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Microstructure refinement of commercial 7xxx aluminium alloys solidified by the electromagnetic vibration technique

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Abstract. This paper examines the microstructure refinement of commercial 7xxx aluminium alloys solidified by the electromagnetic vibration technique (EMV) as a function of vibration frequency, f . The microstructure evolution reveals that at the low frequency of $f = 62.5$ Hz, the solidified microstructure is coarse and with the increase of vibration frequency to $f = 500$ Hz, the grain size becomes the finest and further increase of frequency to $f = 2000$ Hz results in coarsening of microstructures. The refinement mechanism is clarified when considering the significant difference in electrical resistivities of the solid and the liquid in mushy zone, in which both phases coexist and subject to vibration. The frequency-dependent refinement behaviour is revealed when the displacement of the mobile solid and sluggish liquid is taken into account during solidification. In contrast to 3xxx aluminium alloys, no giant compounds have been discerned in the present 7xxx alloy regardless of the solidification condition. The formation of crystalline twin is briefly discussed when considering the vibration condition.

1. Introduction

The 7xxx alloy is one of the mostly widely used aluminium alloys in aerospace engineering. To improve the performance of the alloy during service, it is always desired to produce an ingot with grain-refined microstructures that are of great significance in obtaining enhanced mechanical properties according to the Hall-Petch relation. For the refinement of an aluminium alloy billet, the most widely used technique is to inoculate the melt using TiB_2 compound that has been confirmed to be quite effective for aluminium-based alloys [1]. The problem is that both titanium (Ti) and boron (B) are rare elements with an unstable supply market, which makes the billet expensive and less competitive. Moreover, the addition of the external inoculant may deteriorate deformation properties, in particular, for the deep drawing or press processing and in addition, can increase the cost of recycling process. Hence, it is desirable to develop a novel technique, by which no external additives are required.

The electromagnetic vibration (EMV) technique is based on the Fleming left hand rule, by which a conductor is driven to move periodically upon its initial equilibrium position when an alternating current passes perpendicularly to the direction of the magnetic field. The pioneering work by using the EMV technique to the solidification field may be dated back to Vives [2,3] who investigated a series of aluminium alloys and confirmed that the EMV was effective in grain refinement when EMV was imposed at a certain frequency range. After this great work, some other researchers [4-6] improved and modified the technique and examined the microstructure formation in various metals [7,8] and alloys [9,10]. The present authors [11-13] employed this technique to solidify a series of



magnesium-based alloys and revealed that grain refinement could take place when EMV was imposed at the medium frequency interval.

In this paper, we examine the solidification of a commercial 7xxx aluminium alloy when EMV is added at various vibration frequencies. The solidified microstructure is qualitatively observed and quantitatively measured using an Image Pro software package. The refinement mechanism of the microstructure is clarified when considering the pronounced difference of electrical resistivity between the primary solid and the remaining liquid. The formation of crystal twins during solidification is briefly discussed upon the imposition of EMV processing.

2. Experimental procedures

The 7xxx billet was cast into a fine rod with 6 mm in diameter and then encapsulated into an Al_2O_3 tube. The rod was then stabilized by two graphite electrodes, from which two copper electrodes were connected. A removable arc-shaped carbon heater was covered onto the tube to heat the sample. To monitor the thermal profile during the heating and cooling process, a K-type thermocouple with a sampling rate of 10 scans per second was inserted into an as-prepared hole in the central position of the ceramic tube. More detailed procedures can be found elsewhere [14].

To diagnose the influence of vibration frequency, f , the other two parameters, i.e., magnetic flux density, B_0 , and effective alternating current, J_e , were kept as constants of $B_0 = 10$ T and $J_e = 60$ A. In each EMV run, the experimental operation was conducted with the same procedure as follows; after the sample was melted, overheated, and then held at 1023 K (750 °C) for about 120 seconds, EMV was imposed and simultaneously, the heating power was turned off and the carbon heater was pulled up to cool the sample. When the sample temperature reached 773 K (500°C), the alternating current was switched off and thus EMV was terminated to complete the vibration process.

After the specimen was solidified, it was cut, ground, polished and then etched using the traditional metallographic method. The polarized optical microstructures were observed and characterized quantitatively in order to reveal the structure-frequency dependent relation.

