



Analyzing the Effect of Fe on the Microstructure and Mechanical Properties of a Fibrous AA6110 Alloy

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Received: 3 October 2025 / Accepted: 15 December 2025
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Abstract

In this study, the effect of three different levels of iron impurity on the grain structure and texture of a fibrous AA6110 alloy after hot extrusion was investigated. Microstructural examination was carried out to measure the microtexture and the results were correlated with tensile and bending mechanical properties. Results demonstrated that the fragmented intermetallic particles after extrusion stimulate the nucleation of new grains, therefore refining the grain structure. The thickness of peripheral coarse grains was found to decrease by increasing Fe content due to increasing nano-sized dispersoid precipitation density. Increment Fe-rich intermetallics on the one hand leads to entrapment of Si solute reducing free Si needed for precipitation hardening shown via thermodynamic calculations. The bending formability was found to correlate with the grain structure of the PCG thickness. The results are compared with Part 1 of this research on the study of Fe content in recrystallized alloys. This work helps to a better understanding of recyclability in Al extrusion alloys.

Keywords Al alloys · Texture · Dynamic recrystallization · Fibre texture · Fe-based intermetallics

1 Introduction

The production of lightweight components through a combination of direct chill (DC) casting of aluminum alloys (AA) followed by hot extrusion has proven to be an effective process for both structural and transportation-related applications [1–3]. The Al–Mg–Si series have been frequently produced via hot extrusion methods due to the combination of good extrudability, surface finish, good corrosion resistance [4], and relatively high mechanical properties. These properties make them ideal for structural components and exterior panels in automotive applications, where their high

ductility plays a crucial role in absorbing energy during deformation events, such as vehicle collisions [2, 5]. The performance of these alloys is closely linked to microstructural characteristics like grain structure and crystallographic texture, which in turn are influenced by the alloy composition, production route, and processing parameters [6–11]. Goik et al. [12] demonstrated that increasing the extrusion speed during the production of hollow box profiles reduced the intensity of β -fibre texture components in both recrystallized and fibrous microstructures, while increasing the thickness of the peripheral coarse grain (PCG) zone and enhancing Cube recrystallization texture.

From the chemical composition point of view, the existence of Mg and Si as the main element additions leads to the formation of Mg_2Si precipitates at temperatures below the solvus after a supersaturated solid solution. Precipitation hardening mechanism is the main factor for tailoring the microstructure to obtain desired mechanical properties [13–15]. Other alloying elements also influence the final product properties. For instance, Mn, Cr, and Zr are mainly retained in solid solution after casting and precipitate during homogenization in the form of nano-sized precipitates, so-called dispersoids, preferably at the high-angle grain boundaries (HAGB) [16–20]. Dispersoids hinder grain boundary migration during deformation at elevated temperatures

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preventing grain growth. Using Mn to form dispersoids has been shown to be successful in increasing strength at elevated temperatures in wrought Al–Si series [21–24], Al–Mg [25, 26], and Al–Mg–Si series [19, 27–29]. Iron (Fe), on the other hand, tends to form various intermetallic compounds (IMCs) with Al, Si, and Mn during solidification [30–32], which cannot be dissolved in further heat treatment processes. These particles are detrimental to the mechanical properties of various product components [6, 33]. Analysis of the Al alloy die cast products has shown [34–36] a detrimental trend in ductility and tensile strength of the Al–Mg–Si alloys. However, die cast components have a near-cast structure where morphological aspect of needle-shape IMC particles play an important role.

In addition to the effect of dispersoid forming elements (DFE) [28, 37, 38], highly soluble alloying elements in a face-centred cubic (FCC) matrix as a solid solution can affect the stacking fault energy (SFE), therefore triggering different deformation mechanisms to accommodate deformation depending on the SFE level. In low SFE materials such as stainless steels [39–42], planar slip is favoured and this facilitates twinning. By contrast, in high SFE materials such as aluminium alloys, the material hardly forms twinning and dislocation cross-slip dominates instead [12, 43]. Whereas the effect of iron on AA6xxx alloys has been discussed widely in terms of solidification, phase composition and influence on precipitation hardening [44–46], its influence on grain structure and texture after industrial scale extrusion remains ambiguous. Goik et al. have studied the fibrous 6082 and 6005 alloys in terms of Fe addition and claimed that iron helps retain fibrous microstructure due to an increase in the dispersoids' fraction; although, it's not clear whether this effect is a result of Fe addition or DFEs (Mn and Cr) [12]. Despite the high Mn content, stronger recrystallization and grain growth was observed due to particle-stimulated nucleation (PSN) [44, 47]. The Fe-based IMCs, particularly α -Al(FeMnCr)Si, can influence the texture during thermomechanical processes. By the accumulation of deformation in the surroundings of IMCs, a set of newly equiaxed grains possessing a texture that is deflected from the standard one can occur [48]. Although PSN is a well-known phenomenon and reported in cold-rolled AA8xxx [49, 50] up to 0.7 wt%, hot-extruded AA7xxx [51],

rolled Al–Mg alloy [52], it has not been reported in extruded Al–Mg–Si profiles. Our observations of PSN helps to clarify the effects of Fe addition to microstructural and mechanical properties in extrusion industry of the Al–Mg–Si alloys.

Although AA6xxx series alloys are widely used and essential for promoting sustainability and reducing carbon footprints through recyclable materials, the impact of Fe as a single impurity in an industrial fibrous alloy after hot extrusion has not been thoroughly explored especially considering the effect of particle stimulated nucleation and subsequent consequences on grain structure, texture, and dynamic recrystallization. To address this, an AA6110 alloy exhibiting a fibrous grain structure was investigated. Another part of this research work focusing on the effect of Fe in recrystallized AA6008 alloy has been published elsewhere. While alloys with fibrous structure exhibit high strength, recrystallized alloys having dominant cube texture show high ductility mainly used for crush performance. We have reported a complex behaviour when additional Fe results in formation of more IMC compounds within the Al matrix [Under Review]. A comprehensive microstructural and mechanical investigation were carried out on the influence of Fe content in hot extruded profiles.

2 Materials and Methods

The AA6110 with a fibrous microstructure was used for this study. It should be noted that the effect of Fe on recrystallized AA6008 is currently under review elsewhere. Three variations of the AA6110, having Fe content of 0.2wt%, 0.3wt%, and 0.5 wt% were direct chill (DC) cast into billets, homogenized [53], and extruded into flat strips as shown schematically in Fig. 1 in an industrial scale. The aim is to keep all influential process parameters and chemical composition constant except for the level of Fe. The 0.2Fe, 0.3Fe, and 0.5Fe names will be used for identification of the alloys henceforth.

Firstly, the extruded samples were artificially aged to T6 temper according to suggested cycles from Ref. [54] to achieve desired tensile strength. Then, samples were cut from the profile as shown schematically in Fig. 1 for characterisation. Tensile and bending tests were then performed on an Instron universal tensile machine following the standard procedures according to BS EN ISO 6892-1 [55] and VDA 238-100 [56]. The bending test is performed in a 3-point bend test, where a sample supported on both sides is loaded at its centre point and bent to a specific angle or until the test sample fails. The average bending angle is then measured to report as a criterion of formability.

For microstructural examinations, the longitudinal cross-section of samples was prepared using standard

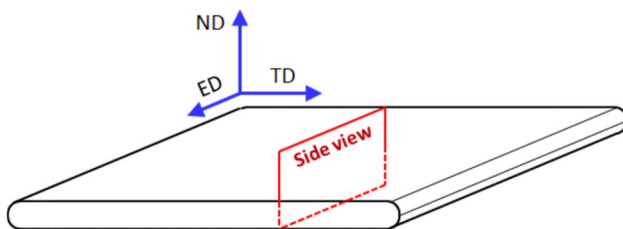


Fig. 1 Schematic representation of extruded profile and sampling region from the side view

metallographic procedures, polished to colloidal silica, followed by electropolishing using 30% nitric acid in methanol at $-30\text{ }^{\circ}\text{C}$ at 15 V. For EBSD analysis, a scanning electron microscope (SEM) (TESCAN Magna) was used at an accelerating voltage of 15 keV and 30 nA probe current to obtain electron back-scattered diffraction (EBSD) data as well as energy dispersive spectroscopy (EDS). Extraction of the EBSD results was carried out using Oxford Aztec v6.0 and data acquisition using Aztec Crystal v3.1. TEM was used for observation of Nano-sized dispersoid particles. Samples were prepared by grinding thin specimens down to 100 μm followed by punching a 3 mm disk and jet polishing via Struers TenuPol-5 with the 25% nitric acid in methanol at $-25\text{ }^{\circ}\text{C}$. The Talos F200i field emission transmission electron microscopy (FETEM) facility was used equipped with EDS Bruker. Thermodynamic calculations were performed using Pandat software and PanAl2021 database.

