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The effect of heating rate and soaking time on microstructure of an advanced high strength steel

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Abstract

This work focuses on the effect of soaking time on the microstructure during ultrafast heat treatment of a 50% cold rolled low carbon steel with initial ferritic-pearlitic microstructure. Dilatometry analysis was used to estimate the effect of heating rate on the phase transformation temperatures and to select an appropriate inter-critical temperature for final heat treatments. A thorough qualitative and quantitative microstructural characterization of the heat treated samples is performed using a wide range of characterization techniques. A complex multiphase, hierarchical microstructure consisting of ferritic matrix with embedded martensite and retained austenite is formed after all applied heat treatments. In turn, the ferritic matrix contains recrystallized and non-recrystallized grains. It is demonstrated that the ultrafast heating generally results in finer microstructure compared to the conventional heating independently on the soaking time. There is a significant effect of the soaking time on the volume fraction of martensite of the ultrafast heated material, while in the samples heated with conventional heating rate it remains relatively unchanged during soaking. Recrystallization, recovery and phase transformations occurring during soaking are discussed with respect to the applied heating rate.

Keywords: steel, ultrafast heating, microstructure, transmission Kikuchi diffraction, texture

1. Introduction

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29 Steels have been the most widely used materials all over the world and are likely to remain
30 a key material of choice in construction and manufacturing. Steel manufacturing is a
31 multistage process, where the heat treatment of (semi-)final product (in form of sheet, rod,
32 wire) to a great extent determines its microstructure and, hence, its properties. The current
33 approach for steel heat treatment is based on homogenization of microstructure at elevated
34 temperatures (either at austenitic or intercritical temperatures) and cooling with controlled
35 rate often followed by further treatment to form the required microstructure [1]. In 2011,
36 Cola *et. al.* [2] proposed an idea to apply ultrafast heat treatment for manufacturing advanced
37 high strength steels (AHSS) with microstructures as heterogeneous as those processed via
38 conventional heat treatments. This treatment was initially referred to as ‘flash processing’
39 [2], and other terms such as ‘ultrashort annealing’ [3] and ‘ultrafast heating’ [4–7] are widely
40 used for this process in the recent literature. Ultrafast heat treatment is based on heating the
41 material with the heating rate in the range of 100 to 1000 °C/s to an intercritical temperature,
42 very short soaking at this temperature followed by quenching. The whole process lasts just
43 a few seconds and, therefore, is characterized by significantly reduced energy consumption
44 compared to the conventional heat treatments [8].

45 The current state of the art in the effect of ultrafast heat treatment on the microstructure and
46 properties of steels can be summarized as follows. The final microstructure of the ultrafast
47 heat treated steels is determined by three major heat treatment parameters: heating rate, peak
48 temperature and soaking time. Ultrafast heating typically results in grain refinement in
49 interstitial free (IF) [9] and low carbon steels [3–5,10,11], thus, leading to higher mechanical
50 strength. Increasing heating rate shifts the recrystallization temperature to higher values than
51 the one measured at conventional heating rates of 10-20°C/s. Recovery and recrystallization
52 processes concurrently occur during ultrafast heating, and increasing the heating rate
53 decreases the recrystallized fraction of ferrite for a given temperature [5–7,12–14]. The
54 martensite volume fraction in the heat treated steel tends to increase with increasing peak
55 temperature [15]. The initial microstructure strongly influences the properties of steels after
56 ultrafast heat treatment [5]. Particularly, the steels with the initial ferritic-pearlitic
57 microstructure showed lower strength and higher ductility compared to the steels with the
58 initial ferritic-martensitic microstructure [5]. The pre-heating stage at temperatures of 300-
59 400 °C has minor effects on the microstructure evolution during ultrafast heating, though
60 increase of pre-heating temperature results in lower volume fraction of austenite, and hence
61 martensite upon quenching, due to cementite spheroidization [12].

62 Microstructure evolution in steels during ultrafast heating and short soaking at the peak
63 temperature is a very complex phenomenon, as it involves simultaneously recovery,
64 recrystallization, grain growth, phase transformations and diffusion of alloying elements
65 with carbon playing the key role. In most of the basic studies, the isothermal soaking time
66 was taken as short as possible, 0.1- 0.2 s [5,7,12,13]. Such short soaking times cannot be
67 reached during UFH processing of steel on the existing industrial lines and this is a
68 significant obstacle for implementation of the ultrafast heating in steel industry. It was
69 reported that longer isothermal soaking time (30 s) can erase the positive grain refining effect
70 of the ultrafast heating [16]. However, in the current literature there are no systematic studies
71 on the effect of the isothermal soaking time at the peak temperature on the microstructure
72 and properties of steel after ultrafast heating. Fundamental understanding of microstructure
73 evolution is required to enable an easy determination of the optimum soaking parameters for
74 microstructural design in the ultrafast heat treated steels. Therefore, the main objective of
75 the present work is to thoroughly study the effect of soaking time on the microstructure
76 evolution during ultrafast heating of a low carbon steel. Conventional heating of the steel
77 followed by detailed microstructural characterization is also performed for comparison.

78

79 **2. Material and experimental procedures**

80

81 *2.1. Material*

82 A low carbon steel with chemical composition of 0.19 % C, 1.61 % Mn, 1.06 % Al, 0.5 %
83 Si (in wt. %) was selected for this investigation. Alloys with this composition are typically
84 used in the automotive sector as transformation induced plasticity (TRIP) assisted steels,
85 which belong to the 1st generation of AHSS [17–19]. Two kinds of heating experiments were
86 performed: a) dilatometry measurements to determine phase transformation temperatures,
87 and b) annealing tests to the intercritical temperature with varying soaking time followed by
88 quenching. Both types of experiments are described in detail below.

89 *2.2. Dilatometry experiments*

90 As increasing heating rate shifts the recrystallization temperature to the higher values than
91 the equilibrium one or the one measured at conventional heating rates [5,13]. Dilatometry
92 measurements were carried out to determine the phase transformation temperatures A_{C1} and

93 A_{C3} of the studied steel as a function of heating rate. For these experiments, specimens with
94 dimensions of $10 \times 5 \times 1 \text{ mm}^3$ were machined from the as-received material. Tests were carried
95 out in a Bähr DIL805A/D dilatometer (Bähr-Thermoanalyse GmbH, Hüll-Horst, Germany).
96 Specimens were heated up to $1100 \text{ }^\circ\text{C}$ with different heating rates (1, 10, 50 and $200 \text{ }^\circ\text{C/s}$)
97 and holding time equal to 0.2 s. Heating rates above $200 \text{ }^\circ\text{C/s}$ were not applied due to
98 instability of the system in that range of heating rates. A *K*-type thermocouple was welded
99 to the midsection of each specimen to measure their temperature during experiment. The
100 material was then cooled down to room temperature at $-300 \text{ }^\circ\text{C/s}$. The sample
101 expansion/contraction during heating/cooling was recorded, and the obtained dilatometry
102 curves were analyzed. The tangent intersection method was applied to determine the start
103 (A_{C1}) and finish (A_{C3}) temperatures of austenite formation.

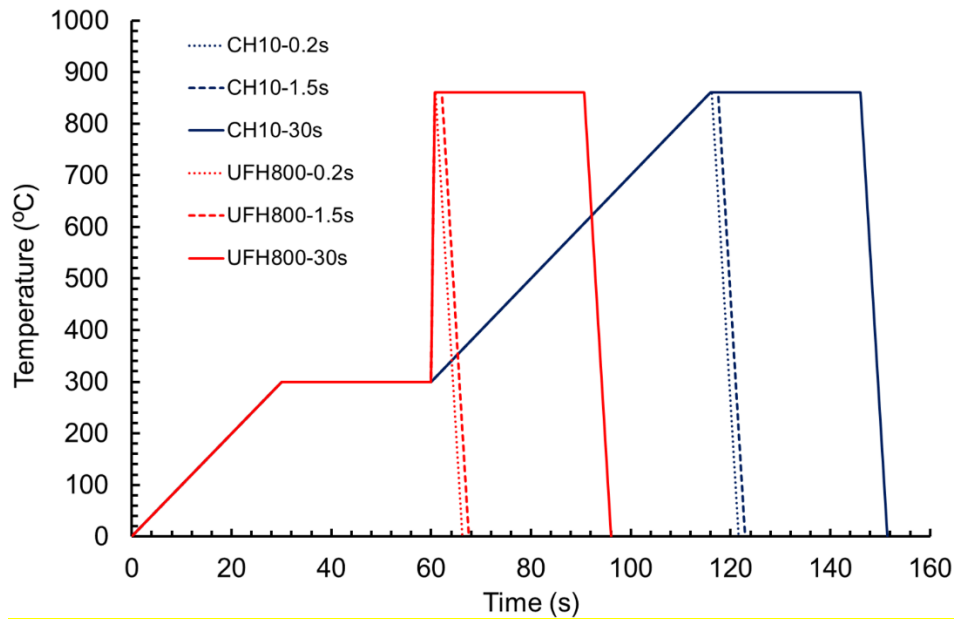
104

105 2.3. Intercritical heat treatments

106 For the intercritical heat treatments, strips of 100 mm in length and 10 mm in width were
107 machined along the rolling direction and heat treated in a thermo-mechanical simulator
108 Gleeble 3800. A *K*-type thermocouple was spot-welded to the midsection of each specimen.
109 Two different types of heat treatment were applied. In both types, the thermal cycle was
110 divided into five stages. On the first and second stages, the specimens were heated at $10 \text{ }^\circ\text{C/s}$
111 to $300 \text{ }^\circ\text{C}$, followed by a soaking period of 30 s at $300 \text{ }^\circ\text{C}$. These stages simulate a preheating
112 in some industrial continuous annealing lines to reduce the thermal stresses during heating.
113 The third stage is heating from $300 \text{ }^\circ\text{C}$ to the peak temperature of $860 \text{ }^\circ\text{C}$ at two different
114 heating rates, $10 \text{ }^\circ\text{C/s}$ (conventional heating or CH) and $800 \text{ }^\circ\text{C/s}$ (ultra-fast heating or UFH)
115 followed by soaking at $860 \text{ }^\circ\text{C}$ for 0.2 s. The processed specimens will be referred to as
116 CH10-0.2s and UFH800-0.2s, respectively. Such a short soaking time (0.2 s) allows to
117 eliminate the effect of annealing time on the microstructure and to focus entirely on the effect
118 of heating rate. The last stage was to cool down the material to room temperature at ~ 160
119 $^\circ\text{C/s}$. The peak temperature of $860 \text{ }^\circ\text{C}$ for intercritical annealing was selected based on the
120 outcomes of the dilatometry measurements (see Section 3.1).

