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Phase quantification in low carbon Nb-Mo bearing steel by electron backscatter diffraction technique coupled with kernel average misorientation

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A R T I C L E I N F O

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A B S T R A C T

An efficient and accurate method is developed for microstructural quantification of complex phases in a low carbon Nb-Mo bearing steel, of which optical micrographs show that it consists of granular bainite and a small amount of ferrite. Our previous work, proposing a method to measure the misorientation angles via electron backscatter diffraction (EBSD) to differentiate granular bainite and ferrite, has been reported. That method is accurate for phase quantification but laborious for the characterization process. To resolve this difficulty, in this study, EBSD combined with a kernel average misorientation (KAM) map is used for phase characterization. Comparisons are made among KAM maps with different kernel sizes (300 to 600 nm) and various step sizes (100 to 600 nm). It is found that a kernel size close to the sub-structure size of granular bainite (500 nm) is optimal for phase identification, while varied step sizes produce relatively invariant results. Therefore, KAM maps can be used for fast and reliable phase quantification, provided that an appropriate kernel size and a large step size are used.

1. Introduction

Low-carbon Nb-bearing high-strength low-alloy (HSLA) steels are widely used in multiple applications [1,2], such as pipelines [3–5], automobiles [6,7] and construction [8,9]. The mechanical properties and related applications of low carbon steels are strongly influenced by their microstructures, which are generally complex and are often composed of various types of body-centered cubic (BCC) structures [4,10–13], including ferrite, Widmanstätten ferrite, bainite and martensite. Those structures have been investigated using optical microscopy (OM) as well as scanning electron microscopy (SEM) and been classified systematically [14], such as with Dubé classification [15]. However, some microstructures cannot be easily differentiated by using conventional metallography methods. For example, polygonal ferrite and granular bainite are both polygonal-shaped and almost identical under OM. Plausible estimations of their respective volume fractions are industrially important, as these two phases have drastically different mechanical properties, but such estimations are practically challenging owing to resemblances in the appearances of the two phases. The present paper attempts to solve this difficulty.

Morphologically, polygonal ferrite is characterized by mainly equiaxed grains with low dislocation densities. In contrast, granular bainite is in the form of fine platelets separated by low-angle grain boundaries, and bainitic ferrite platelets have a significantly higher dislocation density. Techniques with the capability to differentiate these two phases are transmission electron microscopy (TEM) and electron backscattered diffraction (EBSD). TEM can well resolve the substructures of bainite, but phase quantification using TEM is inappropriate due to the limited observation area. On the other hand, EBSD has recently emerged as a technique with sufficient resolution to analyze the substructures of bainite; with the advantages of easy specimen preparation and well-developed automation, a much larger scanned area and routine quantifications are possible. In EBSD [16–20], backscattered Kikuchi patterns are recorded, and the crystal lattices and orientations of individual points can be determined. Commercially-available software can be used for post-experiment analyses for boundary characteristics, grain size, grain size distribution, misorientation angles, and residual strain. However, for phase

