Enrichment of Scandium in Al-Sc Alloys by Vacuum Distillation

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Abstract

Scandium's (Sc) interaction with Aluminium (Al) makes it valuable as an alloying element, not only as microalloying element or in additive manufacturing (*Scalmalloy*®) but particularly for electronics applications such as the current 5G mobile standard, where Aluminium-Scandium-Nitride (AlScN) is utilised. Previous research at IME has identified the aluminothermic reduction of Scandium fluoride (ScF_3) as a promising approach to produce Sc containing Al-alloys in situ, thereby avoiding the classical approach of alloying pure metals. This study explores the potential of vacuum distillation for producing high Sc contents, targeting alloys above 20 wt.% Sc. [1–4]

For vacuum distillation, the strong interaction between Al and Sc, combined with the relatively low initial vapor pressure of Aluminium, poses challenges. However, FactSage™ simulations suggest that significant separation – and thus Sc enrichment – can be achieved under reduced pressures and temperatures exceeding 1350 °C. Especially for low Sc-contents the separation coefficient (β) lies in the magnitude of 10^4. The theoretical enrichment-limit is at the azeotropic point, whereas under the condition of a separation coefficient with great deviation from unity, a concentration of approximately 40 % Sc should be achievable. Practical trials in a vacuum induction furnace have demonstrated successful distillation of almost pure Al in laboratory scale and subsequently enrichment of Sc in the remaining alloy. Yet, control over the process proves difficult.

In conclusion, distillation could be viable option for the enrichment of Sc in Al-Sc alloys. Further development should include an optimisation of the experimental setup and parameters to refine control over the distillation process before scaling up operations. The theoretical enrichment limits of these methods differ; therefore, combining both could yield optimal results.

Introduction

The enrichment of aluminum-scandium (Al-Sc) alloys via vacuum distillation exploits the distinct thermodynamic interactions between aluminum and scandium in both solid and liquid states. In the solid state, Al and Sc form several intermetallic compounds, notably Al_3Sc, Al_2Sc, AlSc, and $AlSc_2$ as can be seen in figure 1. These intermetallic phases exhibit stoichiometric compositions and high thermodynamic stability, evidenced by their melting points significantly exceeding that of pure aluminum. The resultant steep liquidus lines in the Al-Sc phase diagram, particularly at low scandium concentrations, reflect strong atomic interactions and limited solubility of scandium in aluminum. [2], [3], [5–9]

