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Some Considerations on the Structure Refinement in Al-Based Alloys

Ildiko Peter and Bela Varga

Abstract

Grain size is one of the most important characteristics that affect the processing and in turn the properties of alloys. Grain refinement determines many advantages in light alloy casting: it can be achieved using different methods, based on the available technological possibilities and on the performances that one has to obtain. By using grain refinement, important benefits can be reached, for both cast and wrought aluminium alloys: among other, the most important enhancement regards the fine distribution of the second phases, improved castability, reduction of shrinkage porosity, higher mechanical properties, as well as superior fatigue life. The present chapter is not exhaustive on this argument; however, in the first part it reports some current literature data and some perspectives about the grain refinement, while in the second part which has been mostly carried out within a current PhD Thesis focalized on the improvement of the properties of Al-based alloys by physical grain refinement methods, some experimentally obtained results have been presented and discussed.

Keywords: Al-based alloys, structure refinement, grain refinement, metallurgical methods, physical methods, morphology

1. Introduction

Due to their excellent properties (high strength-to-weight ratio compared to Fe-based alloys, good corrosion resistance, etc.), light alloys (Al- or Mg-based alloys) have a key role in various engineering applications. Generally, during traditional casting process, dendritic microstructure is developed. Dendrites can be organised in columnar or equiaxed structures in agreement with the production conditions, which involve among others, the solute concentration, the heat gradient, the solidification velocity, the presence or absence of inoculating elements, etc. The control of the shape and distribution of the grains structure is made up of...
primary importance and really governs the properties of the produced alloy. In many conditions, improvement of the mechanical strength is required, especially in application where structural properties are essential. Thermal treatments and/or reducing the grain size of the constituents are commonly used methods for the improvement of some properties. Addition of grain refiners (GR) determines an increase of the mechanical properties, and in particular, it affects the strength and the fatigue resistance of the alloys, and at the same time, the quality of the cast part and the efficiency of the casting process rises [1]. According to some literature date [2–5] and some own research [6–11], one can state that the grain size is one of the most important characteristics, which affects the processing, and consequently, the properties of alloys. Presence/addition of GR determines many advantages in light alloy casting and it can be achieved using different methods, as will be presented later on. Actually at industrial level, both adding grain refiner and improving solidification conditions are two procedures for grain refinement, especially in case of Al alloys.

The reduction of the grain size has a positive effect on the microstructural homogeneity determining a fine distribution of the second phases leading to an improved castability, lower micro-porosity and shrinkage porosity as well as a reduced segregation development [4, 6].

In any case, the alloy composition has to be carefully considered in order to choose an efficient GR. Even if, Al–5Ti–B master alloy is an efficient GR for wrought Al alloys, its effect is not sufficient in case of foundry alloys: formation of Ti–Si phases occurs following the reaction between the solute Si and Ti determining the weakening of the nucleating of TiB₂ particles in the master alloy, when the Si content in the alloy is higher than 3 wt.% [12, 13].

According to [14, 15], in case of master alloys containing high amount of B and higher than 4–5.5 wt% of Si, an improvement of the grain refining can be obtained. Prior to casting, an inadequate holding time negatively influences the GR efficiency and additionally their agglomeration can take place [16–18]. For Mg alloys, Zr is the ideal GR, but its use is unsuccessful when it contains Al.

Conventional GR for Al alloys are generally produced from Al–Ti–B ternary system [19–22].

On the contrary of Fe-based alloys, in case of light alloys, the primary structure after solidification is maintained and the physical and any structural transformations induced following thermal treatment and/or plastic deformations take place within the primary structure. Any outcomes coming from such technological treatments are influenced by the characteristics of the primary structure. Additionally, light alloys have higher tendency during solidification for the development of the coarse grains with respect to iron-based alloys. From these considerations, it is evident that for light alloys, the influence of the crystallisation process with the aim of the development of fine grains and the good distribution of the different phases within a homogeneous microstructure results very important and it significantly influences the properties of the final product during their use. The schematic illustration of the relation between the structure and the properties is reported in Figure 1. The use of the different methods for the improvement of the structure in case of light alloys significantly influences the properties related to their use and beneficially acts on the technological properties and processing by plastic deformation as well on their behaviour during thermal treatment.
Solidification of the alloys is a process, which involves the passage from the liquid state into a solid state as a result of the energy loss during the release of the heat and shows at least two aspects:

1. Germination: the step when development of the nuclei or crystallisation centre has been realised. The evolution can be:
   - Homogeneous: when the centres have been developing spontaneously and has been growing during the time;
   - Metallurgical processes:
     - Casting
     - Plastic deformation
     - Thermal treatment

![Diagram](http://dx.doi.org/10.5772/intechopen.71450)"Figure 1. Schematic representation of the relation between the structure and the properties."

