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Melt refining and purification processes in Al alloys: a comprehensive study

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TOPICAL REVIEW

Melt refining and purification processes in Al alloys: a comprehensive study

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Abstract

Aluminum and its alloys having lots of advantageous properties are among the most-used metallic materials. So, it is of immense importance to find suitable processes and methods leading to high-quality purified Al melt. In this regard, there are numerous challenges in achieving high purity Al melts, such as its propensity to react with air, oxygen, and water vapor, the presence of a variety of oxide, non-oxide, and solid particle inclusions that lead to the production of pores, cracks, pinholes, and dross, finally adversely influencing the overall quality of the product. The main methods of melt refining are fluxing, floatation, and filtration, but more sophisticated methods have also emerged. The best method for purification can be chosen based on the type of impurities and the desired level of purification. With the industrial development, the need to establish more cost-effective and simpler methods has increased, and in addition, methods should be considered for recycling large volumes of scrap Al parts that contain more impurities. Moreover, achieving high purity melt is also a vital issue for use in specific applications. The present article has been written to discuss the above issues and focus on the study of various methods of aluminum purification.

1. Introduction

The favorable characteristics of aluminum alloys, such as lightweight, exceptional high-strength to weight ratio, excellent corrosion resistance, suitable plasticity and ductility, electrical and thermal conductivity, non-toxicity, resilience, and suitable weldability make the Al and its alloys an ideal material for automobile and aerospace industries. Despite the so-called encouraging attributes, these alloys suffer from the generation of inclusions, pinholes, dross, coarse grain, severe surface crinkles, etc. During the melt processing and casting, these undesirable defects cause several metallurgical defects and cracking, reducing the final quality of products and declining the popularity of the Al alloys in the market [1, 2]. In the process of aluminum alloy smelting, chiefly hydrogen and oxide inclusions pollute the aluminum melt. Molten aluminum during the melting process reacts with air, specifically with water vapor and oxygen resulting in the generation of aluminum oxides and hydrogen. It is known that aluminum is quite prone to react with oxygen leading to the production of Al_2O_3 , Al_2O_2 , or sub-alumina (AlO_2 and AlO) [3]. At the same time, it is also very inclined to absorb hydrogen gas (H) which accounts for about 70%–90% of the total amount of gas in the aluminum melt. Additionally, the main defects of aluminum alloys as pores and slag inclusions are due to solid particles and the remained gas and oxides in the alloy [4, 5]. The accumulation of stress in the periphery of the various pinholes and inclusions substantially deteriorates the mechanical properties of the alloy. Also, the presence of non-metallic inclusions with poor wettability toward aluminum melt is very undesirable since they are suspended in the melt and serve as the nucleation sites of porosities [6]. Moreover, they can adversely affect the melt fluidity and decrease the efficiency

of the riser [7, 8]. Furthermore, the existence of inclusions and other defects can decline the corrosion performance of alloys since they can act as initiation sites for pitting corrosion [9]. Therefore, the refining and purification treatment of the melt is of paramount importance in Al alloys. Accordingly, the main goal of melt refining and purification procedures is to diminish any undesirable substances, such as inclusions, oxides, and hydrogen to an acceptable level to promote the overall performance of the alloy.

Numerous types of inclusions can be found in Al melt, such as carbides, borides, oxides, nitrides, fluorides, iron oxides, chlorides, molten salts, etc [6]. According to the type, wettability, and density of the mentioned inclusions, they can either float or agglomerate inside the melt. For instance, oxides and inclusions with low wettability relative to Al melt are prone to agglomerate, while some inclusions like Al_2O_3 adsorb gas bubbles and float on the melt surface [10]. Without the sophisticated melt refining and purification process, these oxide inclusions will be retained in the aluminum melt, inducing many troubles and leading to the deterioration of mechanical properties, machinability, and surface quality [11]. In addition to oxide inclusions, some kinds of non-oxide inclusions can be found in Al melt, such as TiB_2 , Al_4C_3 , MgAl_2O_4 , etc, these nonmetallic inclusions can lead to poor toughness behavior [12]. The various impurities in Al melt can also be categorized into solid inclusions and dissolved impurities. Solid inclusions typically are simple oxides, silicates, aluminates, exogenous inclusions, and spinel-like phases. The most common type of impurities in this category are Al_2O_3 , MgO , and Al_4C_3 [13]. Also, commonly foreign metals (such as Na, Li, Ca, Fe, Si, Cu) and dissolved gas are found in the dissolved impurities category. The only dissolved gas in this group is hydrogen since it is not prone to fabricate compounds with Al like other gases. Also, Al has a low solubility magnitude toward hydrogen gas, and it has to be removed because it leads to the production of gas pores [13]. Considering the variety of mentioned inclusions, impurities, and pollutants and their substantial adverse effects on the final properties of Al alloys, it is of crucial importance to implement systematic purification and refining processes on Al melt. Hence due to the substantial impact of melt quality on the final microstructure and properties, studying melt quality and its improvement is of high importance. Accordingly, the presented study is aimed to thoroughly address and explain the purification and melt refining processes and techniques. Firstly, traditional melt refining techniques such as fluxing, floatation, and filtration were introduced. Secondly, more recent and modern Al melt purification methods such as sedimentation and distillation technologies, liquation and segregation processes, and high purity refining techniques were discussed. Finally, as examples, the purification of Al scrap and some Al alloys such as Al-Mg and Al-Si series was explained.

2. Traditional melt refining methods

Three fundamental, traditional, and commonly utilized processes to refine aluminum melts with various impurities to meet the requirements include fluxing, floatation, and filtration [14]. These three main processes have similar processing steps which apply to eliminate the variety of inclusion and impurities within the Al melt. These main processing steps involve pre-treatment crucible, casting furnace, degasser, and filter [14], as shown in figure 1. There are numerous methods for Al refinement and purification, each of which has its own pros and cons with specific applications. In this regard, this paper categorizes these technologies in two main groups, including traditional methods leading to low-to-medium purification levels and methods leading to high and ultra-high purity levels. Accordingly, table 1 lists the main traditional methods with their pros, cons, and specific applications.

2.1. Fluxing

One of the popular and most widely adopted procedures to purify the molten Al and remove oxide inclusions such as oxide films is through fluxing. Fluxes are usually solid substances (mixtures of chloride and fluoride salts) that can remove impurities and modify the melt fluidity. The most general solid fluxes include KCl , NaCl , NaF , AlF_3 , and MgCl_2 , and the well-known fluoride salt additions can be named Na_3AlF_6 (cryolite), CaF_2 , KAlF_4 , and Na_2SiF_6 [15]. Fluxes activate simply by furnace's high temperatures and reduce the melting points of all impurities in the crucible and facilitate their oxidation, so the impurities rise to the surface as slag. The prime duty of fluxes is to prevent the oxidation of molten bath that increases by raising the furnace temperature [16]. Moreover, fluxes utilize to diminish penetration of the hydrogen, absorb non-metallic inclusions of the melt, separate the built-up oxides, reduce the entrapped Al content in the dross, evacuate the dissolved hydrogen, assist the Al grain refinement during solidification, modify silicon inclusions, and oxidize excessive magnesium [6, 14]. Usually, fluxes are poured on the melt surface and stirred. They can also be injected into melt with the powder form in an inert gas stream or can be injected effectively by rotary degasser. There are numerous types of fluxes, including grain refiners, cover fluxes, cleaning fluxes, drossing fluxes, degassing fluxes, wall cleaning fluxes, silicon modifiers, and damaging fluxes; each of which has its specific applications and benefits. Overall, fluxing is the most popular procedure for the effective removal of non-metallic inclusions from the Al and Mg

