Quantitative Microstructural Analysis of Direct Chill Casting of Aluminium Alloy by use of QEMSCAN

Satya Prakash Mohapatra[1] and Jagdish Arora[2]
1. General Manager R&D
2. Director P&T
National Aluminium Company, Bhubaneswar, India
Corresponding author: satya.mohapatra@nalcoindia.co.in
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Abstract

Materials characterization provides comprehensive solutions for understanding the intrinsic properties and behaviours of various materials. Quantitative microstructural analysis is crucial in elucidating the fundamental aspects of metal forming, thereby aiding in the design and optimization of materials for specific applications. In the present article, we analyse the direct chill casting of an aluminium alloy using an advanced characterization technique known as QEMSCAN (Quantitative Evaluation of Minerals by Scanning Electron Microscopy). This technique allows for a detailed examination of the microstructural features formed during the direct chill casting process and their subsequent impact on the mechanical and physical properties of the final product. The analysis reveals the formation of unique microstructural characteristics, such as grain size and distribution, which significantly influence the performance and reliability of the cast products. Additionally, we present a case study that demonstrates an innovative method for analysing an Al-Mg-Si alloy casting using QEMSCAN. This method proves to be highly effective for routine testing and quality control of cast products, ensuring consistency and high standards in production.

Keywords: Materials characterization, Quantitative microstructural analysis, Direct chill casting, Aluminium alloy, QEMSCAN.

1. Introduction

Aluminium alloys, particularly the AA 6063 series, are widely used in industries such as construction, automotive, and aerospace due to their excellent extrudability, corrosion resistance, and mechanical properties. Direct chill (DC) casting is a prevalent method for producing aluminium alloy billets, offering advantages in scalability and microstructural control. However, the microstructural features formed during DC casting, such as the distribution and morphology of intermetallic phases (e.g., beta-Al_5FeSi and alpha-$Al_{12}(Fe,Mn)_3Si$), significantly influence the alloy's downstream processing and final properties.

Quantitative microstructural analysis is essential for understanding these features and their impact on material performance. Traditional techniques, such as optical microscopy and standard scanning electron microscopy (SEM), provide limited quantitative data. In contrast, QEMSCAN, which integrates Field Emission Scanning Electron Microscopy (FE-SEM) with Mineral Liberation Analysis (MLA) software, enables precise, automated, and quantitative analysis of microstructural phases.

N. C. W. Kuijpers et al. [6] has elaborated five different quantitative techniques for measurement of Beta to Alpha transformation. Four of the methods are based on morphological characteristics and the fifth one was based on XRD pattern measurement of intermetallic extracted from the matrix. The present work is an unique method based on SEM and EDS technique along with the use of a powerful mineralogical software MLA.

This study leverages QEMSCAN to investigate the microstructural characteristics of DC-cast AA 6063 billets, focusing on the beta-to-alpha phase ratio and its implications for extrusion performance. Additionally, the effect of homogenization treatment on phase transformation is quantified to optimize processing conditions.

2. Experimental Methodology

The AA 6063 alloy primarily contains aluminium, magnesium, and silicon, forms alpha (Mg_2Si) and beta (Mg_2Si) phases during solidification. The beta phase, which is brittle and plate-like, can hinder extrudability, while the alpha phase, with its more equiaxed morphology, is desirable for improved formability. Homogenization treatments are commonly applied to transform beta phase into alpha phase, enhancing the alloy’s suitability for extrusion. Understanding and quantifying these phase transformations is essential for optimizing alloy performance.

2.1 Material & Casting

AA 6063 aluminium alloy billets were produced via direct chill casting. The alloy composition, determined using inductively coupled plasma optical emission spectroscopy (ICP-OES), is presented in Table 1.

Table 1. Composition of the material.

Alloy	Si	Mg	Mn	Ca	Fe	Zn	Ni	Ti
6063	0.55 %	0.55%	0.07%	0.026%	0.18%	0.02%	0.005%	0.018%

The casting parameters are given below in Table 2.

