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Meriem Ben Haj Slama, Nathalie Gey, Lionel Germain, Jean Christophe Hell, Kangying Zhu, et al.. Fast Granularization of Lath-Like Bainite in FeNiC Alloys During Isothermal Holding at M_s+20 K (+20 degrees C). Metallurgical and Materials Transactions A, 2016, 47 (1), pp.15-18. 10.1007/s11661-015-3209-5 . hal-01292800

HAL Id: hal-01292800

<https://hal.science/hal-01292800>

Submitted on 3 Dec 2020

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Communication

Fast Granularization of Lath-Like Bainite in FeNiC Alloys During Isothermal Holding at Ms+ 20 K (+20 °C)

MERIEM BEN HAJ SLAMA, NATHALIE GEY, LIONEL GERMAIN, JEAN CHRISTOPHE HELL, KANGYING ZHU, and SÉBASTIEN ALLAIN

A fast «granularization» of lath-like bainite is observed in an Fe5Ni0.13C (wt pct) alloy during isothermal holding at Ms+ 20 K (+20 °C). The microstructure evolution with holding time is characterized by Scanning Electron Microscopy and Electron BackScatter Diffraction. Granularization starts once the bainitic transformation is finished and degrades the mechanical properties. The granularization involves a rapid decrease in 60 deg $\langle 111 \rangle$ misoriented bainite laths to a dislocated granular bainite, followed by a slower dislocation rearrangement to sub-grain boundaries and more random boundaries. During the granularization, the carbides go through a ripening process.

Bainitic steels have always provided a rich field of study, with many types of bainite already described in the literature. In many cases, these types of bainite have been discovered by virtue of industrial application progresses, resulting in several proposed classifications of bainite. Each classification approach has its own viewpoint and aims at clarifying the links between the observed bainitic microstructures (phases, textures, morphology, and topology) and either the processing conditions (transformation temperatures, cooling rates) or the associated steel properties (toughness, hardness...).

Mehl's classification,^[1] for instance, pays special attention to the nature of the constituent phases and their relative locations in bainite. The classification distinguishes upper bainite consisting of aggregates of ferrite plates separated by untransformed austenite, martensite, or cementite,^[2] from lower bainite that contains additionally intra-lath cementite particles. The classifications of Aaronson *et al.*, Ohmori *et al.*, or Bramfitt *et al.* are based on similar concepts derived from thermo-kinetic analysis.^[3-5]

Recently, Zajac *et al.*^[6] suggested a new definition or classification of bainites that is based on the misorientation angle distribution between bainitic sub-units. Neighboring grains or laths with different crystallographic variants inherited from the same austenite grain exhibits characteristic misorientations fully determined by the $\gamma \rightarrow \alpha$ orientation relationships (OR).^[7] The relative frequency of these variants depends on the transformation conditions. Zajac and coworkers showed that this frequency differs significantly in upper and lower bainites. Upper bainite presents a high proportion of low-misorientation boundaries (with a high peak for misorientation angles in the range of 8 to 20 deg) and a small proportion of high-misorientation boundaries (small peak at misorientations >50 deg). The opposite distribution characterizes lower bainite. The angular domain between 20 and 50 deg contains no characteristic misorientations and instead reveals misorientation angles between sub-units inherited from differently oriented austenite grains. More recently, advanced crystallographic analysis confirmed differences in variant pairing of bainitic ferrite with transformation temperature.^[8,9]

Regardless of the classification approach, bainitic lath-like structures are generally well described. Contrary to this, «granular» bainite has not been well classified in the literature and its forming mechanism is rarely discussed. This form of bainite is often reported in low-carbon steels. It is crudely described as irregular ferrite grains and may contain different second phases (cementite debris, degenerated perlite, and M/A islands). As far as Zajac's approach is concerned, the misorientation distribution between ferrite units in granular bainite is close to random.

granular bainite consists either of coarse plates or entirely of granular shaped bainitic ferrite, with islands of retained austenite and martensite. These authors indicate that one of its

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characteristic features is a lack of carbides. However, granular bainite containing cementite as a second phase has been observed after a continuous cooling treatment.^[14] Bhadeshia reported that granular bainite occurs only in continuously cooled low-carbon steels and cannot be produced by isothermal transformations.^[2] A similar conclusion was drawn by Krauss *et al.*^[15]

In the present work, we suggest an original experiment that indicates the formation of “granular” bainite from the decomposition of an intermediate lath structure during prolonged isothermal treatment. The process is rather fast in the studied alloy. The formation could result from similar mechanisms as those operating during continuous cooling. Thus, this research provides new insights into the formation mechanism of granular bainite and may contribute to the refinement of bainite classification.

