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A serial sectioning technique for evaluating grain and twin boundary precipitate growth kinetics in bulk specimens

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Abstract

A new measurement technique for the growth kinetics of both grain boundary and twin boundary bainite precipitates in bulk specimens is demonstrated. The key advance is the use of coarse-scale serial sectioning to determine the angle made by the grain or twin boundary to the plane of polish, so an explicit stereological correction to the precipitate thickness can be made. This technique is applied to the thickening of bainite at the bay temperature of Fe–0.24C–4Mo, where grain boundary bainite growth kinetics had been measured previously in much thinner specimens using the “bamboo-specimen” method developed by Bradley et al. Several key assumptions implicit in the “bamboo-specimen” method are critically examined using the data obtained from this new technique. Twin boundary bainite growth thickening kinetics, which cannot be obtained using the bamboo method, are also obtained for the first time, and are measured in the same steel.

Keywords: Steel; Bainite; Growth kinetics; Tomography

1. Introduction

Interphase boundaries, especially grain and twin boundaries, are the dominant nucleation sites for many solid–solid phase transformations, diffusional eutectoid decomposition [1] and discontinuous (cellular) precipitation [2] in particular. The kinetics of these reactions have long been of practical interest, since they influence the hardenability of steels, and in stainless steel grades, the sensitivity to corrosion. Of all the products of these reactions that nucleate at grain and twin boundaries, allotriomorphs have traditionally been considered to have the simplest growth process to model. This is because of their single-phase nature[1] (unlike the 2-phase pearlitic and cellular products) and *apparent* lack of crystallographic or structural impediments to growth (unlike Widmanstatten rods, laths or plates). Grain boundary allotriomorphs (GBAs) are precipitates which “grow preferentially, more or less smoothly, along these boundaries” [1,4]; twin boundary allotriomorphs (TBAs) are similar. The idealized

[1] This condition does not hold in steels containing strong carbide-forming alloying elements, such as V, Cr or Mo, where the allotriomorphs contain both ferrite and alloy carbides [3]. However, the allotriomorph morphology does not appear to be significantly altered by the presence of a second phase, in this case, carbides.

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allotriomorph is lens-shaped and grows in a shape-preserving fashion (before impingement), which suggests that atomic attachment occurs at all portions of the (smooth and ideally unfaceted) growth interface with the parent phase. For these reasons, allotriomorphic growth has long been considered an ideal test case for theories of diffusion-controlled precipitate growth.

As separately-nucleated allotriomorphs rapidly impinge as they lengthen along the grain boundary at all but the shortest reaction times and smallest undercoolings, the focus of growth kinetics studies has been on the thickening process normal to the grain boundary. The first carefully controlled measurements of GBA thickening were carried out in high-purity Fe–C alloys [5] using thermionic electron microscopy to make real-time measurements on selected ferrite grain boundary allotriomorphs, as viewed on the surface of bulk specimens. The usefulness of this technique was limited by the considerable scatter in the data resulting from stereological and other errors. A new thickening (and lengthening) measurement technique was applied in a later study by Bradley et al. [6,7], also on high-purity Fe–C alloys. Specimens were austenitized, isothermally reacted for various times and quenched, and the largest GBA measured for each time. The key element of this technique was that the specimens were thinner than the average austenite grain diameter, which allowed the austenite grain boundaries to migrate during austenitization to orientations normal to the specimen surface (forming what's referred to as the "bamboo" structure[2]). Such a grain boundary structure eliminated the stereological problem of determining the angle the grain boundary made to the plane of the polish, so that the thickness measurement of an allotriomorph on the plane of polish gave the true thickness.

This "bamboo-specimen" technique was subsequently applied to determine GBA thickening kinetics in ternary Fe–C–M steels [8–10], where the disagreements between theory and experiment became quite pronounced, especially in Fe–C–Mo alloys near the bay region on their TTT diagrams [10]. Such disagreements have prompted a critical re-examination of the assumptions that underpin this experimental method of allotriomorph thickening kinetics determination.

Assuming first that the ideal "bamboo" grain structure is attained during austenitization, the GBA thickening kinetics data measured from the technique of Bradley et al. [6,7] give a physically meaningful measure of a fundamental growth process only if several key assumptions are satisfied[3]:

(1) The thickest allotriomorph is the first to nucleate, and therefore has the longest time in which to grow.
(2) The thickest allotriomorph (and thus by (1), the first-nucleated one) has the same growth path envelope [12] as all allotriomorphs which nucleated later (i.e., the growth behavior of the thickest allotriomorph is not in some way abnormal.) This is prior to any diffusion field overlap across grains.
(3) Allotriomorphs which nucleate on "bamboo"-structured grain boundaries follow the same growth path envelope as allotriomorphs in bulk specimens.
(4) For cases where the allotriomorphs have impinged to form an allotriomorphic slab, the slab advances at the same rate as an individual allotriomorphic subunit.

To date, only (4) has been tested, and was found to not hold for allotriomorphic bainite formed at the bay temperature in Fe–0.24C–4Mo [13] on account of crystallographic and interfacial structure considerations, which in the past have been ignored or oversimplified. However, assumptions (2) and (3) can still be tested in this alloy independently of assumption (4). Such a test is one objective of this work. (Assumption (1) appears difficult to test in bulk specimens, and will not be considered further.)

The "bamboo specimen" method assumes that the grain boundary at which the precipitate nucleates will migrate during austenitization so as to orient itself perpendicular to the two flat specimen surfaces. Although grain boundaries appear to satisfy this condition [5], it will not hold for twin boundaries, because the special crystallographic relationship that holds between twins in austenite. In particular, twin boundaries are very strongly faceted along {111} in fcc austenites, to such an extent that their freedom of migration to an arbitrary orientation (away from {111}) perpendicular to the specimen faces during austenitization is severely restricted. Because of such constraints, the bamboo specimen method is inappropriate for the determination of twin boundary precipitate growth kinetics. Indeed, no twin boundary precipitate growth kinetics data have been reported in the literature, either by this or other techniques. The development of an alternative technique that can measure

[2] The bamboo structure eliminates the error due to grain boundaries angled with respect to the plane of polish, but does not reduce the error associated with sectioning the allotriomorph other than at its midpoint. This is circumvented by assuming if the single thickest, well formed region is selected it is probably properly sectioned [6].

[3] Assumptions (1) and (2) are general to any technique whose measurements are done solely on the largest precipitate, for example, the maximum nodule radius technique for pearlite [11].