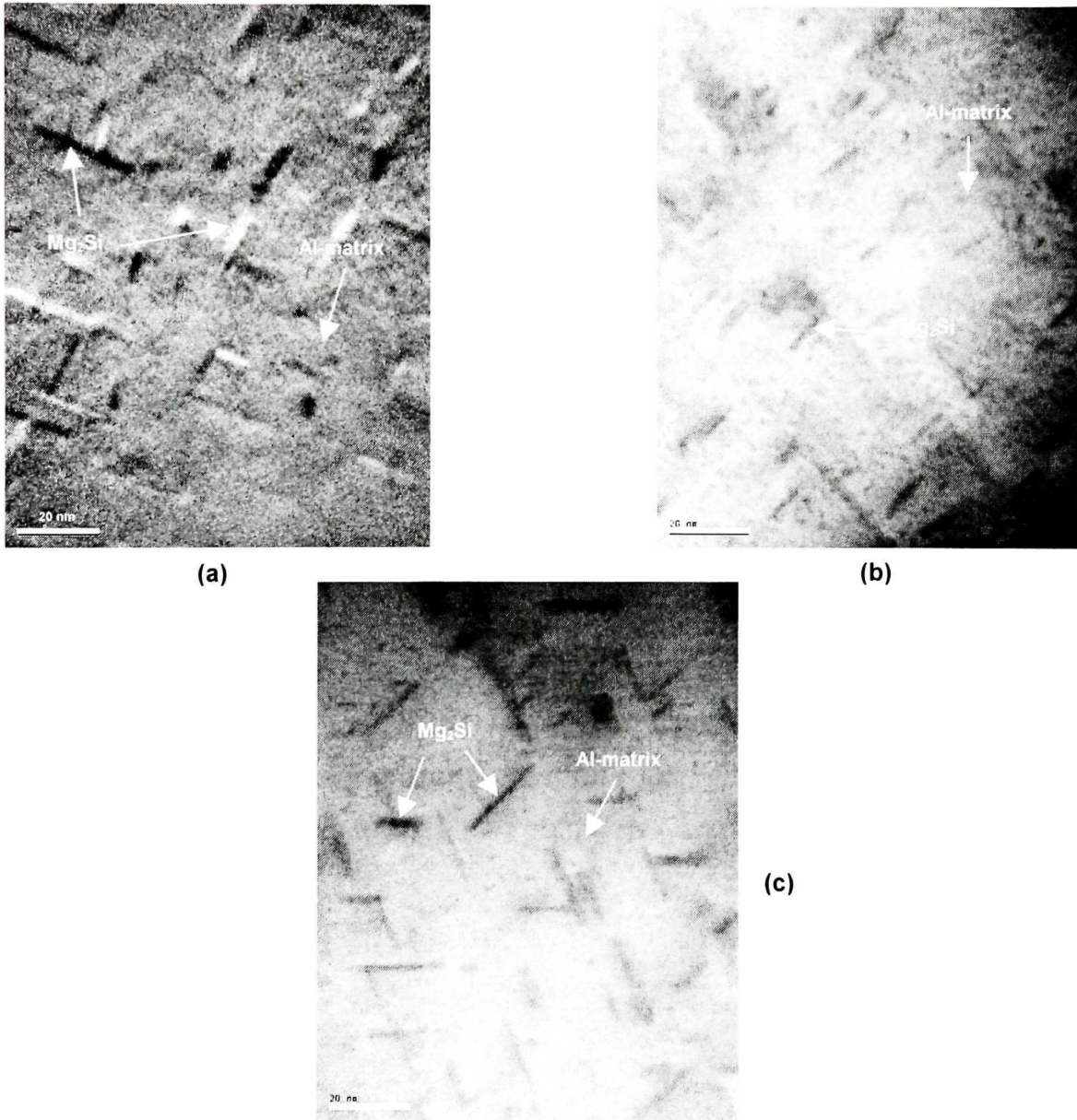


Figure 4.31. Transmission electron micrographs of modified coupons subject to (a) no natural aging, (b) 12 hours and (c) 20 hours and artificially aged 6 hours at 170°C

Table 4.9. Average length and diameter of the precipitates subjected to different natural aging conditions and artificially aged 6 hours at 170°C

Natural Aging Time	Average length	Average diameter
(h)	(nm)	(nm)
0	22.1±4.3	3.3±0.5
12	13.0±4.1	2.5±0.5
20	16.7±3.1	2.9±0.5

The length and diameter distributions are shown in the histograms in Figures 4.32 and 4.33. Regarding the length distribution, it is seen that with no natural aging the distribution is wide, indicating the presence of small and large precipitates. As natural aging advances to 12 hours a shifting to the left side of the histogram is observed indicating a decrease in the size of the precipitates. With 20 hours of natural aging an increase in the length is observed. The diameter distribution with respect of natural aging is again very similar to that of the length.

As in previous cases, the lattice parameter was calculated from the electron diffraction patterns shown in Figure 4.34. The results, 4.06 ± 0.02 Å, 4.03 ± 0.03 Å and 4.04 ± 0.02 Å for 0, 12 and 20 hours of natural aging respectively, are again very close to that of pure aluminum. Once again the differences are believed to be from measurement uncertainties. On the other hand, the diffraction pattern indicates that the precipitates are coherent with the matrix.

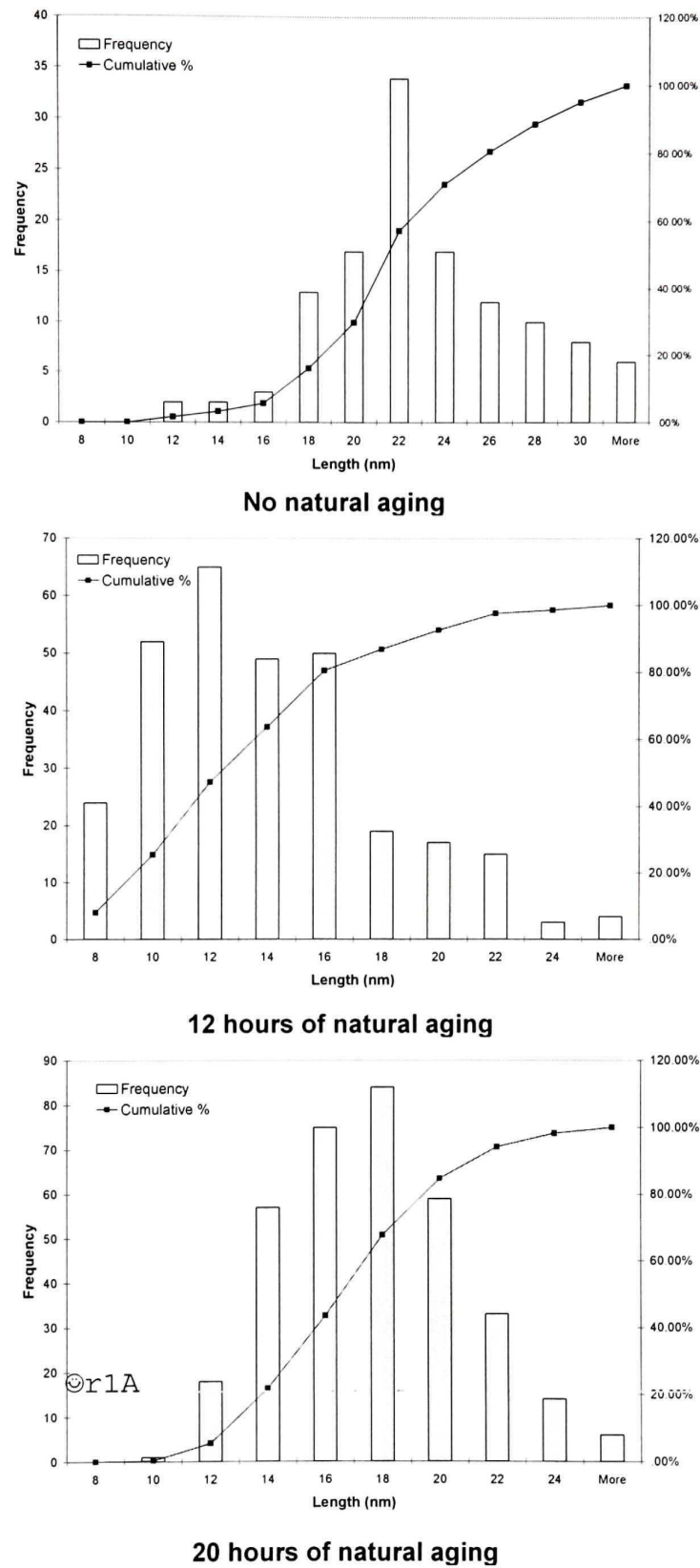
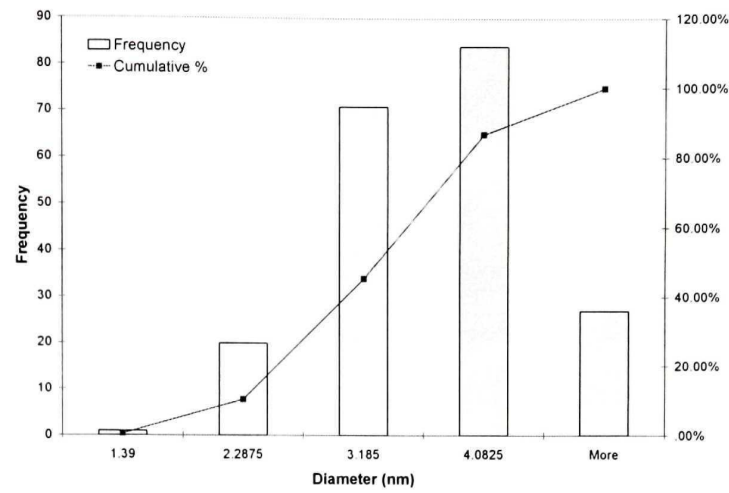
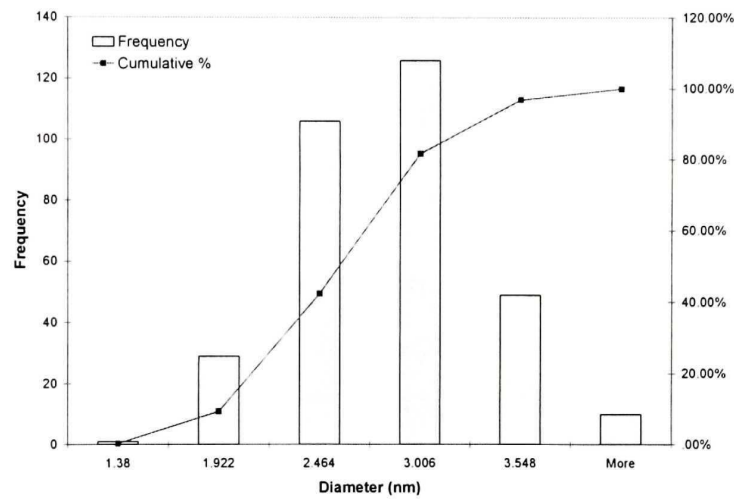