3 Results and Discussion

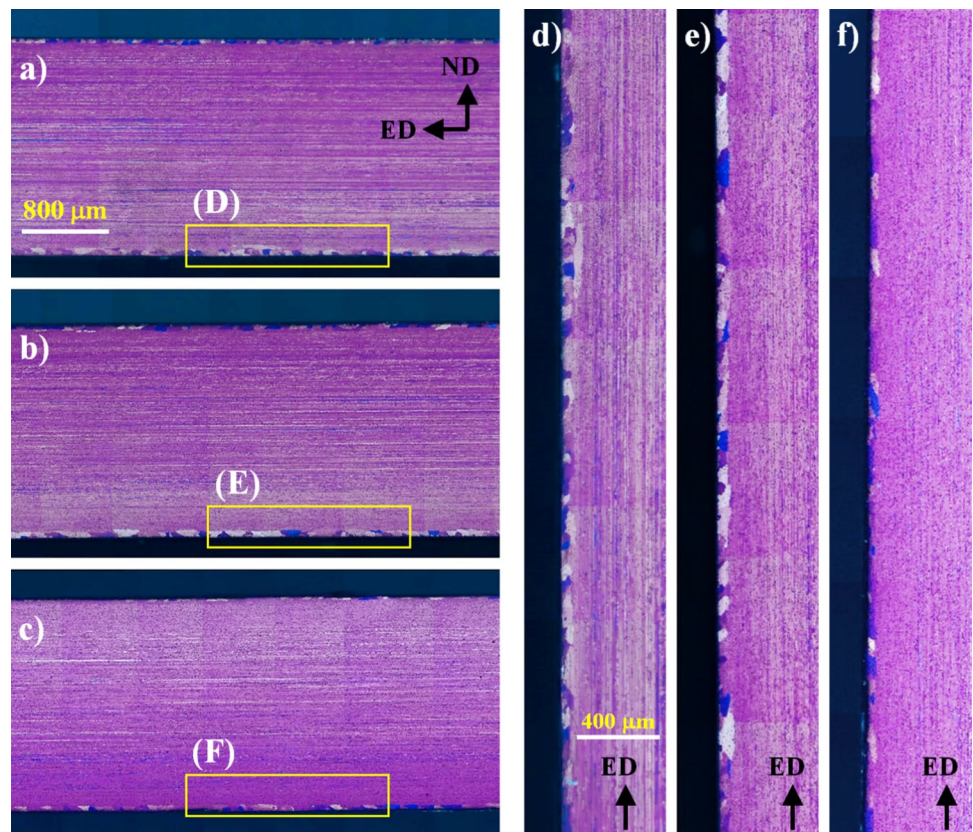
3.1 Effect of Fe on Microstructure

The overview picture of the profiles' microstructure after extrusion is shown in Fig. 2, where it shows fibrous grain structures. The effect of Fe on the peripheral coarse grains (PCG) depth can be seen straightaway. Quantification of

the PCG thickness in extruded alloys has been a common practice in industry [12, 57]. In contrast to the results of Hovden et al. [47], our present investigation of the alloy with a fibrous grain structure concluded that the addition of iron does not affect the fibrous microstructure. Moreover, the higher magnification images at certain points presented in Fig. 2d–f were utilized for analysis of PCG at different extrudate lengths. Statistical measurements show that the PCG depth in this alloy decreases with iron content from $137 \pm 30\text{ }\mu\text{m}$ for 0.2Fe, to $106 \pm 17\text{ }\mu\text{m}$ for 0.3Fe, and to $64 \pm 10\text{ }\mu\text{m}$ for 0.5Fe, respectively. The mechanism of PCG thickness reduction by increasing Fe content will be further discussed in the following section.

The formation of peripheral coarse grains at the extruded profile's surface [57–59] is influenced by chemical composition as well as process parameters. Goik et al. found that increasing Fe+Mn+Cr content resulted in a reduction in PCG thickness [12], but the effect of single iron addition was not studied. Apart from the effect of process parameters, it has been reported that the existence of Mn as a dispersoid-forming element in an AA6082 alloy [28] results in precipitation of dispersoids increasing Zener-drag pressure and preventing recrystallization. Based on optical microscopy observations, one can conclude that the recrystallized alloys do not change their nature due to an increase in iron as the latter does not form pinning particles and Mn content remains rather low. Similarly, increased iron content does

Fig. 2 Optical microscopy images of **a** 0.2Fe, **b** 0.3Fe and **c** 0.5Fe alloys extrudate profiles all at same magnification along with zoomed images for PCG clarification at profile peripheries



not cause any significant PSN effect in fibrous alloys, which means that the dispersoid's pinning pressure still helps to limit grain boundary migration. For both cases, EBSD analysis will help to quantify more precisely the differences in grain size and their orientations, which are determining factors for mechanical properties.

The effect of iron on the area fraction of IMC particles was investigated by quantitative metallography on the BSE images taken at the centre of profile thickness presented in Fig. 3. Visual inspection clearly shows an increasing trend in the Fe-based IMC particles along the extrusion direction due to an extremely low solubility of Fe in Al [60]. Due to a high contrast difference between Al matrix (dark grey) and Fe-IMC particles (white particles) in SEM-BSE images, quantifying the distribution of phases is feasible via image analysis. Thus, 3 images were taken for each condition and image analysis was performed using the ImageJ software to analyze the particles and corresponding area fraction histogram for each sample.

In this alloy, existing iron will contribute in two ways. In addition to the formation of Fe-IMC particles during solidification, secondary Nano-sized dispersoid particles also form during high-temperature homogenization treatment due to the presence of Mn in fibrous AA6110. The size, distribution, and number density of dispersoid particles depend on the process parameters and chemical composition. By studying the effect of Mn and Zr addition to AA6082, Elasheri [20] et al. have reported that dispersoid precipitation can effectively reduce PCG thickness layer and high retention of strong deformation texture. Osterreicher et al. [61] have studied the effect of process parameters (*i.e.* cooling and heating rate as well as homogenization temperature) on the microstructural evolution of an AA6xxx series alloy. Since the dispersoid particles prevent nucleation by pinning grain boundaries as explained by the Zener drag pressure effect [48, 57], it can be inferred that at the same processing condition, an increase in density of dispersoid particles results in reduction of PCG thickness. The difference between the size, distribution, and composition of the Fe-based IMCs and dispersoid particles were measured from high-angle

annular dark field (HAADF) scanning transmission electron microscopy bright field (STEM-BF) representative images of alloys in different Fe contents presented in Fig. 4.

A quantification of both Fe-containing IMCs and dispersoid precipitates comparison was carried out and plotted in Fig. 5 obtained from an average of 7 images at each condition. The IMCs were analyzed via BSE-SEM images (one sample image is shown in Fig. 3) and dispersoids were analyzed from HAADF-STEM images (one sample image is presented in Fig. 4). According to Fig. 5a, the area fraction of IMC particles increases by increasing Fe content; but the circularity remains the same. Minoda et al. have reported a similar trend in increasing size and number density of Fe-based IMC particles by increasing Fe content [62]. Due to the complexity of Fe-based IMC formation during solidification, and its inability to dissolve during homogenization treatment, and processing [63], the circularity of the IMCs does not change by increasing Fe content. The quantitative particle analysis of dispersoids depicted in Fig. 5b shows a similar trend in number density increment by increasing Fe content in this alloy.

To estimate the evolution of phases and composition by changing Fe content of the alloy, thermodynamic calculations were performed and presented in Fig. 6. The fibrous alloy, which contains high Mn content, shows a simple phase composition-temperature relationship and only $\alpha\text{-AlFe}_2\text{Si}_2$ can form (Fig. 6b). As shown in the phase fraction calculations in Fig. 6b with an increase in Fe content, the total fraction of Fe-based IMCs increases sharply, while the Si solute in the (Al) matrix decreases. This is noteworthy to mention that the Si in Al matrix is the available free Si dissolved in Al after some Si content is being consumed by Fe-based IMCs. This affects the precipitation hardening response which will be discussed later. Conclusively, it can be derived that the addition of Fe has contributed to the addition of Fe-rich IMCs as well as dispersoid precipitations when comparing thermodynamic calculations (Fig. 6) with experimental area fraction measurements (Fig. 5).

