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Parts repairing and microstructural refinement of high-pressure die cast aluminum alloys through friction stir processing for bulk production

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A B S T R A C T

A key challenge in the production of high-grade automotive aluminum components through the High-Pressure Die Casting (HPDC) process is the imperative to minimize imperfection. In addressing this concern, this study utilizes friction stir processing (FSP), a widely recognized intense plastic deformation technique. FSP is applied to systematically alter the microstructure of HPDC Al-4Mg-2Fe, a prominent alloy extensively used in the die-casting sector. By using the pass strategy to incorporate both one-pass and two-pass approaches, the microstructure is selectively altered to establish a defect-free processed zone. The utilization of FSP demonstrates its efficacy in breaking aluminum dendrites and acicular silicon particles, leading to a uniformly dispersed arrangement of equiaxed silicon particles within the aluminum-based matrix. In addition, FSP eradicates porosity and disintegrates needle-like Fe particles, resulting in a more refined and homogeneously distributed structure. Subsequently, the material's mechanical properties processed by FSP were assessed in the longitudinal direction concerning the processing axis and then compared with those of the original base material.

The microstructural refinement and reduction in porosity induced by FSP result in a notable enhancement in hardness, with an increase of 23 % after one pass and 37 % after two passes. The substantial improvement in mechanical properties during the FSP process is predominantly attributed to modifications in the morphology, refinement, and dispersion of intermetallic particles within the matrix. This improvement is further complemented by the ultrafine dispersion of casting defects.

This study underscores the efficacy of FSP as a valuable tool for modifying microstructures and improving mechanical properties in HPDC Al-4Mg-2Fe alloys. Such advancements align with the lightweighting objectives pursued by the automotive industry.

Introduction

In major global economies, automobiles hold a crucial position, yet their presence contributes to energy consumption and environmental impact. In response, automotive original equipment manufacturers (OEMs) have placed a high priority on improving fuel efficiency. One effective strategy involves the reduction of weight in light-duty vehicles, achieved by replacing heavy steel structural components with aluminum alloy castings (Nakata et al., 2006). Due to their lightweight nature, improved castability, commendable corrosion resistance, and favorable mechanical properties, aluminum alloys produced through HPDC present a compelling opportunity for reducing the weight of light-duty vehicles. This goal is accomplished through the replacement of structural elements within an automobile's framework with substantial, thinly walled, and intricately formed castings (Benedyk, 2010). Nevertheless, for High-Pressure Die Casting (HPDC) components to effectively serve as structural elements, it is essential for them to demonstrate a balanced combination of strength, ductility, fracture toughness, and, in certain instances, improved fatigue properties. Numerous HPDC aluminum alloys often incorporate significant amounts of iron (Fe) to alleviate die-soldering problems and address microstructural features arising from the casting process, including porosity and inclusions. Regrettably, these attributes have a detrimental impact on the mechanical properties, thereby restricting their suitability for application as structural components in an automobile's body.

The existence of porosity within castings can lead to the initiation of cracks under mechanical stress. Research in alloy development has pinpointed certain HPDC aluminum alloy compositions, such as Silafont,

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Castasil, Aural, etc., characterized by superior mechanical characteristics (Samanta et al., 2022). Similarly, advancements in die-casting techniques, specifically vacuum-assisted HPDC, have played a role in minimizing casting flaws, such as porosity (Niu et al., 2022) However vacuum-assisted HPDC process is costly.

The targeted local processing of a conventional HPDC component has the potential to eliminate defects and adverse microstructural features that contribute to a decline in mechanical properties, particularly in areas of stress concentration. If successful, this approach could facilitate the use of aluminum alloy components produced through HPDC methods, rather than relying on the above HPDC alloy compositions or processes. This marks a notable advancement in the pursuit of cost-effective lightweighting. HPDC alloy, commonly employed in the die-casting process, is widely utilized in manufacturing automotive components like gearbox cases and engine brackets. Nevertheless, challenges arise in the form of reduce hardness and ductility due to the presence of gas and shrinkage porosity. Additionally, the inclusion of silicon and iron-containing particles, coupled with the existence of substantial second-phase particulates in HPDC aluminum alloys, further contributes to these adverse effects (Outmani et al., 2017). Hence, it is crucial to carry out microstructural adjustments and alleviate porosity to improve both the hardness and ductility of HPDC aluminum alloys.

Friction Stir Processing (FSP) has emerged as a sophisticated thermomechanical technique, which can affect the material microstructure by the concurrent effect of heat and deformation (Mishra et al., 2000). The FSP procedure involves a rotating tool that imparts substantial plastic deformation to the workpiece material, leading to intricate material flow and mixing (Karthikeyan et al., 2010) . FSP has been proven as an effective process in altering the microstructural characteristics of cast aluminum alloys (Ma et al., 2022) and alloys containing magnesium (Luo et al., 2020). Cavaliere et al. (Cavaliere and De Marco, 2006) explored the influence of Friction Stir Processing (FSP) on the mechanical and microstructural characteristics of magnesium alloys manufactured via High-Pressure Die Casting (HPDC). Santella et al. (Santella et al., 2005) have reported microstructural modification of cast aluminum alloys by FSP. Samanta et al. (Samanta et al., 2022) have reported the microstructure refinement of HPDC A380 alloy by FSP. In another publication, Samanta et al. (Samanta et al., 2024) also reported the enhancement in toughness and tear resistance of HPDC aluminum alloy by FSP. FSP can contribute to the reduction of porosity and the refinement of the microstructure in cast materials, leading to improved performance (Sun and Apelian, 2022). The flow of material and the alteration of microstructure in Al-Si-based cast alloys during FSP are significantly influenced by two essential process parameters: tool rotational speed and traverse velocities (Karthikeyan et al., 2009). Furthermore, the refinement and mechanical properties of these alloys are influenced by both the number of passes and the extent of their overlap (Z.Y. Ma et al., 2006). Aktarer et al. (Aktarer et al., 2015) investigated the effect of two pass FSP on the microstructural and mechanical properties of as-cast binary alloy. Wang et al. (Wang et al., 2021)explored the impact of multipass FSP on both the microstructural and mechanical properties of cast aluminum alloy. The increment of mechanical and microstructural properties has been observed. Prior research has demonstrated the efficacy of FSP in eradicating porosity while also disintegrating needle-shaped Si particles and Al dendrites in sand-cast A356 alloy (Z.Y. Ma et al., 2006), alloy produced by investment cast Al-7Si-0.6Mg (Jana et al., 2010) alloy produced by die-cast A383 (Nakata et al., 2006), and alloy produced by steel mold-cast A380 (Çamurlu and Ünal, 2011). Sun et al. (Sun et al., 2014) anticipated the impact of microporosity on the mechanical properties and fracture behavior of HPDC AM50 magnesium alloy. Ghio et al. (Ghio and Cerri, 2023) investigated the corrosion behavior of HPDC AZ91 Alloy and the impact of FSP at elevated rotational speeds. Additionally, FSP can disperse large second-phase particles, promoting a uniform distribution with consistent shapes in aluminum castings (Santella et al., 2005). The cumulative influence of these alterations in microstructure resulted in enhanced strength and ductility (Rao et al., 2015). Furthermore, the elimination of porosity reduced potential sites for crack initiation, thereby enhancing fatigue resistance failure (Nelaturu et al., 2018). FSP exhibits the capability to improve not only the machinability (Guru et al., 2015)but also the tribological properties such as friction, lubrication, wear (Mahmoud and Mohamed, 2012)and corrosion resistance of cast Aluminum alloys (Rao et al., 2014) .

HPDC Al-4Mg-2Fe is a newly developed alloy containing Fe-rich intermetallic compounds that is extensively being used in battery frame structure (Patel et al., 2023) and automotive industries because of low density, high ductility, excellent weldability, castability and high corrosion resistance (Liu et al., 2021). Nevertheless, challenges arise due to the presence of porosity and large eutectic Si and Fe-containing particles which restrict their overall performance and compromise the material properties. The literature study on HPDC alloy revealed that mostly HPDC A380 alloy have been studied extensively for microstructural refinement. Fe-rich intermetallic compounds HPDC Al-4Mg-2Fe have not been used for microstructural refinement by using FSP or any other techniques.