121 To study the effect of soaking time at both heating rates (CH and UFH), additional heat
122 treatments were performed with higher soaking time (1.5 s and 30 s). The new generated
123 conditions are referred to as CH10-1.5s and CH10-30s for the CH treatment, and UFH800-
124 1.5s and UFH800-30s for the UFH treatment. All applied thermal cycles are schematically

125 presented in (Figure 1). In all samples, a minimum length of 10 mm of the homogeneously
126 heat treated zone was verified by microhardness measurements.



127

128 **Figure 1:** Schematic representation of the different heat treatments applied to the studied material.
129 (For interpretation of the references to color in this figure, the reader is referred to the web version
130 of this article).

131

132 2.4. *Microstructural characterization*

133 A thorough microstructural characterization of the samples heat treated in a thermo-
134 mechanical simulator (Figure 1) was performed. Specimens for scanning electron
135 microscopy (SEM) studies were ground and polished to a mirror-like surface applying
136 standard metallographic techniques with final polishing using OP-U (colloidal silica). The
137 polished specimens were etched with 3 vol.% Nital solution for 10 s. Examination of the
138 microstructure was performed using a FEI Quanta™ 450 FEG-SEM operating at an
139 accelerating voltage of 15 kV. Microstructure was observed on the RD–ND plane.

140 Specimens for electron backscatter diffraction (EBSD) analysis were ground and polished
141 following the same procedure as for SEM images. Orientation imaging microscopy (OIM)
142 studies were performed using a FEI Quanta™ Helios NanoLab 600i equipped with a
143 NordlysNano detector controlled by the AZtec Oxford Instruments Nanoanalysis (version
144 2.4) software. The data were acquired at an accelerating voltage of 18 kV, a working distance
145 of 8 mm, a tilt angle of 70°, and a step size of 65 nm in a hexagonal scan grid. The orientation
146 data were post-processed using HKL Post-processing Oxford Instruments Nanotechnology

147 (version 5.1©) software and TSL Data analysis version 7.3 software. Grains were defined as
148 a minimum of 4 pixels with a misorientation higher than 5°. Grain boundaries having a
149 misorientation $\geq 15^\circ$ were defined as high-angle grain boundaries (HAGBs), whereas low-
150 angle grain boundaries (LAGBs) had a misorientation $<15^\circ$. Textures are represented as
151 orientation distribution functions (ODFs) using Bunge notation [20]. The ODFs were
152 derived from the EBSD scans by superimposing Gaussian distributions with a half-width of
153 5°. The resulting ODF was represented as a series expansion of spherical harmonics
154 functions with a maximum rank of the expansion coefficient $L = 16$. Texture and grain size
155 calculations were made using scans having area of $\sim 6000 \mu\text{m}^2$ which contains at least 1100
156 grains. The volume fractions of transformed/untransformed grains and
157 recrystallized/recovered ferritic grains were determined by a two-step partitioning procedure
158 described in [5,21]. In this procedure, grains with high ($>70^\circ$) and low ($\leq 70^\circ$) grain average
159 image qualities are separated in a first step, allowing to distinguish between untransformed
160 (ferrite) and transformed (martensite) fractions, respectively. In the second step,
161 recrystallized and non-recrystallized ferritic grains are separated using the grain orientation
162 spread criterion: Grains with orientation spread below 1° are defined as the recrystallized
163 grains, while grains with an orientation spread above 1° are defined as the non-recrystallized
164 ones [22]. It should be noted that another grain average misorientation based criterion was
165 employed in our recent report [14] for separation of recrystallized/non-recrystallized grains.
166 Comparison of these two different criteria via analysis of numerous EBSD scans carried out
167 in this work has shown, that the criterion utilized in the present manuscript yields better
168 results. The microstructure was characterized on the plane perpendicular to the sample
169 transverse direction (the RD–ND plane).

170 X-ray diffraction (XRD) experiments were carried out to determine the retained austenite
171 volume fraction and its carbon concentration. Specimens with a surface of $10 \times 5 \text{ mm}^2$ were
172 prepared following the same procedure as for the EBSD analysis. The measurements were
173 performed using a Bruker D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany)
174 equipped with a VANTEC position sensitive detector and using Co K_α radiation ($\lambda = 1.78897$
175 \AA), an acceleration voltage of 45 kV and current of 35 mA. The measurements were
176 performed in the 2θ range from 45° to 130° with a step size of 0.035° and a counting time
177 per step of 3 s. The volume fraction of retained austenite was calculated using the Jatzcak
178 model as described in [23]. The austenite carbon concentration, X_c , was estimated from its
179 lattice parameter, a_γ . The latter was determined from the austenite peak position as [24]:

180
$$a_{\gamma}=0.3556+0.00453 X_c +0.000095 X_{Mn} +0.00056 X_{Al} \quad (1)$$

181 where a_{γ} is the austenite lattice parameter in nm and X_i represents the concentration of the
182 alloying element i in wt. %. The effect of silicon and phosphorous is not taken into account,
183 as it is negligible compared to other elements considered in Eq. (1).

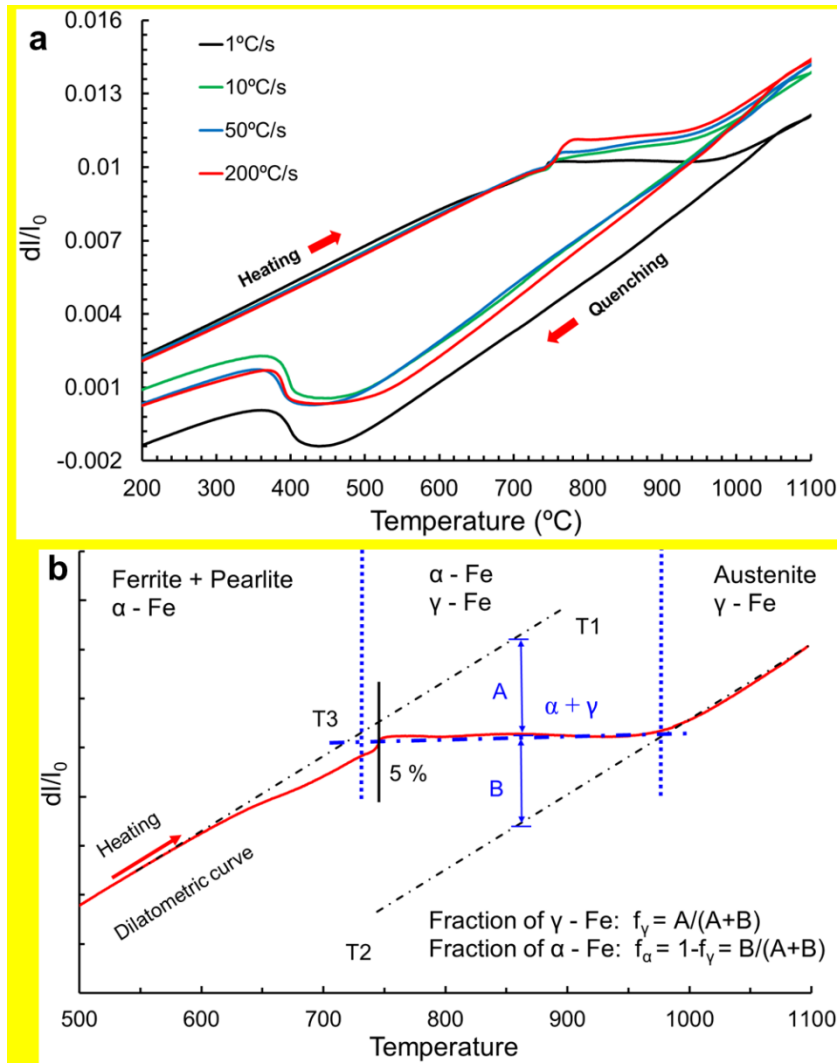
184 In order to carry out a thorough characterization of nanoscale constituents in a rapid manner,
185 in 2012 Keller *et al.* proposed a novel approach called transmission Kikuchi diffraction
186 (TKD) analysis [25]. It is based on performing an EBSD analysis in transmission mode. The
187 method requires very thin samples, similar to those for TEM characterization, and a
188 conventional SEM equipped with EBSD detector. It can also be combined with transmission
189 electron microscopy (TEM) analysis. Due to the low thickness of sample, typical SEM
190 voltages are sufficient for electrons to interact with the material and pass through, to finally
191 be captured by the EBSD detector. TKD offers better spatial resolution (< 10 nm) than
192 EBSD, allowing the resolution of nanoscale microstructural constituents having 10-30 nm
193 in size [26,27]. It has been successfully used to analyze oxides and nitrides in aluminium
194 alloys [28] and stainless steels [29,30], as well as martensite and retained austenite in bainitic
195 steels [31]. In this work, for TKD and TEM studies, the samples were ground to a thickness
196 of 100 μ m and disks of 3 mm in diameter were subsequently punched out. The disks were
197 further thinned in a Struers Tenupol-5 via twin-jet electropolishing until a central hole
198 appeared. The used electrolyte was composed of 4 % vol. HClO₄ in 63 % water-diluted
199 CH₃COOH under 21 V at 20 °C and a flow rate equal to 17. TKD data were collected by an
200 EDAX-TSL EBSD system attached to a FEI Quanta™ 450-FEG-SEM under the following
201 conditions: accelerating voltage of 30 kV, working distance of 4 mm, tilt angle of - 40°, a
202 beam current of 2.3 nA corresponding to the FEI spot size of 5, aperture size of 30 μ m. TKD
203 measurements were performed with the step size of 10 nm. The orientation data were post-
204 processed using TSL Data analysis version 7.3 software. TEM images were acquired in a
205 Jeol (S)TEN JEM-2200FS operated at 200 kV and equipped with an aberration corrector of
206 the objective lens (CETCOR, CEOS GmbH) and a column electron energy filter (omega
207 type). XRD, TEM and TKD measurements were performed on samples CH10-0.2s,
208 UFH800-0.2s, UFH800-1.5s and UFH800-30s.

