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43 Article

44 **Microstructure in non-standard heavy section ductile iron castings: influence of solidification time and**
45 **casting size**

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52 **Abstract**

53 Ductile iron (DI) represents an optimal solution for saving material and costs in producing large
54 heavy-section castings in the energy sector. It aimed to investigate the influence of very long
55 solidification time (3, 10 and 20 h) in different casting zones (casting center and transition zone) on
56 the microstructure and mechanical properties of non-standard heavy-section ferritic DI (EN-GJS-
57 400-15) castings. The different solidification conditions significantly influenced the microstructure
58 (graphite and ferrous matrix). The extent of phenomena such as degenerate graphite, solidification
59 defects, hard carbides, and intergranular pearlitic areas, and the microstructural coarsening were
60 proportional to the solidification time and attributable to the combined effect of limited undercooling,
61 solid solution diffusion mechanisms, and segregation phenomena. For comparable solidification
62 time, the transition zone was characterized by larger nodules, comparable nodularity, and lower
63 nodule count than the casting center due to more effective diffusion phenomena during cooling.
64 Moreover, the lower segregation phenomena in the transition zone reduced the amount of perlite and
65 carbides in the intercellular zones. Hardness was only slightly influenced by the different
66 solidification conditions and did not represent a reliable indicator of the microstructural
67 inhomogeneities. These results are essential to refine casting simulations for producing large ferritic
68 DI castings, considering the wide microstructural variability within non-standard heavy-section
69 castings caused by significantly different solidification conditions.

70 **Keywords:** Heavy-section casting; Ferritic ductile iron; Microstructure; Solidification defects;
71 Graphite degeneration; Hardness

72 **1 Introduction**

73 Today, industrial design and production are oriented towards reducing the environmental impact of mechanical
74 components and require materials suitable for this purpose. Ductile irons (DI), known as nodular irons or spheroidal

75 graphite irons, offer sustainable and cost-effective solutions for various industrial sectors, such as automotive, heavy
76 industry, and energy. The low pouring temperature and high recyclability reduce the consumption of energy and
77 raw materials and, consequently, the environmental impact of their industrial production [1–2]. Furthermore,
78 excellent castability, good mechanical properties (high tensile strength, good wear resistance, and ductility), and
79 lower cost compared to steels with comparable mechanical properties make DI an effective alternative for producing
80 large structural parts with complex geometry, such as railway brake discs, drive shafts, heavy-duty wheels, and
81 gears, without using joints or welding [3, 4]. For these reasons, DI have experienced continuous global growth since
82 1965 (the only case with aluminum alloys among casting materials), which is expected to continue, considering the
83 growing use of DI for producing wind turbine components and nuclear storage barrels, encouraged by the ever-
84 increasing demand for renewable energy sources [5].

85 DI have a composite-like microstructure constituted by graphite nodules dispersed in a ferrous matrix (ferritic or
86 pearlitic), depending on chemical composition, casting conditions, and heat treatment. The long solidification times
87 of heavy-section ductile iron (HSDI) castings generally promote the formation of a ferritic matrix due to a depletion
88 of carbon in the matrix in favor of the growth of graphite nodules [6, 7]. The complex solidification conditions that
89 occur in HSDI castings induce strong microstructural variability and have a negative impact on the mechanical
90 properties (strength, ductility, fatigue resistance, and impact resistance) of the alloy, making the design of large
91 structural components difficult [8]. In particular, long solidification times increase the risk of forming defects such
92 as non-metallic inclusions, shrinkage voids, undesired segregation phenomena of low-melting elements and carbide
93 precipitation at grain boundaries, or degenerated graphite, such as chunky graphite, spiky graphite, exploded
94 graphite, and compacted graphite [9–11]. The HSDI castings present a high sensitivity to tramp elements deriving
95 from recycled steel scrap used as filler materials for DI melting since the long solidification times favor the local
96 segregation of these elements at the grain boundaries, thus worsening the mechanical behavior of the alloy [4, 12].
97 This is significant because the content of undesirable elements in steel scrap, such as Cr, Mn, Mo, Nb, and V, is
98 expected to increase in the coming years due to the growing content of alloying elements in steels to obtain higher
99 mechanical properties [12–14].

100 Over the last five years, several studies were conducted to identify possible solutions (modification of the
101 chemical composition, identification of possible correlations between solidification time, microstructure and
102 mechanical properties, optimization of heat treatments) to counteract the decrease in mechanical performance due
103 to the complex solidification conditions in ferritic HSDI castings, given the growing interest in their use in the
104 ecological transition. Feng et al. [15] highlighted the positive effect of Bi in eliminating coarse graphite caused by
105 the degeneration of the excessive spheroidizing inoculation in HSDI, while Yamamoto et al. [16] showed the
106 beneficial effect of Fe–Si–Ca–Zr and Fe–Si–Ca–Ba post-inoculants in increasing the nodule count and impact
107 toughness of ferritic HSDI. Sertucha et al. [17] showed the positive effects of the combined use of Sn, Sb, and rare
108 earths (RE) to decrease chunky graphite appearance, reducing its negative impact on the room temperature
109 mechanical properties of small castings, with a controlled solidification procedure to simulate the solidification
110 conditions in the HSDI castings.

111 Borsato et al. [9] showed the combined effect of poor inoculation treatment and long solidification time (3 h) on
112 the mechanical properties of the EN GJS 700-2, which led to a decrease in static and cyclic mechanical properties
113 due to a significant increase in grain size, graphite degeneration, segregation of alloying elements and shrinkage
114 voids. Similarly, Benedetti et al. [1] reported an increase in microstructural anomalies (shrinkage voids and graphite
115 degeneration) in the EN-GJS-400-15 by increasing the solidification time (from 2 h 45 min to 12 h) and a consequent
116 decrease in the ultimate tensile strength, elongation to failure and rotating bending fatigue strength by 27.2%, 73.6%,
117 and 30.7%, respectively. A comparable trend was reported by Borsato et al. [10] for a solution-strengthened ferritic
118 DI: the fatigue strength was reduced by 33% when the solidification time was increased from 10 min to 10 h due to
119 an increase in the number and size of defects, such as graphite degeneration and shrinkage porosities, which acted
120 as crack initiation sites.

121 Other studies emphasized the effects of heat treatments on HSDI castings. Tong et al. [18] evaluated the effects
122 of a series of quenching-tempering treatments on ferritic ductile iron poured into a sand mold of wall thickness >
123 110 mm, showing the best mechanical properties after austenizing at 880 °C, quenching, and tempering at 570 °C
124 due to the formation of granular pearlite in the matrix. Gundlach et al. [19] demonstrated the efficiency of the air-
125 cooling from an intercritical (IC) temperature in increasing the strength and ductility of ductile iron in sections up
126 to 250 mm, owing to the formation of an acicular pearlite in ferrite structure.

127 However, the latest studies on HSDI castings were conducted on small batches of laboratory castings that
128 reproduce long solidification times or samples extracted from Y-blocks, which are hardly capable of reproducing
129 the real production conditions of very large HSDI castings. As reported by several authors [20–22], significant
130 differences in graphite nodule and ferrous matrix characteristics were found when experimental samples were
131 extracted from different zones of large castings compared to samples produced under controlled experimental
132 solidification conditions. The microstructure and mechanical properties of non-standard-sized HSDI castings can,
133 in fact, significantly deviate from data obtained from small castings under near-ideal controlled solidification
134 conditions obtained in the laboratory, which are only able to partially reproduce the heterogeneous microstructure
135 and typical defects found in large real components. Furthermore, no data about HSDI castings characterized by
136 sections greater than 200 mm are currently reported in the EN 1563:18 standard, representing a further limitation
137 for their design and use. This is where the need arises to obtain empirical data by directly studying the influence of
138 different solidification conditions in different areas of out-of-standard HSDI castings, thus enabling the
139 development of more accurate predictive solidification and mechanical behavior models [11, 23]. In this scenario,
140 this work aims to provide a starting point for the redefinition of the design rules of HSDI castings characterized by
141 wall thickness larger than 350 mm, currently absent in the literature, providing experimental data and valuable
142 solidification time-microstructure-defect correlations to develop future predictive models. In particular, the effect
143 of very long solidification times was directly evaluated on air-cooled heavy-section castings, thus differing from
144 previous studies [3, 6, 7, 9]. Ferritic DI castings (nominally, EN-GJS-400-15 according to EN 1563:18) were
145 produced in the form of cubes with sides in the range between 350 and 1000 mm to reproduce different solidification
146 times (from 3 to 20 h) in different casting zones (casting center and transition zone). Five Italian foundries were
147 involved in the research project to obtain representative results. This led to the production of castings being
148 independent from a specific foundry practice, strengthening the validity of the experimental data and differentiating

149 this work from what is currently reported in the literature [3, 6, 7, 9–11, 23], where the data were obtained from
150 experimental material provided by individual founders. In-depth microstructural characterization was performed
151 using optical and electron microscopy for an accurate microstructure-solidification time correlation. Finally,
152 hardness tests were carried out to assess the influence of long solidification times on the mechanical properties of
153 the alloy.

