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ORIGINAL PAPER

Solidification and Heat-treatment Conditions Affecting the Tensile Properties and Fracture Feature of an Automotive AlSiMg Alloy

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Abstract
In this work, a study on the interrelationship between the solidification and heat-treatment processes parameters with tensile properties and fracture feature was performed with an automotive AlSiMg alloy. For that, samples of the horizontally solidified Al7Si0.3 Mg (wt.%) alloy were subjected to the T6-heat treatment, and tensile tests were performed on both investigated samples, under conditions established from the literature. The solidification conditions such as growth and cooling rates and the secondary dendritic spacing (V_L and T_R and λ_2, respectively) and their effects on the ultimate tensile strength (σ_{UTS}) and elongation (E%) were evaluated. Higher σ_{UTS} values were observed in the heat-treated samples and finer microstructures resulted in a better E% performance. Analysis by SEM/EDS fractography on both samples showed a mix between brittle and ductile fractures, constituted by cleavage facets, secondary cracks, facets covered with micro-voids, tear ridges and dimples. This allowed the deduction of the occurrence of a transition of a transition from ductile to brittle fracture along the solidified ingot as well as the predominance of brittle fracture in the heat-treated samples.

Keywords Transient solidification · Heat treatment · Microstrutcure · Fracture · Tensile properties

1 Introduction

The use of metallic alloys in the various sectors of engineering and industry is based mainly on mechanical properties, which can be very well programmed. The products by these alloys are able to withstand the loads they are subjected to when in service [1]. It is known that these properties depend largely on the structure which is closely related to chemical compositions and processing conditions, among which heat treatments (HT) are some of the most used [1]. Heat treatments of metallic alloys are accredited as one of the most important methods of materials manufacturing and developing of new technologies throughout human history. Through controlled heating and cooling operations, the materials properties undergo considerable variations as a result of changes in macrostrutcture and microstructure characteristics [1].

Grain refinement, dissolution of secondary phases, distribution and morphology changes of phases have been studied over the decades and are linked to the enhancement of mechanical properties in heat treated aluminum alloys [2–6]. Shivkumar, Apelian and collaborators [5, 7, 8] studying Al-Si-Mg alloys observed that HT changes silicon morphology and that the kinetics of spheroidization are affected by the as-cast microstructure. Similarly, addition of sodium, strontium, antimony and bismuth has been investigated as a method of chemical modification of Si-crystal, and studies indicate that machinability, tribology and mechanical properties are significantly affected [9–16].

Recent studies reported that the microstructure scale of directionally solidified as-cast ingots has effects on HT parameters, and plays a crucial role in the extent of morphological transition of the undissolved phases from acicular

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to spheroidized in aluminum alloys [17, 18]. In a previous work published by the authors regarding AlSiMg automotive alloy subjected to T6-heat treatment, it was stated that spheroidized-like eutectic Si particles and Chinese script iron-rich phase were favored after HT in refined microstructure scale conditions, resulting in different behaviors between as-cast and HT samples subjected to responses to microhardness and wear resistance [6, 19].

Microstructural scale, phases morphology and HT are conducive to changes the properties of automotive AlSiMg alloy separately or in conjunction with each other, to develop desired and optimum properties in the casting [20–24]. In this regard, a series of works were recently developed with the automotive casting Al7Si0.3 Mg (wt.%) alloy, which in a first stage aimed to analyze the effects of solidification conditions on the microstructure and mechanical properties [21–23]. In this step, the influence of the thermal solidification parameters, such as growth and cooling rates on the length of the dendritic microstructure as well as on the size, distribution and morphology of the Mg_2Si/Fe intermetallic compounds (IMCs) and, in turn, on the hardness and tensile properties were investigated. In a second stage [6, 19], T6-heat treatment under different conditions was applied to the as-cast samples resulting from the work of References [21–23].

Studies [20, 21] obtained as-cast ingots of the Al7Si0.3 Mg alloy (wt.%), and typical solidification microstructures observed by both works consisting of a dendritic network with Al-rich primary and secondary phases (Al_α, Si crystals and IMCs). Finer dendritic microstructures were observed for higher cooling rates values. From the as-cast samples obtained by Lima et al. [21], Souza et al. [22] and Barbosa et al. [23], analyses on the influence of the solidification parameters on the microhardness (HV) of the aforementioned alloy were performed. In addition, Souza et al. [22] evaluated the Mg and Cu addition in the Al-7Si alloy (wt.%), depicted higher HV values for the ternary Al-7Si-(0.3 Mg;3Cu) alloys (wt.%), while Barbosa et al. [23] noted higher HV values on IMCs and Si crystals in relation to the Al-rich primary phase (Al_α).

More recently, comparative studies on solidification and T6-heat treatment conditions in Al-7Si-xMg (wt.%) cast aluminum alloys have been carried out [6, 18, 19, 24]. Barbosa et al. [6] and Azevedo et al. [19] analyzed the interrelation between the horizontal solidification and treatment processes parameters on the microhardness and wear performance from the work of References [22, 23]. Chen et al. [24] developed investigations to analyze the influence of Mg composition and HT conditions on the mechanical properties of Al-7Si-xMg cast aluminum alloys (x ≅ 0.26, 0.4 and 0.58 wt.%). In this sense, the present work is a continuity of investigations [6, 18, 21–23] which, although broad, still left a large gap in terms of a more complete mechanical analysis and, thus, aims to present a study on the correlation between tensile properties and microstructural parameters and fracture feature before and after thermal treatment by T6.

