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Citation for the original published paper (version of record):

Hernando, J C., Elfsberg, J., Dahle, A., Diószegi, A. (2019)
Evolution of primary austenite and its influence on eutectic microstructures during
coarsening of Fe-C-Si alloys
Materialia, 7: 100391
<https://doi.org/10.1016/j.mtla.2019.100391>

Access to the published version may require subscription.

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Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:hj:diva-43552>

Evolution of primary austenite during coarsening and impact on eutectic microstructure in Fe-C-Si alloys

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Abstract

The evolution of primary austenite morphology during isothermal coarsening has been studied in the three main Fe-C-Si alloys used in industry, LGI, CGI, and SGI. The dendritic microstructure increases length scale during coarsening accompanied by fragmentation and coalescence of austenite crystals. The morphological parameters, SDAS, M_γ , D_{ID}^{hyd} , and D_γ show a linear relation with the cube root of coarsening time, $t^{1/3}$, with similar rates for the three different Fe-C-Si alloys. The eutectic microstructures after coarsening of primary austenite in CGI and SGI alloys are not significantly affected by the surface area of primary austenite and the size of the interdendritic regions. Fraction, nodularity, shape distribution of graphite particles and the number of nodules and eutectic cells are similar when studied as a function of coarsening time. These results suggest that the nucleation frequency in CGI and SGI, and the growth of eutectic microstructures in CGI, are not significantly influenced by the morphology of primary austenite.

Keywords: Solidification, Coarsening, Dendrites, Austenite, Eutectic, EBSD, CGI, SGI

1. Introduction

Fe-C-Si alloys, also known as cast iron, remain dominant in energy generation, machinery, and heavy vehicle applications despite the development of novel alloys and materials over the last decades. The great variety of properties combined with design flexibility and cost efficiency achieved by advanced casting technologies are the reasons for the longevity and stable use of these alloys. Lamellar graphite iron (LGI), with graphite in the shape of lamellae or flakes, has been a traditional industrial cast iron alloy for decades. The discovery of new types of graphitic alloys presented an excellent opportunity for designers and producers in competition with other metallic alloys. In spheroidal graphite iron (SGI), also known as nodular or ductile iron, the predominant graphite shape is spheroidal, while in compacted graphite iron (CGI) an intermediate worm-like graphite shape, also called vermicular graphite is the main characteristic [1, 2]. In 1966, SGI represented about 5% of the world's production, dominated by LGI, which counted for 75% of the total world's casting production by tonnage. Nowadays, SGI applications have grown steadily counting for 25% of the world's casting production [3]. For many years, CGI was considered as undesired scrap material from SGI production [2], until its excellent set of properties granted CGI full recognition. In the 70s, CGI started to be produced intentionally due to its excellent balance between thermal and mechanical properties. Thus, SGI and CGI offer an excellent set of

tunable properties added to the standard LGI alloys and have become popular for high-temperature applications, such as cylinder heads for heavy-duty engines.

The microstructure formed during solidification controls the final properties of Fe-C-Si alloys [4]. Solidification of hypoeutectic Fe-C-Si alloys begins with the nucleation and growth of primary austenite [5] that typically forms dendritic microstructures [6] which morphology is controlled throughout solidification by coarsening [7]. Coarsening becomes especially important after dendritic coherency, when dendrites impinge on each other, grow competitively and the feeding shifts from mass to interdendritic [8].

The classical Ostwald ripening relation based on curvature induced remelting controls the coarsening of dendritic microstructures [9, 10]. Coarsening minimizes the interfacial free energy of the solid-liquid region reducing its total interfacial area [11] and increasing its length scale [12, 13]. Remelting of smaller arms at the expense of thickening of the large arms occurs during coarsening [14]. Subsequently, further coarsening leads to dendrite fragmentation [15] and coalescence [13].

The secondary dendrite arm spacing (SDAS) is a characteristic of dendritic microstructures [16, 17]. The SDAS follows a linear relation with the cube root of total solidification time, $t^{1/3}$ [6, 18, 19]. However, it has been observed that after long coarsening times the microstructure shows a globular appearance [20] which complicates an accurate measurement of the SDAS. Shape-independent morphological parameters based on stereological relations [21] such as the interfacial area are used to describe the morphological evolution of dendritic microstructures during coarsening [7]. Recently, three-dimensional investigations allowed the measurement of the local surface area per unit volume of the entire dendritic microstructure [4, 12].

Subsequently, when the eutectic composition is reached, the eutectic liquid solidifies in the interdendritic regions [22] and solidification is completed. The eutectic microstructure is comprised of graphite and eutectic austenite. Graphite is the first phase nucleating during the eutectic reaction. Nucleation of graphite occurs on pre-existing inclusions in the liquid [23-27]. For LGI, complex sulfides (Mn,X)S [28] that nucleated on complex oxides of Al, Si, Zr, Mg, and Ti [23, 24, 27] act as nuclei. In CGI and SGI, the nuclei are complex Mg silicates (MgO.SiO₂) [2] that nucleated on the external layer of MgS and CaS sulfides [25].