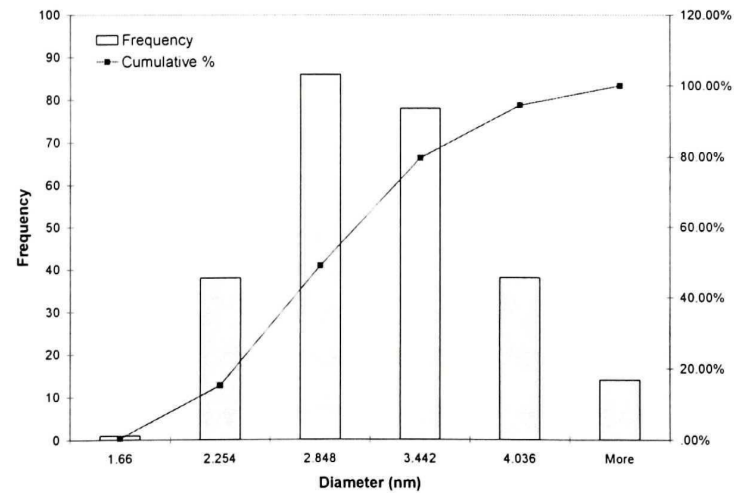
Figure 4.32. β'' - Mg_2Si length distribution in coupons subjected to different natural aging conditions and artificially aged 6 hours at 170°C



No natural aging



12 hours of natural aging



20 hours of natural aging

Figure 4.33. β'' - Mg_2Si diameter distribution in coupons subjected to different natural aging conditions and artificially aged 6 hours at 170°C

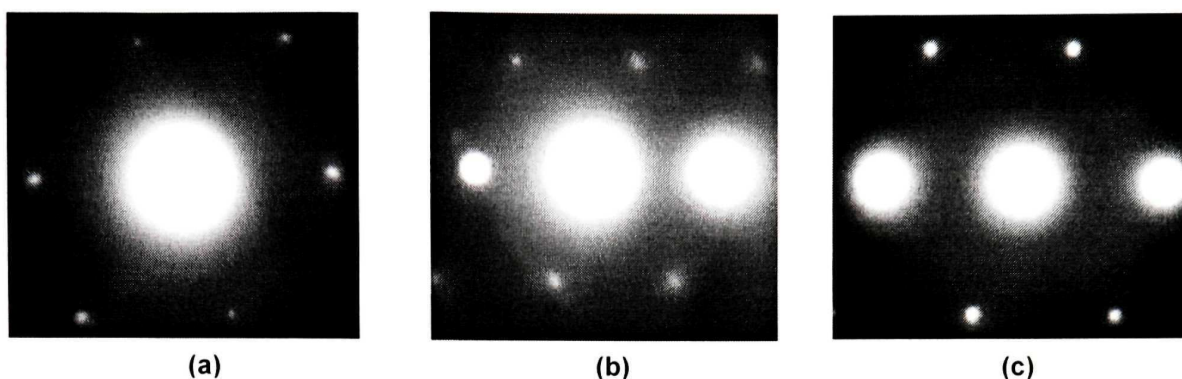


Figure 4.34. Electron diffraction patterns of modified coupons natural aged for: (a) 0 hours, (b) 12 hours and (c) 20 hours and artificially aged 6 hours at 170°C.

In order to more deeply understand the influence of natural aging, a study of the electrical conductivity and microhardness of unmodified coupons artificially aged at 185°C was also done.

Figure 4.35 shows the effect of natural aging time on the electrical conductivity of the coupons artificially aged at 185°C. It is seen that the conductivity increases as natural aging time increases. This behaviour indicates an increased precipitation with natural aging. The effect of natural aging time on the matrix microhardness is given in Figure 4.36 where a slight decrease in its value is seen with 6 hours of natural aging. A recovery in the microhardness value is observed with natural aging times of 12 and 20 hours.

From the results obtained from the electrical conductivity measurements it was expected to find an improved microhardness with natural aging due to the increased precipitation, but this was not the case. It is a fact that when the artificial aging is carried out at a relatively high temperature not only does the precipitation of Mg_2Si occurs, but that of Si also occurs [68,76]. The Si precipitation contributes to an increase in the value of the electrical conductivity and in a way masks the real effect of the natural aging. It is also a fact that this behaviour is not seen at lower artificial aging temperature due to their incubation condition [88].

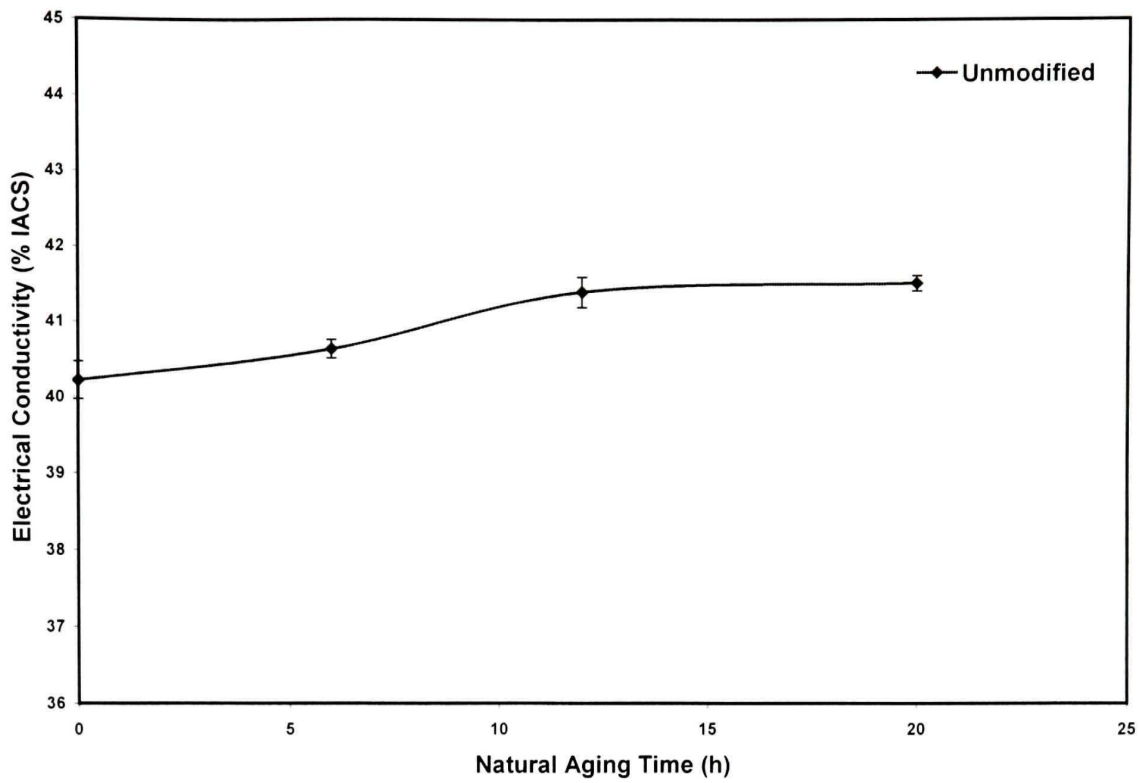


Figure 4.35. Effect of natural aging time on the electrical conductivity of unmodified coupons artificially aged 6 hours at 185 °C