2 Experimental Procedure

In this work, an Al7Si0.3 Mg alloy ingot (wt.%) was obtained by means of a horizontal water-cooled solidification device. Figure 1 shows a complete assembly scheme of the furnace used in the solidification experiments. The experimental apparatus used to achieve horizontal solidification of studied allo, shown in Fig. 1a, was designed in such a

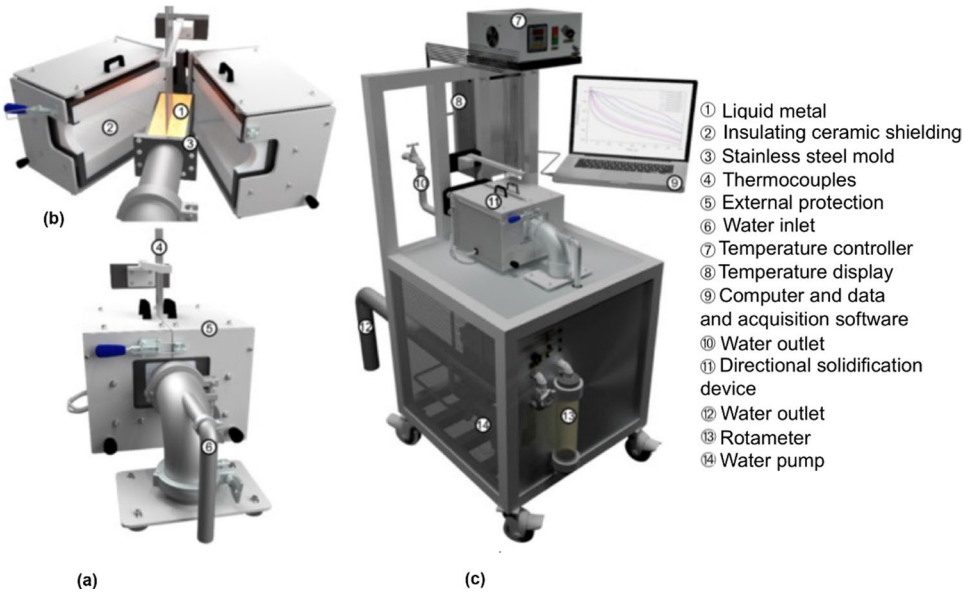

Fig. 1 Details of the solidification furnace: **a** and **b** horizontal solidification device showing the water injection system, ingot mold and thermocouples and **c** complete assembly of the horizontal solidification furnace

way that the heat was extracted only through a water-cooled system placed in the lateral mold wall, promoting horizontal solidification. A stainless steel mold was used which was 150 mm of long, 60 mm of wide, 60 mm of high and 3 mm of thick. Experiment was performed with superheat of 10% above the *liquidus* temperature (T_L) of Al-7Si-0.3 Mg (wt.%) [6, 19, 21].

Experimental temperature curves were obtained and used to determine the solidification thermal parameters, such as growth and cooling rates (V_L and T_R, respectively). Additional details on the operational procedures of the experiments can be consulted in our recently published works [6, 19, 21–23].

In our recent works [6, 19], the precipitation treatment by T6 has been carried out on as-cast samples of the horizontally solidified investigated alloy under the same conditions of this work. It is important to emphasize that the operational parameters for application of the T6-thermal treatment followed guidelines of ASTMB-597-92-Standard Practice for Heat Treatment of Aluminum Alloys, that is: solution treating performed during 3 h at 520 ± 2°C, followed by quenching in warm water (70 ± 2°C), aging for 1, 2, 3, and 4 h at 155 ± 2°C and air-cooling, as shown by the thermal cycles in Fig. 2.

Specimens for tensile tests were prepared from the as-cast ingot, as shown in Fig. 3. In order to guarantee the reproducibility of the results, six samples were prepared for each position, three for analysis of the solidification conditions (V_L, T_R and λ_2) and the other three to evaluate the HT conditions. Thus, from the six as-cast specimens, three were submitted to the T6 -treatment, according to the thermal cycle of Fig. 2, considering the aging time equal to 3 h, since the highest microhardness value (93.23HV on the Al rich phase) was obtained for this condition [6]. The tests were carried out according to ASTM standards: E384/11 and E8M/04. They have been conducted with a strain rate 0.05 mm $s^{-}1$ [25] and the investigated properties include ultimate tensile strength (σ_{UTS}) and elongation (E%).

In turn, as-cast and HT samples were subjected to microstructural characterization. The length of the microstructural scale was analyzed using secondary dendritic spacing (λ_2). Once the λ_2 values were measured, following the technique from the literature [26, 27], they were correlated with solidification parameters and tensile properties of both investigated samples. Image processing MOTIC system and the Image J software were used to measure the secondary dendrite arm spacings, and at least 20 measurements were taken for each selected position along the casting length. A scanning electron microscope (SEM TESCAM,VEGA LMU) coupled to an energy dispersion spectrum (EDS X-MAX 20, Oxford) was used to investigate fracture feature of both as-cast and heat treated specimens.

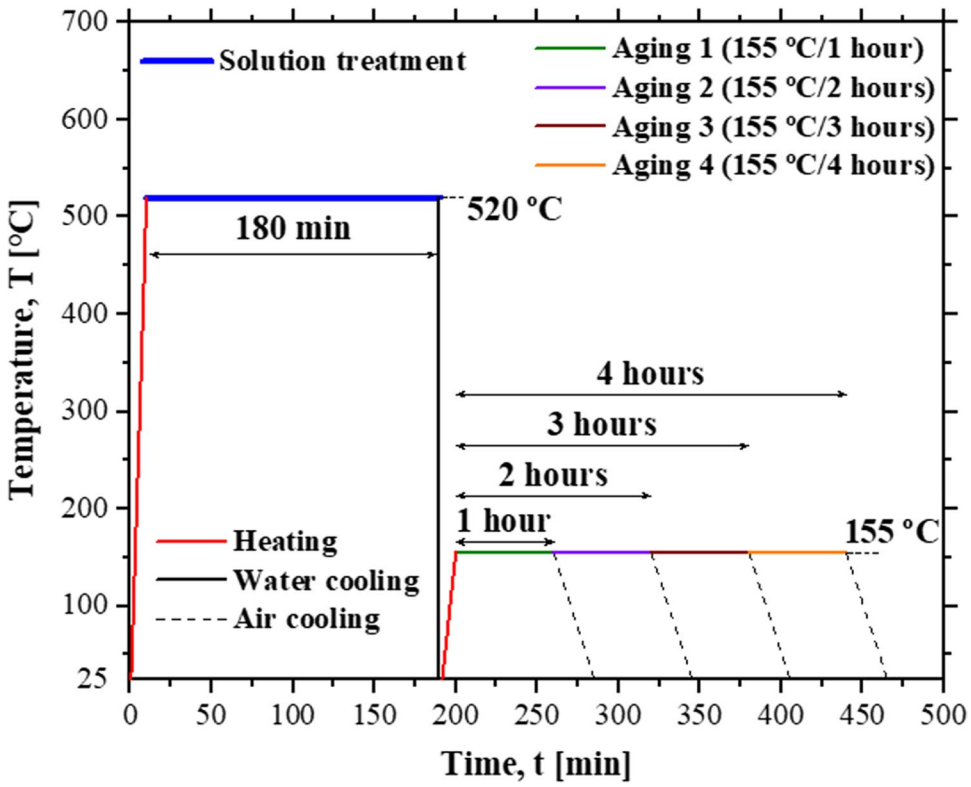

Fig. 2 Thermal cycle showing the operational parameters of the three stages of the T6 -treatment