Trace elements control the predominant growth direction of graphite [29-31]. The formation of LGI is favored in the presence of surface-active elements, such as S, O, Al, Ti, As, Bi, Te, Pb, and Sb, often called anti-compacting or anti-spheroidizing elements. On the other hand, reactive elements, such as Mg, Ce, Ca, Y, and La, promote the formation of SGI and are called compacting or spheroidizing elements.

The amount of trace elements required to promote a change in the growth direction of graphite is low [2]. The presence of O and S is the most detrimental to the formation of CGI and SGI due to their strong tendency to reduce the surface energy of the iron/graphite interface [1, 29, 30]. The impact of dissolved O on the nodularity of the final iron is critical [32, 33]. It is well-known that the formation of CGI and SGI requires low amounts of dissolved O and S, which are usually present in the base materials used to produce Fe-C-Si alloys. The practical way to reduce the level of O and S is by reacting them with certain reactive elements. This treatment is called nodularization, and Mg is the central element used for this purpose [34]. Ce or rare earth elements (RE) mixtures are also used for this purpose [2, 35, 36]. The production window for CGI is narrow [35], showing an abrupt transition to LGI [37]. Additionally, it is challenged by the fading of Mg over the time [37-40] which needs to be monitored [41].

In LGI and CGI, graphite and eutectic austenite grow cooperatively forming eutectic cells [42]. In LGI, the graphite lamella leads the eutectic interface into the liquid, with austenite growing behind [43]. The eutectic cells are usually smaller than the primary austenite grains [44]. The crystallographic orientation of the austenite in the eutectic cells is identical to the primary austenite crystal [5, 45].

Eutectic cells in CGI consist of eutectic austenite and vermicular graphite, which has a highly branched three-dimensional morphology [46-48]. Several observations suggest that in this case austenite leads the growth and constrains the growth of graphite.

The eutectic transformation in SGI proceeds in a significantly different way. Independent graphite nodules nucleate and grow in the liquid in a divorced eutectic [49].

Mechanical properties of cast iron have been traditionally related mainly to graphite shape and distribution [42]. This promoted a significant number of studies on the role of eutectic microstructures for mechanical properties of cast iron, while the role of the primary austenite has usually received less attention [45]. However, recent studies have shown that the morphology of primary austenite controls the mechanical properties of hypoeutectic LGI [50] and CGI [51]. At the same time, the size of eutectic cells in LGI was correlated to the SDAS [41], suggesting a relationship between the morphologies of primary and eutectic microstructures. Consequently, understanding the morphological evolution of primary austenite during solidification of Fe-C-Si alloys is of major importance to control the mechanical properties.

The scope of this work is to study the morphological evolution of primary austenite during coarsening for the three leading families of Fe-C-Si alloys; LGI, CGI and SGI. The characterization of primary austenite during coarsening is performed using two-dimensional morphological parameters. Additionally, the influence of primary austenite morphology on the eutectic microstructure is studied for CGI and SGI alloys.

2. Experimental

2.1. Base material

Two base alloys have been used in this study. An industrial hypoeutectic LGI and a hypoeutectic SGI. The SGI was used to produce CGI. The nodularization treatment of the SGI was performed with FeSiMg. Si-based inoculant was added during pouring. The chemical compositions of the alloys (Table 1) were obtained by optical emission spectroscopy (OES) [52] on graphite-free white chilled samples produced immediately before the pouring of the alloy into sand molds. Both alloys were cast in furan resin bonded sand molds producing 50 mm diameter and 300 mm long rods. The rods were machined to a weight of 400 g, yielding approximately 38 mm diameter and 42 mm long rods.

Table 1. Chemical composition of the base alloys (wt. pct). Carbon equivalent $CE=C+1/3 (Si+P)$.

	C	Si	Mn	P	S	Cu	Mg	CE
Base LGI	3.3	1.8	0.58	0.034	0.086	0.9	-	3.9
Base SGI	3.4	2.5	0.68	0.030	0.010	0.9	0.063	4.2

2.2. Preliminary Experiments (PE)

Preliminary experiments (PE) were conducted to study the solidification of LGI, CGI and SGI alloys. The cylindrical rods were remelted at 1450 °C in an Al₂O₃ crucible in an electrical resistance furnace. A flow of Ar gas of 99.999% purity was introduced at the bottom of the furnace [37]. Different holding times were investigated to find the optimal conditions for producing the desired graphite morphologies. Next, the furnace was switched off and the samples were allowed to solidify.

For LGI, a holding time of 30 min was applied at 1450 °C to produce lamellar graphite, Fig. 1a. For CGI and SGI, Mg fading controls the presence of dissolved O in the melt and hence the morphology of the graphite. The SGI was remelted and held for 10 min at 1450 °C to produce SGI after furnace solidification, Fig. 1b. CGI was produced from the SGI base alloy by remelting and holding for 80 min at 1450 °C, Fig. 1c.