In this research, FSP is employed to selectively modify the microstructure of a plate made of HPDC Al-4Mg-2Fe alloy. The primary focus is on exploring the impact of microstructural refinement by the number of passes. The study explores the influence of FSP on addressing cast porosity and modifying needles like Si and Fe-containing particles. Furthermore, a comparative analysis is conducted between the hardness, microstructure, and porosity of HPDC Al-4Mg-2Fe alloy, and the ones of the material subjected to FSP.

Experimental method

HPDC Al-4Mg-2Fe plates with approximately 3.0 mm thickness were employed for the FSP experiment. The chemical composition of the material is outlined in Table 1. The plates were in an as-cast state, and no heat treatment was administered to them.

FSP trials were carried out using a Tri Flat welding tool manufactured from H13 steel, featuring a shoulder diameter of 12 mm, pin diameter of 6 mm, and height of 3 mm. The experiments were executed in force control mode, achieving a penetration depth of 3 mm, with the tool rotating counterclockwise at a tilt angle of 1° Multiple experiments were conducted, manipulating tool rotational speed from 1500 to 2000 RPM, traverse velocity from 5 to15 mm/s, and force from 4 to 4.5 kN to determine a region free from surface defects post-FSP. Some of the authors have optimized the process parameters in previous research (Bates et al., 2023). Ultimately, a tool rotation speed of 2000 rpm, a traverse velocity of 10 mm/s, and a force of 4 kN were chosen.

An experimental campaign consisting of both single-pass and two-pass operations was conducted along an identical trajectory and in the same direction using a robotic welding machine. Metallographic samples, with dimensions of 30 × 15 mm, were obtained by cutting both the HPDC Al-4Mg-2Fe plate and the FSPed plates in a direction perpendicular to the tool traverse. Subsequently, these specimens were mounted in a Supplement 3000 automatic mounting machine and mechanically polished. The surfaces underwent preparation through standard metallographic techniques and were electro-etched using a 2 % NaOH solution reagent. Optical microscopy was then employed to observe and document the distribution of porosity, Fe particles, and second-phase particles. The analysis of micrographs for quantifying microstructural features and porosity in both as-cast and FSPed samples utilized the Opensource ImageJ software. Scanning Electron Microscopy (SEM) and energy-dispersive X-ray Spectroscopy (EDS) were utilized to assess the microstructure and distribution of elemental composition both prior to and following Friction Stir Processing (FSP). Microhardness evaluations of the FSPed specimens and HPDC Al-4Mg-2Fe were performed using a Vickers hardness tester, applying a 200 g load at a distance of 0.7 mm from the upper surface of the rectangular samples. Three rows of microhardness tests were conducted along longitudinal lines at 0.5 mm

Table 1
Chemical composition of HPDC Al-4Mg-2Fe (Liu et al., 2021).

Element	Mg	Fe	Si	Cu	Mn	Zn	Ti	Al
chemical composition	4.0–4.6	1.5–1.7	<0.2	<0.2	<0.14	<0.29	<0.21	Bal

intervals. The microhardness evaluations were performed using the DURAMIN-40 AC 1 hardness tester.

Results and discussion

Macrostructure and microstructure

In Fig. 1(a), optical micrographs showcase the appearance of a polished HPDC Al-4Mg-2Fe in its original, as-cast condition. Porosity is evident in the original HPDC, and coarse acicular Fe particles are dispersed along the edges of the primary aluminum dendrites are depicted in Fig. 1(b). A substantial number of pores are observable, with variations in size and density depending on the location. Regions closer to the die wall exhibit higher density and smaller casting pores. In contrast, the mid-wall region displays more extensive pores. Fig. 1(c) illustrates the SEM image of the as-cast HPDC Al-4Mg-2Fe, aiming to showcase the aluminum dendrites, an acicular silicon structure in the eutectic region, porosity, and needle-shaped iron particles.

Large needle-shaped silicon (Si) and iron (Fe) particles were distributed along the boundaries of the primary aluminum dendrites, with a dendrite size ranging between 100 and 200 μm. Some carbon contents have also been observed at higher magnification. The presence of carbon content is due to mounting as the base material doesn't contain carbon in their compositions.

Fig. 2(a) shows the optical micrographs of the joint cross section for the one Pass FSP HPDC sample processed under the selected conditions. The processed region exhibits notably reduced porosity compared to the adjacent, unaffected HPDC material. FSP induces a significant thermo-mechanical solicitation, involving elevated temperatures and intense plastic deformation, thus resulting in dynamic recrystallization and consequential alterations in the microstructure. The stirring action of the tool leads to the disruption of the dendritic microstructure. This disruption facilitates effective mixing of the material, resulting in a more homogeneous microstructure. The combination of intense plastic deformation along with the heat generated from friction and deformation serves as the primary driving force for solid-state microstructural alterations. These elements effectively hinder the growth of recrystallized grains, resulting in the formation of fine-grain microstructures. This fine-grain microstructure significantly contributes to improved strengthening and toughening efficiency.

As a result of FSP, acicular Fe particles and aluminum dendrites undergo considerable fragmentation, leading to a uniform distribution of smaller broken Fe particles within the aluminum matrix, as depicted in Fig. 2(b), with the corresponding SEM micrograph shown in Fig. 2(c). The size and aspect ratio of Fe particles in the FSP HPDC decreases with the increase in the number of passes, as illustrated in Fig. 3.

Remarkably, employing a two-pass Friction Stir Processing (FSP) with 100 % overlapping leads to an augmentation in the quantity of small-sized iron (Fe) particles. (Fig. 3b). The SEM image of the 2-pass FSPed HPDC alloy (Fig. 3c) aims to visualize the aluminum dendrites, an acicular Si particles, porosity, and needle-like Fe particles. A distinct recrystallized fine-grained microstructure, devoid of any dendritic structure, was noted in the alloy subjected to two-pass FSP. The presence of fine grains in the FSP-treated as cast alloy can be attributed to the substantial plastic deformation and thermal exposure experienced during the FSP. In addition to this, the significant density of finely dispersed Si and Fe particles, distributed uniformly, is believed to have played a crucial role in achieving a refined recrystallized grain structure. This outcome is attributed to the dynamic recrystallization (DRX) facilitated during the FSP process, with particle-simulated nucleation (PSN) influencing the process. Furthermore, FSP effectively eradicates porosity in the originally cast HPDC alloy.

Several key observations can be derived from microstructure analysis. Firstly, FSP induces substantial reductions in both the size and aspect ratio of Fe particles, along with a notable decrease in porosity. Secondly, at 2000 RPM tool rotational rate results in a further decrease

