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1 Revisiting the formation mechanism of intragranular 2 κ -carbide in austenite of a Fe-Mn-Al-Cr-C 3 low-density steel

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16 Abstract

17 It was usually believed that the formation of intragranular κ -carbide in γ -austenite
18 was attributed to spinodal decomposition followed by ordering reaction. In this work,
19 near-atomic scale characterization of an austenite-based Fe-20Mn-9Al-3Cr-1.2C
20 (wt. %) low-density steel, using (high-resolution) scanning transmission electron
21 microscopy and atomic probe tomography, reveals that the initially-formed κ -carbides
22 (2-3 nm in particle size) are featured with an ordered L'1₂ structure but without
23 detectable chemical partitioning. Further, the Gibbs energy of the FCC phase obtained
24 by thermodynamic calculations always shows a positive curvature (i.e. $\frac{d^2G}{dx^2} < 0$) with
25 the variable contents of Al and C in the temperature range 400-800 °C. Both the
26 results demonstrate that the ordered nuclei of κ -carbide can form directly in the
27 disordered γ -austenite rather than through the well-known spinodal
28 decomposition-ordering mechanism. The extremely low nucleation barrier is due to
29 the similar lattice structure, same composition and complete coherency between the
30 γ -austenite matrix and κ -carbides.

31 **Keywords:** Low-density steel, κ -carbide, Spinodal decomposition, Atom probe
32 tomography (APT), Thermodynamic calculation.

33
34 Precipitation strengthening promoted by nano-scale κ -carbides in austenite-based

35 Fe-Mn-Al-C low-density steels has received much attention since such shearable and
36 coherent precipitates within the γ -austenite matrix significantly enhance strength
37 without sacrificing too much elongation [1-6]. The ideal stoichiometry of
38 intragranular κ -carbide is $(\text{Fe, Mn})_3\text{AlC}$, and it has a perovskite crystal structure
39 designated as $E2_1$ in the Strukturbericht classification [1]. Based on observations of
40 electron diffraction patterns and calculations of structure factors, an elemental
41 site-occupancy in the κ -carbide was summarized as that Fe and Mn atoms were
42 located at face center positions, Al atoms at corner positions, and C atom at body
43 center octahedral ($\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$) interstitial position of this structure [1, 7, 8]. It was
44 reported that the κ -carbide could initially form at the solid solution state [4, 9-11], and
45 its coarsening occurred during isothermal aging treatments in the temperature range
46 450-600 °C [1, 12-14].

47 It has long been believed that in most austenite matrix alloys, the formation of
48 intragranular κ -carbide is through spinodal decomposition and following ordering
49 reaction [1, 15, 16]. Its formation sequence is generally described as follows [1]: a
50 spinodal decomposition divides high temperature γ -austenite into the solute-lean (C
51 and/or Al) phase γ' and the solute-rich phase γ'' ; then the solute-rich phase γ''
52 transforms into the ordered $L1_2$ phase, in which Al and Fe/Mn atoms are located at
53 corner and face center sites, respectively; finally, the κ -carbide with $E2_1$ structure
54 forms by further ordering of C atoms. Usually, the non-stoichiometric κ -carbide ($(\text{Fe,}$
55 $\text{Mn})_3\text{AlC}_x$) is observed being referred as $L'1_2$ ordered phase.

56 In the early years, conventional transmission electron microscopy (TEM) and
57 X-ray diffraction (XRD) were used as the main tools to provide experimental
58 evidence supporting the spinodal decomposition-ordering mechanism by observing
59 the modulated structure [17], diffuse satellites around the (020) diffraction spots in
60 electron diffraction patterns and XRD sideband peaks around the (200) reflections [16,
61 18]. Over recent years, the composition uncertainty of κ -carbide was quantitatively
62 resolved at near-atomic scale by atom probe tomography (APT) [4, 7, 12]. The
63 concentration profiles with sinusoidal curves across the γ -austenite matrix and
64 developed κ -carbide were found to confirm the occurrence of spinodal decomposition
65 during κ -carbide precipitation [12, 19].