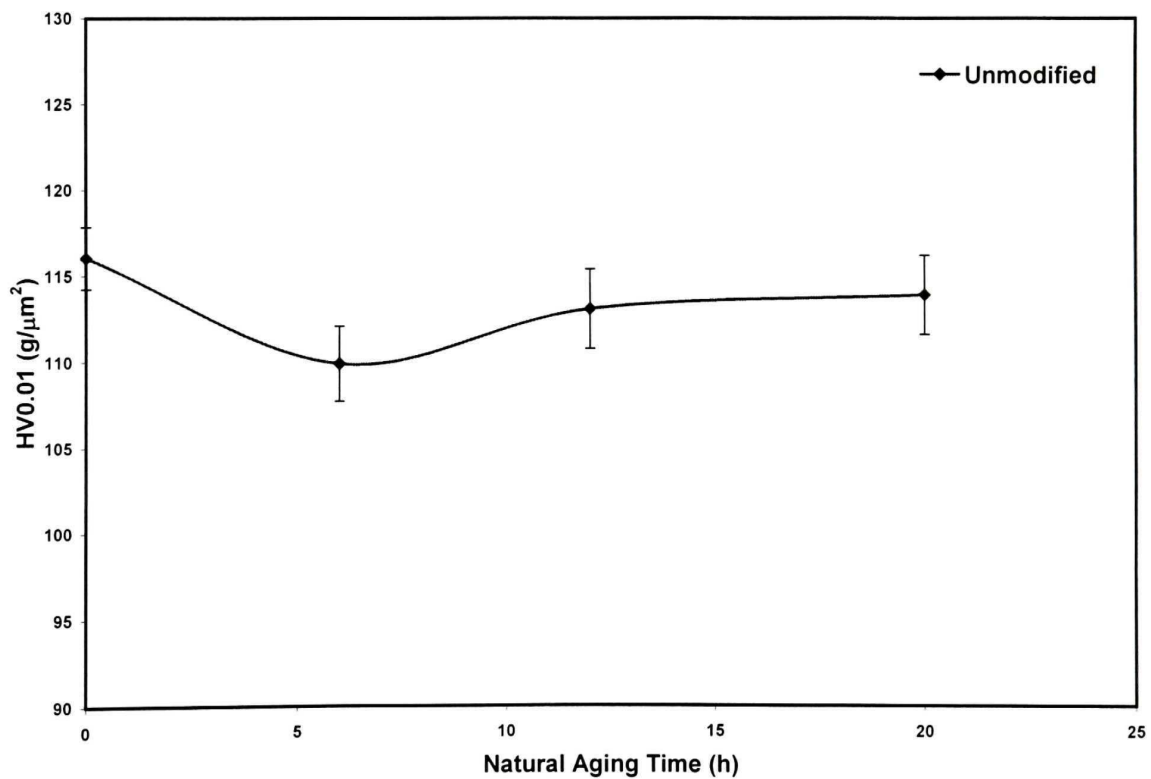


Figure 4.36. Effect of natural aging time on the matrix microhardness of unmodified coupons artificially aged 6 hours at 185°C

The analysis of the results obtained in this section indicates that the behaviour of Al-Si-Mg alloys subjected to natural aging will depend on two phenomena occurring at the same time: (a) a slow formation and growth of precipitates, and (b) the decrease in supersaturation.

In the case of the samples artificially aged after quenching the achievable properties will be completely dependant on the precipitation process that can be obtained from a highly supersaturated matrix (high driving force). On the other hand, when natural aging occurs, the precipitation will be a function of the small precipitates (clusters) formed with natural aging and the precipitates that can be formed from a solid solution with a markedly decreased supersaturation. Thus, when samples are natural aged and then artificially aged at 155°C, their properties will be lower due to the fact that the driving force for precipitation will decrease, making the precipitation process more difficult and the contribution of the precipitates to the final properties less than if no natural aging were to occur.

When artificial aging temperatures higher than 155°C (i.e. 170°C and 185°C) are used after natural aging a recovery in the properties after a certain time of natural aging is observed. This is believed to happen due to the increased precipitation from the solid solution and the increased growth of the clusters (precipitates) formed during natural aging. The recovery in properties will be faster at higher temperature due to the increased growth rate of the precipitates.

4.5 Artificial Aging

The influence of different artificial aging temperatures and times on the electrical conductivity is shown in Figure 4.37. In this figure, it is easily observed that the electrical conductivity increases as the treatment temperature and time increases. This behaviour can be explained by the fact that the electrical conductivity is highly affected by the quantity of solute in the matrix. Increased precipitation will result in higher conductivity values. Regarding the comparison between unmodified and modified coupons at 155°C no differences between both conditions are observed. At 170°C a small difference in the conductivity value is observed with 9 hours of treatment. When the coupons are aged at 236°C consistent differences in the conductivity value between unmodified and modified coupons are observed. This clearly indicates an influence of strontium on the precipitation behaviour of AlSiMg alloys.

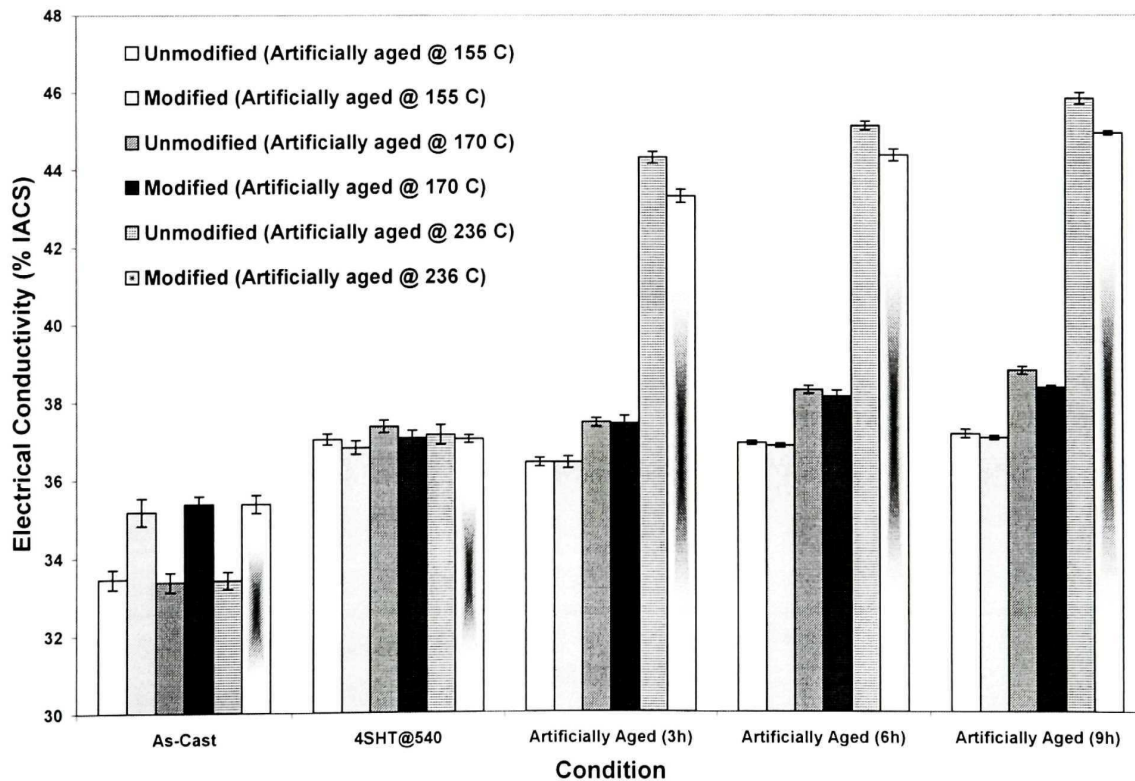


Figure 4.37. Electrical conductivity values of unmodified and modified coupons during different stages of the heat treatment

Figure 4.38 shows the value of the matrix microhardness of samples artificially aged at different temperatures and times. It can be observed that the higher microhardness values are observed with an aging temperature of 170°C. Lower microhardness values are obtained with an aging temperature of 155°C. It is also observed that with an aging at 236°C overaging is achieved. At 170°C a maximum microhardness value is achieved with 6 hours of treatment time. At 155°C a slow microhardness value increase with time is observed. At 9 hours of treatment a value of approximately 104 g/μm² is achieved. This indicates that more than 9 hours of treatment are needed to reach the maximum microhardness. At 236°C a continuous decrease of microhardness with time is observed. The effect of strontium on microhardness is only observed at high temperature and resulted in an increase of less than 5% in the microhardness.

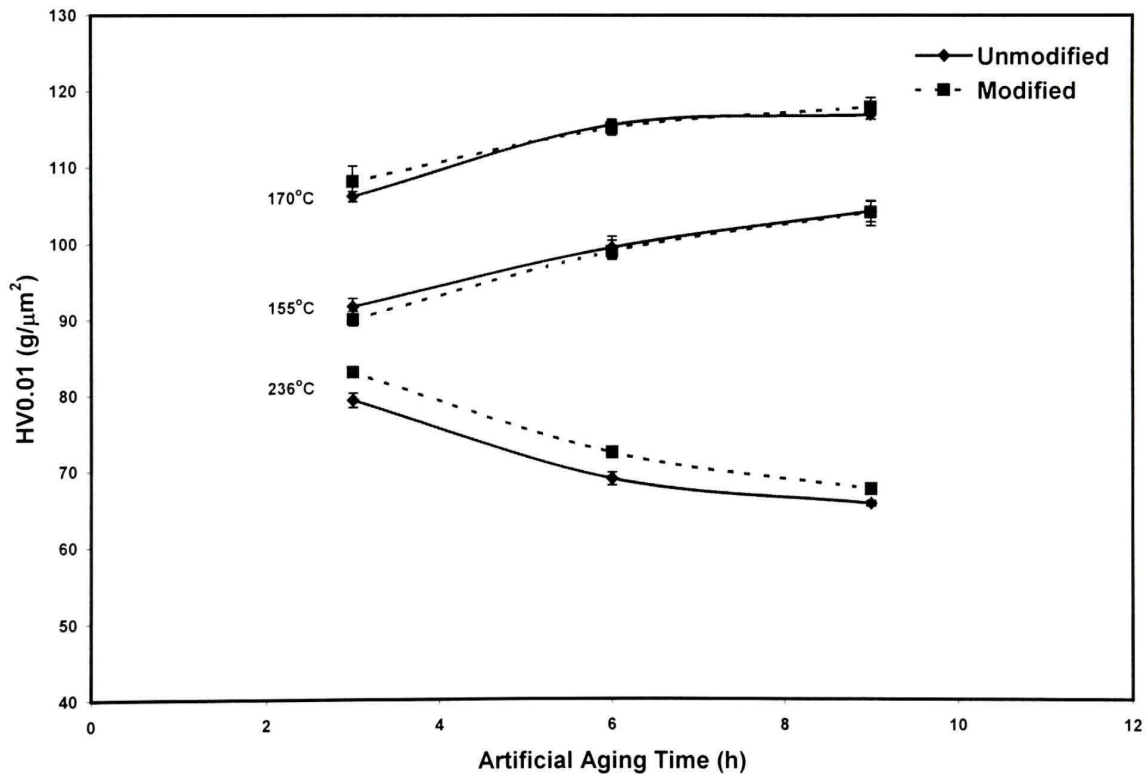


Figure 4.38. Matrix microhardness values of unmodified and modified coupons artificially aged at different temperatures and times

Figure 4.39 shows the average precipitate size measured in samples artificially aged at 155°C and 236°C at different times. It is clearly noted that the precipitation kinetics at the low temperature is very slow indicating that the necessary time to reach maximum hardness will be long. On the other hand, coupons artificially aged at higher temperature will reach maximum properties in a short time, but overaging is also reached sooner.