To evaluate the crystalline orientations corresponding to the grain structure in each investigated condition, EBSD



Fig. 3 SEM/BSE images taken at the same magnification at **a** 0.2Fe, **b** 0.3Fe, and **c** 0.5Fe content accompanied by histogram of corresponding area fraction

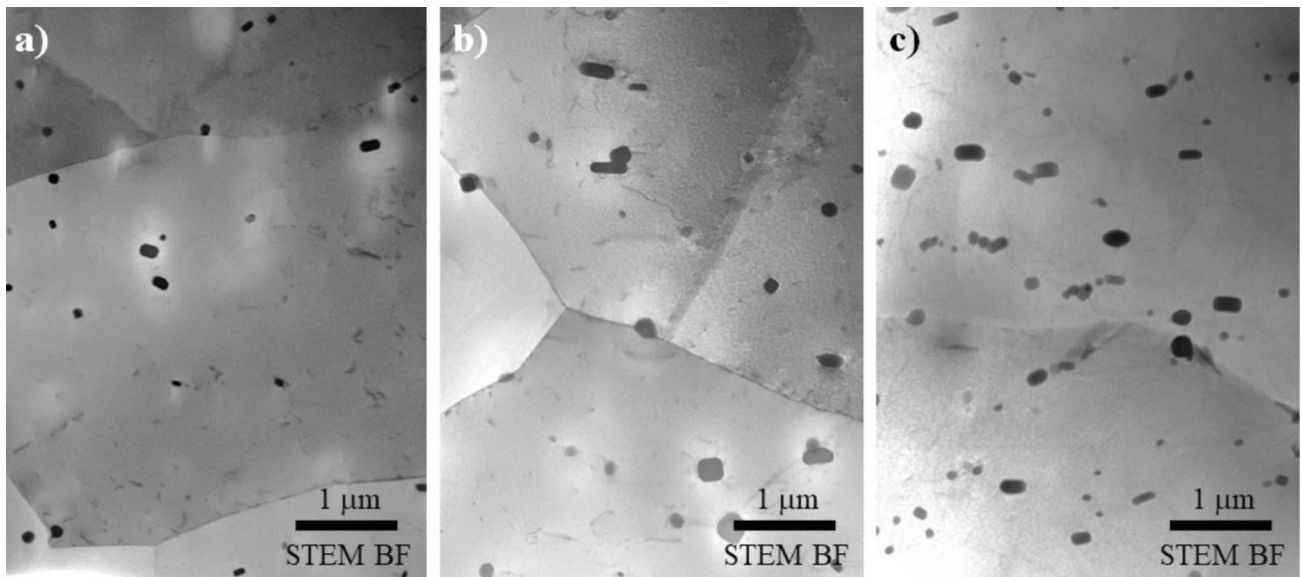


Fig. 4 STEM-BF images obtained from dispersoids precipitation taken from **a** 0.2Fe, **b** 0.3Fe, and **c** 0.5Fe alloys

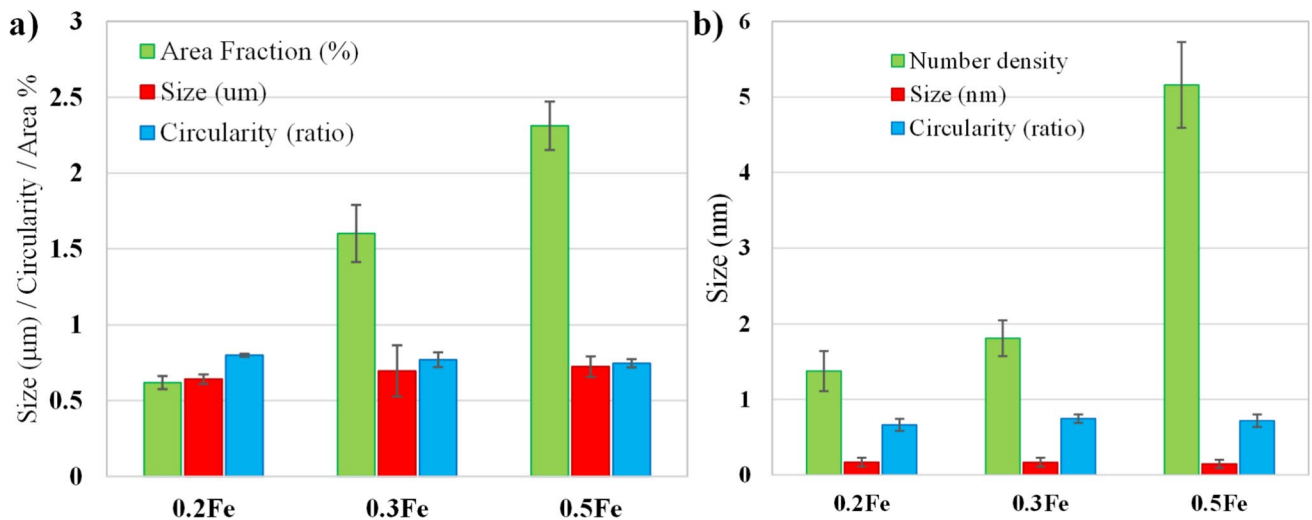


Fig. 5 Quantification of **a** Fe-based IMC particles Area fraction, size, and circularity analyzed from BSE images and **b** Number density (count per μm^2), size (nm), and circularity of nano-sized dispersoid precipitates analyzed from STEM HAADF images at various Fe content

analysis was performed over the whole cross-sections of all samples at a randomly selected area as shown in inverse pole figure (IPF) maps of the X-axis (Extrusion Direction—ED) in Fig. 7. It is noticeable that the grain structure consists of a peripheral recrystallized coarse grain (PCG) at the surface, a subsurface fibrous grain structure (denoted by green color), and central fibrous region (purple combination) (Fig. 7a–c). A fibrous grain structure at the centre of profile thickness possesses a combination of 4 texture components known as β -fibre. For fibrous alloys, the influence of Fe is not straightforward from the images, therefore complementing the analysis with the fraction of texture components will be performed.

To further discuss the evolution of texture for each specific region of the longitudinal cross-section (Fig. 8), a representative orientation distribution function (ODF) map was obtained and presented in Fig. 6 at 0.2Fe conditions. For the quantification of texture components analysis of ODF maps was carried out by the FCC texture components that are summarized in Table 1.

According to the ODF data, the centre of thickness follows a β -fibre texture where all components of Brass (Fig. 8a at ϕ_2 0°), Copper (Fig. 8a ϕ_2 45°) and S (Fig. 8a ϕ_2 60°) contribute to the central fibrous region. The recrystallization Cube component appears to be present as shown in Fig. 8a ϕ_2 0° which has also been reported by Goik et al. in fibrous AA6082 [12]. At the subsurface parts of the alloy,

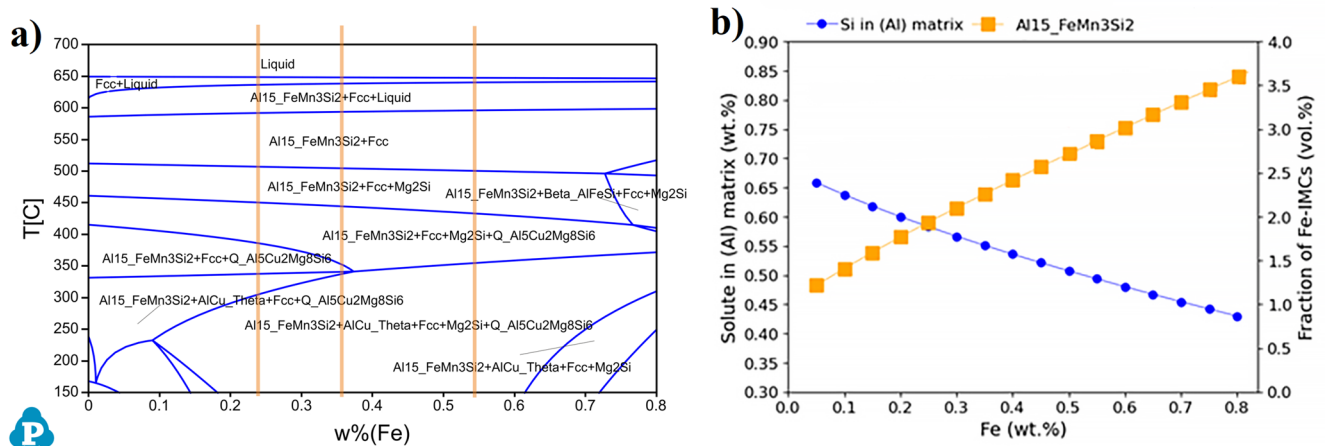


Fig. 6 Thermodynamic **a** pseudo-binary phase diagram calculations versus Fe content with **b** solute Si and Fe-based IMCs phase fraction calculations by changing Fe content

