
THE EFFECT OF THERMOMECHANICAL REGIM ON MICROSTRUCTURE AND WEAR RESISTANCE BEHAVIOR OF EXPERIMENTALLY CASTED Al-Si-Mg ALLOY

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ABSTRACT

AL-1.5% Si-0.8% Mg alloy has been locally prepared and used to study the feasibility of using low temperature thermo-mechanical treatment (LTMT) for improving the wear resistance. Specimens for this study have been prepared from the homogenized as-cast ingot. The specimens were subjected to solution treatment followed by plastic deformation for 8, 15 and 17 % reduction in thickness before aging at 150, 170 and 190 °C for 4 hours. The microstructure investigation showed an increase in the relative amount of Mg₂Si with increasing the degree of deformation. X-ray diffraction analysis revealed a reduction in the lattice parameter of the matrix (Al-based solid solution) of the investigated alloy with increasing the percentage reduction in thickness. Hardness measurements showed that with increasing the percentage of cold work of Al-Si-Mg alloy with excess Si leads to a decrease in the maximum attainable hardness of artificially aged articles. For naturally aged articles, the contrary was true. Wear measurements of the investigated alloy under a load of 3.9 N for 500 m counter a cast iron disc revealed that the wear rate depends mainly on the hardness values of the articles. The investigation revealed that low temperature thermo-mechanical treatment of the studied alloy is not practically feasible for wear application. On the other hand, conventional heat treatment is more feasible.

KEYWORDS: Thermo-mechanical treatment; Cold work; X-ray diffraction; Hardness measurement; Wear measurements.

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INTRODUCTION

Al-Mg₂Si alloys of the 6xxx series of Al-base alloys offer attractive advantages as candidate material in future industrial applications. These advantages include weight reduction due to the low density of Mg₂Si, better mechanical properties at medium temperatures and low cost [Zhanz et.al. 2000]. Some perspective applications are in the automotive industries for engine components such as cylinder heads, engine blocks and engine pistons [Hatab et.al. 2005]. The field of aviation also makes use of this alloy [Miao et.al 1999]. These alloys are commonly used in the age hardening condition. The wear resistance of these alloys is relatively low, however, if free Mg is present in the solid solution the amount of Mg₂Si dissolved will be lower and the aging effect too [Aouabdia et.al. 2005]. In order to use the Mg₂Si at maximum, additional Si content, above that required for the formation of Mg₂Si, is used. The excess Si is taught to enhance the wear resistance of these alloys. Thermo-mechanical treatment may cause an increase in the alloy hardness and thus in wear resistance. The tensile properties of Al-Li alloy was found to be improved by [TMT Gao et.al. 2014]. The fatigue properties of Al-Si alloy was improved by repeated [TMT Umezawa 2005]. The enhancement in mechanical properties of Al-Mg-Si alloy containing 3 wt. % Li was attributed to a more uniform distribution of increasing refined Al₃Li precipitates as a result of [TMT Din et.al. 2014]. The application of deformation was found to lowers the depth of penetration intercrystalline corrosion(ICC) and raises the fatigue characteristics at a high level of mechanical properties in Al-Mg-Si-Cu-Zn alloys [Makhsidov et.al. 2014]. This research work aims to investigate the feasibility of using thermo-mechanical treatment to improve the wear resistance of Al-Mg₂Si alloys with excess Si.

EXPERIMENTAL PROCEDURE

In order to achieve the aim of the present research work, an Al-Si-Mg alloy with excess Si content has been locally prepared. The master alloy used was Al-4.67 wt. % Si. The master alloy was melted with pure aluminum sheets under atmospheric pressure. Then Mg strips was depth in into the melt (700 gram master alloy, 1000 gram aluminum, 36 gram pure manganese). The chemical composition of such alloy is given in Table (1). In order to eliminate coring and micro segregation, caused by the relatively fast solidification, the as-cast ingot was homogenized at 530° C for six hours followed by air-cool-

ing. The homogenization temperature was selected as high as practically possible but below the eutectic temperatures for both the Al-Mg₂Si quasi-binary system ($t_{e1}=595^{\circ}\text{C}$) and the Al-Si binary system ($t_{e2}=577^{\circ}\text{C}$).

Table 1: Chemical composition analysis of the alloy used in the present research work

Element	Si	Mg	Cu	Fe	Ti	Sn	Zn	Al
Wt%	1.508	0.80	0.228	0.758	0.82	0.14	0.241	bal

The homogenized ingot was cleaned and cut into plates 45x250x5mm by means of a wire-cutting machine. Eight plates were cut from the ingot to be utilized for the thermo-mechanical treatment investigation. Faces of plate samples were ground by means of SiC abrasive paper up to 400 grit.