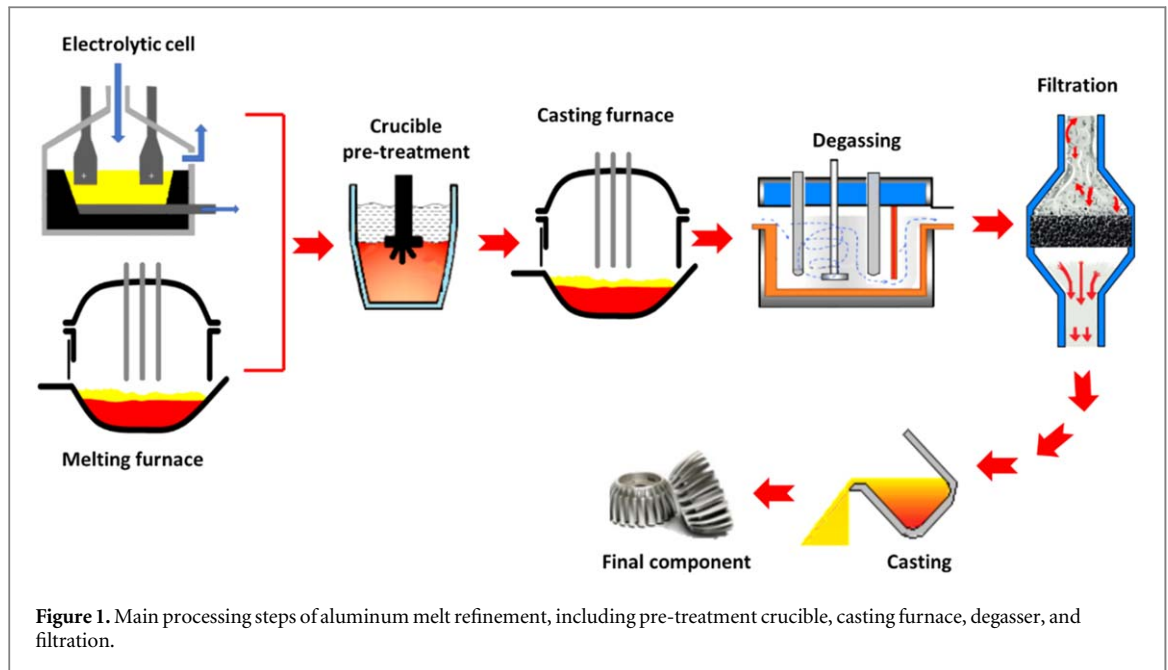
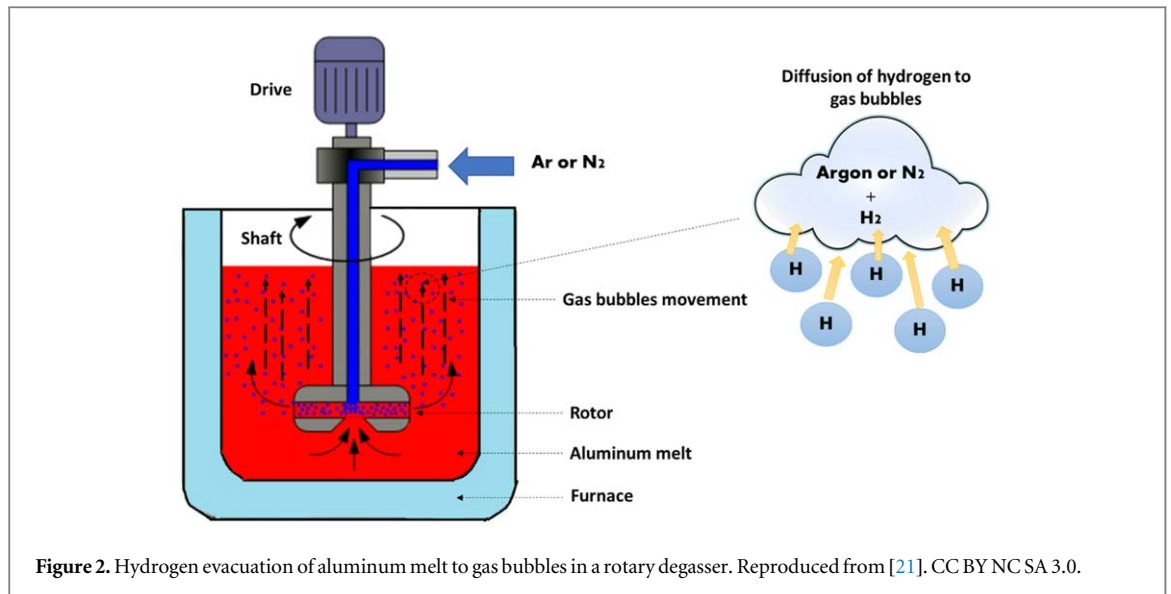


Table 1. Main traditional Al melt purification methods with their pros, cons, and specific applications.

Process	Pros	Cons	Main application
Fluxing	Removing oxide inclusions, reducing the penetration of hydrogen, absorbing non-metallic inclusions, assisting grain refinement, melt modification, having a lot of kinds for various applications.	Need solid additions, low contact with molten Al leading to low purification performance, needs to the high amount of fluxing materials, toxicity, utilization of toxic substances, it is not able to produce high purity levels of Al melts.	Removal of non-metallic inclusions.
Floatation	Easy transformation of hydrogen gas to melt surface, providing high surface area-to-volume ratio, eliminating the use of harmful chlorine and fluorine-containing salts, it can remove non-metallic solid particles.	Usually needs rotary systems, need to fast prevention of off-gas, it is a step-wise process, can remove non-metallic solid particles, it is not able to produce high purity levels of Al melts.	Reduction of hydrogen and inclusions content.
Filtration	It can remove any contaminants, non-metallic inclusions, trapped oxides, extraneous particles, etc, it has numerous kinds with specific applications.	The type of used filter has an influential effect, it is not effective for finer inclusions, it is not able to produce high purity levels of Al melts.	Removal of relatively large inclusions.
Sedimentation	Able to remove elements with a tendency toward oxidation, can separate both oxide films and primary intermetallic compounds.	Smaller inclusions are settled much slower than the large ones to the bottom of the furnace.	Removal of intermetallic phases.
Distillation	Efficient and effective removal of impurities such as Sb, Bi, Al, Au, Ag, Cu from melts.	Need to reach the boiling point of elements, need precise temperature control, needs vapor collection and condensation, the complexity of design and optimization, expensive method.	Removal of specific elements from Al melt.
Liquation	Efficient and effective removal of impurities with low melting points.	The necessity of having divergence in the melting point, needs high temperatures and expensive.	For the refinement of metals with a low melting point that has impurities with high melting points or vice versa.

melt. The solid fluxes (generally inorganic salts) soak up the inclusion particles, fabricating a conglomerate that can be easily separated from the Al melt by flotation and from the pure melt through sedimentation. This phenomenon is directly relied upon in their densities [17].

Recently, fluxes such as fluoride, chlorine salt, and carbonates are used extensively in the aluminum processing industry. Fluxes have a great potential in removing magnesium, calcium, sodium, etc from Al melt, they are also can act as catalysts to reach the equilibrium oxidation reactions. This leads to the fabrication of



more stable chlorides and fluoride forms so they can be collected easily as sedimentation or dross materials based on the resultant density. For instance, AlCl_3 addition will initiate the following reaction $\text{Mg} + \text{Cl}_2 \rightarrow \text{MgCl}_2$ and production of MgCl_2 with a lower density compared to Al melt so that it can be collected as dross [18]. The main limitations of fluxes relate to their insufficient contact with molten Al, preventing the full removal of inclusions. Thermodynamically, inclusions are prone to transfer from molten Al to molten flux. However, inclusions are covered by a layer of molten Al which hinders their movement toward molten flux. If this interface layer becomes thin and torn, the movement process is facilitated [3]. Another drawback can be due to the necessity to use a large magnitude of fluxes to attain the desired melt condition. For example, it was reported that for full-removal of about 1.0 kg of magnesium, 2.95 kg of chlorine should be used [18]. In addition, chlorides and fluorides lead to the production of toxic and dangerous gases that subsequently should be filtered. Therefore, the issue of providing the necessary contact, required flux magnitude, and toxicity issues should be considered by using fluxes.

2.2. Floatation

Flotation as an efficient procedure in reducing hydrogen and inclusions content in the melt has been used widely in the Al melt refining and purification processes. In this regard, bubble floatation is among the proficient purification technologies for Al melt treatment in which thousands of bubbles are purged into the melt by the flush of inert gas bubbles. These bubbles convey the inclusions in suspension to the melt surface to be eradicated [19, 20]. Flotation methods are also applicable in copper and precious metals and are among the initial processing steps following the casting furnace. The floatation procedure can also be referred to as degassing, in which a combination of argon, nitrogen, and chlorine gasses are utilized to clear away the hydrogen through floatation. As the melt gets in touch with the rising gas bubbles, the hydrogen atoms diffuse to the bubble periphery surface and combine in order to form hydrogen gas. Hence, hydrogens are collected inside the bubbles, and bubbles are float on the surface of the melt. So, they can be transferred easily to the outside of the system. Nowadays, foundries usually utilize rotary degassers. During this method, inert or chemically inactive gas such as argon or nitrogen is disposed of by a rotating shaft and rotor. The fabricated large number of fine bubbles have a very high surface area-to-volume ratio suitable for collecting a large amount of hydrogen gasses in melt [21]. In fact, a large surface area leads to effective and quick diffusion of hydrogen into the gas bubbles resulting in equalizing activity of hydrogen in liquid and gaseous phases, consequently full-hydrogen removal without any need to use harmful chlorine and fluorine-containing salts. In this regard, figure 2 schematically shows a rotary degasser. In order to avoid the re-entering of hydrogen into the melt, the off-gas should be cleared immediately. Usually, the hydrogen removal is performed in a step-wise manner so two or more rotary degassers are used in sequence. Moreover, a low amount of chlorine gas should be used in this process to attain full-hydrogen removal since it can also remove additional alkali elements. Additionally, the presence of small bubbles helps to remove the non-metallic solid particles through floatation [22]. Moreover, it should be noted that Al melt flow has a considerable impact on floatation efficiency, Mirgoux *et al* [19] reported that enhanced fluid flow can improve the collision probability between the bubbles and unwanted inclusions leading to attain better purification levels. Also, during the turbulent fluid, flow inclusions are more prone to collide with each other, leading to agglomeration and improving the distribution of inclusion size.

