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EBSD characterization of the eutectic microstructure in hypoeutectic Fe-C and Fe-C-Si alloys

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Hypoeutectic Fe-C and Fe-C-Si model alloys were produced at different solidification conditions. Copper mold casting yields low cooling rates promoting the formation of a eutectic microstructure, which is characterized by two morphologies: elongated cementite plates and a rod structure growing perpendicular to the plates, i.e. austenite rods in a cementite matrix. Electron beam surface remelting generates a mainly plate-like eutectic due to rapid solidification. The microstructures were characterized by light-optical microscopy and electron backscatter diffraction (EBSD). The latter allows for a spatially resolved investigation of the growth crystallography of the eutectic phases. Thereby, a possible existence of crystallographic orientations relationships between cementite and austenite within the plate-like eutectic was assessed experimentally. The eutectic phases were found to grow largely crystallographically independently. Moreover, ferrite and eutectic cementite within the decomposed eutectic microstructure frequently comply with the Bagaryatsky or the Pitsch-Petch orientation relationship. Complementary X-ray diffraction (XRD) analysis reveals a pronounced cementite {002} texture in the microstructure produced by mold casting. Characteristic changes in the lattice parameters indicate that as-cast cementite is non-stoichiometric.

1. Introduction

The solidification characteristics and the microstructure of the metastable cementite/austenite eutectic in low-alloy white cast iron – typically referred to as *ledeburite* – have been investigated extensively at different solidification conditions during the last decades. Conventional casting methods [1–9], unidirectional solidification [10–12] and rapid solidification processes [13–21] have been employed. A very comprehensive study on the growth of ledeburite was published by Hillert and Steinhäuser [1] using light-optical microscopy with polarized light complemented by X-ray diffraction (XRD). They suggest the following model for the microstructural development of the white eutectic. The formation of ledeburite starts with the growth of faceted cementite (θ) plates spreading into the melt (*edgewise growth*; see Fig. 1a). The growth of the plates is fastest in $[001]_\theta$ and slowest in $[010]_\theta$.[1] The second phase to form in the ledeburite is austenite (γ) nucleating on cementite. Austenite grows along the $(010)_\theta$ facets as two-dimensional dendrites. As solidification proceeds, cementite penetrates through this γ dendrite and a rod-like structure, i.e. γ rods in a θ matrix, is established perpendicular to the main growth direction. The rod-like growth occurring into $[010]_\theta$ direction is also referred to as sidewise growth (see Fig. 1a). Park et al. [10] have shown by directional solidification experiments that the rods form at the eutectic cell walls when a planar growth front is unstable. New plates might develop in $[001]_\theta$ from the rod structure which is, again, denoted edgewise growth. The competition of cooperative sidewise and non-cooperative edgewise growth may result in several steps visible in the ledeburitic microstructure (Fig. 1b).

Rickard and Hughes [3] have described a *plate-like* eutectic in white cast iron which is formed at larger undercooling. Its microstructure does not show rod-like features (Fig. 1c) as opposed to the ledeburite eutectic investigated in [1]. Moreover, Song et al. [8,9] have coined the term *network-like* cementite for microstructures in which eutectic cementite seems to be continuous, i.e. individual θ plates appear interconnected and are hardly distinguishable (Fig. 1d). In order to avoid confusion about the nomenclature of θ/γ eutectic morphologies in the course of the present work, eutectic cementite with a plate-like or lamellar appearance in metallographic cross-sections will be referred to as *plate-like* (*edgewise* growth in $[001]_\theta$ dominating) independent of solidification conditions and the degree of interconnection between plates. Microstructural features with honeycomb-like appearance, i.e. austenite rods in a cementite matrix, will be denoted *rod-like* (*sidewise* growth in $[010]_\theta$).

Considering the apparent variety of the θ/γ eutectic morphologies, the question arises whether a crystallographic orientation relationship

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[1] Indices and directions refer to the *Pnma* space group setting for cementite in the present work where it holds $c < a < b$ for the lattice parameters. Note that many older works including Ref. [1] used the *Pbnm* setting with $a < b < c$, leading to a corresponding permutation of the indices.