In the first step of the low temperature thermo-mechanical treatment (LTMT), seven plates were heated to 530°C for 2 hrs. The plates were then water- quenched from the solution treatment temperature. One plate was left untreated to represent the as – cast homogenized state. A lab-scale rolling mill was used to perform the deformation step of the LTMT. Three different thickness reductions were selected; namely 8 %, 15 % and 17 %. Larger thickness reductions were not feasible because of intensive cracking. Two plates were rolled to each of the selected thickness reductions. One plate was left without mechanical deformation to represent the solution- treatment condition.

Three different aging temperatures have been attempted within the frame of this investigation. These were 150°C, 170°C and 190°C. Equal duration annealing for 4 hours has been performed. The deformed plates of different thickness reductions and a part of the solution treated plate have been subjected to aging at each of the selected temperatures.

Furthermore, specimens of the deformed plates and the solution-treated plate have been subjected to aging at room temperature for 48 hrs.

Hardness test

Hardness has been selected to characterize the mechanical properties of the material after each step of treatment. Hardness measurements have been performed using Zwick 3112 hardness tester. Vickers hardness principle under a load of 1 kg has been utilized. At least 10 measurements have been conducted to characterize each state. Hardness has been measured for the cast

condition, the solution-treatment condition, on each of the rolled plates with different reductions of thickness, and for each of the specimens aged at various temperatures for the specified time interval.

Microstructures investigation

Specimens $10 \times 15 \text{ mm}^2$ of the homogenized as well as the solution treated alloy and the plates deformed to each of the explored thickness reductions have been cut by means of wire spark cutting machine for microstructure investigation.

The specimens were ground conventionally with SiC abrasive paper of grit № 220, 320, 400, 600, 800 in sequence. The specimens were further polished using normal (N) and fine (F) alumina paste. For revealing the microstructure etching has been performed with the polished specimens using Keller's reagent. The microstructure of both the as-polished and the etched specimens was studied and micro graphed by means of an optical microscope equipped with photographic facilities.

X-ray diffraction analysis

In order to follow any massive phase transformation, which may take place on the sub-microscopic scale during the plastic deformation of the solution treated plates of the explored thickness reductions; $20 \times 20 \text{ mm}^2$ of those plates were cut using wire spark machine. These specimens were subjected to x-ray diffraction analysis using x-ray diffractometer of the type BRNKER AXS Mode D5005. Copper k_α radiation of $\lambda = 1.540 \text{ \AA}$ has been utilized for the purpose of this work.

Wear measurements

Specimens ($4.65 \times 10 \times 20 \text{ mm}$) of the solution treated as well as of each of the deformed plates have been prepared. The surface to be wear tested has been ground using SiC abrasive paper of successive grits up to 400 grit. Wear measurements have been performed by means of locally manufactured pin on disc wear testing machine, Figure (1). Wear testing was carried out against a cast iron disc of hardness $H_v = 230$. The disc was rotating at 30 rpm affecting a sliding speed of 0.36 m/s at the centre of the specimen pin surface. Wear testing was accomplished under a force of 3.9 N for a sliding distance of 500 m . The specimens were weighed before and after the test, using a sensitive Balance type PRODIT MODEL BCA 200 (with four decimals accuracy) and the weight lost by the specimens during the test was determined.