2.3. Filtration

Filtration of molten metals is very similar to filtration of aqueous suspensions, and it has almost the same principles. Filtration methods can be used productively to remove any contaminants, non-metallic inclusions, trapped oxides, extraneous particles, etc in the molten metal. During the filtration procedure, the liquid metal passes through a packed bed of refractory particles, screens, ceramic foams, etc to separate any second-phase particles in the size range of 1 to 30 μm . The inclusion removal rate largely depends on the filter type, the porosity of the filter, filter roughness, length of the bed, filter area, melt temperature, melt flow rate, etc [23, 24]. There are numerous types of filters, including bed filtration (BF), ceramic foam filters (CFF), first rigid media filters (RMF), bonded particle tube filters (BPF), two-stage filter systems, surface-active filter systems, etc Note that generally, the used filtration mechanisms in foundries are from two major types of cake and bed filtration, in which these two types usually happen in a combined and sequential manner. In the initial stages, cake filtration occurs by sieve effects separating larger inclusions. This method is often used for Al melt with high content of large-sized inclusions. In contrast to cake filtration, bed filtration is more usual for Al melt but it has some complexities. The bed filtration process happens through direct collision or adhesion of inclusions to/at the filter surface, sedimentation based on gravity or inertia forces, and particles' collision by Brown's movement or fluid dynamic effects. Bed filters (BF) usually consist of Al_2O_3 balls or chips, having a size range of 2–8 mm [13]. In addition to the BF method, utilization of ceramic foam filter (CFF) produced from labyrinthic structured ceramic materials is among the widespread and popular purification methods of molten aluminum due to its high porosity, high surface area, economic nature, and simple application [25]. It was reported that bonded ceramic particle filters can enhance the efficiency of the filtration process up to 90% for the inclusions in the range of 10 to 25 μm . However, they are not so much effective for finer inclusions. In this regard, high porosity CFF types can significantly improve the filtration performance for the case of finer particles. Also, the utilization of active coatings can prevent the filter blockage and help to trap the non-metallic inclusions [25].

3. Recent and more modern Al melt purification methods

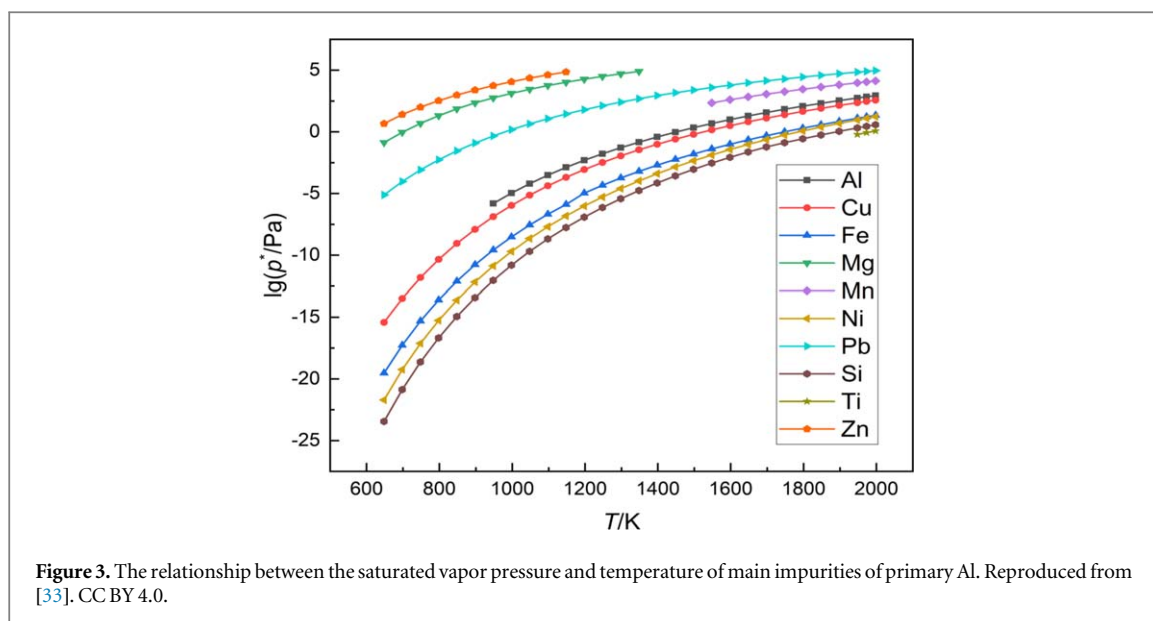
3.1. Sedimentation and distillation technologies

In sedimentation processes usually, inclusion particles with high densities are forced by the use of additional melt holding time and energy consumption to settle to the bottom of the furnace melt. Additionally, this process can be applied to any existing metallic elements in a melt with a tendency toward oxidation, such as calcium and magnesium elements in Al melt [18]. Since sedimentation methods are based on Stokes law, smaller inclusions are settled much slower than large ones to the bottom of the furnace. Primary intermetallic compounds and impurities can be precipitated and sedimented from the Al melt, and sedimentation processes are able to separate both oxide films and primary intermetallic compounds. It was reported that iron removal can be fulfilled with the rates of 24% up to 71% by sedimentation separation processes [26, 27]. Annadurai *et al* [28] reported the successful removal of Fe-rich intermetallic compounds from 356 secondary Al alloy through sedimentation of liquid aluminum by alloy addition. In this regard, sedimentation temperature and the relevant conditions have been measured through thermodynamic evaluations and solidification curves. It was deduced that isothermal sedimentation of Fe content can be achieved by the addition of 0–2 wt% Mn and Cr into Al melt. Consequently, the Fe-rich sludges, including $\text{Al}(\text{FeMn})\text{Si}$, $\text{Al}(\text{FeCr})\text{Si}$, and $\text{Al}(\text{FeMnCr})\text{Si}$ are produced and sedimented in which Mn increases the sludge particle size, and Cr refines and increase the aspect ratio of precipitate morphology [28]. It was found that Fe and Mn content can affect the precipitation and sedimentation process and the morphology of precipitations [26]. It was determined that a larger Mn/Fe mass ratio leads to lower Fe fractions in the melt [27]. The sedimentation process can considerably affect the overall liquid melt quality due to the separation of Fe content and subsequent mechanical properties of the final product by preventing the possible oxide-induced crack formation [29].

In addition to sedimentation methods, distillation processes can also be utilized for the purification of Al melt. In the distillation process, a metallic melt is kept at a regulated temperature and vapor pressure range. In order to remove the specific element from the melt, the melt is heated above the boiling point of this specific element while the temperature should be kept under the boiling point of Al and the other existing metals. Meanwhile, in the case of precious impurities, vapor collection and condensation can also be done. One of the well-known categories of distillation processes is in the basis of the vacuum and very low-pressure conditions known as the vacuum distillation method.

3.1.1. Vacuum distillation method

Vacuum distillation based on distillation processing usually is implemented under reduced pressure. This prevents easy distillation at ambient pressures and makes the purification of compounds more proficient with saving time and energy. This technique separates various compounds according to their differences in boiling



points. Also, it is mostly utilized when achieving the boiling point of the desired compound is troublesome or leads to the compound decomposing [30]. It should be noted that reduced pressures decline the boiling point of compounds. This reduction can be calculated using a temperature-pressure nomograph through the Clausius-Clapeyron relationship [31]. From theoretical prospect in the prediction of the power of impurities' separation by vacuum distillation, the saturated vapor pressure and the separation coefficient b are two main criteria, so under the same conditions, more saturated vapor pressures can lead to easy volatilization into the gas state. The success of the separation process can be understood from the condensation of elements on the cooling area, proving that some elements successfully escape from the liquid melt. Accordingly, the saturated vapor pressure and temperature can be related to equation (1) [32].