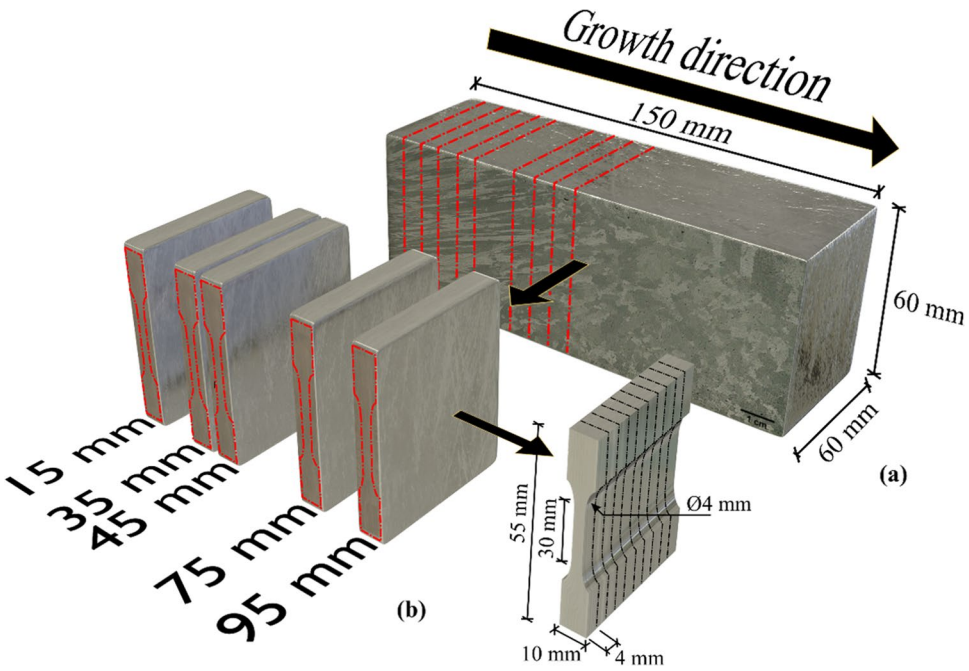

Fig. 3 **a** As-cast ingot of the investigated alloy, resultant from the solidification process and **b** cheme of sample removal from the ingot resulting for tensile tests

3 Results and Discussion

It is known that the solidification thermal parameters exert effects on the length of the microstructural scale. It has been evaluated by the experimental results achieved considering the solidification conditions assumed in the present work, such as the growth and cooling rates (V_L and T_R, respectively), which were obtained from experimental thermal data resulting from horizontal. The thermocouples readings, represented by temperature–time profiles shown in Fig. 4a, were used to generate a plot of position from metal/mold interface (P) as a function of time (t) corresponding to the liquidus front passing by each thermocouple. Very similar experimental points were obtained, and then, a single equation given by $P = 3(t)^{0.76}$ was proposed. The derivative of this function, that is, dP/dt provided V_L values, as shown in Fig. 4b. The experimental $T_R = f(P)$ profile was calculated by considering the time derivative of each cooling curve right after the passage of the *liquidus* isotherm (T_L) by each thermocouple [6, 19, 21–23, 28–30]. It is clearly observed that the cooled mold imposes high V_L and T_R values close to the heat transfer interface, which decrease with the increasing formation of the solid layer. Mathematical power equations given by the general expression $A.P^{-a}$ characterize the V_L and T_R variation with the position in the as-cast ingot. This significantly influenced the dendritic network, inheriting finer dendritic microstructures for higher V_L and T_R values, as can be seen in Fig. 4c. As observed, it has been represented by experimental power expressions in the forms of Constant. $V_L^{-2/3}$ and Constant.$T_R^{-1/3}$, which characterize the λ_2 coarsening laws with the solidification thermal parameters.

In our works [6, 19, 21, 23] the λ_2 values were measured from the cooled interface, in both as-cast and heat-treated samples, processed under the same conditions assumed in the present work. The expressions resulting from the investigations, which characterize the growth laws of λ_2 of the aforementioned samples are presented in Fig. 5. It was observed that the HT has affected the length of the dendritic microstructure scale, since the λ_2 values came be greater in the treated samples by T6, as seen in Fig. 5. It was also observed that finer microstructures contributed to the dissolution of the Mg_2Si-intermetallic compound within the Al-rich matrix [6, 19, 24], as well as potentiated the spheroidization process of the eutectic Si crystals during the solution treatment, as depicted by the microstructures above the graph in Fig. 5. Finer microstructures have also allowed better dissolution of Al_2Cu and obtaining spherical-like Si particles during Treatment by T6 in AlSiCu alloys [17, 18, 25, 28].

Figures 6 and 7 present, for both investigated specimens, the results obtained from the tensile tests for two as-cast and heat-treated samples from the heat transfer surface, and studied mechanical properties, respectively. Higher mechanical strengths were achieved for heat-treated samples, as can be seen in Fig. 6a and b. It is observed that the analyzed properties performance along the position in the ingot has not allowed to obtain a mathematical relationship as a function of the solidification parameters (V_L, T_R e λ_2), especially for the σ_{UTS} values, as seen in Fig. 7a, although finer solidification microstructures have inherited a slight improvement in deformation (E%), as noted in Fig. 7b. This induced the calculation, for