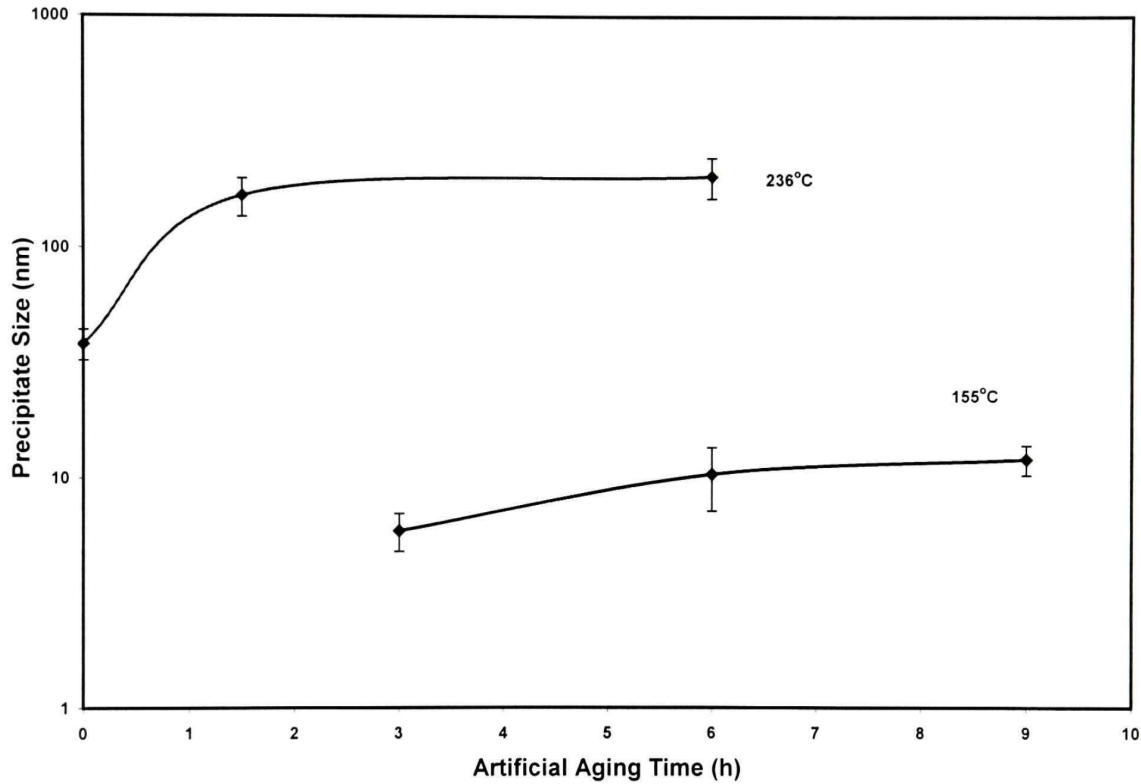


Figure 4.39. Average precipitate size values of modified coupons artificially aged at 155°C and 236°C at different times

TEM micrographs showing the Mg_2Si precipitates present in modified coupons artificially aged for 6 hours at 155°C, 170°C and 236°C are shown in Figure 4.40. In these images, it is seen that the length of the precipitates increased with temperature. The average length of the precipitates is given in Table 4.10. Moreover, Figure 4.41 shows that the precipitate length increases in an exponential fashion as the alloy moves from an aged to an overaged condition.

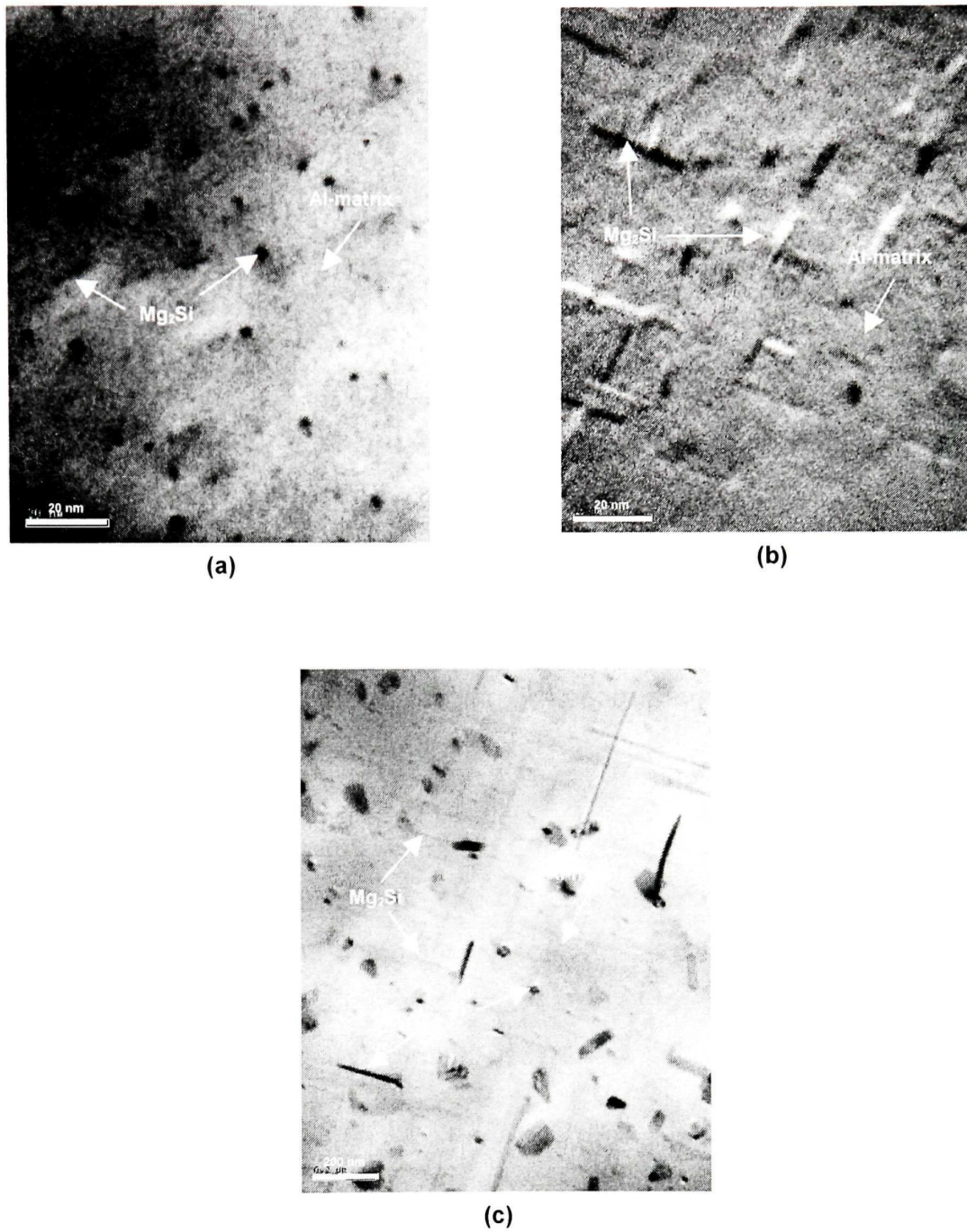


Figure 4.40. Transmission electron micrographs of modified coupons artificially aged at (a) 155°C, (b) 170°C (c) and 236°C for 6 hours

Table 4.10. Average length of the precipitates artificially aged at 155°C, 170°C and 236°C for 6 hours

Artificial Aging Temperature (°C)	Average length (nm)
155	10.4±3.2
170	22.1±4.3
236	203.2±40.8

Figure 4.42 shows an Arrhenius plot constructed in order to calculate the activation energy (E_a) needed to carry out the precipitate growth process. This plot was constructed considering the change of the precipitate length as seen in Table 4.10. It is important to notice that this plot is the most basic way to determine the activation energy and it does not give the total value of the activation energy required for the precipitation process, since precipitation includes in our case nucleation and growth.

The obtained activation energy of 65.4 KJ/mol is lower when compared to the activation energy necessary to carry out the precipitation hardening process ($E_a=130$ KJ/mol) [89]. As mentioned above this activation energy does not consider the energy necessary for the dispersion of the precipitates (nucleation). This reinforces the fact that hardening is not only dependent on the precipitate size but also on precipitate distribution.