Figure 1: Micrographs of **(a)** LGI, **(b)** SGI, and **(c)** CGI. Samples etched in a picric-based reagent [53].

Thermal analysis was conducted in the PE. The temperature was recorded using two type-S thermocouples. One thermocouple was located in the center of the sample (T_{center}) and the other next to the inner wall of the crucible (T_{wall}) at the same depth. Fig. 2 shows the cooling curve for the SGI. Thermal coherency was determined as the maximum difference in temperature between the two thermocouples after nucleation (i.e., the first minimum in Fig. 2) [8]. Nucleation (T_N) and coherency (T_{coh}) temperatures of the LGI, CGI, and SGI are shown in Table 2. At coherency, the dendrites have impinged on each other and coarsening becomes the dominant growth mechanism [8].

Table 2. Nucleation (T_N) and coherency (T_{coh}) temperatures, coherency time (t_{coh}) and coarsening temperature (T_{iso}) from the preliminary experiments for each alloy.

	T_N (°C)	T_{coh} (°C)	t_{coh} (s)	T_{iso} (°C)
LGI	1205	1191	1020	1175
CGI	1180	1167	1040	1163
SGI	1178	1168	848	1161

Figure 2: Cooling curves and determination of thermal coherency for SGI.

2.3. Isothermal Coarsening experiments (ICE)

Isothermal coarsening experiments (ICE) were conducted at temperatures below coherency but safely above the eutectic temperature and are shown in Table 2 and Fig. 3. For each alloy, an isothermal treatment was applied to promote the coarsening of primary austenite. The samples were remelted and held at 1450 °C for the times described above for each alloy; 30 min for LGI, 80 min for CGI, and 10 min for SGI. Next, the samples were cooled inside the furnace until the coarsening temperature was reached. The samples were held at this temperature for different times. Primary austenite was coarsened at 1175 °C in LGI, 1163 °C in CGI and 1161 °C in SGI as shown in Table 2. Fig. 3 shows the ICE for SGI.

Different coarsening times were studied, as shown in Table 3. Samples were quenched in agitated water at room temperature. The morphological evolution of primary austenite in LGI was studied for different coarsening times up to 5760 min (4 days). However, isothermal coarsening in CGI and SGI was only conducted on coarsening times up to 90 min with are closer to solidification times in industrial cast components [54].

An additional sample for 0 min of isothermal treatment was quenched from the thermal coherency temperature of each alloy. These samples allowed the observation of the initial coherent dendritic microstructure.

Table 3. Isothermal coarsening time (in minutes)

Isothermal coarsening time (min.)										
LGI	0	30	90	180	360	720	1440	2880	4320	5760
CGI and SGI	0	10	30	60	90	-	-	-	-	-

Figure 3: Cooling curve and isothermal coarsening treatment for SGI.

2.4. Coarsening and furnace cooling experiments (CFCE)

After the ICE, an additional series of experiments was conducted to study the possible effect of the primary austenite morphology on the eutectic microstructures of CGI and SGI. In this series of experiments, the samples were subjected to the same procedure as in the ICE, but in this case, the samples were cooled to room temperature inside the furnace after the isothermal treatment. Therefore, similar cooling conditions were applied during solidification after coarsening (as in the non-coarsened samples from the PE). The isothermal coarsening times are shown in Table 3. Fig. 4 shows an example of the temperature recordings for these experiments for SGI.

Figure 4: Coarsening and furnace cooling experiments (CFCE) for SGI.

2.5. Microstructural characterization

The samples were sectioned in the middle of the longitudinal axis, subjected to metallography and etched with a picric-based solution to reveal the microstructure [20, 53]. Microstructural analysis was performed by optical microscopy. The micrographs of the quenched samples were transformed into binary images where dendrites are represented in black. Microstructural analysis was performed with Olympus Stream Motion Desktop software 1.9.1.

The secondary dendrite arm spacing, SDAS, commonly used in literature to characterize coarsening [16] was measured including at least 3 secondary dendrite arms parallel to the primary arm.

The perimeter, P_Y , and area, A_Y , of the primary austenite were measured on the same binary images. Morphological shape-independent parameters were calculated to perform a two-dimensional quantitative analysis of the morphological evolution of primary austenite during coarsening.

The modulus of primary austenite, M_Y [μm] (Eq.1), is the ratio of the volume of an austenite crystal, V_Y , and the surface area, S_Y . Some authors refer to this ratio as the specific interfacial area, S_s [13]. It is related to the modulus of geometry employed in Chvorinov's analytical calculation of solidification time [55], as it is the volume of a solidifying domain relative to its cooling surface. S_Y is approximated by a relation to the perimeter of austenite in an area, P_Y , applying the stereological relationship by Underwood [21], $S_Y \approx \frac{4}{\pi} P_Y$.

$$M_Y = \frac{V_Y}{S_Y} \approx \frac{\pi A_Y}{4 P_Y} \quad (1)$$

The hydraulic diameter, or modulus, of the interdendritic region, D_{ID}^{Hyd} [μm] (Eq.2), describes the interdendritic region as the ratio between its volume, V_{ID} , and the surface area, S_{ID} .

$$D_{ID}^{\text{Hyd}} = \frac{V_{ID}}{S_{ID}} \approx \frac{\pi A_{ID}}{4 P_{ID}} \approx \frac{\pi (A_T - A_Y)}{4 P_Y} \quad (2)$$

where: V , A , S , and P , are volume, area, interfacial surface area, and perimeter, respectively. The subscripts T , Y , and ID , are total, primary austenite, and interdendritic region, respectively.