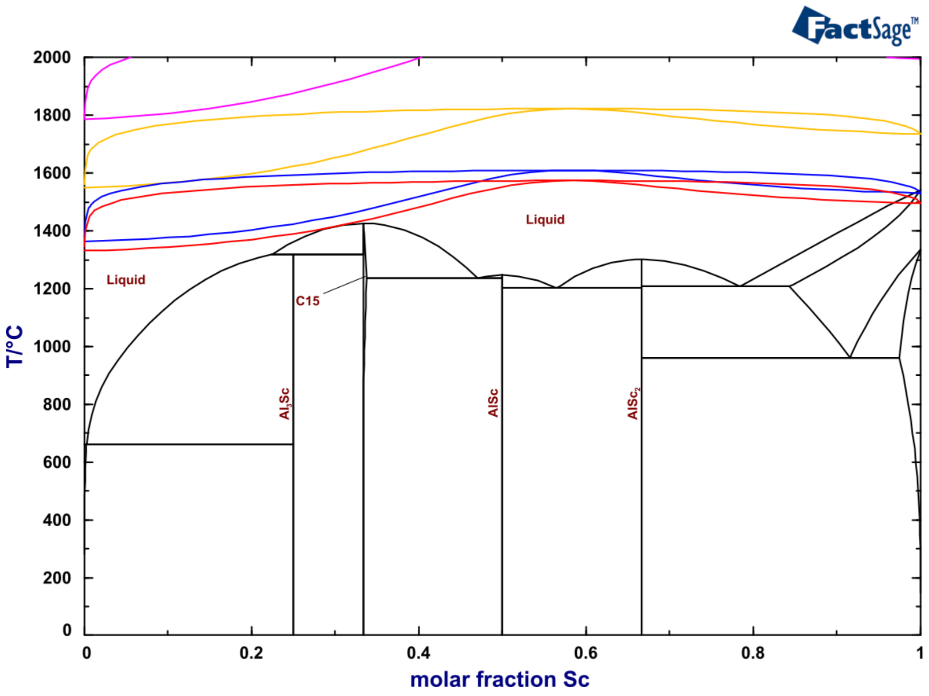

Figure 1: Phase diagram Al-Sc at different pressures. The phase regions with a gaseous component (G and G + L) are represented by colored lines. According to a decreasing pressure in decreasing order with temperature: $p = 10^{-2}$ atm (magenta), $p = 10^{-3}$ atm (yellow), $p = 10^{-4}$ atm (blue), p = 6.5 - 10-5 atm (red). Created with FactSage™, [10].

In the liquid state, the Al-Sc melt demonstrates significant short-range ordering due to preferential associations between unlike atoms. This behavior strongly deviates from ideal solution theory and is characterised by a negative mixing enthalpy, indicating exothermic interactions and the formation of Al-Sc atomic clusters. The Modified Quasichemical Model in the Pair Approximation (MQMPA) effectively describes these thermodynamic properties by considering the formation of Al-Sc pairs within the melt. [5], [8], [9], [11]

Phase diagrams of the Al-Sc system at reduced pressures illustrate the influence of pressure on phase equilibria, particularly affecting the liquidus and solidus lines. Under vacuum conditions (e.g., in figure 2 on the right), the boiling and dew points of the components are lowered, and the vapor-liquid equilibrium (VLE) conditions shift favorably for distillation. Specifically, the boiling point of aluminum decreases, allowing selective vaporisation at temperatures between approximately 1360 °C and 1600 °C, depending on the scandium concentration.

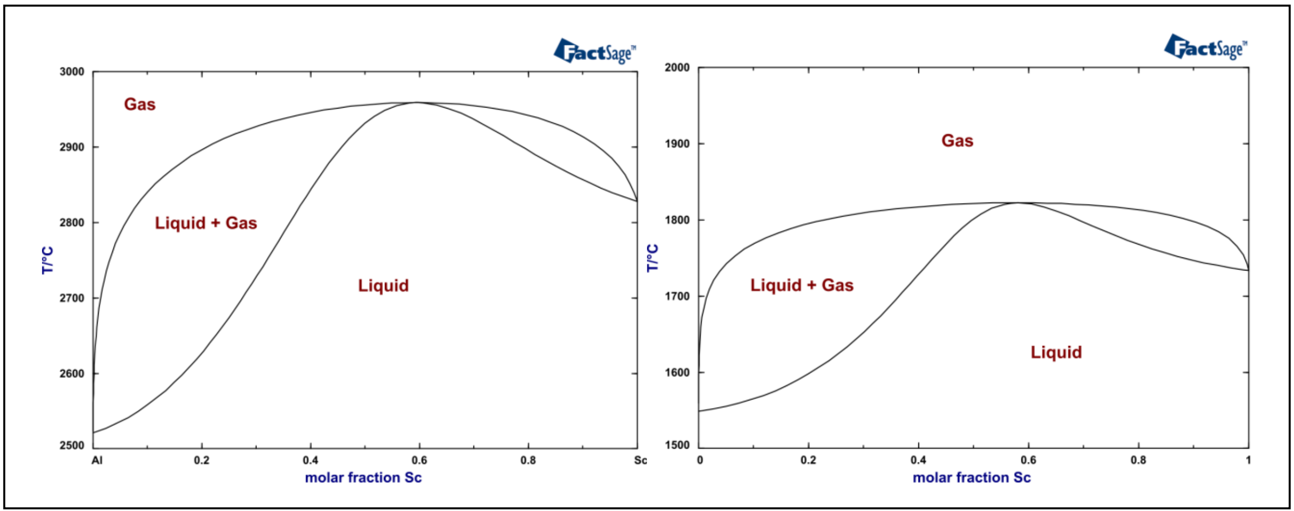

Figure 2: Phase diagrams Al-Sc at different pressures. Left: 1 atm; right: 10^{-3} atm. Created with FactSage™, [10].