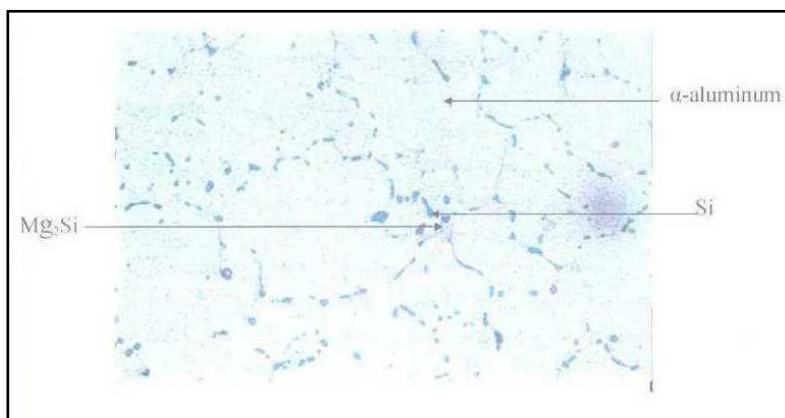


Figure 1: Locally manufactured wear testing machine

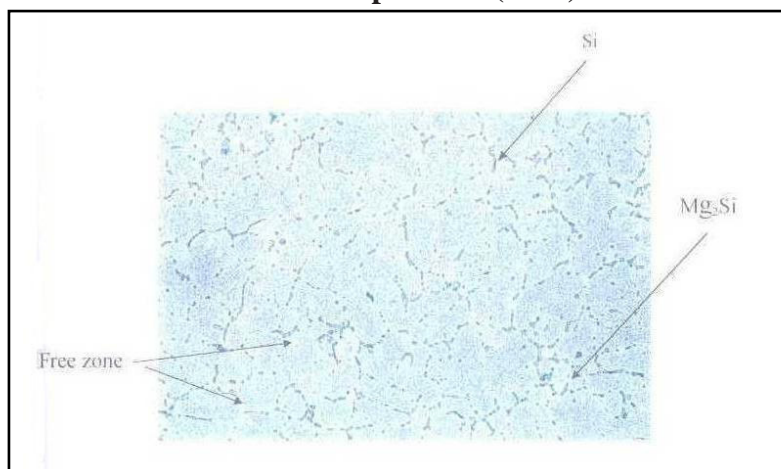
RESULTS AND DISCUSSION

The chemical composition, Table (1), indicates that the starting material had a Si+Mg content of 2.308 % which is within the frames of the majority of 6xxx series of wrought aluminum alloy. However the Mg/Si ratio equals 0.53, which is well below the stoichiometric value of 1.73 corresponding to Mg_2Si formation. This means that the structure will contain excess free Si ranging at $1.508 - 0.807/1.73 \approx 1.042$ %. Hence, free Si is expected in the microstructure of the homogenized cast material. Representative microstructures of the polished and etched surface of the starting material are represented in Figure (2). The polished microstructure, Figure (2) a, indicates grey-blue rounded or elongated particles forming a network resembling grain boundaries on the fair-grey background. Some violet fine precipitates participate in delineation of what seems to be grain boundaries. The blue-grey particles seem to be free Si crystallites solidifying at grain boundaries. The violet fine precipitates are probably those of Mg_2Si crystals also solidified at grain boundaries due to the slow cooling taking place during furnace cooling following the homogenization annealing. After etching, Figure (2) b, the background becomes mottled

and the grain boundaries with the blue and violet particles delineating them become surrounded by a thin fair unmottled zone. The mottling of the background is the result of the chemical interaction of the α -aluminum solid solution and the very fine precipitated particles with the etching reagent. The thin fair unmottled zones, surrounding the grain boundaries, are the precipitation free zones formed as a result of rapid diffusion of solute atoms towards the grain boundaries to form precipitates in the early stage of precipitation. This takes place before homogeneous precipitates within the grains (far from the grain boundaries) could nucleate.



a- As polished (x600)



b- Etched (x300)

Figure 2: Representative microstructure of the homogenized ingot (T=530°C, 6 hrs).

Typical etched structure of a specimen subjected to solution treatment is showing in Figure (3). The micrograph reveals a grained structure with a noticeable amount of grain boundary precipitates. Some pores are also seen. Grain boundaries became sharp and well defined. Mottling of the grain inside has been considerably reduced. Moreover, the precipitate free zones, surrounding the boundaries disappeared. These observations indicate that the majority of bulk precipitates as well as grain boundary precipitates have been dissolved during heating for solution treatment. The dissolved precipitates have been retained in the solid solution during water quenching, because their re-precipitation has been suppressed.

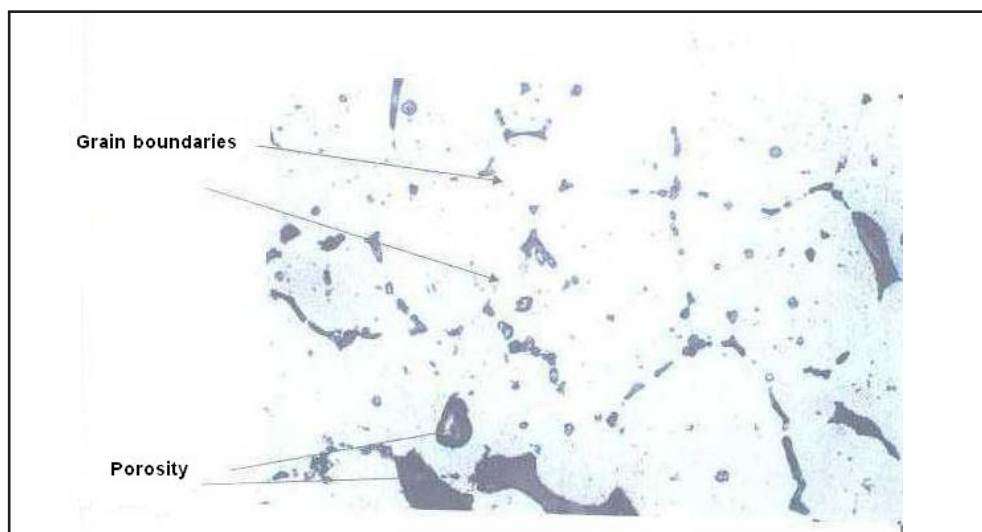


Figure 3: Microstructure of the solution treated specimen (x600)