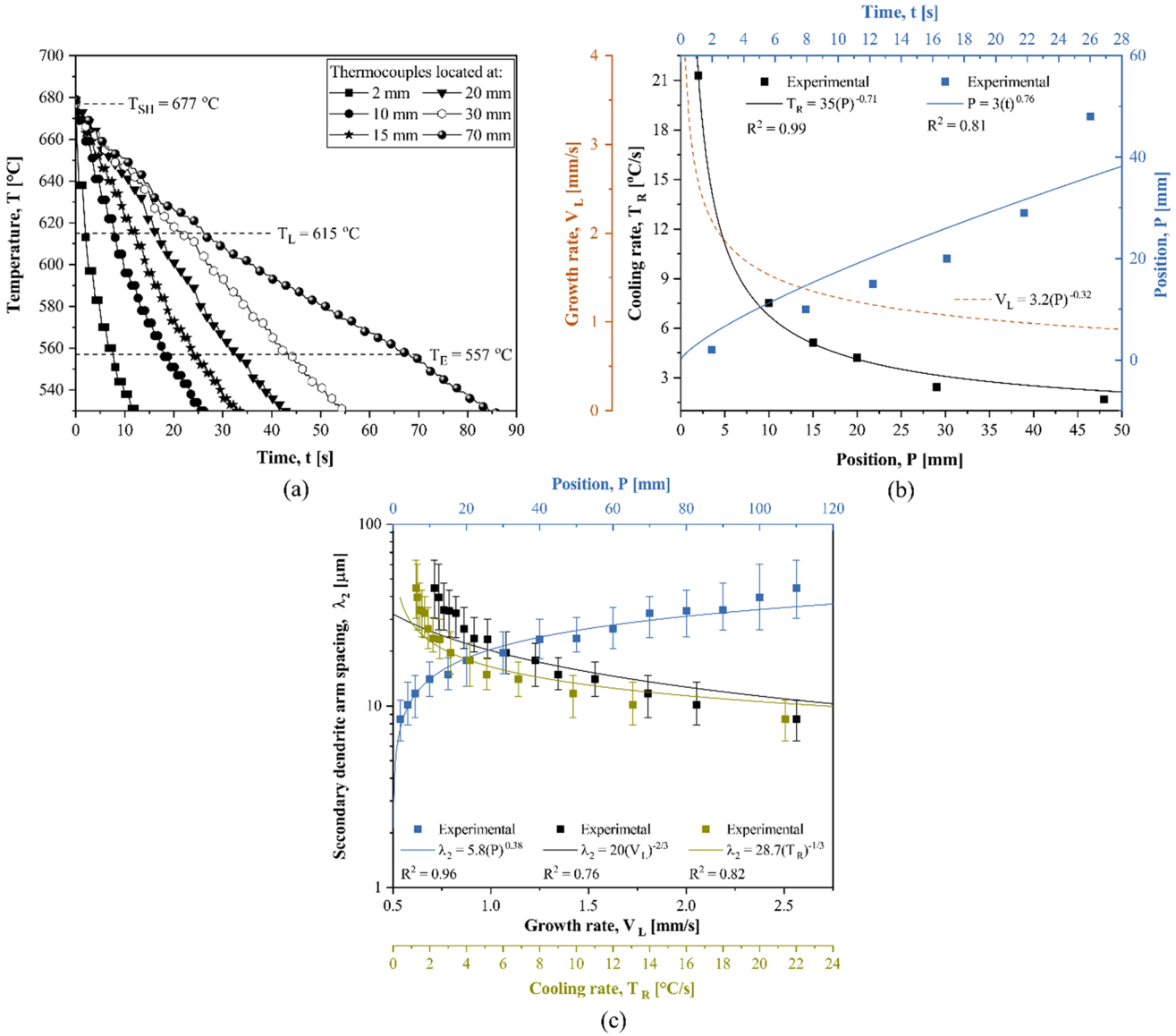

Fig. 4 Transient thermal and microstructural parameters, resulting from the horizontal solidification process: **a** experimental temperature curves for 6 thermocouples from the heat -transfer interface; **b** Growth and cooling rates (V_L and T_R); and **c** secondary dendritic spacing (λ_2) as a function of the position in the ingot, V_L and T_R. R^2 is coefficient of determination

both studied samples, of a resulting average value from the averages of each position, as shown in Fig. 7c. High σUTS and E% values can be observed for heat-treated samples, that is, increases from 132.4 to 194.4 MPa (≅47%) and 5.8 to 6.1% (≅ 5.5%) have been achieved with the HT, confirming the precipitation hardening without deleterious effects on the deformation. In general, the increase in mechanical resistance achieved with HT does not cause damage to deformation, as can be seen in Fig. 7c. Precipitation hardening also have been observed in the results of HV from the work of Reference [6] for the horizontally solidified and heat-treated Al7Si0.3 Mg alloy in the same conditions of this work.

However, Souza et al. [25] have established an inverse mathematical relationship of σ_{UTS} and E% with λ_2 for the horizontally solidified Al7Si3Cu alloy, that is, they have observed higher σ_{UTS} and E% values for smaller secondary dendrite arm spacing, but HV and yield strength (σ_{YS}) did not vary with λ_2. In turn, Souza et al. [18] have submitted samples of the horizontally solidified Al7Si3Cu alloy to the T6-heat treatment and observed the hardening of the alloy as well as higher HV values with increasing aging time. An investigation [24] has reported higher σ_{UTS}, σ_{YS} and E% values for finer microstructures in as-cast samples and heat treated by T6 of Al7SixMg alloys, also observing higher values of the aforementioned mechanical properties after