66 However, very recent TEM and APT results obtained in a Fe-30Mn-9Al-1.2C
67 (wt. %) lightweight steel surprisingly indicated that the formation of an ordered
68 structure was earlier than chemical partitioning of any solute elements during the
69 early stage of κ -carbide precipitation [4]. This observation obviously challenged the
70 general understanding of the κ -carbide formation process. In fact, the study of
71 κ -carbide mainly focused on the coarsened κ -carbide at the aged states where both
72 $L'1_2$ ordered structure and chemical partitioning were observed. Comparatively, the

73 study of the κ -carbide formation at the initial stage is still very limited due to tiny size
74 of κ -carbide in the range of a few nanometers and complete coherency with
75 γ -austenite. Therefore, the understanding of κ -carbide formation process at the initial
76 stage has still been insufficient. The objective of this work is to revisit the formation
77 mechanism of κ -carbide in an austenite-based Fe-20Mn-9Al-3Cr-1.2C (wt. %)
78 low-density steel at near-atomic scale by probe-corrected (high-resolution) scanning
79 TEM ((HR)STEM), APT and thermodynamic calculation.

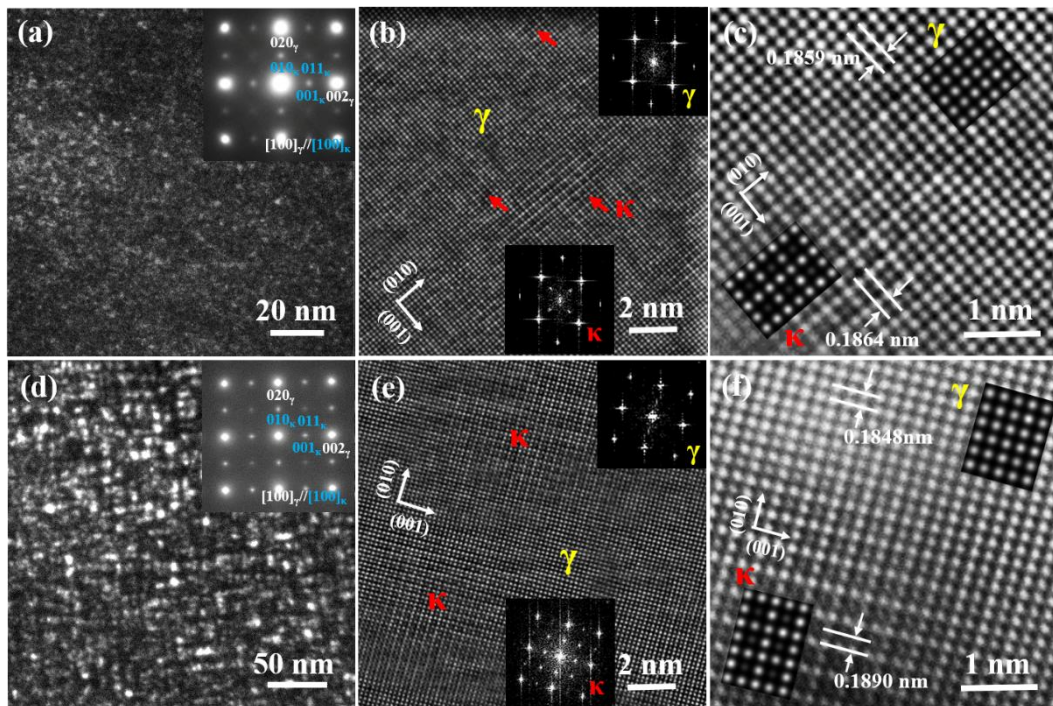
80 As detailed in our previous works [9, 20], the steel sample was prepared by
81 centrifugal casting under near-rapid solidification in an argon atmosphere. Molten
82 metal was poured directly into a copper mold and solidified at a cooling rate higher
83 than 10^3 K/s [21]. Isothermal aging was carried out under vacuum at 600 °C for 9 h,
84 followed by furnace cooling. Chemical composition of the as-cast steel, measured by
85 an inductively coupled plasma-atomic emission spectrometry (ICP-AES) and a
86 carbon-sulfur analyzer (CS2800), was Mn 19.70 wt. %, Al 8.86 wt. %, Cr 2.90 wt. %,
87 C 1.21 wt. % and Fe balance.