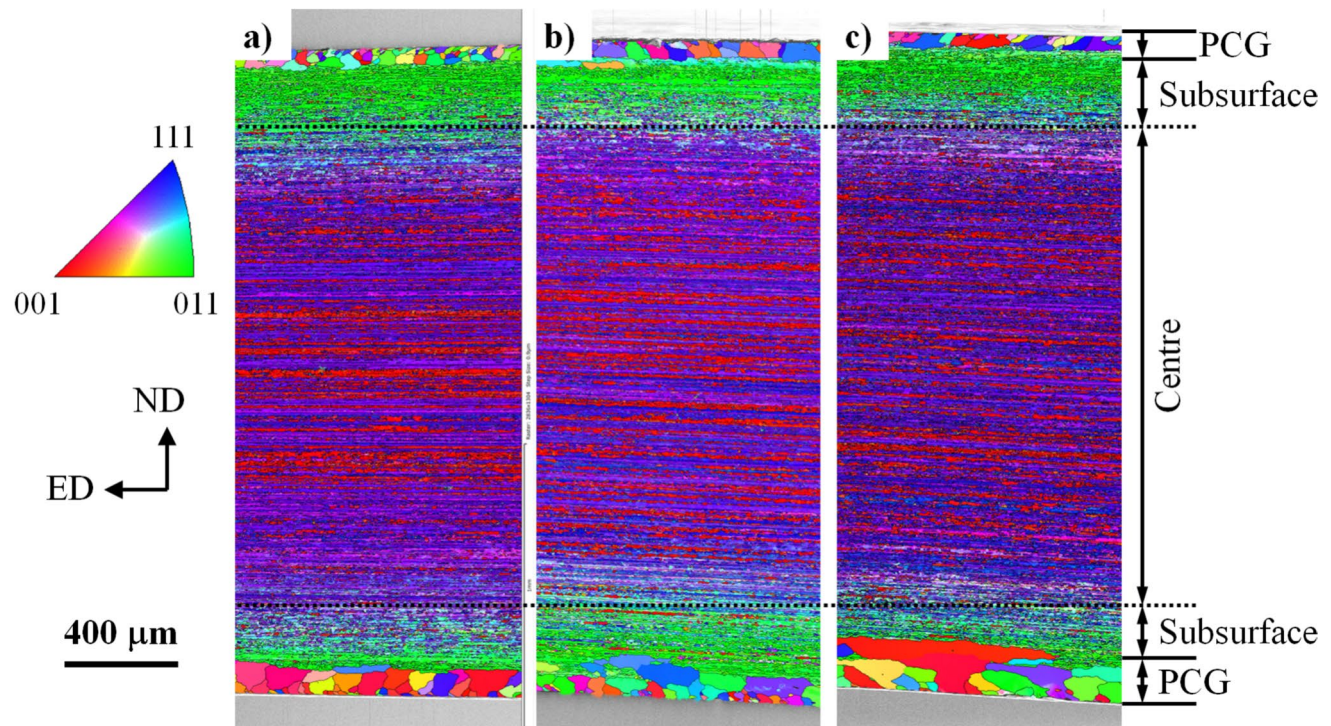


Fig. 7 EBSD IPF maps at **a** 0.2Fe, **b** 0.3Fe, and **c** 0.5Fe taken from the side of the profiles where the extrusion direction is parallel to the X-axis. All images are at same magnification

the β -fibre texture disappears gradually, and B-fibre starts to appear as shown in Fig. 8b ϕ_2 45°. The PCG at the surface of the extrudate also has a component of the Goss and Goss/Brass (Fig. 8c).

To explain the effect of iron addition on the microstructure and properties, each of the component fractions was calculated based on the texture components obtained from the EBSD maps (Fig. 7) and plotted in Fig. 9. It is evident that all components of the β -fibre contribute to the fibrous region at the centre of the extruded profile considering that

the distribution of each component depends on the activation of slip systems during deformation. Hirsch has stated that in aluminium alloys containing very low amount of constituent elements, the stacking fault energy is relatively high, and the twinning rarely takes place [64]. From the perspective of alloying elements, Cu addition in aluminium has been shown to increase SFE while Mg reduces the SFE as in the AA5xxx series [41, 42, 65]. However, in the present study the individual additions of Mg, Si and Cu elements are less than those in AA5xxx series. The addition of Fe up to

Fig. 8 ODF maps calculated from each part of EBSD maps shown in Fig. 7 at **a** centre of thickness, **b** subsurface, and **c** PCG surface

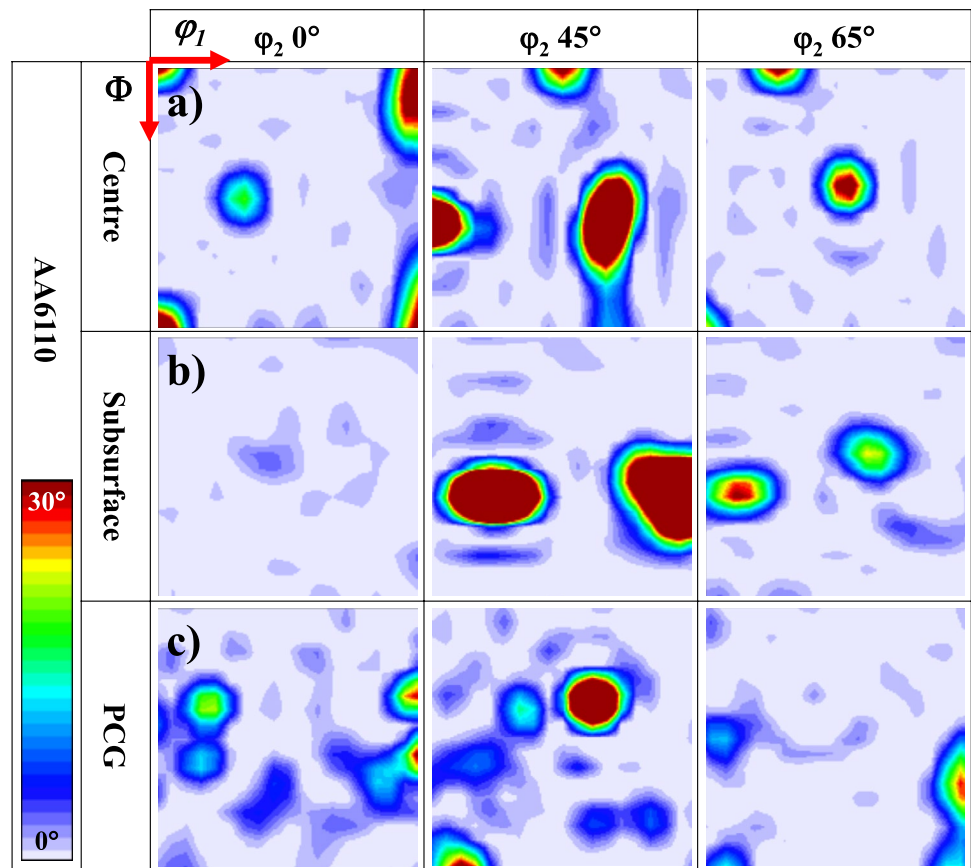


Table 1 Various texture component names with respect to each corresponding Euler Angles

Texture Component		Orientation	Euler angles		
			ϕ_1	Φ	ϕ_2
Recrystallization fibre	Cube	$\{001\} \parallel \langle 100 \rangle$; $\langle 100 \rangle \parallel ED$	0/90	0	0/90
	β -fibre				
β -fibre	Goss	$\{011\} \parallel \langle 100 \rangle$	0	45	0/90
	Brass	$\{011\} \parallel \langle 211 \rangle$	35	45	0/90
			54	90	45
	S	$\{123\} \parallel \langle 634 \rangle$	59	37	63
			52	74	33
			27	57	18
	Copper	$\{112\} \parallel \langle 111 \rangle$	90	35	45
			39	65	26
	B-fibre	$\{\bar{1}12\} \parallel \langle 110 \rangle$; $\langle 110 \rangle \parallel ED$	0	55	45
	$\bar{B}$	$\{1\bar{1}2\} \parallel \langle \bar{1}10 \rangle$	60	55	45

the level of 0.7 wt% is believed not to change the SFE due to poor solubility and the aluminium is still the dominant element determining the SFE.

Grain size refinement is another consequence of Fe addition as shown in Fig. 10a as the area fraction of large grains (over 200 μm) is less frequent by increasing Fe content (e.g. 0.5Fe). The area-weighted average grain size data was

measured from EBSD results based on ASTM E2627 considering $HAGB \geq 10^\circ$. Based on the average grain size values in Fig. 10, it is evident that the grain refinement has occurred in the centre part. The mechanism of grain refinement via iron IMCs has been explained by Wang et al. [66]. They mentioned that particles having a diameter of more than 1 μm , mostly $\alpha\text{-Al(Fe,Mn)Si}$ IMCs, have the potential to act as substrates for recrystallization utilizing PSN [47]. One should point out the difference between dispersoids in pinning GBs with IMCs in grain refinement. According to Nes [67], the wrought microstructure contains two dominating structural particles within the matrix: the bimodal coarse particles (i.e. micro-sized Al(Fe,Mn)Si IMCs and coarse Mg_2Si particles; and fine particles (i.e. nano-sized Al(Fe,Mn)Si and Mg_2Si precipitates). The micro-sized particles promote recrystallization through PSN that are recognised as the increment of the stored energy in the deformation zone around them. Conversely, the nano-sized particles impede recrystallization process by exerting a Zener pinning force [28, 68]. The Zener pinning force offsets the driving force of recrystallization while stored energy because of plastic deformation enhances the recrystallization. This balance determines the microstructure of the material after deformation.

