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Crystallographic analysis of lenticular martensite in Fe–1.0C–17Cr stainless steel by electron backscatter diffraction

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ABSTRACT

The crystallography of lenticular martensite, which formed in coarse austenite grains (size about 80 μm) after subzero treatment at −196 °C (liquid nitrogen) for different holding times, was investigated using electron backscatter diffraction (EBSD). For the sample treated with 15 min of isothermal holding, more than 50 martensite plates (with a thickness of larger than 1 μm) that formed within a coarse austenite grain were employed to obtain the pole figures. The pole figures clearly indicated that the individual plate of lenticular martensite approximately adopted the Kurdjumov–Sachs (K–S) orientation relationship with respect to the austenite matrix. For the sample treated with 30 s of isothermal holding, a few martensite plates that formed in variant pairings in a coarse austenite grain were analyzed. The results showed that zigzag couplings (including spear couplings), the major product of plate martensite, had an absolute dominance of a specific variant pair (V1/V17). The orientation gradient within a lenticular martensite plate was also measured using convergent beam electron diffraction (CBED). The evidence strongly suggests that the spread in diffracted intensity within pole figures is related to the misorientation gradient within the lenticular martensite plate.

1. Introduction

The morphology of ferrous martensite, which is contingent on the chemical composition and martensite start temperature (Ms), is generally of three types: lath, lenticular, and thin-plate [1–23]. Lath martensite forms in the highest temperature range in low-carbon steels (up to about 0.6C wt%) and some Fe–Ni alloys [1–5]. The high dislocation density usually observed within lath martensite results from the accommodation strains induced by transformation. Thin-plate martensite, which forms in the lowest temperature range in high alloyed steels, consists of a set of uniformly spaced transformation twins crossing throughout the plate [6,7]. Lenticular martensite forms at the intermediate temperature between lath martensite and thin-plate martensite. Under an optical microscope, typical lenticular martensite resembles a symmetrical lens divided in half by a straight-line midrib [6,7]. In fact, the substructures of lenticular martensite are more complicated than those of thin plate martensite. Transmission electron microscopy has revealed that lenticular martensite contains three regions: the midrib, the extended twinned region, and the untwinned region [6–8]. The midrib is composed of highly dense, regularly spaced transformation twins. The extended twins have a lens-like feature; they emanate laterally from the midrib region and form an approximately symmetrical pattern of parallel vertex pillars [6]. In the untwinned region, several sets of screw dislocations with a high density of tangled dislocations can be observed [8]. The transformations of both thin-plate martensite and lenticular martensite are initiated at the midrib region. However, during growth, the former retains the lattice-invariant deformation mode of twinning, while the latter has a combination of twinning and slip modes [6].

Extensive work has been done on the crystallographic and morphologic analyses of lath martensite by Maki et al. [2–4]. They proposed that the morphology of lath martensite transformed from a prior austenite grain can be regarded as a hierarchic structure: parent grain–packet–block–sub-block–individual lath. An individual lath, the basic unit for building lath martensite, is a few microns long and about 0.5 μm thick. Lath martensite generally adopts the Kurdjumov–Sachs (K–S) orientation relationship (OR) with respect to the austenite matrix, and 24 independent crystallographic variants of lath can be developed within a single crystal of austenite, as shown in Table 1. Four groups can be assigned to the four independent $\{111\}_\gamma$ planes in a given austenite crystal. In each of the four groups, six independent variants have a common parallel relationship of close-packed planes. Because martensite variants within one packet have nearly the same habit plane, it is therefore appropriate to conclude that only one of these four groups constitutes a packet structure; in other words, a packet may consist of six variants, which belong to the same group. Using the nomenclature used by Morito et al. [3], the four packets can be expressed as follows: Group 1 (V1–V6), Group 2 (V7–V12), Group 3 (V13–V18), and Group 4 (V19–V24). A packet may be subdivided into three parallel blocks, each consisting of two sub-blocks (two different variants). For example, the three blocks of the "Group 1" packet are V1–V4, V2–V5, and V3–V6,

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Table 1
24 variants of Kurdjumov–Sachs (K–S) orientation relationship.