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Heterogeneous: when the development takes place close to the existent germs on the surface of the melt (on the wall of the die, near to some hold-up in the melt or during the precipitation of some phases in mini-crystal forms) and can be favoured by the introduction of some particles as modifier into the melt;

Mechanical: such effect is induced by mechanical ultrasonic vibration and/or by using turbulent convection currents.

2. Development and growing of the crystals by the combination of the atoms from the melt on the crystal surface in form of monoatomic layers with critical size representing bi-dimensional germs [23].

During very slow cooling, a preferential direction of the heat release could be observed. During the development of dendrites, the dendrite arms are growing encountering during their expansion other neighbouring dendrites. When crystallisation finishes, the evolution continues on the perpendicular direction with respect to the former one and the whole crystal grows into a complex form.

Industrially, there is the need to obtain enhanced quality product. To realise this, one has to consider and satisfy some of the following conditions:

- To increase the cooling rate and the number of the crystalline germs, simultaneously with the reduction of the solidification time in some regions of casting;
- Managing the heat transfer process and the mass transfer, which directly influences the size of crystalline germs, determining the development of a fine equiaxed structure;
- Homogenising as much as possible the structure;
- Degassing the melt;
- Reducing the presence of internal stress avoiding in this way the crack formation [24].

The solidification process can be governed by the control of the time required for the cooling and acting directly on the alloy, on the casting profile or on the final product. The control of the solidification is significantly complex especially when the alloy shows high shrinkage in liquid state and during solidification.

**Figure 2** summarises the most important methods used in order to act on the solidification. Application of a magnetic stirring field to the melt, which solidify has a grain refining effect too. There are two types of magnetic stirring:

- Electromagnetic stirring, which stimulates the development of an equiaxed crystalline zone; refinement of the solidification structure occurs with a decrease of the inclusions content and
- Permanent magnetic stirring, which has some benefits over the first one, because it consists of lower cost tools, high magnetic field intensity and with no skin effect.
Moving backward the stirring direction of the electromagnetic stirring, the oxide film on the melt surface can be protected, which is important for maintaining it undamaged. Contrarily, the layer can be fragmented by the forced melt flow losing its protective character. Some studies [23, 25, 26] reveal how the effect of a magnetic field on the crystal direction and on the phase arrangement during solidification is a valid method to increase the physical properties of the alloy.

In [23, 27, 28], the authors have demonstrated that the rotating magnetic field can be effectively used to reduce the segregation appearance during solidification and to enhance the structure of metallic alloys.

In order to shortly appreciate the variations taking place during solidification, Figure 3 reports, schematically, the enhancement of the structure using high cooling rate contemporary with the addition of some chemical element: the refinement of the structure and consequently, the improvement of the general behaviour is evident.

Figure 2. Schematic representation of the most important methods used for the refinement of the structure.

Figure 3. Schematic representation of the main modifications in hypoeutectic Al-Si alloy.
2. Experimentally used devices and methods

In the following part, some results of the current research performed within a recent PhD Thesis oriented to the enhancement of the characteristics of Al-based alloys by physical grain refinement methods that have been presented and talked over, including some real case studies.

The purpose of using the dynamic methods is twofold, as follows:

A. For enhancing the structure of the alloys reaching a fine microstructure and a homogeneous product, both chemically and structurally and

B. For reducing the scrap formation during casting with a consequent time saving possibility.

Both foundry and deformable alloys have been used for such investigations. The study is realised analysing:

• The effect of the vibration during solidification on the development of the microstructure, lowering the porosity content and furthermore enhancing the mechanical properties;

• The effect of magnetic stirring applied on the melt, considered as a clean solution for the hating and for the treatment of the alloys; such approach allows, with no any interaction, the necessary thermal and mechanical energy for obtaining a homogeneous alloy with fine grains and enhancement of the mechanical properties;

• Solidification with high cooling rate, leading to positive results concerning the refinement of the structure, a method which allows obtaining high performance product with no any thermal treatment.