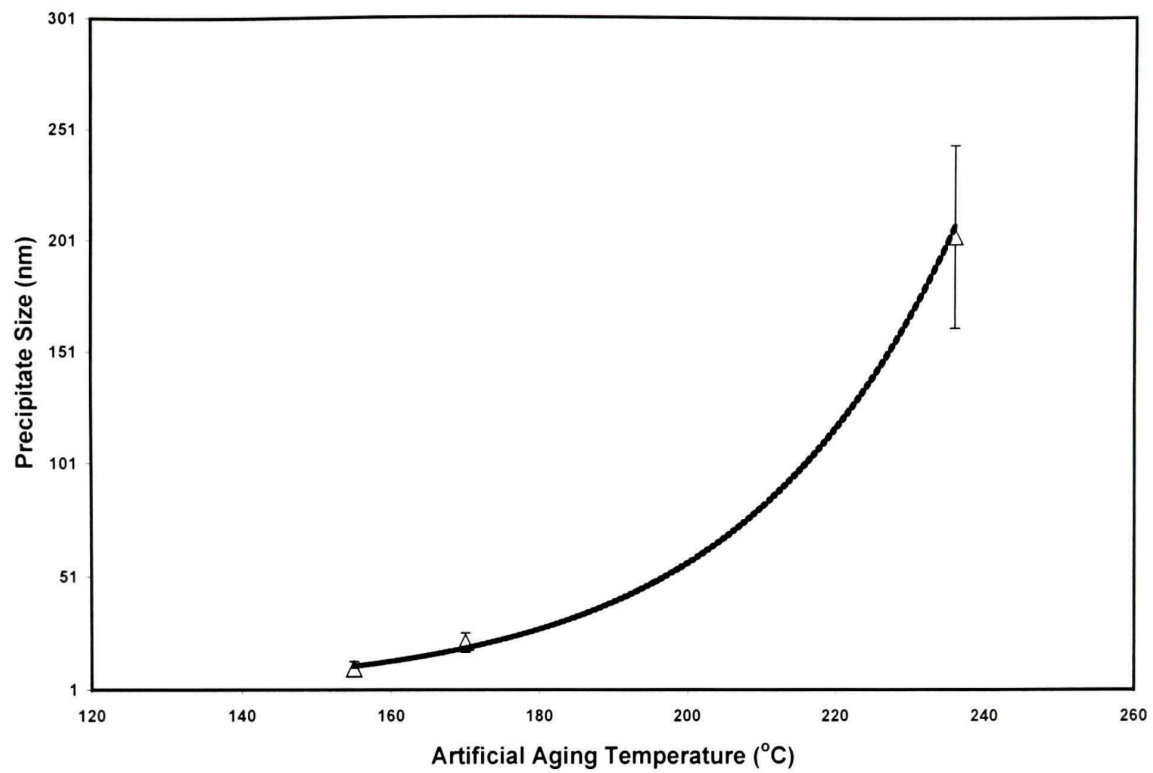


Figure 4.41. Average length of the Mg_2Si precipitates in modified coupons as a function of the artificial aging temperature

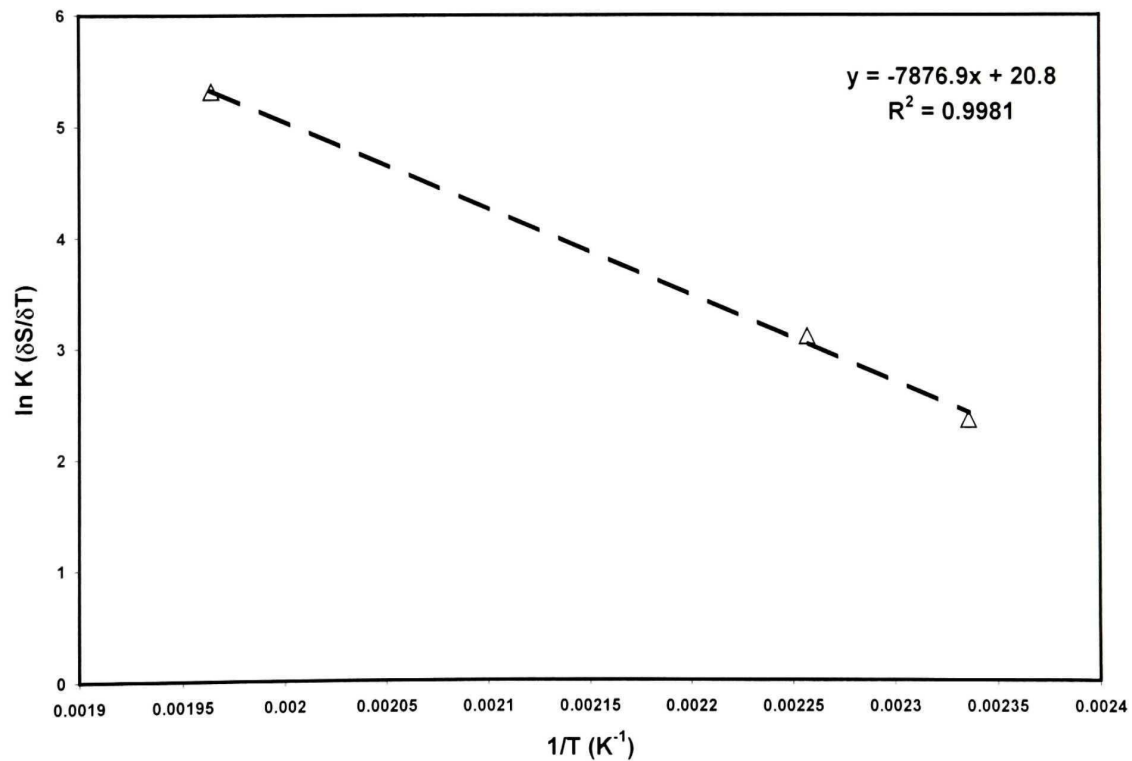


Figure 4.42. Arrhenius plot used for the calculation of the activation energy needed for Mg_2Si precipitate growth

Another very interesting observation that can be made in Figure 4.40 (c) is that silicon precipitates are also observed besides the Mg_2Si in the sample artificially aged at 236°C . This is corroborated in Figure 4.43 where a line scan and an EDS analysis of the particles present at this condition are shown. In order to develop a better understanding of the precipitation process occurring in the Al-Si-Mg alloys a deeper analysis of the phenomena occurring at 236°C was carried out.

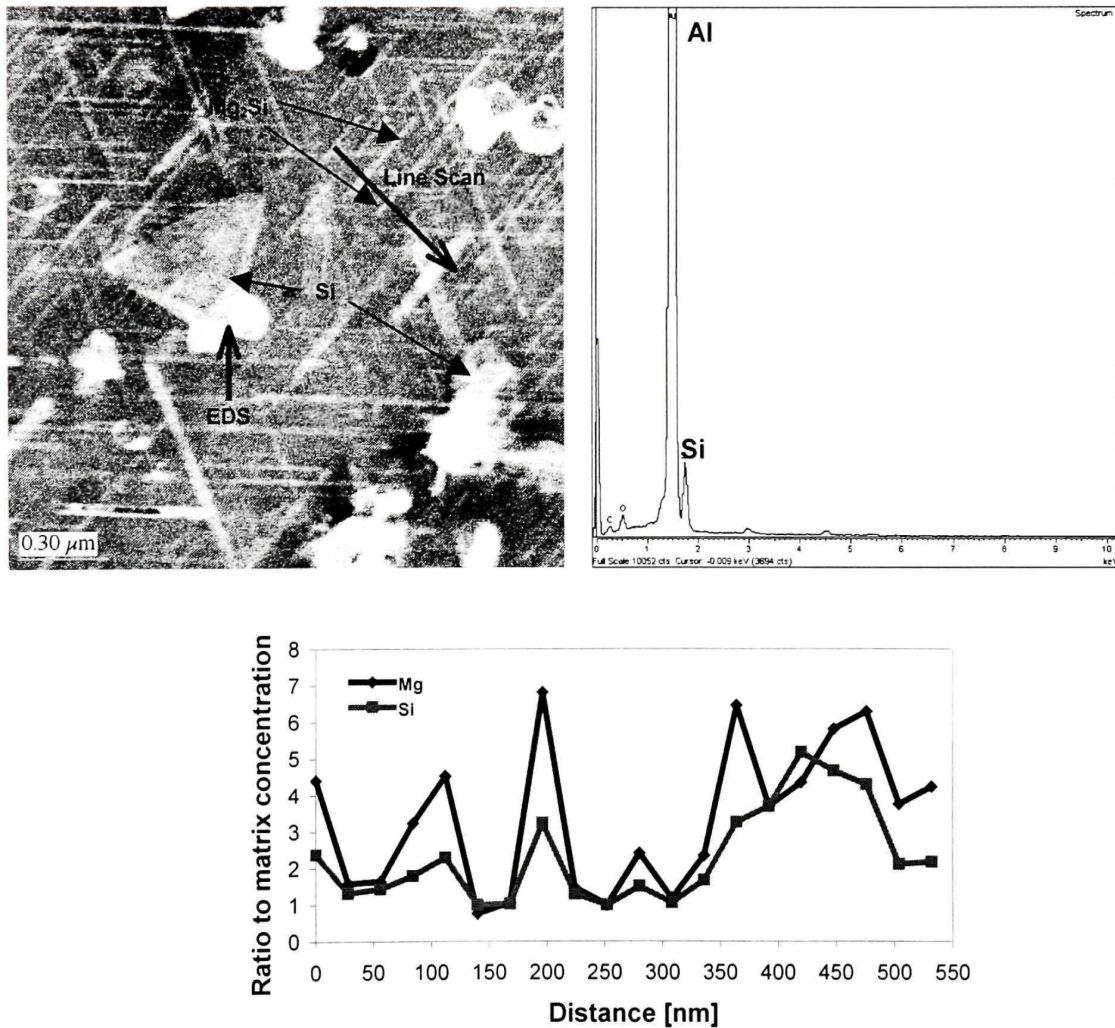


Figure 4.43. Line scan and EDS analysis showing the composition of the particles present in samples artificially aged at 236°C

In Figure 4.44 the electrical conductivity of unmodified and modified coupons subjected to an artificial aging temperature of 236°C at different times is shown. It is observed that the modified coupons consistently show a lower electrical conductivity value than those unmodified indicating that strontium plays a role in the precipitation process of Al-Si-Mg alloys.