The shortest distance between the centers of gravity of austenite crystals, D_Y [μm], describes the spatial distribution of austenite crystals.

The volume fraction, f_Y , is estimated as the ratio between A_Y and A_T (Eq.3).

$$f_Y \approx \frac{A_Y}{A_T} \quad (3)$$

Several characteristics of the eutectic microstructure were obtained on random micrographs of a minimum area of 24 mm² for each specimen. The fraction, number and nodularity of graphite particles of the CFCE samples were determined according to the ISO 16112:2006(E) standard [56]. Additionally, the number of eutectic cells in CGI was measured on radial sections of the CFCE samples.

2.6. EBSD and EDS characterization

The quenched samples of the ICE in LGI were investigated in a JEOL JSM-7001F Scanning Electron Microscope (SEM). The samples were prepared by mechanical polishing

followed by electrochemical polishing. Electron Backscatter Diffraction (EBSD) analysis was carried out with an EDAX detector at a fixed acceleration voltage of 20 kV and a step size of 1 μm . EBSD maps were analyzed using the TSL-OIM v.7.3 software package. Energy Dispersive X-ray Spectroscopy (EDS) maps were collected in similar regions to identify the Si distribution.

3. Results and discussion

3.1. Evolution of primary austenite morphology during coarsening

Fig. 5(a-e) shows the morphological evolution of primary austenite as a function of isothermal coarsening time in the quenched samples of LGI. Fig. 5(a) shows the sample quenched at coherency and 0 min of isothermal holding. The microstructure consists of highly branched equiaxed dendrites and several primary crystals are observed. This microstructure is consistent with a coherent dendritic network where the equiaxed crystals have impinged on each other [57]. The remaining liquid solidified as ledeburite.

After 30 min of isothermal holding, it is apparent that significant coarsening has already occurred and the secondary dendrite arms appear thicker, Fig. 5(b). The dendritic network structure is still visible. In Fig. 5(c) the length scale of the secondary dendrite arms has increased further, and they show a spheroidal morphology after 360 min of isothermal holding.

The microstructure after 24 h (1440 min) of isothermal holding shows heavily coarsened, almost globular, primary austenite crystals, Fig. 5(d). Remnants of the dendritic microstructure are observed as a regular spacing of crystals, but it is possible that they are not interconnected in three dimensions. Compared to Fig. 5(c) there are fewer crystals/globules. Indications of coalescence are observed as highlighted by the arrows.

The sample coarsened for 72 h (4320 min) contains fewer and larger globules, Fig. 5(e). The globules have an irregular, non-spherical appearance that is a signature of coalescence as marked with arrows.

EBSD was performed to investigate the crystallography of primary austenite. The sample quenched after coherency shows a highly dendritic microstructure with clearly identifiable secondary dendrite arms, Fig. 6(a). The crystallography of all austenite is identical, i.e. they are the same crystal.

The microstructure of the sample coarsened for 30 min; Fig. 6(b) shows a dendritic appearance of coarsened primary austenite. Most of the primary austenite in Fig. 6(b) belong to the same crystal. This observation is highlighted in the inverse pole figure (IPF) map of austenite shown in Fig. 7(a). In Fig. 6(c), after 360 min of isothermal coarsening, larger globules are observed, and many have the same crystallographic orientation.

After 24 h (1440 min) of isothermal coarsening, the globules have increased in size, Fig. 6(d). In this case, the globules have different crystallographic orientations, Fig. 7(b). These differences indicate that they are detached from each other, floating freely in the liquid and therefore their spacings are no longer determined by the classical coarsening mechanism. The fragmentation sequence during coarsening was recently studied by Cool and Voorhees [15]. They found that fragmentation is a result of local capillary induced remelting. The study showed that coalescence has a greater impact on microstructural coarsening than fragmentation. This is in accordance with the observations in Fig. 5(a-e).

The micrographs of the samples from the CFCE are shown in Fig. 5(f-j) for CGI and Fig. 5(k-o) for SGI. In CGI the primary austenite is easily observed together with eutectic cells. However, it is difficult to distinguish the primary microstructure in the isothermally coarsened SGI samples due to the divorced eutectic thus, indicating that accurate quantitative characterization of primary austenite morphology in fully solidified samples of SGI is difficult.