The composition of the investigated steel was Fe-0.13C-5Ni (wt pct). Ni is known to improve the hardenability. Compared to Mn, Ni has the advantage of avoiding micro-segregation issues and speeds up the bainitic transformation.

A 15 kg ingot was cast in a vacuum furnace and hot- and cold rolled, respectively, to the thicknesses of 3 and 1 mm. Samples were cut from this 1-mm-thick cold-rolled plate and thermally treated using Bahr dilatometer. After austenizing for 5 minutes at 1093.15 K (820 °C), the samples were rapidly cooled [95 K/s (95 °C/s)] to 653.15 K (380 °C) (=Ms + 20 K (20 °C)) and held for 50, 1800 seconds, and 5 hours. The dilatometry curves showed that no ferrite transformation occurred and that the initial bainitic transformation was finished after 50 second (Figure 1).

The microstructures of the three samples were characterized in detail through electronic microscope images and through crystallographic orientation maps acquired with the JEOL 6500F Field Emission Gun-SEM equipped with the Oxford-instrument EBSD system (Nordlys II CCD camera and AZTec acquisition software). For the EBSD measurements, the samples were

mechanically polished through 0.25 μm diamond paste and finished with colloidal silica. EBSD data were acquired in ‘refined accuracy’ mode.^[16] This mode improves the angular resolution. Experiments confirmed that low misorientation angles as small as 0.2 deg were reliably detected. These low misorientations are especially of interest for the present study as they indicate the presence of geometrically necessary dislocations formed during the transformation. The software “Merge 2” was used to depict the advanced crystallographic features of bainite.^[7] Following EBSD studies, the samples were Picral-etched to image carbides. Additional Transmission Electron Microscopy was performed on a JEOL JEM-2100F. Finally, Vickers hardness tests were used to highlight the effect of microstructure evolution on the mechanical properties. Ten measurements were averaged to reach the reported values.

The EBSD maps in Figure 2 evidence the evolution of the ferritic matrix with holding time: the initial lath-like morphology (Figure 2(a)) became fully granular after 5 hours (Figure 2(b)). This granularization phenomenon was found to be detrimental to the steel mechanical properties, as the hardness decreased from 265 to 214 HV.

After 50 seconds of holding at 653.15 K (380 °C), the microstructure typically featured lath-like bainite. The lath size ranged from 100 to 300 nm. A high density of very low misorientations (white boundaries in Figure 2(a)) was detected inside the laths or blocks of low-misorientation laths. However, adjacent laths were most frequently separated by high-angle boundaries (red in Figure 2(a)). Most of these boundaries were misoriented by 60 deg about the $\langle 111 \rangle$ axis. This corresponds to self-accommodating pairs of variants whose formation involves opposite shears in the same habit plane,^[17] which are characteristic of lower bainite according to Zajac’s classification.

Advanced processing of the EBSD data with Merge 2 showed that the bainitic structure was (1) inherited from prior austenite grains with an average grain size of 30 μm (2) with the mean OR described by the set of Euler angles: (3.6, 46.3, 3.6 deg). This OR deviates from the Kurdjumov–Sachs OR by 1.32 deg for the plane normals and by 2.54 deg for the direction. Such deviations from the KS OR were already reported in Reference 18. Most of the laths were grouped as packets of variants sharing the same habit plane, with a packet size less than 10 μm .

After 5 hours of isothermal holding, surprisingly the lath-like bainite was fully transformed to granular bainite (Figure 2(b)). The grain size ranged between 5 and 10 μm , on the same order of magnitude as the prior lath packets. The bainite grains exhibited mostly irregular shapes, with locally wavy boundaries that could be traced to prior growing laths. Moreover, they often contained networks of low-angle boundaries (white in Figure 2(b)). Most of the bainitic grains were randomly misoriented. Even though misorientations >50 deg remained numerous (red in Figure 2(b)), the characteristic 60 deg $\langle 111 \rangle$ boundaries were no longer observed.