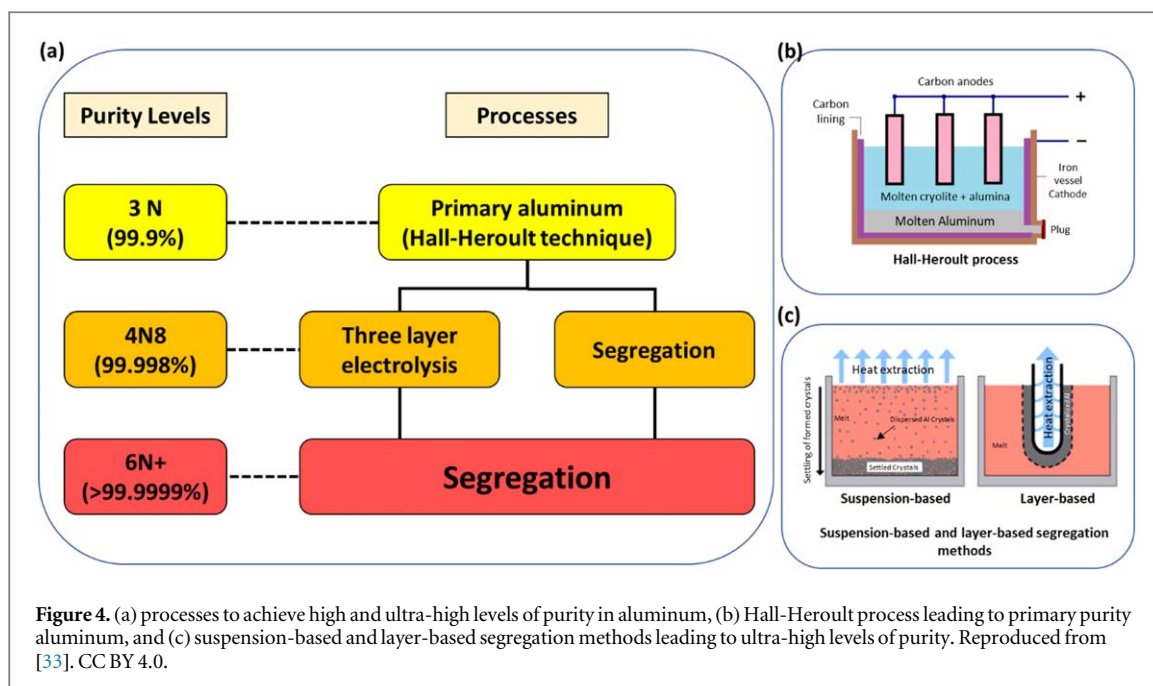
$$\log p^* = A.T^{-1} + B. \log T + C.T + D \quad (1)$$

where p^* and T denote the saturated vapor pressure (Pa) and temperature (K), respectively. Also, A , B , C , and D are evaporation constants. In this regard, figure 3 represents the relationship between the saturated vapor pressure and temperature of main impurities of primary Al. According to figure 3, it can be deduced that Zn, Mg, Pb, and Mn have higher vapor pressures than Al. So, they can be preferentially volatilized and separated by the distillation method. While some elements like Fe, Ni, and Si cannot be volatilized and will remain in the melt, elements with quite similar vapor pressure to aluminum, such as Cu are very difficult to be removed by distillation [33]. Accordingly, the Al distillation process can be performed in two steps. The first step can separate the volatile impurities such as Zn, Mg, Mn, and Pb, and the second step of distillation can be utilized to distill and purify the Al itself from residual impurities.

The vacuum distillation method having numerous advantages such as excellent recovery performance, impurity removal in the metallic state, simplified flowsheet, economic nature, and ecological superiority mostly is used to refine impurities such as iron, silicon, magnesium, and zinc from aluminum melts, the influential parameters during vapor distillation of Al alloys are vapor pressures, temperature, time of distillation, and the state of the initial melt [34]. Accordingly, Wei *et al* [34] reported that in the high vacuum condition (~ 18 Pa) and high temperature (~ 800 °C) and processing duration of ≥ 30 min, the zinc constituent of Al melt can conveniently be declined from 4.86% to 0.666%. In the first stages of distillation after 15 min, the Zinc content reached 1.55%, then by increasing the duration up to 60 min the content decreased to 0.666%. Unfortunately, further increases in processing time did not cause significant changes in zinc content. In another study, Katsuaki *et al* [35] investigated the vacuum distillation of 7075 Al alloy and studied the vaporization rate of the alloy content within vapor by using the Langmuir formula and Raoult law. The results show a good agreement between simulation and experiment under the vaporization condition of 1500 °C. They concluded that vacuum distillation can successfully separate Cu, Al, Cr, and also Mg and Zn elements.

3.2. Liquation processes

Liquation-based processes are among the efficient metal-refining methods rooted in variations of the solubility of impurities during melt cooling [36]. These processes are performed by heating the material till one of the constituent elements begins to melt while the others are still solid. Subsequently, the liquid melt is removed and separated from the other, and collected. Initially, the liquation method was used to extract antimony minerals



from ore. It should be noted that liquation-based techniques are utilized for the refining of metals with a low melting point that has impurities with high melting points or vice versa. In fact, the system must have a divergence in the melting point of the constituents. For instance, Li *et al* [37] utilized a selective liquation procedure to extract pure Al from an Al-Si alloy by adopting zinc as the solvent. During this selective liquation process, the Al with high purity in the Al-Si alloy is dissolved in the zinc melt, producing a type of α -solid solution with zinc. This leads to the precipitation of the silicon and iron-rich phases and Al-Si-Fe intermetallic. They finally transform into massive grains and accumulate as slags that can easily be separated. The Al extraction rate has been reported to be in the range of 35%–40% [37]. Zhao *et al* [36] utilized a liquation process to purify metallurgical grade silicon and efficiently remove the undesirable B and P elements. This is carried out due to the low melting temperature of Si by alloying with metal.

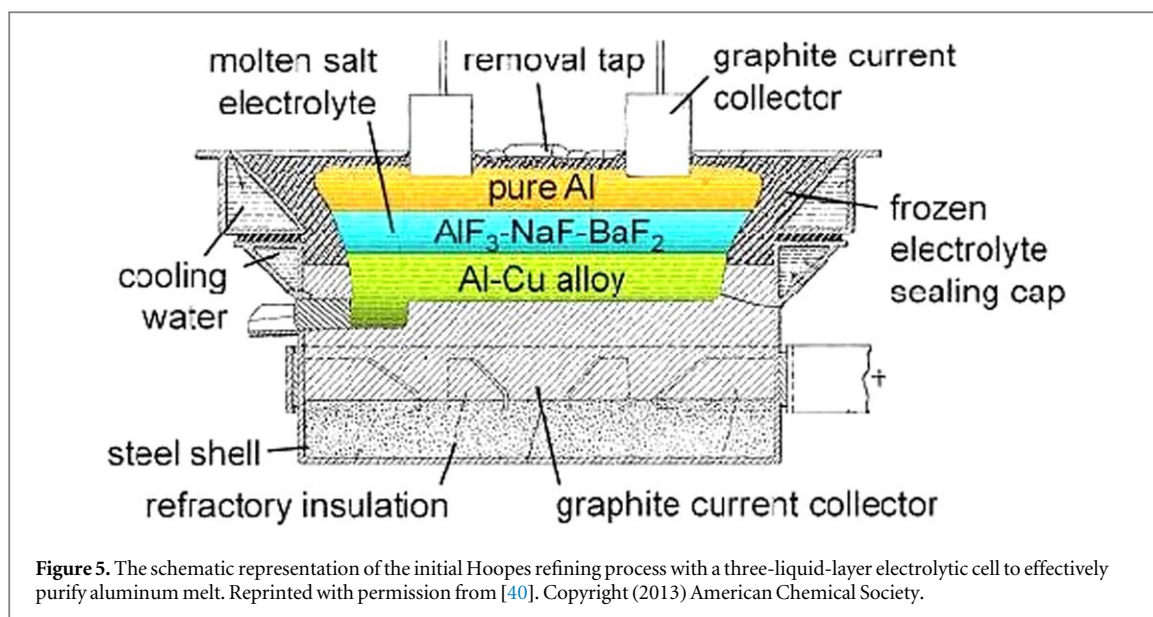
3.3. High purity purification techniques

High purity Al has unique properties such as low magnetic permeability, high strength, and plasticity at low temperatures, elevated thermal and electrical conductivity, etc and after oxidation, it will have substantial corrosion resistance and electrical insulation property [33]. Therefore, high purity Al products can be utilized in the integrated circuits, substituting Cu and Au wires. These high purity level Al wires can also be utilized in very large-scale integrated (VLSI) circuits and they can be used in superconductor devices running at cryogenic temperature ranges up to -269°C [33]. Other possible applications of ultra-pure Al products are transistors interconnects (5 N purity), magnetic disks substrates (6 N purity), low-temperature superconducting magnets (6 N purity), semiconductors, integrated circuits (IC) fabrication (4 N8 purity), anode foils for electrolytic capacitors (6 N purity), cathodes for organic-light emitting diode (OLED) (6 N purity), sputtering targets (6 N purity), liquid crystal display (LCD) panels (6 N purity), etc [38].

It is possible to achieve high purity aluminum products on an industrial scale through three-layer electrolysis procedures and fractional crystallization methods as well as their simultaneous employment as series can even lead to ultra-high-purity aluminum products. For unusual and non-industrial scale applications, vacuum distillation and organic electrolysis can also be utilized [33]. Currently, in order to achieve the purities surpassing the primary production (99.5%–99.9% produced from Hall-Heroult technique), mainly the three-layer electrolysis and the segregation (fractional crystallization) techniques are utilized [18]. The processes to achieve ultra-high-purity aluminum have been shown in figure 4. As it can be seen, the three-layer electrolysis method is not capable to reach purity levels higher than 4N8–5N, and production of higher purities (5N+) needs a contribution of both methods in which three-layer electrolysis should be continued by segregation or could be achieved solely by segregation and through some processing repetitions [38].