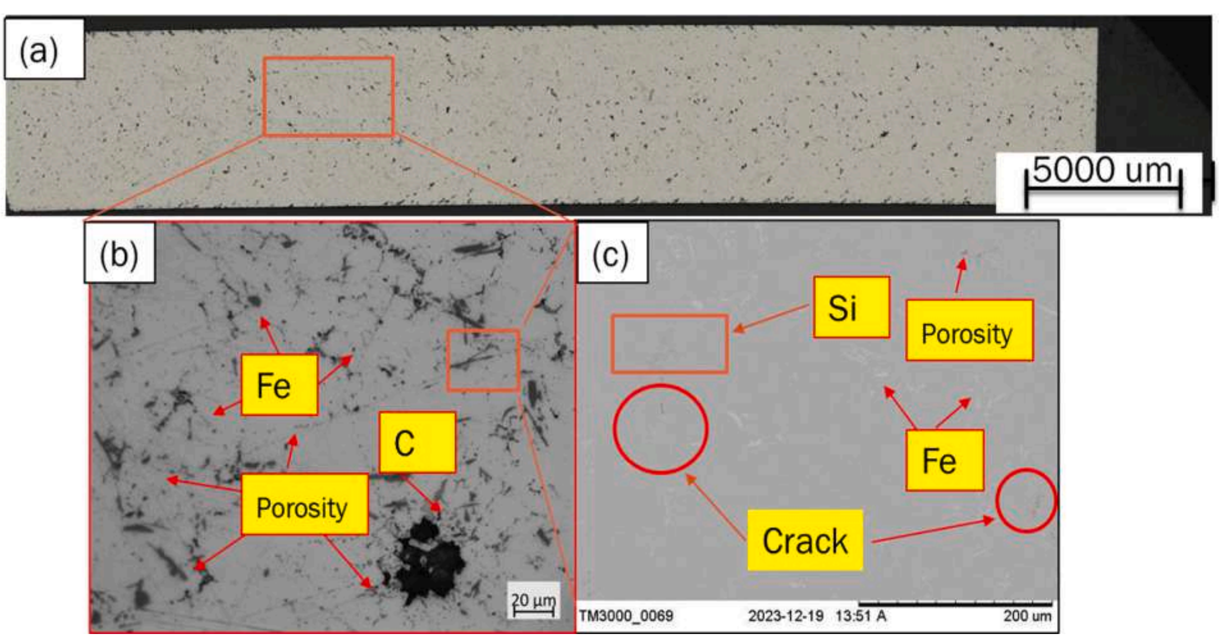

Fig. 1. (a) cross-sectional micrograph of HPDC Al-4Mg-2Fe sample (b) Optical micrographs illustrate the structure and morphology of Fe particles (c) SEM micrograph illustrating the as-cast microstructure of the surface region.

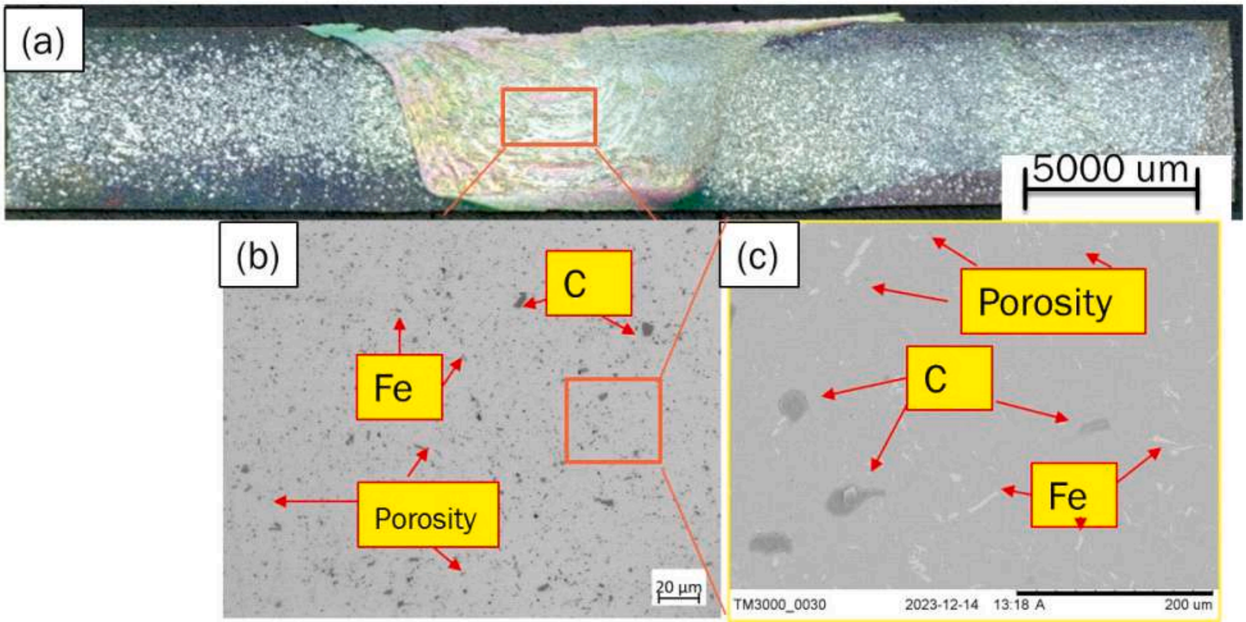

Fig. 2. (a) cross-sectional micrograph of one pass FSPed HPDC Al-4Mg-2Fe alloy sample (b) Optical micrographs illustrate the structure and morphology of Fe particles (c) SEM micrograph illustrating the as-cast microstructure of the surface region.

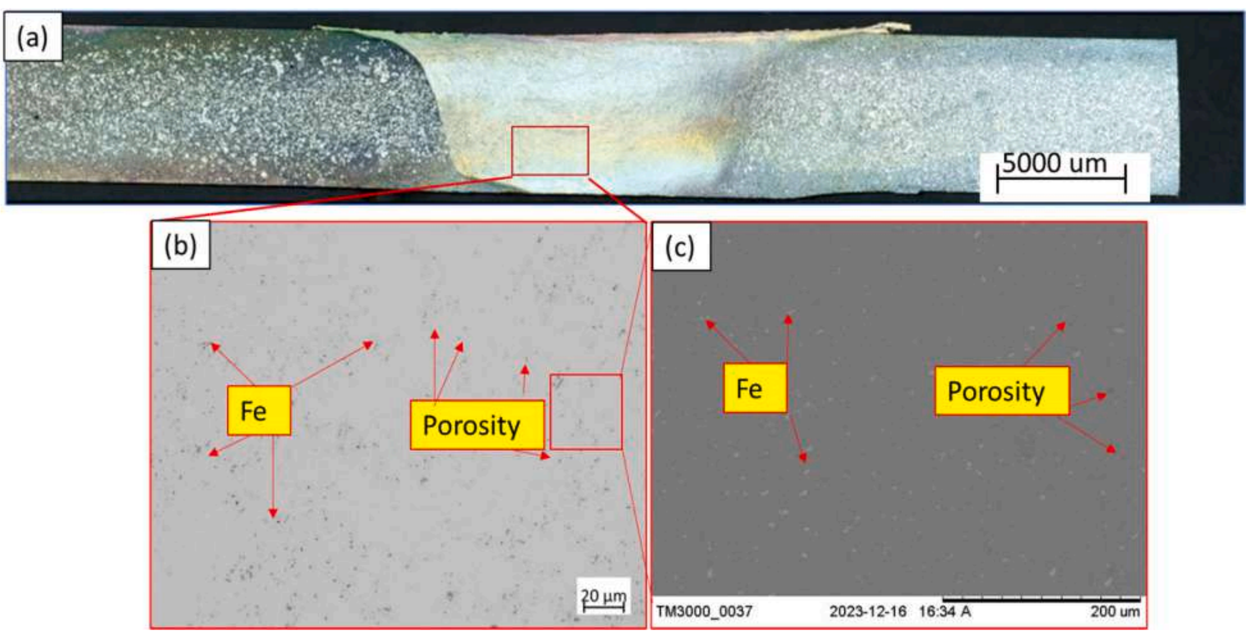

Fig. 3. (a) cross-sectional micrograph of two passes FSPed HPDC Al-4Mg-2Fe alloy (b) Optical micrographs illustrate the dendritic structure and morphology of Fe particles in (c) SEM micrograph illustrating the as-cast microstructure of the surface region.

in both the size and aspect ratio of Fe particles, as well as the porosity level. The decrease in Fe particle size is attributed to an augmented presence of smaller particles and a concurrent decrease in the quantity of larger particles. Thirdly, increasing the number of FSP passes with 100 % overlapping leads to a reduction in the Fe particle size and increases the count of small-sized Fe particles while eliminating porosity.

The characteristics of porosity were examined and quantified utilizing an ImageJ analyzer paired with an optical microscope. Polished samples were employed in this instance for the assessment of porosity parameters. Fig. 4 provides the optical micrographs of the samples used for porosity measurement. For HPDC Al-4Mg-2Fe all, the porosity volume fraction and aspect ratio values are 0.84 and 5.4 ± 2.4, respectively.