154

155 **2 Experimental**

156

157 **2.1 Casting production**

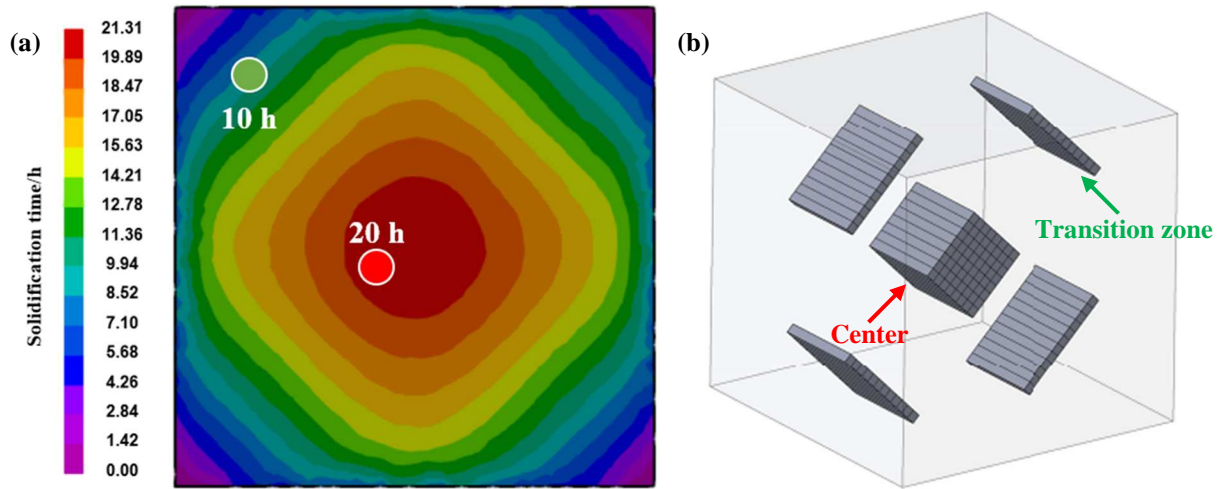
158 Five different foundries were involved in casting large-sided cubes: (i) 350 mm³, (ii) 700 mm³, and (iii) 1000 mm³.
159 The cube sizes were assessed by a preliminary numerical simulation according to target solidification times (3, 10
160 and 20 h) at the casting center, with a tolerance of $\pm 10\%$. For the sake of simplicity, the mold was considered to
161 have already been filled with liquid iron at a starting temperature of 1340 °C, therefore avoiding the simulation of
162 any casting system. Around the mold cavity, a sand thickness of 300 mm at an initial temperature of 20 °C was
163 considered (as in real conditions). The whole system (sand mold and iron casting) was modeled with finite elements
164 modeling (FEM). The FEM analysis divided the solid into 10 mm cubes, thus evaluating the centers of adjacent
165 cubes as single points and calculating their interaction via heat transfer equations. As in real conditions, no coolers
166 were considered. The cast iron and sand properties as a function of temperature were taken from the database of the
167 numerical code (Magmasoft©) used to make the casting model: the properties of the cast iron with C = 3.5 wt.%
168 and Si = 2.5 wt.% were chosen. Finally, the heat transfer coefficient (HTC) value between the sand and the alloy as
169 a function of temperature is given in Table 1. The values of this critical parameter are the result of the calibration
170 procedure and experience carried out over several years by the foundries that performed the simulations for this
171 work.

172 Table 1 Heat transfer coefficient (HTC) as a function of temperature used in simulation

T/°C	20	600	1100	1200	2000
HTC/(W m ⁻² K ⁻¹)	300	500	600	800	800

173 Material removal operations were performed to analyze the solidification conditions from blanks obtained by the
174 different zones of the cubes according to the casting simulation (Fig. 1). For each cube, the blanks were extracted
175 by saw cutting, without thermally altering the metallurgical state of the cast iron, and according to the cutting scheme
176 in Fig. 1b, to obtain samples representative of the solidification conditions reported in Table 2. The position of the
177 blanks was based on the solidification simulations within the tolerances mentioned above. The sectioning resulted
178 in the creation of 30 mm × 30 mm × 300 mm blanks from which the samples for the microstructural analyses were
179 obtained.

180



181

182 Fig. 1 Casting simulation of 1000 mm × 1000 mm × 1000 mm cube (a) and extraction zones (center and
 183 transition zone) of specimens from 1000 mm³ cube (b)

184 Table 2 Designation of analyzed conditions. Blanks are extracted from different areas of cubes to evaluate influence
 185 of solidification time and position on microstructure and mechanical properties of EN-GJS-400-15

Designation	Cube	Extraction zone	Solidification time
3C	350 mm × 350 mm × 350 mm	Center	3 h
10C	700 mm × 700 mm × 700 mm	Center	10 h
10T	1000 mm × 1000 mm × 1000 mm	Transition zone	10 h
20C	1000 mm × 1000 mm × 1000 mm	Center	20 h

186 The study of different solidification times at the center of massive castings aimed to evaluate their effects on the
 187 microstructure and defects. Additionally, as highlighted in Fig. 1 and Table 2, also blanks extracted from the
 188 transition zone of 1000 mm × 1000 mm × 1000 mm cubes (with a solidification time of 10 h) were studied to
 189 compare the solidified microstructures at the center of the cubes with those of areas characterized by comparable
 190 solidification times but different solidification conditions (transition zones).

191 The DI used for the castings was the ferritic EN-GJS-400-15 (according to EN 1563:18). Each foundry melted
 192 and poured a standard chemical composition (Table 3), using a comparable casting temperature (1340 °C), and an
 193 appropriate casting system. To avoid graphite degeneration, Sb was introduced in the following concentrations: $25 \pm 5 \times 10^{-6}$
 194 $\pm 5 \times 10^{-6}$ for the 350 mm side castings and $50 \pm 10 \times 10^{-6}$ for the remaining castings, while inoculation and
 195 spheroidization treatments were left to the experience of each foundry. After solidification of the castings, the
 196 chemical composition of each cube was verified by each foundry using optical emission spectroscopy (Table 3) to
 197 assess its compliance with the EN 1563:18 standard.

198 Table 3 Nominal chemical composition (wt.%) of EN-GJS-400-15 according to EN 1563:18 and average chemical
 199 composition of each cube (size 350, 700 and 1000 mm³), evaluated by optical emission spectroscopy

C	Si	Mn	Cu	Ni	Cr	Mo	Sn	V	Ti	S	P	Mg
---	----	----	----	----	----	----	----	---	----	---	---	----

Min	3.5	2.2	0.1	-	-	-	-	-	-	-	-	-	0.04
Max	3.7	2.5	0.3	0.1	0.02	0.04	0.02	0.004	0.015	0.02	0.01	0.04	0.06
350 mm ³	3.57 ± 0.03	2.36 ± 0.12	0.17 ± 0.05	0.07 ± 0.04	0.01 ± 0.01	0.03 ± 0.01	0.00	0.00	0.01 ± 0.005	0.01 ± 0.01	0.005 ± 0.005	0.034 ± 0.005	0.05 ± 0.01
700 mm ³	3.60 ± 0.05	2.31 ± 0.10	0.18 ± 0.04	0.08 ± 0.04	0.01 ± 0.01	0.03 ± 0.01	0.00	0.00	0.01 ± 0.005	0.01 ± 0.01	0.005 ± 0.005	0.035 ± 0.007	0.05 ± 0.01
1000 mm ³	3.59 ± 0.04	2.31 ± 0.10	0.18 ± 0.06	0.07 ± 0.05	0.02 ± 0.01	0.02 ± 0.01	0.00	0.00	0.01 ± 0.005	0.01 ± 0.01	0.005 ± 0.005	0.035 ± 0.008	0.05 ± 0.01

200 For the mold, silica sand was used in sufficient quantity to cover the casting. Metal brackets held the sand mold
201 shape during casting, and coolers and feeders were avoided. The removal of the brackets occurred when a surface
202 temperature of 400 °C was reached, according to the cooling simulations (Table 4).

