Influence of a High Static Magnetic Field on the Morphology of Primary Al$_3$Fe Phase in Al-3\%Fe Alloy

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Abstract. It had been done the experiments of the solidification on Al-Fe alloy under a high static magnetic field (10T). The effect of high magnetic field on the morphology of primary Al$_3$Fe phase in Al-3\%Fe alloy solidification structure has been investigated by analyzing the microstructures. The experimental results show that the variation of the morphology of Al$_3$Fe phase was obvious under a high static magnetic field, and them changed to particle-like and short needles from needle-like, and they were arranged in chains along the direction of magnetic field to form oriented layered structure. The critical nucleation work reduced and the nucleation rate increased under the applied field, and the magnetic interaction caused by the field can suppress the growth of needle-like Al$_3$Fe phase, both of them resulted in the particle-like and short needles grains of primary Al$_3$Fe phase to nucleate and grow preferentially. Under the action of magnetic moment and the magnetic interaction force a high static magnetic field, the grains of Al$_3$Fe rotated and then polymerized, and finally formed chain arrangements and layer structures.

Keywords. High static magnetic field, Al-Fe alloy, primary Al$_3$Fe phase, magnetic interaction.

1. Introduction

Due to the low solid solubility of iron in aluminum, generally existing Al$_3$Fe strengthening phase in Al-Fe alloy structures, this gave the Al-Fe alloy excellent properties such as high hardness, heat resistance, wear resistance and corrosion resistance [1], and Al-Fe alloy still kept the characteristics of aluminum alloy low density, all kinds of these characteristics were important requirements of material properties for engines and aerospace components. Therefore, the development of Al-Fe alloys had broad application prospects.

Although the intermetallic compounds such as Al$_3$Fe in Al-Fe alloy had all the above excellent performances, their brittleness was also very great. Moreover, their shapes were mostly needles or flakes, which would seriously split the matrix and become a stress concentration source, this significantly reduced the mechanical properties of Al-Fe alloy. Therefore, for a long time, various methods have been tried to improve the microstructures of Al-Fe alloys so as to enhance their properties. Added Mg elements to the casting of Al-10\% Fe alloy by Li Rongde et al., so that the Al$_3$Fe phase in the alloy structure changed from developed needle shape to dendritic one, small needle (piece) one and irregular block one(flowershape) [2]. It was concluded that the addition of Mg caused the primary Al$_3$Fe phase to branch and inhibited the growth of the primary Al$_3$Fe phase, making the
structures refinement. Doping Sc and Zr in Al-Fe alloy elements, as a result of Sc, Zr and Al atoms had a stronger interaction, formed Al-Sc, Al-Zr and Al-Sc-Zr segregation areas in the alloy melt, and the precipitations of fine dispersion of Al$_3$Sc and Al$_5$Zr particles were taken place. As heterogeneous nucleating core, it had a strong grain refining effect on Al matrix grains and primary Al$_3$Fe phase grains [3]. Adding 1% Ni to Al-10Fe alloy and preparing sample by plasma injection method could refine α-Al grains and reduce the size of primary Al$_3$Fe particles [4]. The particles of primary Al$_3$Fe phase in Al-Fe alloy extruded bar prepared by the hot-top semicontinuous casting was dispersed and refined. At the same time, doping Zr and Cu can also improve the creep resistance of Al-Fe alloy extruded bar [5]. The microstructure and morphology of Al-Fe alloy can also be influenced by special preparation methods. For example, when mechanical alloying and spark plasma sintering processes were used to prepare block Al-Fe alloys, large particles with layer structure and fine strip or spot Al-Fe intermetallic compound phases appeared in the structures [6]. By applying ultrasonic wave during the solidification of Al-Fe alloy, the microstructures with fine and uniform distribution grains can be obtained [7]. By rapid solidification, the finer grains can be formed in Al-Fe alloy and accompanied by a large number of dispersion phases, which can improve the heat resistance of the alloy [8]. In recent years, the thermal speed treatment process developed (namely, the alloy liquid was overheated to a higher temperature above the liquidus, and then was rapidly cooled to a lower temperature before pouring) get a certain refining effect on the Al$_3$Fe phase [9]. Before casting, alternating current was applied into the melt of Al-Fe alloy for a certain period of time, the Al$_3$Fe phase in the solidified structure changed from thick needle-like sheet to fine needle-like, flower-like and pin point-like, and the tensile strength of the alloy was greatly improved [10]. According to the characteristics of the large density difference between the primary iron phase and the alloy liquid, a functionally gradient material with radial gradient distribution of the iron phase along the radial direction can be obtained by using centrifugal casting method [11-13].