The combined analysis of the VLE and the separation coefficient β (equation 1) provides critical insights into the distillation process. Due to the strong deviations from ideality and intermolecular interactions, the Al-Sc system exhibits azeotropic behavior with a maximum boiling point at approximately 57.8 % Sc. At the azeotropic composition, β approaches unity (β = 1), signifying that the vapor and liquid phases have identical compositions, rendering simple distillation ineffective beyond this point.

$$\beta_i = \frac{\gamma_i}{\gamma_j} \cdot \frac{p_i^0}{p_j^0} = \frac{y_i}{y_j} \cdot \frac{x_j}{x_i} \tag{1}$$

For most scandium concentrations below the azeotrope, the separation coefficient is significantly greater than one (see figure 3), indicating that aluminum is considerably more volatile than scandium under these conditions. For pressure of 10^{-4} atm, at a scandium concentration of 0.001% and a temperature of approximately 1362 °C, the separation coefficient is $\beta \approx 68{,}289$. At a higher concentration of 13 % scandium, β still remains favorable at 8700.5 (boiling point 1381.7 °C). Only when the concentration is more than 40 % β lies in the lower range of magnitude 10^2 leading to scandium losses.

Thus, a working vacuum between 10^{-3} atm and 6.5×10^{-5} atm is ideal. Operating at pressures higher than 10^{-3} atm results in worse separation and increased boiling temperatures whereas a pressure lower than 6.5×10^{-5} atm is inadvisable due to the formation of solid intermetallic phases that impede mass transfer which can be verified on the basis of Figure 1, red lines.