203 Table 4 Times required for each analyzed cube to reach surface temperatures defined by simulation data

Cube	400 °C	300 °C
350 mm × 350 mm × 350 mm	34 h	42 h
700 mm × 700 mm × 700 mm	109 h	131 h
1000 mm × 1000 mm × 1000 mm	170 h	200 h

204 **2.2 Microstructural and mechanical characterization**

205 Fifteen samples for each condition (three samples for each of the five foundries) were extracted by saw cutting;
206 attention was paid to avoid thermal alteration of the microstructure. Microstructural analyses were carried out on
207 the samples using optical microscopy (OM) and field emission-gun scanning electron (FEG-SEM) microscopy,
208 equipped with energy-dispersive X-ray spectroscopy (EDS). Metallographic samples were embedded in conductive
209 resin, ground by emery papers from 180 grit up to 1200 grit, and polished by diamond suspensions from 9 to 1 μm,
210 according to the ASTM E3 standard. Finally, metallographic samples were etched with 4% Nital reagent (4 mL
211 HNO₃-96 mL ethyl alcohol) according to the ASTM E407 standard. Image analysis was done using ImageJ software
212 to identify nodularity, *N* (%), nodule count, *NC* (units/mm²), nodule size, *NS* (mm²), ferritic grain size, *FG*
213 (intercepts/mm), and pearlitic area, *PA* (%). Analysis areas for graphite characterization were carried out
214 considering the size of the graphite nodules due to the long solidification times. Three different images were
215 captured per sample, each measuring 3.2 mm × 5.7 mm, for a total analysis area per sample of approximately 55
216 mm². For the graphite analysis, the ISO 945-4:2019 standard was applied as far as possible, considering no

217 references for sections larger than 200 mm. In particular, nodularity for each area was evaluated according to Eq.
 218 (1):

$$219 \quad Nod = \frac{A_V + A_{VI}}{A_{all}} \quad (1)$$

220 where *Nod* is nodularity, in %; A_V and A_{VI} represent the total area of graphite particles with roundness ≥ 0.6 to ≤ 1.0
 221 (only form V and VI particles by ISO 945-4:2019 are included as spheroidal graphite particles in the nodularity
 222 calculation); and A_{all} is the total area of all graphite particles that meet the size criteria, excluding the particles that
 223 intersect the border of the area of analysis. Roundness is a shape factor calculated as the area of a graphite particle
 224 divided by the area of the circle where the diameter is the maximum Féret diameter of the same graphite particle.

225 Nodules along the edges and nodules characterized by a Féret diameter less than 30 μm (class 6 and lower
 226 according to the ISO 945-4:2019 standard) were excluded from the calculation of nodularity and average nodule
 227 size. Based on the magnification applied for quantitative analysis, this threshold was set to eliminate measurement
 228 errors introduced by scratches, noise, and small pores. Only the particles with a minimum roundness equal to 0.6
 229 (according to the ISO 945-4:2019 standard) were analyzed for nodule count. Furthermore, areas with degenerated
 230 graphite were manually counted as degenerate and excluded from the images to facilitate automated analysis of the
 231 nodules.

232 According to the ASTM E112-13 (2021) standard, the intercept method was used to measure the ferritic grain
 233 dimension. The pearlitic area was evaluated using the percentage of occupied area within the analysis area.

234 Brinell hardness (HB30) was carried out according to the ASTM E10-18 standard with a 2.5-mm steel ball, 187.5
 235 kg load, and dwell time of 10 s to assess the correlation between solidification time and mechanical properties.
 236 Hardness was evaluated on each specimen and calculated as the average of at least three measurements for each
 237 sample.

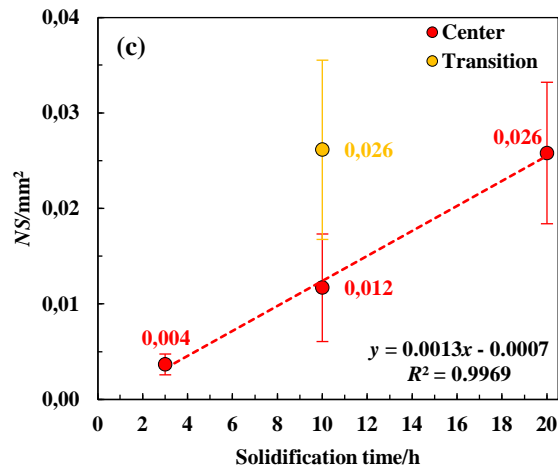
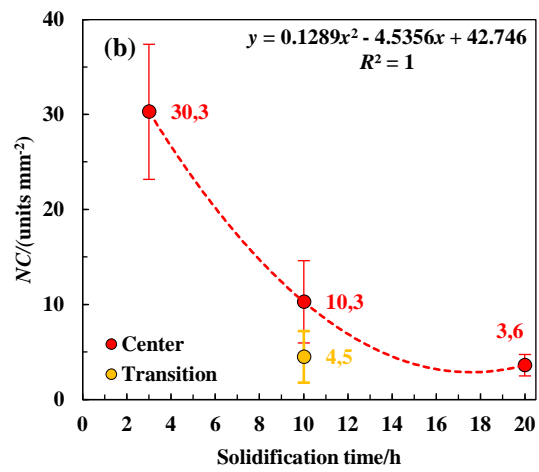
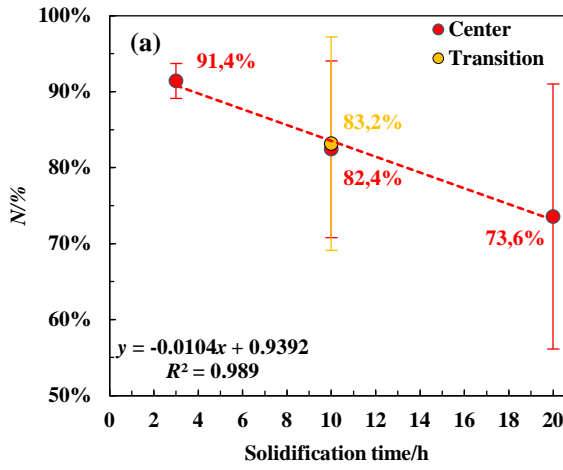
238 **3 Results and discussion**

239 Significant microstructural differences were observed between the different solidification conditions
 240 (solidification time: 3, 10 and 20 h, and casting position: casting center (C), and transition zone (T)) on the
 241 microstructural characteristics of the EN-GJS-400-15, as summarized in Table 5 and Fig. 2 and highlighted by
 242 representative microstructures shown in Figs. 3 and 4.

243 Table 5 Results of image analysis measurements carried out on specimens characterized by different solidification
 244 conditions: *N* (%), *NC* (units/mm²), *NS* (mm²), *PA* (%) and *FG* (intercepts/mm)

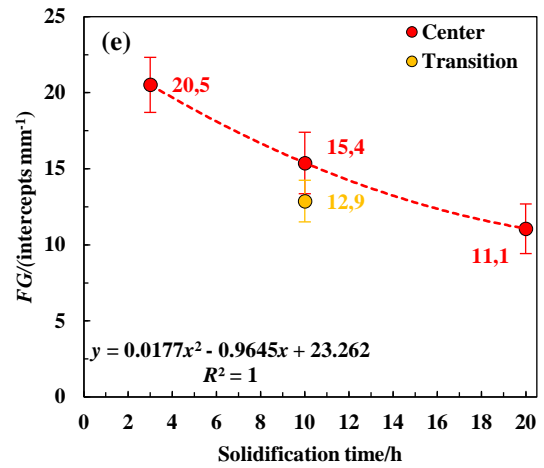
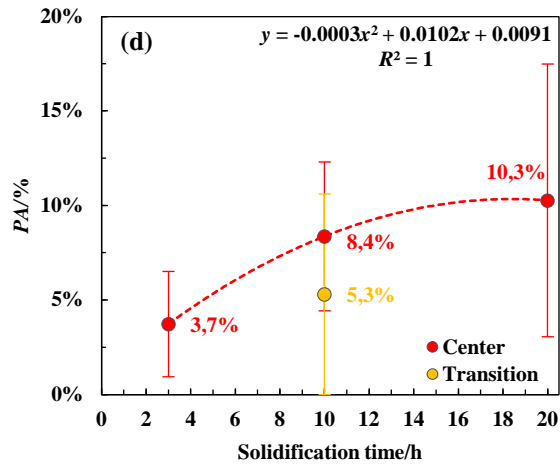
3C			10C			10T			20C	
Min	Max	Average	Min	Max	Average	Min	Max	Average	Min	Max
88.9	95.6	91.4 ± 2.3	57.1	90.7	82.4 ± 11.6	50.2	95.8	83.2 ± 14.0	43.3	91.1

units	22.6	41.0	30.3 ± 7.1	5.3	19.0	10.3 ± 4.3	1.7	12.2	4.5 ± 2.8	2.0	6.3	3
mm ²	0.002	0.005	0.004 ± 0.001	0.006	0.017	0.012 ± 0.003	0.010	0.039	0.026 ± 0.009	0.019	0.036	0.0
%	0.0	7.9	3.7 ± 2.8	3.3	18.3	8.4 ± 3.9	0.0	13.8	5.3 ± 4.1	1.5	25.2	7.2
intercepts	19.1	23.1	20.5 ± 1.4	11.4	19.5	15.4 ± 2.0	10.5	15.7	12.9 ± 1.4	7.9	13.6	1
												1.6