It was noted that the porosity at the die-wall of the HPDC sample was notably lower than that observed in the mid-wall regions of the HPDC Al-4Mg-2Fe sample. The number of pores within the Stir Zone (SZ) diminishes considerably compared to the base metal (BM) due to the stirring and forging actions of the FSP tools. As depicted in Fig. 4b, both the volume fraction and aspect ratio of porosity notably decrease after one pass of FSP.

The processed region displays markedly lower porosity compared to the adjacent, unaffected HPDC material. With an increase in the number of passes, there is a further reduction in porosity. FSP induces a significant breakdown of Si particles and their redistribution. The

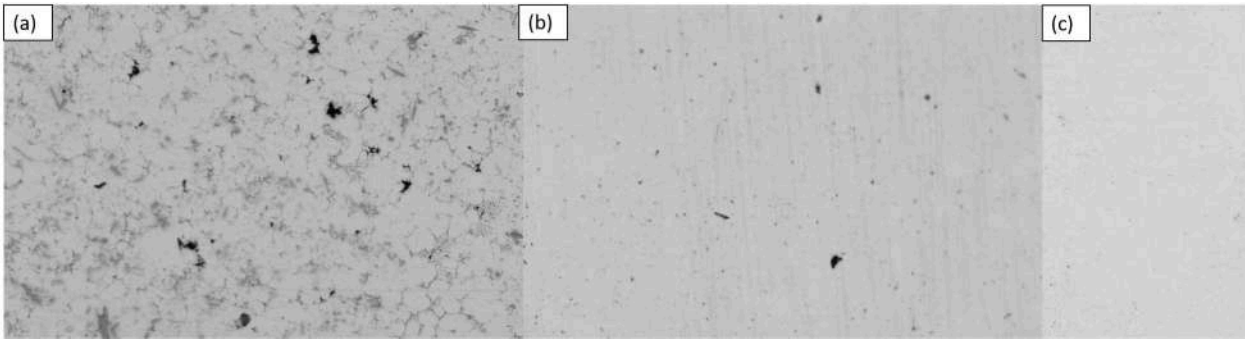

Fig. 4. Optical micrograph showing porosity and particle distribution in (a) as cast HPDC Al-4Mg-2Fe alloys (b) one pass FSP (c) two pass FSP sample.

conventional dendritic microstructure is absent, and the Si particles undergo considerable refinement after two passes, as shown in Fig. 4c, which presents a higher magnification image of the nugget, highlighting the extent of Si particle refinement after FSP. FSP results in more than a 50 % reduction in aspect ratio in particle size and shape, both in terms of mean and maximum values. From table 2 it was observed that the percentage of porosity and aspect ratio decreases as the number of passes increases. The continual increase in the number of passes in FSP leads to a reduction in the average size of Si and Fe particles. This phenomenon is ascribed to the mechanically induced fragmentation of particles occurring after each successive FSP pass. Likewise, the composite's porosity content decreases from 0.84 % to 0.05 % with an increase in the number of tool passes to two. A direct correlation is noted between the porosity content and grain sizes of FSP-processed Al-25Mg_2Si alloys. According to Barmouz et al. (Barmouz and Givi, 2011), an increased number of Friction Stir Processing (FSP) cycles/passes leads to enhanced interfacial bonding and densification. This result is ascribed to enhanced particle-matrix refinement and a decrease in the number of pores in the substrate composite. The findings of Barmouz suggest that large Mg_2Si particles are surrounded by numerous pores, leading to an elevated porosity level. This phenomenon can explain the decrease in the porosity content of HPDC Al-4mg-2Fe alloys as FSP passes increases.

Fig. 5 depicts the size of grain and structure of HPDC Al-4Mg-2Fe alloys highlighted using light microscopy. The initially received material (Fig. 5a) exhibited a conventional cast structure marked by floating crystals, a result of rapid cooling, embedded within the eutectic phase. At higher magnifications, sub grains were also visible within the grain boundary regions. The average size of grain was 40 μm for HPDC Al-4Mg-2Fe alloys. Each grain exhibited multiple secondary dendrites, characterized by a secondary dendritic arm spacing ranging between 20 and 30 μm. It is worth mentioning that it is an average grain size. Some grains were larger, with size of around 50–100 μm, depending on the location of measurement. A comprehensive examination of the transverse cross-sections shown in Fig. 5(b) reveals the outcome of the one-pass FSP on HPDC Al-4Mg-2Fe. The images illustrate the unique formation of an elliptical 'onion' structure at the specimen's center, marked by finely recrystallized and equiaxed grains. FSP, leveraging its heightened temperature and severe plastic deformation, initiates recrystallization, leading to grains with a distinctly different structure in comparison to the original cast material. FSP resulted in notable grain refinement within the nugget region due to extensive plastic deformation and frictional heating. FSP entirely disrupted the dendritic microstructure of the cast alloy. The majority of grains exhibited an equiaxed morphology, with occasional instances where some grains aligned parallel to either the advancing or retreating side. Significant grain size reduction is observed through two-pass FSP. The average grain size after one pass and two passes decreases to 7 μm and 4 μm, respectively. This is because increasing the number of passes in FSP promotes heightened dynamic recrystallization. This phenomenon is accountable for the augmentation of the new locations of nucleation, resulting in decrease in grain sizes within the stirred nugget zone. The impact of the number of passes on heat input during FSP of HPDC Al-4Mg-2Fe alloy is a pivotal factor in optimizing both the microstructure and mechanical properties of the end product. With an increasing number of passes in FSP, there is a corresponding elevation in cumulative heat input. In a two-pass scenario, examination of the results (refer to Fig. 5) reveals a consistent reduction in grain size from 7 to 4 μm after one pass and two passes, respectively. The refinement of grains in FSP specimens is attributed to the occurrence of discontinuous Dynamic Recrystallization (DRX), as evident from the obtained results. Notably, the investigation did not identify any grain coarsening after the completion of two passes. This absence of coarsening underscores the effectiveness of the FSP process in maintaining fine grains in the alloy. This phenomenon can be attributed to the presence of dispersed particles (Fe and Si) within the cast aluminum, which act as impediments to the movement of grain boundaries. Fig. 6 provides a visual representation of the average grain size comparison among the HPDC, one pass, and two passes FSP.

A comprehensive analysis have been conducted facilitated by SEM-EDS-based elemental mapping, can offers valuable insights into the extent of microstructural refinement achieved by FSP. Fig. 7 presents the results of EDS mapping analysis for various elements, including Al, Mg, Fe, and Si as well as their distribution before and after FSP. These maps revealed a non-uniform distribution of these elements before FSP (Fig. 7b, 7c, 7d, 7e). Following FSP, a significant improvement in the distribution of these alloying elements was observed, showing a visibly uniform distribution (Fig. 7g, 7h, 7i, 7j). The FSP tool induced plastic deformation in the matrix and mixed the different elements, promoting uniformity in the stir region. Optimal uniform distribution of dissolved elements was achieved through the two-pass FSP, refining the alloy (Fig. 7l, 7m, 7n, 7o). Iron-containing phases were dissolved and uniformly dispersed throughout the alloy. In particular, in Fig. 7(a), it is visible how the as-cast HPDC Al-4Mg-2Fe showcases features such as Al dendrites, Si structure, porosity, and needle-like Fe particles. Following a single pass of FSP, as depicted in Fig. 7(f), there is a noticeable transformation with fragmented and uniformly dispersed Si particles within the processing zone, effectively eliminating the dendritic microstructure seen in the as-cast state. Moreover, Fe particles undergo significant refinement, especially the needle-shaped particles known to adversely impact the tensile and fatigue properties of cast Al alloys. The coarse precipitates, predominantly consisting of Si and Fe components, take on a rod shape, while some larger precipitates in the spherical form

Table 2
Influence of FSP on Porosity volume fraction (vol.%) and aspect ratio of as cast HPDC Al-4Mg-2Fe alloys.

