

Effect of Sever Plastic Deformation on The Microstructure of Al-Si/Mg₂Si in Situ Composites

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ABSTRACT

In this work, the Al-Si/Mg₂Si in-situ composites were fabricated using casting technique, followed by repetitive equal-channel angular pressing (ECAP) to refine the microstructure. (ECAP) as sever deformation was carried out through a die made of high chromium carbon steel at 250°C for four passes of deformation. Microstructures investigation was conducted using an optical microscope, electron back scattering, EDS, elemental mapping and electron backscattered diffraction (EBSD). After sever deformation, the results showed fragmentation of eutectic Mg₂Si and eutectic Si, reduction of the primary Mg₂Si particle size, and fragmentation of the dendrite of Mg₂Si particles to smaller ones. On other hand, the columnar α -Al phase changed to nearly equiaxed grains after severe deformation. The results of (EBSD) revealed that the grain size of the in site composite matrix showed decrease to less than 5 μ m after four passes, and the grains after deformation have more orientation of homogenization, and there are fine grains less than 1 μ m in size surrounded by high angled boundaries

keywords: in-situ composites, sever plastic deformation

INTRODUCTION

The Al-Si-Mg system has been proved a very promising system for the production of Al matrix in situ composites, with the Mg₂Si intermetallic phase being the in situ formed reinforcing phase, establishing a new group of particulate metal matrix composites. Al-Mg₂Si composites have high potential as structural materials because an intermetallic compound of Mg₂Si exhibits low density (1.99 g/cm³), high melting point (1085°C), high hardness (4.5 $\times 10^9$ Nm⁻²), low thermal expansion (7.5 $\times 10^{-6}$ K⁻¹), and high Young modulus (120 GPa) [1]. In situ Mg₂Si may precipitated using liquid state processes (casting) or using solid state processes (powder metallurgy). The solid state routes are expensive, therefore efforts have been made to produce such composites by a casting process (2). However, conventional casting process usually produces coarse Mg₂Si phases and these particles themselves are hard and brittle which would produce stress concentration at the tips or edges of the particles, leading to cracking at the grain boundary or in the Mg₂Si particles themselves, thereby reduce the mechanical properties, especially elongation [3]. Investigation have been carried to modify the eutectic and primary Mg₂Si by adding some elements to the alloys melt [4,5]. However, the improper control of some process parameters of this technique can lead to deleterious defects in cast composites such as porosity and non-uniform distribution of the particles in the matrix (6).

.Some efforts have been conducted to modify the microstructure of Al- Mg₂Si in situ composites by modifying the casting process and using different heat treatment regimes[7]. It has been recognized that the repetitive equal-channel angular pressing(ECAP) is one of the most effective methods in producing bulk ultra-fine grained materials with sub micro or nanocrystalline structure for a wide range of materials[8-10]. Few issues were found to demonstrate the modification in situ composites by deformation, Hence ,the aim of the present work is to focus on the effect of ECAP on the microstructure of the in-situ Al- Mg₂Si composites.

Experimental Procedure

Materials

Al-23Si master alloy and commercial purity aluminum supplied from General Company for Electrical Industries in Wazirya (Baghdad); Al-23Si alloy, pure aluminum were used as starting materials to prepare (Al-7%Si and Al-15%Si) alloys. High purity magnesium (99.89%) supplied from the chemical shop in Baghdad was added to fabricate Al-Si/Mg₂Si in-situ composites

Fabrication of insitu composites

About 250 gram of the Al-23Si master alloy and pure aluminum with the required proportions was charged into the clay-graphite crucible and melted at about 750 °C in electric resistance furnace. The melt was held at this temperature for approximately 15 min for homogenization melt temperature and composition. After that, the crucible was drawn from the furnace, and the melt was stirred using an electrical motor equated with a stainless steel rod, introduced into the melt. After stirring, the melt was poured in to steel mold to produce the Al-7Si and Al-15Si alloys.

To fabricate Al-Si/Mg₂Si in situ composites different weight percentages(5,12%) of magnesium lumps, warped in an aluminum foil, were added in to the melt and pressed under the surface of the melt to prevent oxidation , (15% Mg was added in to the melt as a balance of oxidation loss). A laboratory flux (AlCl₃) was used as a degassing agent. After completing the addition of magnesium , the stirring was stopped and the mixture was reheated in the furnace for 10 min and then poured into a preheated low carbon steel mould .Table(1) show the chemical compositions of the prepared composites. Chemical analysis was done at the Specialized Institute for Engineering Industries, using Thermo ARL 3460, Optical Emission Spectrometer .