Table 2. Casting parameters.

Casting parameters	**Values**
Billet diameter	7 inches
Casting speed	126 mm/min
Water flow rate	78 L/min
Water temperature	38 °C
Metal pouring temperature	705 °C

2.2 Homogenization Treatment

Samples underwent a three-stage homogenization cycle:

a. Ramp up to 580 °C at 50 °C/h
b. Hold at 580 °C for 6 hours
c. Controlled furnace cooling at 30 °C/h to 300 °C, followed by air cooling

This cycle is intended to promote transformation of the metastable β-AlFeSi phase into the more rounded α-AlFeSi phase.

The micrographs of both as cast and homogenized sample are shown in Figures 1 and 2.

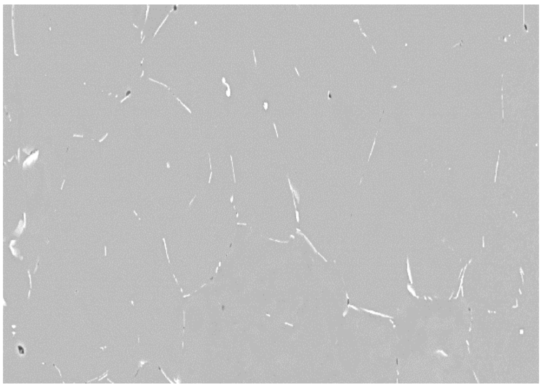


Figure 1. As cast sample micrograph.

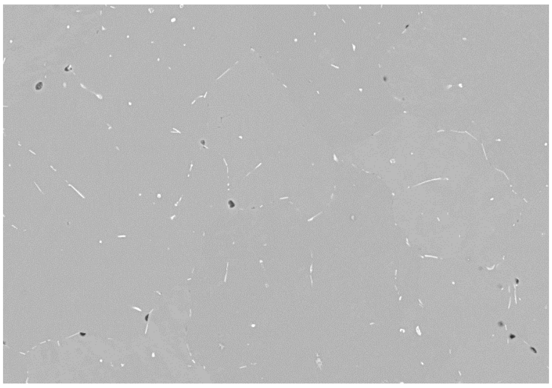


Figure 2. Homogenized sample micrograph.

The figure above clearly depicts the characteristics of transformation beta phases to alpha phases. In order to quantify the percentage of conversion QEMSCAN instrument was used.

2.3 QEMSCAN Setup

i. **Instrument**: FEI Quanta 650 FEG SEM with MLA software
ii. **Voltage**: 20 kV
iii. **Beam current**: 2 nA
iv. **Scan resolution**: 2 µm per pixel
v. **Area analyzed**: 10 mm^2 per sample
vi. **Data collected**: Backscattered electron images, EDS spectra, and phase mapping

MLA software was calibrated to distinguish between β-AlFeSi and α-AlFeSi phases based on characteristic elemental ratios and morphology.

2.4 Microstructural Analysis

Microstructural characterization was conducted using a QEMSCAN system, comprising an FEI Quanta 650 FEG SEM equipped with MLA software. Samples were prepared by sectioning the billets, polishing to a 1 µm finish, and etching with 0.5 % HF solution to enhance phase contrast. QEMSCAN analysis was performed at an accelerating voltage of 20 kV and a beam current of 2 nA. The MLA software identified and quantified phases based on backscattered electron (BSE) imaging and energy-dispersive X-ray spectroscopy (EDS) data. The beta-to-alpha phase ratio and particle size distribution were measured across multiple regions of interest (ROIs) to ensure statistical reliability.

The MLA software does image processing based on analysis of all regions of interest and reference library. The parameters of interest are selected based on which image is classified. The data set comprising each phase as single data are tabulated with its characteristics like length, breadth, area, perimeter, angularity, circularity, form factor etc. Then percentage of conversion from beta to alpha was calculated using simple statistical tools.