The grain refinement as a result of Fe addition in hot extrusion of the AA6110 fibrous alloy is an indication of

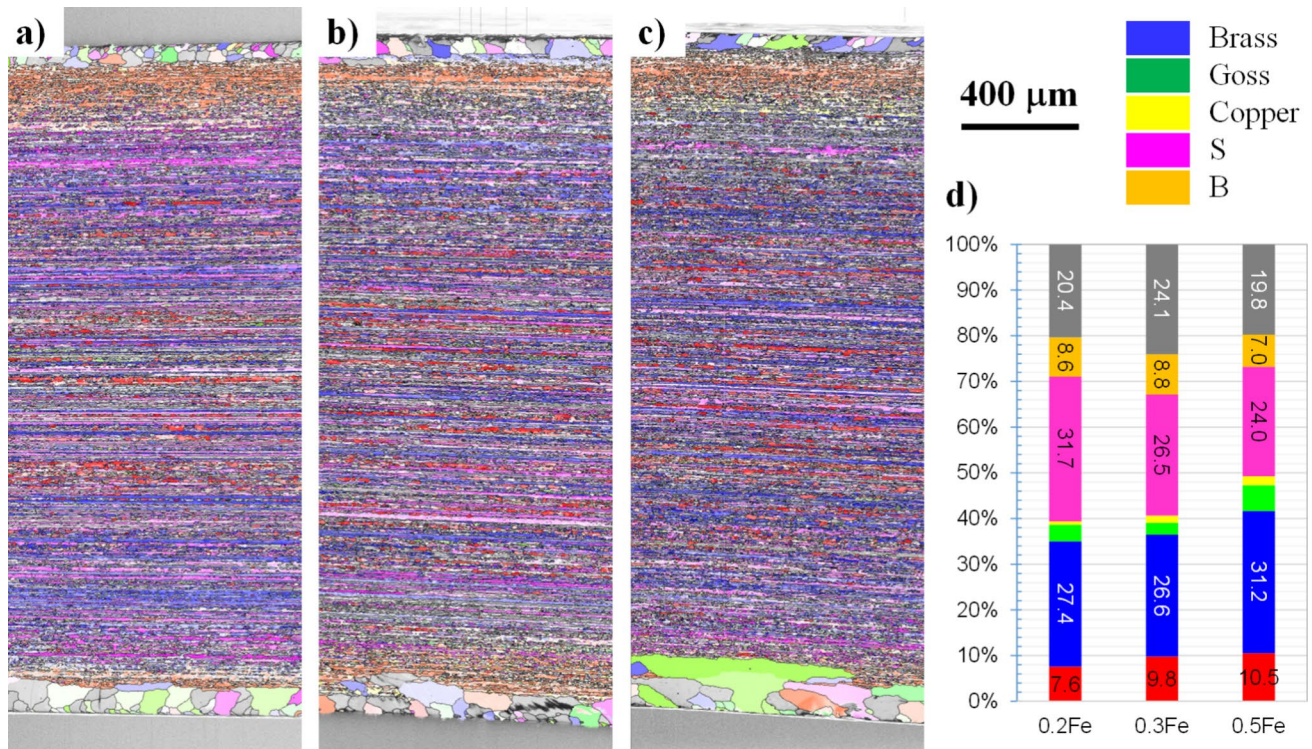


Fig. 9 Texture components distribution map obtained from EBSD data at **a** 0.2Fe, **b** 0.3Fe, **c** 0.5Fe with **d**) quantitative fraction. All images have the same magnification

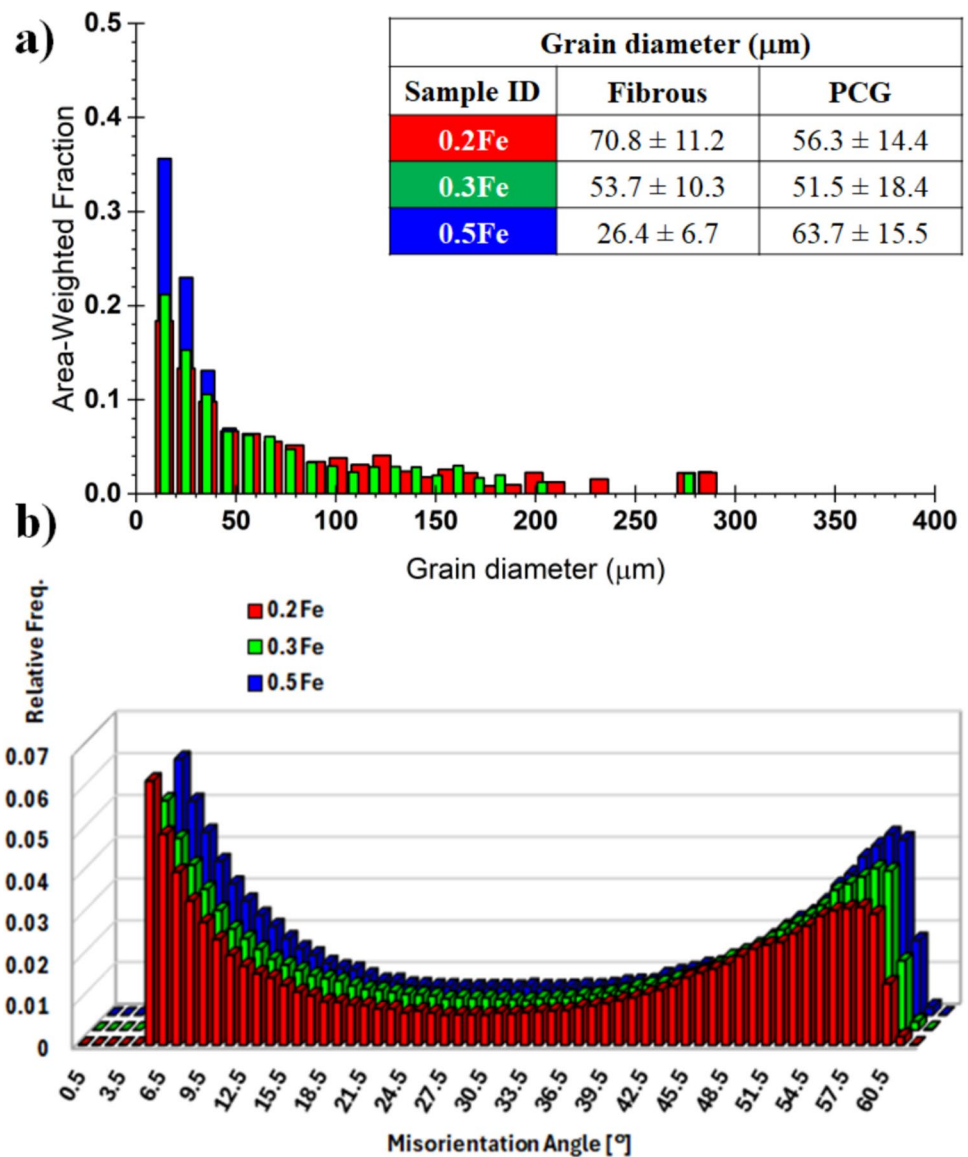
manipulation of dynamic recrystallization during high temperature deformation. This was further elaborated via looking into the misorientation angle distribution obtained from the fibrous region of Fig. 7 and plotted in Fig. 10b. It can be inferred that the misorientation angle frequency of all samples has two distinctly separated peaks at low angle (5°) and at high angle (58°). The HAGBs are showing and increasing frequency by increasing Fe content (from 0.2Fe-red to 0.3Fe-green and 0.5Fe-blue) which is an indication of increasing higher fraction of HAGB leading to a finer grain size. This is an indication of facilitating recrystallization through PSN which will be discussed further.