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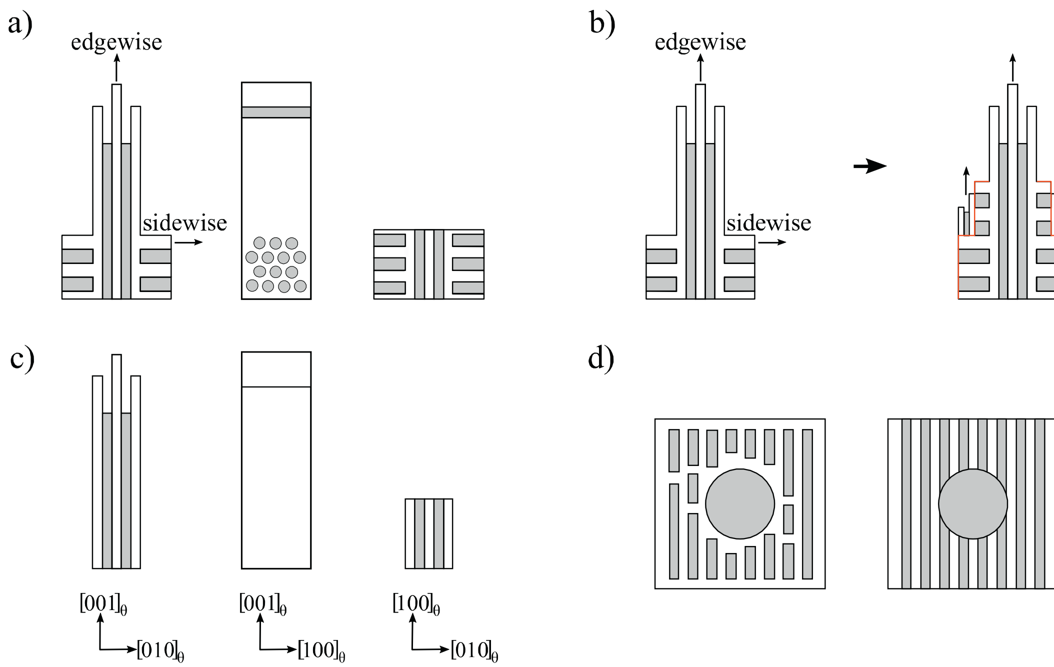

Fig. 1. Schematic sketch of the formation of the cementite/austenite eutectic based on drawings presented in [1–3] (cementite θ: white; austenite γ: grey). (a) Ledeburite with cementite plates growing edgewise in $[001]_\theta$ and austenite rods growing sidewise perpendicular to $(010)_\theta$. (b) Formation of steps due to competition of edgewise and sidewise growth. (c) Plate-like θ/γ eutectic resulting from (virtually) absent rod-like growth. (d) Eutectic microstructure around dendrite arm with continuous (network-like) appearance of cementite (left) and discontinuous cementite (right).

Table 1
Crystallographic orientation relationships reported for Widmanstätten and grain boundary cementite precipitates in austenite. Experimental deviations from the ideal ORs are indicated. Pitsch and Thompson-Howell OR are expressed conventionally (con) and in terms of close-packed planes (ccp).

Pitsch (< 5°) (con) [22]	Pitsch (ccp) [27]	Thompson-Howell (< 2°) (con) [23]	Thompson-Howell (ccp) [27]	Farooque-Edmonds (< 3°) [24]	Zhang-Kelly (< 1°) [26]	Zhou-Shiflet (< 5°) [25]
$(100)_\theta \| (110)_\gamma$	$[100]_\theta \| [10\bar{1}]_\gamma$	$[100]_\theta \| [10\bar{1}]_\gamma$	$[100]_\theta \| [10\bar{1}]_\gamma$	$(100)_\theta \| (0\bar{2}1)_\gamma$	$(\bar{2}20)_\theta \| (\bar{1}\bar{1}1)_\gamma$	$[100]_\theta \| [031]_\gamma$
$[010]_\theta \| [\bar{2}25]_\gamma$	$(03\bar{1})_\theta \| (\bar{1}1\bar{1})_\gamma$	$[010]_\theta \| [\bar{4}1\bar{4}]_\gamma$	$(031)_\theta \| (\bar{1}1\bar{1})_\gamma$	$(010)_\theta \| (\bar{5}12)_\gamma$	$[110]_\theta \| [\bar{1}10]_\gamma$	$[010]_\theta \| [51\bar{3}]_\gamma$
$(001)_\theta \| (5\bar{5}4)_\gamma$	$[0\bar{1}\bar{3}]_\theta \| [\bar{1}\bar{2}\bar{1}]_\gamma$	$[001]_\theta \| [181]_\gamma$	$[01\bar{3}]_\theta \| [\bar{1}\bar{2}\bar{1}]_\gamma$	$(001)_\theta \| (112)_\gamma$	$[001]_\theta \| [112]_\gamma$	$[001]_\theta \| [2\bar{1}3]_\gamma$