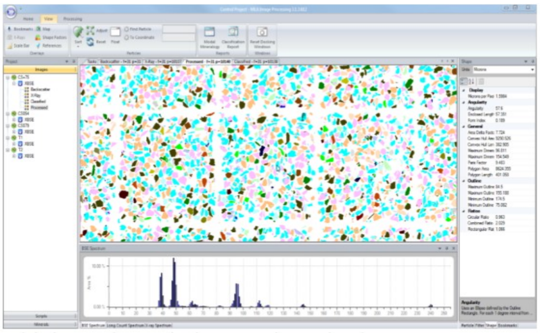


Figure 3. Image processing.

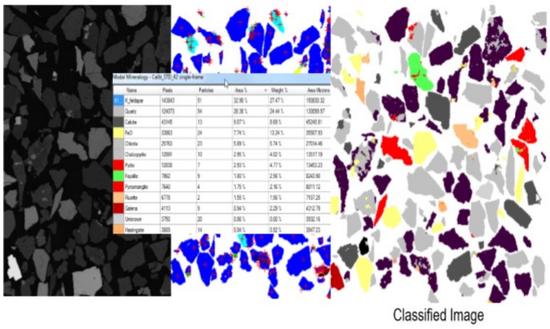


Figure 4. Image classification.

3. Results and Discussion

3.1 Microstructural Features of As-Cast Billets

QEMSCAN analysis revealed a heterogeneous microstructure in the as-cast AA 6063 billets, characterized by a matrix of aluminium with dispersed intermetallic phases. The primary intermetallic phases identified were beta-Al_5FeSi (needle-like morphology) and alpha-$Al_{12}(Fe,Mn)_3Si$ (spherical or cuboidal morphology). The beta phase was predominantly located at grain boundaries, while the alpha phase was more uniformly distributed.

The needle-like beta-Al_5FeSi particles, with an average length of 10–20 µm, were found, which are the main reasons to cause micro-cracking during extrusion, leading to surface defects and increased extrusion pressure. This is attributed to the brittle nature of the beta phase and its tendency to align along the extrusion direction, creating stress concentrations.

3.2 Effect of Homogenization

Post-homogenization, QEMSCAN analysis indicated a significant reduction in the beta phase with a corresponding increase in the alpha phase. The transformation was accompanied by a morphological change, with the needle-like beta particles converting into spherical alpha particles (average diameter: 5–8 µm). This phase transformation takes place due to diffusion of Fe and Si during homogenization, facilitated by the high temperature and controlled cooling rate.