With the development of superconducting technology, high magnetic field has been widely used in material science. When the alloy solidified in the magnetic field, regular structures of directional arrangement can be acquired [14-17], and it can be formed layered structures that solidified in the gradient magnetic field because of migratory phenomenon [18], all of which provided a new research direction for the preparation of autogenous composite materials. In addition, alloy materials undergo heat treatment in stable magnetic field refined the microstructures [19], and solidification in gradient high magnetic field made the grains of microstructures refine [20]. So far, few studies have been reported to improve the microstructures of Al-Fe alloys by high static magnetic field. In this paper, the effects of magnetic field on the morphology of Al$_3$Fe phases were researched when Al-3% Fe (mass fraction) alloys solidified from 10T high static magnetic field, and the mechanism of action on magnetic field was also discussed.

2. Experimental Materials and Methods

The Al-Fe master alloys were made from industrial pure aluminum (99.99%, mass fraction, the same below) and pure iron (99.99%), and the melting crucible and casting mould were made of ordinary graphite. To prevent oxidation, protecting slag (1:1 KCl/NaCl salts) were firstly joined in the graphite crucible and it was placed in a furnace to heat to 880°C, after melting slag, took out the crucible to join metals Al, and then put it into the furnace insulation at 880°C for 10 min, the metal Fe was added after Al completely melted. After waiting for Fe completely melted fully, mixing alloy liquid, then pouring the liquid into a mold made alloy rods of 10 mm diameter, length of about 80 mm, and the composition of alloy bar for Al-3%Fe (mass fraction). These alloy rods were cut into small segments of 20–30 mm, encapsulated in graphite tubes with an aperture of 10 mm and placed in a superconducting high magnetic field for solidification experiments. The experimental equipment was shown in figure 1. Superconducting strong magnet generated a longitudinal magnetic field, the strength of which was continuously adjustable from 0-14T. The heating furnace was placed in the cavity of the magnet, and a copper jacket water cooling system was installed between the furnace and the chamber wall of the magnet. The temperature of the heating furnace was controlled by WZK-1 temperature controller, and
the controlling precision of temperature was ±1°C. The graphite tube containing the sample was connected with the stainless steel rod by screw, and one end of the stainless steel rod was fixed above the cavity. The sample can be suspended in the heating furnace by adjusting the length of the steel rod. The sample was heated to 780°C at a heating rate of about 10°C/min to make it melt completely, kept warm for 40min, and then cooled to solidify at a cooling rate of about 18°C/min. After solidification, the sample was removed from the graphite tube and cut open along the longitudinal and cross sections. The metallographic samples were made by inlaying, pregrinding, polishing and corroding (1%NaOH solution for corrosive), and the microstructures of the samples were analyzed by Leitz optical microscope.

3. Experimental Results

Figure 2 shew the low magnification solidified structures (×100) of the cross section on Al-3%Fe under different solidification conditions. Among them, figure 2(a) was the microstructures of the as-cast parent alloys, figure 2(b) was the microstructures of the samples after remelting and solidifying at 780°C when there was no magnetic field, and figure 2(c) was the microstructures of the samples after remelting and solidifying at the same temperature when 10T high static magnetic field was applied. As can be seen from figure 2, the parent alloy’s structure was composed of typical primary Al3Fe phases and matrix. The Al3Fe phases were mainly acicular (gray-black), with a small amount of granular and blocky (seeing figure 2(a)). By comparing the microstructures of the remelting samples with that of the as-cast parent alloy samples without magnetic field, it can be seen that after remelting and solidifying, the Al3Fe phases remained acicular, while the granular and block Al3Fe phases increased (seeing figure 2(b)). In general, the microstructures of the as-cast state and remelting state were very similar, and the morphology of the Al3Fe phase was no substantially different. This indicated that the morphology of Al3Fe phase in the parent alloy cannot be changed by remelting and solidifying, and it also declared that the Al3Fe phase in the Al-Fe alloy was very stable. However, after applying the magnetic field, the remelted and solidified structures of the samples were changed significantly. There were no acicular Al3Fe phases in the structures, and the Al3Fe phases had been coarsened and appeared plum shape (seeing figure 2(c)).
Figure 2. The low magnification solidified structures of the cross section on Al-3%Fe under different solidification conditions. (a) as-cast mother alloy; (b) 0 T, solidified from 780°C at cooling rate 18°C/min; (c) 10 T, solidified from 780°C at cooling rate 18°C/min.