Fig. 5(f) shows the as-cast microstructure resulting from uninterrupted solidification during furnace cooling. Compared to the microstructure in Fig. 5(a) the dendritic microstructure in Fig. 5(f) includes the effect of coarsening during solidification after coherency. Equiaxed dendrites with secondary arms are clearly observed.

Samples isothermally coarsened for 10 and 30 min, Fig. 5(g) and (h) show dendritic microstructures coarsening with time. In Fig. 5(i) and Fig. 5(j), after 60 and 90 min of isothermal coarsening, the microstructure contains austenite globules. Indications of coalescence are observed in the micrographs as highlighted with arrows.

Overall the morphological evolution of primary austenite is similar to that observed in the quenched samples of LGI Fig. 5(a-e).

Figure 5: Micrographs from the ICE of LGI, quenched and color-etched samples after (a) 0 min, (b) 30 min, (c) 360 min, (d) 24h, and (e) 72h of isothermal coarsening. Micrographs from the CFCE of CGI after (f) 0 min, (g) 10 min, (h) 30 min, (i) 60 min, and (j) 90 min of isothermal coarsening, and SGI after (k) 0 min, (l) 10 min, (m) 30 min, (n) 60 min, and (o) 90 min of isothermal coarsening. Please note that the micrographs of LGI have a different scale than the micrographs of CGI and SGI.

Figure 6: Crystallographic orientations in the quenched samples of LGI. Inverse pole figures after (a) 0 min, (b) 30 min, (c) 360 min and (d) 1440 min (24 h) of isothermal coarsening. RD and RA are Radius Direction and Rotational Axis of the sample, respectively.

Figure 7: Inverse pole figure (IPF) maps of austenite in the quenched samples of LGI. Inverse pole figures after (a) 30 min and (b) 1440 min (24 h) of isothermal coarsening. RD and RA are Radius Direction and Rotational Axis of the sample, respectively.

3.2. Quantitative characterization of primary austenite morphology during coarsening

The morphology of primary austenite was quantitatively characterized in the quenched samples of LGI, CGI and SGI alloys. The morphological parameters introduced in the experimental section, M_γ , D_γ and D_{ID}^{hyd} , are shown in two different ways. First, the results of the LGI alloy, which was studied for long coarsening times, are shown to describe the full morphological evolution of primary austenite during coarsening. Second, the parameters for CGI and SGI alloys, measured for coarsening times up to 90 min, are shown together with the results corresponding to the same coarsening times for LGI.

Fig. 8 shows the SDAS as a function of the cube root of coarsening time, $t^{1/3}$, for the three alloys. The initial values for SDAS correspond to SDAS at the coarsening temperature after coherency. LGI has the largest SDAS which is slightly larger than SGI and SDAS for CGI is significantly smaller.

All alloys have a linear relation to $t^{1/3}$ in agreement with the classical Ostwald ripening relation [9, 58] and similar to results for other alloys reported in the literature [6] valid in systems ranging from Al-Cu [14, 59], Pb-Sn [4] and Fe-Ni [60].

The coarsening rate calculated for the three alloys shows no significant differences. CGI shows a slightly lower rate. This difference could be caused by a difference in alloy composition, surface tension and solid fraction [6, 11, 61]. Future work is required to elucidate the mechanism responsible for these differences.

As seen in Fig. 5(d,e), (i,j) and (n,o) the morphological changes occurring during coarsening make the observation of secondary dendrites arms challenging with increasing coarsening time. The globular appearance of the microstructure suggests that there is no regular arrangement of the globules relative to each other therefore making SDAS challenging to measure. Hence, the error in SDAS increases for increasingly coarsened microstructures, as seen in Fig. 8. This suggests that SDAS is only valid for relatively short coarsening times. Shape-independent scale parameters should be considered for a more accurate characterization of primary austenite morphology for longer coarsening times.

Figure 8: SDAS as a function of coarsening time in the quenched samples of LGI, CGI, and SGI after the ICE. Error bars show 95% confidence intervals.

The modulus of primary austenite, M_γ (Eq. 1), is a shape-independent length scale parameter. During coarsening, the dendritic microstructure reduces its free energy by

reducing its surface area and thus increasing its length scale. The modulus reflects the reduction of the specific surface area of primary austenite during coarsening.

The results for LGI, Fig. 9(a), show a linear relationship between M_γ and cube root of coarsening time, $t^{1/3}$, even for very long isothermal coarsening times, i.e., 4 days. A similar linear relation to $t^{1/3}$ is also observed for CGI and SGI, as seen in Fig. 9(b). Thus, the three alloys show a similar relation between M_γ and coarsening time, indicating no significant influence of the alloy composition, e.g., Mg content, on the morphology of primary austenite for the range of compositions studied.

These results agree with recent literature studying the evolution of interfacial area in dendritic microstructures of different alloys [4, 13] reporting a relation to $t^{1/3}$ for coarsening up to long times.

Figure 9: The modulus of primary austenite, M_γ , in the quenched samples (a) of LGI, and (b) LGI, CGI and SGI after the ICE as a function of coarsening time.