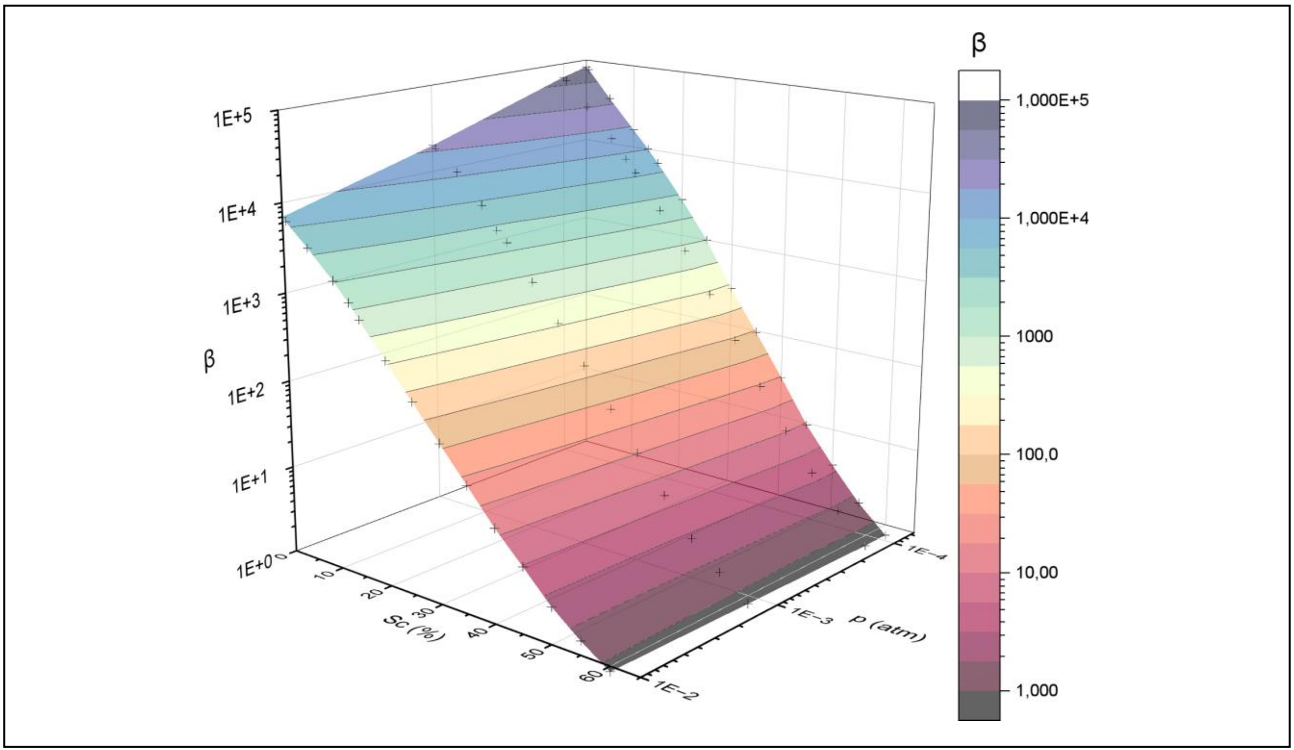

Figure 3: Progression of the separation coefficient with the scandium content and the pressure. Crosses: concrete values of the equilibrium simulation of the range 0.001 – 60 % Sc in steps of 5 %. Additional information for 0.6 % and 13 % Sc. Calculated with the function module in FactSage™, [10].

Material and Methods

The following sections contain information on the AlSc-alloy and setup used for vacuum distillation. Lastly, the experimental procedure will be described.

Material

A 20 wt.% aluminium-scandium (Al-Sc) alloy was used as the primary material for the enrichment studies via vacuum distillation. The alloy was initially provided as a large rectangular block weighing approximately 20 kg, with dimensions of 35 cm × 35 cm × 9 cm. The block exhibited multiple cracks and pores and possessed a silvery-metallic appearance akin to pure aluminium.

Due to the impracticality of utilising such a large piece for small-scale experiments, the alloy was subdivided into smaller segments. The block was sectioned using a carbide-tipped saw blade into pieces measuring roughly 2.5 cm × 2.5 cm, while maintaining the original thickness. The resulting pieces weighed in the high double-digit to low triple-digit gram range. To ensure consistency in experimental conditions, pieces weighing approximately 100 g were selected for each trial. Any significant weight deviations were corrected by further trimming the pieces using a metal saw or by chipping with a hammer and chisel. The prepared alloy pieces were then cleaned with ethanol and dried using compressed air to remove any residual particles and contaminants.

For compositional analysis, both X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectrometry (ICP-OES) were employed. The internal XRF analysis indicated a scandium content of 18.571 wt.%, while the external ICP-OES analysis showed a scandium content of 19.73 wt.%, both slightly below the nominal 20 wt.% target. Additionally, energy-dispersive X-ray spectroscopy (EDX) was performed on selected areas of the alloy, revealing an average scandium content of 21.2 wt.% with a relative uncertainty of ±0.3 wt.%. Minor discrepancies in scandium content were attributed to analytical limitations and sample heterogeneity. Gas content analysis, conducted using an ELTRA Elementrac ONH-p analyser, revealed minimal levels of dissolved oxygen, nitrogen, and hydrogen, indicating low levels of interstitial impurities.

Microstructural characterisation was performed using scanning electron microscopy (SEM) coupled with EDX. SEM images revealed two primary phases: a light grey intermetallic Al_3Sc phase present in cubic or dendritic forms up to 400 µm in size, and a medium grey α-aluminium matrix. Occasionally, a third dark grey phase was observed, appearing as circular to elliptical regions within the matrix. The samples displayed significant porosity, with pores often located between the Al_3Sc-grains. EDX analysis confirmed the presence of aluminium and scandium as the main constituents but also detected minor amounts of impurities such as silicon, tantalum, carbon, oxygen, and nitrogen. Notably, tantalum-rich particles appeared as bright spots in the SEM images, likely originating from the alloy production process.

Experimental Setup

The vacuum distillation experiments were conducted using a custom-designed setup to facilitate the evaporation of aluminium from the Al-Sc alloy under reduced pressure. The apparatus shown in figure 4 consists of two primary components: the evaporation unit and the condensation unit.

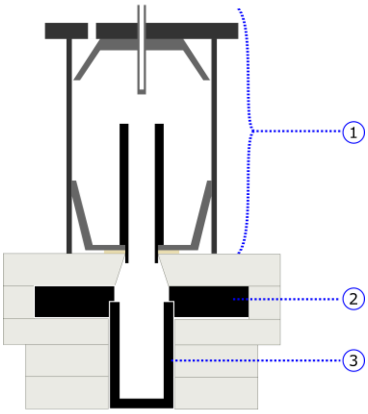

Figure 4: Schematic sketch of the experimental setup with evaporation and condensation unit. (1) Condensation unit (grey), (2) graphite lid (black), (3) graphite crucible (black). Beige symbolises the surrounding refractory mat, yellow symbolises refractory paper.