B-fibre is reported to take place in severe plastic deformation (SPD) processes such as equal channel angular pressing (ECAP) and high-pressure torsion (HPT) [12, 69–72]. Here, in the extrusion process, the friction due to the contact between the die and the material during the extrusion process results in the shear deformation of the Al matrix at the contact zone with the extrusion die. Based on experimental results and finite element modelling, Mahmoodkhani et al. have studied the surface grain structure during extrusion. The authors concluded that the shear component can be controlled via die geometry management [73–75]. The formation of B texture shown in EBSD IPF map of Fig. 11a as a result of die shear has a direct correlation with the formation of equiaxed PCG grains. In fibrous alloys, this can

be prevented by using a choke bearing at the die bearing. Similar observations were separately reported by Goik et al. [12] and Chen et al. [71]. According to Negrozio et al. [57] the mechanism of PCG occurrence depends on simultaneous existence of high DRX, high temperature, and high strain rate. Wherever satisfied (mostly at the surface where shear strain exists and temperature exceeds rapidly), the driving force for recrystallization surpasses the Zener pressure retarding force and equiaxed grain nucleate and grows. If conditions are met, Goik et al. have demonstrated [12] that the equiaxed PCG grains tend to consume all B texture shear grains from a crystallographic viewpoint. As indicated by black arrows in Fig. 11b–d, there's a close correlation between $\{110\}$ planes of PCG and shear textured grains). Thus, the reason that prevents PCG grain growth lies in the inhibition of grain boundary migration because of increasing number density of dispersoid precipitation as shown in Figs. 4 and 5b.

The presence of Fe-rich IMCs has an impact on grain structure by decreasing grain size in the bulk structure as reported earlier in Fig. 10. To observe the effect of Fe-based IMCs on grain refinement, a simultaneous EBSD/EDS map was obtained from 0.5Fe condition in the fibrous region, as shown in Fig. 12. Enrichment of Fe, Si, and Mn (Fig. 12c–e) accompany the presence of IMCs (red colour areas in Fig. 12a). According to the IPF map in Fig. 12b, the IMC

Fig. 10 Statistical **a** grain size in quantitative numeric with average weighted fraction and **b** misorientation angle distribution obtained from EBSD data in Fig. 7 fibrous region



particles are surrounded by tiny grains within 2–5 μm size range, which have an orientation different from the typical $\langle 111 \rangle$ and $\langle 001 \rangle$ seen in fibrous microstructures, as indicated by yellow arrows. Dynamic recrystallization was reported to take place during high-temperature extrusion of an Al–Mg–Si alloy as presented by Zhang et al. [7]. Therefore, recrystallization due to PSN can also occur by dynamically after high residual strain is being imposed to the surrounding matrix by IMC particles [76–79].

3.2 Effect of Fe on Mechanical Properties

The tensile properties shown in Fig. 13a, c show a slight reduction in yield strength (TYS) as well as ultimate tensile strength (UTS) by increasing iron content. The effect of iron in AA6xxx series has already been well-established [16]. On one side, it has a high tendency to consume Si by forming β

(in cast structure) and α -AlFeSi (after homogenisation treatment) IMCs. Since Si provides a strengthening effect in both its solid solution and through the formation of Mg_2Si precipitates, reducing free Si will result in a reduction of precipitation hardening. This was proven via thermodynamic phase fraction calculation in Fig. 6b. Additionally, the formation of IMC particles has been said to affect mechanical properties by resisting deformation while the surrounding matrix deforms easily. The result is the formation of cracks from the broken IMC particles [34, 44, 80, 81].

Bendability versus tensile properties is a valuable comparison for assessing the ductility of an engineering material for automotive applications. Since the bendability of the material depends on the surface characteristics of the grains, PCG thickness and texture components of the peripheral grains are important. In AA6110 having a hybrid grain structure (e.g. fibrous and recrystallized PCG), various

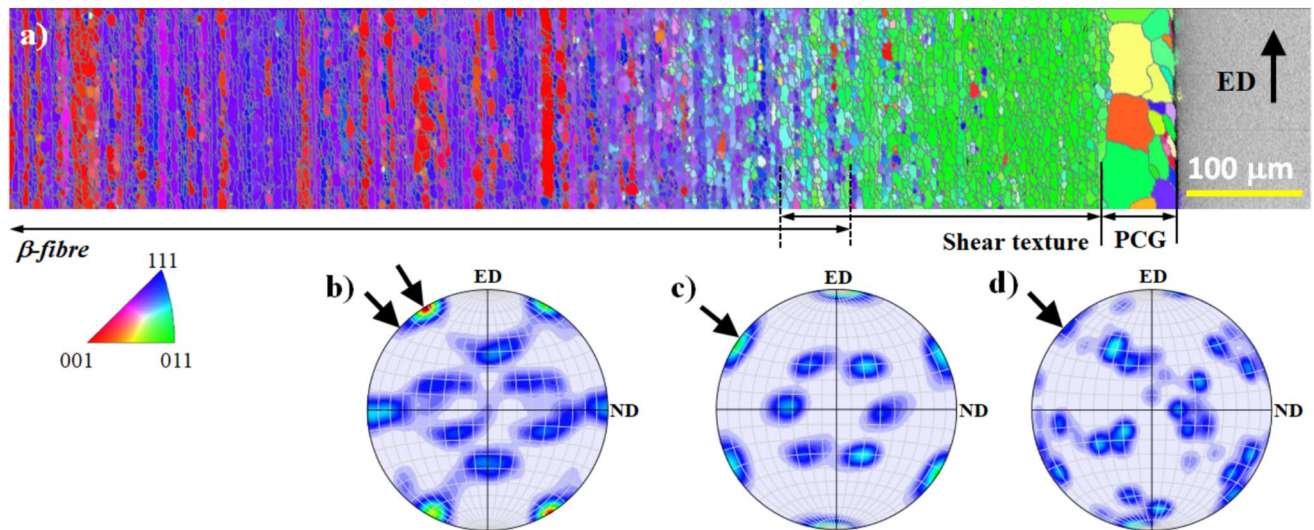


Fig. 11 a) EBSD IPF map of surface to the centre of thickness distinguishing three layers of equiaxed PCG, fibrous shear texture at the subsurface accompanied by corresponding PF of b) β -fibre, c) B shear texture, and d) PCG gradually emerging toward the centre of thickness

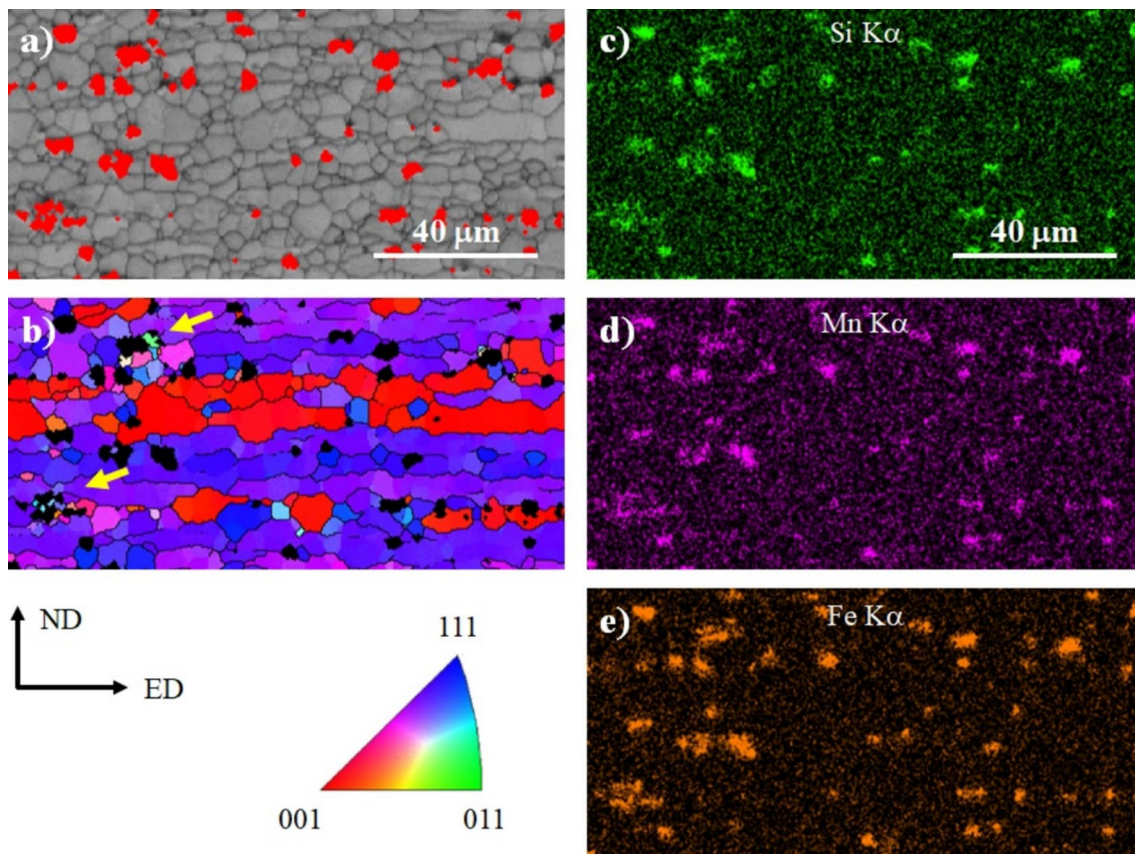


Fig. 12 Observation of grain refinement around the Fe-based IMCs via EBSD a) Phase map and b) IPF on Al matrix excluding IMC particles and EDS c) Si $K\alpha$, d) Mn $K\alpha$, and e) Fe $K\alpha$ mapping

parameters affect the bendability of the material such as PCG thickness, hardening behavior, anisotropy, and alignment of IMC particles [82]. Our observations on Fig. 13b show that by increasing the Fe content, the bending angle of

the material increases slightly while experiencing a tensile strength decrease. As presented in Fig. 2, increasing Fe content reduces PCG thickness. The mechanism for mechanical properties dependence to increasing Fe content can be

