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Unusual martensite-formation kinetics in steels: Observation of discontinuous transformation rates

Sarah Loewy^{a,b,*}, Bastian Rheingans^b, Sai Ramudu Meka^a, Eric J. Mittemeijer^{a,b}

^a Max Planck Institute for Intelligent Systems (formerly Max Planck Institute for Metals Research), Heisenbergstraße 3, D-70569 Stuttgart, Germany ^b Institute for Materials Science, University of Stuttgart, Heisenbergstraße 3, D-70569 Stuttgart, Germany

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Abstract

In order to study the role of the potentially thermal activation of martensite formation in systems which are classically considered as "athermal", the austenite \rightarrow martensite transformation kinetics of a maraging steel (FeNiCoMo), forming lath martensite, was investigated by dilatometry and differential thermal analysis (DTA), supplemented by electron backscatter diffraction, X-ray diffraction and transmission electron microscopy analysis of the microstructure. No dependence of the transformation rate on cooling rate could be observed and thus any possible activation energy has to be very small. Dilatometry as well as DTA measurements revealed an unusual transformation behavior during the martensite formation: a train of transformation-rate maxima occurs, which was found to be reproducible for different specimens, heating rates and specimen geometries. This phenomenon was attributed to the formation of the highly hierarchical microstructure of blocks of martensite laths, grouped in packages of parallel blocks: the observed train of transformation-rate maxima is caused by the simultaneous, concerted formation of blocks in different packages in all grains. The simultaneity results from the defined formation conditions for a block as established by the formation of the preceding adjacent block within the package. © 2013 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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1. Introduction

Martensite formation occurs by a diffusionless, cooperative movement of atoms transferring the parent (austenite) crystal structure into the product (martensite) crystal structure without change of composition. The transformation sets in at a certain degree of undercooling at the characteristic martensite start temperature M_s . Due to the development of interfaces and plastic as well as elastic deformation associated with the formation of martensite, M_s can be considerably lower than the temperature T_0 , the temperature at which the chemical Gibbs energy difference of the austenite phase and the martensite phase is zero. Frequently, relatively high cooling rates are required to suppress competitive diffusional reactions (e.g. a massive transformation or a bainitic transformation¹) that can occur at temperatures between T_0 and M_s [1]. One exception is the class of so-called maraging steels, iron-based alloys which contain nearly no carbon, but a high content of nickel (or chromium) as the main substitutional alloying element as well as other alloying elements, such as cobalt and molybdenum: for these alloys the martensitic transformation can be induced upon imposing (very) modest cooling rates.² Age-hardening subsequent to martensite

^{*} Corresponding author at: Max Planck Institute for Intelligent Systems, Heisenbergstraße 3, D-70569 Stuttgart, Germany.

E-mail address: s.loewy@is.mpg.de (S. Loewy).

¹ Several different definitions of the characteristics of a bainitic transformation exist (cf. e.g. Refs. [25–27]), in some cases leading to ambiguous differentiation between martensite and bainite in ultra-low-carbon steels.

² Critical cooling rates of less than 1 K s⁻¹ as compared to cooling rates of more than 10^4 K s⁻¹ for classical martensitic steels [28].

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formation (hence "maraging") leads to the precipitation of coherent intermetallic compounds in the martensitic matrix, causing a strong increase in hardness with an excellent combination of strength and toughness [2].

In iron-based alloys, the different morphologies of martensite can be divided in two major types: plate martensite and lath martensite. Plate martensite grows in higher alloyed steels as lenticular-shaped plates whereas ironbased alloys with a carbon content up to 0.3 wt.% and/or nickel up to 28 wt.% typically show a lath-type morphology. Upon the formation of lath martensite, an initial austenite grain is subdivided into several packages of parallel blocks, each block consisting of sub-blocks of multiple martensite laths [3]. The orientation relations between the different hierarchical elements of the lath martensite microstructure are highly correlated and can be indicated in good approximation by the Kurdjumov-Sachs (K-S) orientation relation between austenite (γ) and martensite (α') (K–S is not perfectly realized) [4]: the common habit plane in one package is of $\{111\}_{\alpha'} || \{011\}_{\alpha'}$ -type, each package showing all six possible variants of the K-S OR for only one given variant of the habit plane. One block within one package consists of two types of sub-blocks showing two of the above indicated six variants of the K-S OR. A sub-block consists of multiple laths showing only one variant of the above indicated two variants of the K-S OR, with only slight mutual misorientation [4,5].