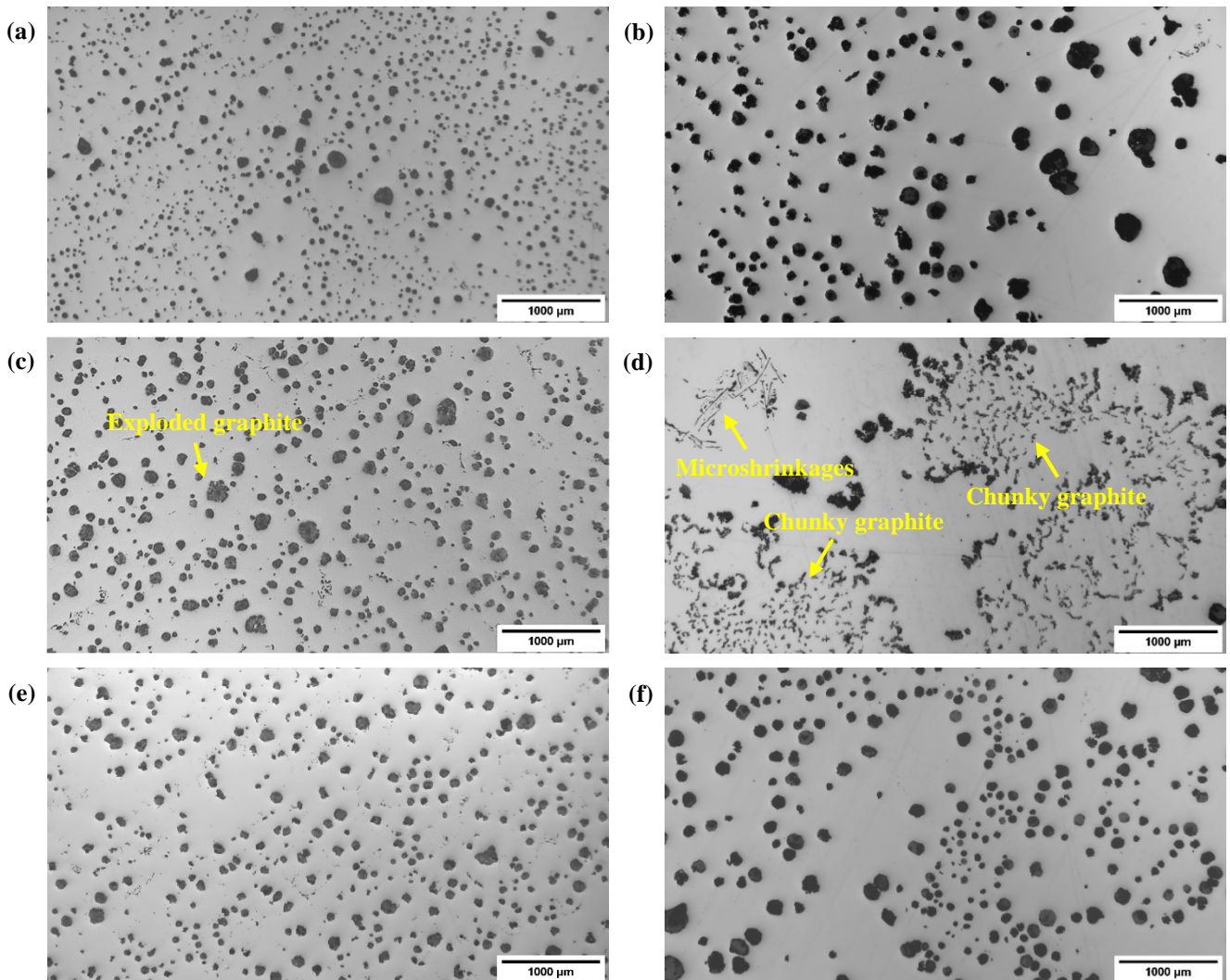
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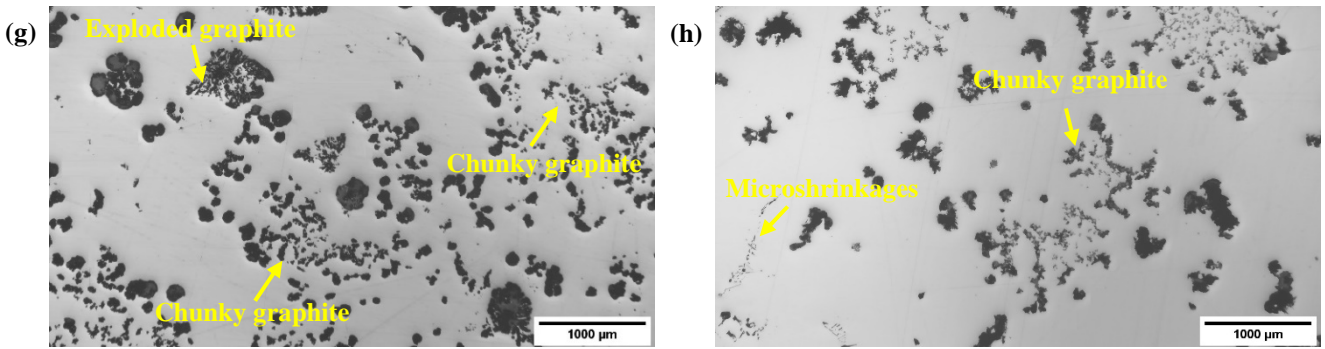
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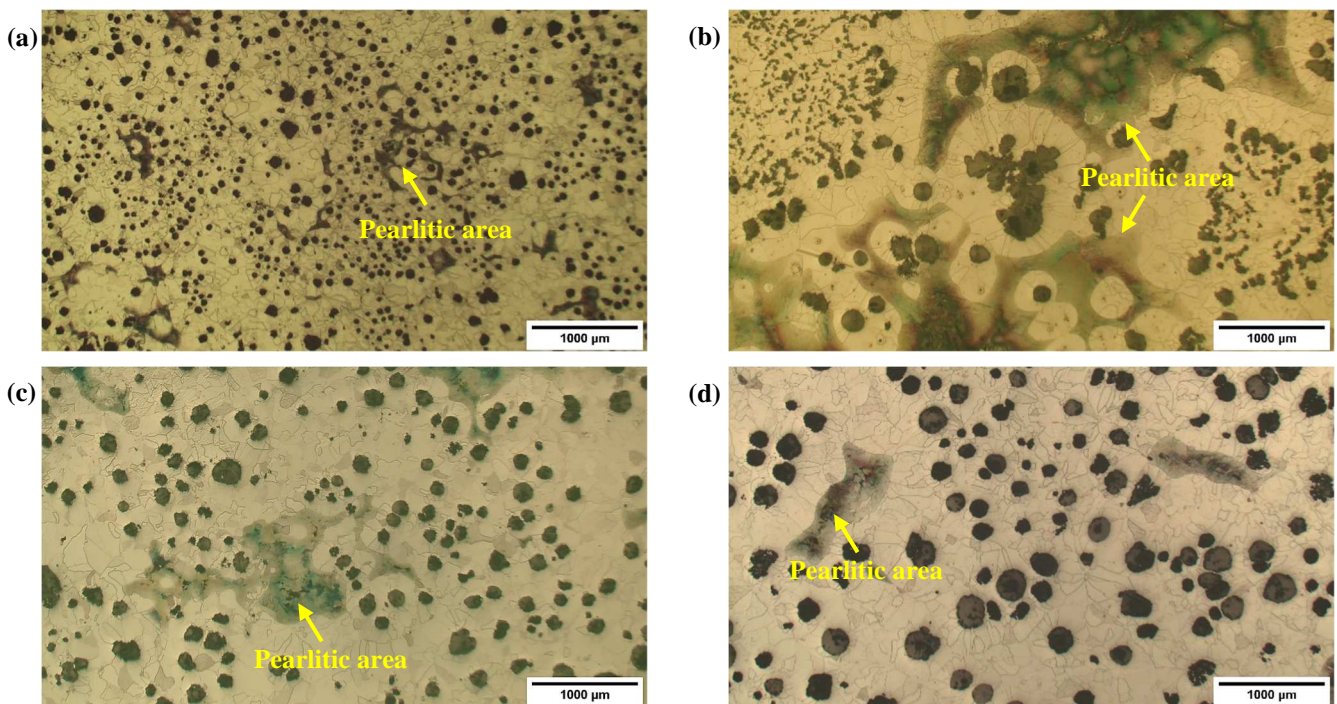
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248 Fig. 2 Effects of different solidification times (3, 10 and 20 h) and casting positions (casting center and transition
 249 zone) on microstructural characteristics of EN-GJS-400-15. **a** Nodularity; **b** nodule count; **c** nodule size; **d** pearlitic
 250 area extent; **e** ferritic grain size. R^2 , referred to as the square of the correlation, measures the proportion of variation
 251 in the dependent variable that can be attributed to the independent variable.