Table (1): Chemical compositions of the in-situ composites

No.	V%	Ti%	Zn%	Cr%	Mg%	Mn%	Cu%	Fe%	Si %	Al%
1	0.007	0.004	0.214	0.005	4.36	0.028	0.038	0.250	6.72	Bal.
2	0.016	0.003	0.514	0.025	11.39	0.071	0.046	0.271	6.88	=
3	0.012	0.012	0.237	0.013	5.81	0.037	0.029	0.265	15.45	=
4	0.026	0.013	0.688	0.038	11.54	0.086	0.021	0.353	15.15	=

Repetitive equal-channel angular pressing (ECAP)

Billets with(12 x 12 x 45)mm dimensions were machined from the as-cast composites and pressed through a die(manufactured by the researcher), made of high chromium carbon with a sharp-cornered 90° (the internal angle is 90° between the horizontal and vertical channels) using Instron machine model (WDW-200E) ,20 KN capacity . The situ composites were pressed at 10 mm min⁻¹ at temperature 250° C. The billet was first inserted into the die and then together heated to the test temperature by using four pin heaters with thermocouple and digital control unit followed by holding at this temperature for 15 min; finally, the billet was pressed under a

constant speed of 5mm/min. The billet was continuously rotated 90° clockwise after each pass of ECAP up to four passes. For minimizing the effect of friction, Graphite was used as lubricant during the deformation .Figure (1) shows the ECAP press; ECAP die and the samples after severe deformation by ECAP.

Microstructure Observation

The microstructures of the samples were examined by an optical microscope and scanning electronic microscope using both secondary and backscatter electron imaging by (SEM LEO 1450 and EDS Detector(Model 7353) , at the University of Ferdowsi in Mashhad and at the University SAINS in Malaysia. The as-cast and the in-situ composites were prepared using standard metallographic procedures, grinding, polishing and etching. The polished surface was etched by a solvent of 5% nitric acid, 5% acetic acid, 20% water, and 60% ethanol, on other hand ,the electron backscattered diffraction (EBSD) analysis was done directly after 1 μ m diamond polishing then electro polished using (20% hydrochloric acid and 80% ethanol) at 20 volt, for polishing time 25 second . (EBSD) analysis was done at Missouri University.