Evaporation Unit

The evaporation unit comprised a high-purity graphite crucible serving as the container for the alloy. Graphite was selected due to its chemical stability with Al-Sc melts at high temperatures and its compatibility with induction heating. The crucible had an outer diameter of 6 cm, a wall thickness of 1 cm, and a height of 10 cm. This size was chosen to limit the amount of alloy required per experiment and to minimise the risk of uncontrolled aluminium vapor release. The crucible was positioned such that only its lower 2 cm were within the induction coil's effective zone to reduce excessive heating of the crucible lid.

For temperature measurements, two Type B thermocouples were inserted into 20 mm-deep holes drilled into the bottom of the crucible. The crucible, along with its graphite lid, was wrapped in layers of refractory insulation material (Isofrax®-1400) to minimise heat losses and protect the furnace components. This insulation also served to shield the induction coil from thermal radiation.

Condensation Unit

Positioned above the evaporation unit, the condensation unit was designed to collect the evaporated aluminium. It consisted of a steel cylinder topped with a steel lid, from which a condensation bell was suspended. A graphite riser tube connected the crucible to the condensation chamber, directing the aluminium vapour towards the cooler surfaces where it could condense. An aluminium collection vessel was placed beneath the condensation bell to gather the condensed metal. The entire assembly was carefully aligned and insulated to the bottom with refractory materials to maintain temperature gradients favourable for condensation and to ensure structural stability within the vacuum environment.

Vacuum Induction Furnace and Heating

The experiments were carried out in a vacuum induction furnace (VSG 025) equipped with a water-cooled induction coil operating at a nominal frequency of 4 kHz and a maximum power output of 20 kW. The furnace chamber was evacuated using a combination of a Leybold DK 90 rotary vane pump and an additional Roots blower, achieving pressures down to approximately 5×10^{-2} mbar. The reduced pressure was crucial for lowering the evaporation temperature of aluminium and facilitating its separation from scandium.

Experimental Procedure

Before each experiment, the furnace chamber and components were meticulously cleaned to prevent contamination, and all parts of the setup that could experience weight changes were weighed. Approximately 100 g of the prepared 20 wt.% Al-Sc alloy were loaded into the graphite crucible. The crucible and its lid were assembled, wrapped with insulation, and placed on refractory bricks within the induction coil to achieve the desired positioning.

The furnace was then evacuated to the target pressure. Temperature and pressure data were recorded continuously using appropriate sensors and data acquisition systems. Induction heating was initiated at a power setting of 10 kW or 14 kW to provide controlled heating rates. The change in power was made after a new, thicker graphite lid had to be used. The alloy was heated until the aluminium began to evaporate, typically indicated by a plateau or inflection in the temperature curve and a corresponding rise in pressure due to aluminium vapour. Throughout the experiment, only intermittent power interruptions allowed for accurate temperature readings, as the thermocouples were affected by electromagnetic interference from the induction coil.

The power was supplied until the end-point criteria were met. Specifically, the experiment was terminated when the temperature increase over a five-minute interval fell below 3 K, indicating that the system had reached a steady state of evaporation. In later experiments as parameter variation, a threshold of less than 10 K temperature increase over five minutes was used as the criterion for process completion which proved to show no significant difference.

After the distillation process, the furnace was allowed to cool under vacuum to prevent oxidation of the residual scandium-rich alloy and the condensed aluminium. Upon reaching ambient temperature, the system was vented with inert gas, and the condensed aluminium and the residual alloy were retrieved for analysis. Post-experiment weighing of the components allowed for mass balance calculations to assess the effectiveness of the distillation process.

Results

The vacuum distillation experiments of Aluminium-Scandium (Al-Sc) alloys yielded several distinct products located at various points within the experimental setup. These allowed to calculate the mass balance and to conduct different analysis methods.

Products

Distillate Collection and Characteristics

In all successful experiments, notable amounts of distillate were found adhering to the lower end of the riser pipe. This distillate ranged from fine powders to larger, often spherical particles shown in figure 5, with masses varying between 2.2 g and 20.2 g. The coloration of these particles varied from light to dark grey, indicating differing surface characteristics or impurity levels. The size and mass of the distillate correlated with the end temperature of the experiment and the extent of evaporation achieved. Higher temperatures generally produced larger masses of distillate due to increased evaporation rates. The distillate was mechanically removed from the riser pipe for subsequent analysis.