252 Fig. 3 EN-GJS-400-15 microstructures from solidified samples under different solidification conditions. **a, c** 3C
 253 condition; **b, d** 20C condition; **e, g** 10C condition; **f, h** 10T condition. The first image of each solidification condition
 254 shows the best identified microstructural features, the second one the worst.



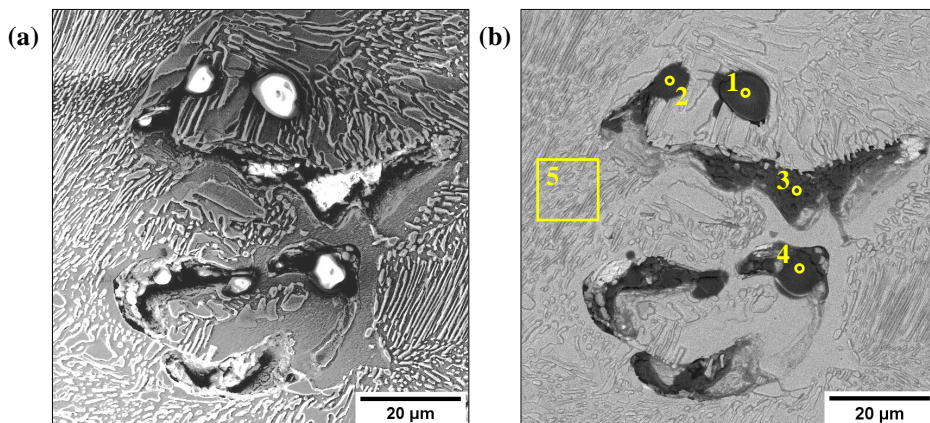
255 Fig. 4 EN-GJS-400-15 microstructures from solidified samples under different solidification conditions. **a** 3C
 256 condition; **b** 20C condition; **c** 10C condition; **d** 10T condition

257 As shown in Fig. 2, the microstructural characteristics of the casting center (in terms of graphite nodules and
 258 ferrous matrix) strongly depend on the increase in solidification times, as also described in the literature for thinner
 259 wall thicknesses [10, 24–26].

260 Focusing on the casting center position (C condition), the increase in the solidification time (from 3 to 20 h) leads
 261 to a decrease in NC (from 30.3 to 3.6 nodules per mm^2) and an increase in NS (from 0.004 to 0.026 mm^2) (Fig. 2a,
 262 c). At the same time, as solidification time increases, the morphology of the nodules “degenerates” as a result of the
 263 formation of exploded and chunky graphite (Fig. 3d, h), with a decrease in N from 91.4% of the 3C condition to
 264 73.6% of the 20C condition (Fig. 2b). Longer solidification and cooling times (Table 4) also lead to an increase in

265 inhomogeneity in terms of morphology and size of the graphite nodules, with very marked differences between
 266 different samples under the same solidification conditions (time and position) (Table 5 and Fig. 2). This result is
 267 also due to the production of castings by different foundries, which amplified the microstructural variability of the
 268 samples under the same solidification conditions due to the complexity of the nucleation and segregation
 269 phenomena. Large nodules are associated with primary graphite growth due to solid solution diffusion mechanisms
 270 below the eutectic temperature [22]; prolonged exposure to high temperatures due to extremely long cooling times
 271 (Table 4) leads to continued growth of primary nodules. However, the asymptotic value of NC equal to 3.6 nodules
 272 per mm^2 may indicate a limit in the increase in nodule size imposed by the solid solution diffusion of C atoms and
 273 the consequent achievement of the maximum mean nearest neighbor distance between nodules, as well as to a limit
 274 in obtaining nodules without generating degenerate graphite.

275 The degenerate graphite, observable for 10 and 20 h of solidification time (Fig. 3d, g, h), results from the
 276 combined effect of limited undercooling and increased liquid supersaturation, negatively influencing the nodule
 277 growth mechanisms. As described by Källbom et al. [25], the solid solubility of rare earth (RE), Mg, S, and O in
 278 austenite is negligible; therefore, these elements gradually enrich the liquid at the solidification front, thus
 279 influencing the growth of the eutectic graphite. On the one hand, the nodulizing effect of Mg and RE makes the
 280 growth of eutectic graphite possible along the crystalline orientation $\langle 0\ 0\ 0\ 1 \rangle$, spheroidizing the graphite particles;
 281 on the other hand, S, O, and excessive Mg and RE content promote the branching of eutectic graphite, which
 282 assumes the chunky or exploded morphology (Fig. 3d, g, h). This condition was favored in the center of the casting,
 283 where the advancement of the solidification front led to a continuous enrichment of the liquid melt in Mg or RE and
 284 the subsequent degeneration of the graphite nodules. An example is the presence of distinct endogenous inclusions
 285 such as magnesium oxides (MgO) in the intercellular region (Fig. 5) formed during solidification due to the
 286 continuous enrichment of Mg in the residual melt and its reaction with dissolved O. The action of O is doubly
 287 damaging: on the one hand, it traps Mg and favors the formation of degenerate graphite; on the other hand, it forms
 288 non-metallic MgO compounds that worsen the mechanical properties of the castings. Therefore, it is essential to
 289 control the MgO formation during pouring.



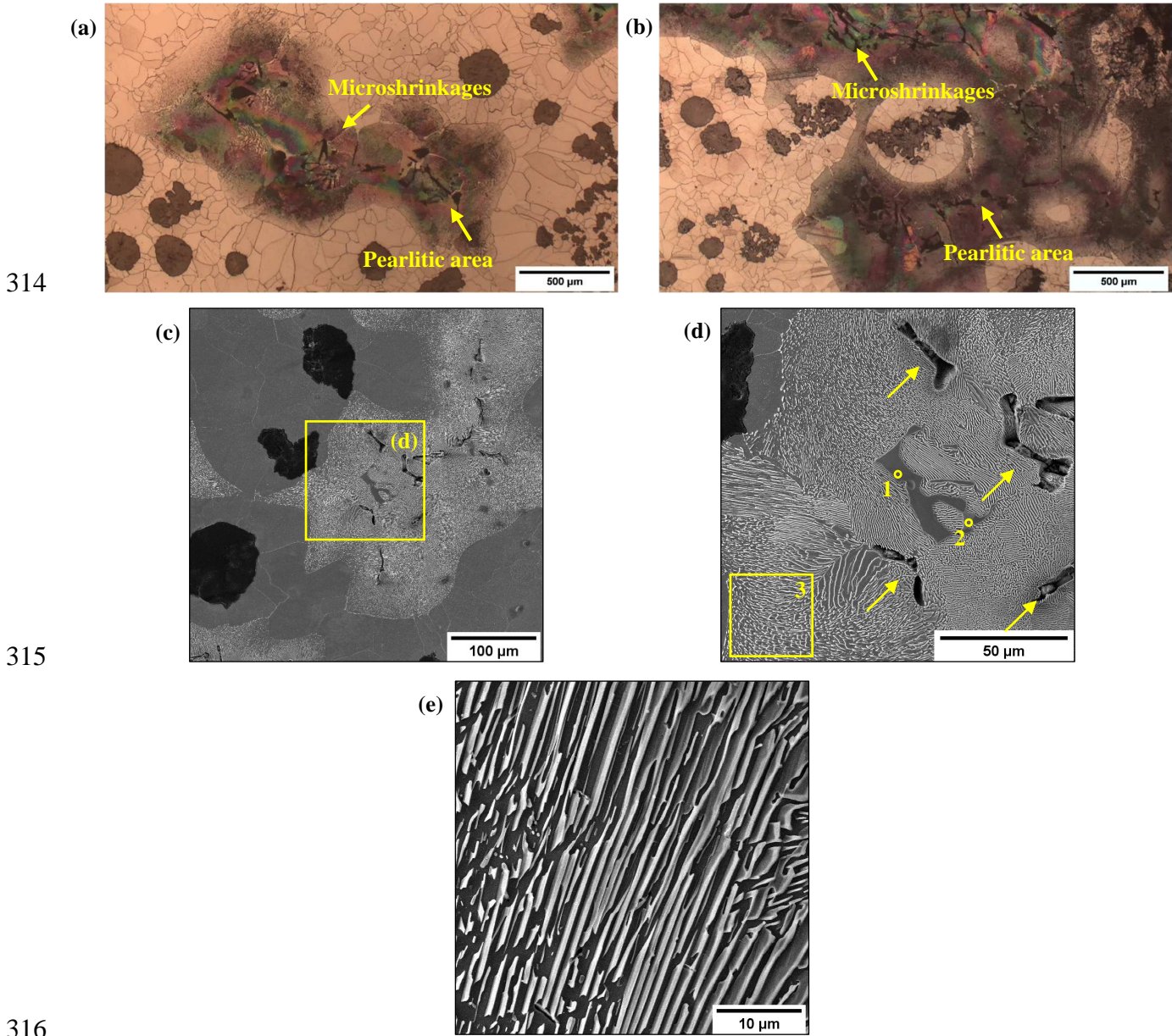
Normalized atomic concentration/%	C	O	Mg	Si	P	S	Fe
1	18.0	57.4	21.0	0.0	1.9	0.0	1.7