The alloys, with the chemical composition used for the research have been casted and details on such part have been found in some previously published paper [6–12]. The vibration system, the electromagnetic stirring system and the device used as electromagnetic induction system have been projected and realised during the present research and here is shortly presented. Results on the benefits of the physical treatment performed, with some comparison related to other similar condition for the solidification have been reported and discussed integrated with some preliminary considerations related to their eventually technological transfer. Additionally, enhancement of the structure following high cooling rate using different processing methods: melt spinning, static and dynamic casting have been recalled with some consideration about the final benefits. The analysis are carried out and the results obtained can be used as guide-line for people working in this area in an economically convenient way, saving time, energy and materials.

The research has been carried out using binary and more complex alloys, belonging to the families: Al-Si, Al-Cu, Al-Zn and Al-Mg. The chemical compositions have been reported in the previously published papers [6–12].

The mechanical vibration of the melts during solidification has been performed using a laboratory device, usually employed in a dental laboratory with the details reported in Figure 4. The parameters adopted for the experiment are as follow:
- Voltage: 110 V – 220 V/60 Hz ± 10%
- Power: 100 W
- Acceleration: 0.8–0.07 m/s²
- Velocity: 2.8–0.22 mm/s
- Amplitude: 0.07
- Dimension: 14 × 13.5 × 11.3 cm
- The device projected for the research has made possible the variation of the frequency in the range of 4–94 Hz.

Castings have the size as reported in Figure 5 and the K type thermocouple has been introduced in the position indicated in figure, at about ¾ height of the crucible’s total length.

Castings has been obtained in a static and dynamic mode and the chemical composition has been selected to evaluate as well as the influence of the working condition on the nature of the phases developed during solidification (primary phase, solid solution and intermetallic particles, eutectic phase). Structural modification has been monitored following the dynamic solidification condition for the hypoeutectic Al-Si foundry alloy and for the deformable Al-Cu, Al-Zn, Al-Mg alloys. The effect of the dynamic condition on the areas containing columnar crystals has been assessed by observing some samples when solidified with high cooling rate, using cooling channels placed around the crucible (Figure 6). Directed solidification has been performed on AlSi7Mg0.3 and on AlZn10Si7 alloy to study the effect of the mechanical vibration on the properties of the columnar disposition of the dendrites.

The tool reported in Figure 7 has been used for the study of the solidification in rotating electromagnetic field. The parameters adopted are: tri-phasic power supply, nominal power 1.5 kW, frequency 50 Hz.

 Casting, industrially, has been performed using the forms reported in Figure 8, and they have been carried out using different cooling rates (guaranteed by the different material used for the casting).

![Figure 4](http://dx.doi.org/10.5772/intechopen.71450)
Figure 5. The shape and the size of the forms used for the casting; (a) crucible in refractory material and (b) thermocouples placed for monitoring the cooling [10].

Figure 6. Scheme of the casting (a), crucible with the cooler and the thermocouples before located casting (b) and the samples with no vibration and under vibration [10].

Figure 7. Experimental set-up (a): A-inductor coils, B-casting shape in refractory material, C-Al alloy, D-thermocouples; (b): Rotating electrical field generator, (c) command block and (d) system for the registration of the frequency [8, 10].
The experimental device (Figure 9) realised for the simulation of the solidification in different cooling rates has the benefits to associate the cooling technology of the melt jet on spinning disc with the principle of the moulds feeding from low pressure casting procedure. In case of low pressure casting, the transfer of the liquid metal from the furnace to the die cavity placed on the top of the furnace, is guaranteed by an overpressure acting on the liquid alloy enclosed in the crucible, which is hermetically sealed. As a result, the liquid metal is forced to go vertically through the connecting tube. In case of the experimental device indicated in Figure 9,
the working gas (Ar) introduced by the cover 6 acts upon the liquid metal 4 from the crucible 3 causing the fused metal raising in the feeding tube 5 followed by the melt placement on the rotating disc. The furnace and port-crucible is (1), respectively (2).