88 TEM examination was carried out using a FEI Tecnai G2-20 twin and a
89 Thermofisher Titan Themis 300 microscopes, operated at 200 and 300 kV,
90 respectively. The latter was equipped with a probe aberration corrector allowing to
91 acquire (HR)STEM images. The probe size was set to 0.1 nm with a convergence
92 semi-angle of 22.5 mrad. Collection angle of the high-angle annular dark-field
93 (HAADF) imaging detector was in the range 80-150 mrad. TEM specimens were
94 prepared by twin-jet electrolytic polishing at -30 °C and 30 V, with a mixed solution
95 of 10 % perchloric acid and 90 % alcohol. Dr. probe software was used for
96 atomic-scale HAADF image simulation [22]. Standard two-step electro-polishing was
97 used to prepare needle-like specimens for APT using a local electrode atom probe
98 instrument (CAMECA LEAP 4000 XHR), in laser-pulsing mode with a repetition rate
99 of 200 kHz, a pulse fraction of 15 %, and a specimen temperature of 50 K.
100 Post-treatment of APT data was done using IVAS 3.6.8 software (CAMECA software
101 suite). The peak decomposition algorithm in IVAS was used to resolve the peak
102 overlaps among Fe, Al and Cr at 27 Da in mass spectrum, which has been proven to
103 significantly improve counting statistics [23, 24].

104 Thermodynamic calculations were performed by Thermo-Calc software using
105 previously-created thermodynamic database of low-density steels [25-29].
106 Partitioning model based on the Compound Energy Formalism (CEF) was employed
107 to describe disordered and ordered forms (such as $L1_2$ and $L1_0$) of the FCC phase in
108 the database and the detailed calculation process was given in [Supplementary](#).

109 The DF-TEM images (in Fig. 1 (a) and (d)) show that the homogeneously

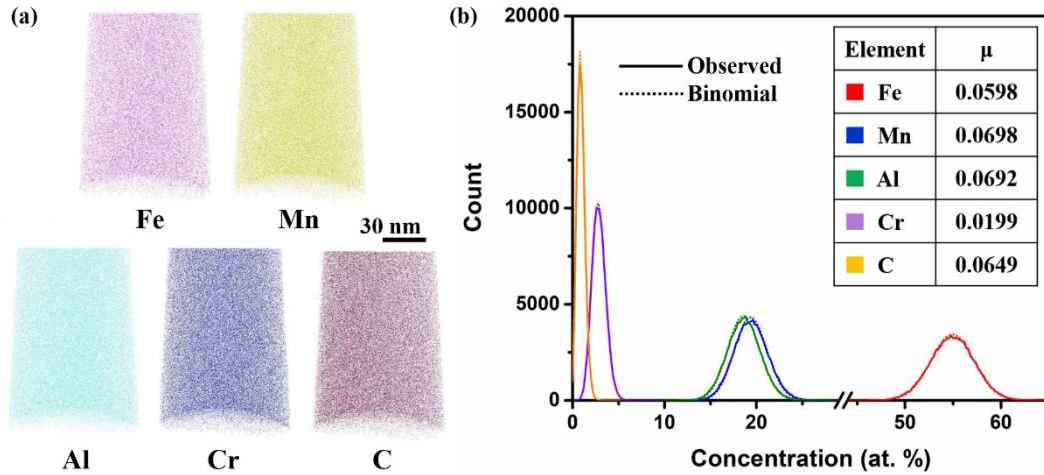
110 dispersed κ -carbide precipitates are present at the as-cast and aged states, and have the
 111 average sizes of around 3 and 10 nm, respectively. Being consistent with the previous
 112 results [1], the selected area diffraction patterns (SADPs, insets in Fig. 1 (a) and (d))
 113 show that the κ -carbides have a cube-on-cube orientation relationship with the
 114 γ -austenite: $[100]_{\kappa} // [100]_{\gamma}$ and $(100)_{\kappa} // (100)_{\gamma}$. By measuring the $\{100\}$ and $\{110\}$
 115 superlattice reflection intensity in SADP shown in Fig. 1 (a) and (d), the intensity
 116 ratios of $I_{(011)}/I_{(001)}$ in the as-cast and aged samples are 0.52 and 0.25, respectively.
 117 This suggests that the ordering of C atoms in the body-center octahedral sites is
 118 gradually improved with the growth of κ -carbides. It was reported that the intensity of
 119 the $\{100\}$ superlattice spots is higher than the $\{110\}$ ones in $L'1_2$ structure [17, 30,
 120 31]. Since theoretically the intensity of the superlattice reflections is proportional to
 121 square of the structure factor (F_{hkl} , h , k and l are the Miller indices), the $L'1_2$ structure
 122 with partially C ordering can be distinguished [32]. Contrasts in an HAADF image is
 123 proportional to $Z^{-1.7-2}$ [33], meaning that the bright contrast indicates Cr- ($Z=24$), Mn-
 124 ($Z=25$) and/or Fe-rich ($Z=26$) atomic columns, while dark contrast represents Al-
 125 ($Z=13$) and/or C-rich ($Z=6$) atomic columns in this case. Thus, the individual
 126 κ -carbide precipitates can be identified using FFT patterns shown in Fig. 1 (b) and (e),
 127 which have the fully coherent interface with the γ -austenite matrix and no interfacial
 128 misfit dislocation is visible. Although the exact atomic occupations of Cr and Mn are
 129 unknown, the contrasts of the κ -carbides approximately correspond to those of the
 130 simulation results using the $E2_1$ structural model shown in Figs. 1 (c) and (f).