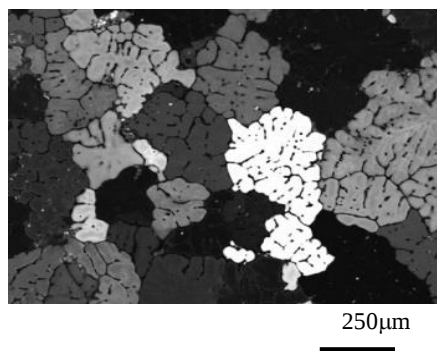


Figure 1. Polarized optical structure of a 7xxx alloy solidified at the casting state without EMV.

3. Results

In order to elucidate the influence of EMV on microstructure formation, the microstructure solidified under a usual casting state without EMV is included in figure 1, which consists of coarse dendrites with well-developed trunks (for easy description, hereafter $f = 0$ Hz). Here it should be supplemented that the sample without EMV is solidified at the same cooling rate as that with EMV, which ensures the microstructures to be comparable.

Figure 2 depicts the polarized optical microstructures of the sample solidified at various frequencies of (a) $f = 62.5$ Hz, (b) $f = 125$ Hz, (c) $f = 250$ Hz, (d) $f = 500$ Hz, (e) $f = 1000$ Hz and (f) $f = 2000$ Hz, respectively. Note that both figure 1 and figure 2 are observed at the same magnification. Qualitatively, one can see that in comparison with the structure formed at $f = 0$ Hz, the microstructure yielded at

$f = 62.5$ Hz is much fine. Overall, the size of equiaxed dendrites decreases and the number of fine grains increases. When the vibration frequency is further increased to $f = 125$ Hz and $f = 250$ Hz, the structures become finer and finer and more and more independent equiaxed grains can be observed. In contrast, there are only a few premature dendrites, as highlighted by a circle in figure 2(c). When the alloy is solidified at $f = 500$ Hz, the microstructure consists of uniform and equiaxed round grains, as shown in figure 2(d). With the increase of the vibration frequency to $f = 1000$ Hz, some undeveloped dendrites once more can be discerned, as circled in figure 2(e). As EMV is carried out at $f = 2000$ Hz, the microstructure becomes coarse with a number of developed dendrites, as depicted in figure 2(f). The general microstructure evolution versus the vibration frequency exhibits a V-shaped tendency with a minimum average grain size at medium frequency of $f = 500$ Hz and comparatively large grain sizes at both low and high frequency regimes.

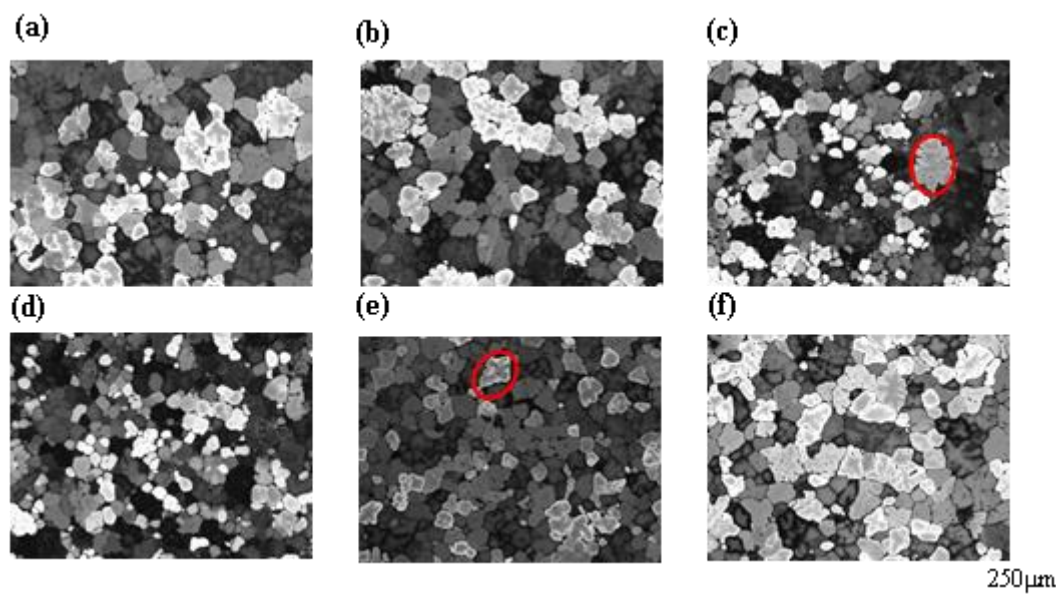


Figure 2. Polarized optical microstructures of the 7xxx aluminium alloy solidified at the vibration frequency of (a) $f = 62.5$ Hz, (b) $f = 125$ Hz, (c) $f = 250$ Hz, (d) $f = 500$ Hz, (e) $f = 1000$ Hz and (f) $f = 2000$ Hz, respectively.