The diameter of the feeding tube (5) corresponds to 1 mm and the distance to the disc results to be 5 mm. The disc rotation speed is 2600 rot/min and the working gas pressure is 1.5 bars. The aforementioned parameters have an important role, since they establish the thickness of obtained ribbons. Major details on such devise used can be found in [10].

3. Experimental study results

3.1. Casting in steel die, refractory material die under vibration

AlCu4 and AlSi7Mg0.3 alloys have been casted in steel die, refractory material with no vibration and with vibration (f = 50 Hz, a = 15 m/s², v = 14 mm/s, A = 0.07 mm). The cooling curves are reported in Figure 10. Higher cooling rate can be observed when the alloy is subjected to vibration compared to the static cooling condition, confirmed by the microstructural analysis: the finest microstructure corresponds to the high cooling rate and in the presence of vibration for both alloys, as reported in Figure 11.

Directed solidification has been performed on AlSi7Mg0.3 and AlZn10Si7 alloy to investigate the influence of the mechanical vibration on the properties of the columnar zone. For this experiment, cylindrical casted samples positioned on Cu rings (Ø30 × 13 mm, weight 22 g) have been used. The thermocouples for the registration of the temperature variation as a function of time has been mounted on the top and on the bottom part of the refractory mould at a distance of 15 mm from the exterior part of the sample, as reported in Figure 12. The experiment has been carried out both with no vibration and under vibration conditions, using different parameters and varying the frequency in the range of 50÷90 Hz. The resonance frequency is 62 Hz and the other parameters adopted are: acceleration = 8.8 mm/s²,
velocity = 99 mm/s² and amplitude = 0.7 mm. The distance from the cooler has been considered during the microstructural analysis and the most important areas (top, middle and bottom) have been analysed.

The finest grains have been obtained for the areas situated at the bottom part, which is in direct contact with the cooler and as the distance increases, the microstructure becomes coarser as reported in Figure 13. Following vibration, the roughness of the sample increases (Figure 14).

The results have the same tendency concerning the microstructure evolution (Figure 15) and for the roughness in case of AlZn10Si7 alloy (Figure 16) as well. There are some differences as their workability concerns: the second one is harder and seems to be more sensible to the impurities, which are on their surface. In this case, there are segregation of intermetallic particles made of Fe. With no vibration, trans-crystallisation is observed (columnar crystallisation), while in case of vibration, the equiaxed grain crystallisation has been observed.

On the upper part of the sample with vibration, some darker zones have been observed, while in the region in contact with the cooler development of finer grains have been observed. The lighter and longer appearance segregations are made of Fe, coming from the scraps re-used and this is one of the causes related to the more difficult workability. The bottom part of the sample is made of the solid solution and contains segregation of Si (darker colour than the Fe-made particles).

Industrially employed alloys have been investigated. In the case of hypoeutectic AlSi7Mg0.3 alloy is assumed that enhancement of the microstructure determines refinement of the microstructure and the modification of the eutectic phase. For the eutectic composition AlSi12CuMgNi alloy with complex composition, the vibration is of interest both for the structure of the dendrites and for the morphology of the eutectic and for the shape and distribution of the intermetallic particles segregation. For the binary AlCu4 alloy, it is important that the refinement of the dendrites of solid solution and for the development of non-equilibrium eutectic phase.
3.2. Effect of the vibration and of the cooling velocity on the solidification condition

Comparison of the effect of the vibration and of the cooling velocity on the solidification condition has been carried out. For the thermal analysis, samples of size 14×80×160 mm have been used, labelled as C00. Figure 17 (left) reports the results of the thermal analysis for the AlSi7Mg0.3 alloy. The analysis of the cooling curves indicates that in case of vibration, the...
Figure 12. Set-up of the directed solidification experiment [10].

Figure 13. OM microstructures of the different areas within the AlSi7Mg0.3 alloy after directed solidification [10].

Figure 14. Photographs showing the roughness of the samples in AlSi7Mg0.3 alloy with and with no vibration (a), top part of the sample under vibration (b) and with no vibration (c) [10].
The cooling rate is higher. In both cases, the liquidus temperature is 613.35°C, while the solidus temperature (eutectic) for the vibrated sample is 1.25°C lower than the sample without vibration (Figure 17 right hand side).