Specimen	Porosity (vol.%)	Aspect ratio
as-cast HPDC	0.84	5.4 ± 2.4
1-pass FSP	0.11	2.1 ± 1.1
2-pass FSP	0.05	1.7 ± 0.7

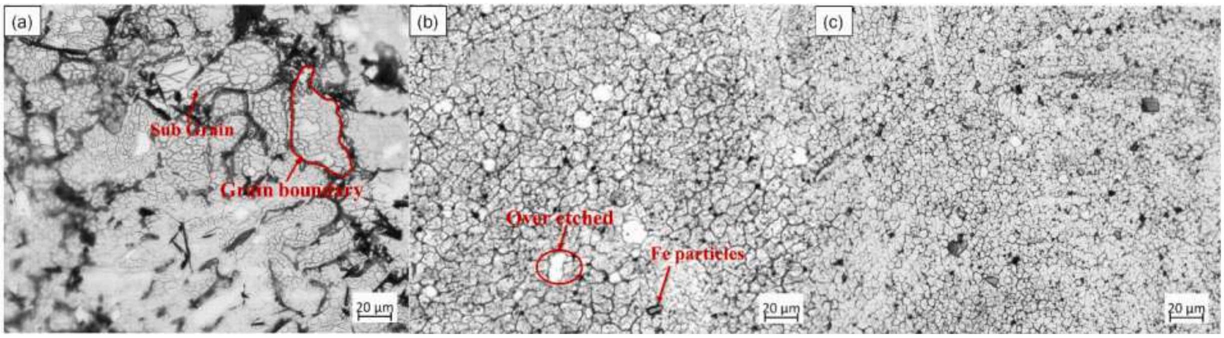

Fig. 5. Optical microstructure of (a) as received HPDC Al-4Mg-2Fe (b) one-pass FSP-grained refined structure (c) two-pass FSP-grained refined structure.

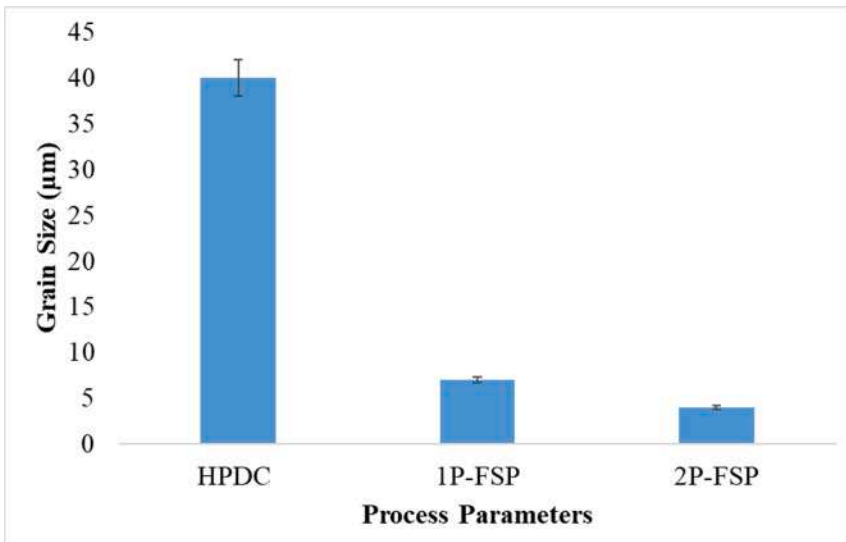

Fig. 6. Grain size comparison of HPDC, one pass and two passes FSP.

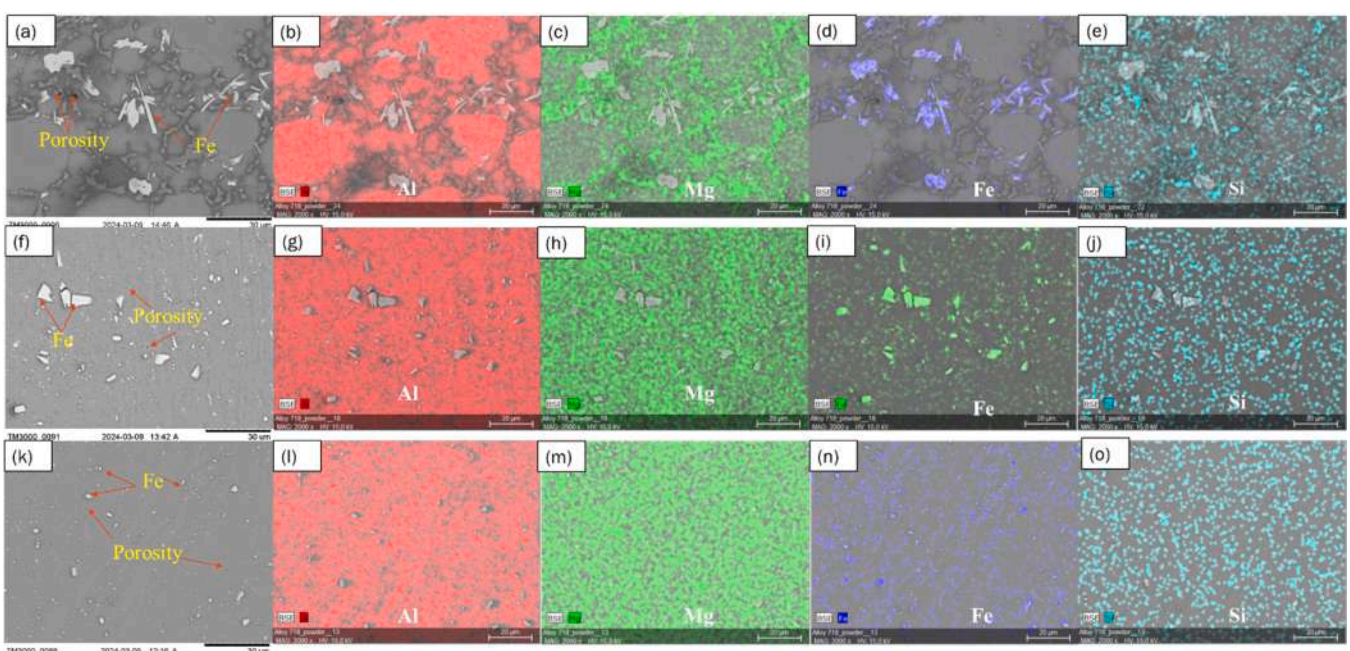

Fig. 7. SEM and EDS map comparison of (a-e) as received HPDC Al-4Mg-2Fe (f-j) 1-pass FSPed HPDC Al-4Mg-2Fe (k-o) 2-pass FSPed HPDC Al-4Mg-2Fe.

are composed solely of Al-Fe combinations. This indicates that the significant Fe particles in HPDC alloy undergo a transformation into smaller, rod-shaped particles through breakage during the Friction Stir Processing (FSP) process. Increasing the number of passes results in a more even distribution of Si and Fe second-phase particles, leading to a reduction in porosity in the region, as shown in Fig. 7(k). Moreover, the stirring action of the tool disintegrates the Si, Fe, and Cu phases in the as-cast HPDC Al-4Mg-2Fe alloy into smaller particles, enhancing the potential for diffusion into a solid solution at elevated temperatures. The flow of material induced by stirring also contributes to the dissolution of Fe into the Al matrix.

A few significant observations have come to light. Initially, the HPDC Al-4Mg-2Fe alloy displayed gas pores resulting from the casting process, which were effectively eliminated by FSP through intricate flow of material and plastic deformation. Before FSP, the microstructure of HPDC Al-4Mg-2Fe exhibited dendritic patterns Si particles in the Al-Si eutectic regions, Fe, and Cu-containing particles. FSP fragmented and evenly redistributed these phases, leading to the formation of a homogeneous wrought microstructure. The phases containing Fe and Cu experienced significant refinement, becoming fully enveloped by the Al matrix, indicating complete wetting of grain boundaries. FSP led to the comprehensive disintegration of dendrites, fragmentation of eutectic colonies, and refinement of Si phases. This alteration in microstructure, along with the elimination of porosity, was easily noticeable.