209

210 **3. Results and discussion**

211 *3.1. Dilatometry*

212 **Figure 2a** represents the typical dilatometry curves for the samples tested with different
213 heating rates. The A_{C1} temperature was determined at 5 % volume fraction of the
214 transformed phase calculated by the lever rule (as shown in **Figure 2b**). Such relatively high
215 percentage of the transformed phase was selected as a criterion due to complexity of the
216 microstructure evolution during heating, which involves various processes (carbide
217 dissolution, recovery and recrystallization of ferrite, formation of austenite as observed in
218 [32–34] and described in Section 3) resulting in A_{C1} temperature range. Once the sample is
219 fully austenitic at the A_{C3} phase transformation temperature, the expansion becomes linear
220 with the temperature. The martensite start temperature M_s corresponds to the point on the
221 dilatation curve, where the contraction of austenite during quenching is replaced by
222 expansion due to the formation of martensite. As it is seen from **Table 1**, all three
223 transformation temperatures, A_{C1} , A_{C3} and M_s , tend to increase with the increasing heating
224 rate.



225

226 **Figure 2:** a) Schematic diagram of an experimental dilatometry curve (measured at 1 $^{\circ}\text{C/s}$) to
 227 calculate A_{C1} and A_{C3} temperatures via tangent intersection principle and lever rule; b) Dilatometry
 228 curves from dilatometry tests with different heating rates. (For interpretation of the references to
 229 color in this figure, the reader is referred to the web version of this article).

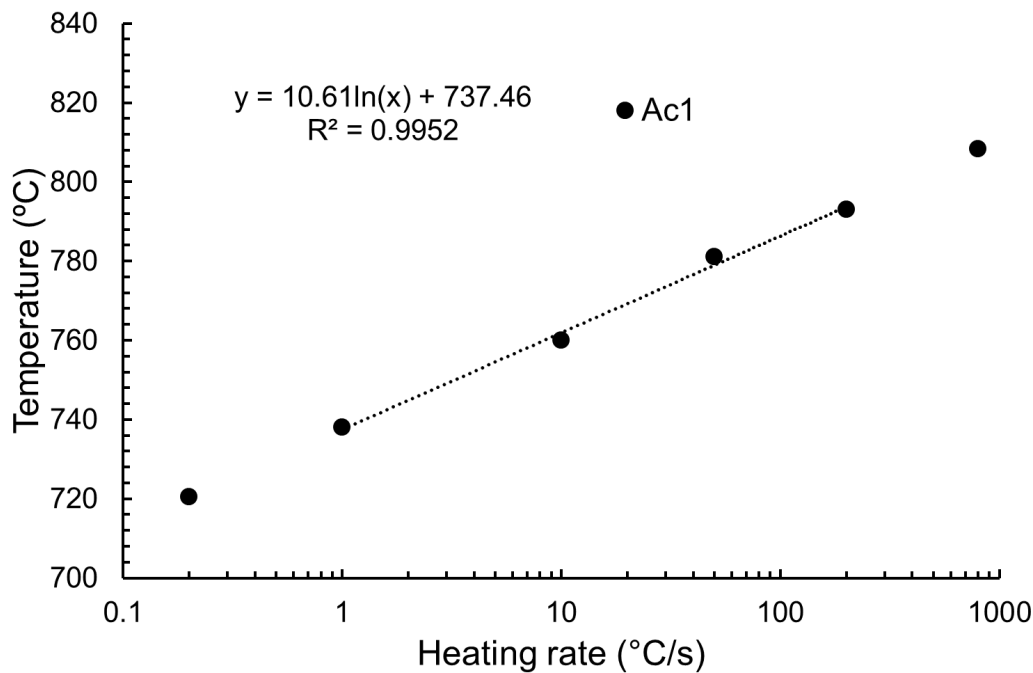
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231 **Table 1:** Effect of the heating rate on the phase transformation temperatures: A_{C1} , A_{C3} and M_s .

Heating rate ($^{\circ}\text{C/s}$)	A_{C1} ($^{\circ}\text{C}$)	A_{C3} ($^{\circ}\text{C}$)	M_s ($^{\circ}\text{C}$)
1	738	968	483
10	760	969	489
50	781	971	498
200	793	983	530

232

233 For the A_{c1} , the pronounced increase from 738 to 781 °C occurs at the lower heating rates
234 ranging from 1 °C/s to 50 °C/s. On the other hand, the A_{c3} temperature just slightly grows
235 from 968 to 971 °C in that temperature range jumping up to 983 °C at 200 °C/s. It can be
236 hypothesized, that this variation of the A_{c1} temperature is determined mainly by nucleation
237 and growth rate of austenitic grains. The nucleation rate at the given elevated temperature
238 grows with the increasing heating rate, since the latter suppresses the recovery effects,
239 resulting in higher density of lattice defects at the given temperature, which, in turn, promote
240 phase nucleation. The growth rate of the nucleated austenitic grains is controlled by carbon
241 diffusion [7] and solute drag effect (by Mn atoms in the studied steel) [35]. Therefore, at the
242 early stages of phase transformation, the austenite volume fraction at the given temperature
243 decreases with increasing heating rate. Both factors result in increasing A_{c1} temperature with
244 rising heating rate. It should be noted that similar results were earlier published in [36]. In
245 this study, a linear dependency of A_{c1} on the heating rate (Figure 3) on the semi-log plot is
246 observed. Similar tendency of A_{c1} on the heating rate for ferritic-pearlitic microstructure has
247 been reported in [37,38]. The nucleation and growth depend on the heating rate exponentially
248 [38]. Moreover, the extrapolation of this behavior to low heating rates (0.2 °C/s) shows an
249 equilibrium temperature of 720 °C, which is very close to the theoretical one (723 °C), thus
250 confirming the linear character of this dependence. Therefore, this approach can also be used
251 to predict the A_{c1} temperature at high heating rates. Particularly, for 800 °C/s, the A_{c1}
252 temperature is about 808 °C (Figure 3). On the other hand, the dependence of A_{c3}
253 temperature on the heating rate is less pronounced. Similar observations were reported
254 earlier in [39]. Therefore, the intercritical temperature of 860 °C was selected as the peak
255 temperature for both CH and UFH treatments (see Section 2.3).



256

257

Figure 3: Effect of heating rate on the A_{c1} temperature.

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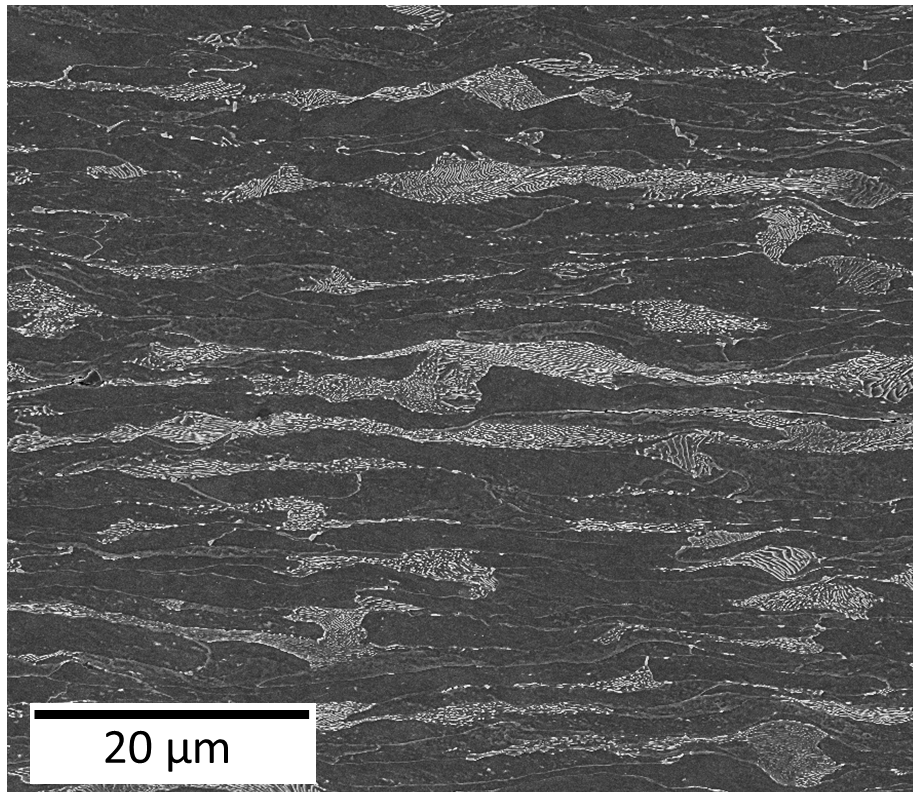
270 3.2. SEM characterization

271

272

273

The supplied material shows a typical cold rolled microstructure consisting of elongated grains of deformed ferrite with volume fraction of 76 % and pearlite with volume fraction of 24% (Figure 4Error! Reference source not found.).