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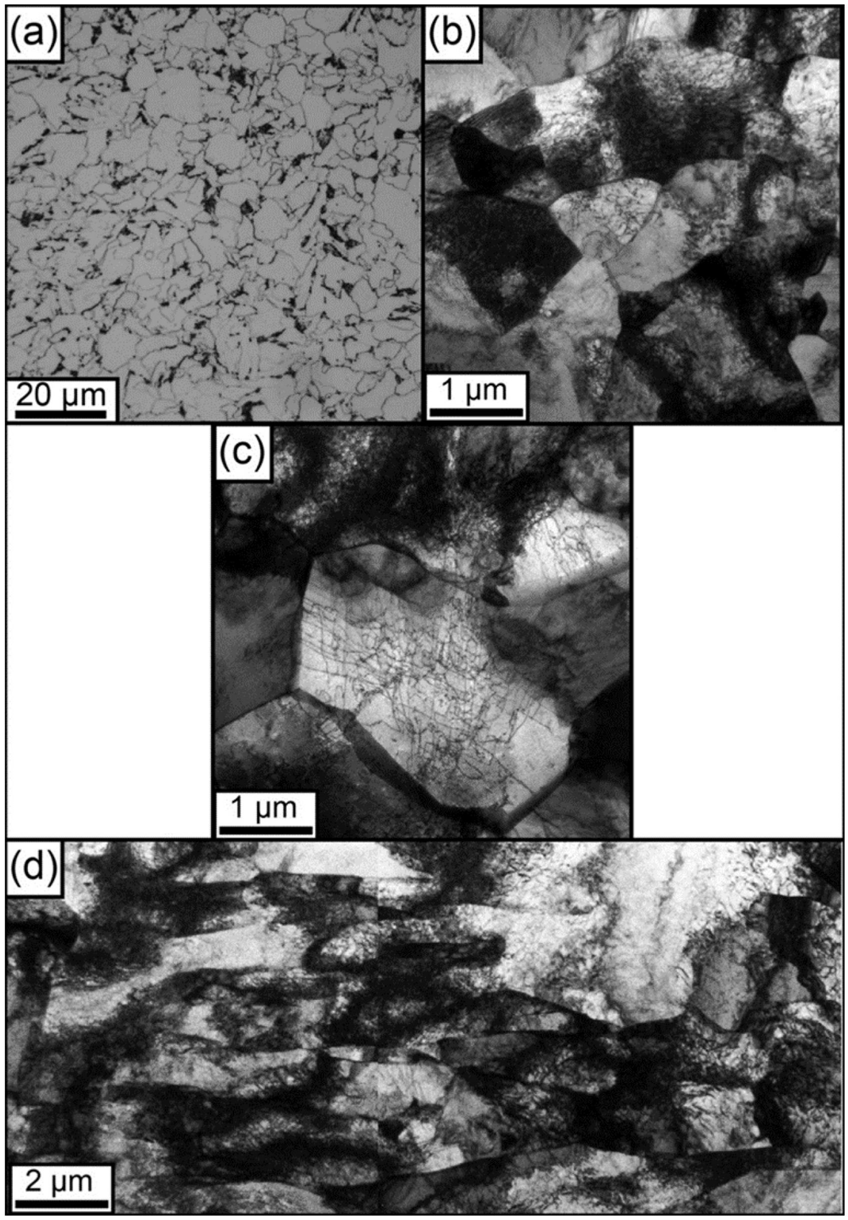

Fig. 1. (a) Optical micrograph of Exp. steel; (b) TEM image of the small grain size (~1 μm) ferrite; (c) large grain size (~4 μm) ferrite; (d) parallel lath structures inside granular bainite sheaves.

identification using EBSD, tough issues still remain for phases of the same crystallographic structure, such as those commonly encountered in steels. It is still difficult to differentiate between the aforementioned granular bainite and polygonal ferrite, or between bainite and martensite in low carbon steels. Recently, many researchers [11,12,21–23] have devoted their efforts to developing reliable methods for the identification of phases of the same crystallographic structure. For example, image quality (IQ) [24–30] is related to the degree of lattice imperfection, such as dislocation densities and sub-grain boundaries inside the grain. Wu et al. [28] found that in their IF steels, the poor IQ regions increased with the cooling rate, and the increase was attributed to a higher dislocation density. In addition, DeArdo et al. [29] quantified phases in low carbon steels using IQ spectra, where multiple peaks should occur because each phase is supposed to have a distinct IQ value, and quantifications were obtained by calculating the area of every peak. The method is called multi-peak analysis. However, bainite and martensite, both commonly found in low carbon steels, have high dislocation densities, and the IQ peaks of the two cannot be separated, limiting the use of multi-peak analysis on complex phase steels. In addition, this IQ-based method has several shortcomings. One is that the quality of diffraction patterns strongly depends on the preparation and contamination of the specimens. Another is that differences in analysis software, SEM and operator variables lead to different quantification results. An alternative method to distinguish phases in complex phase steels was developed by Shrestha et al. [23], who investigated ultra-thin cast steels with diverse microstructures, including acicular ferrite, polygonal ferrite and bainite. In their approach, morphological features such as grain boundary misorientation, grain size, aspect ratio and mean misorientation were taken into consideration, and the criteria for determining each phase were developed. For example, acicular ferrite was distinguished with its high aspect ratio. However, an efficient method for distinguishing granular bainite from ferrite has yet to be established. One feasible way to differentiate granular bainite from ferrite, recently developed by Huang et al. [11,12], is summarized as follows. The first step is the construction of the point-to-origin misorientation angle profile of every polygonal BCC