2	20.8	59.0	17.7	0.0	2.2	0.1	0.2
3	20.2	56.8	19.7	0.1	1.2	0.2	1.8
4	20.8	58.8	19.1	0.0	0.4	0.0	0.9
5	23.6	1.6	0.0	1.6	0.0	0.0	73.2

290 Fig. 5 High magnification SE (secondary electrons) (a) and BSE (Back-scattered Electrons) (b) images of MgO
 291 inclusions in intercellular regions of a 20C sample. EDS analysis of panel “5” in (b) shows composition of pearlite
 292 (ferrite and cementite lamellae, Fe₃C)

293 For atomically smooth (faceted) interfaces, such as graphite, the spheroidization process requires large driving
 294 forces that necessitate high undercooling and supersaturation of the nodulizing elements in the liquid melt [5]. As
 295 the wall thickness and solidification times increase, the driving forces become too low to promote spheroidal
 296 graphite. However, they are sufficient to form lamellar graphite, as also reported in [21, 27], resulting in a spiral
 297 growth mechanism on the “c” axis of graphite crystals capable of forming dendritic (coarse and exploded) graphite,
 298 observable in Fig. 3d, g, h as aggregates or clusters of graphite particles. The distribution of the chunky graphite is
 299 not uniform, and it is possible to identify fine chunky graphite (Fig. 3d) and coarse chunky graphite (Fig. 3g)
 300 depending on the solidification time and extension of the affected areas.

301 An additional factor that increases the formation of degenerate graphite is the increase in the content of interfering
 302 elements in the intercellular zones, which favors the formation of blocky graphite (Al, Ca, Ce, Ni, and Si) or lamellar
 303 graphite (As, B, Bi, Cu, Pb, Sb, and Sn) [28, 29]. In particular, as the solidification time increases, the interfering
 304 elements have enough time to diffuse onto the boundary surface of the graphite melt, thus interrupting the
 305 spheroidization of graphite and promoting another graphite morphology. Comparable mechanisms led to a three-
 306 fold increase in the *PA* extension, moving from the 3C to the 20C condition. As solidification times increase, the
 307 transformation from austenite to ferrite is promoted by C atom diffusion, thus limiting the formation of pearlitic
 308 zones in small and homogeneously distributed intergranular areas in the ferritic matrix (Fig. 4a). However, longer
 309 solidification time facilitated segregation phenomena and the accumulation of pearlite promoter elements in the
 310 liquid melt, such as Cr and Mn, leading to the formation of large intergranular pearlitic areas within a coarse ferritic
 311 matrix, and microshrinkages promoted by the precipitation of carbides (Fig. 6a, b). HSDI castings were also affected
 312 by a significant coarsening of the ferritic matrix linearly dependent on the solidification time (Fig. 2e): moving from
 313 the 3C to the 20C condition, the increase in *FG* is equal to 45.8%.

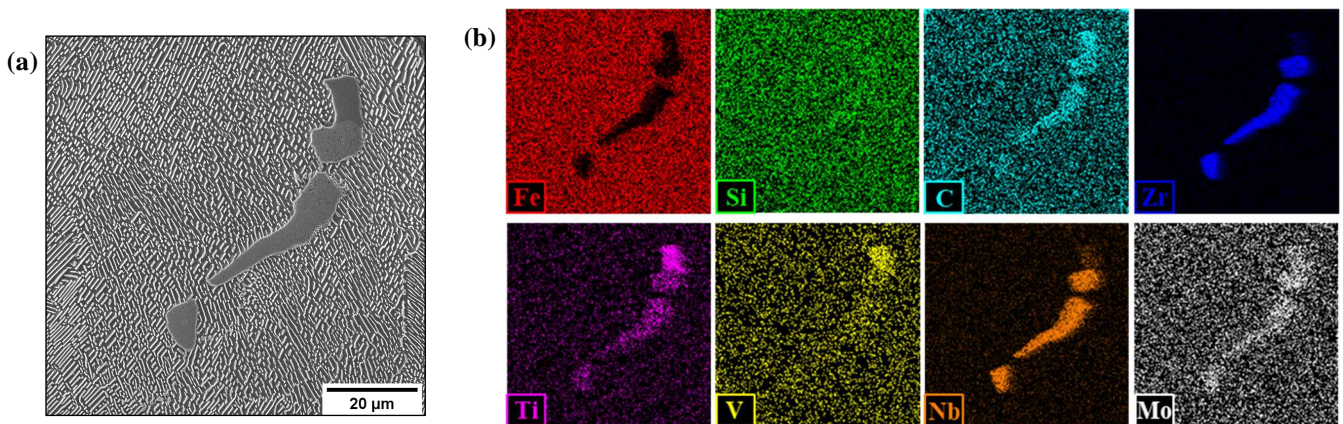


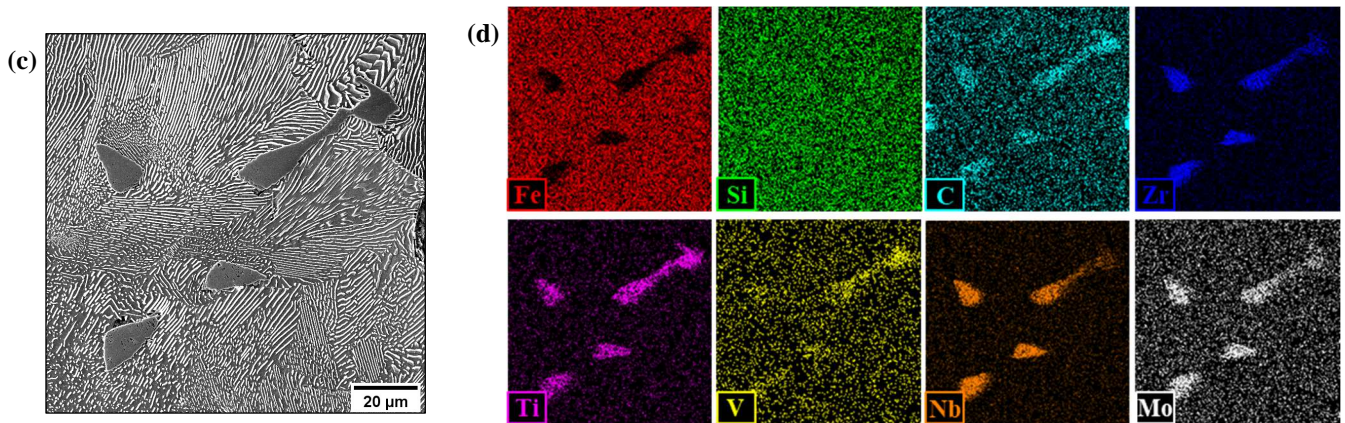
316

	C	Mg	Si	P	S	Ti	V	Mn	Fe	Cr	Cu	Zr	Nb
1	49.8	0.04	0.00	0.01	0.00	31.6	10.6	0.00	1.6	0.00	0.01	2.51	2.45
%	50.3	0.04	0.09	0.00	0.00	12.4	3.34	0.03	3.6	0.00	0.12	27.9	1.41
	21.4	0.03	1.54	0.00	0.00	0.01	0.24	1.16	74.9	0.52	0.14	0.00	0.00