	γ orientation	Axis angle pair (α′)[a]	Equivalent axis angle pair (α′)	PG[b]
V1	$(1\ 1\ 1)[\bar{1}\ 0\ 1]$	[1.000 0.000 0.000]/0°	–	A1
V2	$(\bar{1}\ \bar{1}\ \bar{1})[\bar{1}\ 0\ 1]$	[0.577 0.577 0.577]/180°	[0.576 0.577 0.578]/60.00°	A5
V3	$(1\ 1\ 1)[0\ 1\ \bar{1}]$	[0.000 −0.707 −0.707]/120°	[−0.001 −0.707 −0.707]/60.00°	D5
V4	$(\bar{1}\ \bar{1}\ \bar{1})[0\ 1\ \bar{1}]$	[0.966 0.065 0.065]/180°	[0.000 0.707 −0.707]/10.53°	A2
V5	$(1\ 1\ 1)[1\ \bar{1}\ 0]$	[0.000 0.707 0.707]/120°	[−0.001 0.707 0.707]/60.00°	D2
V6	$(\bar{1}\ \bar{1}\ \bar{1})[1\ \bar{1}\ 0]$	[0.418 0.642 0.642]/180°	[−0.001 −0.707 0.707]/49.47°	D1
V7	$(1\ \bar{1}\ 1)[1\ 0\ \bar{1}]$	[0.471 0.342 0.813]/180°	[0.577 0.578 −0.577]/49.47°	A6
V8	$(\bar{1}\ 1\ \bar{1})[1\ 0\ \bar{1}]$	[0.667 0.742 0.075]/180°	[−0.575 0.576 0.582]/10.53°	A3
V9	$(1\ \bar{1}\ 1)[\bar{1}\ \bar{1}\ 0]$	[0.742 0.650 0.167]/90°	[0.614 −0.182 0.767]/50.51°	D3
V10	$(\bar{1}\ 1\ \bar{1})[\bar{1}\ \bar{1}\ 0]$	[−0.087 −0.900 0.428]/120°	[0.739 0.463 −0.489]/50.51°	C2
V11	$(1\ \bar{1}\ 1)[0\ 1\ 1]$	[0.075 0.167 −0.983]/90°	[−0.933 −0.355 −0.065]/14.88°	B2
V12	$(\bar{1}\ 1\ \bar{1})[0\ 1\ 1]$	[−0.856 −0.043 0.515]/120°	[0.356 −0.603 −0.713]/57.21°	D6
V13	$(\bar{1}\ 1\ 1)[0\ \bar{1}\ 1]$	[−0.075 −0.167 0.983]/90°	[−0.355 0.933 0.065]/14.88°	B4
V14	$(1\ \bar{1}\ \bar{1})[0\ \bar{1}\ 1]$	[0.087 0.900 −0.428]/120°	[0.490 −0.463 0.739]/50.51°	C6
V15	$(\bar{1}\ 1\ 1)[\bar{1}\ 0\ \bar{1}]$	[0.667 −0.742 −0.075]/90°	[−0.739 −0.246 −0.628]/57.21°	B6
V16	$(1\ \bar{1}\ \bar{1})[\bar{1}\ 0\ \bar{1}]$	[0.742 0.650 0.167]/180°	[−0.659 0.660 0.361]/20.61°	B1
V17	$(\bar{1}\ 1\ 1)[1\ 1\ 0]$	[0.524 0.407 0.748]/180°	[0.659 0.363 −0.659]/51.73°	C1
V18	$(1\ \bar{1}\ \bar{1})[1\ 1\ 0]$	[−0.770 0.149 −0.621]/120°	[0.719 0.303 −0.626]/47.11°	C3
V19	$(1\ 1\ \bar{1})[\bar{1}\ 1\ 0]$	[−0.742 −0.650 −0.167]/90°	[0.186 −0.767 −0.615]/50.51°	C4
V20	$(\bar{1}\ \bar{1}\ 1)[\bar{1}\ 1\ 0]$	[0.856 0.043 −0.515]/120°	[−0.356 −0.713 0.604]/57.21°	D4
V21	$(1\ 1\ \bar{1})[0\ \bar{1}\ \bar{1}]$	[0.053 0.984 0.171]/180°	[−0.955 0.000 0.296]/20.61°	A4
V22	$(\bar{1}\ \bar{1}\ 1)[0\ \bar{1}\ \bar{1}]$	[0.770 −0.149 0.621]/120°	[0.302 −0.626 −0.719]/47.11°	C5
V23	$(1\ 1\ \bar{1})[1\ 0\ 1]$	[−0.667 0.742 0.075]/90°	[0.246 0.628 0.739]/57.21°	B5
V24	$(\bar{1}\ \bar{1}\ 1)[1\ 0\ 1]$	[0.075 0.167 0.983]/180°	[0.912 −0.410 0.000]/21.06°	B3