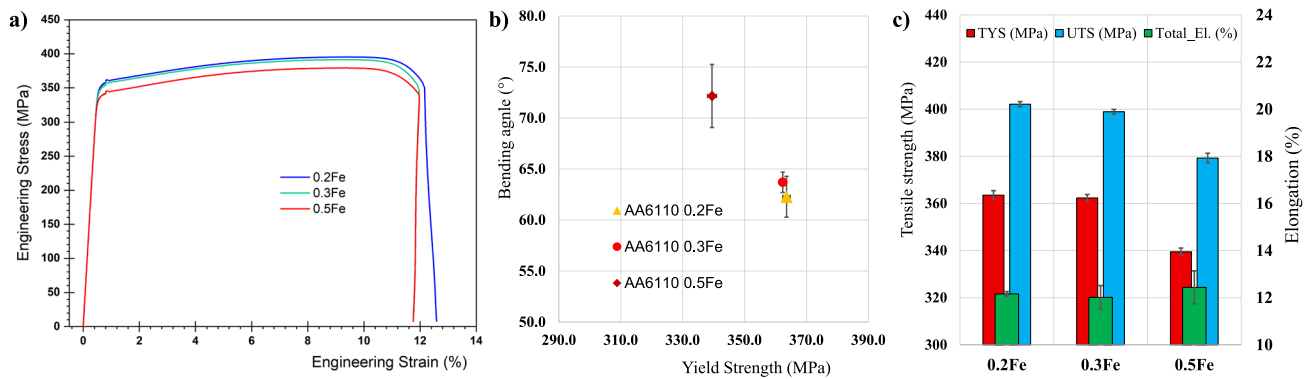


Fig. 13 Result of mechanical **a** tensile yield, ultimate strength, and total elongation and **b** bending angle versus tensile yield strength having different Fe contents

attributed to different factors. Firstly, an enhanced level of α -Al(FeMn)Si IMCs by increasing iron as shown in Fig. 3, quantitatively measured in Fig. 5a, and thermodynamically calculated in Fig. 6. The IMC particles consume more Si resulting in less strengthening precipitates formation (*i.e.* Mg_2Si) [62, 63]. Secondly, the addition of iron not only increases the number density of IMCs but also enhances the precipitation of nano-sized dispersoid particles as presented in Fig. 4 and quantitatively measured in Fig. 5b. The dispersoid particles prevent grain migration by increasing Zener pressure proposed by Zener [83] and explained by Liu et al. [84]. The result is inhibition of recrystallization and grain growth from the peripheral grains that were demonstrated on EBSD IPF map of Fig. 11. Conclusively, the PCG thickness reduces by increasing Fe content. Thirdly, Elasheri et al. [82] have elucidated the relationship between surface characteristics of AA6xxx extrudate profiles such as PCG thickness and bending properties and concluded that the thickness of the PCG layer has a direct correlation with bending angle. Due to the undesirable recrystallization texture that was observed at peripheral grains (as shown in Fig. 9), cannot tolerate tension and compression on either side of the surface during bending resulting in loss of ductility and formation of crack that propagates through thickness.

Engineering stress–strain curves have shown a profound plastic deformation prior to fracture in all alloys (Fig. 13a). after UTS, the localized necking has led to a reduction in thickness followed by a swift propagation of crack. Assuming that all samples follow the same fracture mode, one example of fracture surface at 0.5Fe was observed under electron microscope for further examination and presented in Fig. 14. From Fig. 14a, we can witness a 45° inclination of fracture surface with respect to the applied load direction. This is because although tensile load was applied horizontally, the material experiences maximum shear on the planes at $\pm 45^\circ$ activated by the FCC slip system [85, 86].

At the periphery, intergranular (IG) fracture was observed where crack follows grain boundary regardless of IMC trace path as shown in Fig. 14c, f. The reason was explained to be due to the GB decohesion. At the central fibrous region; however, the IMC network dominates the path of crack tip growth as shown on the cross-section at Fig. 14b and top fracture surface in Fig. 14e.

Further analysis was performed on the bending samples using optical microscopy on the anodized samples. Figure 15 shows the overall grain structure of the bent samples. It's evident that the deformation has led to the curvature of fibrous during deformation. The cracking that has sheared through the fibers can also be observed in 0.5Fe condition (Fig. 15c).

To further analyze the microstructure of bending tested samples, one sample from maximum Fe content was analyzed by low magnification EBSD to observe the grain structure in Fig. 16. Band contrast (BC) image (Fig. 16a) clearly demonstrates the abundance of deformation at both the compression and tension sides of the bent samples. Shear band formation is the response of the material to relaxing after further deformation [87]. The accumulation of shear bands followed by further applying deformation is the most suitable position for crack growth and failure [82]. In addition, the grain boundaries possessing IMC particles are also prone to void formation as a result of boundary decohesion [88]. This might be the reason for the start of crack formation and propagation from the coarse surface grains (PCG) in the form of intergranular fracture.

A comparison of residual strains from the GND map in Fig. 16c clearly justifies that the fibrous structure is capable of withstanding deformation by distributing the applied stress throughout the whole microstructure. Furthermore, the crystallography of each grain with respect to applied stress is responsible for activation of slip system to further tolerate the deformation. Comparing GND maps in Fig. 16 and IPF maps in Fig. 7, it can be inferred that the PCG