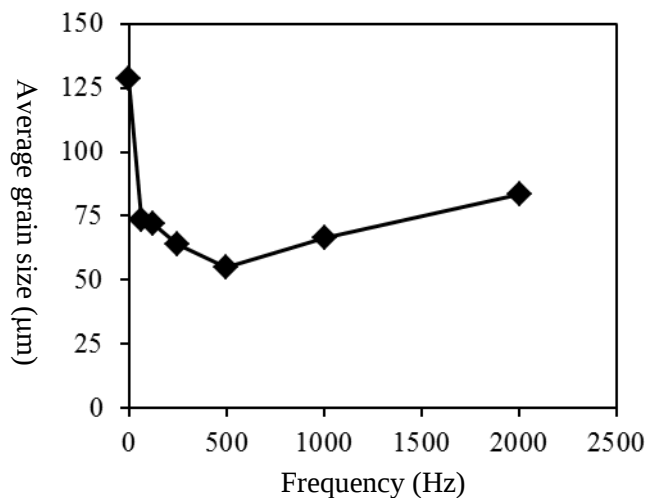


Figure 3. The measured average grain size of solidified alloys as a function of vibration frequency for the present 7xxx alloys.

To characterize the microstructure frequency-dependent relation quantitatively, the crystal grain size is measured using an Image Pro software package. Figure 3 indicates the average grain size as a function of vibration frequency, which exhibits a similar variation tendency as qualitatively stated previously. The average grain size is rather large of about 128 μm in diameter at $f = 0$ Hz and decreases drastically to about 73 μm at $f = 62.5$ Hz. When vibration frequency is increased from $f = 125$ Hz to $f = 500$ Hz, the average grain size continuously decreases from around 72 μm to the minimum of 54 μm . Further increase the frequency to $f = 1000$ Hz will result in an increase in average grain size to about 66 μm .

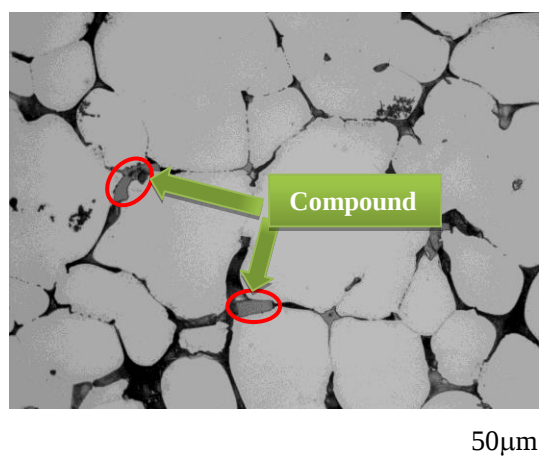


Figure 4. An optical microstructure showing the morphology of intermetallic compounds in the present 7xxx alloy

When the alloy is solidified at the present maximum frequency of $f = 2000$ Hz, the average grain size becomes as large as 83 μm . From the presentation above, it is clear that the optimum frequency for achieving grain-refined microstructures in the present 7xxx aluminium alloy is around 500 Hz when the alloy is solidified at $B_0 = 10$ T and $J_e = 60$ A. Actually, this kind of “V-shaped” transition behaviour has been confirmed in magnesium alloys [11-13].