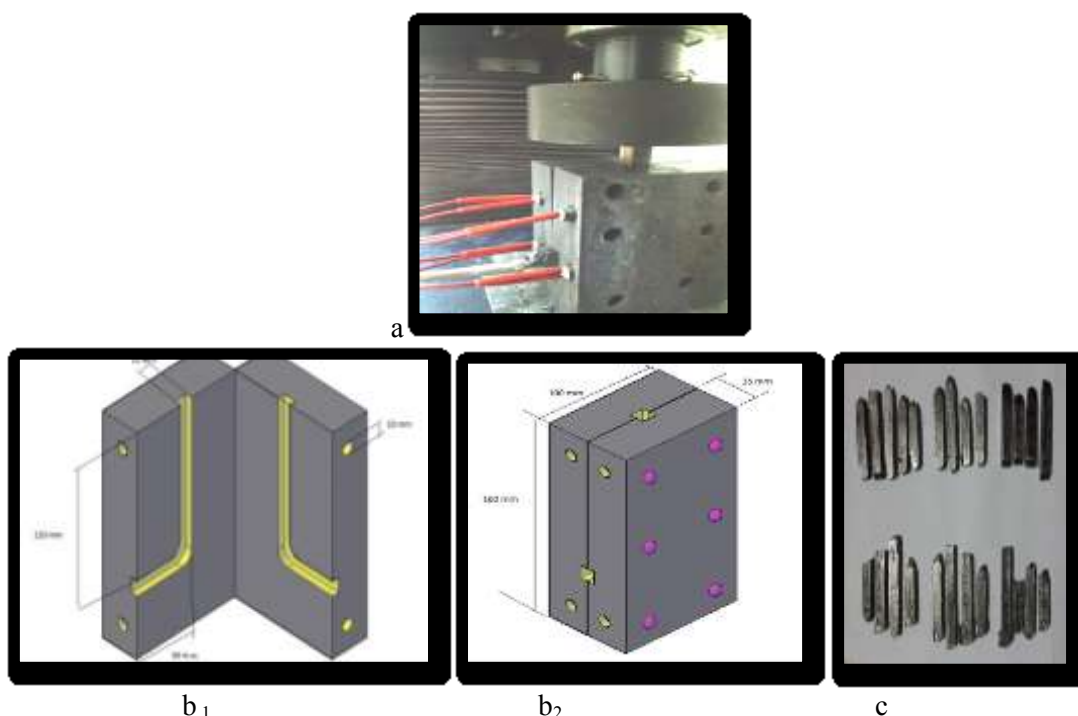


Figure (1): The ECAP press, die and the samples after severe deformation by ECAP.
a: ECAP press , b₁&b₂:ECAP die ,c: the samples after ECAP .

Results and Discussion

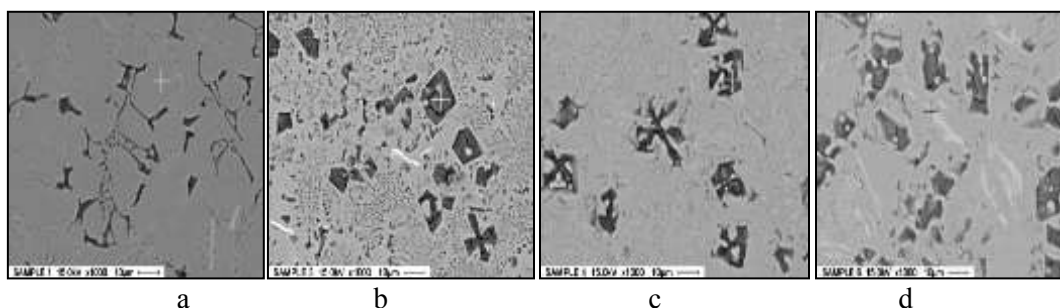
Microstructure Observation

According to alloys composition Al-Mg₂Si equilibrium phase diagram [11], and ternary Al-Mg₂Si-Si phase diagram [12] , the theoretical estimated Mg₂Si and the solidification path of prepared in-situ composite are summarized in table(2).From the table ,it can be seen that the prepared in-situ composites contain different percentages of Mg₂Si and different retained primary silicon.

Table (2): Estimated Mg₂Si and solidification path of prepared in-situ composites

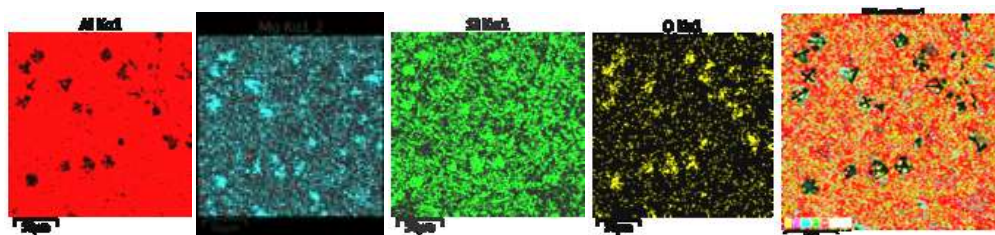
Prepared alloy	Estimated composition	Solidification sequence according to equilibrium phase diagram
Al-7Si-5Mg	Al-8Mg ₂ Si-4Si	$L \rightarrow L1 + \alpha\text{-Al} \rightarrow L2 + (\text{Al} + \text{Mg}_2\text{Si}) + \alpha\text{-Al} \rightarrow (\text{Al} + \text{Mg}_2\text{Si}) + (\text{Al} + \text{Si} + \text{Mg}_2\text{Si}) + \alpha\text{-Al}$
Al-7Si-12Mg	Al-19Mg ₂ Si	$L \rightarrow L1 + \text{Mg}_2\text{Si} \rightarrow L2 + \text{Mg}_2\text{Si} \rightarrow (\text{Al} + \text{Si} + \text{Mg}_2\text{Si}) + (\text{Al} + \text{Mg}_2\text{Si}) + \text{Mg}_2\text{Si}$
Al-15Si-5Mg	Al-8Mg ₂ Si-12Si	$L \rightarrow L1 + \text{Mg}_2\text{Si} \rightarrow L2 + (\text{Si} + \text{Mg}_2\text{Si}) \rightarrow (\text{Al} + \text{Si} + \text{Mg}_2\text{Si}) + (\text{Si} + \text{Mg}_2\text{Si})$
Al-15Si-12Mg	Al-19Mg ₂ Si-8Si	$L \rightarrow L1 + \text{Mg}_2\text{Si} \rightarrow L2 + \text{Al} + \text{Mg}_2\text{Si} \rightarrow (\text{Al} + \text{Si} + \text{Mg}_2\text{Si}) + (\text{Al} + \text{Mg}_2\text{Si}) + \text{Mg}_2\text{Si}$