The micrographs in Fig. 5 also suggest a significant change in the spatial distribution of primary austenite occurring together with the reduction of the surface area during coarsening. This can be characterized by the distance between the centers of gravity of primary austenite crystals, D_γ . The results for LGI, Fig.10(a), indicate that D_γ increases linearly with the cube root of coarsening time, $t^{1/3}$, even for very long coarsening times. However, D_γ shows larger scatter for coarsening times longer than 1 day. For CGI and SGI, D_γ also increases with a linear relationship with the coarsening time, $t^{1/3}$, as shown in Fig.10(b). D_γ shows a similar rate as a function of coarsening time for all alloys.

Figure 10: The distance between austenite crystals, D_γ , in the quenched samples (a) of LGI, and (b) LGI, CGI and SGI after the ICE as a function of coarsening time.

The changes in spatial distribution of primary austenite necessarily result in morphological changes of the interdendritic region.

The modulus, or hydraulic diameter, of the interdendritic region, D_{ID}^{hyd} , is a function of the reduction of surface area and spatial redistribution of primary austenite during coarsening. Following an equivalent method to M_γ , D_{ID}^{hyd} is calculated as the ratio between the volume of the interdendritic region and its surface area (Eq. 2).

In Fig. 11(a) D_{ID}^{hyd} is shown as a function of the cube root of coarsening time, $t^{1/3}$, for LGI. D_{ID}^{hyd} shows a linear relationship with $t^{1/3}$ up to 1 day of isothermal coarsening. However, D_{ID}^{hyd} shows a decreasing trend for longer coarsening times.

Figure 11: The hydraulic diameter of the interdendritic region, D_{ID}^{hyd} , as a function of coarsening time in the quenched samples of **(a)** LGI, and **(b)** LGI, CGI, and SGI after the ICE. The measured area fraction of primary austenite, f_γ , as a function of coarsening time of **(c)** LGI, and **(d)** LGI, CGI and SGI.

After these long coarsening times, the morphological changes occurring in the microstructure are so drastic that a 2-D section is not sufficient to represent the spatial distribution of the coarsened austenite [62]. There is significant scatter in LGI for samples coarsened for more than 1 day, as seen in Fig. 11(a), where D_{ID}^{hyd} appears to decrease. This is due to the local variation of f_γ , which is a result of the coalescence of detached austenite crystals occurring for long coarsening times. The lower values of D_{ID}^{hyd} are a result of the relationship with the austenite area fraction, f_γ , measured in the micrographs f_γ , shows small variations between samples of the same alloy, as shown in Fig. 11(c-d), which is due to the severe coarsening of the microstructure. As shown in the literature [62], selecting a finite number of control volumes in a two-dimensional investigation results in an inaccurate characterization of f_γ for heavily coarsened samples. f_γ reported in this work is measured on one two-dimensional section of the sample which could be the reason for the scatter for

long coarsening times. The large scatter in D_γ , Fig. 10(a), is also the result of the same limitation.

D_{ID}^{hyd} also increases with a linear relationship with the coarsening time, $t^{1/3}$, for CGI and SGI showing a similar rate, Fig. 11(b). The effect of local variations in measured austenite area fraction, f_γ , can be observed for SGI, with a larger scatter in the values as a function of coarsening time.

3.3. Eutectic microstructures after coarsening of primary austenite

The morphology of primary austenite during coarsening determines the interdendritic channels where nucleation and growth of the eutectic microstructure occurs. This suggests a possible relation between primary austenite morphology and eutectic microstructure. The influence of primary austenite morphology on the eutectic microstructure for CGI and SGI is studied in the samples from the CFCE. The eutectic microstructures of CGI and SGI after different isothermal coarsening times can be observed in Fig. 5(f-o).

Eutectic cells comprised of vermicular graphite particles and eutectic austenite are observed in the eutectic microstructures in the color-etched samples of CGI, Fig. 5(f-j). Primary austenite in the micrographs shows the clear effect of increasing coarsening time (described earlier). However, the eutectic cells do not seem significantly affected by coarsening time. The eutectic cells have a near-spherical appearance and are of similar size in all the micrographs.

Micrographs of the color-etched SGI samples, Fig. 5(k-o), show nodular graphite particles surrounded by eutectic austenite, consistent with the completely divorced eutectic during solidification of SGI [42]. The number of nodules and size distribution appear similar in all the micrographs.

Quantitative characterization of the graphite in CGI and SGI samples was performed on as-polished samples from the CFCE. Micrographs of CGI after 0, 30 and 90 min of isothermal coarsening are shown in Fig. 12. Fig. 13 shows the micrographs of SGI after 0, 30, 60 and 90 min of isothermal coarsening. The number of graphite particles appears to be unaffected by coarsening time and seems similar in all the micrographs of CGI, Fig. 12, and in all the micrographs of SGI, Fig. 13.

Figure 12: Micrographs from the CFCE for CGI after **(a)** 0 min, **(b)** 60 min and **(c)** 90 min of isothermal coarsening.

