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# Microstructure and mechanical properties of Al-3Fe alloy processed by equal channel angular extrusion

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**Abstract.** Al-Fe alloys are attractive for applications at temperatures beyond those normally associated with the conventional aluminum alloys. Under proper solidification condition, a full eutectic microstructure can be generated in Al-Fe alloys at Fe concentration well in excess of the eutectic composition of 1.8 wt.% Fe. The microstructure in this case is characterized by the metastable regular eutectic Al-Al<sub>6</sub>Fe fibers of nano-scale in diameter, instead of the equilibrium eutectic Al-Al<sub>3</sub>Fe phase. In this study, the microstructure and mechanical properties of the Al-3Fe alloy with metastable Al<sub>6</sub>Fe particles deformed by equal channel angular extrusion were investigated. Severe plastic deformation results in a microstructure consisting of submicron equiaxed Al grains with a uniform distribution of submicron Al<sub>6</sub>Fe particles on the grain boundaries. The room temperature tensile properties of the alloy with this microstructure will be presented.

## 1. Introduction

In general, alloys such as Al-Si alloys and Al-Fe alloys with chemical compositions close to or beyond the eutectic point could not be subjected to conventional plastic deformation due to the fact that at least one of eutectic phases in the alloy of considerable volume fraction is brittle. It has been recently shown that the above difficulties associated with Al-Si alloys could be readily overcome by combination of direct chill casting and plastic deformation provided the fibrous eutectic Si phase was transformed into fine Si particles prior to deformation [1]. The resulting microstructure consists of fine equiaxed Al grains with fine Si particles distributed on the Al grain boundaries.

Al-Fe alloys have been the subject of much study in the past primarily driven by the requirement to develop Al alloys with a higher temperature capability than conventional Al alloys [2-6]. To avoid the deformation difficulty of the cast Al-Fe alloys in the conventional processing, severe deformation routes have been employed to generate bulk materials from not only cast alloys but also rapidly solidified Al-Fe alloy powders [4] and ribbons [5] as well as the elemental Al and Fe powders [6].

In the 70's of the last century, Hughes and Jones [7] showed that the solidification microstructure of Al-Fe alloys varied depending on the growth velocity during directional solidification. An entire metastable eutectic microstructure of Al-Al<sub>6</sub>Fe could be obtained in alloys with Fe contain up to 5.5 wt.% while the equilibrium eutectic composition is only 1.8 wt.% Fe. They further showed that the rod-like metastable eutectic Al<sub>6</sub>Fe phase is quite stable at elevated temperatures and could be transformed into nano-sized particles upon heating [8]. It should be noticed that, from open literature, no attention had ever been paid to the utilization of microstructure of full eutectics of Al-Al<sub>6</sub>Fe in Al-Fe alloys, even though much work has been devoted into Al-Fe systems aiming at developing high temperature Al alloys.

In this work, we introduce a new method to generate a binary Al-3Fe alloy with an extremely fine microstructure based on normal casting and severe plastic deformation (SPD) by equal channel angular processing (ECAP) which was inspired by our research on Al-Si alloys. The mechanical properties of thus processed alloys will be presented.

## 2. Experiments

High purity (99.99%) Al and Fe were used to prepare Al-Fe alloy. Melting of aluminum was carried out in a high purity alumina crucible with an induction furnace. A certain amount of Fe was added into molten aluminum at 850°C to make a nominal composition of Al-3%Fe in weight. The melt was kept at this temperature in the furnace for 30 minutes to ensure all Fe was dissolved and then cooled to a temperature of 790°C in the furnace



by cutting off the power supply. The alloy melt was stirred vigorously in the air and poured into an iron-made die at 780°C to make bars of 12mm in diameter and 70mm in length. Before SPD by ECAP, the bars were annealed at 550°C for 10 hours. The ECAP was performed in a 90° die with back pressure at room temperature using route A with 4 and 8 passes. The microstructure of Al-3Fe alloys were investigated using a Leica DMI 5000M optical microscope and a Zeiss Ultraplus scanning electron microscopy (SEM) following standard metallographic methods of sample preparation. For preparation of samples for transmission electron microscopy (TEM) studies, cut materials were thinned to 50µm by mechanical polishing and punched into 3mm disks. The thinned foils were then twin-jet electropolished in a solution of 30% nitric acid in Methanol at -20°C. Conventional TEM observations were performed using a FEI Tecnai F20 electron microscope operated at 200 KV. The specimens for tensile tests were electrical-discharging cut from ECAP formed samples parallel to the direction of the extrusion. The dimensions of the gauge section the specimens were 1.5mm in thickness, 3mm in width and 5 mm in gauge length. Tensile tests were performed on an Instron 5848 microtester at a speed of 0.5 mm/min. A contactless MST LX300 laser extensometer was used to calibrate and measure the strain of the samples upon loading.