Figure 3. Effect of a high magnetic field on the microstructures of Al-3%Fe alloy. Among them, (a), (c) and (e) were the microstructures of cross sections, and (b), (d) and (f) were ones of longitudinal sections. (a), (b) as-cast mother alloy, none magnetic field; (c), (d) 0 T, solidified from 780°C at cooling rate 18°C/min; (e), (f) 10 T, solidified from 780°C at cooling rate 18°C/min.
In order to further observe and analyze the influence of strong magnetic field on the microstructure of Al-Fe alloy, the high magnification (×500) microstructures of the cross-sections and longitudinal sections of Al-3% Fe samples under different solidification conditions were analyzed. The solidification structures were shown in figure 3. It could be seen from figure 3(a) and 3(b) that both the cross section and the vertical section in the as-cast master alloy, the Al$_3$Fe phases in the solidified structure were needle-shaped and distributed in a disordered manner. After remelting and solidification, the Al$_3$Fe phases in the transverse and longitudinal sections were mostly needle-like and a few granular, and Al$_3$Fe phases were still randomly distributed, as shown in figure 3(c) and 3(d). Under the action of strong static magnetic field, the Al$_3$Fe phases in the transverse and longitudinal sections of remelted samples were mainly composed of granular and short-needle-like Al$_3$Fe phases, and the Al$_3$Fe phases in the cross section of remelted samples were arranged in regular chains along perpendicular to the direction of magnetic field, as shown in figure 3(e). The Al$_3$Fe phases in the longitudinal section of remelted samples were arranged in a similar regular pattern along parallel to the magnetic field direction, as shown in figure 3(f). Based on the experimental results of Al$_3$Fe phase alignment across and longitudinal sections of the sample, it was shown that Al$_3$Fe phases were arranged in regular layers and plane orientation happened when Al-Fe alloy solidified in a high static magnetic field.

4. Discussion
The above experimental results showed that the long acicular Al$_3$Fe phase in the remelted solidification Al-Fe alloy disappeared and the granular and short-needle-like phases increased with the addition of magnetic field, which indicated that the thermodynamic state and growth mechanism of Al$_3$Fe phase took place alteration under the action of magnetic field. The specific discussion was as follows:

4.1. Influence of Magnetic Field on Nucleation Rate
When the magnetic field was applied, the critical nucleation work of spontaneous nucleation was that [21]:

\[
\Delta G_n = \frac{16 \pi \sigma_{\text{SL}}^3}{3 (\Delta G_v + \Delta G_M)^2}
\]

\(\sigma_{\text{SL}}\) was the interfacial tension between the solid and liquid phases; \(\Delta G_v\) was the Gibbs free energy difference of the forming unit volume solid phase; \(\Delta G_M\) was the change of magnetic Gibbs free energy \(G_M\), which was expressed as follows:

\[
\Delta G_M = -\frac{\Delta \chi^{s-L} B^2}{2 \mu_0}
\]

In the equation, \(\chi^L\), \(\chi^S\) were the volume susceptibility of the liquid and solid phases of the alloy, respectively, and \(\Delta \chi^{s-L} = \chi^S - \chi^L\). \(B\) was magnetic induction intensity of the field. Equation (1) showed that after applying the magnetic field, the critical nucleation work decreased, which was favorable for nucleation. In addition, according to the nucleation rate equation [22]:

\[
l = l_0 \exp(-\frac{\Delta G_n^*}{kT}) \exp(-\frac{\Delta G_d}{kT})
\]