131

132 Fig. 1 (HR)(S)TEM characterization of the as-cast (a-c) and aged steels (d-f): (a) and (d) are

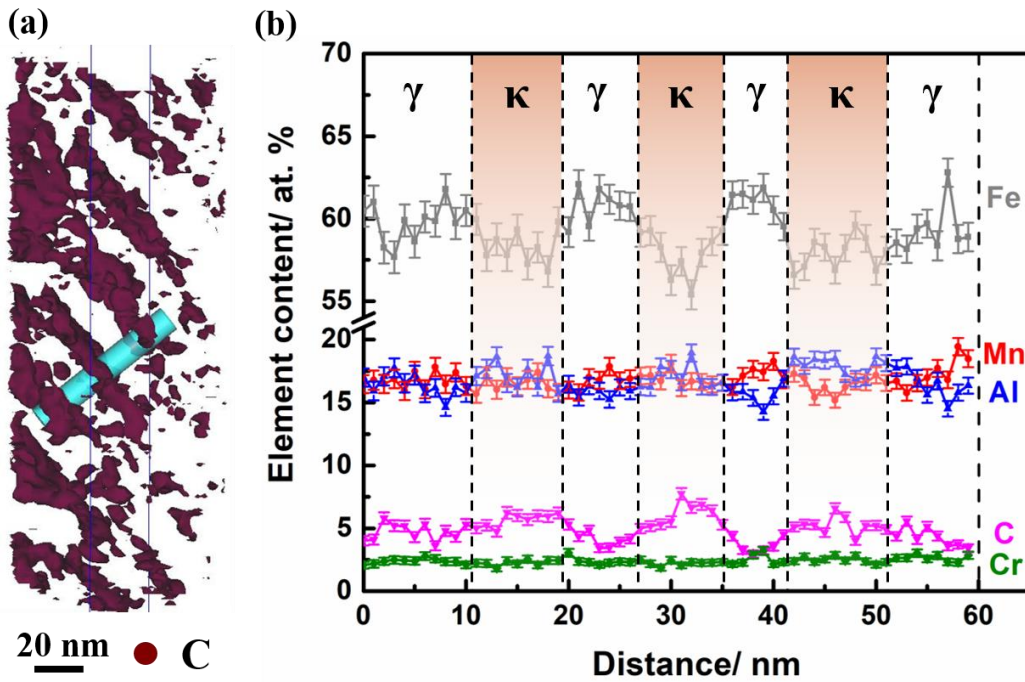
133 dark-field (DF) TEM images, insets are corresponding SADP of $[100]_{\gamma}$ zone-axis; (b) and (e) are
 134 filtered HRSTEM-HAADF images along $[100]_{\gamma}$ zone axis, insets are corresponding *fast Fourier*
 135 *transform* (FFT) patterns of γ -austenite and κ -carbide; (c) and (f) are zoom-in images of (b) and (e)
 136 respectively, overlapped images are simulated HRSTEM-HAADF images using κ -carbide ($E2_1$
 137 Fe_3AlC) and γ -austenite (FCC Fe) structure models.



138
 139 Fig. 2 APT characterization of γ -austenite in the as-cast steel: (a) Fe, Mn, Al, Cr and C elemental
 140 maps; (b) frequency distribution analysis obtained by comparing the observed experimental results
 141 and the binomial simulation, and the values of normalized homogenization parameter μ are listed
 142 in the inserted table in (b).