Fig. 8(a) and 8(b) report the results of the EDS analyses for various alloy conditions. It was observed that the chemical composition, as determined by EDS analysis of both the parent material and the stir zone, aligns with the specified requirements. Coarse precipitates are evident due to higher concentrations of Al, Cu, Mg, and Mn elements in homogenized alloys. Figs. 9(a) and 9(b) represent the EDS analysis and mass percentage at one pass FSPed of HPDC Al-4Mg-2Fe alloy while Figs. 10(a) and 10(b) represent the EDS analysis and mass percentage at two pass FSPed of HPDC Al-4Mg-2Fe alloy. By comparing the mass percentage graph, results shows that with the increment of number of passes the percentage of Fe and Si contents decreases. This can be attributed to the breaking down of eutectic large-size Si and Fe particles into small particles by FSP which uniformly mixes it into the aluminum matrix.

Hardness test

Fig. 11 depicts the Vickers hardness across the cross-sections of the considered specimens, including one pass, two passes, and the as-cast HPDC alloy. Continuous measurements were conducted at 0.5 mm intervals horizontally along a row of points situated approximately 0.8 mm below the surface. Additional measurements were taken in the second and third rows at the same intervals and depth. The average of these three rows was used in the results.

The results indicate an increase in the hardness of HPDC alloys with an increasing number of passes. The mean hardness values for HPDC, one-pass, and two-pass specimens are 69, 85, and 95 HV, respectively, indicating a 23 % increment in one pass and a 37 % increment in two passes. At some point, the hardness value exceeds 100 HV in the two-pass specimen. This observation is assigned to the enhanced fragmentation of Si and Fe particles and the refinement of the Al matrix grain structure previously observed in the composite with the increasing of number of FSP from one to two. The uniform dispersion of Fe particles and the strengthening effects on the matrix grain size, resulting from two pass FSP of HPDC alloys, contribute to the observed increase in hardness. In quantifying the fragmentation of particles, it was observed that the average area of dispersed particles decreased from 0.8 μm^2 in one pass to 0.28 μm^2 in two passes. This reduction in particle size may have contributed to the increase in hardness from 23 % to 37 %, as depicted in Fig. 7. Moreover, the notable reduction in grain size from 7 to 4 μm following one pass and two passes likely contributed significantly to the observed increase in hardness. The reduction in porosity content in the composites may also contribute to the enhanced hardness. The fine and evenly distributed Fe and Si particles in the two-pass FSP processed material are believed to hinder indentation loading and impede the movement of deformation/dislocations within the composite. Enhanced hardness is associated with the inhibition of grain boundary sliding (GBS), aligning with the Hall-Petch relation. The synergistic impact of high dislocation density caused by FSP, dynamic recrystallization leading to grain refinement, particle dispersion to micro size scale, and uniformity collectively contribute to enhanced hardness in the two-pass FSP treatment of HPDC alloy. These factors may be accountable for the observed enhancement in hardness in composites subjected to the two-pass FSP process. It is noteworthy that the hardness value near the upper surface of the FSPed sample is higher than the lower surface. During FSP, there is a significant thermal gradient within the nugget zone, with higher temperatures near the top and lower temperatures towards the bottom. The temperature variations can influence the kinetics of phase transformations and the evolution of microstructure, impacting hardness increment near the top surface. The top surface, in direct contact with the rotating FSP tool, undergoes higher levels of plastic deformation and thermal exposure. As a consequence, this surface tends to exhibit a more refined microstructure, potentially resulting in higher hardness due to increased dislocation density and grain refinement. Interestingly, during porosity measurement, it was noted that the porosity was significantly lower in the top region of the sample, closer to the die wall, in comparison to the middle region, which displayed higher porosity. Upon quantification it was observed that Volumatic porosity of as cast HPDC Al-4Mg-2Fe alloy at die wall region decreased to 0.53 ± 0.05 % compared to 0.84 ± 0.08 % in the middle wall region. Following a single pass of FSP, the volumetric porosity notably decreased to 0.073 ± 0.05 % in the die wall region. Remarkably, after two passes of FSP, the porosity in the die wall region was nearly eliminated. This correlation between porosity and hardness suggests that the higher hardness near the upper surface may indeed be a contributing factor to the observed low porosity levels in that region.

A detailed comparison of Vickers hardness reveals fluctuations in hardness values in the base material, ascribed to the existence of

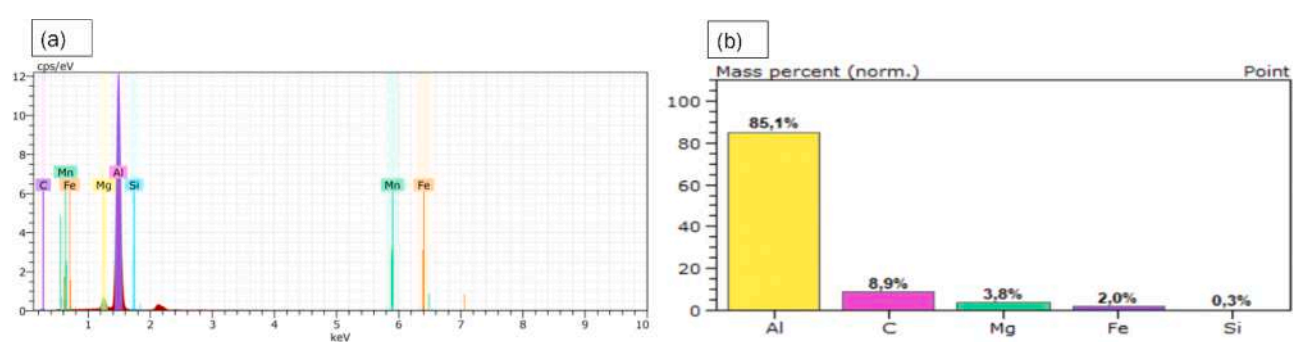

Fig. 8. (a) EDS analysis of as received HPDC Al-4Mg-2Fe alloy and (b) their mass percentage.

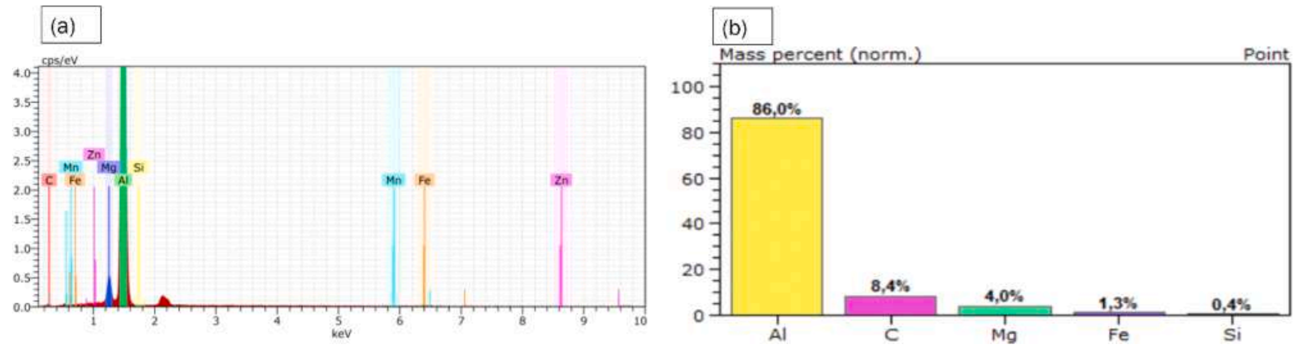

Fig. 9. (a) EDS analysis of as-received one pass FSPed HPDC Al-4Mg-2Fe and (b) their mass percentage.