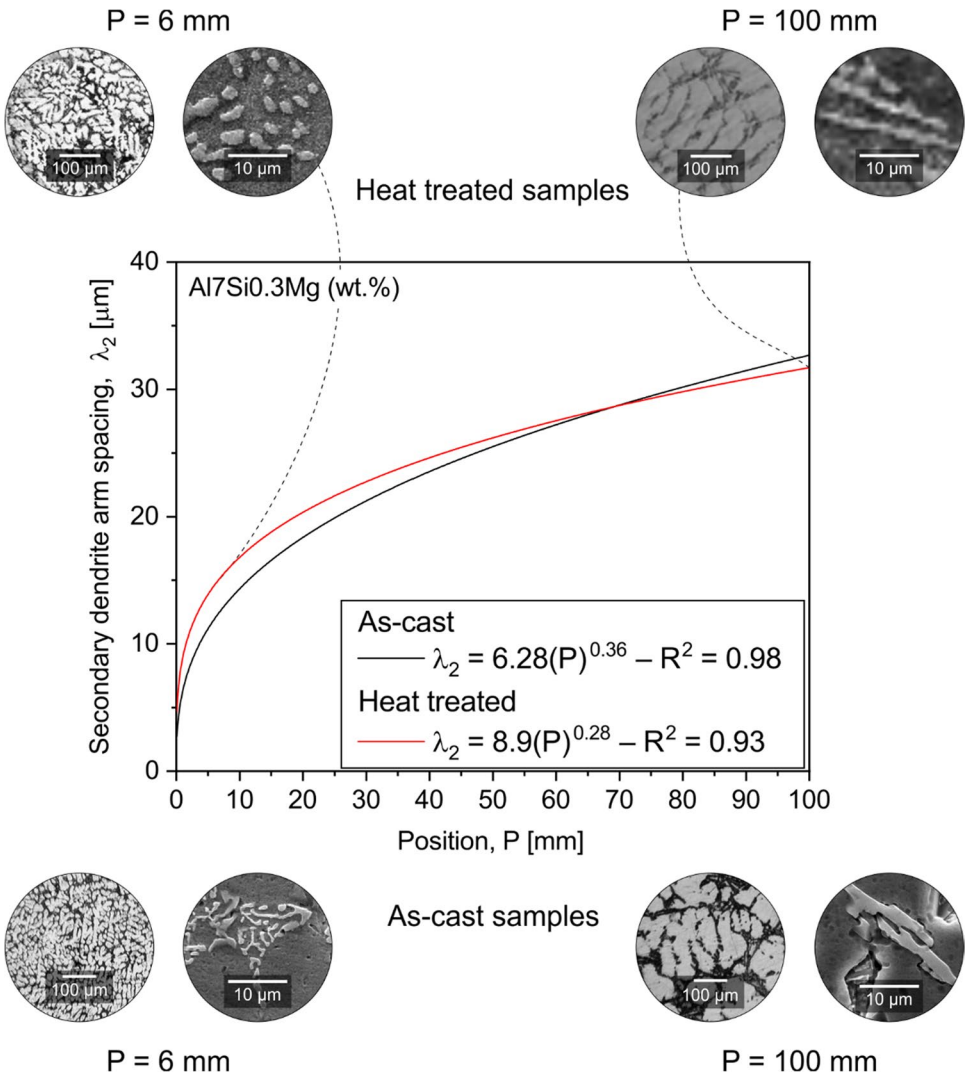

Fig. 5 Comparative curves of λ_2 evolution for both analyzed samples, showing microstructural evolution for two positions from the cooled base

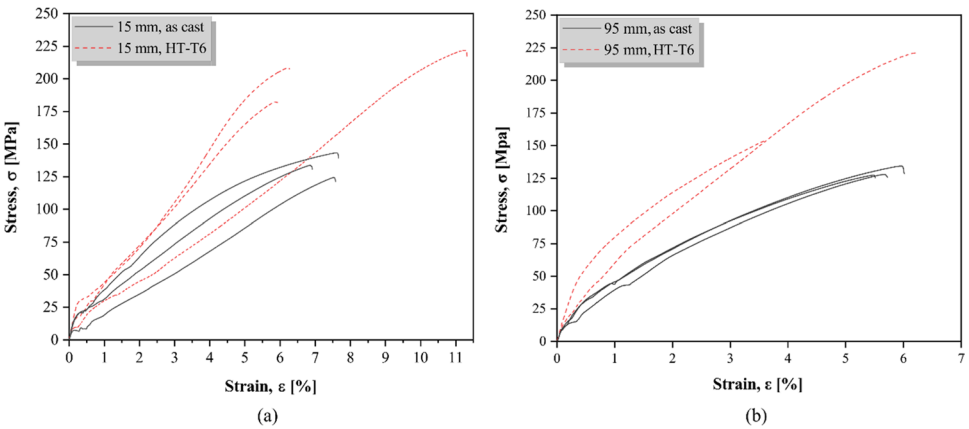

Fig. 6 Stress x Strain diagrams for two as-cast and heat-treated samples in positions from the cooled base: (15) mm and (b) 75 mm

HT, as well as proposing an inverse mathematical relationship of σ_{UTS}, σ_{YS} and E% with a function of λ_2.

In our research [19], the HT applied under the assumed conditions after solidification process of the investigated automotive alloy affected the dendritic microstructure scale, since higher λ_2 values have been obtained in T6/heat-treated samples. This has influenced the wear resistance of the Al7Si0.3 Mg (wt%) alloy, since lower wear volumes and