317 Fig. 6 Microshrinkages in correspondence with pearlitic area in 10C (a) and 20C samples (b). High magnification
 318 SE images and EDS analysis of intercellular phases segregated in intercellular regions. c Pearlitic island; d micro
 319 shrinkages (yellow arrows) and complex (Ti Zr, and V) carbide (points 1 and 2) embedded into a pearlitic structure;
 320 e detail of pearlitic lamellar structure. EDS analysis of box "3" in (d) shows composition of pearlite (lamellas of
 321 ferrite and cementite, Fe_3C)

322 Hard carbides are present in the intercellular pearlitic regions due to the reduced solubility of carbide-promoting
323 elements in austenite. The area fraction of pearlite and the number of carbides strongly depend on the chemical
324 composition and segregation of pearlite- and carbide-promoting elements in the remaining liquid during eutectic
325 solidification. In particular, Ti, V, Mo, and Nb are present in small amounts in common ferrous charge materials
326 and are typical residual elements in HSDI castings. Although present in very low quantities in the furnace charge,
327 they strongly segregate at the cell boundaries during austenite solidification from the liquid phase, reaching
328 concentrations up to 1000 times higher than those allowed in the nominal concentration (Table 3), thus promoting
329 the formation of Ti-, V-, Mo-, and Nb-based carbides (Fig. 7). In addition, the long solidification times strongly
330 impact nodule density (Table 5 and Fig. 2) and thus the diffusion distance for C and other elements during
331 solidification and cooling in the solid state. This is particularly true under the 10C, 10T, and 20C conditions, where
332 a more pronounced segregation of the promoting elements was favored, increasing the area fraction of carbides
333 formed at the end of solidification. During DI solidification, carbide precipitation can occur directly from the liquid,
334 at the early stage, or at the end of austenite solidification. Considering the long solidification times, the low content
335 of carbide promoters in the chemical composition (Table 3), and the position of the carbides in the microstructure
336 at the intergranular region, embedded into pearlitic regions, the carbide precipitation could mainly occur in the last
337 part of the austenite solidification due to very significant segregation phenomena of carbide promoter elements
338 toward the remaining liquid during eutectic solidification [13]. The analysis highlighted a higher carbide content
339 and microshrinkages in the pearlitic areas of the 10C and 20C samples, characterized by a more extensive pearlitic
340 area. Another result that can be observed from Figs. 4b and 6a, b is the proximity of a large number of carbides
341 around the degenerate graphite. As described in [13, 30], the formation of carbides hinders the diffusion of C atoms
342 from austenite to graphite through the ferritic shell, favoring the formation of chunky graphite due to the kinetic
343 disadvantage for the growth of nodules and the thermodynamic conditions for the graphite nucleation at the
344 austenite-liquid interface.

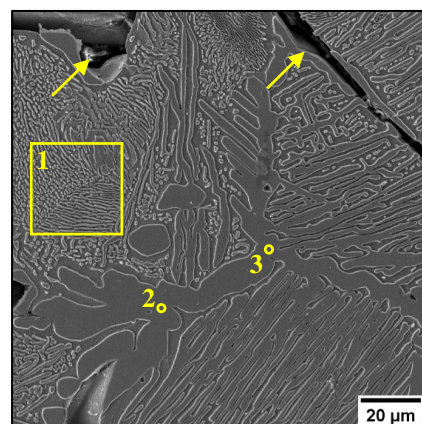




345 Fig. 7 SE images (a, c) and EDS maps (b, d) of Ti-, V-, Mo-, Zr-, and Nb-based carbides embedded into a pearlitic
 346 structure

347 In conclusion, segregation phenomena of minor elements at the interface (carbide formers such as Cr, Ti, and V,
 348 pearlite promoters such as Mn and Cr, nodularizer and inoculant elements such as Mg, Zr, and RE) induced the
 349 formation of large intergranular pearlitic islands in the ferritic matrix (Fig. 6a–e). In addition to “single” carbides,
 350 high-entropy carbides are identified in the intercellular regions (Fig. 7); as described by Kan et al. [31], rock-salt
 351 crystal structure carbides, such as TiC, ZrC, and NbC, are completely inter-soluble, and, if melted together with Fe,
 352 they precipitate out of the melt and form a single high-entropy carbide phase. Carbides are also at least partially
 353 responsible for the formation of microshrinkages since they have a smaller volume than the ferritic matrix and
 354 graphite after solidification (Figs. 6d and 8). Moreover, very long solidification times, as occurs in the 20C
 355 condition, can also lead to the formation of large Fe₃C lamellas in the pearlitic structure, increasing the discontinuity
 356 with the ferrous matrix (Fig. 8). As reported in [32] and shown by EDS analysis in Fig. 8, Cr and Mn have a high
 357 impact on the formation of the intergranular pearlitic area: they decrease the eutectoid equilibrium temperature at
 358 which austenite decomposes into ferrite and graphite, thus leading to a decrease in the carbon diffusion coefficient
 359 during the ferritic reaction and favoring the formation of pearlite.

360



Normalized atomic concentration/%	C	Mg	Si	P	S	Ti	V	Cr	Mn	Fe	Ni	Cu
1	17.7	0.05	1.02	0.00	0.00	0.03	0.14	0.24	1.16	79.50	0.08	0.11
2	26.3	0.02	0.10	0.03	0.03	0.00	0.12	0.62	1.11	71.65	0.03	0.01
3	26.5	0.06	0.05	0.00	0.00	0.00	0.18	0.59	1.19	71.42	0.03	0.00

361 Fig. 8 Large Fe₃C lamellas within pearlitic structure into 20C microstructure. Yellow arrows indicate
362 microshrinkages within pearlitic area

363 Significant differences were observed even for the samples characterized by the same solidification time (10 h)
364 but by different extraction positions (casting center (10C) of 700 x 700 x 700 mm³ cube or transition zone (10T) of
365 1000 mm × 1000 mm × 1000 mm cube). The 10T condition is characterized by a *NS* approximately double that of
366 the 10C condition and a lower *NC* (−56.3%), while the *N* is comparable (82.4% vs. 83.2%, respectively). At the
367 same time, the *PA* of the 10T condition is smaller than the 10C condition (−36.9%), and the *FG* is coarser, with an
368 increase equal to 16.2% (Fig. 2).

369 The differences between the 10T and 10C microstructures (Figs. 3 and 4) can be explained by considering three
370 main phenomena which take place during solidification in HSDI castings: (i) graphite nucleation and growth, (ii)
371 ferritic grain formation and coarsening, and (iii) segregation of minor elements.

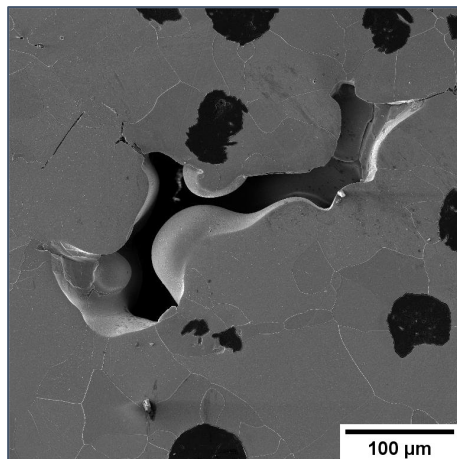
372 The formation of a non-homogeneous microstructure in HSDI castings, characterized by large pearlitic islands
373 (Fig. 5), is promoted by segregation of pearlitic promoters such as Mn and Cr in the intercellular regions of the
374 ferritic matrix. Therefore, the formation of large pearlitic islands affects the casting center (10C condition) more
375 than the transition zone (10T condition) due to the segregation of alloying elements into the last fraction of liquid
376 that solidifies (Fig. 2d).

377 The size and morphology of graphite in as-cast DI result from crystallization within the liquid and growth in the
378 austenitic shell controlled by C atom diffusion. Instead, solid solution diffusion mechanisms below the eutectic
379 temperature increase the *NS* and reduce the *NC* due to C atom diffusion. In particular, dissolution of smaller particles
380 and deposition onto larger particles occurs to reach a more thermodynamically stable state where the surface-to-
381 area ratio is minimized (Ostwald ripening mechanism). Considering the longer time necessary for the largest cube
382 (1000 mm × 1000 mm × 1000 mm) to cool (200 h to reach 300 °C on the surface) compared to the medium cube
383 (700 mm × 700 mm × 700 mm) (131 h to reach 300 °C on the surface) (Table 4), the 10T samples are subjected to
384 high temperature for a longer time than the 10C samples, even if the solidification times are comparable. Therefore,
385 castings with a higher thermal modulus favor the formation of a lower number of larger graphite nodules in the
386 transition zone.