X-ray diffraction

In order to identify the phases present in the structure of the homogenized specimen, X-ray diffraction analysis has been performed. The obtained patterns were analyzed by the computer system attached to the X-ray machine. Figure (4) introduces the obtained pattern. Eight strong peaks were observed. The d-values of these peaks were compared with those representing the possible phases, which may be formed by the atomic species comprising the alloy. The most probable phases are Al, Si, Mg_2Si and $\text{Al}_{3.21}\text{Si}_{0.47}$. Such comparison leads to the following conclusions:

- (i) The strongest peaks of homogenized specimen correspond to those of

aluminum but with slightly larger d-values. It is evident that these peaks are due to diffraction from planes of Al-base solid solution of larger lattice parameter.

- (ii) The absence of the Si peaks indicates that the amount of free Si is below the method's sensitivity.
- (iii) Since all Mg and a large portion of Si are dissolved in the Al-base solid solution during solution treatment, it is not surprising that the peaks of Mg_2Si are absent.
- (iv) The peaks of $Al_{3.21}Si_{0.47}$ phase is either absent or superimposed with those of aluminum.
- (v) There are three minor peaks that do not fit any of the diffracting planes of the mentioned phases. However, the strongest peak of them matches the peak of the complex phase $(Mg_2Al(SiAl)O_5(OH)_6)$ called “chromeasite”.

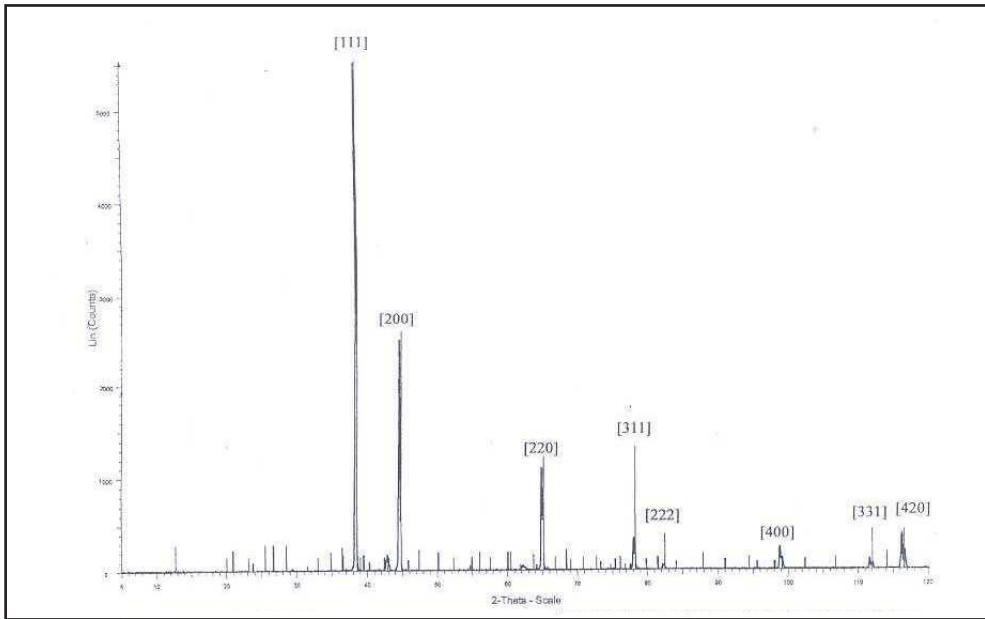


Figure 4: X-ray diffraction chart of homogenized alloy

Effect of cold work

The effect of percentage of cold work on the Vickers hardness of the solution treated specimens is presented in Figure (5). The effect is almost linear; the hardness increment is 10 Vickers units per 10 % reduction in thickness.