143 The APT result in Fig. 2 (a) shows that the distributions of all the five solute
 144 elements (Fe, Mn, Al, C and Cr) are visibly uniform in γ -austenite of the as-cast steel.
 145 According to the TEM-DF image shown in Fig. 1 (a), the density of the κ -carbides
 146 with a size of 3 nm is almost $3/100 \text{ nm}^2$. It is estimated to have at least 294 observable
 147 κ -carbide particles in the 2-dimensional surface of the APT-reconstructed map if
 148 chemical ordering exists. However, no chemical partitioning caused by the formation
 149 of tiny κ -carbides (confirmed by TEM in Fig. 1 (a-c)) is detected. As shown in Fig. 2
 150 (b), the observed data (solid line) fit with the binomial random distribution model
 151 (dotted line), and the values of the normalized homogenization parameter μ are all
 152 close to 0, indicating the random distribution of all the elements at the near-atomic
 153 scale [34-36]. Comparatively, the APT result of the γ -austenite in the aged steel (Fig.
 154 3(a)) shows that the reconstructed intragranular κ -carbides are distributed along a
 155 certain direction. The concentration profile in Fig. 3(b) shows the clear fluctuation of
 156 the different elements traversing the γ -austenite and κ -carbides. The κ -carbides are
 157 obviously rich in C and Al and poor in Fe, while the distributions of Mn and Cr
 158 between the γ -austenite and κ -carbide are almost uniform. Thus, putting together the
 159 HRSTEM and APT results clearly indicates that the chemical partitioning of solute

160 elements is unnecessary for the formation of the ordered κ -carbides.

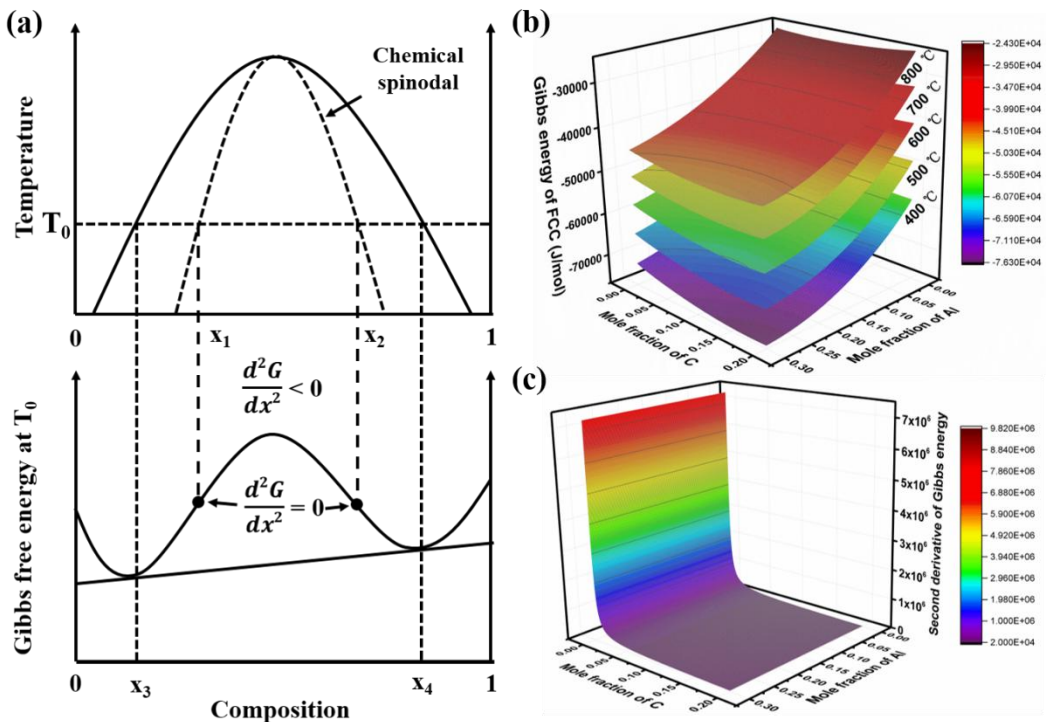


161

162 Fig. 3 APT characterization of γ -austenite in the aged steel: (a) reconstructed C map showing

163 intragranular κ -carbides with an iso-concentration surface at 6 at. % C; (b) 1 D concentration

164 profile generated by the 10 nm diameter virtual cylinder in (a).