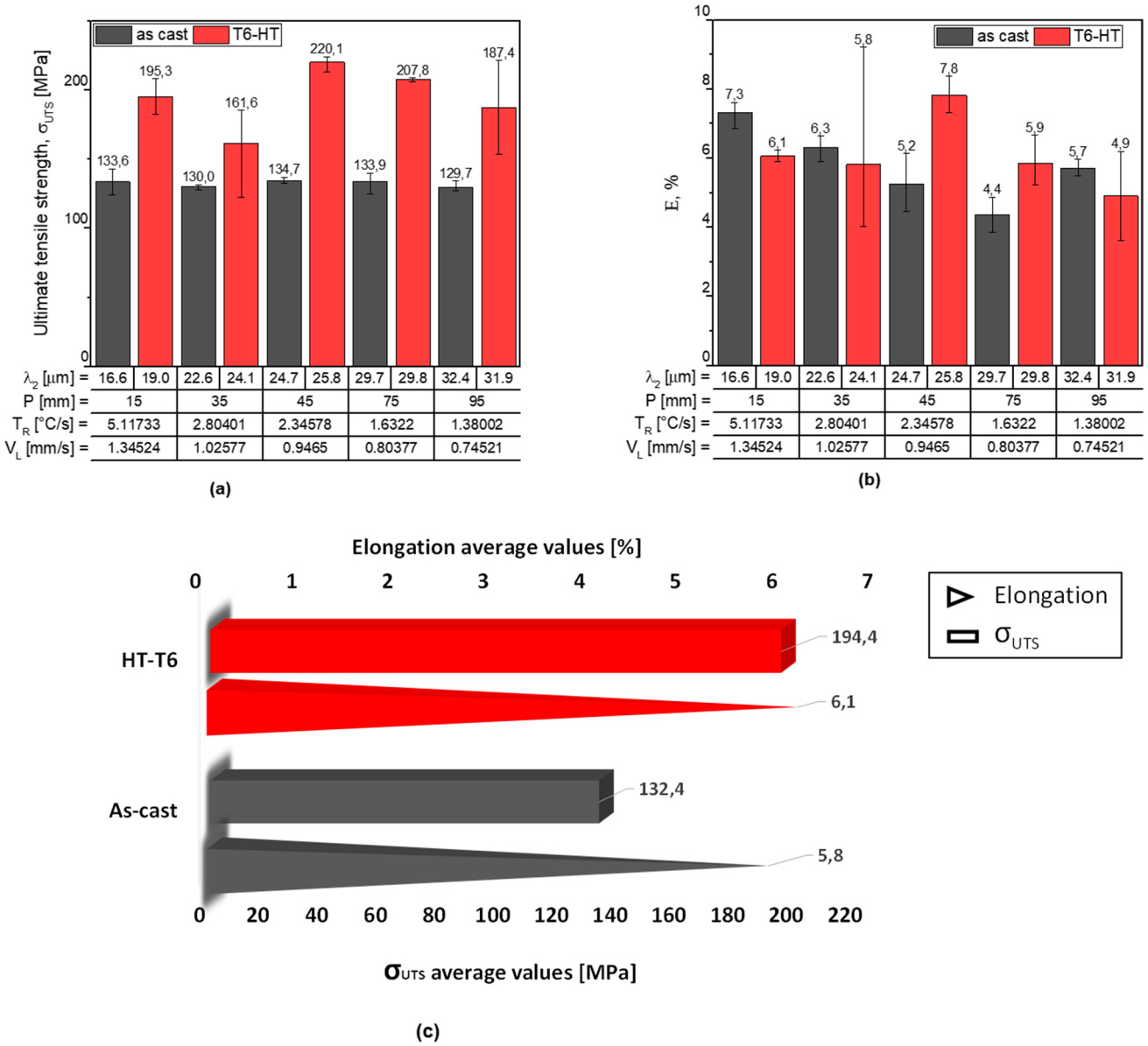

Fig. 7 Experimental results of the studied properties resulting from the tensile tests: **a** and **b** ultimate tensile strength and elongation vs. solidification parameters, respectively and **c** resulting average values of ultimate tensile strength and elongation, calculated for all position of both investigated samples

rates were achieved in the as-cast and heat-treated samples for finer and coarser microstructures, respectively. Thus, the results obtained for E%, shown in Fig. 7b, in which the finer microstructures present the highest E% value, are consistent with the results of the wear behavior for the heat-treated samples [19]. This can also be attributed to the degeneration of tertiary dendritic branches during solution treatment, as observed in [6]. Correlations between wear rate and structure were proposed for as-solidified and heat-treated Al 2 Mg-(5 and 8)Zn (wt%) alloys, and the results, in general, showed that better wear resistance is associated with higher hardness and strength. However, depending on the characteristics of the microstructure and contact aspects, the wear resistance can have an opposite behavior, inducing the inappropriate selection of the material condition.

A more complex dendritic network of the Al-rich primary phase, consisting of primary, secondary and tertiary branches, may affect the mechanical strength the as-cast alloy. It is known that interdendritic spacing, and the precipitates distributed along the interdendritic regions, acts as strength-increasing mechanisms, creating barriers to dislocation movement during plastic deformation, playing a fundamental role in the tensile results and in the fracture characteristics of metal alloys. In addition, finer dendritic

microstructures can also increase the mechanical strength of as-cast products. The degeneration of the dendritic network with heat treatment, such as the collapse of the tertiary arms, for example, may be decreasing this barrier.

Figures 8, 9, 10, 11 show the SEM images and element scans mapping by EDS of the fractured areas for two as-cast and heat-treated samples, revealed in two samples solidified under different solidification thermal parameters conditions. In general, it is observed that the features of all fracture surfaces of the studied samples indicate a mix between brittle and ductile fractures, constituted by cleavage facets, secondary cracks, facets covered with micro-voids, tear ridges and dimples. In addition, the Mg_2Si/IMCs-like Mg element, initially segregated within the interdendritic regions of the as-cast samples, as noted by scan mappings in Figs. 8 and 10, has been dissolved in the Al-rich matrix during the solution treatment, as can be verified in Figs. 9 and 11 as well as has been reported in our works [6, 19]. Regarding the morphology and fracture modes analysis from the SEM fractographies of as-cast regions, it has shown a predominance and better distribution of small dimples against cleavage facet regions in position close to the heat-transfer surface, deducing the occurrence of a transition from ductile to fragile fracture with decreasing cooling rate as well as with increasing secondary dendritic spacing, as seen in Figs. 8 and 10, respectively. This fracture feature can be associated by the elongation performance observed along the as-cast ingots, since higher and lower E% values have been obtained for positions near and far from the cooled base, respectively, as reported in Fig. 7b. Souza et al. [25] also reported for the horizontally solidified Al7Si3Cu alloy (wt.%) a transition from ductile to brittle fracture along the position of the as-cast ingot, with the authors attributing it to the decrease in T_R as well as the increase in λ_2. The increase in the cooling rate and, consequently, the refinement of the dendritic microstructure, have caused transitions in both fracture path from transgranular to intergranular as well as in the fracture mode from quasi-cleavage for dimple [24].

In turn, the presence of dominant cleavage regions is observed in the heat-treated fractured samples, as seen in Figs. 9 and 11, allowing to admit the predominance of brittle fracture in both analyzed positions. It has been observed more extensive cleavage areas in the furthest position from the cooled base, as noted in Fig. 11. This has inherited a deformation behavior similar to that observed in as-cast samples, i.e., lower E% values have been achieved in heat-treated positions far from the heat-transfer surface, as can be seen in Fig. 7b.