### 3. Results and discussions

#### 3.1. Solidification microstructure of Al-3Fe alloy

In the periphery of the cast bars the microstructure is featured by Al dendrites with fine eutectics among them. The dendrites are roughly perpendicular to the surface of the bar. Typical microstructure in the periphery is shown in the left part in Fig. 1a. The entire periphery of the bar forms a “ring” of about 1.5 mm in thickness covering the rest of the body. Inside the region covered by the “ring”, the microstructure consists of big colonies of extremely fine eutectics of Al-Al<sub>6</sub>Fe separated by distinct boundaries, as shown in the right part of Fig. 1a. However the eutectics within the colonies may vary either in growth directions or fineness. Fig. 1b shows the eutectic colonies in the center of the bars, the eutectic Al-Al<sub>6</sub>Fe and the boundaries of the colonies are very evident. The passes in the cast bars were studied with X-ray diffraction (XRD) and the results showed that the dominant phases in the alloys are Al and Al<sub>6</sub>Fe phases. Negligible amount of equilibrium intermetallic Al<sub>3</sub>Fe phase was detected by XRD. High magnification observation of the eutectic reveals that the Al<sub>6</sub>Fe phase is rod-like about 100 to 200 nm in diameters and the length can be several tens µm.

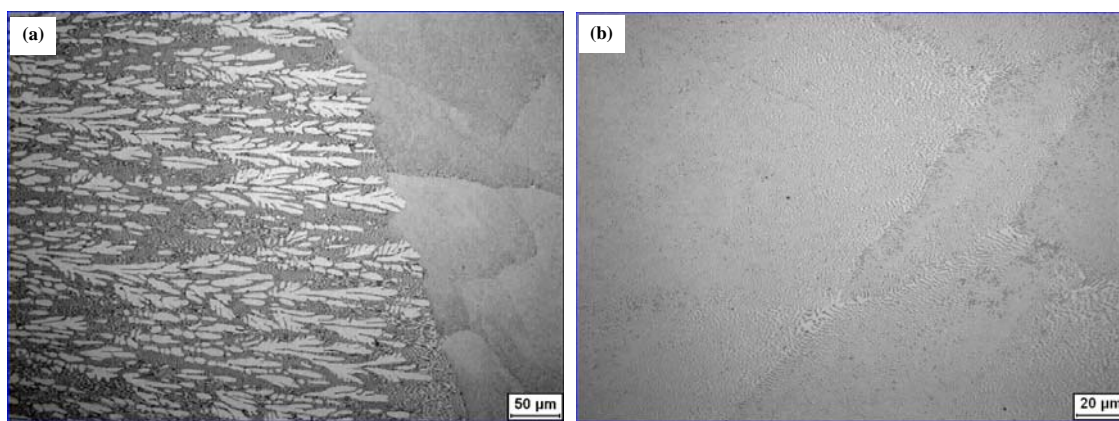


Fig.1 Optical micrographs showing (a) Al dendrites in the periphery of the cast bars perpendicular to the bar surface and (b) entire eutectics within the eutectic colonies with clear boundaries.

According to Hughes and Jones [7], for Al-3Fe alloy, within the growth velocity range between about 0.3 mm/s to 3 mm/s, the solidification microstructure is characterized by full Al-Al<sub>6</sub>Fe eutectics and the interphase spacing varies with growth velocity. The growth velocity in the directional solidification experiments could be well controlled thus a uniform microstructure could be always obtained. In the present work, the thermal field in the solidifying melt varied with time, consequently different morphologies of the eutectics in the eutectic colonies resulted as shown in Fig.1b. Al dendrites microstructure plus fine eutectics in the left part of Fig.1a in the periphery area is due to the rapid cooling provided by the cast-iron die and the fact that the heat transfer direction is perpendicular to the surface of the die in the beginning of the solidification. It therefore could be anticipated that the growth velocity of the Al dendrites is beyond 3 mm/s.