274

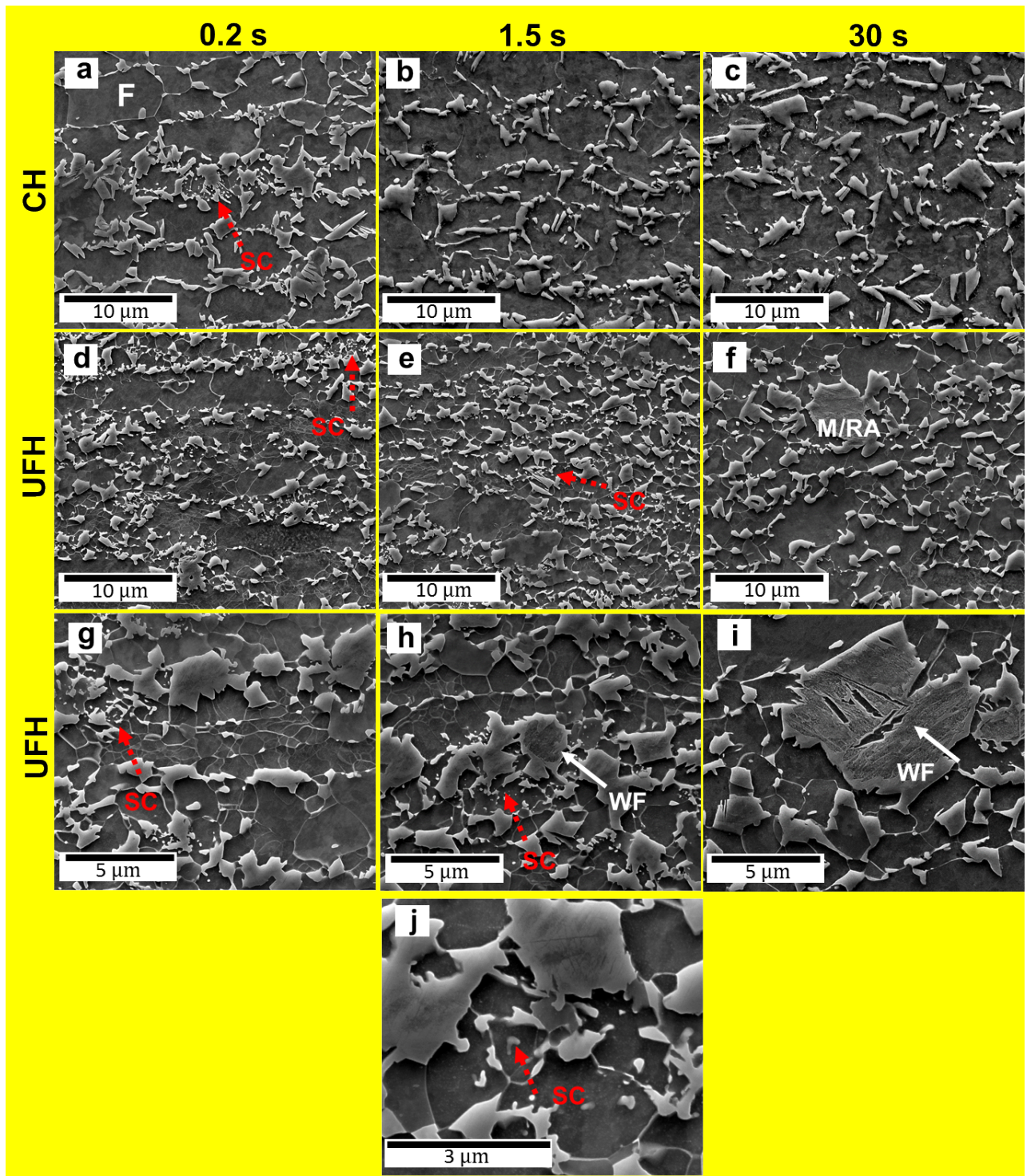
275 **Figure 4: Initial ferritic-pearlitic microstructure of the steel after 50 % cold reduction, being ferrite**
276 **in grey and pearlitic colonies in white.**

277

278 The microstructure after CH treatment with soaking time of 0.2 s, 1.5 s and 30 s **is presented**
279 **in Figure 5a,b,c**, respectively, whereas remaining images illustrate the microstructure after
280 **UFH treatment**. In all cases, the material presents a complex microstructure formed by a
281 ferritic matrix (consisting of recrystallized and recovered ferritic grains) with embedded
282 martensite and retained austenite grains. However, it strongly depends on the applied heat
283 treatment parameters. During CH treatment, the material presents a similar microstructure
284 independently on the soaking time, while the latter has very significant effect on the
285 microstructure **formed after** UFH treatment.

286 CH treatment generates a ferritic matrix with homogeneous microstructure consisting of
287 equiaxed grains, as previously observed in [5]. On the other hand, **UFH results in the matrix**
288 **microstructure consisting of fine equiaxed grains and larger elongated grains** surrounded by
289 martensitic grains. The large grains may grow from the heavily deformed ferrite located in
290 the vicinity of pearlite colonies, as the latter are not able to accumulate high plastic strain
291 during rolling. Hence, the higher energy stored in the heavily deformed ferritic areas leads
292 to a faster grain growth [40]. Some Widmanstätten ferritic grains are also observed in the

293 UFH samples after soaking for 1.5 and 30 s (marked by white arrows on **Figure 5h,i**)
294 possibly formed at the early stages of cooling. Those ferrite plates are surrounded by bainite.
295 Spheroidized cementite (SC) is also observed in samples UFH-0.2s and UFH-1.5s (marked
296 by red dashed arrows on SEM micrographs presented on **Figure 5**). It is related to the short
297 time (0.2 – 1.5 s) of the heat treatment, as reported previously by Castro Cerdá *et al.* [5,43],
298 and fully dissolved after soaking for 30 s. A very small region with spheroidized cementite
299 particles was also observed in the CH-0.2s sample, although its amount is negligible (**Figure**
300 **5a**).



301

302 **Figure 5:** SEM micrographs showing the effect of heating rate (10 and 800 °C/s) and soaking time
 303 (0.2 to 30 s) on the microstructure: a), b) and c) correspond to 10 °C/s for 0.2, 1.5 and 30 s,
 304 respectively; d), e) and f) correspond to 800 °C/s for 0.2, 1.5 and 30 s, respectively. Higher
 305 magnification images g), h) and i) show microstructures heated at 800 °C/s for 0.2, 1.5 and 30 s,
 306 respectively; j) higher magnification image of spheroidized cementite (SC) in the sample heated at
 307 800 °C/s for 1.5 s. Spheroidized cementite is marked by dashed red arrows, while white arrows
 308 indicate Widmanstätten ferrite (WF). Ferrite is marked as F, and M/RA stands for martensite/retained
 309 austenite. Etched with Nital (3%).

310

311 3.3. EBSD characterization

312 EBSD technique was used to precisely quantify and characterize the different
313 microconstituents formed in the material after both heat treatments. The results of EBSD
314 analysis are outlined in Table 2. CH treatment leads to a microstructure mainly formed by a
315 ferritic matrix, whose volume fraction remains constant (~ 86–87 %) and martensite volume
316 fraction slightly increases from 10.6 % to 12.5 % with the soaking time. As volume fraction
317 of ferrite does not vary with soaking time (i.e. the amount of intercritical austenite formed
318 at the peak temperature does not depend on the soaking time), the martensite increment can
319 be attributed to the partial transformation of austenite into martensite by deformation during
320 sample preparation. This indicates that retained austenite is less stable caused by the
321 homogenization of carbon distribution in its interior after longer soaking times. Although
322 the UFH process generates similar microstructure with the same microstructural
323 constituents, there are significant variations in the volume fractions of different phases with
324 respect to the CH treatment. The volume fraction of ferrite noticeably decreases with
325 increasing soaking time from 90.9 % at 0.2 s to 75.9 % at 30 s, while the volume fraction of
326 martensite shows the opposite trend. As the volume fraction of retained austenite remains
327 stable (2.1 – 2.2 %), it is possible to assure that the decrease of ferrite fraction is directly
328 associated to the formation of martensite. On the other hand, the difference in ferrite and
329 martensite volume fractions between CH and UFH conditions can be explained by the
330 spheroidization of cementite during heating. First, the nucleation of austenite occurs at the
331 α /cementite interface [44]. With conventional heating (CH), the cementite spheroidizes [7]
332 reducing the amount of preferable sites for austenite formation and resulting in longer
333 soaking time to reach the equilibrium. The main fraction of the inter-critical austenite is
334 transformed into martensite during cooling. On the other hand, during UFH treatment the
335 peak temperature is reached in less than 1 s which dramatically reduces the amount of
336 spheroidized cementite and, thus, increases the driving force for austenite nucleation at the
337 more favorable α /cementite interfaces.

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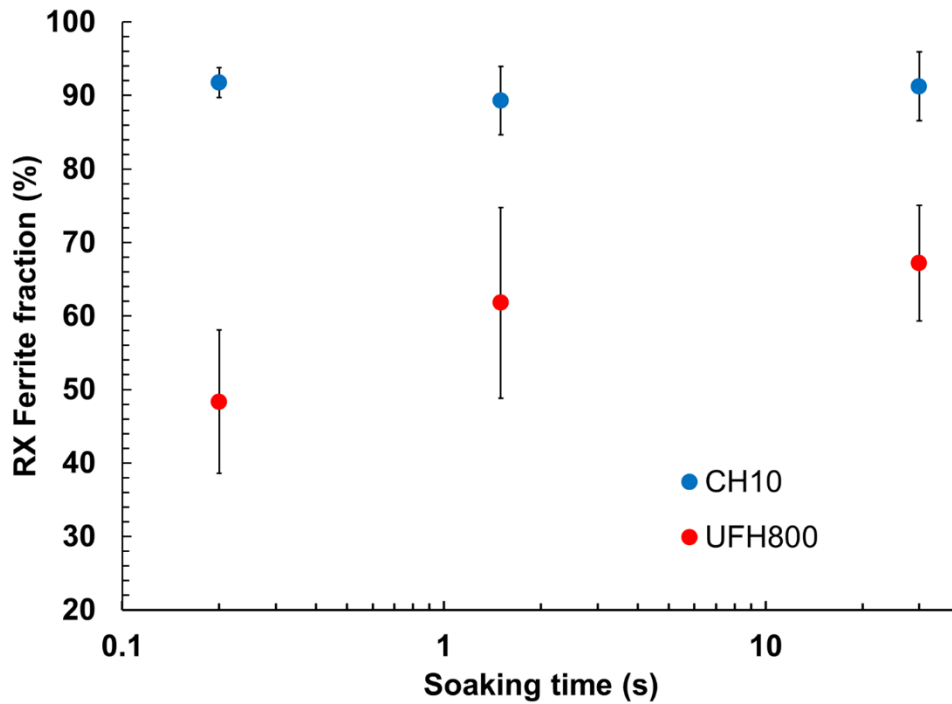
Table 2: Effect of the heating rate and soaking time on the volume fractions of phases present in the studied material.