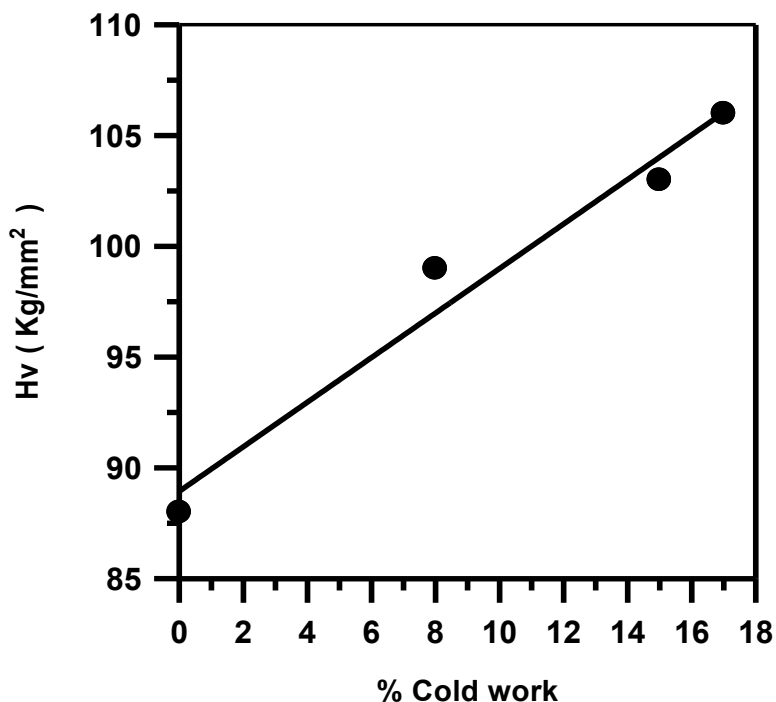


Figure 5: Effect of % C.W on the hardness of solution treated specimens

The X- ray diffraction charts of specimens subjected to increasing reduction in thickness after being solution treated, show almost the same peaks as for the solution treated specimen (deformation free), with a slight shift. Comparison of the d-values of the same peaks after deformation with that of the solution treated specimen reveals a noticeable decrease in the lattice parameter of the Al-based solid solution with increasing the degree of cold work, the lattice parameter becoming even less than that of pure Al. Several processes could have participated in this phenomenon. (a) The accommodation of compressive stresses caused by the rolling process could decrease the lattice parameter. However the amount of lattice defects (point defects and dislocations) created during the same process would have outbalanced the effect of compressive stresses. (b) Under the action of the compressive stresses the larger solution atoms (e.g. Mg) dissolved in the Al-based solid solution are likely migrate via the created lattice defects (vacancies and dislocations) to the fine grain boundary precipitates (e.g. Mg_2Si) leading to their growth. (c) Some of these solute atoms would migrate for short distances forming

zones, metastable intermediate phases which may accumulate around dislocations. These phenomena are likely to interfere with the subsequent aging processes accelerating or retarding them.

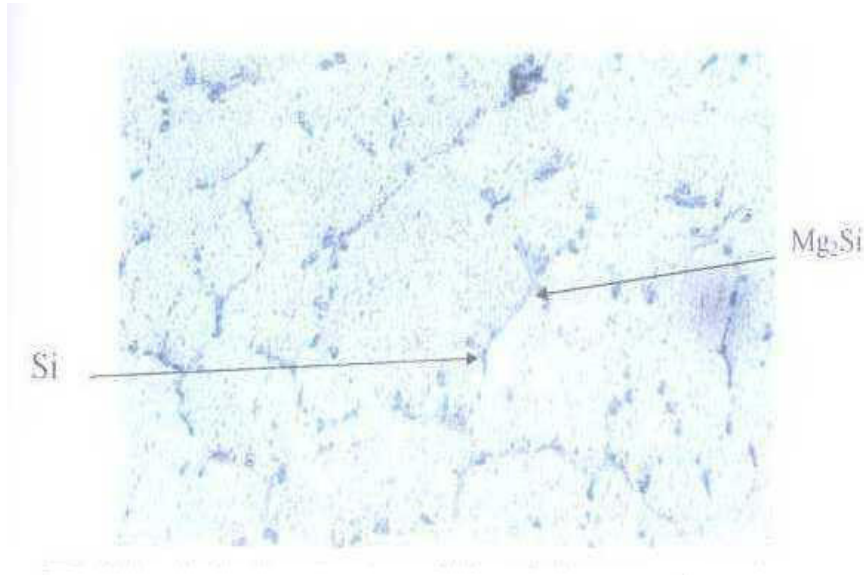
Two additional points should be mentioned here. The first is that with increasing the degree of cold work from 15% to 17% the lattice parameter did not decrease any more but in fact it is slightly increased. This can be explained by the increased amount of porosity occasionally encountered before deformation. In such a case deformation would have been accommodated into the pores rather than in the material giving rise to smaller compressive stresses. The second point concerns the high angle peaks of the diffraction charts. These peaks (at $\sim 112^\circ$ and 116°) in the chart corresponding to the solution treated specimens show a doublet due to the nature of X- ray beam applied. Higher angle planes diffract $K_{\alpha 1}$ and $K_{\alpha 2}$ radiation separately giving rise to sharp doublet. The chart of the specimen deformed 8% shows less sharp splitted peaks. However, with increasing the percentage deformation the splitting gradually disappears. After 17 % deformation, the peaks become singlet.

