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Kinetics of anomalous multi-step formation of lath martensite in steel

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Abstract

A steel containing 16 wt.% Cr, 5 wt.% Ni and 3 wt.% Cu was transformed into martensite by applying isochronal, i.e. constant rate, cooling followed by isothermal holding. The formation of martensite was monitored with dilatometry. A series of retardations and accelerations of the transformation was observed during isochronal cooling for cooling rates ranging from 1.5 to 50 K min⁻¹. The cooling rate in the isochronal stage was observed to influence the transformation rate in the isothermal stage. Electron backscatter diffraction was applied to determine the morphology of the martensite, which was of lath type, and to investigate the microstructure of the material. No influence of the cooling rate on the scale of the microstructure was observed. The series of retardations and accelerations of the transformation is interpreted in terms of the combined effect of the strain and interfacial energy introduced in the system during martensite formation, which stabilizes austenite, and autocatalytic nucleation of martensite. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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1. Introduction

Martensite in iron-based alloys can have lath or plate morphology $[1-8]$. Lath martensite shows an internal sub-structure with a high density of dislocations $[2,3]$, while plate martensite is partially $[4-7]$ or fully $[8]$ internally twinned.

The microstructure of lath martensite is highly hierarchical. Austenite grains are sub-divided by martensite formation at four length scales [\[2,3,9\]](#page--1-0). Firstly, austenite grains are divided into groups of laths with the same habit plane, so-called packets. Each packet contains several blocks, which are groups of laths with one or two coupled (see Refs. $[1-10]$) variants of the orientation relationship with the parent austenite, defined as sub-blocks. Block boundaries are high-angle boundaries, while sub-block boundaries are low-angle boundaries. The individual laths

represent the smallest level of sub-division. The formation of lath martensite is thermally activated (i.e. timedependent) [\[10\].](#page--1-0)

The microstructure of plate martensite, which includes martensites of lenticular shape, so-called lenticular martensites [\[1,10\]](#page--1-0), does not present a hierarchical structure $[1,10,11]$, but the formation of the martensite plates progressively partitions the prior austenite grains [\[11\].](#page--1-0) Lenticular martensite is composed of an internally twinned central midrib surrounded by a region with a high density of dislocations [\[5–7\]](#page--1-0) (i.e. slipped martensite). The slipped regions grow by thermal activation $[10,12]$, while the for-mation of midribs proceeds athermally [\[10\]](#page--1-0).

Time-dependent formation of lath $[13-16]$ and lenticular [\[17\]](#page--1-0) martensites may yield a microstructure with a scale dependent on the thermal cycle applied during martensite formation, i.e. cooling rate $[13-15,17]$ and with the pres-ence of isothermal steps [\[16\].](#page--1-0)

Regardless of the morphology of martensite, martensite formation in iron-based alloys is characterized by strong

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autocatalytic nucleation, which is promoted by the elastic and plastic strains in the surrounding austenite as induced by the forming martensite [\[18,19\]](#page--1-0).

Autocatalytic nucleation of lath martensite may take place at all different scales of the microstructure [\[20–23\].](#page--1-0) Autocatalytic nucleation was demonstrated within singular blocks and packets [\[20\]](#page--1-0), inter-packets [\[21,22\]](#page--1-0) and in neighbouring austenite grains [\[22,23\]](#page--1-0). Similarly, autocatalytic nucleation of plate martensite may occur within the same prior austenite grain [\[28\]](#page--1-0), or may spread the transformation in neighbouring austenite grains [\[24–27\]](#page--1-0).

As a consequence of the fact that nucleation of martensite occurs spontaneously in a minor number of prior austenite grains, the kinetics of martensite spread largely contributes to the overall kinetics of the transformation [\[24–27\].](#page--1-0) For example, autocatalytic nucleation of plate martensite may result in burst kinetics [\[11,29\]](#page--1-0). In a burst, a large portion of material is transformed instantaneously in a single event $[29]$, yielding sudden $[11,30]$ and irreproducible [\[11\]](#page--1-0) transformation steps.

In Refs. [\[11,30\],](#page--1-0) anomalous observations were reported where the steps appear smoothed into a train of retardations and accelerations of the transformation, such that the kinetics of the transformation was characterized by a series of transformation rate maxima. Specifically, multiple retardations and accelerations of the transformation during continuous cooling have been reported several times for the transformation of face-centred-cubic (fcc) austenite into body-centred-cubic (bcc) microstructure constituents in steels, as ferrite $\lceil 31-34 \rceil$, bainite $\lceil 35 \rceil$ and martensite with lath $[36-38]$ and lenticular morphology $[11,30,39-41]$, respectively.