3.3.1. Hoopes process and three-layer electrolysis

The as-discussed traditional Al refining technologies cannot satisfy the requirements to attain high purity Al melt ($>99.97\%$ or 3N7). To this end, in early 1920, Dr William Hoopes of the Alcoa Research Laboratories in



collaboration with Dr Francis Frary invented the Hoopes refining Process [39], as illustrated in figure 5 [40]. By 1938, the purity reached 99.996% in France through the modified Hoopes process. This process is also called a three-layer process involving three density separated layers consisting of a bottom layer with an aluminum-copper alloy serving as the anode, a middle layer with a molten electrolyte, and the top layer of molten purified aluminum. Aluminum is added to the anode section, and its purity gradually increases as it electrolytically moves towards the cathode layer since impurities cannot migrate. This process has three electrolytic layers that require high temperatures of 700 to 900 °C and high energy amounts. For instance, the initial production uses approximately 14 kWh kg⁻¹ energy. Due to the required high temperature and considerable energy consumption, this method is only suitable for high purity Al production [41]. Moreover, Unlike the discussed Hoopes refining Process that necessitates the use of high temperatures and considerable energy amounts, lower temperature electrolysis has a much more economic nature and can also lead to the production of high-quality Al parts up to 99.89% purity [28]. In this process, the need to be refined aluminum (serves as an anode) is put in the anhydrous aluminum chlorides solution as an ionic liquid, but unfortunately, this method cannot be used in melt [42, 43].

The modern three-layer electrolytic cell is continuously improved to increase the purification level and reduce costs. In this regard, figure 6 schematically shows the main construct of three-layer electrolysis in which the outside shell is steel while the inside section of the enclosure has an anode role made by carbon block with refractory bricks for heat preservation. The wall which is in contact with the melt is fabricated by refractory magnesia bricks, and there is a cathode manufactured from graphite in the upper part of the chamber [44]. In this technology, the commercial-grade Al is dissolved in copper to produce an Al-Cu master alloy with about 33% Cu to act as a high-density anode (3.4~3.7 g.cm⁻³) at the bottom of the chamber. In fact, Cu is used to increase the density and reduce the melting point. The electrolyte which is usually a composition of fluorides or chlorides such as Na, Ba, Al, Ca, and Mg is located in the middle layer with ~2.8 g.cm⁻³ density, while the refined high-purity Al with ~2.3 g.cm⁻³ density accumulates in the upper layer. These density differences and the presence of the electrolytic layer in the middle layer certify the stability and full separation of the Al-Cu and high-purity Al layers during electrolysis [41]. After several processing repetitions, the Al purifies further, and the impurities are concentrated in the anode. Overall, the electrolysis process is founded on the differences in electrode potential of metallic ions present in the electrolyte.

3.3.2. Segregation methods

Unidirectional solidification (or zone melting) and fractional crystallization are two categories of segregation methods that are designed based on the thermodynamic behavior of dilute eutectic binary systems. The first one as an expectant method still is in the research and development phase and can be used to purify bars of aluminum metal [45]. In this method, performing a precise control over melting and resolidification drives the undesired impurity and inclusions to migrate and concentrate in a specific region. This is usually done by the gradual movement of the Al bar into a ring-shaped furnace, producing a moving mobile molten zone. As the Al bar cools, the refined Al crystals are fabricated, and impurities and inclusions are kicked back into the molten zone that subsequently can be removed. The rate of this kickback procedure governs the purity degree and recrystallization speed [46]. On the other hand, the other method known as fractional crystallization refining is

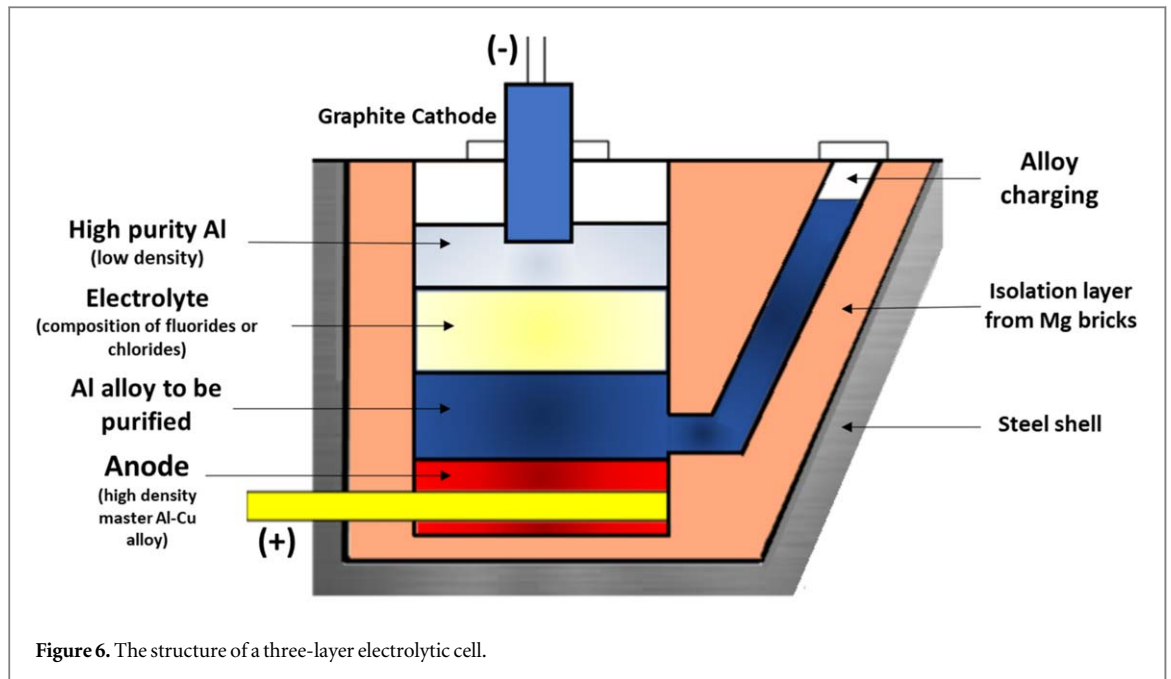


Figure 6. The structure of a three-layer electrolytic cell.

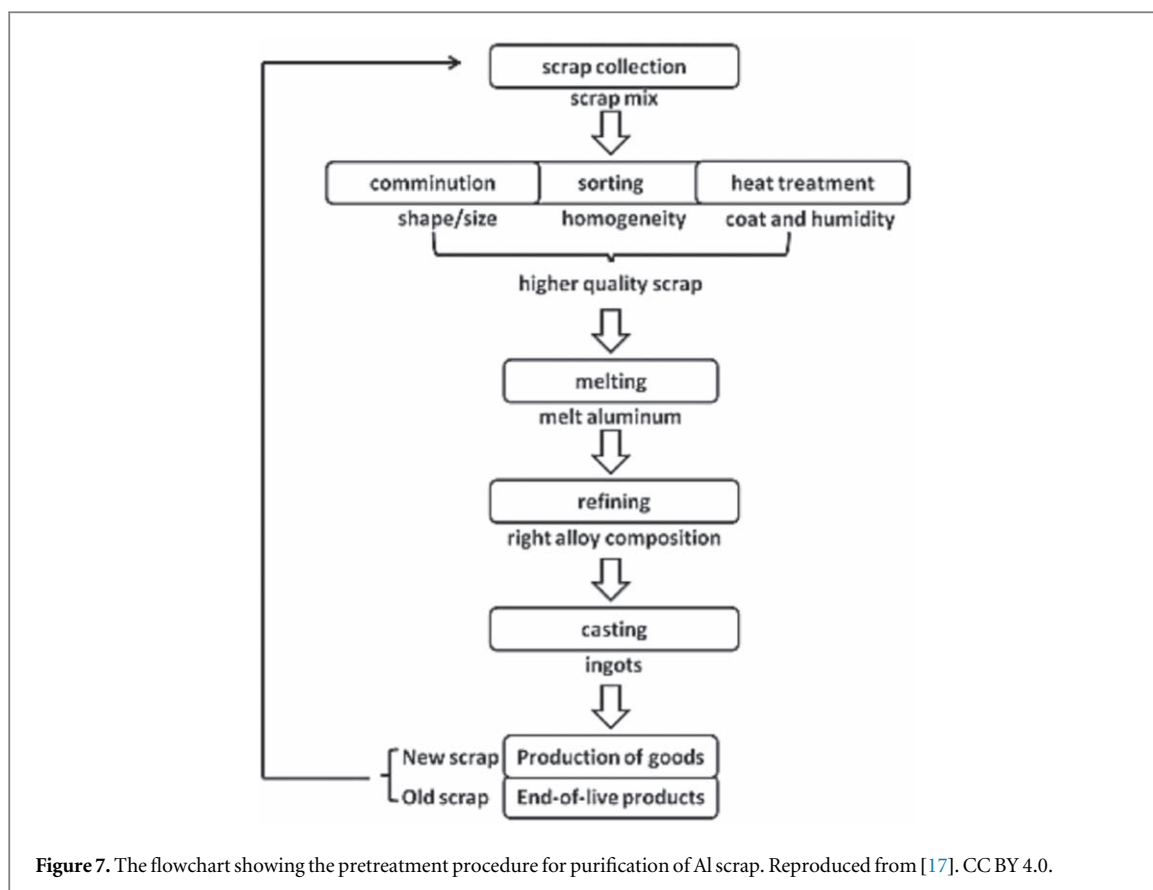
generally be utilized to remove any inclusions and impurities from Al to fabricate very highly refined Al (>99.97%). The fractional crystallization refining method can also be used to refine scraps of Al alloys such as the 5XXX series since it leads to high purity Al with low cost [47]. During the fractional crystallization refining method, in order to produce Al crystals, the surface of the melt is cooled in a very rapid manner so that the refined crystals settle to the bottom of the furnace and the residual melt accumulates from the impurities. Initially, the remaining liquid with a high density of impurities is discharged from a furnace called downgrade. Subsequently, the refined crystals in the bottom of the furnace are remelted and extracted, called an upgrade. This procedure can be performed in multiple manners in order to attain high purity metals.