Metallographic examination

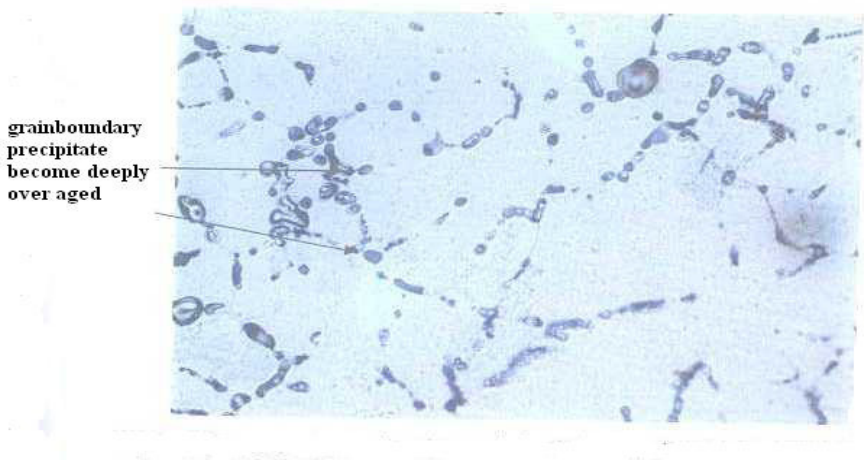
The polished and etched microstructure of the specimen cold rolled to 8% reduction in thickness is shown in Figure (6). Figure 6 (a), there is no appreciable change with respect to the solution treated (undeformed) specimen. The microstructure shows some bluish (probably Si) and violet particles of the form of Chinese script (probably Mg_2Si) delineating grain boundaries. However the amount of Mg_2Si is higher in this case. This may be due to inhomogeneous distribution of Mg_2Si in the as cast ingot. Figure 6 (b) introduces the same deformed structure but after etching to reveal the grain boundaries. The grain boundary precipitate particles became deeply over-aged probably due to the high residual stresses concentrated at grain boundaries, where harder particles are situated.

The microstructure of similar specimens but with higher percentage of cold reduction of thickness is showing in Figure (7). The violet particles show the characteristic Chinese script appearance of Mg_2Si . Figure 7 (a) of 15% reduction shows larger amount of the blue particles(Si phase) as compared with the microstructure of Figure 7 (b) of 17% reduction, This is indicative of the poor homogeneity of the as-cast structure. Etching of these specimens

showed very rapid over-etching around the grain boundary precipitate particles before the grain boundaries became seen. This is again due to the increased residual stresses at places of matching the hard precipitate particles with the soft grains.

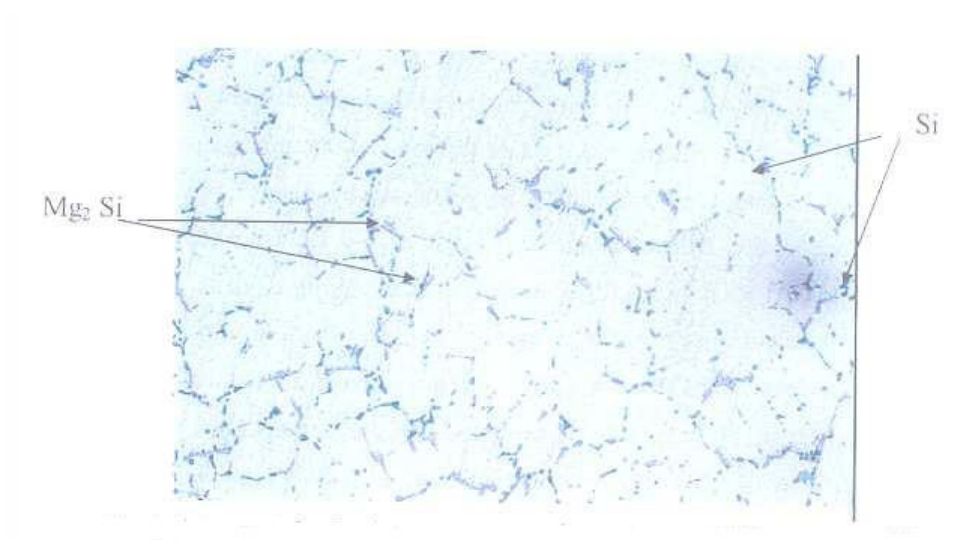


(a)- As polished (unetched)