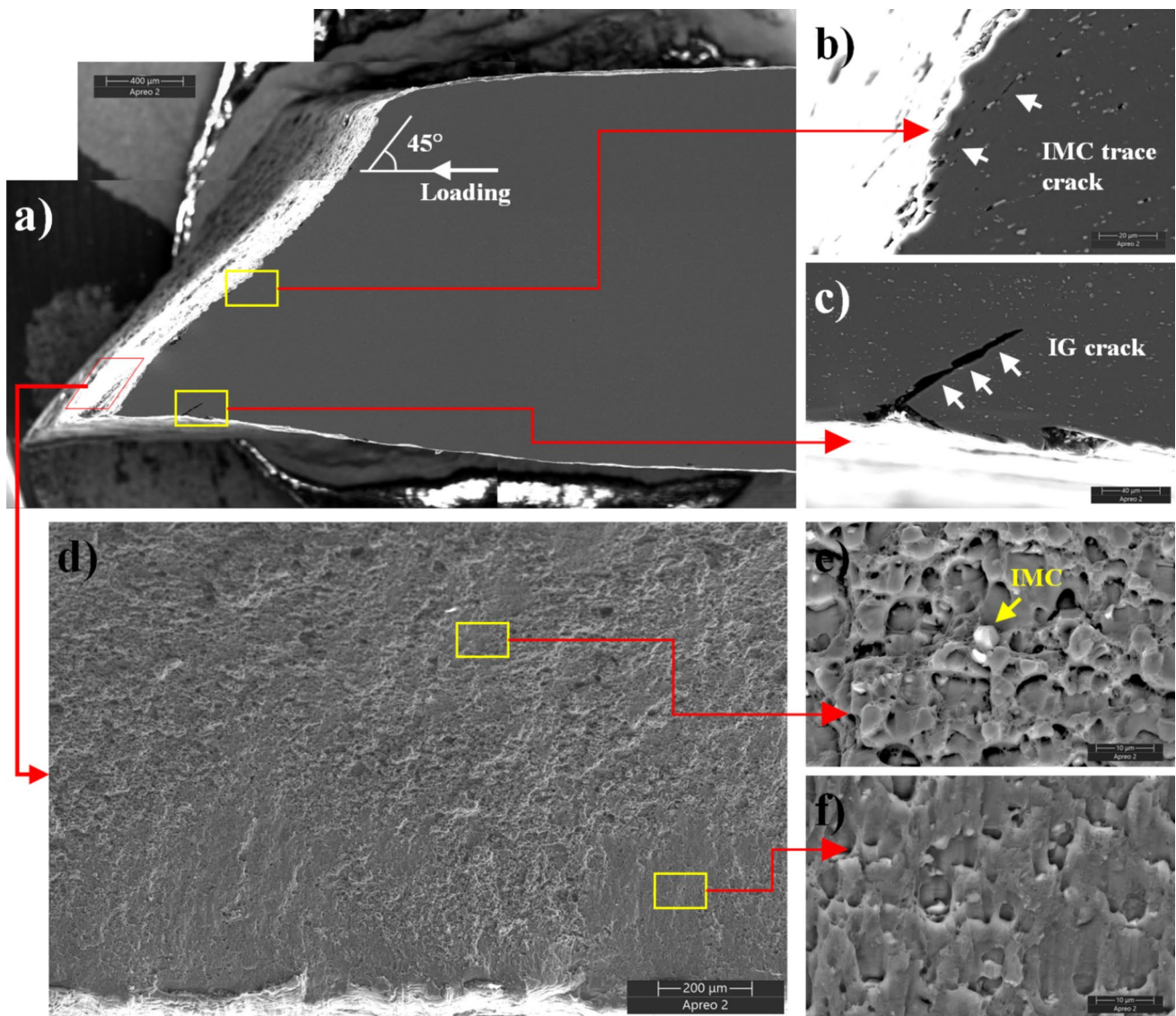


Fig. 14 Fracture surface analysis of the 0.5Fe tensile tested sample from **a, b, c** cross-section side view and **d, e, f** top fracture surface

grains are less tolerant to applying deformation relative to the fibrous structure acting as an initiation of failure during bending.

In conclusion, Fe impurity addition to AA6110 extrusion alloys can lead to a set of complex effects. It is obvious that the entrapment of part of the Si content in IMC particles leads to a decrease in precipitation hardening. Increasing iron content reduces PCG thickness which affects bendability of the alloy. The IMC particles also act as nucleation site for newly recrystallized small grains having a texture far from β -fibre. When comparing recrystallized alloys with fibrous alloys, one can conclude that the nature of Cube texture has been known to have lower strength and high ductility. The Fe addition in recrystallized AA6008 has increased the total IMC content. PSN has led to grain refinement in equi-axed grain structure, too. From the perspective

of mechanical properties, a reduction in tensile properties have been similarly observed in recrystallized alloy, too. On the other hand, bendability of the recrystallized alloys has observed to decrease by increasing Fe content. It was found that the reason is due to the modification of the PCG layer in recrystallized alloys (proved by EBSD texture components) where intergranular crack nucleates from the surface and propagates towards the thickness. Whereas the bendability was slightly improved in fibrous alloys mainly due to a decrease in thickness of PCG layer where crack is nucleated from the surface but hinders in fibrous region.

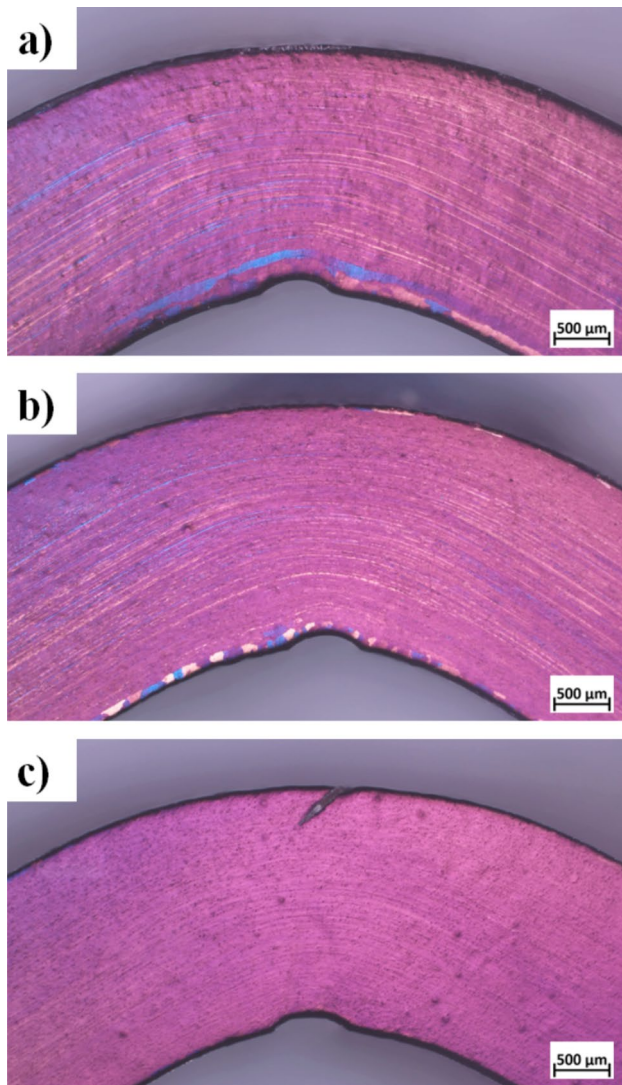


Fig. 15 Optical microscopy anodized images of bent samples at **a** 0.2Fe, **b** 0.3Fe, and **c** 0.5Fe Fe content

4 Conclusions

In this paper, the effect of single impurity Fe addition on the AA6110 fibrous alloy was investigated from the viewpoint of microstructural and mechanical properties. The extruded flat strip profiles were analyzed by optical and electron microscopy before and after bending tests and following conclusions were made:

- The Fe addition increases the area fraction and size of the α -Al(Fe,Mn)Si intermetallic particles while it does not affect their morphology proven by thermodynamic calculation and image analysis.
- The β -fibre texture is dominant in the fibrous region accompanied by a layer of B-fibre shear texture at subsurface and PCG grains at the surface.

- The thickness of peripheral coarse grains (PCG) is found to decrease by increasing Fe content in this alloy due to increasing the fraction of nano-sized dispersoid particles which inhibit grain growth.
- Increased Fe content increases the Fe-based IMC area fraction and number density of dispersoid precipitates while reducing the free solute Si. However, it does not alter the tensile strength and bending properties drastically. Thus, the alloy is capable of maintaining the required properties despite additional Fe content.

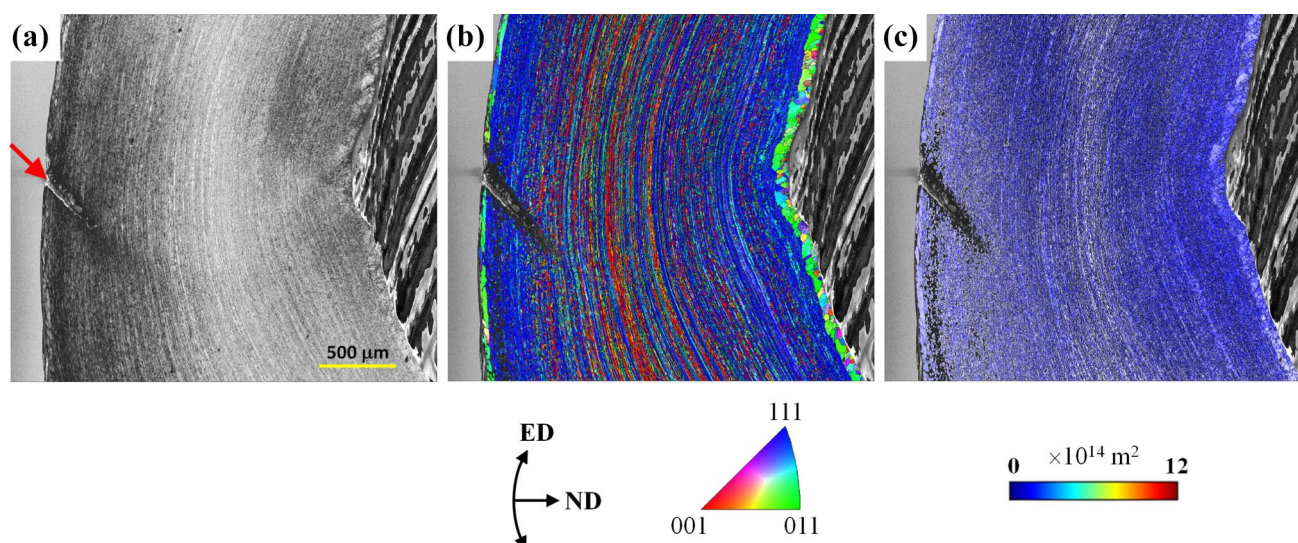


Fig. 16 EBSD low magnification **a** BC, **b** IPF at TD (Z-axis), and **c** GND maps of bending tested sample at 0.5Fe condition

Acknowledgements The author would like to appreciate help and support from Constellium, UK.

Funding This work was not supported by any project.

Data Availability Data is available upon a reasonable request from the corresponding author.

Declarations

Competing interests 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.

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