Figure (2) shows the back scattered electron images of as-cast in-situ composites. It was observed that the micro structure of in situ composite (Al-8Mg₂Si-4Si) consists of chine's script like shaped eutectic Mg₂Si together with eutectic silicon located at the grain boundaries and periphery of α -Al matrix, as shown in Figure (2-a). It can be seen from the figure that no primary Mg₂Si particles exist in this composite this can be attributed to the small amount of Mg₂Si (8%) which is lower than that of the pseudo eutectic composition (13.9wt %) as in Al-Mg₂Si phase diagram.

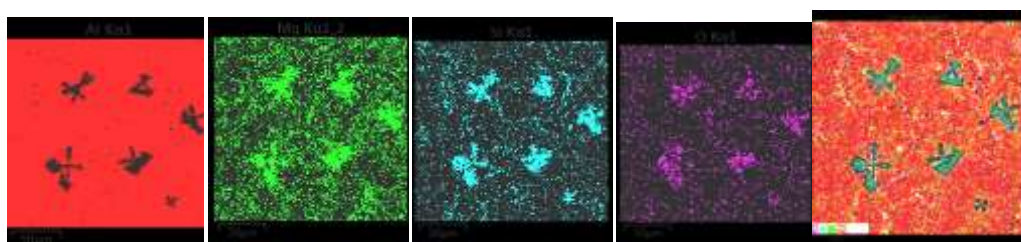


Figure(2) Back scattered electron image and corresponding EDS spectra of prepared as-cast in-situ composites a:Al-8Mg₂Si-4Si ,b:Al- 19Mg₂Si, c:Al- 8Mg₂Si-12Si, d:Al- 19Mg₂Si-8Si

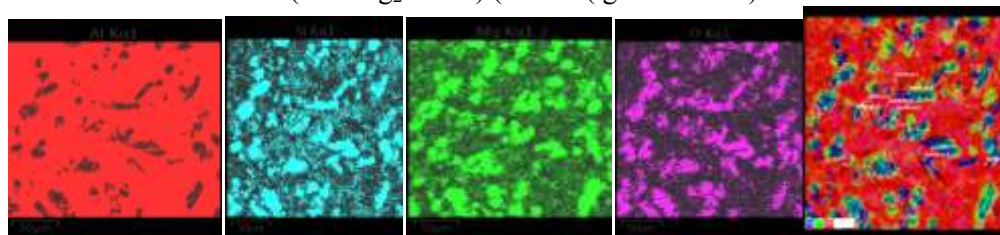
With increasing magnesium content to 12 wt% , theoretical Mg₂Si amount is increased to 19 % and the composite composition was converted to hypereutectic composition of Al-Mg₂Si phase diagram ,and the microstructure of(Al-19Mg₂Si) contained primary Mg₂Si particles embedded in (α -Al+Mg₂Si) binary eutectic, surrounded by (α -Al+Mg₂Si+Si)ternary eutectic as shown in Figure (2-b) .With increasing Si content in the alloys as shown in Figure (2- c), the morphology of theMg₂Si changed from chine's script like shaped in(Al-8Mg₂Si-4Si) to cross-like

as cast Al-8Mg₂Si-12Si in situ composite

Mg₂Si phase(blue & green color), Al phase(red color), binary eutectic(Al+Mg₂Si)(red+(blue & green)) ,ternary eutectic (Al+ Mg₂Si +Si) (red +(blue& green)+blue

as cast Al-19Mg₂Si in situ composite.