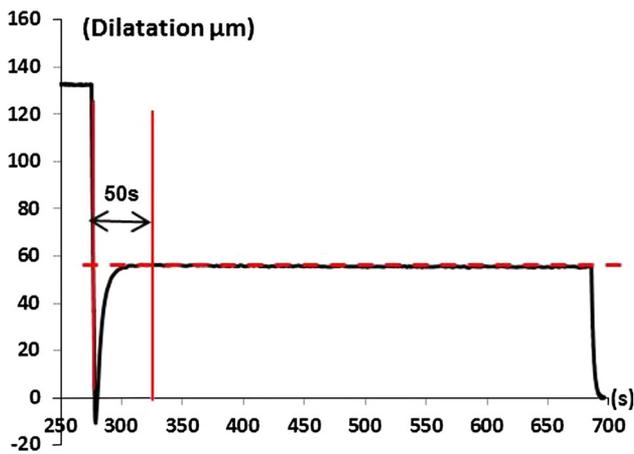


Fig. 1—Time-dependent dilatometry curve during isothermal holding at 653.15 K (380 °C) showing the end of the bainitic transformation after 50 s.

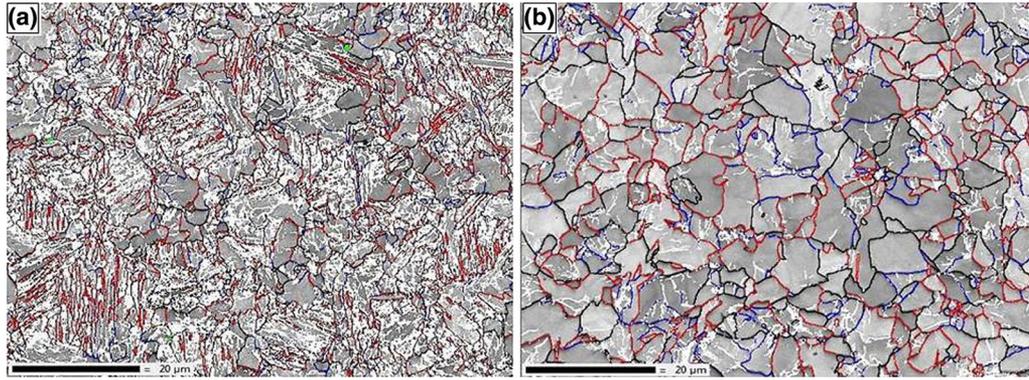


Fig. 2—Pattern quality and misorientation maps of samples annealed at 653,15 K (380 °C) for (a) 50 s (predominantly lath-like microstructure) and (b) 5 h (entirely granular microstructure). The boundaries have been highlighted as a function of their respective misorientations: 0.5< white <3, 3< blue <20, 20< black <45, and red >45 deg. For color interpretation, the reader is referred to the web version of this article.

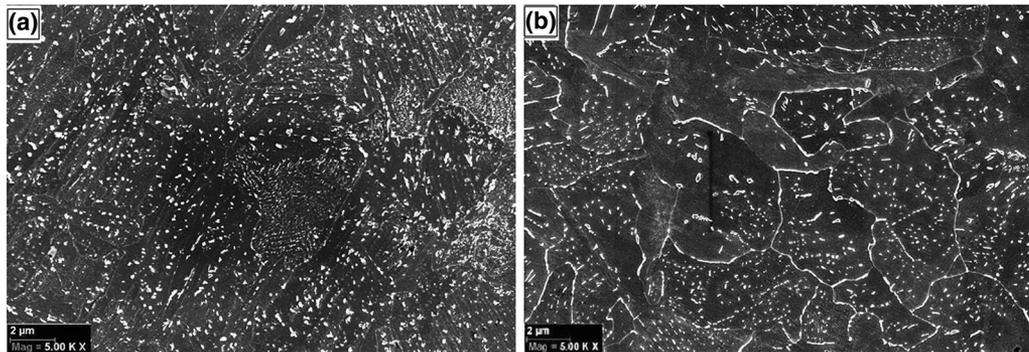


Fig. 3—Distribution of carbides in the sample treated at 653,15 K (380 °C) for 50 s (a) and 5 h (b).

Besides the ferritic matrix evolution, carbide ripening was also observed with holding time, as seen in Figure 3. After 50 seconds, while the carbides were primarily located between the laths, they also appeared inside the laths with the characteristic morphological orientation^[19] (Figure 3(a)). Thus the initial microstructure was a mixture of lower and upper bainite, according to Mehl's classification. After 5 hours, a carbide ripening process was clearly observed; the smallest carbides had dissolved and precipitated as discontinuous films at high-angle boundaries of granular bainite. Small carbide-free zones appeared next to the boundary precipitates (carbon-depleted zones). Carbides initially located between the bainitic laths remained arranged in parallel lines within the bainitic grains, as seen in Figure 2(b). This result was confirmed by TEM.