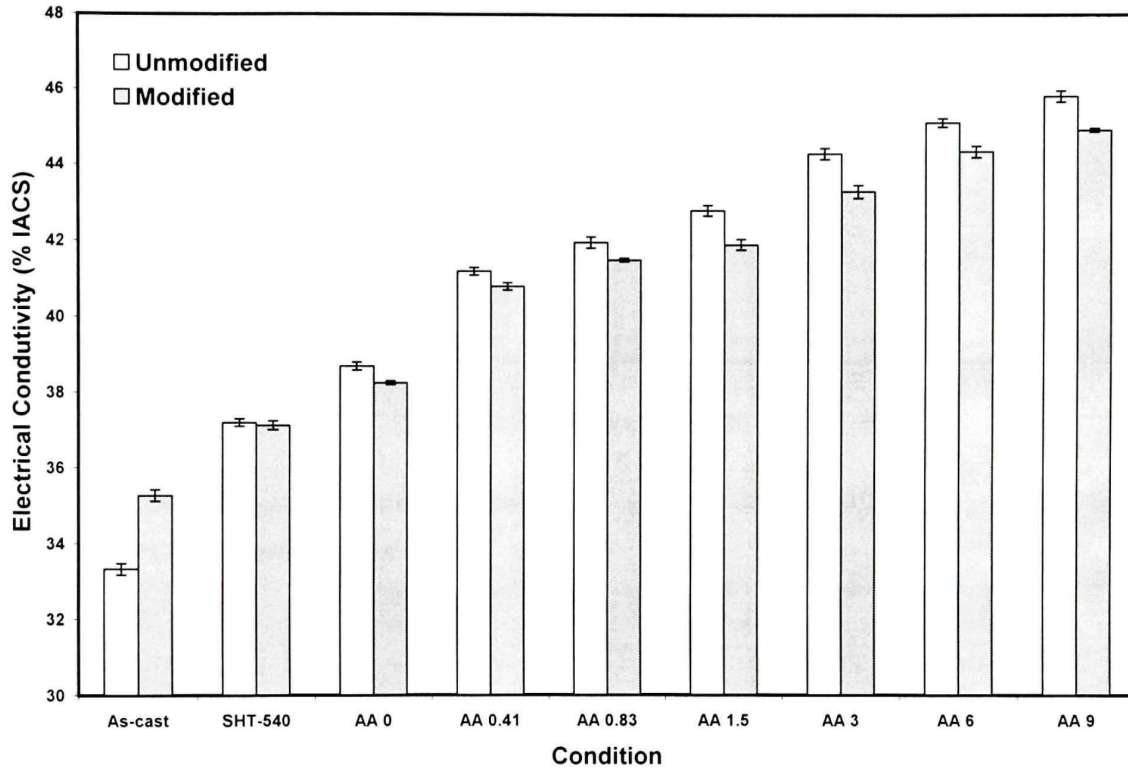


Figure 4.44. Electrical conductivity values of unmodified and modified coupons artificially aged at 236°C at different times

Figure 4.45 shows the influence of different artificial aging times at 236°C on the matrix microhardness of unmodified and modified coupons. In this figure it can be observed not only that the microhardness decreases as the artificial aging proceeds, but also that the unmodified coupons consistently present a lower microhardness value. This again indicates that the strontium modification plays a role in the precipitation process of this alloy. Moreover, it is worthy to note that the coupons heat-treated for only the time necessary to reach 236°C (time = 0) showed a microhardness similar to those reached with 9 hours of aging at 155°C.

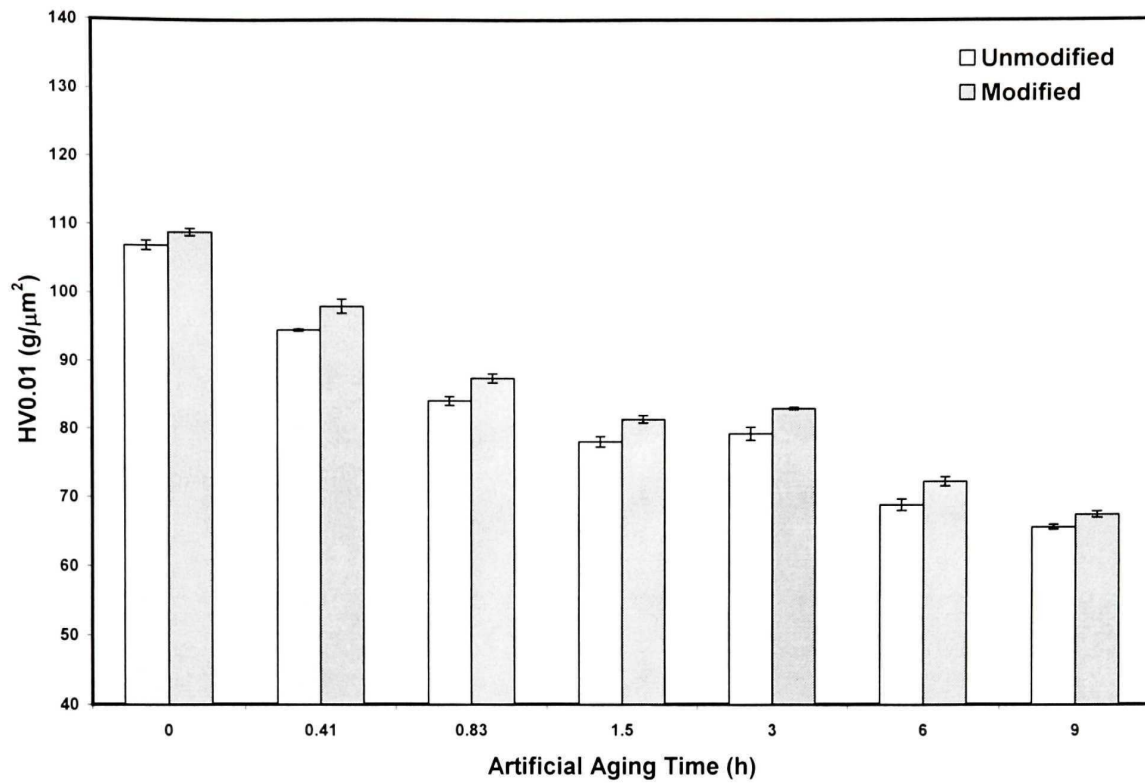
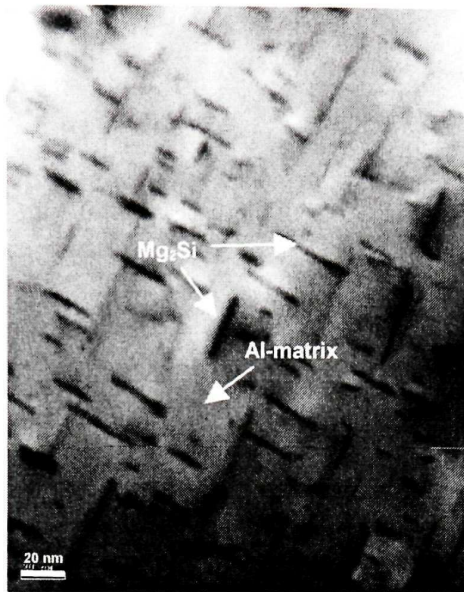
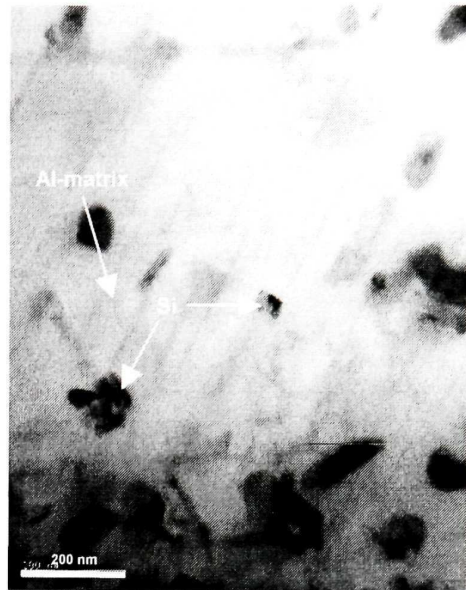


Figure 4.45. Matrix microhardness values of unmodified and modified coupons artificially aged at 236°C at different times

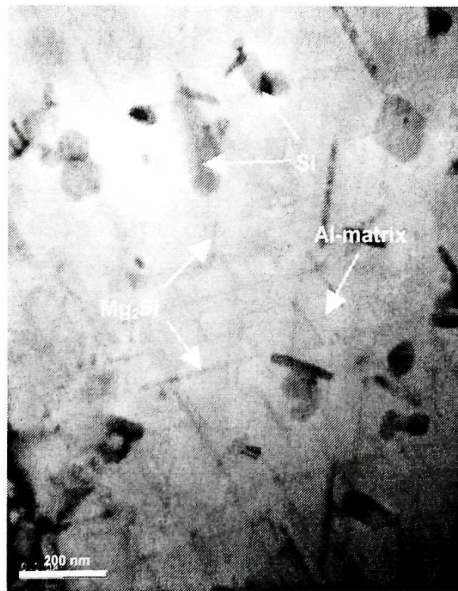
Figure 4.46 and 4.47 show the TEM micrographs of unmodified and modified coupons artificially aged for 0, 1.5 and 6 hours at 236°C. The analysis of these micrographs allows some conclusions to be reached about the role of strontium modification in the precipitation process of Al-Si-Mg alloys. The average precipitate length was measured in at least 10 different photos in each condition. The measurements revealed that the average Mg_2Si precipitate length of unmodified and modified alloys does not change after approximately two hours of aging at 236°C. This is shown in Figure 4.48.