Condition (s)	CH			UFH		
	0.2	1.5	30	0.2	1.5	30
Ferrite (%)	86.3 ± 2.4	87.4 ± 2.7	85.8 ± 1.6	90.9 ± 4.0	85.3 ± 2.8	75.9 ± 4.6
Martensite (%)	10.6 ± 1.7	10.8 ± 1.6	12.5 ± 1.6	6.9 ± 3.2	12.6 ± 3.1	22.0 ± 3.0
Retained austenite (%)	3.1 ± 0.7	1.8 ± 0.6	1.7 ± 0.1	2.2 ± 0.4	2.1 ± 0.3	2.1 ± 1.9

340

341 The morphology of the ferritic matrix in the CH and UFH heat treated samples also presents
342 significant differences. The EBSD analysis revealed both recrystallized and recovered grains
343 in the ferritic matrix. **Figure 6** represents the fraction of recrystallized ferrite in the ferritic
344 matrix for all analyzed conditions. It is seen that, while the CH treatment leads to a
345 homogeneous ferritic matrix, where almost 90 % of ferrite is recrystallized, the UFH
346 processing generates a matrix microstructure formed by recrystallized and non-recrystallized
347 (i.e. recovered) ferritic grains. After UFH treatment, the volume fraction of recrystallized
348 ferrite increases from ~50 % after 0.2 s to ~67 % after 30 s. So, while the recrystallization
349 process is completed during CH treatment already **after soaking for 0.2 s**, it is delayed during
350 UFH process. Similar observations were previously reported in **[43,45,46]**. This effect is due
351 to the competition of different processes, such as austenite formation and further grain
352 growth, reducing the driving force for recrystallization. For short soaking time (0.2 s), the
353 recrystallization is the controlling process, which results in a very low martensite volume
354 fraction (**Table 2**), similar to the CH treatment, and a significant volume fraction of
355 recrystallized ferrite present in the material (**Figure 6**). However, **after soaking for longer**
356 **time (1.5 – 30 s)**, other processes become dominant over recrystallization, such as the
357 nucleation and growth of austenite into ferrite and ferrite grain growth **[10,16]**. The first
358 effect results in the higher volume fraction of martensite present in the UFH800-30s (**Table**
359 **2**) and the decrease in volume fraction of recrystallized ferrite with increasing soaking time

360 from 1.5 to 30 s (Figure 6). The latter effect is discussed more in detail below (Figure 7 and
361 Figure 8).

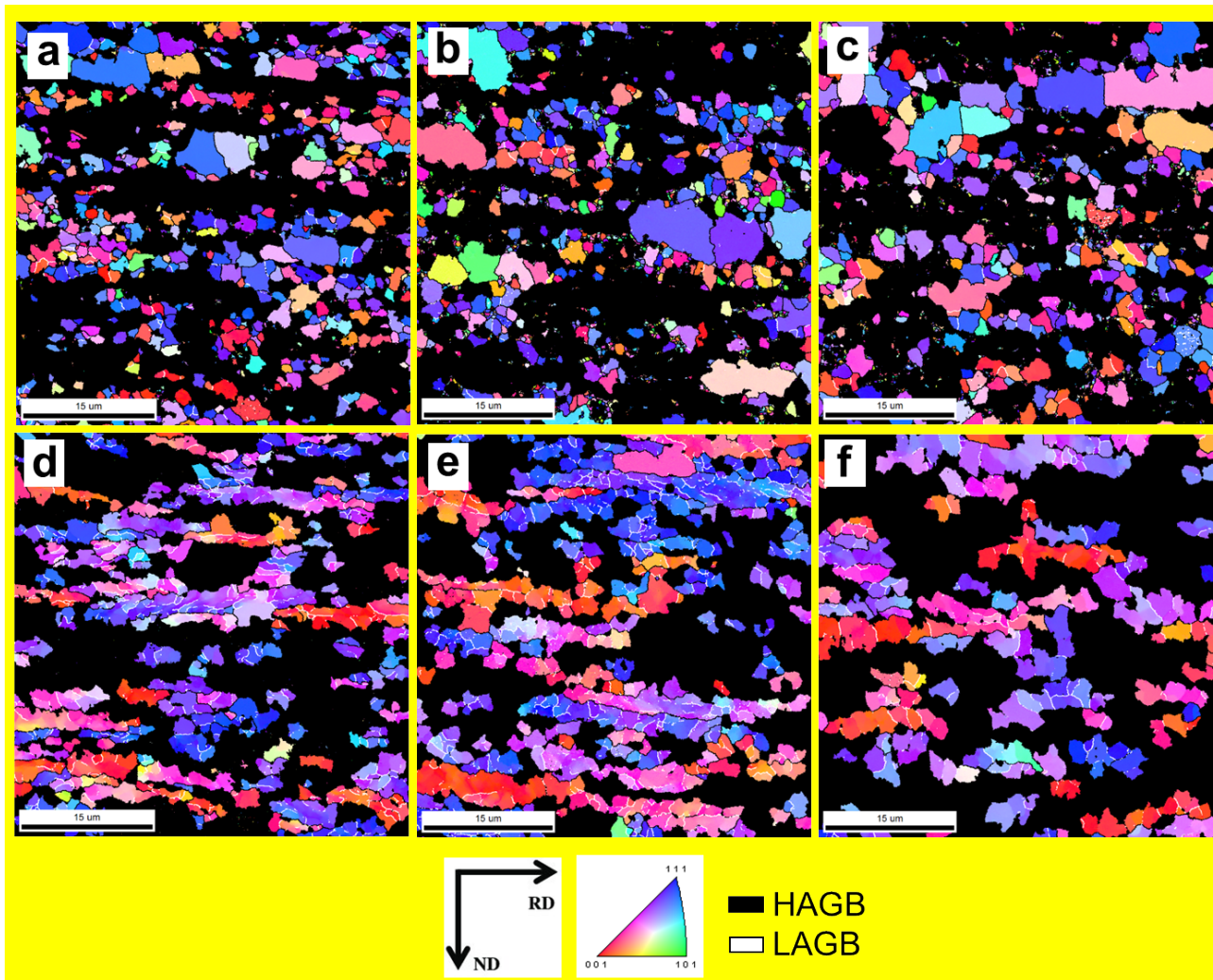


362

363 **Figure 6:** Evolution of volume fraction of recrystallized ferrite with respect to the total fraction of
364 ferrite with heating rate and soaking time. (For interpretation of the references to color in this
365 figure, the reader is referred to the web version of this article).

366

367 **Figure 7** represents the IPF maps for recrystallized (a, b, c) and non-recrystallized (d, e, f)
368 ferrite after UFH for 0.2, 1.5 and 30 s, respectively. It is seen in **Figure 7a,b**, that the vast
369 majority of the grains are in the early stage of growth, presenting a size $\leq 1.5 \mu\text{m}$, although
370 it is possible to observe grains which have fully recrystallized and grown, i.e. grains without
371 LAGBs and with low misorientations in their interior. This observation was also reported by
372 Castro Cerda *et al.* [5]. When soaking time increases to 30 s, the fraction of fine grains
373 decreases due to their growth, and the presence of larger grains is more evident (**Figure 7c**).
374 The non-recrystallized grains demonstrate significant misorientation in the interior of the
375 grains **indicating** formation of substructure independently on the applied soaking time
376 (**Figure 7d,e,f**).

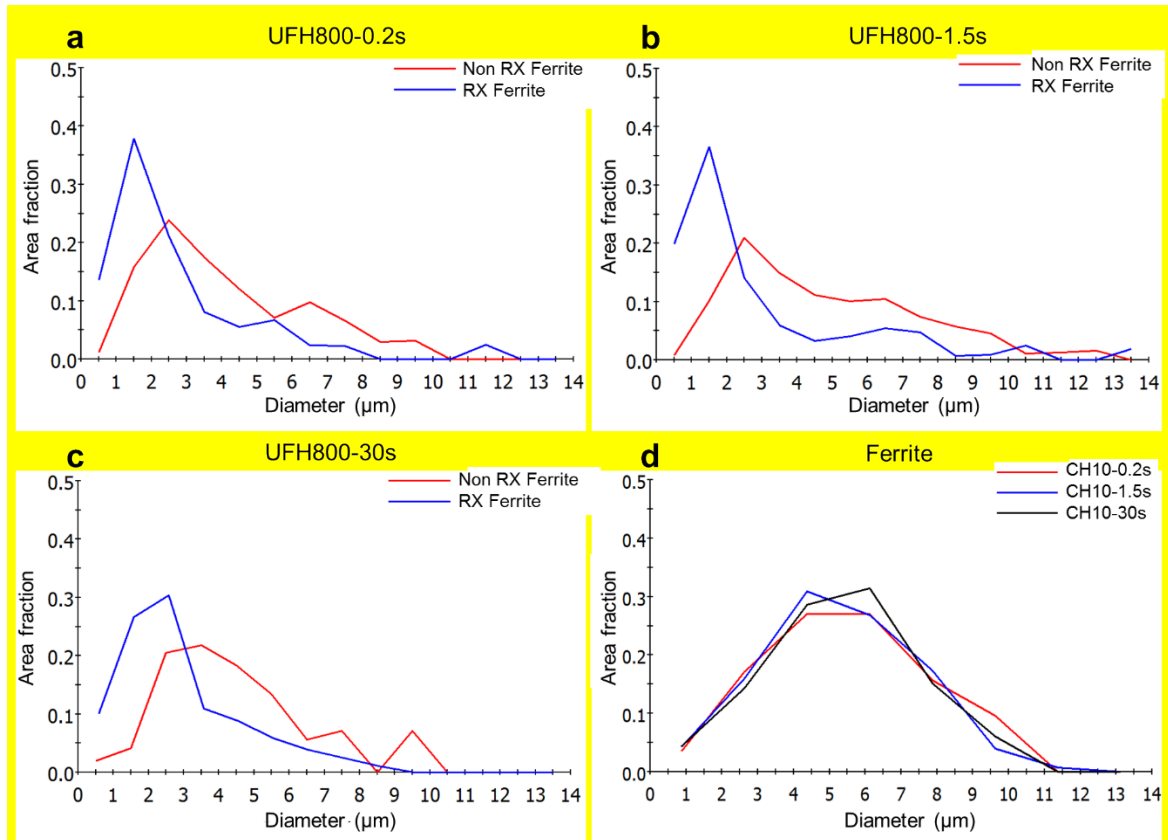


377

378 **Figure 7:** IPF maps after UFH treatment showing the recrystallized (a, b, c) and non-recrystallized
 379 (d, e, f) ferrite after 0.2, 1.5 and 30 s, respectively. HAGBs are shown in black and LAGBs in
 380 white. (For interpretation of the references to color in this figure, the reader is referred to the web
 381 version of this article).