Therefore, the cleavage planes seem to be closely associated with the silicon crystals present in the microstructure, thus, the larger and more faceted the Si particles, the fracture will tend to be more brittle, with a predominance of cleavage planes, whereas when refining and spheroidizing the Si-crystals, ductile fracture mechanism will prevail. Was observed

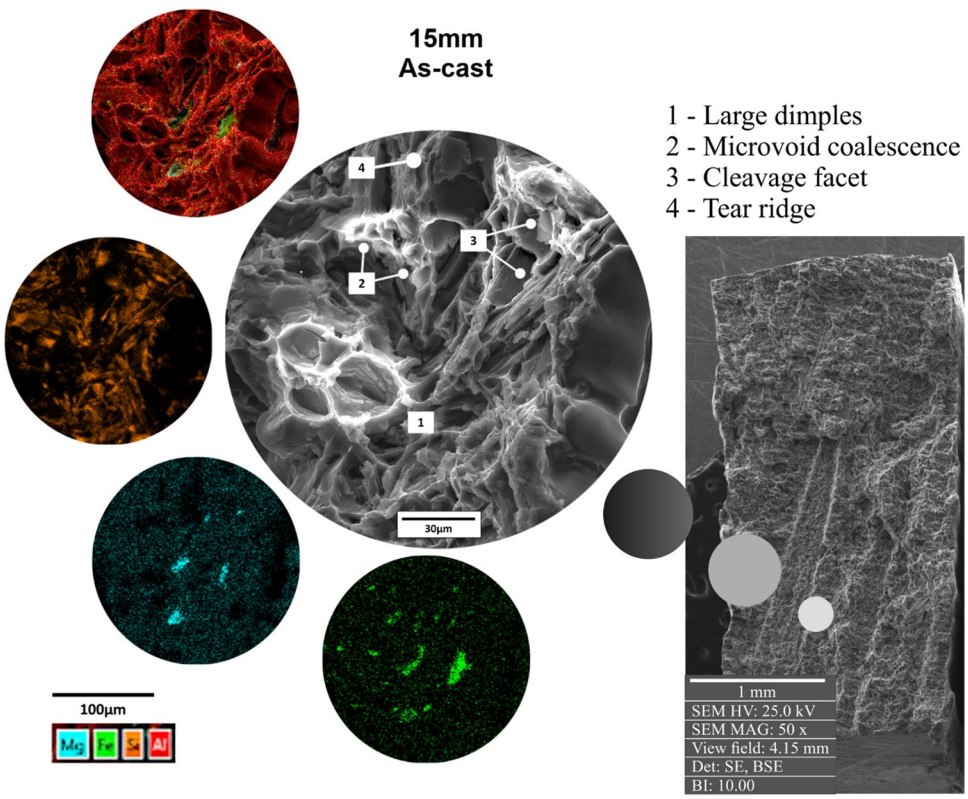

Fig. 8 SEM fractographies and elemental scanning mapping by EDS for both investigated as-cast samples in the ingot equal to 15 mm

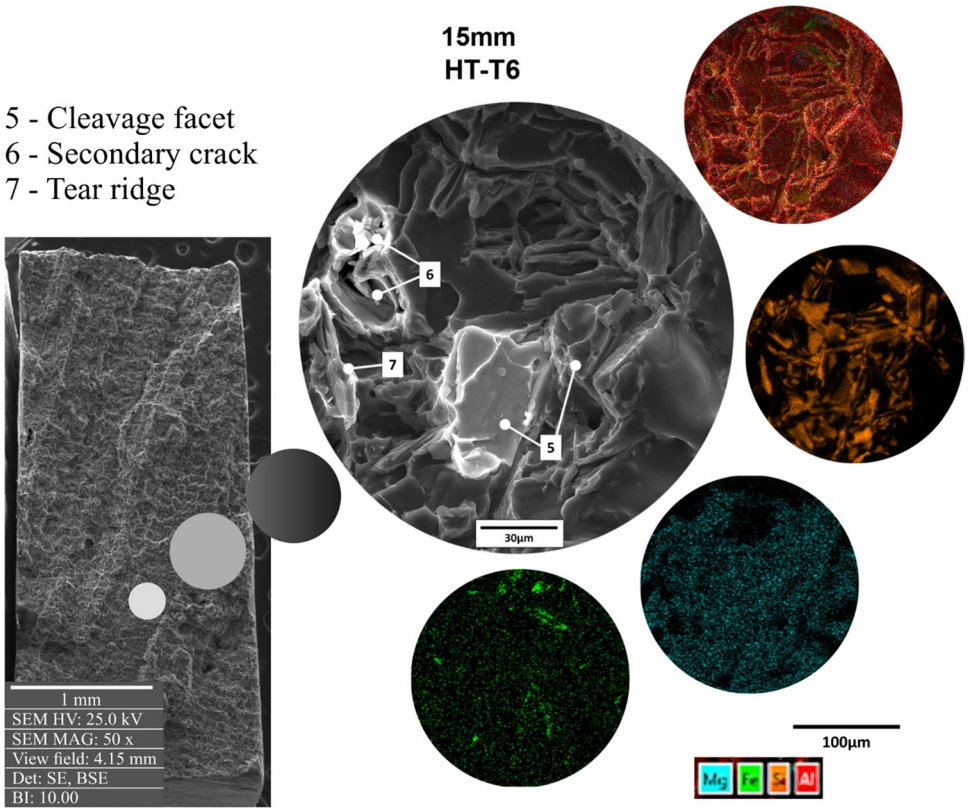

Fig. 9 SEM fractographies and elemental scanning mapping by EDS for both investigated HT samples in the ingot equal to 15 mm