165

166 Fig. 4 (a) Schematic phase diagram of miscibility gap and the corresponding free energy curve

167 [37]; (b) calculated Gibbs energy of FCC phase as a function of mole fractions of Al and C in

168 Fe-20Mn-9Al-3Cr-1.2C (wt. %) steel at the temperature range 400-800 °C; (c) calculated second
169 derivative (curvature) of the Gibbs energy at 600 °C in (b).

170 Thermodynamically, the spinodal decomposition is a transformation without
171 activation energy barrier and can result in a decreased total Gibbs energy [37]. When
172 it occurs to the high temperature γ -austenite, the γ' and γ'' phases form with the
173 change of C and Al contents. Due to the same FCC crystal structure of the γ' and γ''
174 phases, the Gibbs energy of both phases will move along the same Gibbs
175 energy-composition curve. To ensure the decreased total Gibbs energy of FCC phase,
176 the Gibbs energy-composition curve has a negative curvature [37], i.e. $\frac{d^2G}{dx^2} < 0$, as the
177 same as the composition range between x_1 and x_2 shown in Fig. 4 (a). A small
178 composition fluctuation decreases its total free energy to give rise to the spinodal
179 decomposition by up-hill diffusion. Outside this composition range, a small
180 composition fluctuation results in an increase of the free energy, which makes the
181 alloy unstable. To decrease the total free energy, an energy barrier must be overcome
182 so that the nucleation and growth mechanism dominates. The calculated results (Fig.
183 4(b)) show that the evolution trend of Gibbs energy of the FCC phase is the same at
184 the large temperature range 400-800 °C. At each given temperature, the Gibbs energy
185 decreases with increasing the mole fractions of Al and C, while its curvature remains
186 always positive (the case of 600 °C shown in Fig. 4(c) and those of the other
187 temperatures in [Supplementary](#)) ruling out the spinodal decomposition of $\gamma \rightarrow \gamma' + \gamma''$.
188 It should be further noted that the calculated Gibbs energy in Fig. 4 (b) originates
189 totally from the disordered FCC phase. The fact that the ordering reaction of $\gamma'' \rightarrow$
190 $L1_2$ phase has no contribution indicates that it is thermodynamically unfavorable.

191 According to the classical nucleation theory in solids [37], the minimum
192 activation energy barrier for the κ -carbide precipitation in γ -austenite matrix is
193 described as:

$$194 \quad \Delta G^* = \frac{16\pi\gamma_{\kappa/\gamma}^3}{3(\Delta G_V - \Delta G_S)^2} \quad (1)$$

195 where $\gamma_{\kappa/\gamma}$ is interfacial energy between the κ -carbides and γ -austenite, ΔG_V is
196 chemical driving force of κ -carbides per unit volume and ΔG_S is elastic strain energy.
197 In equation (1), the interfacial energy and elastic strain energy can be further
198 expressed as [38]:

$$199 \quad \gamma_{\kappa/\gamma} = \frac{\varepsilon(c-c_0)^2}{d^2} \quad (2)$$

$$200 \quad \Delta G_S = \frac{E\delta^2(c-c_0)^2}{1-\nu} \quad (3)$$

201 with lattice misfit $\delta = \frac{a_\gamma - a_\kappa}{a_\kappa}$ (4)

202 where ε is pair interaction energy, c is nucleus composition, c_0 is nominal composition,
 203 d is interface thickness, E is Young modulus, ν is Poisson coefficient, a_κ and a_γ are
 204 lattice parameters of the κ -carbides and γ -austenite, respectively.