In addition to the microstructure formation in the 7xxx aluminium alloy, whether or not the coarse intermetallic compounds can be yielded is another great concern in engineering, because the giant compounds are detrimental to make the alloy brittle during deformation processing. Yoshikawa et al. [15] summarized the conditions for the intermetallic compound formation upon crystallization in the 7075 alloy. We calculated the equivalent solute amount for compound formation of the present alloy and found that the total equivalent solute amount of the alloy is still less than the critical boundary for compound formation, indicating that it is hard to yield giant compounds. However, this does follow that no compound can be found in the alloy. On the contrary, intermetallic compounds can be identified at grain boundaries when the microstructure is observed under a large magnification. Figure 4 shows an optical microstructure with a high magnification. Very fine compound particles with the size of about tens micrometres can be identified, as highlighted in red ellipse in figure 4. In comparison with the considerable amount of Mn in 3xxx alloys, the entire volume of intermetallic compounds in the present 7xxx alloy is rather small and negligible. As the present authors [16] have examined the formation and refinement of compounds in 3xxx alloys in detail, here we omit to repeat the topic.

4. Discussion

From the experimental results presented above, one can readily raise two questions as follows; i.e., (1) why can EMV produce grain refined microstructures compared with that formed at the usual casting state and (2) why the microstructure evolution is frequency-dependent in the present 7xxx alloy, that is, why can the “V-shaped” transition behaviour be identified?

The first question deals with the refinement mechanism that has been a great concern for decades and one of the most striking mechanisms was that cavitation occurred during vibration and thus resulted in grain refinement [17]. Here it should be mentioned that by using a high speed digital imaging system, Shu et al. [18] have confirmed that cavitation could indeed take place in liquid at ultrasonic frequency band and can induce breakup of crystals from dendrite arms, which can lead to multiplication of grains and thus result in refinement. However, in terms of microstructure refinement using the EMV technique, the most effective frequency regime is usually at sonic frequency range of hundreds of hertz, e.g., in the present 7xxx alloy and other magnesium-based alloys [11-13], which are two orders of magnitude lower than the well-accepted definition for ultrasonic frequency, i.e., higher than 16000 Hz in physics. So it is doubtful whether or not it is suitable to transplant the phenomenon in ultrasonic band to sonic regime. Furthermore, there have been no reports that cavitation can really occur during sonic vibration.

With this discrepancy in mind, the present authors propose a new model when considering the electrical properties of the solid and liquid in mushy zone. The electrical resistivities of pure Al melt at the melting temperature is $242 \text{ n}\Omega\cdot\text{m}$, whereas the value is $110 \text{ n}\Omega\cdot\text{m}$ [19] for the solid at the same temperature, i.e., only about half in quantity as that of liquid. For an Al alloy, the overall electrical resistivity of the liquid is estimated to be about twice of that solid. As the solid and the liquid in the mushy zone are to be a parallel circuit, the electric current in the solid, $J_e(s)$, is about twice that in the liquid, $J_e(l)$, according to the Ohm's law, i.e., $J_e(s) \approx 2J_e(l)$. Hence, according to Newton's second law of motion and electromagnetic theory, the solid in the mushy zone is subject to twice Lorentz force of that liquid and thus the solid is driven to move with twice acceleration of that liquid due to their similar densities. Consequently, the solid moves faster with a higher velocity and thus results in a longer displacement in comparison with that of the liquid within half vibration cycle, which have been quantified elsewhere in detail [11].

Based on the analysis stated above, it is concluded that uncoupled motion occurs between the mobile solid and the surrounding sluggish liquid. Considering that the mushy zone is confined within an alumina tube rather than in free space, one can readily tell that melt flow can be initiated to take place during EMV processing, which has been demonstrated to be effective in promoting grain multiplication during solidification [20].

As far as the second question is concerned, we should consider the melt flow intensity that is closely related to vibration frequency. The non-synchronic motion between the primary mobile solid and the sluggish liquid produces the relative leading displacement, Δs , which decreases per a square relation with the increase of vibration frequency [11]. At $f = 50 \text{ Hz}$, the relative displacement of the leading solid spans about 64 mm, which is much larger than the diameter with 6 mm for the Al_2O_3 tube. Under an extreme condition that a solid particle is driven to move across the diameter, the time duration for movement is about $\Delta t = 6/(60 \cdot 2f) = 0.001 \text{ s}$ within a half cycle, i.e., only 10% of the half cycle interval of 0.01 s. This indicates that within nearly 90% duration time of a half cycle, solid particle are impeded to stop around the tube wall and kept to be still as the sample is encased in the sealed container. This means that at this vibration frequency, the total moving time for solid is rather short while the dormant time is quite long. Therefore, only weak melt flow can be induced at this low frequency and thus microstructure is relatively coarse.