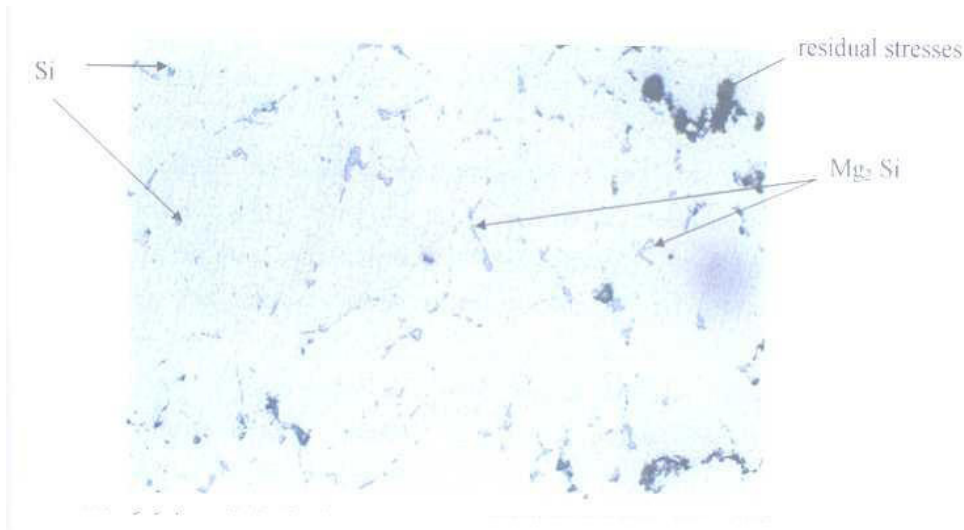


(b) - Etched

Figure 6: Typical microstructure of the solution-treated specimens, further subjected to cold rolling for 8 % reduction in thickness(x600)



(a)-15 % reduction in thickness



(b)-17 % reduction in thickness

Figure 7: As-polished microstructure of cold-rolled specimens (after solution treatment) (x300)

Effect of aging

The effect of the aging temperature for constant time interval on the hardness of specimens deformed for different degrees after solution treatment is summarized in Figure (8). The following conclusions can be withdrawn:

- (i) For all reductions in thickness, with increasing the aging temperature up to 170 °C the hardness increases. However, on increasing the temperature above 170°C the hardness sharply drops because of over-aging. The maximum obtainable hardness is achieved on aging at 170 °C.
- (ii) The hardness increment in aging at temperatures up to 170 °C is maximum for the specimens not subjected to intermediate deformation. With increasing the percentage cold work the hardness increment decreases. The lowest increment is obtained after deformation for 17 % reduction in thickness (within the frame of this investigation).
- (iii) For all reductions in thickness aging at 190 °C for 4 hrs leads to over-aging and softening.

The same data of Figure (8) but taking the deformation as an argument and the aging temperatures as a parameter displayed in Figure (9). The following conclusions can be made.

- (i) For natural aging (at room temperature) the higher, the percentage of cold work the higher is the hardness.
- (ii) For artificial aging at all of the investigated temperatures (150-190 °C) the effect of deformation is reversed, with increasing the percentage cold work the hardness obtained after 4 hrs is reduced. Based on this information it may be concluded that deformation accelerates the aging process, may be eliminating the formation of the coherent and semi-coherent precipitates, thus reducing the maximum attainable hardening effect within the explored range of temperature. The adverse effect of the extra Si particles situated at grain boundaries is quite clear.