The unexpected location of the distillate – accumulating at the riser pipe rather than in the designated condenser – suggests that the vapor jet could not sustain itself over the intended distance, likely due to insufficient vacuum. This observation points to potential improvements in the setup, such as adjusting the riser pipe length or enhancing the vacuum conditions to facilitate more effective distillation.

Figure 5: Photos of the distillate found at the bottom of the riser tube from selected experiments. Riser tube diameter is 2.5 cm.

Residual Alloy in the Crucible

The majority of the non-evaporated metal remained in the crucible, displaying notable characteristics. The residual metal exhibited a predominantly golden color (figure 6, right) with surface patterns consisting of black hexagonal formations (figure 6, left) up to 5 mm in size. These patterns indicate potential microstructural changes or phase formations resulting from the distillation process.

The golden metallic layer extended notably up the walls of the crucible, suggesting significant interaction between the molten alloy and the crucible material. Beneath this surface layer, the metal retained a shiny, metallic appearance, consistent with unaltered alloy regions. The variation in surface coloration and texture highlights the effects of temperature gradients and material interactions within the crucible during distillation.

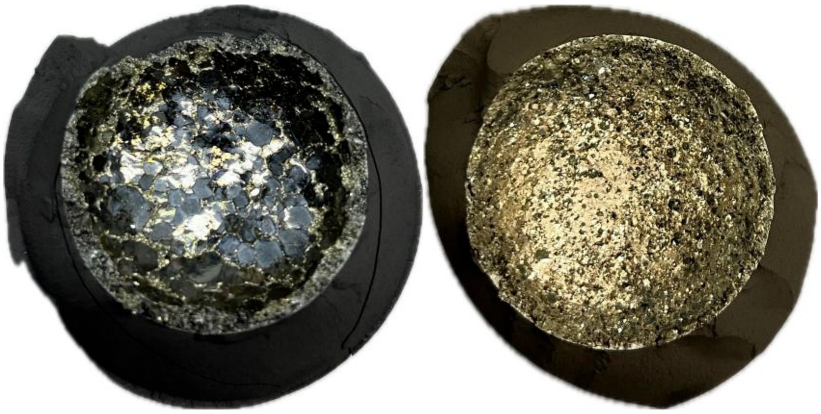

Figure 6: Metal phases of the distillations with AlSc20 with residual surrounding graphite. Metal diameter is 4 cm.

Condition of the Condensation Unit and Surroundings

Minor deposits and small metallic particles were observed within the condensation unit, particularly on the bell (Glocke) and at the vapor jet temperature measuring point. These deposits exhibited a spectrum of colors but were generally too thin or sparse for effective sampling and analysis. A consistent white deposit was also noted on the exterior surfaces of the crucible and lid, especially in experiments with higher distillate masses. This deposit is likely composed of alumina (Al_2O_3) or scandium oxide (Sc_2O_3), formed from oxidation of vaporised metal species upon contact with traces of oxygen or cooler surfaces.

Material characterisation

The analytical characterization focused on the AlSc20 experiments to determine the efficiency of scandium enrichment via vacuum distillation and to understand the compositional changes in the residual alloy and distillate.

EDX analyses were performed on samples extracted from both the upper and lower sections of the residual alloy within the crucible. The results indicated an enrichment of scandium in the residual alloy where a correlation was observed between the end temperature of the experiments and the scandium content in the residual alloy. Higher end temperatures facilitated greater evaporation of aluminium, thus enhancing scandium enrichment, visualized by figure 7, left. This relationship is further supported by figure 7, right, plotting the Sc-content against the distilled mass fraction hinting that the evaporated material must be indeed aluminium.