With the increase of vibration frequency, the relative displacement decreases. When $f = 500 \text{ Hz}$, Δs is about $570 \text{ }\mu\text{m}$ that is larger than the thickness of the boundary layer and thus it is sufficient to move out of the regime of the solute boundary layer. In this case, a stable solute distribution boundary cannot be established ahead of advancing front and hence, refinement can be effectively produced without dormant time. When frequency is further increased to $f = 2000 \text{ Hz}$, Δs is decreased to about $35 \text{ }\mu\text{m}$ that is much less than the thickness of the layer. In this case, although uncoupled movement occurs between solid and liquid, this short distance is still within the boundary regime and thus it cannot break out the hierarchy of solute distribution during solidification. This is why the microstructure once more becomes coarse. Considering the relation between the relative displacement

and vibration frequency one can readily tell the origin of the “V-shaped” microstructure evolution during EMV processing.

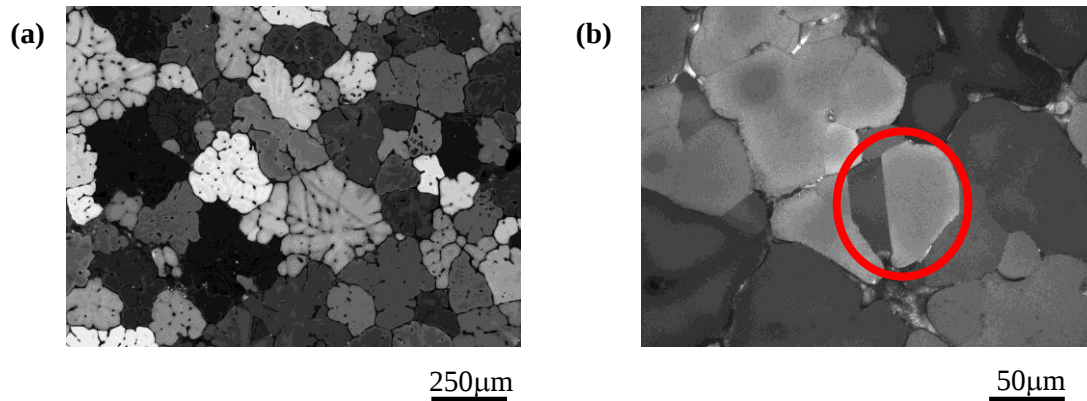


Figure 5. The microstructure solidified (a) at the usual casting state without EMV showing no crystal twins and (b) with EMV indicating deformation twins in the present face-centred cubic aluminium alloys due to the imposition of Lorentz force

The impact of Lorentz force, for one aspect, can produce refined structures during EMV processing, and for the other aspect, can result in the formation of crystal twins. Figure 5 depicts two microstructures solidified (a) without EMV at the usual casting state and (b) with EMV. For the aluminium alloy with a face-centred cubic structure, there are virtually no twins in the as-cast alloy [21], which can be verified in figure 5(a). When EMV is imposed, the Lorentz body force is added to the specimen. The Lorentz force is directional, while crystallographic orientation of grains is random, as the polarized microstructures reveal in figure 2. In this case, deformation twins may be produced when the Lorentz force is higher than the critical resolved shear stress for twin formation for those grains, in which their easy deformation crystallographic directions are consistent with that of the Lorentz force. This is why twins can be observed in certain grains, as highlighted in figure 5(b) in red circle while no twins can be discerned in other grains.

5. Conclusions

We examined the microstructure formation of the 7xxx aluminium alloy subject to EMV processing. The microstructure is coarse at the low vibration frequency and the average grain size decreases with the increase of frequency and eventually reaches the minimum of 54 μm at $f = 500$ Hz and then increases gradually when frequency is increased up to 2000 Hz in the present experiment. The refinement should be attributed to the uncoupled movement between the mobile solid and sluggish liquid because of a significant difference in electrical resistivity for the solid and liquid at high temperature. As the relative leading distance is strongly dependent on frequency, the “V-shaped” microstructure evolution behaviour can be achieved. No giant intermetallic compounds have been found regardless of imposition condition of vibration while, in terms of deformation twins, they can be yielded only in certain grains due to the directional feature of Lorentz force when EMV is imposed during solidification.

Acknowledgements

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