382 The evolution of the grain size distribution for recrystallized ferrite is clearly visible and
 383 quantified in **Figure 8a,b,c**, where the grain size is plotted vs. the area fraction for the
 384 UFH800-0.2s, UFH800-1.5s and UFH800-30s, respectively (blue lines). It is observed that
 385 the mean peak shifts to higher values and widens. For instance, in the samples UFH800-0.2s
 386 and UFH800-1.5s the fraction of grains with a size below 1.5 μm is 52 % and 56 %, respectively,
 387 while after longer soaking it decreases to 36 % indicating the growth of the
 388 small grains nucleated at shorter times. A second peak at higher grain size is noticeable
 389 indicating the presence of the large grains mentioned above. The intensity of the second peak
 390 decreases with soaking time, as the microstructure becomes more homogeneous (**Figure 8**
 391 c). The histogram of grain size distribution for non-recrystallized ferritic grains (red lines in
 392 **Figure 8**) presents a similar character in comparison to the recrystallized ones. The primary

393 peak shifts to the higher values becoming wider, when soaking time is increased. The
 394 fraction of grains having size above 2.5 μm increases from 59 % at 0.2 s to 68 % at 1.5 s to
 395 73 % after 30 s. This effect can be produced by the coalescence of grains after partial
 396 recrystallization indicated by the presence of HAGBs. Nevertheless, the non-recrystallized
 397 grains are larger compared to the recrystallized ones after all soaking times. On the other
 398 hand, the ferritic matrix in the CH condition is formed mainly by recrystallized equiaxed
 399 grains, and its microstructure is not affected by soaking time (**Figure 8d**).



400

401 **Figure 8:** a), b), c) Representation of the equivalent circle diameter (ECD) versus area fraction for
 402 recrystallized (RX) and non-recrystallized (Non RX) ferrite after UFH with soaking for 0.2, 1.5 and
 403 30 s, respectively; d) grain diameter versus area fraction for ferrite after CH treatment. Data are
 404 obtained from the EBSD measurements. (For interpretation of the references to color in this figure,
 405 the reader is referred to the web version of this article).

406

407 It is well known that high heating rates lead to a smaller grain size [6,10,13,47,48], as it is
 408 shown for the studied steel in **Figure 8**. This is caused, among other reasons, by the short
 409 time given to the α/α interface to grow. On the one hand, after CH treatment the
 410 recrystallization and grain growth processes are completed independently on the applied
 411 soaking time. The grain size is also not affected by soaking time, as intercritical austenitic

412 grains act as barriers for the ferritic grains suppressing their further growth. On the other
413 hand, the UFH treated conditions show a bimodal distribution of grain size. The presence of
414 the two differentiated regions on the histograms can be rationalized by the interplay of two
415 main effects:

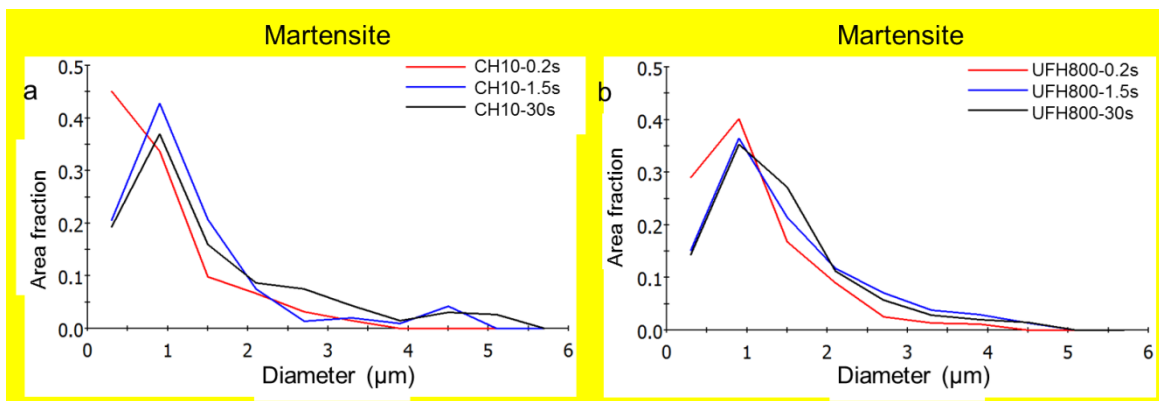
416 (1) the effect of the initial heterogeneous microstructure related to different amounts of strain
417 accommodated by individual ferritic grains, as shown in **Figure 4Error! Reference source**
418 **not found.**;

419 (2) the effect of heating rate. A higher heating rate results in a recrystallization process taking
420 place at higher temperatures, as discussed above, and, thus, in a higher nucleation rate due
421 to the high density of defects [13,43,48].

422 The nuclei formed within the highly deformed areas possess higher driving force to grow
423 and coalesce due to the high energy stored during cold rolling, resulting in the larger grains.
424 On the other hand, nuclei generated within the less deformed regions present reduced driving
425 force for growth. Moreover, due to the short time of the heat treatment, remains of individual
426 cementite particles (which were not completely dissolved during inter-critical annealing)
427 located at grain boundaries effectively pin grain boundaries suppressing grain growth and
428 coalescence [49–51] (**Figure 5g,h,i**). As the material is heated up to an intercritical
429 temperature, another important factor comes into play: Formation of austenite and its growth
430 competes for the energy stored in the material. The austenitic grains nucleate in carbon
431 enriched areas, i.e. within pearlitic colonies. It can be assumed that the intensive nucleation
432 of austenitic grains takes place within pearlitic colonies which were severely deformed,
433 rotated or broken during cold rolling, resulting in reduction of distance between cementite
434 plates. As is well known, the austenite nucleation rate is inversely proportional to the inter-
435 lamellar spacing of pearlite [12]. The austenite grows firstly into the pearlite until it is
436 dissolved and then into ferrite, as it is seen in **Figure 5**. Competition of all these processes
437 during UFH treatment results in the microstructure with finer grains (**Figure 5, Figure 8**).

438 **Figure 9** represents the equivalent circle diameter of martensite plotted versus area fraction.
439 For the CH condition, at short soaking time (0.2 s) most of the martensite grains were formed
440 from ultrafine austenitic grains, as the major peak lies below 1 μm (**Figure 9a**). Increasing
441 soaking time up to 1.5 s, the curve shifts to the right, indicating the growth of the earlier
442 formed nuclei. Finally, after annealing for 30 s, the decrease of the main peak intensity is
443 accompanied by increase in the area fraction at 3 μm , displaying that the austenite has

444 entered the growth stage after the nucleation after short soaking times. In the case of the
 445 UFH800-0.2s, the curve is similar to the CH condition with the same soaking time. However,
 446 the fraction of larger grains having a size of 4-5 μm increases. This behavior can indicate
 447 that the austenite nucleation is accompanied by a growth, due to the fact that the material
 448 has higher energy compared to the CH condition because of the low amount of spheroidized
 449 cementite and the higher carbon gradients present in the material, both produced by the rapid
 450 heating. It is more pronounced after 1.5 s, where the main peak has reduced, but there is an
 451 increase of the fraction of larger grains. The result of this effect is the rise of the martensite
 452 fraction in the overall microstructure. Finally, after 30 s the peak spreads to higher values,
 453 as it happens in the ferrite, showing an intense growth of the austenite grains during soaking.



454
 455 **Figure 9:** Martensite ECD vs area fraction for CH (a) and UFH (b) for different soaking times: 0.2
 456 s, 1.5 s and 30 s.

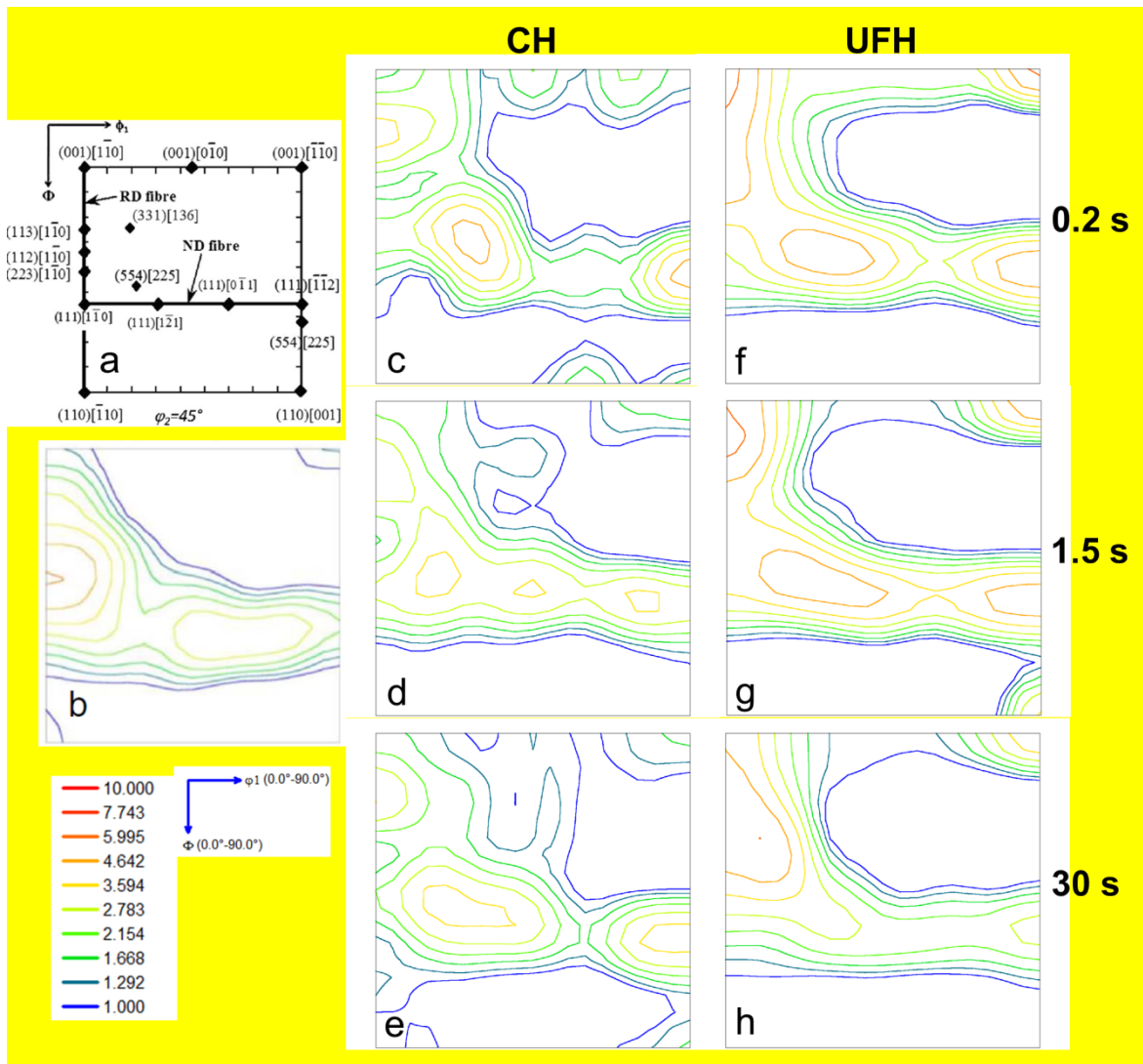
457

458 3.4. Texture analysis

459 To analyze evolution of the preferable crystallographic orientation of ferritic grains, texture
 460 analysis was carried out for all studied conditions. **Figure 10a** represents the ideal positions
 461 of the most important texture components in BCC lattice, while **Figure 10b** shows the
 462 orientation distribution function (ODF) of the initial cold-rolled material. **Figure 10c,d,e**
 463 display the ODFs for the CH samples annealed for 0.2, 1.5 and 30 s, respectively, while
 464 **Figure 10e,f,g** represent the UFH conditions soaked for 0.2, 1.5 and 30 s, respectively. The
 465 initial cold-rolled material is represented by the ND $\{111\}\langle uvw \rangle$ and RD $\{hkl\}\langle 110 \rangle$ fibers,
 466 with a maxima corresponding to $\{111\}\langle 110 \rangle$ components. Similar texture was found
 467 previously in cold-rolled low carbon steels [52,53]. On the other hand, the CH samples
 468 (**Figure 10c,d,e**) present an opposite curvature in the ND fiber compared to the initial cold-