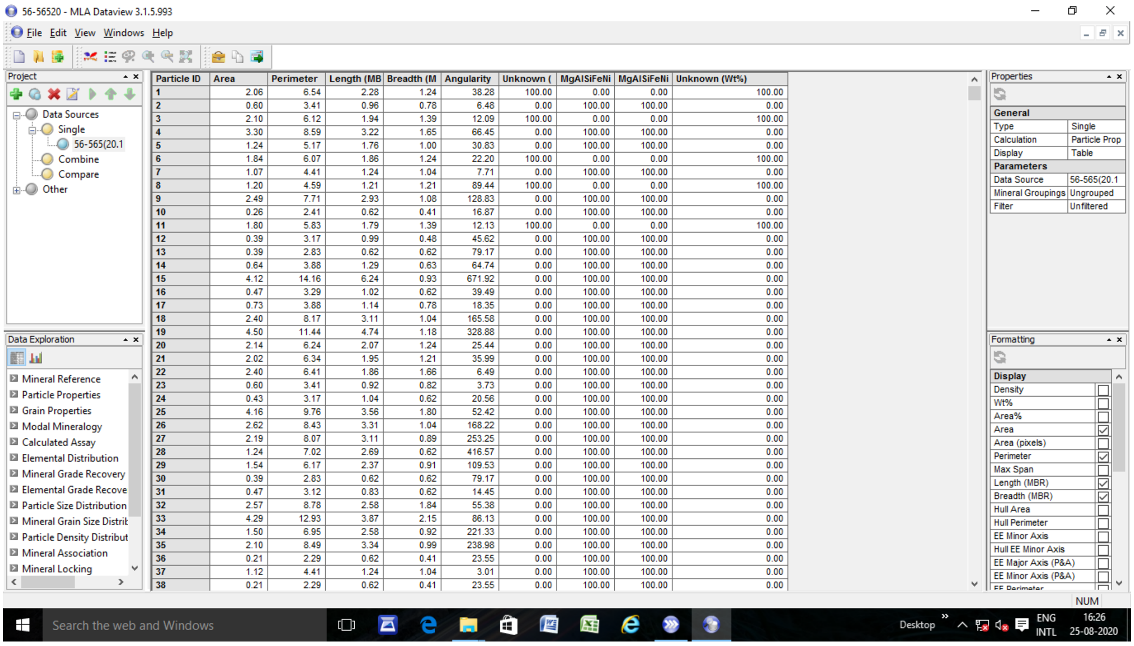
3.3 Quantitative Insights from QEMSCAN

QEMSCAN's ability to provide high-resolution, automated phase identification and quantification was critical to this study. The technique enabled precise measurement of phase fractions, particle sizes, and spatial distributions, which are difficult to achieve with conventional SEM or optical microscopy.

The example of data view shown by QEMSCAN is presented in Figure 5.

The conversion efficiency calculated based on the MLA data analysis is of the order of 92 %. This transformation improved the microstructural homogeneity and reduced the possibility of crack initiation during extrusion. Conversion percentage above 90 %, calculated according to Equation (1) is considered as very good homogenization level.

$$\text{Conversion Efficiency} = \frac{\beta_{initial} - \beta_{final}}{\beta_{initial}} \times 100 \tag{1}$$



Particle ID	Area	Perimeter	Length (MB	Breadth (M	Angularity	Unknown (	MgAlSiFeNi	MgAlSiFeNi	Unknown (Wt%)
1	2.06	6.54	2.28	1.24	38.28	100.00	0.00	0.00	100.00
2	0.60	3.41	0.96	0.78	6.48	0.00	100.00	100.00	0.00
3	2.10	6.12	1.94	1.39	12.09	100.00	0.00	0.00	100.00
4	3.30	8.59	3.22	1.65	66.45	0.00	100.00	100.00	0.00
5	1.24	5.17	1.76	1.00	30.83	0.00	100.00	100.00	0.00
6	1.84	6.07	1.86	1.24	22.20	100.00	0.00	0.00	100.00
7	1.07	4.41	1.24	1.04	7.71	0.00	100.00	100.00	0.00
8	1.20	4.59	1.21	1.21	89.44	100.00	0.00	0.00	100.00
9	2.49	7.71	2.93	1.08	128.83	0.00	100.00	100.00	0.00
10	0.26	2.41	0.62	0.41	16.87	0.00	100.00	100.00	0.00
11	1.80	5.83	1.79	1.39	12.13	100.00	0.00	0.00	100.00
12	0.39	3.17	0.99	0.48	45.62	0.00	100.00	100.00	0.00
13	0.39	2.83	0.62	0.62	79.17	0.00	100.00	100.00	0.00
14	0.64	3.88	1.29	0.63	64.74	0.00	100.00	100.00	0.00
15	4.12	14.16	6.24	0.93	671.92	0.00	100.00	100.00	0.00
16	0.47	3.29	1.02	0.62	39.49	0.00	100.00	100.00	0.00
17	0.73	3.88	1.14	0.78	18.35	0.00	100.00	100.00	0.00
18	2.40	8.17	3.11	1.04	165.58	0.00	100.00	100.00	0.00
19	4.50	11.44	4.74	1.18	328.88	0.00	100.00	100.00	0.00
20	2.14	6.24	2.07	1.24	25.44	0.00	100.00	100.00	0.00
21	2.02	6.34	1.95	1.21	35.99	0.00	100.00	100.00	0.00
22	2.40	6.41	1.86	1.66	6.49	0.00	100.00	100.00	0.00
23	0.60	3.41	0.92	0.82	3.73	0.00	100.00	100.00	0.00
24	0.43	3.17	1.04	0.62	20.56	0.00	100.00	100.00	0.00
25	4.16	9.76	3.56	1.80	52.42	0.00	100.00	100.00	0.00
26	2.62	8.43	3.31	1.04	168.22	0.00	100.00	100.00	0.00
27	2.19	8.07	3.11	0.89	253.25	0.00	100.00	100.00	0.00
28	1.24	7.02	2.69	0.62	416.57	0.00	100.00	100.00	0.00
29	1.54	6.17	2.37	0.91	109.53	0.00	100.00	100.00	0.00
30	0.39	2.83	0.62	0.62	79.17	0.00	100.00	100.00	0.00
31	0.47	3.12	0.83	0.62	14.45	0.00	100.00	100.00	0.00
32	2.57	8.78	2.58	1.84	55.38	0.00	100.00	100.00	0.00
33	4.29	12.93	3.87	2.15	86.13	0.00	100.00	100.00	0.00
34	1.50	6.95	2.58	0.92	221.33	0.00	100.00	100.00	0.00
35	2.10	8.49	3.34	0.99	238.98	0.00	100.00	100.00	0.00
36	0.21	2.29	0.62	0.41	23.55	0.00	100.00	100.00	0.00
37	1.12	4.41	1.24	1.04	3.01	0.00	100.00	100.00	0.00
38	0.21	2.29	0.62	0.41	23.55	0.00	100.00	100.00	0.00