205 At the initial stage of the κ -carbide formation, the fully coherent interface
 206 between the κ -carbide and γ -austenite matrix is revealed in Fig. 1 (b). The interfacial
 207 energy of κ -carbide precipitates in Fe-Mn-Al-C steel was calculated to be 0.025 J/m²
 208 [29]. For comparison, it is much lower than the interfacial energy of 0.115 J/m²
 209 necessary to form GP zones from the Al matrix in Al-Cu alloy [39]. The lattice misfit
 210 (δ) between the κ -carbide and γ -austenite is estimated to about 0.27 % in term of the
 211 HRSTEM-HAADF image in Fig. 1 (c), which results in the low ΔG_S (measured lattice
 212 parameters in [Supplementary](#)). More importantly, the nearly consistent composition
 213 between the κ -carbides and γ -austenite matrix revealed by APT (see Fig. 2) indicates
 214 that the critical nucleation composition of κ -carbide can be very close to that of the
 215 γ -austenite matrix, which makes the value of $(c - c_0)$ close to zero. All the results
 216 mean that there are both very low interfacial energy and elastic strain energy at the
 217 initial stage of κ -carbide formation. Therefore, the activation energy barrier to the
 218 nucleation of κ -carbides in γ -austenite matrix should be rather small. In addition, the
 219 large undercooling, due to a high cooling rate of more than 10³ K/s during the
 220 near-rapid solidification [21], also increases the driving force for κ -carbide
 221 precipitation [37, 40].

222 Table 1 Quantitative chemical compositions of γ -austenite matrix and κ -carbide precipitates
 223 in the as-cast and aged steels obtained by APT analyses (in at. %).

Steel	Phase	Fe	Mn	Al	C	Cr
As-cast	γ/κ	58.42 ± 0.45	16.73 ± 0.11	17.50 ± 0.25	4.87 ± 0.25	2.48 ± 0.11
Aged	γ	60.41 ± 0.50	16.71 ± 0.13	15.95 ± 0.27	4.39 ± 0.26	2.54 ± 0.07
	κ	56.00 ± 0.29	16.43 ± 0.12	18.22 ± 0.22	6.37 ± 0.25	2.45 ± 0.17

224 Further, density functional theory calculations [41, 42] suggest that the Fe, Mn
 225 and Cr atoms are usually considered to be located at the face center positions of the
 226 ideal (Fe, Mn)₃AlC lattice. Assuming that such positions in the initially formed
 227 κ -carbide are completely occupied and as shown in Table 1, the occupancy rates of Al
 228 and C atom positions are determined to be 67.6 % and 18.8 %, respectively. This
 229 indicates that there is a large amount of quenched-in vacancies in the κ -carbides
 230 effectively promoting nucleation by increasing diffusion rates or relieving misfit
 231 strain energies [37]. Simultaneously, the presence of vacancies is a critical way to
 232 achieve ordering structures in non-stoichiometric phases [37]. The formation of an

233 ordered structure by nucleation only requires the short-range movement of atoms in a
234 crystal lattice, which is much easier to achieve than the spinodal decomposition [43].

235 With the growth of κ -carbides after the aging treatment, their composition
236 gradually approaches the equilibrium composition with the higher contents of Al and
237 C. The interfacial energy and elastic strain energy also increase by the increased
238 composition difference ($c - c_0$) between the κ -carbides and γ -austenite matrix (see
239 Table 1), and the increased lattice misfit (δ) of 2.27 % obtained by HRSTEM-HAADF
240 image (Fig. 1 (f)) and equation (4). The increased lattice misfit (δ) resulted from the
241 partitioning of Al and C to the κ -carbides [4, 7, 12]. This indicates that nucleation of
242 the equilibrium κ -carbides directly in the γ -austenite matrix is difficult. Therefore,
243 there must be a change in composition and associated crystal structure accompanying
244 with the nucleation and growth process.

245 In summary, the formation mechanism of the intragranular κ -carbide in
246 γ -austenite of the austenite-based Fe-20Mn-9Al-3Cr-1.2C low-density steel was
247 revisited in terms of (HR)STEM, APT and thermodynamic calculation. It was
248 experimentally observed that the κ -carbide precipitates of 2-3 nm in particle size were
249 homogeneously distributed in γ -austenite of the as-cast steel. They corresponded to the
250 ordered $L'1_2$ structure without elemental partitioning at near-atomic scale. The
251 thermodynamic calculations indicated that the Gibbs energy of the FCC phase always
252 had a positive curvature with the variable contents of Al and C in the temperature
253 range 400-800 °C. These results clearly show that the formation of the intragranular
254 κ -carbide in the as-cast and aged steels obeys the nucleation and growth mechanism
255 rather than the well-known spinodal decomposition-ordering mechanism. The similar
256 lattice structure, same composition and complete coherency between the γ -austenite
257 and κ -carbide resulted in an extremely low nucleation barrier. With the growth of
258 κ -carbides after aging, their composition tended to be stable, which increased the
259 composition fluctuation between the γ -austenite and κ -carbide.

260

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