Figure 4 superimposes the three misorientation angle distributions obtained from all EBSD maps acquired on the samples treated for 50, 1800 seconds, and 5 hours at 653,15 K (380 °C). These are statistically much more representative than the maps shown in Figure 2, since they correspond to data acquired over $150 \times 120 \mu\text{m}^2$ EBSD areas (vs $80 \times 70 \mu\text{m}^2$ sized maps of Figure 2).

With increasing holding time from 50 seconds (end of the bainitic transformation) to 1800 seconds, the most important change was the decrease in high-angle boundaries between laths [mainly the 60 deg $\langle 111 \rangle$ —see Figure 2(b)]. Further analysis of the EBSD data showed that the granular structure already covered 90 pct of the

sample treated for 1800 seconds at 653,15 K (380 °C). This microstructure evolution also resulted in a drop in hardness from 265 HV after 50 seconds to 221 HV after 1800 seconds (88 pct of the total hardness decrease observed after 5 hours). The granularization process was thus surprisingly fast for the studied alloy composition. Meanwhile, only a small fraction of the low-misorientation boundaries were removed after 1800 seconds [see Figure 2(b)]. ECCI and TEM images confirmed that granular bainite contained a dense network of dislocations.

The most important change in terms of misorientation distribution from 1800 seconds to 5 hours was the reduction of very low misorientations in favor of random orientation distributions [see Figures 2(a) and (b)]. This reduction resulted from dislocation rearrangement to sub-boundaries and random boundaries in the fully granular structure.

Thanks to coupled EBSD and FEG-SEM observations, we have experimentally identified a fast granularization process of lath-like bainitic structures during an isothermal holding treatment of an Fe-0,13C-5Ni steel. Our result shows that the granularization phenomenon starts directly once the lath-like structure is formed (after 50 seconds of holding time) and decomposes into two main steps:

- (1) Block of laths, free of high misorientations, develop early within a packet of laths; the highly misoriented laths within those packets shrink, and

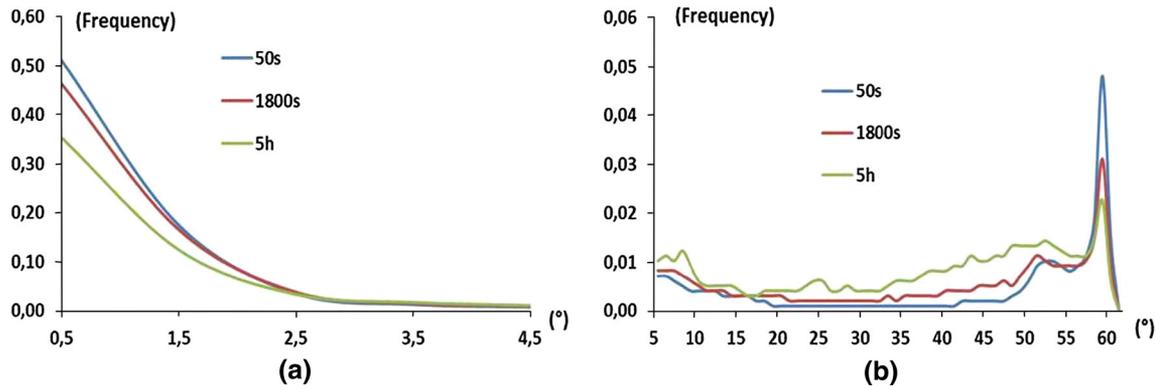


Fig. 4—Angular misorientation distribution of three samples treated for 50 s, 1800 s, and 5 h. (a) Low and (b) high misorientation frequencies of the global distributions (ranging from 0.5 to 62 deg) are separated into two figures to adjust at best the frequency axes. For color interpretation, the reader is referred to the web version of this article.

the inter-lath carbides remain arranged in parallel lines within highly dislocated bainitic grains.

- (2) Carbide ripening process gives rise to discontinuous carbide films precipitating at high-angle boundaries of granular bainite. At the same time, dislocation rearrangement leads to the development of sub-boundaries and random boundaries in the granular microstructure.

This work was supported by the French State through the program “Investment in the future” operated by the National Research Agency (ANR) and referenced by ANR-11-LABX-0008-01 (LabEx DAMAS) and was also supported by the Region Lorraine. The authors would like to thank Professor Martin Crimp for fruitful discussion.

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