[a] Axis angle pairs (with respect to martensite plates) relating variant number V1 to other variants that may form within the same austenite crystal.
[b] Plate Group 1: (A1, B1, C1 and D1); Plate Group 2: (A2, B2, C2 and D2); Plate Group 3: (A3, B3, C3 and D3); Plate Group 4: (A4, B4, C4 and D4); Plate Group 5: (A5, B5, C5 and D5); Plate Group 6: (A6, B6, C6 and D6).

where each block is made up of two sub-blocks with a misorientation of about 10°. The sub-block contains several individual laths with a very small misorientation (1–2°). Through electron backscatter diffraction (EBSD) coupled with transmission electron microscopy, comprehensive orientation image microscopy (OIM) of lath martensite has been achieved [3,4].

Unlike the laths of lath martensite, the plates of lenticular martensite and thin-plate martensite form in isolation rather than in packets within a prior austenite grain [6,7]. Hereinafter, both lenticular martensite and thin-plate martensite are called plate martensite. The size of an individual plate is limited by the prior austenite grain size and by the preceding martensite plate, which inhibit the growth of a fresh plate [6]. When the first plate of martensite forms, it can induce a new embryo of a secondary plate for further transformation. During transformation, the austenite is not as uniformly eliminated as occurs with lath martensite, so the morphology of plate martensite is rather more complex than that of lath martensite. The resulting non-parallel variant pairings of plate martensite are presumed to be associated with an orientation relationship for mutual self-accommodation [10,11]. The related investigation of lenticular martensite or thin-plate martensite in iron alloys can be seen in the following works: Fe–1.80C (wt.%) [5], Fe–30.70Ni–0.28C (wt.%) [11], Fe–31.0Ni–0.02C (wt.%) [12], Fe–28.0Ni–0.41C (wt.%) [13], Fe–29.6Ni (wt.%) [14], Fe–33Ni (wt.%) [15], Fe–Pt [16], Fe–20.0Ni–0.73C (wt.%) [17], Fe–1.40C–12.0Cr (wt.%), Fe–1.0C–7.2Cr (wt.%) [18], 9 Cr steel (wt.%) [19], Fe–30Ni (at.%) [20], and Fe–30Ni (wt.%) [21]. In the recent EBSD works [14,19,20], the corresponding pole figures of martensite variants in a given austenite matrix did not show sharp orientations of the poles, as could be expected with well-defined variant orientations. This issue was apparently involved with plastic deformation in the austenite to accommodate the shape strain of martensite during the course of transformation. Consequently, significant misorientation gradient within the lenticular martensite plate occurred. However, no detailed investigations to elucidate the spreading orientation of poles in the pole figures have been reported yet. The present work first focused on this aspect.

The crystallography of variant pairings of lenticular martensite has been intensively studied in some previous research [10,11], from which the concept of the plate group was established. Following this concept, Stormvinter et al. [5] recently performed a related investigation on Fe–1.8C steel using EBSD. They presented a trend of plate group formation with the dominance of variant pairing in the specimen composed of high quantities of plate martensite, where hard impingement obviously occurred as the martensite platelets from different nucleation sites grew in contact with each other. It should be noted that the plates can appear to be adjacent to one another for two reasons: autocatalytic nucleation (the formation of one plate may trigger the growth of another) and hard impingement (plates which have formed at completely separate sites may come into contact as a consequence of impingement). It is fundamentally difficult to distinguish between variant pairing and hard impingement in specimens composed of high quantities of plate martensite. Therefore, in this work, care was taken to examine the cases of exact variant pairings (zigzag, spear, and kink types) in specimens with a few martensite plates forming in the austenite grains to avoid the interference of hard impingement. The orientation relationship for each variant pairing was characterized by axis–angle pair. In contrast, in the previous work [5], the variant pairing frequency data were displayed with the length fractions of inter-variant boundaries. For the present investigation, the detailed analysis of the orientation relationships of the variant pairs of lenticular martensite plates will be illustrated in the 'Results and discussion' section. The results will be compared with those of the previous work [5].

2. Experimental procedure

The as-received material was a commercially wrought AISI 440C stainless steel bar (with a diameter of 50 mm), produced by Gloria Materials Technology, Taiwan, through four-folded forging of a cast slab at 1130 °C and annealing at 870 °C, followed by furnace cooling to ambient temperature. The chemical composition of the steel was Fe–1.0C–17.4Cr–0.45Mo–0.40Mn–0.38Si (wt.%). The pieces of steel