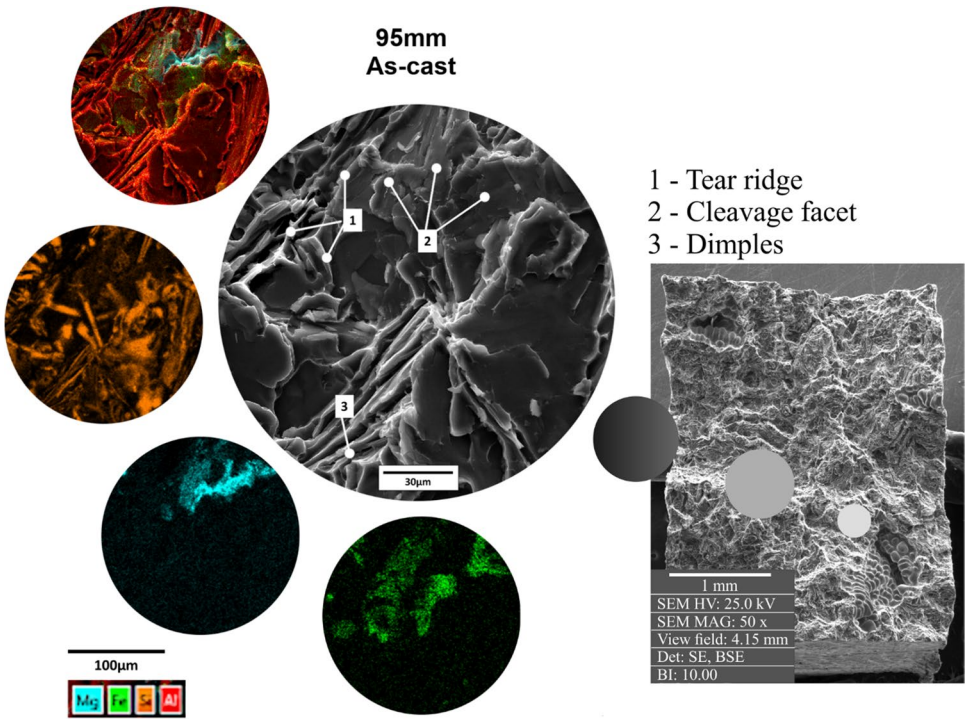

Fig. 10 SEM fractographies and elemental scanning mapping by EDS for both investigated as-cast samples in the ingot equal to 95 mm

that the presence of Mg_2Si-IMC increases the density of dimples in fracture surfaces possibly because this phase acts as a deformation energy absorber, promoting controlled nucleation of microvoid in the interdendritic region. On the other hand, for HT specimens the Mg_2Si-IMC particles are solubilized from the interdendritic region to the α-Al matrix,

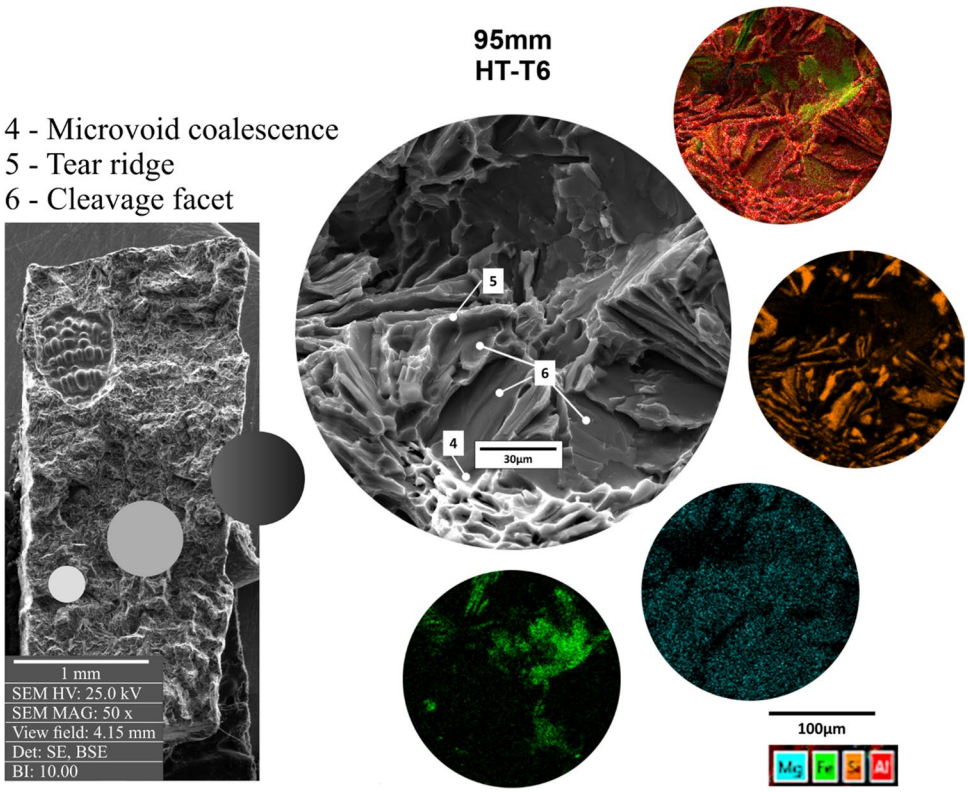

Fig. 11 SEM fractographies and elemental scanning mapping by EDS for both investigated HT samples in the ingot equal to 95 mm

leading a direct interaction between Si-crystal (high elasticity module) and α-Al phase (low elasticity module), increasing the occurrence of brittle fracture.

4 Conclusion

The main conclusions obtained from this study are:

1. Ultimate tensile strength values were achieved for heat-treated samples, proving the effectiveness of HT under the assumed conditions. Despite the hardening of the Al-rich matrix, this has not influenced the elongation performance, since in general the E% values remained very close in both investigated samples, even being slightly better.
2. Under the assumed conditions, finer microstructures have inherited better deformation conditions for both solidification and heat-treatment samples.
3. Analysis from the SEM/EDS fractrographs of both as-cast and heat-treated samples has shown a perfect dissolution of the Mg_2Si-IMCs in the matrix during the solution heat treatment. In addition, EDS scan mappings have depicted finer Si and Fe particles in a-cast samples, for higher and lower T_R and λ_2 values, respectively. This has inherited better conditions for obtaining spherical-like Si crystals after the solution treatment and probably contributed to the good E% performance.
4. From the analysis of fracture features performed on both as-cast and heat-treated samples, it was observed by the fracture morphologies in the as-cast samples the predominant presence of dimples and cleavage for finer and coarser microstructures, respectively, deducing the occurrence of a transition from ductile to brittle fracture modes along the length of the ingot. While in samples treated by T6, cleavage areas became prominent, suggesting a predominance of brittle fracture.

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Author Contributions All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed Carolina Rizziolli Barbosa, Hugo André Magalhães de Azevedo, Sharlane Costa, João Eduardo Pinto Castro Ribeiro, José Carlos de Araújo Cardoso Filho, Thiago Antônio Paixão de Sousa Costa, and Otávio Fernandes Lima da Rocha. The first draft of the manuscript was written by Otávio Fernandes Lima da Rocha and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Data Availability All data generated or analysed during this study are included in this published article.

Declarations

Consent for Publication Not applicable.

Competing Interests The authors declare no competing interests

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