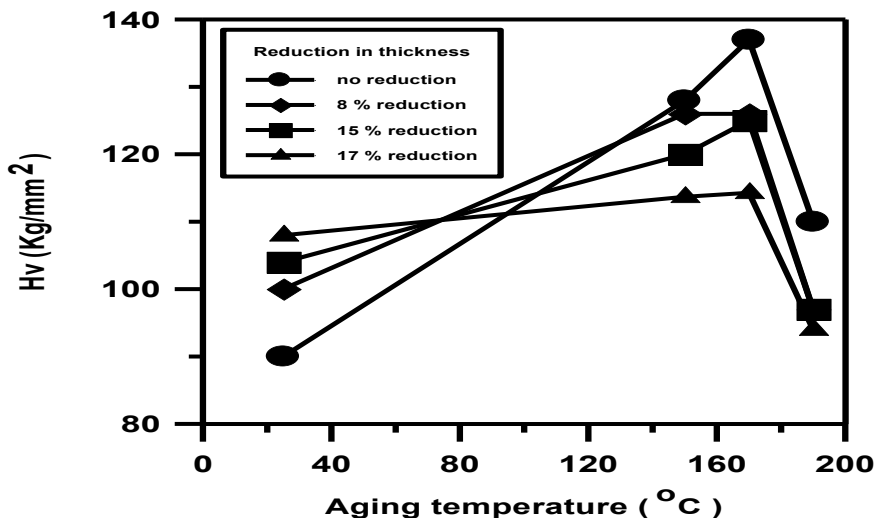


Figure 8: Effect of aging temperature on the hardness of deformed specimens

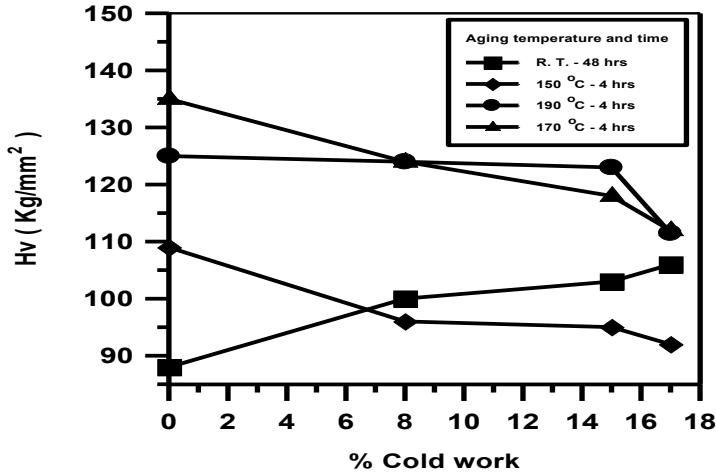


Figure 9: Effect of the degree of cold work on the hardness of aged specimens

Wear measurements

The effect of the percentage cold work on the wear weight loss counter cast iron disc (230 H_v) for 500 meter of sliding under 3.9 N of load for the specimens aged at different temperatures are shown in Figure (10). The weight loss after aging follows the same pattern for all the investigated temperatures. With increasing the percentage cold work the weight loss increases almost linearly up to 15 % cold work, and then increases abruptly. On the other hand, increasing the isochronal (4 hrs.) aging temperature up to 170°C reduces the weight loss. Higher aging temperatures, for the same time interval, leads to over-aging and softening, as a result the wear weight loss increases. It is worth to mention here that the least wear loss is obtained in undeformed specimens aged for 4 hrs. at 170°C.

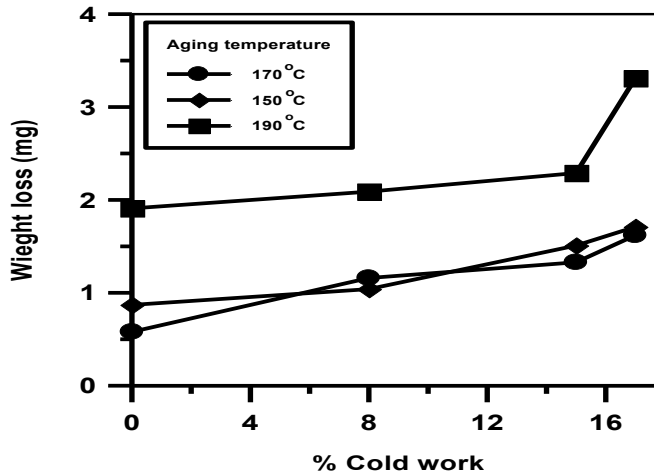


Figure 10: Effect of the % deformation on the weight loss due to wear of specimens aged at different temperatures

The correlation between the hardness and the weight loss due to wear is given in Figure (11). It can be fairly concluded that with increasing the hardness, regardless of the regime used, the weight loss will linearly decrease. Finally, it can be concluded that for wear applications, low temperature thermo-mechanical treatment of Al-Si-Mg alloys is not feasible. However, heat treatment rather than LTMT seems to be more feasible.

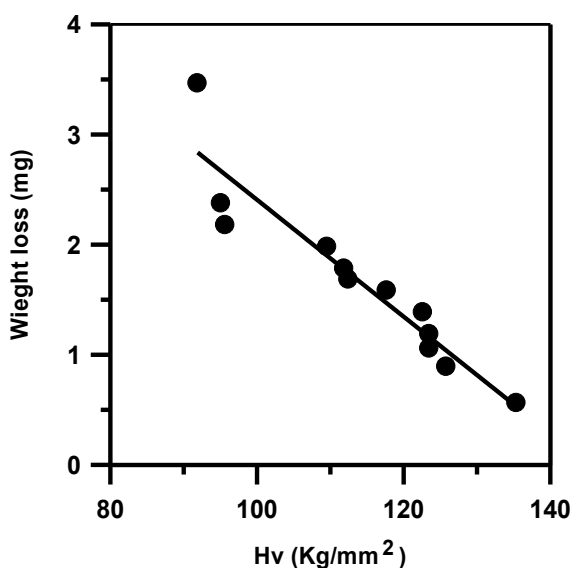


Figure 11: correlation between the wear weight loss and the hardness

CONCLUSIONS AND RECOMMENDATIONS

Based on the performed experimental work the following conclusions can be drawn:

1. Low temperature thermo-mechanical treatment of Al-Si-Mg alloys with excess free Si particles is not practically feasible for wear applications. Conventional heat treatment is more feasible.
2. Plastic deformation by rolling following solution treatment leads to a reduction of the lattice parameter of the matrix Al-based solid solution in Al-Si-Mg alloys.
3. Increasing the percentage cold work of Al-Si-Mg alloys with excess silicon leads to a decrease in the maximum attainable hardness of the artificially aged articles. For naturally aged articles the contrary is true, i.e. with increasing the degree of cold work the maximum attainable hardness increases.

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