It can be noticed that the eutectic transformation has a more evident appearance in the presence of vibration. The maximum cooling velocity in liquid state is 4.6°C/s and 6.7°C for the non-vibrated and vibrated sample, respectively. In case of the sample solidified under vibration, the primary solidification and the eutectic transformation take place at higher cooling velocity compared to the situation with no vibration. The period of the transformation is significantly different for the two samples: in case of vibration, a decrease of both the extent of the crystallisation and of the period of the eutectic transformation of about 50 and 31%, respectively occurs. Such reduction is determined by the increase of the cooling velocity.

Figure 15. OM microstructures of the different areas within the AlZn10Si7 alloy after directed solidification [10].

Figure 16. Photographs showing the roughness of the samples in AlZn10Si7 alloy with and with no vibration [10].
For the AlCu4 alloy, the maximum cooling velocity in liquid state is (Figure 18):

- 1.15°C/s for the sample with no vibration;
- 1.6°C/s and 2.1°C/s for the samples solidified in refractory moulds;
- 17°C/s for the sample solidified in steel moulds (C00).

It can be noticed that as the vibration increases, the cooling velocity significantly increases also in the biphasic domain (liquid + solid). C0 corresponds to the liquidus temperature, the peaks labelled as C1 makes in evidence the temperature when the development of solid solution dendrites concludes, while C2 (very evident) indicates the formation of the eutectic Al-Al$_2$Cu phase. The region correspondent to the formation of the eutectic is preceded by the overcooling of the alloy, intensified by the vibration of the melt. The sample, solidified under vibration, shows higher roughness: intensification of the heat exchange between sample and mould occurs, appreciated by the value of the overall heat transfer coefficient, which in the presence of vibration has a favourable effect on the interaction between the alloy and the
material made up the mould. [7, 10] reports more details on the calculation on the overall heat transfer coefficient, based on the cooling curves.

Figure 19 reports, for comparison, the optical microstructure of the alloys investigated after solidification (no vibration and under vibration). In the case of hypoeutectic AlSi7Mg0.3 alloy (Figure 19 top), the vibration during solidification determines, first of all, the reduction of the size of the separation of Si and of the intermetallic particles, both in length and width. The α solid solution dendrites becomes more uniform and of rounded shape, with no significant variation of their size (130 μm). For the AlSi12CuMgNi alloy (Figure 19 middle), the secondary dendrite arm spacing under vibration is reduced (from about 70–40 μm) and the proportion of the eutectic phase is significantly increased and refinement of the Si particles occurs. In the case of the binary AlCu alloy (Figure 19 bottom), the effect of the vibration determines the reduction (from about 245 to 200 μm) of the secondary dendrite arm spacing and more globular grains have been obtained. Additionally, the Al,Cu particles become more fragmented.

As cooling velocity increases, the dendritic parameter decreases. Under vibration, germination of the gas bubbles is favoured and there is no promising condition for their expulsion from the melt because of the reduction of the viscosity and of the formation of the solid phase.

Figure 19. OM microstructure of the alloys investigated: Top: AlSi7Mg 0.3, middle AlSi12CuMgNi and bottom AlCu4; left hand side with no vibration, right hand side under vibration during solidification [9, 10].
The results obtained indicate an increase of the overall heat transfer coefficient value. However, they do not determine the enhancement of the cooling velocity in such a level to provide a significant modification of the casting structure. The schematic interdependence of such elements are summarised in Figure 20.

The effect of the rotator electromagnetic field on the structural evolution of light alloys has been further investigated. The casting of some cylindrical samples (Ø30 x 100 mm) in ZnAl4Cu1 and AlSi7Mg0.3 alloy has been performed in refractory mould. Under the electromagnetic field action, the melt is subjected to the centrifugal force, like in the traditional centrifugal casting, driving the liquid metal against the die walls of the mould. In the refining procedure, such effect can be considered as a limiting factor and has to be minimised using a refractory material placed on the superior part of the mould when the electromagnetic field is applied. During the experiments, it has been found that the effect of the centrifugal electromagnetic field really has an effect when the frequency arrives to a certain value. At the same time, when the electromagnetic field is coupled, the rotation speed of the melt caused by the electromagnetic phenomenon in the system coil-melt decreases and do not permit sufficient grade of agitation. For this reason, after reaching the frequency, coupling-decoupling and the modification of the rotation senses have been carried out.