In general, segregation methods are designed and fabricated based on the differences between the solubility of impurities in liquid and solid phases of base metal. This solubility difference can be evaluated by distribution coefficient ' k ' in equation (2), indicating the fraction between the concentration of a solid (C_S) and liquid (C_L) phases for particular impurity in the melt. These impurity concentrations can be attained through a lever rule utilization and a binary phase diagram [46]. Therefore, impurities having $k < 1$ magnitude can be segregated and accumulated in the liquid phase improving the Al purification. Rarely, $k = 1$ occurs during no segregations, and this impurity will be found equally in the matrix.

$$k = C_S / C_L \quad (2)$$

3.3.3. Suspension- and layer-based segregation techniques

Although numerous techniques are designed with the basis of segregation processes, they can be categorized into two principal methods of suspension- and layer-based segregation, as schematically shown in figures 4(b) and (c). In the course of crystallization, if the generated crystals begin to grow, attach preferably to a surface, and produce a layer, the technique is called layer-based. While for the case of freely development and suspension of generated crystals in the melt, the technique is entitled to suspension-based segregation [48]. The suspension-based method has a larger solid/liquid interface zone than the layer-based counterpart. So, during the suspension-based method, the fabricated crystal having greater pureness and slightly higher melting point than the periphery melt environment settles slowly down to the bottom of the chamber. On the other hand, the layer-based technique has another superiority in which no further processes are required to separate the crystals from the melt since the crystallization stage occurs on the previous crystallized layer, and the solid phase can grow continuously on the generated purified solid layer resulting from the accumulation of purified crystals. Lots of modern techniques are branched out from these two main methods. For the case of the suspension-based approach, new techniques were introduced, including the Alcoa process [47] and Corus crystallizer [48]. Also, zone refining [49–51], Pechiney [52], cooled finger [38, 53], and directional crystallization [54] methods are branches of layer-based technique.



4. Purification of Al alloys

4.1. Al scrap purification

In comparison to copper and steel, the recycled amount of Al scraps is lower just about 35% so it is of considerable importance to improve the techniques and procedures to increase this magnitude to upper than 50%. Recycling of Al scrap is very economic and it was estimated that only one ton of recycled Al scrap can lead to saving up 14,000 kWh energy, eight metric tons of bauxite, 6300 liters of oil, 7.6 m³ of landfill with an average total exhaust emission of about 350 kg CO₂ [17]. The main challenge in Al scrap recycling is the chemical composition since scraps are usually originated from different Al alloys. Therefore, the pretreatments such as sorting, heat treatment, and comminution are necessary, the pretreatment flowchart is shown in figure 7. After the second step including sorting, comminution, and heat treatment that leads to the production of higher quality Al scraps, the melting procedure is done. Then, the melted Al product is gone to be processed by refiners that are able to add necessary alloying elements and eliminate the undesired elements and inclusions, leading to the production of de-oxidized Al and foundry Al alloys [55]. Subsequently, the melt can be diluted or downgraded depending on the applications. In the case of downgrading, the resultant low-alloyed scrap is used in the fabrication of alloys having higher alloying contents. While, during dilution, the resultant molten scrap is diluted by utilization of primary Al or low-alloyed scrap in order to decline the concentration of alloying elements. Additionally, the resultant molten scrap can be upgraded by several methods including traditional methods such as fluxing, floatation, filtration, and modern techniques such as three-layer electrolysis, segregation methods, Hall-Heroult process, liquation, sedimentation and distillation technologies, etc.

4.2. Al-Mg alloys

The presence of impurities and inclusions in Al-Mg (5XXX) alloys can lead to deficient stability, poor quality, and low performance of the final product, so the purification process of Al alloys with high Mg content is of great importance. Also, the presence of any impurities in various Al-based alloys can affect the resultant properties, such as fatigue, monotonic and dynamic mechanical properties, etc, and even the performance of utilized processing methods, including severe plastic deformation, extrusion, corrosion, etc [56–62]. In this regard, Ma et al [63] studied the melt purification process and the effect of the various refiners on the defects generation and microstructure of 5083 alloys. They extracted an optimized rotary impeller degassing procedure to purify the 5083 alloys with the following conditions: a gas flow of 1.2–2.0 l s⁻¹, rotary speed of 250–400 r min⁻¹, and a

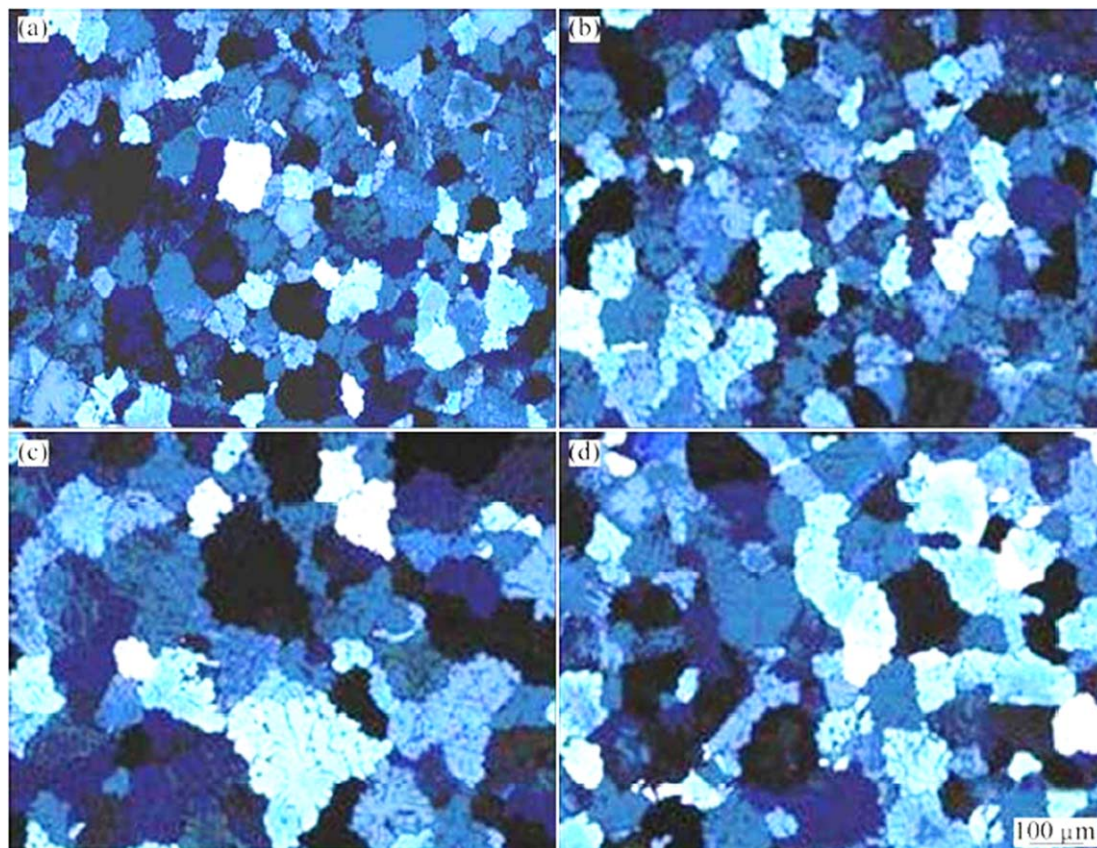
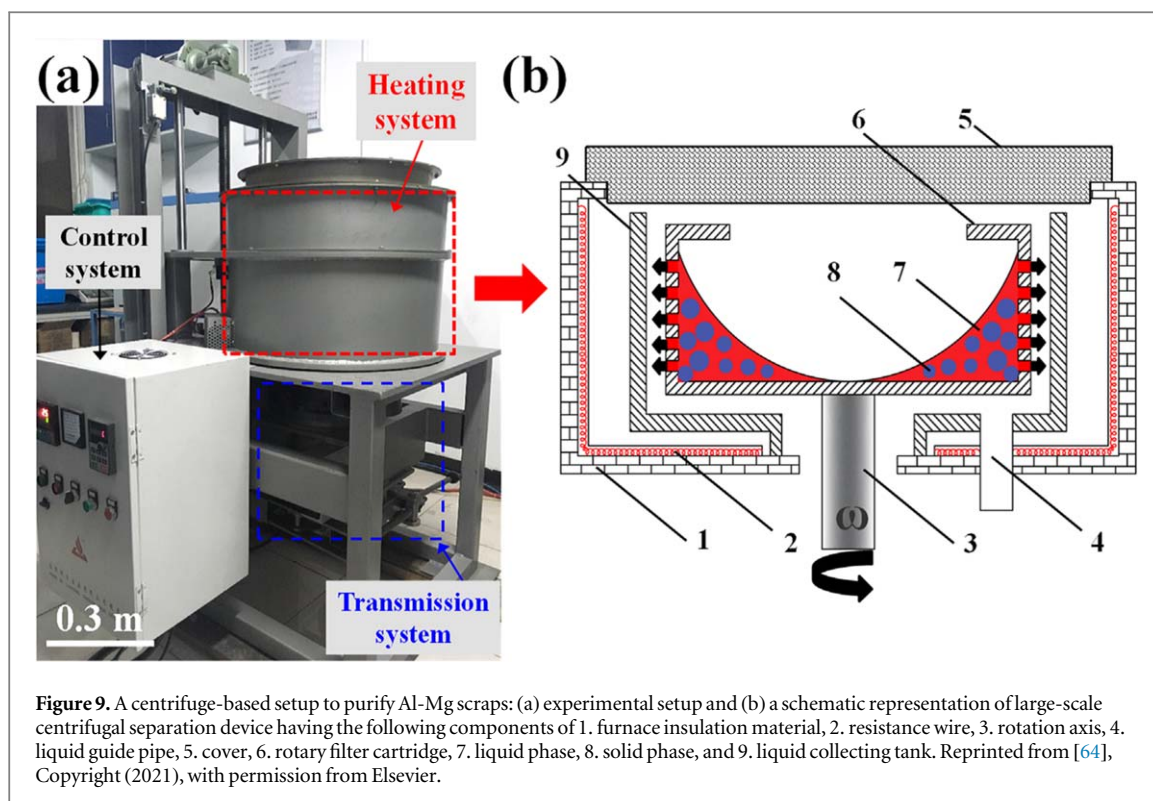