0 Hours

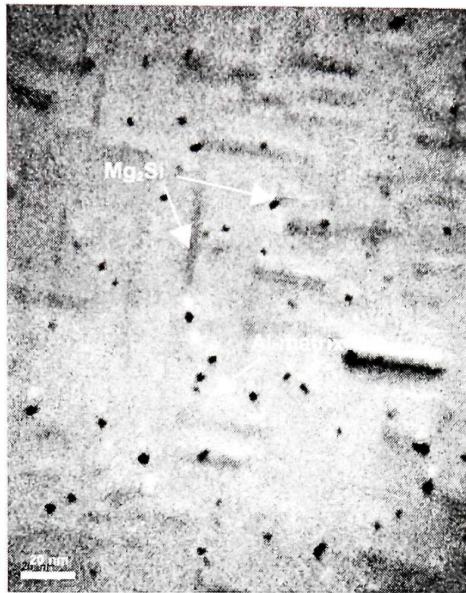


1.5 Hours

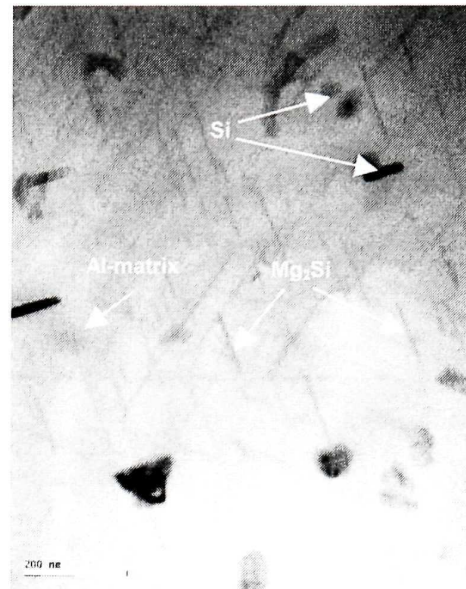


6 Hours

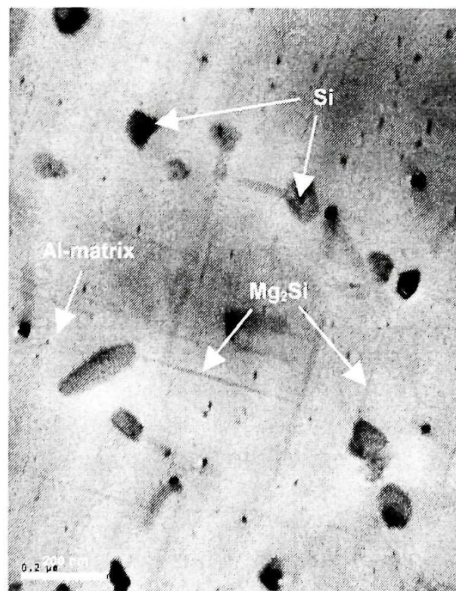
Figure 4.46. TEM micrographs of unmodified coupons artificially aged at 236°C at different times



0 Hours



1.5 Hours



6 Hours

Figure 4.47. TEM micrographs of modified coupons artificially aged at 236°C at different times

The second observation made on the TEM micrographs was that unmodified samples contained larger quantities of silicon precipitates. In order to verify this observation, image analysis of the area percentage occupied by silicon was carried out on 10 micrographs. The results obtained indicate that indeed unmodified samples release more silicon from the matrix than the modified ones. This is shown in Figure 4.49. The meaning of these results is that the strontium does affect the precipitation process of the Al-Si-Mg alloys by suppressing the precipitation of silicon out of the aluminum matrix.

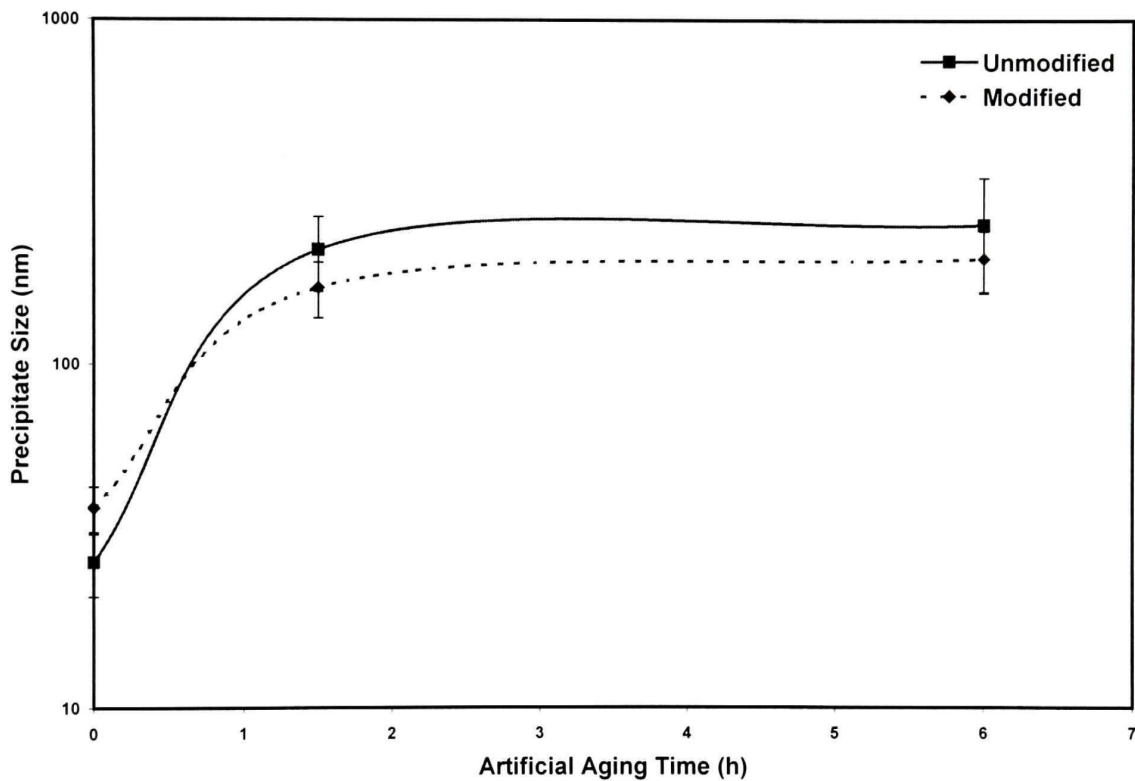


Figure 4.48. Average precipitate length of unmodified and modified coupons artificially aged at 236°C at different times

Another important observation made during this work was that a high artificial aging temperature (236°C) combined with the time necessary to heat up the furnace from room temperature to 236°C, was enough to obtain microhardness values close to those exhibited by samples artificially aged for 9 hours at 155°C. Thus, a comparison of the mechanical properties of samples

artificially aged at 155°C for 6 hours and those artificially aged at 236°C for 30 minutes.

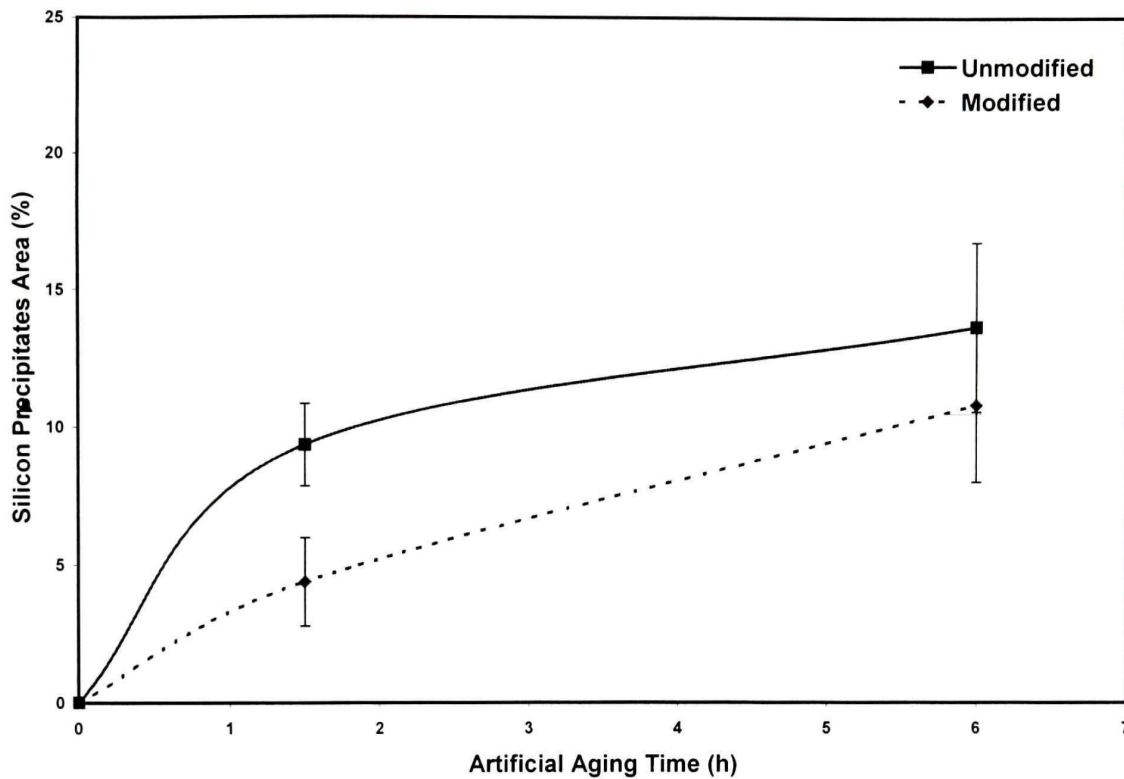


Figure 4.49. Average silicon precipitate percent area of unmodified and modified coupons artificially aged at 236°C at different times

Table 4.11 summarizes the results obtained from the tensile tests performed on test bars artificially aged at 155°C and 236°C. In this table it is noted that the UTS obtained with both treatments temperatures is very similar. On the other hand, in the case of the YS a large difference (~16%) can be observed between the treatments. This difference indicates that a more effective precipitation hardening process is occurring at 236°C. The elongation seems to decrease when the artificial aging has been carried out at 236°C. However, the decrease is not completely clear due to the large standard deviation present.