469 rolled microstructure and lower intensity in the RD fiber. Both effects can be associated with
470 the recrystallization in the ferritic matrix [4]. In the UFH conditions (Figure 10f,g,h), the
471 ODFs display texture similar to the initial cold-rolled condition (Figure 10b), with a strong
472 intensity in the ND fiber components, indicating that complete recrystallization has been
473 delayed. However, its intensity is reduced with increasing soaking time. This effect can be
474 attributed to onset of recrystallization during intercritical annealing for >1.5 s and increasing
475 fraction of recrystallized grains with soaking time revealed by EBSD analysis (Figure 6,
476 Section 3.3), as the initial ND fiber grains in the cold rolled steel present the higher stored
477 energy [54].

478 The alpha fiber in the UFH treated material is also affected by soaking time. While a
479 significant fraction of gamma fiber components recrystallized during UFH due to higher
480 energy stored during cold rolling (compared to the alpha fiber components) [55,56], a lower
481 fraction of alpha possesses energy (i.e. driving force) sufficient for recrystallization. So the
482 RD fiber intensity is retained to large extent during UFH treatment.



483

484 **Figure 10:** Effect of heating rate and soaking time on the orientation distribution function (ODF) of
 485 the studied material for $\phi_2=45^\circ$ in the Euler space; a) Ideal BCC texture components for $\phi_2=45^\circ$ in
 486 the Euler space; b) ODF of the initial cold rolled material, reproduced from [5]; c), d) and e) ODF
 487 corresponding to the CH conditions annealed for 0.2, 1.5 and 30 s, respectively; f), g) and h)
 488 correspond to the UFH conditions soaked for 0.2, 1.5 and 30 s, respectively.

489

490 3.5. XRD analysis

491 XRD measurements were carried out to analyze the evolution of retained austenite and its
 492 carbon content with soaking time. The results are listed in **Table 3** and compared to the
 493 values obtained by TKD (see Section 3.6).

494 **Table 3.** Effect of the heating rate and soaking time on the retained austenite volume fraction and
495 its carbon content measured by XRD and TKD analysis.

Condition	XRD		TKD
	(%)	% C (wt.)	(%)
CH10-0.2s	7.9	0.77	4.8
UFH800-0.2s	6.6	0.80	8.1
UFH800-1.5s	6.9	0.77	4.9
UFH800-30s	5.2	0.70	4.4

496

497 After short annealing (soaking for 0.2 s), the CH sample presents a higher retained austenite
498 fraction compared to the UFH condition. The CH treatments lead to phase fractions closer
499 to the ones at the equilibrium condition since there is more time for the austenite to nucleate
500 and grow (Table 2). In the CH10-0.2s sample, taking into account fractions of both phases
501 (i.e. retained austenite measured by XRD in Table 3 and martensite determined by EBSD in
502 Table 2), the total fraction of austenite formed during intercritical annealing is close to 20
503 %. The effect of soaking time on the retained austenite volume fraction for the UFH samples
504 has two different trends. For short soaking times (0.2 s, 1.5 s), both nucleation and growth
505 of intercritical austenite take place, as it is observed from the martensite fraction (see Section
506 3.3). Then, the volume fraction of austenite rises slightly from 6.6 % to 6.9 % with increasing
507 time within the short range (Table 3). This effect indicates, that the nucleation stage plays a
508 more important role compared to the growth stage, as there is a significant austenite fraction,
509 which retains after rapid cooling, with a carbon concentration similar to the CH condition.
510 Eventually, when the soaking time increases up to 30 s, the austenite fraction at the peak
511 temperature increases due to the longer time to nucleate and grow, as there is a significant
512 fraction of martensitic grains having a size below 1 μm (Figure 9), but its carbon
513 concentration decreases up to 0.7 % reducing the amount of retained austenite down to 5.2
514 %.

515 The volume fractions of retained austenite measured by XRD (Table 3) are considerably
516 higher than the values determined by EBSD (Table 2). This effect is produced by the large
517 difference in the depth of the analyzed area being approximately 1 μm for XRD and 50 nm
518 for EBSD [57]. As is well known, the metastable retained austenite generates a local increase
519 in volume during transformation into martensite [58]. As phase transformation on the surface
520 allows an easier accommodation of this volume change, the surface retained austenite grains

521 are more prone to phase transformation during sample preparation, that reduces the amount
522 of retained austenite detected by EBSD [57]. Meanwhile, XRD is able to detect retained
523 austenite present in the bulk material, which has not transformed into martensite. Moreover,
524 it should be noted that although the spatial resolution of the EBSD is reasonably high (65
525 nm in step size), it is not sufficient for detection of the finest austenite grains present in the
526 microstructure, revealed by TEM analysis (see Section 3.6). Similar conclusions were drawn
527 for other steel grades containing metastable austenite, such as Q&P steels in [59,60].

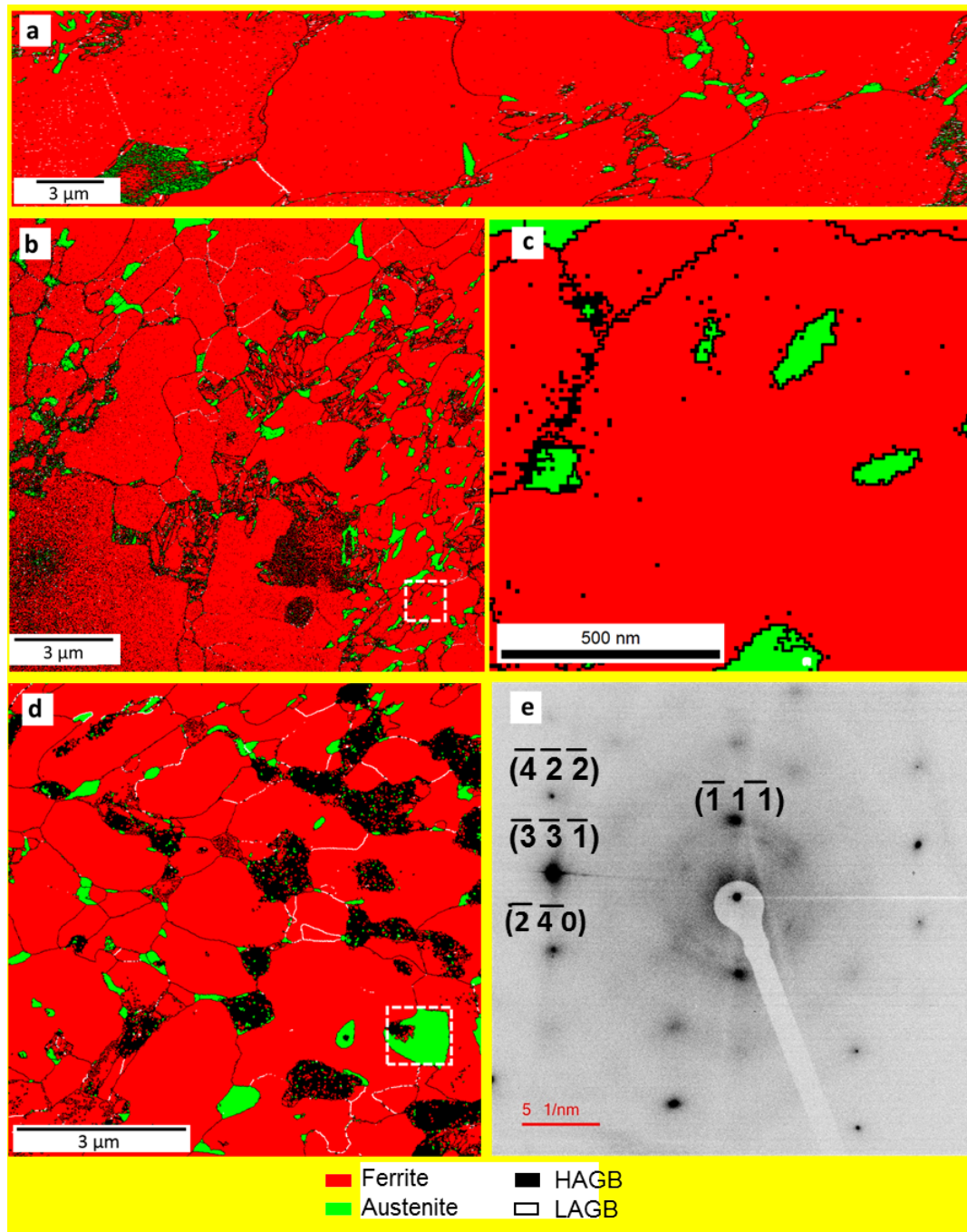
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529 3.6. TEM and TKD analysis

530 To study the evolution of microstructure during soaking on nanoscale, TKD analysis
531 combined with TEM characterization were carried out on CH10-0.2s and UFH after 0.2, 1.5
532 and 30 s samples. Figure 11 represents the phase maps of the different samples analyzed by
533 TKD. They are in a good accordance with the outcomes of the EBSD measurements
534 presented above (see Section 3.3). Larger ferritic grains are observed in the CH10-0.2s
535 samples (Figure 11a) compared to those seen in the UFH samples (Figure 11b,c,d). In
536 addition, the CH treatment results in equiaxed ferritic grains without LAGBs in their interior
537 (Figure 11a) due to the longer treatment time, while the UFH leads to an inhomogeneous
538 microstructure with varying grain size and a higher fraction of LAGBs (Figure 11b,c,d).