General	
Type	Single
Calculation	Particle Prop
Display	Table
Parameters	
Data Source	56-565(20.1
Mineral Groupings	Ungrouped
Filter	Unfiltered

Display	
Density	☐
Wt%	☐
Area%	☐
Area	☑
Area (pixels)	☐
Perimeter	☑
Max Span	☐
Length (MBR)	☑
Breadth (MBR)	☑
Hull Area	☐
Hull Perimeter	☐
EE Minor Axis	☐
Hull EE Minor Axis	☐
EE Major Axis (P&A)	☐
EE Minor Axis (P&A)	☐

Figure 5. Example of data view.

The transformation of another campaign with heat treatment at 575 °C, investigated through QEMSCAN is shown below in Figures 6 and 7. The conversion efficiency was found to be 88 % in QEMSCAN analysis.

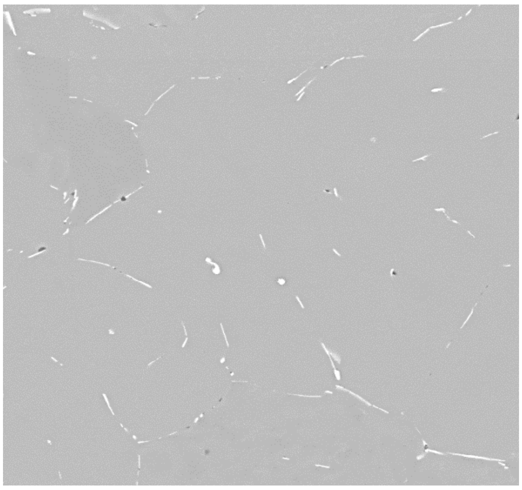


Figure 6. As cast sample micrograph.