From the viewpoint of microstructure uniformity, full eutectic microstructure as shown in Fig.1b is preferred. It is believed that under proper conditions, the dendritic microstructure shown in Fig.1a could be eliminated. At present, before further processing, the periphery of the bars was machined out to maintain a uniform starting microstructure.

### 3.2 Microstructure of the ECAP processed Al-3Fe alloy

Fig.2 shows the microstructure in the center of the Al-3Fe alloy bar after thermally annealed at 550°C for 10 hours. The morphology of Al<sub>6</sub>Fe phase was transformed from rods into fine particles typically of 100 nm to 700 nm in length during annealing, depending on the initial eutectics. It is well known that the thermodynamically stable phase in Al-Fe alloy is Al<sub>3</sub>Fe. The microstructure in the annealed sample indicated that metastable Al<sub>6</sub>Fe phase in fact is quite stable at elevated temperature and the spheroidization of the eutectic Al<sub>6</sub>Fe rods is the consequence of the reduction of the surface free energy [9]. The boundaries of the eutectic colonies still exist after annealing. In some cases, on the boundaries the Al<sub>6</sub>Fe particles could be seen evidently larger than that inside the colonies and a zone free of the Al<sub>6</sub>Fe particles like PFZ in aging hardening Al-alloys always accompanies these large particles. It is then believed that the PFZ-like zones were formed because of the coarsening of the Al<sub>6</sub>Fe particles on the boundaries of the eutectic boundaries.

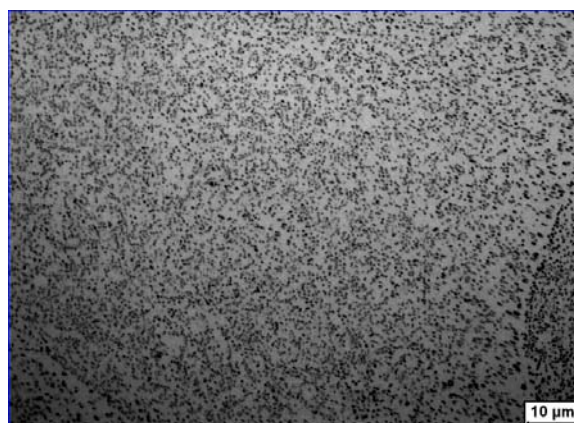


Fig.2 optical micrograph showing spheridized eutectic Al<sub>6</sub>Fe particles.

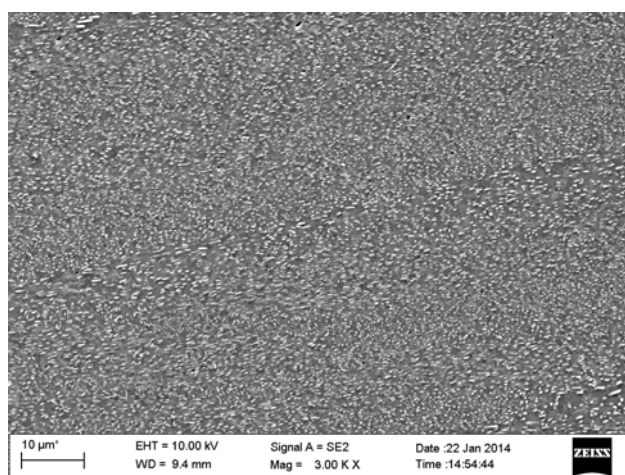


Fig.3 SEM micrograph showing microstructure formed by ECAP, note the elongation of the eutectic colonies and the different morphologies of Al<sub>6</sub>Fe particles within the colonies.

The spheroidization of the eutectic Al<sub>6</sub>Fe made plastic deformation possible without cracking of this brittle intermetallic compound. Fig.3 shows the SEM microstructure of ECAP processed Al-3Fe alloy in the longitudinal direction after 8 passes. It can be seen that eutectic colonies were elongated and the size and the morphologies of the Al<sub>6</sub>Fe particles are roughly the same within each elongated colonies. Compare to the undeformed eutectic colony shown in Fig.2, the large share strain provided by ECAP is evident and because

route A ECAP was employed, the flow of the Al matrix as well as the  $\text{Al}_6\text{Fe}$  particles was only limited within the eutectic colonies. The deformation microstructure is thus not ideally uniform from this point of view. The Al grains and the particle dispersion among the Al grain matrix were examined by TEM. Fig.4a and Fig.4b show TEM micrographs of Al-3Fe alloy ECAP processed after 4 and 8 passes respectively in the regions while the dominating  $\text{Al}_6\text{Fe}$  particles are round-shaped. The  $\text{Al}_6\text{Fe}$  particles mostly sit or across the Al grain boundaries. It is worth to know that there is no much difference on the Al grain size in the 4 and 8 pass processed samples, it is about 500nm.