In the equation, \(l_0\) was a constant, and \(\Delta G_n^*\) was the Gibbs free energy for the formation of critical size embryos, which can be expressed as critical nucleation work \(\Delta G_n\). \(\Delta G_d\) was related to the diffusion of atoms and called diffusion activation energy, and \(k\) was Boltzmann constant. \(l_1\) and \(l_2\)
represented the nucleation rate in the absence and presence of magnetic field, respectively, then:

$$\frac{I_2}{I_1} = e \exp \left( \frac{\Delta G_{a1} - \Delta G_{a2}}{kT} \right)$$  \hspace{1cm} (4)$$

Substituting equation (1) (when there was no magnetic field) into equation (4), and then:

$$\frac{I_2}{I_1} = C \exp \left[ \frac{1}{\Delta G_g^2} \frac{1}{(\Delta G_g + \Delta G_M)^2} \right]$$  \hspace{1cm} (5)$$

Here, $C$ was the coefficient related to the surface tension of the nucleation position, and $C$ was a constant under certain solidification conditions. For the non-spontaneous nucleation, the same expression can be obtained as equation (5), except that the coefficient $C$ in the equation was related not only to the surface tension at the nucleation position, but also to the wetting angle.

It can be seen from equation (5) that $I_2/I_1$ increased exponentially with the increase of $\Delta G_M$, indicating that the nucleation rate of Al$_3$Fe phase increased significantly after magnetic field was applied. From the point of view of metallography, increasing nucleation rate was one of the conditions for grain refinement through phase transition. Therefore, after the solidification of Al-Fe alloy in magnetic field, a large number of granular and short-needle-like Al$_3$Fe phases appeared in the solidification structure.

4.2. Influence of Magnetic Interaction

For ferromagnetic materials, the interaction energy between magnetic particles can be represented by magnetic dipoles as follows:

$$U = \frac{\mu_0}{4\pi} \left( \frac{\vec{m}_1 \cdot \vec{m}_2}{r^3} - \frac{3(\vec{m}_1 \cdot \vec{r})(\vec{m}_2 \cdot \vec{r})}{r^5} \right)$$  \hspace{1cm} (6)$$

In the equation, $\mu_0$ was the vacuum permeability, $\vec{m}_1$ and $\vec{m}_2$ were the dipole moments, $\vec{r}$ and $r$ were the position vector and distance between the two magnetic dipoles, respectively. When a non-ferromagnetic material was magnetized in a magnetic field, the particles had a dipole moment due to magnetization. In a conventional magnetic field, their value was very small and their interaction was weak, so the interaction was not easy to be observed. However, in a high magnetic field, such the magnetic interaction became more obvious and had been observed by researchers [23]. According to equation (6), the interaction force between the two grains was [17].

$$F = \frac{1}{4\pi \mu} \frac{m_1 m_2}{r^2}$$  \hspace{1cm} (7)$$

Here, $\mu$ was the magnetic permeability of Al$_3$Fe phase grains in the matrix, $m_1$ and $m_2$ were the magnetic pole strength (or magnetic dipole moment) of grain 1 and grain 2, respectively, and $r$ was the distance between magnetic poles.

The nucleation rate increased when Al-Fe alloy solidified in a high magnetic field, resulting in a large number of primary Al$_3$Fe grains at the beginning of solidification. The magnetic interaction of these grains due to magnetization was determined by equation (6), and this effect inhibited the growth of the Al$_3$Fe phase along the preferred orientation of the two smooth small crystal faces (100) and (001) in the crystal direction [010], meanwhile, the growth of the rough (100) and (001) planes (non-small face) did not occur when alloying elements were doped [2]. As a result, when the sample solidified in
the high magnetic field, there were granular and short-needle-like phases of Al$_3$Fe in the solidified structures (see figure 3(e), 3(f)).