(OR) between cementite and austenite during eutectic growth exists. Is a specific low-energy interphase boundary established or do the (largely uncooperatively growing) phases rather grow crystallographically independently? No crystallographic orientation relationship has been reported for the as-solidified θ/γ eutectic but $(041)_\theta \| (101)_\gamma / [100]_\theta \| [\bar{3}10]_\gamma$ [1], which is presumed to be valid solely during the stage of nucleation. However, $(041)_\theta$ is perpendicular to $[100]_\theta$ whereas $(101)_\gamma$ is not perpendicular to $[\bar{3}10]_\gamma$. Thus, the OR cannot be fulfilled. On the other hand, several crystallographic relationships are known for cementite precipitates in austenite, i.e. Pitsch [22], Thompson-Howell (TH) [23], Farooque-Edmond (FE) [24], Zhou-Shiflet (ZS) [25] and Zhang-Kelly (ZK) [26]. These are listed in Table 1.

The direct investigation of the θ/γ eutectic in low-alloy white cast iron is complicated due to the limited existence range of austenite. Primary and eutectic austenite are not retained but decomposed, e.g. to cementite and ferrite (α), if the cooling rate after solidification is not sufficiently high. It might be expected that the crystallographic nature of cementite and ferrite formed upon austenite decomposition is related to the crystallography of the eutectic cementite. Thus, orientation relationships between ferrite and eutectic cementite could exist, i.e. Bagaryatsky [28], Isaichev [29] and Pitsch-Petch [30,31]. These are given in Table 2 following the nomenclature of [32]. Indeed, the occurrence of Bagaryatsky OR in ledeburitic microstructures has been reported [33].

The aim of the present work is to elucidate the crystallographic nature of the θ/γ eutectic paying tribute to the early work of Hillert and Steinhäuser [1]. In doing so, hypoeutectic Fe-C and Fe-C-Si model alloys were produced by two casting procedures associated with different solidification rates. These are conventional copper mold casting and electron beam surface remelting. The latter yields cooling rates up to $10^4\,Ks^{-1}$ [34]. All of the θ/γ eutectic morphologies previously discussed can be produced that way. The microstructures were analyzed by electron backscatter diffraction (EBSD) with respect to phase constitution and crystallography. Knowledge of the crystallographic orientation of adjacent microstructural features allows determining misorientations and testing the validity of orientation relationships. Although low-alloy white cast iron was already subjected to EBSD analysis [8,9,35], no detailed crystallographic investigation on the microstructure was carried out. In particular, no EBSD measurements of as-solidified eutectic cementite in contact with eutectic austenite have been published for low-alloy cast iron yet to the knowledge of the present authors. Besides, EBSD data are available for austenite and carbides in as-cast high-chromium cast iron [36–38].

2. Experimental

Fe-C and Fe-C-Si model alloys were produced from pure Fe granules (99.98%), Si lumps (99.9999%) and graphite rods (99.9995%) supplied by *Alpha Aesar*®. Fe-C samples containing 3.5 wt% carbon are denoted Fe-3.5C. Fe-C-Si samples containing 3.5 wt% carbon and 1.5 wt% silicon are referred to Fe-3.5C-1.5Si. The raw material was induction-melted in alumina crucibles in argon atmosphere and cast into copper molds to manufacture plates sized 80 mm × 80 mm × 5 mm. Material