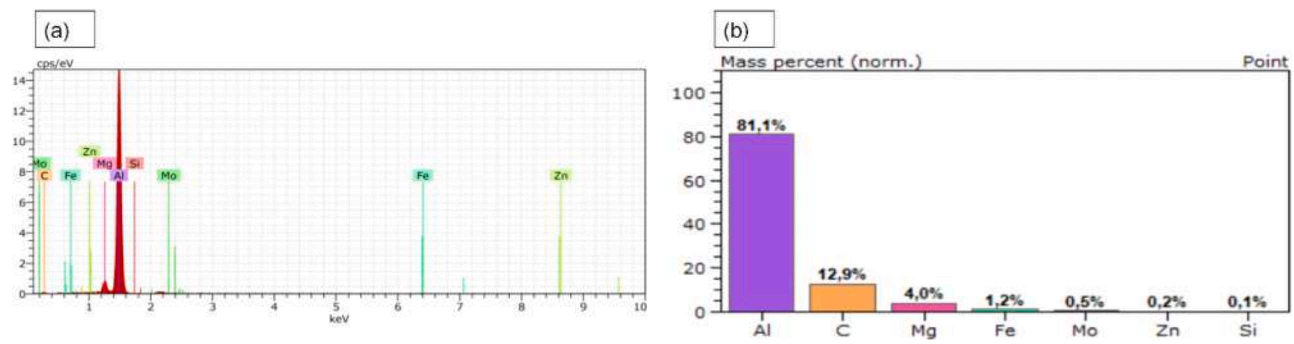

Fig. 10. (a) EDS analysis of as received two pass FSPed HPDC Al-4Mg-2Fe.and (b) their mass percentage.

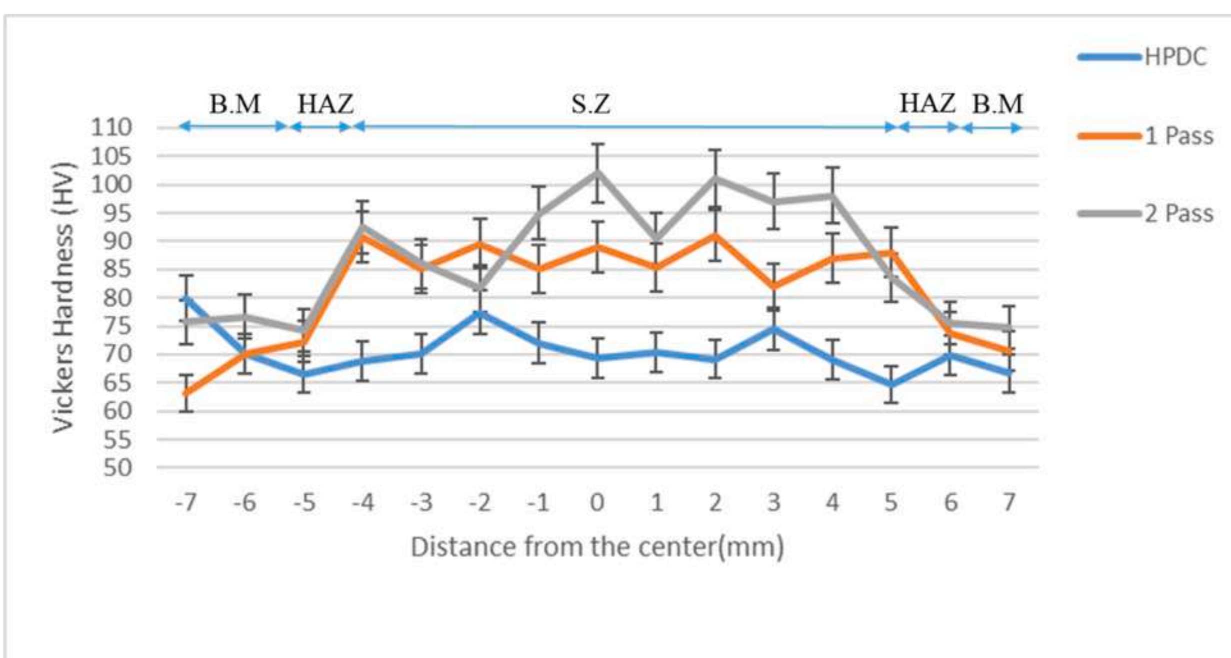

Fig. 11. Microhardness profile comparison of the HPDC Al-4Mg-2Fe and FSPed specimen.

porosity and the substantial size of Fe particles. The strengthening impact is more evident in the specimen subjected to a two-pass process compared to the one-pass, attributed to the existence of finer grains, complying to the material combining law, and increased hardness. The significant permanent strain and heat produced during FSP, combined with the tool's motion, fosters dynamic recrystallization, resulting in a well-organized grain structure and the mechanical breakdown of inherent grain barriers. Consequently, the alloy subjected to two-pass FSP exhibits a significantly refined grain structure in comparison to both the as-cast HPDC alloy and the alloy subjected to one-pass FSP. Therefore, the enhancement in hardness in the two-pass FSP-treated alloy stems from a combination of dispersion strengthening and a refined grain structure. The fluctuation of hardness in the S.Z of two passes have been observed. This fluctuation of hardness may be attributed to local grain size variation due to grain structure and presence of porosity at the measured point. Kumar et al. (Kumar et al., 2021) have observed the similar phenomena in fluctuations of hardness. Upon examining the fluctuating points in hardness, it became evident that the indentation at these particular points contains porosity.

Conclusion

The study investigates the impact of FSP on the mechanical and microstructural behavior of HPDC Al-4Mg-2Fe alloy, with the findings summarized as follows:

- The microstructure of as cast HPDC Al-4Mg-2Fe alloy comprises significant solidification porosity, aluminum dendrites, acicular silicon, and iron-rich particles.
- FSP induces substantial changes in the microstructure by minimizing porosity and disassembling aluminum dendrites. It also fragments needle-shaped silicon and iron-rich particles, along with other particulates, redistributing the fragmented particles consistently across the processed zone.
- The size of grains in the Stir Zone (SZ) experiences a significant reduction in one pass and two passes compared to base materials, indicating remarkable Dynamic Recrystallization (DRX) in this region.
- Increasing the number of passes results in a further decrease in the average grain size and the Fe-rich intermetallic, thereby extending the processed region.
- In comparison to the base material, the hardness at one pass and two passes of FSP increased by 23 % and 37 %, respectively. The enhanced hardness properties result from a combination of factors, including the elimination of porosity, microstructural refinement, homogenization of silicon, and second-phase particulates, along with precipitation strengthening induced by severe plastic deformation and thermal cycles during FSP.

CRediT authorship contribution statement

Muhammad Adnan: Writing – original draft. **Gianluca Buffa:** Visualization. **Livan Fratini:** Project administration. **Vivek Patel:** Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Mattias Igestrand:** Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

References

Aktarer, S.M., Sekban, D.M., Saray, O., Kucukomeroglu, T., Ma, Z.Y., Purcek, G., 2015. Effect of two-pass friction stir processing on the microstructure and mechanical properties of as-cast binary Al-12Si alloy. Materials Science and Engineering: A 636, 311–319. https://doi.org/10.1016/j.msea.2015.03.111. Jun.

Barmouz, M., Givi, M.K.B., 2011. Fabrication of in situ Cu/SiC composites using multi-pass friction stir processing: Evaluation of microstructural, porosity, mechanical and electrical behavior. Compos Part A Appl Sci Manuf 42 (10), 1445–1453. https://doi.org/10.1016/j.compositesa.2011.06.010. Oct.

Bates, W.P., et al., 2023. Properties Augmentation of Cast Hypereutectic Al–Si Alloy Through Friction Stir Processing. Metals and Materials International 29 (1), 215–228. https://doi.org/10.1007/s12540-022-01207-7. Jan.

Benedyk, J.C., 2010. Aluminum alloys for lightweight automotive structures," in Materials, Design and Manufacturing for Lightweight Vehicles. Elsevier Ltd 79–113. https://doi.org/10.1533/9781845697822.1.79.