4.3. Analysis of Orientation Mechanism on Layered Plane

When the materials solidified in the magnetic field, the enhanced or precipitated phases with magnetic anisotropy (such as particles or fibers) were easy to rotate orientation and polymerize, and then formed ordered textures. The process could be roughly divided into three stages: nucleation growth, orientation and polymerization [24]. A high magnetic field was applied in this study, there were granular and short acicular Al$_3$Fe phase in the solidified structure of remelted sample (see figure 3(e), 3(f)). It was obvious that the short spiculate phase of Al$_3$Fe had been rotated to the direction of parallel magnetic field (see figure 3(f)). The results show that the Al$_3$Fe phase in the longitudinal section of the solidified sample rotated in the direction of magnetic field and formed regular arrangement, and this was also observed in the cross section of the sample. This indicated that the Al$_3$Fe phases had taken place plane orientation in the magnetic field and formed regular layer structures parallel to the magnetic field. The phenomenon of layered structure formation had been observed by researchers in the solidified structures of Al-Ni alloy in the magnetic field [25]. Studies had shown that there were two possible orientation mechanisms for magnetic anisotropic crystals in the magnetic field [26]: one was formed by rotation, and the other was formed by preferential growth of grains in the magnetic field. According to the theoretical model of rotation orientation, the fundamental reason why magnetic anisotropic crystals were affected by magnetic moment in the magnetic field was that the direction of magnetization of crystals was not parallel to the direction of magnetic field, which made the crystals rotate and form orientation. The strong magnetic field induced the paramagnetic crystal to be oriented parallel to the axis of the crystal with the greatest magnetic susceptibility, and the diamagnetic crystal to be oriented perpendicular to the axis of the crystal with the greatest absolute magnetic susceptibility. The influence of magnetic field on grain orientation was mainly due to the different magnetic susceptibility of grains in different directions. The grains were subjected to magnetic torques in the magnetic field. According to the magnetic theory [27], the magnetic moment received by the grain was:

$$T_m = \frac{1}{2\mu_0} V \Delta \chi B^2 \sin 2\alpha$$

(8)

Among them, $\Delta \chi = \chi_1 - \chi_2$, $\chi_1$ and $\chi_2$ were the magnetization rates along the grain direction of easy and difficult magnetization axes, respectively, and the two directions were perpendicular to each other. $B$ was the magnetic induction intensity; $\alpha$ was the angle between the applied magnetic field and the direction of the easy magnetization axis. The rod or small needle crystals had magnetic crystal anisotropy, and the magnetization axis of easy was in the rod or needle direction. The needle crystals completely rotated orientation under the magnetic moment $T_m$ of equation (8), as shown in figure 3(e) and figure 3(f). The experimental results show that the short-spined Al$_3$Fe phases were oriented along the direction of magnetic field. According to the rotation orientation mechanism, Al$_3$Fe was paramagnetic phase.

The effect of magnetic field on nucleation growth was mainly manifested in the refinement of structures. The addition of magnetic field increased the magnetic Gibbs free energy, resulting in a significant augmentation in nucleation rate, and then a large number of granular and acicular Al$_3$Fe phases were produced in the solidified sample. The granular and acicular Al$_3$Fe phases were arranged in chains due to the magnetic interaction between the grains. By introducing grain volume and volume susceptibility in equation (7), it could be obtained that:

$$F = \frac{1}{4\pi \mu_0^2} \frac{V_V}{r^2} \chi_1^2 B^2 \cos^2 \alpha$$

(9)
Here, $\chi_e$ was the volume susceptibility of Al$_3$Fe phase grain in the direction of easy magnetization axis, and $V_1$ and $V_2$ were the volumes of grain 1 and grain 2, respectively. For non-ferromagnetic materials, such as paramagnetic and diamagnetic materials, their magnetic susceptibility $\chi_e$ was very small, in the conventional magnetic field, the magnetic interaction between grains was very weak, $F$ was often ignored. However, in a strong magnetic field (such as 10T order of magnitude), the magnetic interaction between grains could no longer be ignored. Researches had shown that the force $F$ could reach the order of magnitude of the mutual attraction between liquid molecules [17], under which the chain structures in the sample might be generated.

5. Conclusion
(1) When Al-3% Fe alloy solidified in a strong static magnetic field, the morphology of primary Al$_3$Fe phases changed significantly. The Al$_3$Fe phases in the structures changed from acicular to granular and short spiculate.

(2) Al$_3$Fe particles formed by solidification of samples in a strong static magnetic field were arranged in chains along the direction of magnetic field, and formed a planar oriented layer structures.

(3) During the solidification of Al-Fe alloy, the growth of acicular Al$_3$Fe phase was inhibited by the addition of a strong magnetic field.

(4) Experimental results and theoretical analysis show that Al$_3$Fe was paramagnetic phase.

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