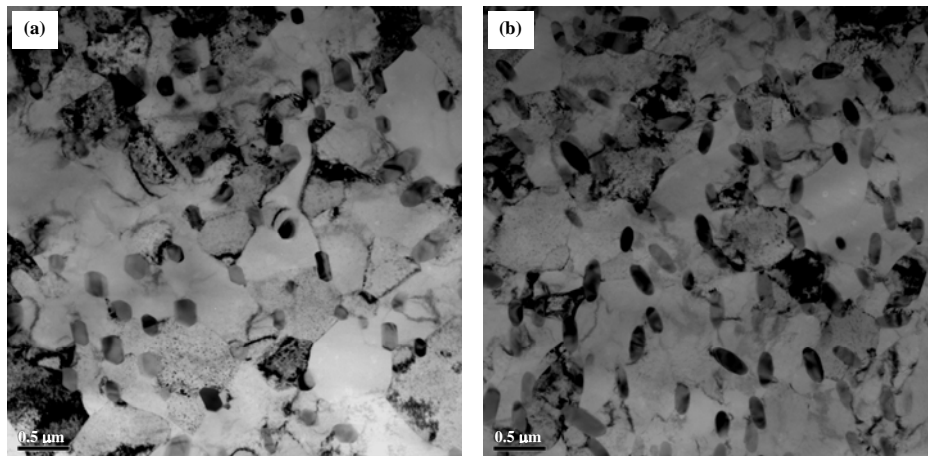


Fig.4 TEM micrographs showing  $\text{Al}_6\text{Fe}$  particles on Al matrix of equiaxed grains after (a) 4 passes and (b) 8 passes of ECAP.

There are many reports on the microstructural evolution of Al-Fe alloys during SPD processing started from different microstructure [3]. The unique microstructure of Al-Fe alloys prior to ECAP in this study, gave rise to the resulting microstructure as shown in Fig.3 and Fig.4. It should be pointed out that the formation of the phases in Al-Fe system is very complex, thus not only the feature of Al matrix but also that of the particles reported in literature is often various. Nevertheless, ECAP on the Al-3Fe alloy is efficient to generate fine microstructure of submicron meter in size. One thing remaining is the reason why the grain size of the Al matrix is insensitive to the ECAP passes. In the case of hot extrusion of Al-Si alloys while the Si particles several micrometers in size were obtained in the same way as  $\text{Al}_6\text{Fe}$  particles in this study, a microstructure consisting of equiaxed Al grains with Si particles on the grain boundaries was always the consequence, on the condition that the extrusion ratio was beyond some critical value [10]. This kind of microstructure never appeared in the cold deformed Al-Si alloys. However a short annealing at 300°C immediately led to a microstructure similar to that of hot extruded Al-Si alloys. This suggests that it is dynamic recrystallization which is contributable to the microstructure formation during hot extrusion of Al-Si alloys. At a constant deformation temperature and deformation rate, the up limit of the Al grain size is set by the volume fraction and the size of the Si particles which play role on the coarsening of the Al grains [11]. For Al-3Fe alloy, on the condition of lack of firm evidence, we postulate that the rise of the temperature together with the large plastic strain induced during ECAP triggered dynamic recrystallization of the high purity Al matrix; the growth of the Al grains was then inhibited by the dispersion of the extremely fine  $\text{Al}_6\text{Fe}$  particles in space leading to fine Al grains. This is to say that once the microstructure like that in the samples after 4 passes is formed by dynamic recrystallization, the next pass will repeat the microstructure form in the previous one making the grain size insensitive to the passes.