Mg₂Si phase(green & blue color), binary eutectic(Al+Mg₂Si)(red+(green & blue)) ,ternary eutectic (Al+ Mg₂Si +Si) (red +(green & blue)+blue

as cast Al-19Mg₂Si-8Si in situ composite

Si phase, Mg₂Si phase, binary eutectic (Al+Mg₂Si) ,ternary eutectic (Al+ Mg₂Si +Si)

Figures (3) show elemental mapping of as cast in-situ composites prepared from alloys contain different amount of magnesium and silicon shape in (Al-8Mg₂Si-12Si) in situ composite (Fig-2a& b). The cross-like shape Mg₂Si in (Al-19Mg₂Si) changed to coarse irregular dendrite shape in (Al-19Mg₂Si-8Si) in situ composite, as shown in Figure (2-d). Micro structure observation, the shape and size of primary Mg₂Si and eutectics phases depended on the Mg percentage.

The results of microstructure observation have shown that the shape and size of primary Mg₂Si and eutectics phases of resulting composite depended on the Mg and Si additions as shown in elemental mapping image in Figure (3). The microstructure difference can be explained as follows: The as cast composites were cast at the same temperature and solidified under the same conditions, thus according to Al-Mg-Si ternary phase diagram, the different alloys have different liquids temperature, this mean the actual under cooling for each alloy is different, therefore the composites will achieve different under cooling then the resulting microstructure will changed.

The pseudo binary-eutectic is converted to ternary eutectic system when excessive Si is added in to alloys. Accordingly primary Mg₂Si solidified first from the liquid followed by binary eutectic reaction to form either Al or Si eutectic with Mg₂Si. afterward the ternary eutectic forms if extra Si is added in to the alloys [13]

Microstructure after repetitive equal-channel angular pressing

The microstructure of the initial as cast and repetitive equal-channel angular pressed in situ composite (prepared from Al-7Si and Al-15Si with different amounts of Mg, from 1 pass up to 4 passes), is shown in Figure (4). This figure reveals that the eutectic Mg₂Si and eutectic Si have been fragmented, primary Mg₂Si particle size has been reduced with increasing passes of ECAP and the dendritic Mg₂Si particles have been fragmented to smaller ones. On the other hand, the columnar α -Al phase changes to nearly equiaxed grains after repetitive deformation.

Figure (5) shows a comparison between the initial as cast Al-8Mg₂Si-4Si in situ composite microstructure, and those obtained after (ECAP). BSE image manifests that the eutectic Mg₂Si and coarse eutectic Si for the deformed in situ composite sample became fragmented when compared to the as cast sample. After one pass of ECAP, the eutectic Mg₂Si and eutectic Si were broken and accumulated on the α -Al grain and aligned along the metal flow direction or with an angle about 45° to ECAP direction, as shown in figure (5- b). After four passes, the Mg₂Si eutectic was transformed to fine particles and distributed throughout the matrix, as shown in figure (5- c). The change in eutectic is due to the shear stress imposed by ECAP, resulting in the fragmentation of the larger eutectic structure of the as cast composite. Observation also indicated that the columnar α -Al matrix grains were decreased from the initial of about 28 μ m to 18 μ m after one pass of deformation. After four passes, the α -Al grains change to the nearly equiaxed grains with an average size of 5 μ m.

After four passes of deformation, the coarse and irregular back-bone like Mg₂Si particles in Al-8Mg₂Si-12Si in situ composite (shown in Figure 2) were broken and changed to irregular polygonal shapes. On the other hand, there is a small number of incompletely broken Mg₂Si particles in the matrix, resulting from the fragmentation of the larger particles. In (Al-19Mg₂Si) in situ composite, the primary Mg₂Si particles were broken after four passes of ECAP. The eutectic Mg₂Si and eutectic silicon became finer, as shown in Figure (6). On the other hand, the coarse and dendritic (back-bone like) Mg₂Si particles of Al-19Mg₂Si-8Si in situ composite were broken to several ones, but there were many fine Mg₂Si particles resulting from the fragmentation of the larger particles. Even in the matrix, there is still a lot of coarse Mg₂Si particles; this could be due to a very high hardness of the Mg₂Si particles compared with the matrix Al grains, therefore the shearing stress being enforced by the matrix flow is not enough to break the hard Mg₂Si into small particles.