Transformation kinetics characterized by a series of transformation rate maxima was firstly reported in Ref. [\[11\],](#page--1-0) where time-dependent wavy transformation curves were observed during the formation of lenticular and butterfly [\[1\]](#page--1-0) martensites. Ref. [\[11\]](#page--1-0) considers several Fe–Ni–C, Fe–Ni–C–Mn and Fe–Ni–C–Cr steels. A series of retardations and accelerations of the transformation was observed after numerous instantaneous transformation steps, i.e. bursts. This behaviour was typified as unusual for martensite formation and remained not clarified.

Later, a similar behaviour, now typified as irregular [\[30\],](#page--1-0) was reported in Refs. [\[30,39\]](#page--1-0), where a controlled timedependent multi-step transformation of austenite into lenticular martensite was reported after a single large transformation burst in Fe–Ni–C systems. The mechanism responsible for irregular behaviour was not described in Refs. [\[30,39\].](#page--1-0)

In Refs. [\[40,41\],](#page--1-0) two transformation rate maxima on isochronal cooling were reported during the formation of lenticular martensite in a Fe–Cr–C steel. In Ref. [\[40\],](#page--1-0) the rate maxima were observed to become more pronounced the faster the cooling, up to 5 K min⁻¹, while upon cooling at 15 K min⁻¹ a single rate maximum was reported. The mechanism responsible for multiple transformation rate maxima was not addressed in Refs. [\[40,41\].](#page--1-0)

The observation of a controlled multi-step transformation of austenite into lath martensite was first described in Ref. [\[35\],](#page--1-0) where wavy transformation curves were reported for the first time from the onset of the transformation of fcc austenite into a bcc product in a Fe–Ni alloy. The transformation product reported in Ref. [\[35\]](#page--1-0) was later interpreted as bainite rather than martensite [\[42\].](#page--1-0) In Ref. [\[35\]](#page--1-0) a single transformation rate maximum was reported on cooling at 9 K min^{-1} , while a series of rate maxima was observed on slower cooling at 1 K min^{-1} . The temperature interval for the transformation was shown to be largely independent of the cooling rate. Although it was recognized that wavy transformation curves result from the combined effect of an athermal mechanism and a thermally activated mechanism $[35]$, the origin of such mechanisms remained unrecognized.

The suggestion of a combined effect of an athermal mechanism and a thermally activated mechanism was implemented in Ref. [\[31\]](#page--1-0). In Ref. [\[31\]](#page--1-0) a multi-step massive transformation of virginal austenite into ferrite during isochronal cooling was presented for the first time and the kinetics of the transformation was typified as abnormal. The transformation in Ref. [\[31\]](#page--1-0) was shifted to lower temperature for faster cooling and presented more pronounced transformation rate maxima for faster cooling. Ref. [\[31\]](#page--1-0) considers the Fe–Mn and Fe–Co alloys. Following Ref. [\[31\],](#page--1-0) firstly, ferrite nucleates at several locations in the material and starts to grow. The strain energy which is locally introduced in the system, because of the volume misfit between ferrite and austenite, promotes autocatalytic nucleation of ferrite in front of the austenite–ferrite interface, yielding an acceleration of the transformation. Thereafter, the sudden formation of a large fraction of ferrite introduces a relevant amount of strain and interfacial energy in the system, which counteracts the chemical driving force. The transformation stops when the overall strain and interfacial energy equals the chemical driving force for the transformation. This condition is satisfied at a lower temperature, i.e. at a larger value of chemical driving force, for faster cooling, yielding more pronounced transformation rate maxima for faster cooling. A new acceleration of the transformation is obtained for an additional increase of the driving force upon further undercooling. Evidently, Ref. [\[31\]](#page--1-0) indicated the transformation itself as thermally activated, whereas the athermal character arises from the chemical driving force for the transformation, which depends on temperature only.

Multi-step lath martensite formation kinetics on isochronal cooling was first demonstrated in Refs. [\[36,37\].](#page--1-0) In Ref. [\[38\],](#page--1-0) multi-step transformation of austenite into lath martensite on isochronal cooling remained unrecognized.

In Ref. [\[36\],](#page--1-0) where multi-step transformation kinetics was typified as unusual, a train of transformation rate maxima was reported for the transformation of virginal austenite on isochronal cooling a Fe–Ni–Co–Mo steel. The applied cooling rate ranged from 0.2 to 2.5 K min⁻¹. The transformation rate maxima in Ref. [\[36\]](#page--1-0) were sharper

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