### 3.3. Mechanical properties of the Al-3Fe alloy

Since the samples for tensile tests are not the standard ones, we use terminology of nominal stress-strain curve instead of engineering stress-strain curve. In fact due to the different gauge section dimensions used by different researchers, evaluation of the ductility of the materials according to the recorded elongation has little meaning when necking is considered. Nevertheless from the nominal engineering stress-strain curves of the ECAP Al-3Fe alloy with 4 and 8 passes shown in Fig.5, one can still get the valuable information. Firstly, the high purity Al gains considerable strength from its grain refinement and  $\text{Al}_6\text{Fe}$  particle reinforcement induced by SPD. But with increase of deformation passes from 4 to 8, there is no significant increase of tensile strength. This is consistent with the fact that the grain size in alloy with 4 and 8 passes are similar to each other as shown in Fig.4.

It is therefore anticipated that 8 ECAP passes generated an overallly more uniform microstructure than 4 passes which led to a slight increase of tensile strength and higher elongation. Secondly, both samples after 4 and 8 passes of ECAP show a limited amount of strain hardening after yielding, necking developed in the very beginning of tensile tests. The yield strength (YS) is so closed to the tensile strength (UTS) that it seems no meaning to separate them on the nominal stress-strain curves. It is well known that necking is a result of low strain hardening rate.

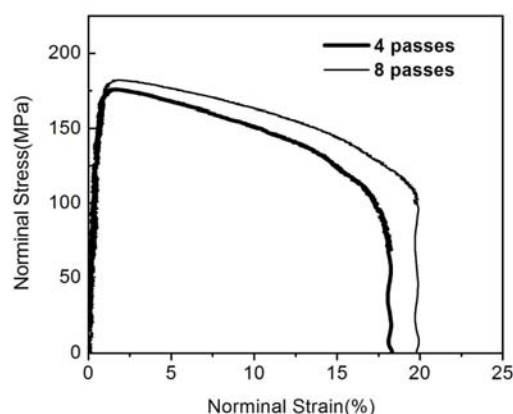


Fig.5 Nominal engineering stress-strain curves for ECAP Al-3Fe alloy with number of passes.

The comparison on the tensile properties of Al-Fe alloy in the present work and in other researcher's work can be made with reference to the work by Stolyarov *et al.* [3] because the processing conditions in [3] were similar to that in this work and the dimensions of the gauge section of the tensile samples selected in the two work differed only 0.5 mm in thickness. In the work of Stolyarov *et al.*, while Al-5Fe alloy was used, ECAP with back pressure of 275MPa resulted in YS of 200 MPa, UTS of 214 MPa and elongation of 4.1% after 8 passes of deformation. In the present work, the tensile strength was about 175 MPa, however the nominal elongation of the sample after 8 passes is about 20%. Keeping in mind that the Al grains was about 400 nm in [3] and 500 nm in the present work, this significant difference in ductility must be the consequence of the remarkable differences on the morphology, size, chemical compositions as well as the way of dispersion of the Fe-bearing particles. It was noticed that in some cases, the ECAP Al-5Fe alloy in [3] also showed phenomenon that the stress-strain curve rapidly reaches UTS after yielding and necking develops. Further work is needed to understand it.

#### 4. Summary

A microstructure free of equilibrium primary and eutectic  $Al_3Fe$  phase was obtained in high purity Al-3Fe alloy bars of 12 mm in diameter by normal solidification. The entire eutectics of Al and  $Al_6Fe$  microstructure dominate in the bars. Annealing at 550°C for 10 hours gave rise to spheroidization of the eutectic  $Al_6Fe$  rod with diameter less than 300 nm. The diameters of the resulting  $Al_6Fe$  particles ranged typically from 100 nm to 300 nm and the length of them ranged typically from 100 nm to 700 nm. The ECAP were performed with 4 and 8 passes, the resulting microstructure is featured by fine dispersion of  $Al_6Fe$  particles on the equiaxed Al grains of 500 nm in size. The similarity on the microstructure in ECAP Al-3Fe alloy with 4 and 8 pass was attributed to dynamic recrystallization induced by high plastic strain and heat generated during ECAP. Tensile tests showed that the UTS of thus formed Al-3Fe alloy is about 175 MPa. The Al-3Fe alloy fabricated in this work possesses good ductility, which can be attributed to the dispersion of fine  $Al_6Fe$  particles among the submicron-sized Al grains. We therefore introduced a new method to fabricate Al-Fe alloy with extremely fine microstructure.

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