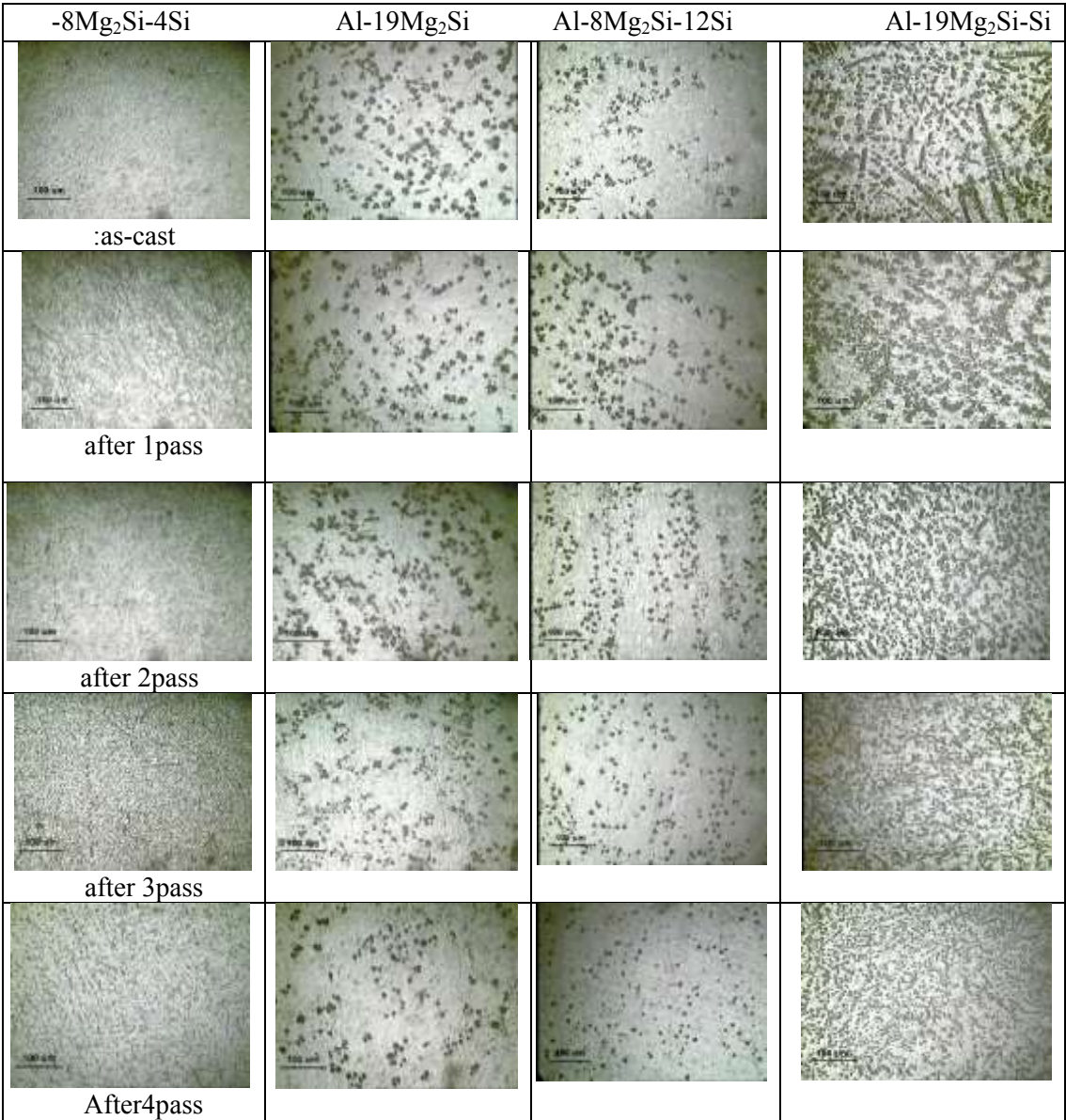
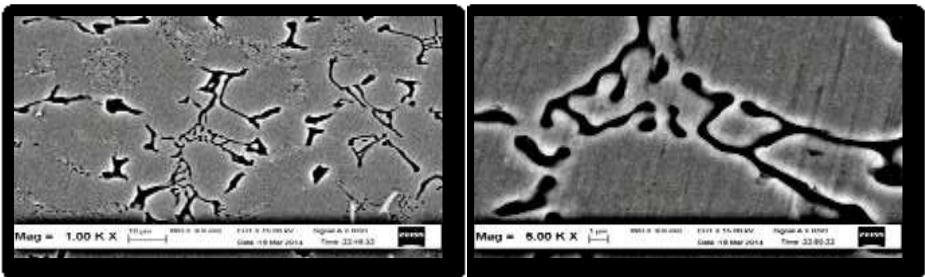