539 Values of retained austenite volume fraction measured by TKD are provided in Table 3.
540 They are higher compared to those determined by EBSD. This effect is caused by higher
541 spatial resolution of the TKD technique, which enables to resolve nanoscale microstructural
542 constituents having 10-30 nm in size [27]. Discrepancies between the volume fractions of
543 retained austenite determined by XRD and those measured by TKD should also be noted.
544 Unlike in the XRD measurements, a very local area is analyzed by TKD which leads to
545 statistically insignificant data. Moreover, the TKD results highly depend on the quality of
546 the studied samples. If the electropolishing step is inhomogeneous, there are significant
547 differences in the foil thickness through the sample. If a local area is too thick, the electrons
548 are unable to pass through and reach the detector, as their initial energy is orders of
549 magnitude less compared to the ones generated in TEM which results in the non-indexed
550 areas. Similar effect occurs when the foil is too thin, as too many electrons cross the
551 specimen and reach the detector [26,61]. Diffraction patterns were taken from different

552 austenitic regions observed by TKD in all samples, in order to prove the presence of austenite
 553 in the material, as it is shown in **Figure 11e**).

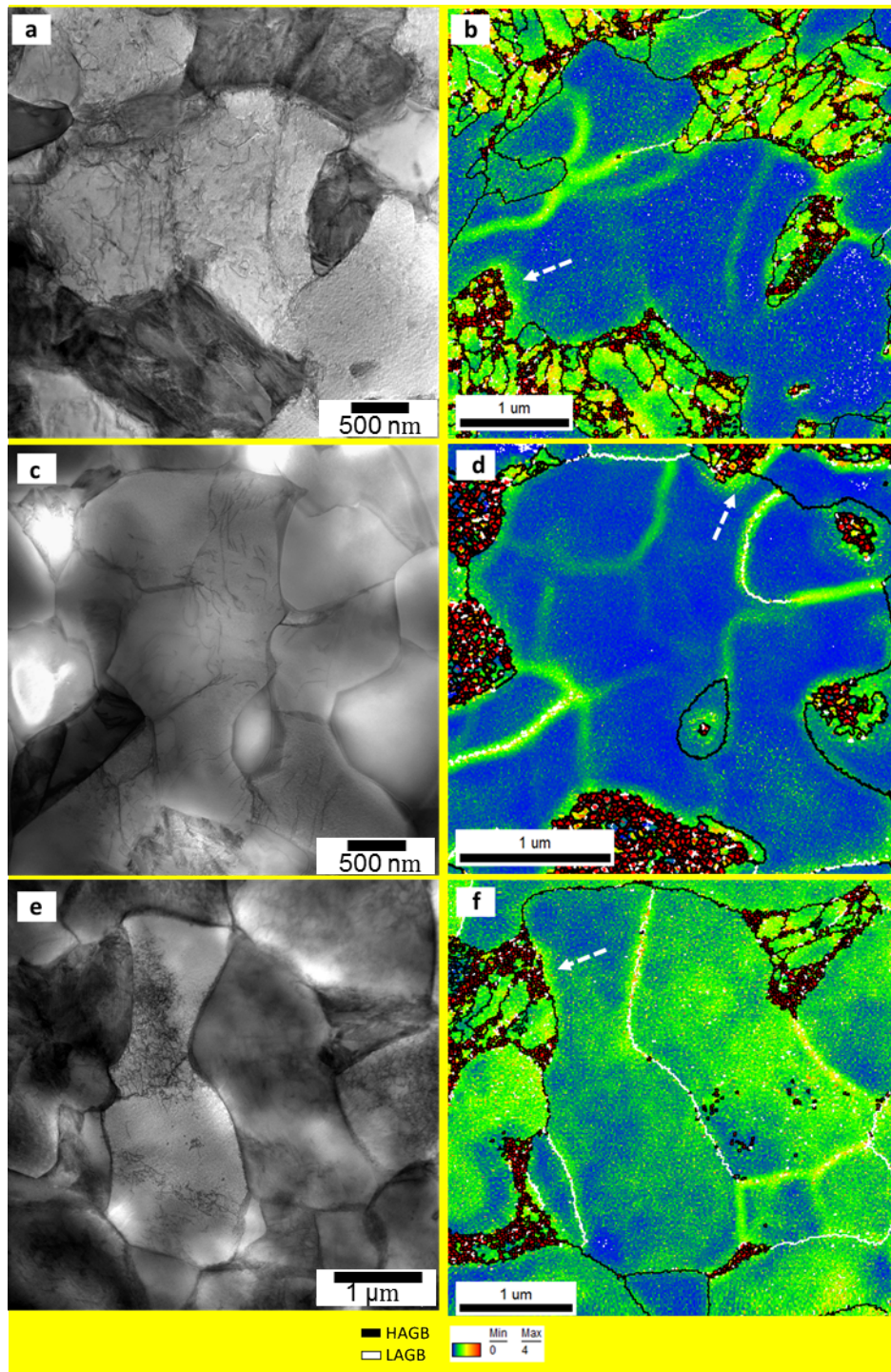


554

555 **Figure 11:** Phase maps obtained from TKD analysis in a) CH10-0.2s and UFH for 0.2 s (b & c),
 556 and 1.5 s (d). Figure c) shows a detailed region in figure b). Figure e) represents the diffraction
 557 pattern of the austenite marked in figure d). Ferrite is shown in red and austenite in green. HAGBs
 558 are represented in black and LAGBs in white. Large regions in black are areas with a confidence
 559 index (CI) lower than 0.1.

560 **Figure 12a,c,e** shows TEM images illustrating microstructure evolution during UFH
 561 treatment of the steel within the non-recrystallized areas (as discussed in Sections above).

562 **Figure 12b,d,f** illustrate the corresponding KAM maps of the corresponding regions
563 extracted from the TKD analysis. Formation of dislocation walls and other configurations is
564 observed after UFH 0.2 s treatment, which are represented in form of lines with local
565 misorientation $< 1^\circ$ on KAM maps (**Figure 12a,b**). Dislocation walls associated to recovery
566 were reported elsewhere [49,62]. Longer soaking time of 1.5 s allows further dislocation
567 climb and rearrangement and onset of LAGBs formation (**Figure 12c,d**). Finally, annealing
568 for 30 s results in formation of an energetically favorable substructure in the grain interior
569 (**Figure 12e**) with local misorientation at LAGBs reaching 4° (**Figure 12f**). In **Figure 12e**,
570 f, enhanced local dislocation density and increased local misorientation are clearly seen also
571 in the ferritic matrix near the martensite/ferrite interface (marked by white arrows). It is
572 related to accommodation of the plastic micro-strain induced by the volume expansion due
573 to the austenite/martensite transformation during rapid cooling. This observation was
574 reported earlier for DP steels [63].



575

576 **Figure 12:** TEM images after UFH treatment for a) 0.2 s, c) 1.5 s and e) 30 s; KAM maps for b)
 577 0.2 s, d) 1.5 s and f) 30 s obtained from the TKD analysis. White dashed arrows indicate the
 578 increase in misorientation in the ferritic matrix due to the martensite formation. (HAGBs in black,
 579 LAGBs in white).

580 The outcomes of this study clearly indicate that the microstructure of the low carbon steel is
 581 very sensitive to the soaking time at the peak temperature during UFH treatment. This
 582 provides an additional tool for microstructural design in carbon steels by manipulating also
 583 the soaking time in addition to the heating rate [5] and initial microstructure [12] of steels.

584 Grain size, volume fraction of martensite, volume fraction of non-recrystallized and
585 recrystallized ferrite can be optimized via the correct balance of the heat treatment
586 parameters, so steels with the excellent combination of high strength and ductility can be
587 manufactured [5]. The approach can be applied to all carbon steels.

588

589 4. Conclusions

590 The effect of heating rate and soaking time on the microstructure of the heat-treated low
591 carbon steel was studied using SEM, EBSD, XRD, TKD and TEM techniques. The
592 following conclusions can be drawn.

- 593 1. A complex multiphase, hierarchic microstructure mainly consisting of ferritic matrix with
594 embedded martensite and retained austenite is formed after all applied heat treatments.
595 There is significant effect of soaking time on the microstructure of the UFH treated steel,
596 while it does not affect the microstructure evolved in the CH treated material.
- 597 2. There is a strong effect of heating rate on the microstructure of the ferritic matrix. The
598 CH treatment results in the ferritic matrix consisting mainly of equiaxed recrystallized
599 grains independently on the soaking time, while fine recrystallized grains and larger non-
600 recrystallized (i.e. recovered) ferritic grains are present in all UFH treated conditions. The
601 fraction of recrystallized ferritic grains generally tends to increase with increasing
602 soaking time. Combined TEM and TKD study proved directly that the recovery process
603 starts with formation of dislocation walls via dislocation climb and rearrangement, which
604 gradually transform into LAGBs.
- 605 3. Volume fraction of martensite tends to increase with increasing soaking time during UFH
606 treatment due to suppression of cementite spheroidization, which, in turn, reduces the
607 amount of energetically favorable sites for austenite nucleation and results in longer
608 soaking time to reach the equilibrium at the inter-critical peak temperature.
- 609 4. Based on the outcomes of the XRD analysis, it is possible to conclude that UFH
610 treatments results in slightly lower amount of retained austenite compared to CH
611 treatment. The amount of retained austenite and carbon content therein tend to slightly
612 decrease with increasing soaking time after UFH treatment due to lower carbon gradients
613 in the material before rapid cooling.

- 614 5. TKD analysis allows to precisely identify and analyze the retained austenite nanograins
615 and other nanoscale elements of the complex microstructure along with the local
616 misorientations due to dislocation generation and **rearrangement**.
- 617 6. TKD and TEM proved that local volume expansion due to austenite-martensite phase
618 transformation during rapid cooling induces dislocations into the ferritic grains.

619

620 **Acknowledgements**

621 MAVT acknowledges gratefully the financial support by IMDEA Innovation Award.

622

623 **Data availability statement**

624 The raw/processed data required to reproduce these findings cannot be shared at this
625 time as the data also forms part of an ongoing study.

626

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