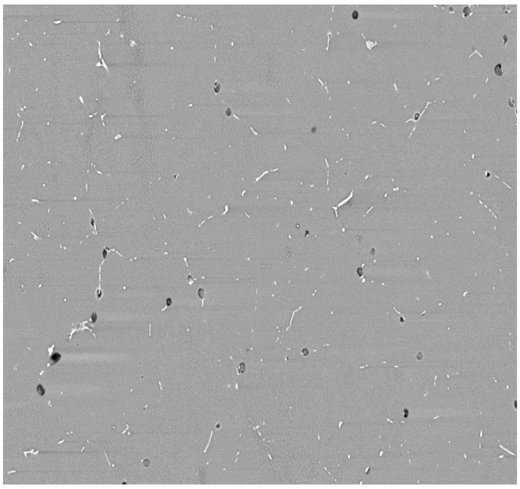


Figure 7. Homogenized sample micrograph.

In order to cross check the QEMSCAN method, several measurements were carried out by Metallurgical microscope (Carl Zeiss, Imager M2m model) with image analysis software. The microscopic analysis was based on three morphological features, length, circularity and aspect ratio. The variation in conversion efficiency between the two techniques is found to be in the range of 4 to 8 %.

The reduction in β-phase and increase in α-phase results in lower extrusion pressures, reduced surface tearing and defects on extruded profiles, and improved die life due to reduced abrasion by brittle intermetallic. The accurate quantification by QEMSCAN allowed pre-screening of billet quality before extrusion, potentially saving downstream processing cost and time.

4. Conclusions

This study demonstrates the efficacy of QEMSCAN as a powerful tool for quantitative microstructural analysis of DC-cast AA 6063 aluminium alloy billets. The key findings are:

a. As-cast billets exhibit a high beta-to-alpha phase ratio with needle-like beta-Al_5FeSi particles detrimentally affecting extrudability.
b. Homogenization treatment at 580 °C for 6 hours reduces the beta-to-alpha ratio achieving more than 90 % conversion of beta to alpha phase.
c. QEMSCAN provides detailed quantitative data on phase fractions and morphologies, enabling precise optimization of casting and processing parameters.
d. The technique can be used as a practical industrial tool for billet certification and process optimization

These findings signify the importance of advanced characterization techniques in aluminium alloy design for specific applications.

5. Acknowledgments

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6. References

1. Mondolfo, L. F. (1976). Aluminum Alloys: Structure and Properties. Butterworths.
2. Dons, A. L., et al. (1999). The role of intermetallic phases in aluminium extrusion. Materials Science and Engineering A, 265(1-2), 6–16.
3. QEMSCAN User Manual. (2020). FEI Company.
4. ASTM E112-13. (2013). Standard Test Methods for Determining Average Grain Size. ASTM International.
5. H. Tanihata, et al.: Effect of casting and homogenizing treatment conditions on the formation of Al-Fe-Si intermetallic compounds in 6063 Al-Mg-Si alloys. J. Mater. Sci. 34 (1999) 1205-1210.
6. N. C. W. Kuijpers et al., Assessment of different techniques for quantification of α-Al(FeMn)Si and β-AlFeSi intermetallics in AA 6xxx alloys. Mater.Char.49 (2002)409-420.
7. Totten, G.E., & MacKenzie, D.S. (Eds.). (2003). Handbook of Aluminum: Vol. 1: Physical Metallurgy and Processes. CRC Press.
8. Hatch, J.E. (1984). Aluminum: Properties and Physical Metallurgy. ASM International.
9. N. C. W. Kuijpers, et al- A model of the β-AlFeSi to α-Al(FeMn)Si transformation in AA 6xxx alloys. Mater. Trans. 44 (2003), 1448-1456.
10. Liu, J., & Kulak, M. (2012). Quantitative microstructural analysis of aluminium alloys using SEM-based techniques. Materials Characterization, 70, 45–53.
11. T Minoda, et.al - The quantitative analysis of α-AlFeSi ratio in a 6063 aluminum alloy billet by X-ray diffraction, Proceedings of ICAA-6 (1998), 339-344
12. N. C. W. Kuijpers, Kinetics of the β-AlFeSi to α-Al(FeMn)Si transformation in Al-Mg-Si alloys , PhD Thesis,2004, NIMR, Netherlands.