Figure (4): Microstructure of as cast and ECAP from1-4 passes for insitu composite prepared fromAl-7Si and Al-15Si alloys.



a:as cast

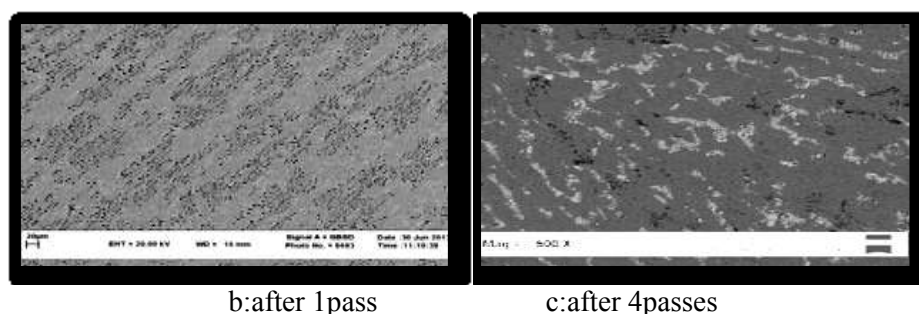


Figure (5)BSE image of:(Al-8Mg₂Si-4Si) before and after ECAP.

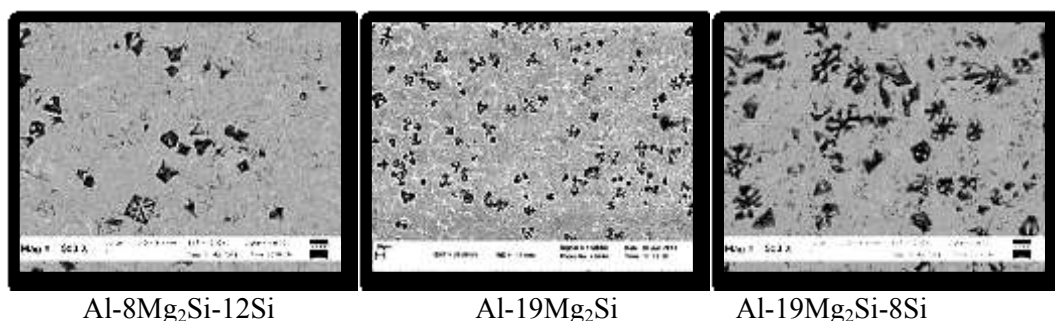


Figure (6)back scattered electron image of in situ composites after 4 passes

Electron Back Scatter Diffraction mapping was used to observe the eutectic modification and grain refinement after severe plastic deformation by ECAP. SEM image and orientation mapping (EBSD map) of Al-Si/Mg₂Si in-situ composites before and after severe deformation is shown in figures (7 and 8) . The white color indicates the Mg₂Si particles and eutectic. Different colors in EBSD indicate different crystallographic orientations .The similar colors of grain indicate similar orientations (little difference between the orientations of grains). In addition, the black line in maps marks the location of high-angle boundaries (the boundaries have a relative misorientation greater than 15°). It was observed that the EBSD maps of as cast mainly contains coarse grains. The grain color and morphology significantly changed after ECAP. The microstructure and the corresponding selected area electron diffraction pattern demonstrate that after 4 passes ECAP, most of the coarse grains whose average size (9.5μm, 15.7μm, 7.35μm and 8.28μm) are substituted by equiaxed grains. Almost all grain sizes of deformed matrix are less than 5 μm. Fine -sized grains (surrounded by high angle boundaries less than 1 μm) are also observed, and the microstructure becomes more homogeneous within the observed area. The changes in the microstructure are associated with the continuous formation of high angle boundaries due to the accumulation of dislocations during deformation. The presence of large misorientations in the vicinities of the boundaries is due to the rapid development of strain gradients near grain boundaries during deformation. The fraction of such boundaries gradually increases during straining, until almost all the grains are bounded by high angle boundaries. Some investigators attributed the grain refinement in sever plastic deformation to continuous dynamic recrystallization [14,15].The mechanisms are as follows :The initial stages of straining introduce high dislocation densities, arranged in cellular substructures. An increase in strain is attended with localizations of plastic flow on a microscopic scale. The dislocation cells evolve into cell blocks that are subdivided by dense dislocation walls. These are essentially dislocation sub -boundaries whose misorientations are appreciably larger than those of common cell walls. Then, various deformation bands begin to appear at medium strains that introduce still larger misorientations. This leads to the subdivision of the original grains into small, heavily misoriented fragments. This process of grains

subdivision during deformation is fundamental to the process of grain refinement by sever plastic deformation [16].