Table 4.11. Comparison of the mechanical properties of tensile bars artificially aged at 155°C and 236°C

Condition	UTS (MPa)	YS (MPa)	Elongation (%)
Unmodified @ 6 hours at 155°C	277.8± 9.4	193.3± 2.5	7.2± 3.5
Modified @ 6 hours at 155°C	286.6 ± 12.2	202.5 ± 4.7	7.0 ± 1.2
Unmodified @ 10 min at 236°C	276.1 ± 5.1	232.7± 6.3	4.2 ± 0.4
Modified @ 10 min at 236°C	282.2 ± 1.2	236.4± 4.2	5.2 ± 1.8
Unmodified @ 20 min at 236°C	269.5 ± 8.7	229.5 ± 6.1	5.1± 2.0
Modified @ 20 min at 236°C	282.3± 3.9	236.4 ± 2.8	5.3 ± 0.5
Unmodified @ 30 min at 236°C	266.7± 4.1	223.5± 2.6	6.3± 2.7
Modified @ 30 min at 236°C	276.8 ± 1.5	228.1 ± 4.7	5.7 ± 2.0

Chapter 5

PRACTICAL IMPLICATIONS AND CONCLUSIONS

As a result of this research work, a new window of opportunities for the heat treatment of Al-7%Si-0.3Mg has been opened. Currently, the T6 and T61 heat treatment of permanent mould Al-7%Si-0.3Mg alloy according to ASTM standard practices [28] consists of:

1. A solution heat treatment at $540\pm 5^{\circ}\text{C}$ for 4 to 12 hours,
2. A quench in water at 65 to 100°C ,
3. A natural aging of 8 hours,
4. Artificial aging at 155°C for 2 to 5 hours.

This research presented in this thesis indicates that significant improvement can be made to this standard process, such as:

1. The solution heat treatment time can be reduced, since very similar matrix microhardness values after artificial aging at 155°C to those of coupons solution heat treated for 4 and 6 hours, are obtained with only 2 hours of solution heat treatment (see Figure 4.4). This recommendation applies only to modified castings due to the positive effect that strontium exerts on the silicon morphology (see Figures 4.1 and 4.2). During the solution heat treatment of large castings a homogeneous soaking is mandatory to achieve this time reduction.
2. In general, the quenching conditions used in this research did not exert an important effect on the matrix microhardness value of the coupons (see Figure 4.11). Compressed air might be used as a quenching medium

depending on the mass of the casting. It is important to remember that high cooling rate quenching induces residual stresses that may cause distortions of the casting, depending on its geometry.

3. The natural aging can be manipulated to obtain the desired properties. Thus, an improved elongation after an artificial aging of 6 hours at 155°C can be obtained if the duration of natural aging is extended, however some strength is sacrificed (see Figure 4.24 and Table 4.5). An optimum elongation can be obtained with a treatment that involves 12 hours of natural aging and an artificial aging of 6 hours at 170°C. Once again the strength is sacrificed. If an optimum strength is required, and a relatively long natural aging is unavoidable, a recovery in the strength is obtained with 20 hours of natural aging (see Figure 4.30 and Table 4.8).

The use of high temperature artificial aging (~185°C) on castings that have been subjected to long natural aging times is proposed since a recovery in strength is observed (see Figures 4.24, 4.30 and 4.36).

4. The artificial aging temperature and time should be chosen depending on the desired properties. If strength is needed, higher artificial aging temperatures and shorter times should be used to obtain the desired strength (see Figure 4.38).

Also, the possible substitution of the traditional isothermal artificial aging for a continuous heating artificial aging could bring new opportunities to optimize the heat treatment time. Table 4.11 and Figure 4.45 clearly demonstrate that similar tensile properties and hardness to those obtained with traditional isothermal artificial aging are obtained with a continuous heating artificial aging from room temperature to 236°C (~20 minutes). This treatment can have a high impact on energy saving during the process and therefore in the economy of the foundry. The practical

limitation to this process is to attain a homogenous heating throughout the casting during the process.

Better properties and energy savings are seen in the future of the heat treatment of Al-7%Si-0.3Mg alloy. Moreover, the understanding of the precipitation process in this nanotechnology era, will allow better designs of the final properties by the manipulation of the size and distribution of precipitates,

Based on the theoretical and experimental investigation the conclusions that can be drawn from this research are summarized as follows:

- Shorter solution heat treatment times can be used in the heat treatment of modified Al-7%Si-0.3Mg alloy since coupons artificially aged at 155°C after a solution heat treatment of 2 h gave an improved matrix microhardness value compare to those solution heat treated at 4 and 6 h. Unmodified alloy can not be subjected to this consideration due to the negative effects that the coarse platelets of Si exert on the mechanical properties.
- A normal precipitate size distribution was found in coupons solution heat treated for 2 hours which showed an improved matrix microhardness value.
- Water quenching temperature does not seem to play a very important role in the Mg_2Si precipitation since the matrix microhardness of coupons quenched under different water quenching temperatures and conditions yield similar values. However, the precipitate size distribution might have a role in the decreased elongation associated with reduced quenching rates.
- Natural aging can be manipulated in order to obtain the desired properties. Its effect depends strongly in its duration and the subsequent artificial aging temperature.

- During the heat treatment of Al-7%Si-0.3Mg alloy not only the precipitation of the Mg_2Si occurs but also that of Si. When strontium is added to modify the morphology of the eutectic silicon, it has an effect on the precipitation as follows:
 - Strontium additions in Al-7%Si-0.3Mg alloy do not influence the precipitation kinetics of Mg_2Si .
 - Strontium additions in Al-7%Si-0.3Mg alloy retard the precipitation of Si.
- Hardening of the Al-7%Si-0.3Mg alloy is obtained with the presence of clusters to ~ 40 nm β'' - Mg_2Si precipitates. The peak hardness appears to be obtained with precipitates of ~ 30 nm size.
- Overaging is seen in the presence of Mg_2Si in the form of needles of ~ 150 nm of length and longer formed during artificial aging at 236°C .
- Only coherent precipitates contribute to the hardening of the Al-7%Si-0.3Mg alloy.
- Precipitate size distribution plays a very important role in the properties of heat-treated Al-7%Si-0.3Mg alloy subjected to natural aging.
- An activation energy based on the measurement of the precipitates size after different heat treatment conditions was calculated. A value of $E_a = 65.4$ KJ/mol was obtained.
- The time necessary to heat from room temperature to 236°C (~ 25 minutes) can be used as a continuous heating artificial aging to obtain similar tensile properties to those found in samples artificially aged for 6 hours at 155°C .

- Si requires more temperature or time to precipitate since Si precipitates begin to appear only when coupons are artificially aged at 236°C for 1.5 hours.

STATEMENT OF ORIGINALITY

The following aspects of the present work are considered to be original contributions to knowledge:

- The effect of strontium modification on the heat treatment of A356 aluminum alloy has been further understood. The use of electrical conductivity, matrix microhardness measurements and transmission electron microscopy helped to identify that strontium addition to A356 aluminum alloy does not influence the precipitation kinetics of Mg_2Si , but it does retard the precipitation of Si.
- The influence of natural aging time on the properties of heat-treated A356 aluminum alloy was studied and relationships with artificial aging temperature have been established. During natural aging supersaturation of the solid solution decreases and clusters (precipitates) are formed. The use of higher artificial aging temperatures (170°C-185°C) in castings that were subjected to natural aging allowed the recovery of the strength due to the faster growth of precipitates formed during natural aging and the enhanced precipitation from the solid solution.
- Tensile tests and matrix microhardness results obtained in this work suggest that a new approach to carry out the artificial aging can be taken. It consists of a continuous heating from room temperature to ~236°C instead of the classical isothermal process. This process allows a reduction of more than 80% of the artificial aging time necessary to obtain similar properties.

- A transmission electron microscopy study involving all the stages of the heat treatment of the A356 aluminum alloy has been done, and quantitative data obtained on the size and type of precipitates formed under various conditions obtained. The findings can be summarized as follows:
 - An influence of the solution heat treatment time on the precipitate size distribution was found. A most normal size distribution was found in samples solution heat-treated for 2 hours.
 - Quenching does not seem to greatly affect the average precipitate size, but it does affect its distribution.
 - Variations on the precipitate size of coupons artificially aged at 155°C with natural aging were detected, but the standard deviation does not allow a clear conclusion to be made. The precipitate size distribution is affected. In coupons artificially aged at 170°C a more noticeable change in the precipitate size is observed and again the size distribution is affected.
 - A hardening effect was observed in the presence of only coherent precipitates with sizes from 5 nm to 40 nm.
 - Overaging was observed in the presence of Mg₂Si needles formed at 1.5 hours at 236°C.
 - The measurement of precipitate length at different temperatures allowed a calculation of an activation energy of 65.4KJ/mol necessary for the growth of precipitates.

- Precipitation of silicon could be observed only after 1.5 hours of artificial aging at 236°C, and a difference of ~50% between unmodified and modified alloy was found. With 6 hours of artificial aging the difference is reduced to ~21%.

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Appendix 1

Typical Strain-Stress Curves for Test Bars Solution Heat Treated 4 hours at 540°C, Water Quenched at 25°C, Natural Aged for 6 hours and Artificially Aged for 6 hours at 155°C.