Figure 8. The effect of various refiners on the as-cast microstructure of 5083 alloy: (a) Al-5Ti wire, (b) Al-5Ti-B wire, (c) Al-3.2Ti-0.32C wire, and (d) Al-5Ti bulk. Reprinted from [63], Copyright (2014), with permission from Elsevier.

purification time of 10–15 min that considerably reduce the gas content in the solid alloy up to $2 \times 10^{-3} \text{ mL g}^{-1}$ or lower. Also, it was proved that the grain refiner led to the substantial refinement of the cast microstructure of 5083 alloys, and Al-5Ti-1B wire manifests the best refining performance. In this regard, figure 8 shows the effect of various refiners on the microstructure of 5083 alloy [63].

In another study, Meng *et al* [64] investigated a novel purification process based on supergravity technology to refine scrap Al-Mg alloys. As known, refining Al-Mg alloys is of technical importance, and they are among the most commonly recycled nonferrous metals. However, unfortunately, the presence of numerous impurities in their scrap is a big challenge [65]. These scraps usually have a variety of impurities such as unfavorable metallic elements (Mn, Cu, Zn, Fe, Ni, Si, etc) and various non-metallic impurities (carbides, oxides, nitrides, etc) [66, 67]. In this regard, Meng *et al* [64] proposed a novel liquation-based supergravity method for simultaneous recycling of Al and Mg scraps. Firstly, Al scarp and $\sim 30 \text{ wt\%}$ Mg scraps are melted together to fabricate the eutectic scrap from Al-Mg alloy. Subsequently, the precipitated intermetallic species are crystallized as primary phases containing impurity elements (mainly Al_3Fe and $\text{b-Al}_5\text{FeSi}$) and separated at the near eutectic temperature. Supergravity technology which was introduced by Ramshaw *et al* in 1981 [68] is an influential physical method to enhance the separation and mass transfer of various phases. Supergravity technology utilizes centrifugal force in order to improve the phase separation performance and micro-mixing through supergravity field application in which the variation between densities or melting points of solid particles and liquid melt leads to gentle distribution and separation of the particles, according to the centrifuging direction [69, 70]. It was confirmed that the supergravity technique can considerably be effective in refining and extracting valuable components from metallurgical wastes [71, 72] and purification of metals [73, 74]. Respectively, Meng *et al* [64] proved the effectiveness of the supergravity technique in Al-Mg scrap purification. They concluded that impurities can be successfully separated from the melted scrap alloy through supergravity filtration and using a centrifuge-based setup, as shown in figure 9. They illustrated that at the conditions of $T = 500 \text{ }^\circ\text{C}$, gravity coefficient of $G = 600$, and $t = 1 \text{ min}$, the Al and Mg recoveries reach 91.6% and 90.1%, respectively. It should be noted that the removal rates of all Mn, Fe, Si, Ni, Cu, Zn, and Cr elements were higher than 50% except for the cases of Cu and Zn. They also demonstrated the applicability of the supergravity technique for industrial applications by manufacturing a large-scale centrifugal separation, as shown in figure 9.