Çamurlu, H.E., Ünal, N., 2011. Friction stir processing and characterisation of A380 cast aluminium alloy. International Journal of Cast Metals Research 24 (6), 357–362. https://doi.org/10.1179/1743133611Y.0000000008. Nov.

Cavaliere, P., De Marco, P.P., 2006. Effect of friction stir processing on mechanical and microstructural properties of AM60B Magnesium alloy. J Mater Sci 41 (11), 3459–3464. https://doi.org/10.1007/s10853-005-5601-9. Jun.

Ghio, E., Cerri, E., 2023. Corrosion Behaviour of High-Pressure Die-Cast AZ91 Alloy in NaCl Solution: Effects of Friction Stir Process at High Rotational Speed. Materials 16 (20). https://doi.org/10.3390/ma16206620. Oct.

Guru, P.R., Khan MD, F., Panigrahi, S.K., Ram, G.D.J., 2015. Enhancing strength, ductility and machinability of a Al-Si cast alloy by friction stir processing. J Manuf Process 18, 67–74. https://doi.org/10.1016/j.jmapro.2015.01.005. Apr.

Jana, S., Mishra, R.S., Baumann, J.A., Grant, G.J., 2010. Effect of friction stir processing on microstructure and tensile properties of an investment cast Al-7Si-0.6Mg alloy. Metall Mater Trans A Phys Metall Mater Sci 41 (10), 2507–2521. https://doi.org/10.1007/s11661-010-0324-1. Oct.

Karthikeyan, L., Senthilkumar, V.S., Balasubramanian, V., Arul, S., 2010. Analysis of first mode metal transfer in A413 cast aluminum alloy during friction stir processing. Mater Lett 64 (3), 301–304. https://doi.org/10.1016/j.matlet.2009.10.068. Feb.

Karthikeyan, L., Senthilkumar, V.S., Balasubramanian, V., Natarajan, S., 2009. Mechanical property and microstructural changes during friction stir processing of cast aluminum 2285 alloy. Mater Des 30 (6), 2237–2242. https://doi.org/10.1016/j.matdes.2008.09.006. Jun.

Kumar, A., Gotawala, N., Mishra, S., Shrivastava, A., 2021. Defects, microstructure and mechanical behaviour upon multi-pass friction stir processing of magnesium alloy with spiral tool path. CIRP J Manuf Sci Technol 32, 170–178. https://doi.org/10.1016/j.cirpj.2020.12.006. Jan.

Liu, C., et al., 2021. Characteristics of Fe-rich intermetallics compounds and their influence on the cracking behavior of a newly developed high-pressure die cast Al–4Mg–2Fe alloy. J Alloys Compd 854. https://doi.org/10.1016/j.jallcom.2020.157121. Feb.

Luo, X.C., et al., 2020. Enhancing mechanical properties of AZ61 magnesium alloy via friction stir processing: Effect of processing parameters. Materials Science and Engineering: A 797. https://doi.org/10.1016/j.msea.2020.139945. Oct.

Ma, Z.Y., Sharma, S.R., Mishra, R.S., 2006. Effect of friction stir processing on the microstructure of cast A356 aluminum. Materials Science and Engineering: A 433 (1–2), 269–278. https://doi.org/10.1016/j.msea.2006.06.099. Oct.

Ma, Z.Y., Sharma, S.R., Mishra, R.S., 2006. Effect of multiple-pass friction stir processing on microstructure and tensile properties of a cast aluminum-silicon alloy. Scr Mater 54 (9), 1623–1626. https://doi.org/10.1016/j.scriptamat.2006.01.010. May.

Mahmoud, T.S., Mohamed, S.S., 2012. Improvement of microstructural, mechanical and tribological characteristics of A413 cast Al alloys using friction stir processing. Materials Science and Engineering: A 558, 502–509. https://doi.org/10.1016/j.msea.2012.08.036. Dec.

Z.Y. Ma, S.R. Sharma, and R.S. Mishra, "Microstructural Modification of As-Cast Al-Si-Mg Alloy by Friction Stir Processing."2022.

R.S. Mishra, M.W. Mahoney, S.X. Mcfadden, N.A. Mara, and A.K. Mukherjee, "HIGH STRAIN RATE SUPERPLASTICITY IN A FRICTION STIR PROCESSED 7075 Al ALLOY," 2000. [Online]. Available: www.elsevier.com/locate/scriptamat.

Nakata, K., Kim, Y.G., Fujii, H., Tsumura, T., Komazaki, T., 2006. Improvement of mechanical properties of aluminum die casting alloy by multi-pass friction stir processing. Materials Science and Engineering: A 437 (2), 274–280. https://doi.org/10.1016/j.msea.2006.07.150. Nov.

Nelaturu, P., Jana, S., Mishra, R.S., Grant, G., Carlson, B.E., 2018. Influence of friction stir processing on the room temperature fatigue cracking mechanisms of A356 aluminum alloy. Materials Science and Engineering: A 716, 165–178. https://doi.org/10.1016/j.msea.2018.01.044. Feb.

X.P. Niu, B.H. Hu, I. Pinwill, and H. Li, "Vacuum assisted high pressure die casting of aluminium alloys."2022.

Outmani, I., Fouilland-Paille, L., Isselin, J., El Mansori, M., 2017. Effect of Si, Cu and processing parameters on Al-Si-Cu HPDC castings. J Mater Process Technol 249, 559–569. https://doi.org/10.1016/j.jmatprotec.2017.06.043. Nov.

Patel, V., et al., 2023. Robotic friction stir welding in lightweight battery assembly of extrusion-cast aluminium alloys. Journal of Advanced Joining Processes 8. https://doi.org/10.1016/j.jajp.2023.100156. Nov.

Rao, A.G., Deshmukh, V.P., Prabhu, N., Kashyap, B.P., 2015. Ductilizing of a brittle as-cast hypereutectic Al-Si alloy by friction stir processing. Mater Lett 159, 417–419. https://doi.org/10.1016/j.matlet.2015.07.006. Jul.

Rao, A.G., Katkar, V.A., Gunasekaran, G., Deshmukh, V.P., Prabhu, N., Kashyap, B.P., 2014. Effect of multipass friction stir processing on corrosion resistance of hypereutectic Al-30Si alloy. Corros Sci 83, 198–208. https://doi.org/10.1016/j.corsci.2014.02.013.

Samanta, A., et al., 2022. Microstructure-refinement–driven enhanced tensile properties of high-pressure die-cast A380 alloy through friction stir processing. J Manuf Process 78, 352–362. https://doi.org/10.1016/j.jmapro.2022.04.027. Jun.

Samanta, A., Lall, A., Das, H., Seffens, R.J., Grant, G.J., Jana, S., 2024. Enhanced toughness and tear resistance of thin-walled High-Pressure Die-Cast aluminum alloys through Friction stir processing. Mater Lett 357. https://doi.org/10.1016/j.matlet.2023.135752. Feb.

Santella, M.L., Engstrom, T., Storjohann, D., Pan, T.Y., 2005. Effects of friction stir processing on mechanical properties of the cast aluminum alloys A319 and A356. Scr Mater 53 (2), 201–206. https://doi.org/10.1016/j.scriptamat.2005.03.040. Jul.

N. Sun and D. Apelian, "Friction Stir Processing of Aluminum Cast Alloys for High Performance Applications."2022 [Online]. Available: www.tms.org/jom.html.
Sun, X., Cao, Z.Y., Liu, H.F., Jiang, W., Liu, L.P., 2014. Predicting the influence of microporosity on the mechanical properties and fracture behavior of high-pressure die-cast AM50 magnesium alloy. Applied Mechanics and Materials. Trans Tech Publications Ltd, pp. 90–94. https://doi.org/10.4028/www.scientific.net/AMM.670-671.90.
Wang, J., et al., 2021. Effect of Multi-pass Friction Stir Processing on Microstructures and Mechanical Behaviors of As-Cast 2A14 Aluminum Alloy. J Mater Eng Perform 30 (4), 3033–3043. https://doi.org/10.1007/s11665-021-05594-7. Apr.