Figure 13: Micrographs from the CFCE for SGI after (a) 0 min, (b) 30 min, (c) 60 min and (d) 90 min of isothermal coarsening.

The measured fraction of graphite is shown as a function of the coarsening time in Fig. 14(a). The fraction of graphite in SGI is around 8.5% and it remains constant for all coarsening times, while the volume fraction in CGI is about 10% and it is also independent of coarsening time. The slight difference in graphite fraction between the alloys may be a result of the graphite morphology, thus underestimating the graphite fraction in SGI. The graphite fraction is a consequence of both alloys having the same C and Si contents. These observations suggest that the amount of graphite is not influenced by the morphology of the interdendritic region, as it remains similar for each alloy after different isothermal times.

Figure 14: (a) Graphite fraction and (b) nodularity as a function of isothermal coarsening time in CGI and SGI samples from the CFCE. Distribution of graphite particle types in (c) CGI and (d) SGI as a function of coarsening time.

The nodularity of the graphite in CGI and SGI is shown in Fig. 14(b) as a function of coarsening time. It can be observed that the nodularity of SGI is around 85% for 0 min coarsening time. There is a gradual decrease in nodularity with increasing coarsening time, reaching 70% after 90 min coarsening. The nodularity of CGI, on the other hand, is very low (around 2%) and shows insignificant variations with coarsening time.

The distribution of graphite particles is analyzed as a function of coarsening time in CGI and SGI based on the roundness shape factor (nodular, vermicular and intermediate) in Fig. 14(c-d). The total number of graphite particles and the number of vermicular graphite particles in CGI are relatively constant for different coarsening times, as shown in Fig. 14(c). Most of the graphite particles are vermicular particles.

In Fig. 14(d), the number of nodules in SGI decreases with increasing coarsening time. The most significant decrease occurs in the first 30 min and is on the order of 25%. Subsequently, the number of nodules does not change much. The number of vermicular particles increases significantly after 10 min and remains constant for longer times. However, the number of intermediate particles increases linearly with coarsening time. The total number of particles seem quite similar after 10 min of coarsening and then it decreases slightly after 30 min remaining constant for longer coarsening times. These observations

agree with the previous result on the nodularity of graphite in SGI shown in Fig. 14(a). The lower proportion of nodular particles leads to a lower nodularity of the sample.

Nodular graphite particles are the result of a separate nucleation and growth event during solidification of SGI. The nucleation of intermediate graphite particles compensates the slight decrease in the number of nodules. These intermediate particles are not considered as nodules due to their lower shape factor, although they have a similar nucleation mechanism. Despite the number of nodules decreasing slightly with coarsening time, the nucleation frequency of eutectic in SGI was not altered, showing a similar total number of particles. Thus, the changes in nodularity are connected to fading of Mg rather than the morphology of primary austenite.

A relationship between vermicular graphite particles and their nucleation frequency cannot be established for eutectic microstructures in CGI where eutectic austenite and vermicular graphite grow cooperatively as eutectic cells [2]. Therefore, the number of eutectic cells is similar to the number of nucleation events occurring during the eutectic reaction. Thus, to study the possible influence of the morphology of primary austenite on the nucleation of eutectic cells in CGI, the number of eutectic cells is measured for different coarsening times radial sections of the color-etched CGI samples, Fig. 15.

The number of eutectic cells in these radial sections as a function of coarsening time is shown in Fig. 16. The number of eutectic cells is not significantly influenced by coarsening time. This indicates that the number of eutectic cells and, thus, the nucleation frequency, in CGI, are not significantly influenced by the morphology of primary austenite.

The similar number of nodular graphite particles and eutectic cells as a function of coarsening time indicates that nucleation is not affected by the morphology of primary austenite and hence by the size and morphology of the interdendritic region. These observations suggest that the nucleation in CGI and SGI, and the growth of eutectic microstructures in CGI, are not significantly affected by the morphology of primary austenite.

These results are in contradiction with previous literature [63], reporting a correlation between the SDAS of primary austenite and the size of eutectic cells in LGI. The reason for this disagreement can be found in the difference in cooling conditions applied. In the experiments by Elmquist and Diószegi [63], the cooling conditions were constant throughout solidification, thus promoting the coarsening of both the primary and eutectic microstructures. In the current study, the coarsening of primary austenite occurs under isothermal conditions, i.e., decoupled from the eutectic reaction, while eutectic solidification occurs under similar cooling conditions in the CFCE reported in this study.

The similar nodularity and morphology of graphite particles for all the CGI samples indicated that the morphology of eutectic microstructures is not influenced by the coarsening of primary austenite. The decrease in the number of nodular graphite particles and hence in nodularity in SGI samples with increasing coarsening times could be explained by Mg fading. Previous studies report a higher rate of Mg fade with higher Mg content in the alloy [37], as is the case for SGI in this work. The isothermal coarsening treatment at 1161 °C in SGI promotes the fading of Mg while coarsening of primary austenite occurs. However, the fading of Mg is lower in CGI alloys, showing an asymptotic behavior until a sudden drop occurs resulting in the appearance of LGI [37, 39]. This also explains the unaffected nodularity for eutectic microstructures in CGI as Mg fading is almost negligible during the isothermal coarsening treatment. Thus, the reason for the slight variation in the nodularity of SGI is not associated with the morphology of primary austenite, but to the Mg fading in SGI.