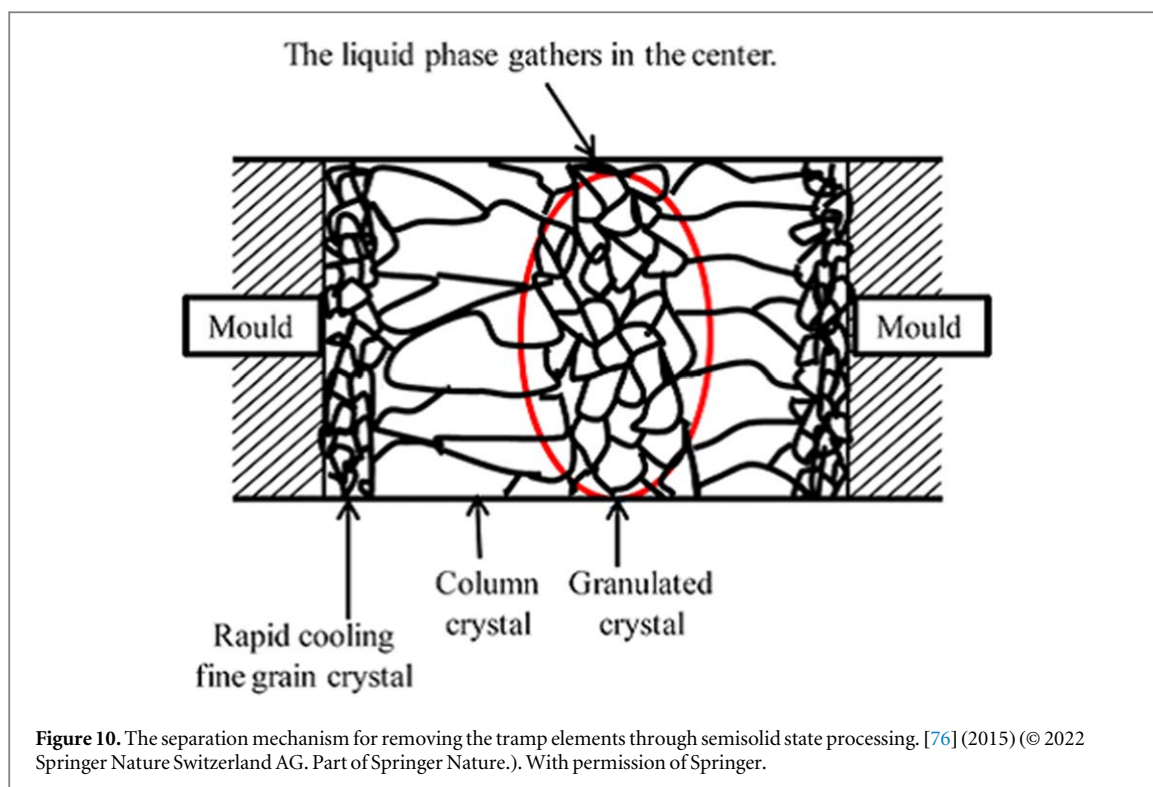


In addition to experimental studies, Buzatui *et al* [75] proposed an optimization modeling through a program of active experimentation (PO_2), according to primary parameters of the filtering process of Al–Mg–Mn alloys (5XXX series). The considered parameters are the initial concentration of impurities (z_3), melt flow rate (z_2), filtration efficiency (η), and temperature (z_1). The modeling results indicated that filtration efficiency (η) improves by reduction of flow rate and increment of the initial impurity concentration, while this efficiency reduces with temperature increment which satisfactorily is in agreement with the experimental observations [75]. Other methods can also be used to refine Al alloys, for instance, Cho *et al* [76] proposed a separation technology to remove tramp elements from Al scraps through semisolid processing. In this regard, backward extrusion in a semisolid state is utilized to produce high purity aluminum, in which partial solidification is used to condense tramp elements in the liquid phase on the grain boundary. Figure 10 schematically illustrates the separation mechanism through semisolid state processing. Firstly, the molten metal begins to solidify from the wall of the enclosure and then gently moves forward to the interior zones. After reaching the semisolid state, the accumulated liquid phase in the central section contains high magnitudes of tramp elements which were extracted by backward extrusion. Several repetitions of the backward extrusion process led to further purifications.

For the case of Al-Mg alloys, the presence of extra Fe magnitudes in the melt can lead to the reduction of the elongation (ductility) and tensile strength while the presence of 0.6 wt% Fe in Al-10Mg-0.5Si alloy can also enhance these properties. Also, increment of 0.2 to 0.8 wt% Fe content in Al-3 wt% Mg and 6 wt% Mg alloys slightly enhances the tensile strength [77]. The improvement of tensile strength with Fe addition also was reported in Al-Mg-Zn and Al-0.7 Mg-1Si wrought alloys [78]. The existence of Fe can even lead to superior corrosion performance since in Al-Mg alloys, Mn converts Al_3Fe into $(Fe, Mn)Al_6$, improving the corrosion resistance of the alloy [79]. This information clearly shows the importance of controlling the Fe content in Al-Mg alloys. Removal of extra iron content can be achieved by several technologies such as precipitation of Fe-rich phases, gravity-based separation methods, filtration and fluxing techniques, centrifugal and electromagnetic separation procedures, electrolysis, fractional solidification, electroslag refining, etc.

4.3. Al-Si alloys

Al-Si alloys have numerous applications in various industries and modern technologies including electronic devices, aerospace, and automobile industry mainly due to their favorable castability, high strength-to-weight ratio, substantial wear and corrosion resistance along with proper tensile, impact, and fatigue characteristics that can be attained through a suitable heat treatment [80]. These advantageous behaviors of Al-Si alloys were attributed to the presence of hard silicon particles arising both from eutectic silicon and primary silicon. The Si usually is added to Al in order to enhance castability, fluidity, and hot tear resistance [81]. Despite the usefulness

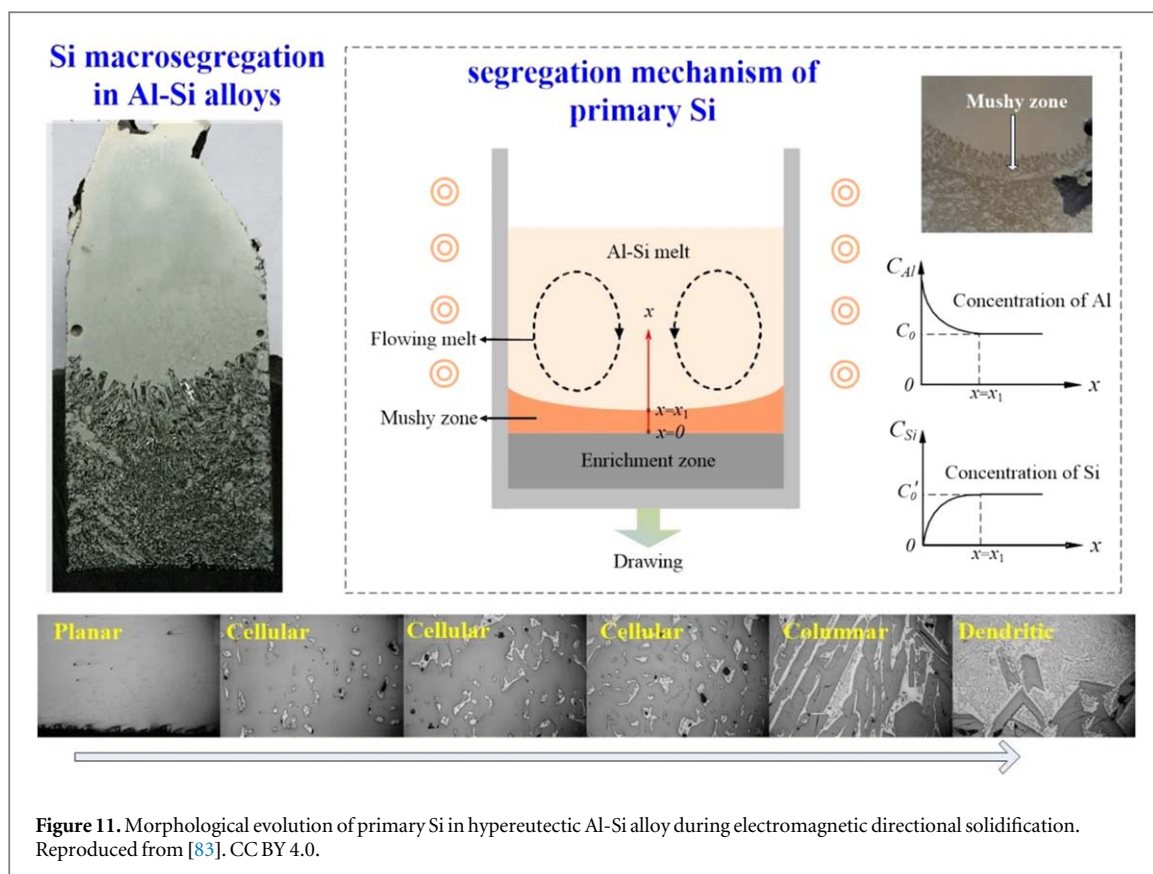


of Si in Al melt, its excess amounts and undesirable Si morphology can lead to serious issues and adversely affect the microstructure and overall properties. To eliminate the high primary silicon content, the melt must be refined through various methods such as electromagnetic directional solidification procedure which is mainly dependent on the growth rate of the primary Si phase, cooling, and stirring conditions, and the solidification rate of the Al-Si melts [82]. In this regard, Jiang *et al* [83] studied the Si segregation behavior in hypereutectic Al-Si alloys during the electromagnetic directional solidification (EMDS) method. They numerically and experimentally proved that extreme primary Si macrosegregation is mainly caused by fluid flow and temperature distribution with the morphological evolution order of planar \rightarrow cellular \rightarrow columnar \rightarrow dendritic, figure 11 briefly illustrates the morphological evolution in hypereutectic Al-Si alloy. In another study, Ren *et al* [84] also used electromagnetic directional solidification to purify Al-Si alloy, showing its effectiveness in melt degassing.

5. Conclusion and future trends

Nowadays, aluminum refining and purification technologies are becoming more and more critical issues due to the increment of Al scraps and the need to obtain higher quality aluminum materials with enhanced properties and specific applications. In this regard, the reasons behind the production of low-quality melts, the verity of impurities and inclusions, techniques and procedures, and purification mechanisms must be comprehensively studied and investigated. The presented paper aims to give a brief overview of this subject with special attention to techniques.

Al melt can have several problems leading to the generation of various inclusions (carbides, borides, oxides, nitrides, fluorides, iron oxides, chlorides, molten salts, etc), pinholes, dross, coarse grain, severe surface crinkles, etc Chiefly hydrogen and oxide inclusions along with solid particles, tramp and extra elements, pollute the aluminum melt. In order to reach a high-quality Al melt, these issues should be solved as much as possible and according to their characteristics including density, wettability, etc, the suitable refining process can be designed and chosen. The main purification processes are fluxing, floatation, and filtration, but there are also many modern techniques usually aimed to produce highly purified Al melt such as sedimentation and distillation technologies, liquation processes, Hoopes process, three-layer electrolysis, suspension- and layer-based segregation methods, etc. Despite great achievements in Al production methods and reaching ultra-pure aluminum alloys, unfortunately, the techniques are still very expensive and often complex, so careful study of the subject can pave the way for simpler and more cost-effective techniques. Accordingly, this article is dedicated and written with a view to this influential issue. It is hoped that by carefully studying the subject, more cost-effective and simpler methods will be gradually formed and developed.



Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Competing interests

The authors declare no conflict of interest.

Data availability

All data generated or analyzed during this study are included in this published article.

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