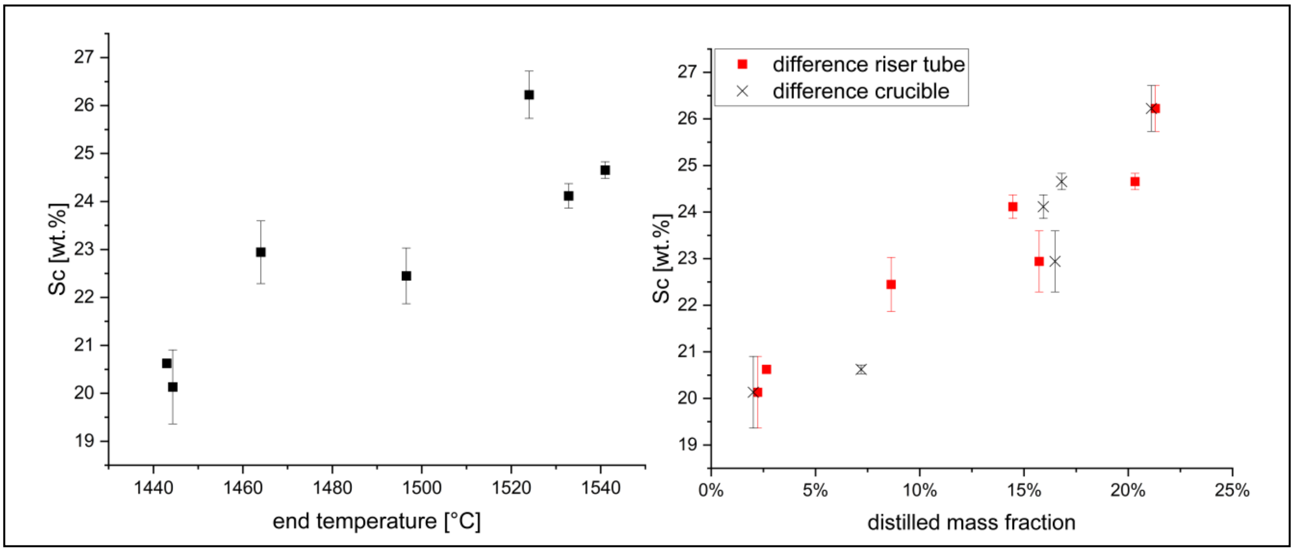

Figure 7: Plot of the Sc content according to EDX against the final temperature (left) and the distilled mass fraction (right).

ICP-OES analyses provided quantitative confirmation of scandium enrichment in the residual alloy and the effectiveness of the vacuum distillation process. In experiment AlSc20_01, the residual alloy obtained from the crucible showed a scandium content of 23.9 wt.%, which is an increase from the initial alloy composition of 20 wt.% Sc. In contrast, the distillate collected from this experiment contained less than 0.05 wt.% Sc, indicating that scandium did not significantly vaporise under the experimental conditions and was effectively retained in the residual alloy.

Additionally, the ICP-OES analysis revealed the presence of carbon in the residual alloy, with a measured content of approximately 0.67 wt.%. The distillate also showed a carbon content of about 0.70 wt.%. The source of this carbon is likely the graphite crucible material, suggesting that some carbon dissolution or carbide formation occurred during the high-temperature distillation process.

Conclusion

Vacuum distillation has proven to be an effective method for enriching scandium in aluminum-scandium alloys, achieving scandium concentrations beyond conventional approaches. Theoretical analyses indicated that optimal separation and enrichment occur at reduced pressures between 10^{-3} atm and 6.5×10^{-5} atm and temperatures exceeding 1350 °C. In practice, the experiments were conducted at actual minimal pressures around 2×10^{-4} atm, in the optimal range. This discrepancy may have contributed to challenges such as the accumulation of distillate in unintended parts of the setup and difficulties in maintaining a stable vapor jet, suggesting that operating within the optimal pressure range is crucial for efficiency. Scandium enrichment was observed in the residual alloy, with scandium content increasing from the initial 20 wt.% to up to a maximum of 26.2 wt.%. Distilled masses of aluminum ranged from 2.2 g to 20.2 g, correlating with higher end temperatures and longer distillation times. Control over the distillation process was further challenged by technical factors, such as the coupling of thermocouples to the induction system. Future work should focus on optimising the experimental setup, including maintaining pressures within the optimal range, enhancing vacuum, and precisely controlling temperature gradients to achieve consistent and scalable results. Combining vacuum distillation with other enrichment techniques may further increase scandium concentration, opening new possibilities for advanced applications in electronics and materials engineering.

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