Figure 15: Micrographs of etched samples from the CFCE for CGI after (a) 0 min, (b) 10 min, (c) 30 min, (d) 60 min and (e) 90 min of isothermal coarsening.

Figure 16: Number of eutectic cells in CGI as a function of coarsening time.

It is interesting that clearly separate near-spherical eutectic cells can be observed in all micrographs in Fig. 15. It is notable that the eutectic cells in many cases have not impinged on each other. This is contrasted by the behavior of spherical eutectic cells in Sr-modified Al-Si alloys [64]. The eutectic cells in the micrographs in Fig. 15 appear as if they were quenched during eutectic solidification. Instead of impinging, they are separated by regions with different color contrast. In these regions, there are graphite particles, austenite and intermetallics. This is somewhat similar to Cu-rich regions surrounding eutectic cells in Sr-modified Al-Si-Cu alloys.

Si is an important element to promote the solidification of Fe-C alloys through the stable diagram and ensure that the carbon-rich phase precipitates as graphite during the eutectic reaction [42]. Si segregates to primary austenite, and the Si content in the liquid decreases during solidification. This results in a partition coefficient, $k = \frac{C_S^{Si}}{C_L^{Si}}$, higher than 1 [65-67]. Hence, it is critical to verify that Si segregation is not significantly altered as a result of the isothermal coarsening treatment because it could influence the eutectic reaction. EDS analysis is used to describe the Si distribution in the quenched samples of LGI from the ICE. In the EDS maps of the samples quenched at 0 and 30 min of coarsening, Fig 17(a-b), we can observe a higher Si content in the primary austenite. These results confirm that $k > 1$ and back diffusion is insignificant. It should be pointed out that while Si is enriched in the austenite, with some Si remaining in the liquid and finally appearing on the eutectic cell boundaries, i.e., last liquid to solidify [68].

Figure 17: EDS maps showing Si distribution in the quenched samples from ICE in LGI after (a) 0 min and (b) 30 min of isothermal treatment. RD and RA stand for Radius Direction and Rotational Axis of the sample, respectively.

4. Conclusions

The evolution of primary austenite morphology during isothermal coarsening has been studied in the three main Fe-C-Si alloys used in industry, LGI, CGI, and SGI. The morphological evolution of the dendritic microstructure is characterized by quantitative stereological two-dimensional parameters and EBSD analysis. Furthermore, the influence of primary austenite morphology on the eutectic microstructure is analyzed. The main findings are:

- The coarsening of primary austenite follows similar kinetics for the three alloys. The surface area of primary austenite is reduced while the overall length-scale increases according to the Ostwald ripening model. Coarsening occurs by fragmentation and coalescence of primary austenite crystals, confirmed by EBSD analysis in LGI.
- The morphological parameters, SDAS, M_V , D_{ID}^{hyd} , and D_V showed a linear relation with the cube root of coarsening time, $t^{1/3}$, with similar rates for the three different

Fe-C-Si alloys; LGI, CGI and SGI. These observations suggest that the morphology of primary austenite is not influenced by the presence of surface-active elements, such as O.

- On the contrary, the presence of O controls the morphology of the graphite. As demonstrated in this study, using a common base SGI, we can tailor the final morphology of graphite and produce nodularity on demand by controlling the fading of Mg and thus the O in the alloy.
- The fraction of graphite, distribution of graphite particles and nodularity are not significantly altered as a function of coarsening time. The differences found in these parameters are associated with the fading of Mg indicating the weak effect of primary austenite morphology on the growth of graphite.
- In the case of SGI, nodularity shows a slight decrease with isothermal coarsening time. This seems associated with a faster fading of Mg for the higher Mg contents in SGI alloys as previously reported in the literature [37].
- The number of nodules in SGI and eutectic cells in CGI as a function of coarsening time indicate that nucleation frequency of eutectic microstructures in CGI and SGI are not influenced by the size of the interdendritic regions, associated with the primary austenite morphology.

5. Acknowledgments

This research was financed by VINNOVA, the Swedish Agency for Innovation, through the research projects CastDesign, grant number (2013-03303) and SPOFIC II, grant number (2013-04720), and KK-Stiftelsen through the research project LeanCast, grant number (2018-0033). The projects are a collaboration between Scania CV AB, Volvo Group Trucks Operation, SinterCast, Swerea SWECAST, and Jönköping University. All support and participating personnel from the above institutions are gratefully acknowledged by the authors. Special acknowledgment is directed towards Isabel María Medina Agudo for her contribution to the experimental work reported in this study.

Conflict of interest

The authors have no conflict of interest to declare.

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