CONCLUSIONS

The main conclusions drawn from the present work are as follows:-

1. The microstructure of as cast composites contained two morphologies of the intermetallic Mg₂Si phase: one is the eutectic Mg₂Si phases and, the other is the primary Mg₂Si particles. the shape and size of these phases depended on the Mg and Si percentage.
2. The ECAP process affected the refinement of the microstructure of the Al-Mg₂Si in-situ composite.
3. After four passes the primary Mg₂Si particle size reduced with increasing passes of ECAP and the dendrite Mg₂Si particles has been fragmented to smaller ones. The coarse grains of the matrix α -Al became smaller and changed to the nearly equiaxed grains
4. The morphology of eutectic is modified, and the size of eutectic and Si particles is reduced after ECAP.

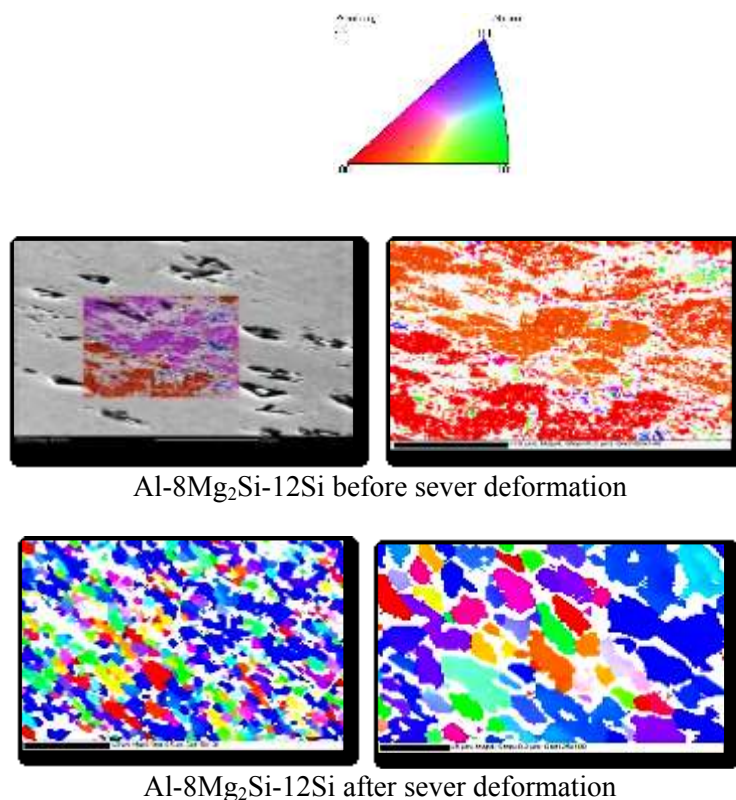


Figure (7) SEM image and orientation mapping (EBSD map) of Al-8Mg₂Si-12Si in-situ composites before and after ECAP

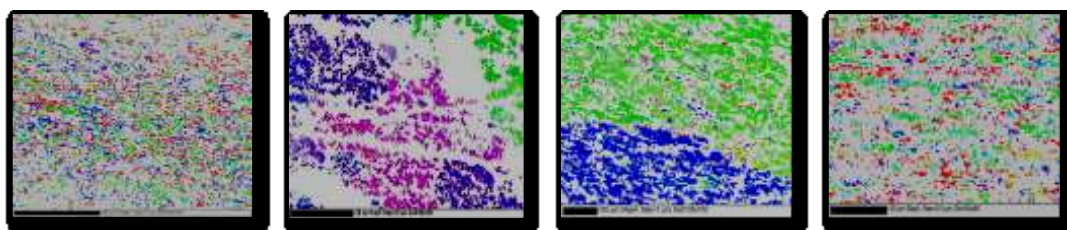


Figure (8) EBSD map of eutectic for in situ composites after severe deformation

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