Such device has been used for the preliminary investigations using a low melting point alloy, in particular ZnAl4Cu1 alloy.

In case of the hypoeutectic ZnAl4Cu1 alloy, a significant modification takes place: the transformation of the columnar dendrite into a more globular shape and the appearance of a higher level (18%) and finely dispersed eutectoid phase can be observed, as pointed out in the phase diagram. During solidification, the electromagnetic field stimulate the rearrangement of the alloying elements. The evolution of the microstructure is reported in Figure 21.

During solidification in the case of AlSi7Mg0.3 alloy, the electromagnetic field determines a decrease of the grain size and the α solid solution globular grains development arises. The eutectic Si does not suffer any significant modification neither in shape nor in size. In the central part of the sample, refining of the α solid solution has been observed and a higher distribution of the eutectic phase has been reached. The most relevant microstructures have been reported in Figure 22.

In the first step of the research, the influence of the cooling velocities (encountered in the different casting technology processes) on the structure development has been analysed.

![Figure 20. Schematic representation of the interdependence between some features affecting the solidification of the alloys.](http://dx.doi.org/10.5772/intechopen.71450)
Higher the cooling rate finer becomes the structure, as reported in Figure 23. When the cooling rate is lower, the presence of acicular Si phase and the separation of the acicular shape particles made of AlFe can be observed. In case of rapid solidification, displacement of the structure in the hypoeutectic zone can be observed with the appearance of an α solid solution. At the same time, the acicular separation of small eutectic Si particles appears. Because of the high cooling rate, Fe is present as AlFeMn intermetallic particles in a polygonal shape (lighter shade than the primary Si particles). In case of casting under pressure, the morphology is close to the hypoeutectic structure, where the separation of the eutectic Si and the intermetallic particles are globular (the length/weight fraction tends to 1) and they are uniformly spread out close to the α solid solution. The chemical analysis carried out confirms that the differences between the structures observed are associated to the different solidification conditions and cooling rate.

The presence of Fe as Al9Fe2Si2 or Al12Fe3Si determines minor plasticity to the alloy, which can be balanced by high cooling rate, decreasing the negative effect especially in the case of secondary foundry alloys, where the presence of Fe is higher.

3.3. Casting using melt spinning technology

Significant structural changes, represented by the appearance of α phase, have been obtained in hypereutectic Al-Cu alloy produced by MS-LP technology. Applying a liquid quenching technique, increased solubility of Cu in the Al matrix was reached up to a value of 25.51 wt%.
After annealing treatment applied to melt-spun ribbons, the coarse separations of the intermetallic phase ($\text{Al}_2\text{Cu}$) was observed, which results to be embedded into the Al matrix with high plasticity and low content of Cu. Due to a grain refined structure of melt-spun ribbons, $\text{Al}_2\text{Cu}$ incoherent phase is uniformly distributed in the annealed ribbons, as illustrated in Figure 24.

### 4. Conclusions

Grain refinement determines many advantages in Al-based alloy casting and it can be reached by both metallurgical and physical methods. By using grain refinement method, for both cast and wrought aluminium alloys, a fine distribution of the phases, better castability, decrease of shrinkage porosity, higher mechanical properties, as well superior fatigue life were obtained.
However, the purpose of this chapter was twofold, it is not exhaustive on this topic. The first section was dedicated to present a short outlook about some possibilities to refine the structure of Al-based alloys based on some literature data and continues with the presentation and discussion of some experimental results obtained recently during the research performed within the PhD Thesis [10] targeted to the improvement of the properties of Al-based alloys by physical grain refinement methods.

The solidification process can be governed by the control of the time required for the cooling and acting directly on the alloy, on the casting profile or on the final product. Generally, the electromagnetic field and solidification under vibration are advantageous determining a decrease of the grain size; consequently in such conditions, a fine microstructure development is favoured. It comes out that significant microstructural changes can be obtained using physical methods, which based on the characteristics of the alloy composition and on the real use of the final component can be managed in such a way to produce a better quality Al-based alloy castings.

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