Martensitic transformations are often categorized as (i) athermal, meaning that the (extent of the) transformation is only dependent on the degree of undercooling, i.e. on the (chemical) driving force available, and (ii) isothermal,³ implying that an activation energy must be overcome for the transformation to proceed and that this process leads to a (discernible) time dependency at constant temperature [5]. For various systems, a clear distinction between the two modes appears difficult and there have been suggestions (see e.g. Ref. [6]) that no principal difference of the two categories of martensite exists apart from a much lower activation energy for transformation in the case of athermal martensite.

Within the framework of a project focusing on the role of (potential) thermal activation of martensite formation in systems hitherto characterized as "athermal" (e.g. Fe–Al alloys [7]), the austenite \rightarrow martensite transformation kinetics of an Fe–18.5 wt.% Ni–9 wt.% Co–5 wt.% Mo alloy, forming lath martensite, was investigated. This alloy is similar to commercially used maraging steels [8], but, in order to focus on the martensitic transformation, excludes secondary alloying elements such as Ti and Al with a stronger tendency towards the formation of intermetallic phases. The transformation behavior was studied by high-resolution dilatometry, applying various different cooling rates. The kinetic analysis was supplemented with

differential thermal analysis (DTA). Electron backscatter diffraction (EBSD), X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used for detailed characterization of the microstructure. In this system, an unexpected, unusual austenite \rightarrow (lath) martensite transformation behavior, i.e. a series of distinct maxima in the transformation rate, was observed. A possible transformation mechanism is proposed.

2. Experimental

An FeNiCoMo alloy rod was prepared by induction melting from elemental Fe (4N), Ni (3N6), Co (3N) and Mo (3N). After a homogenization treatment under argon atmosphere at 1327 K for 100 h, hammering and a second homogenization treatment at 1327 K for 20 h, cylindrical specimens of 10 mm in length and 5 mm in diameter were machined for dilatometry. For DTA, disk-shaped specimens of the same diameter with a height of \sim 1.5 mm were prepared. The alloy composition determined after the homogenization treatment is given in Table 1. The carbon content is negligibly low.

Dilatometric measurements were performed on a Bähr DIL 802 high-resolution dilatometer with a length-change resolution of $\sim \pm 10$ nm [7] under constant argon flow, using a quartz measurement system with a polycrystalline Al₂O₃ specimen as reference. The temperature scale was calibrated for each cooling rate as described in Ref. [9] by measuring the Curie temperature of pure Ni, which is close to the martensite start temperature. Starting from room temperature, the specimens were heated up to 1323 K at 15 K min⁻¹ and annealed for 1 h in order to ensure complete homogenization (dissolution of potential intermetallic compounds) and transition into the austenite phase, and to remove any (deformation) effects from specimen manufacturing. Then, the specimens were cooled down with always the same cooling rate of 5 K min⁻¹ from 1323 to 600 K, i.e. a little above M_s , in order to avoid effects of different thermal histories before the onset of the martensitic transformation, which could lead to e.g. disparate grain growth. Subsequently, the specimens were subjected to cooling rates of 2.5, 1.5, 1, 0.5 and 0.2 K min^{-1} .

In order to investigate the influence of specimen geometry on the transformation behavior, a hollow cylindrical specimen with a wall thickness of 0.4 mm was investigated using a cooling rate of 2.5 K min⁻¹. To study the influence of varying initial austenite grain size, one specimen was subjected to a prolonged annealing at 1323 K for 60 h, followed by cooling down with a cooling rate of 2.5 K min⁻¹.

Supplementary DTA measurements were performed with a cooling rate of 0.2 K min^{-1} on a Netzsch DSC 404C with a heat-flux-DSC measuring head with Pt pans diffusion-bonded on the specimen stage [10], employing the same temperature program as for the dilatometric measurements. For calibration and thermal desmearing of the

³ This martensite is denoted isothermal martensite because its progress of formation can be observed (also) upon isothermal annealing.

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