387 The comparable N can be explained based on the peculiar graphite growth conditions in the center of the HSDI
388 castings [20, 21]. Initially, the graphite nuclei precipitate in the liquid, and then spheroids are formed by the action
389 of Mg and RE; however, during the coarsening of the first nuclei, the morphology of the graphite nodule can
390 degenerate. The development of anomalous structures of the graphite (exploded or chunky graphite) is due to high
391 C atom supersaturation in the remaining liquid and low nucleation potential related to the lack of oxygen-based
392 compounds suitable for graphite nucleation [25] or insufficient undercooling [33]. Consequently, the chunky or
393 exploded graphite formation occurs during solidification and depends on the solidification time. In this condition,
394 the casting center is disfavored compared to the transition zone at equal solidification time due to the more extensive
395 segregation phenomena (e.g., Mg, Mn, Mo, Cr, V, B, Ti) around the coarse eutectic cells, which can negatively
396 impact nodularity [12].

397 Local segregation also occurs in the transition zone of the 1000 mm³ cube due to the large size of the casting.
398 This leads to the formation and growth of chunky graphite cells by diffusion of C atoms through the austenitic
399 matrix and, consequently, to the growth of very large foliated dendrites and branched structures (Fig. 3h) depending
400 on the cooling time. Therefore, the slow cooling rate after solidification favors the growth of chunky and exploded
401 graphite in the 10T condition more than in the 10C condition. In addition, the longer exposition at high temperature
402 negatively affects N (Table 4) and induces the coarsening of the ferrite grains, as reported in Fig. 2e.

403 Despite the excellent castability of DI, interdendritic shrinkage defects (sized up to a Féret diameter of 300 μm)
404 were observed, especially in samples extracted from the casting center for longer solidification times (10C and 20C
405 conditions), as shown in Fig. 9. As a matter of fact, the Mg segregation in the center of the large castings can favor
406 a more extensive formation of interdendritic shrinkage [34]. At the same time, the solidification of the DI as a
407 divorced eutectic, i.e. the graphite nodules surrounded by the austenite shell, hinders the interdendritic feeding and
408 promotes the formation of interdendritic shrinkage, as described in [35].

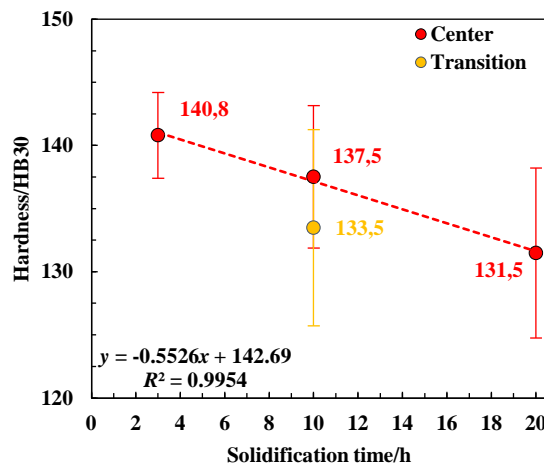


409
410 Fig. 9 SE image of intercellular shrinkage within ferritic matrix (10C sample)

411 As reported in [25, 30, 36], the hardness of DI is essentially the hardness of the ferrous matrix (ferritic and
412 pearlitic grains), increased by the hard carbides located in the intergranular zone. Hardness is minimally influenced
413 by the morphology and size of the graphite, even if the chunky graphite interrupts the continuity of the three-

414 dimensional matrix. However, optimizing the nodule size and nodule density can indirectly increase the hardness
415 of the DI [37]: a high nodule count allows for obtaining a finer and more homogeneous matrix, thus reducing the
416 presence of defects. In addition, the nodule count is related to the nodularity: if the inoculation treatment is
417 performed correctly, it allows for the obtaining of smaller and more spherical nodules, refining the grain structure.

418 Despite the significant differences in the microstructures observed in this study, the hardness varies only by 6.6%
419 (Fig. 10) from the maximum value (3C condition: 140.8 HB30) to the minimum value (20C condition: 131.5 HB30).
420 This result is due to the combined and contrasting effects related to the variation of the microstructural
421 characteristics described in Fig. 2. This is also supported by the increased data deviation from the 3C to the 20C
422 condition (Fig. 10), which agrees with the microstructural inhomogeneity increase in Fig. 2.



423

424 Fig. 10 Effects of different solidification times (3, 10 and 20 h) and extraction zones (casting center, C, and transition
425 zone, T) on hardness of EN-GJS-400-15. R^2 is evaluated only for C samples (different solidification times at casting
426 center)

427 In HSDI castings, the inoculation and spheroidization treatments are challenging due to the casting size and
428 solidification time. As a result, it is more likely to find areas with low nodule count and segregation of elements that
429 favor the formation of pearlite, carbides, or degenerate graphite. These microstructural irregularities significantly
430 impact the tensile and fatigue properties of the castings [3, 4, 9–11, 24]. However, because hardness is negligibly
431 affected by such inhomogeneities and anomalies, it is not a reliable indicator of the microstructure in HSDI castings
432 and, moreover, it doesn't provide reliable information on tensile and fatigue strength.

433 4 Conclusions

- 434
- 435 1. As the solidification time in the center of HSDI varies from 3 to 20 h, the nodule count decreases from 30.3
436 to 3.6 nodules per mm^2 and the nodule size increases from 0.004 to 0.026 mm^2 . At the same time, nodularity
437 reduces from 91.4% to 73.6%. These effects are attributed to an increase in local segregation and lower
undercooling.

- 438 2. For comparable solidification time (10 h), the microstructural characteristics vary in relation to the
439 extraction zone. Specifically, the transition zone (10T) is characterized by an average nodule size
440 approximately double that of the casting center (10C) (0.012 vs. 0.026 mm²), as well as lower nodule count
441 values (4.5 vs 10.3 nodules per mm²). Conversely, nodularity is comparable (83.2% vs. 82.4%,
442 respectively).
- 443 3. Long solidification times favor the formation of chunky graphite, especially in the casting center, where
444 solidification conditions limit undercooling and promote supersaturation of minor elements in the molten
445 liquid.
- 446 4. With increasing solidification time, local segregation phenomena of minor elements increase, thus leading
447 to the formation of carbides in the intercellular area and a three-fold increase in the pearlitic area extension
448 from the 3C to the 20C condition. Conversely, the 10T condition shows a decrease in the pearlitic area
449 extent compared to the 10C condition (5.3% vs. 8.4%, respectively).
- 450 5. The increase in casting wall thickness leads to a ferritic grain coarsening from 3C to 20C condition (from
451 20.5 to 11.1 intercepts per mm). Similarly, the diffusion mechanisms lead to larger grains in 10C than in
452 10T condition (15.4 vs. 12.9 number of intercepts per mm, respectively).
- 453 6. Hardness is slightly influenced by solidification time and varies from the maximum value of 140.8 HB30
454 for the 3C condition to the minimum value of 131.5 HB30 for the 20C condition. This can be related only
455 to the microstructural coarsening of the graphite nodules and the ferrous matrix.

456 In the light of the above, the longer the solidification time, the worse the microstructural characteristics of cast
457 iron due to unfavorable solidification phenomena. However, the evaluation of how much the morphology of the
458 microstructure can worsen in terms of nodule size, degenerated graphite, shrinkage porosity, etc., was never
459 evaluated in real castings despite its importance. Moreover, these data will be helpful in refining casting simulations
460 used to predict microstructure in HSDI castings. This would also allow for the optimization of the design of castings
461 and the reduction of costs and environmental impact. This study pointed out that hardness tests do not highlight the
462 microstructural variabilities within ferritic HSDI castings, which instead profoundly affect the static and cyclic
463 mechanical properties, as reported in the literature. This aspect will be explored more thoroughly in future works
464 by the Authors by analyzing the static and cyclic mechanical properties of the EN-GJS-400-15 in the same
465 solidification conditions. Finally, this study establishes a foundation for developing an international standard for
466 quality control of ferritic HSDI castings with thicknesses exceeding 200 mm, currently outside the existing standard.

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473 **Declarations**

474 The authors declare that they have no known competing financial interests or personal relationships that could have
475 appeared to influence the work reported in this paper. The corresponding author, on behalf of all authors, certify
476 that they have NO affiliations with or involvement in any organization or entity with any financial interest (such as
477 honoraria; educational grants; participation in speakers’ bureaus; membership, employment, consultancies, stock
478 ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest
479 (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials
480 discussed in this manuscript.

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