Available online at www.sciencedirect.com

ScienceDirect

Acta Materialia 59 (2011) 6751–6760

Acta MATERIALIA

www.elsevier.com/locate/actamat

Application of cyclic partial phase transformations for identifying kinetic transitions during solid-state phase transformations: Experiments and modeling

Hao Chen [a,*], Benoît Appolaire [b], Sybrand van der Zwaag [a]

[a] *Faculty of Aerospace Engineering, Delft University of Technology, Kluyverweg 1, 2629 HS Delft, The Netherlands*
[b] *LEM, CNRS/ONERA, 29 Av. Division Leclerc, BP 72, F-92322 Châtillon Cedex, France*

Received 10 January 2011; received in revised form 14 July 2011; accepted 14 July 2011
Available online 5 August 2011

Abstract

A series of cyclic partial phase transformation experiments has been performed to investigate the growth kinetics of the austenite to ferrite phase transformation, and vice versa, in Fe–Mn–C alloys. Unlike the usual phase transformation experiments (100% parent phase → 100% new phase), in the case of cyclic partial transformations two special stages are observed: a stagnant stage in which the degree of transformation does not vary while the temperature changes, and an inverse phase transformation stage, during which the phase transformation proceeds in a direction contradictory to the temperature change. The experimental results have been analyzed using paraequilibrium and local equilibrium diffusional growth models. Only the local equilibrium model was shown to predict the new features of the cyclic phase transformation kinetics. The stagnant stage was found to be due to Mn partitioning, while the inverse phase transformation is caused by non-equilibrium conditions when switching from cooling to heating and vice versa.

Keywords: Local equilibrium; Paraequilibrium; Diffusion; Interface migration

1. Introduction

A full understanding of the growth kinetics of the austenite (γ) to ferrite (α) [1–5] and the ferrite (α) to austenite (γ) phase transformations [6–8] in the intercritical region is of great interest in the production of lean Mn steels, as the austenite and ferrite condition after intercritical annealing is a key factor for kinetics of the phase transformations during further cooling [9–11]. In the last few decades, the $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations have been separately investigated, including both modeling and experiments. Despite considerable efforts, some fine details related to solute partitioning and actual interface conditions remain unclear and new approaches to address these phase transformations are required.

Generally, both $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations involve two parts: nucleation and growth [12]. For nucleation, classic nucleation theory [12], and a modern variant thereof [13], are the most widely used approaches for estimating the nucleation rates in terms of parameters such as the activation energy for nucleation, the Zeldovich non-equilibrium factor, a frequency factor (the rate at which atoms are added to the subcritical nucleus) and the density of available nucleation sites. However, all parameters used to calculate the nucleation rate are difficult, or even impossible, to measure experimentally even by modern techniques [14]. In the past, for the sake of simplicity, the site-saturation nucleation concept [15] has been widely applied to modeling $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations, which unavoidably affects the accuracy of the transformation models and the kinetic parameters obtained from the analysis of the experiments.

There are two classic concepts for describing growth kinetics of solid-state phase transformations: the interface-controlled mode and the diffusion-controlled mode.

---

[*] Corresponding author. Tel.: +31 (0)152788177; fax: +31 (0)152784472.
E-mail address: hao.chen@tudelft.nl (H. Chen).

1359-6454/$36.00 © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.actamat.2011.07.033

In the interface-controlled mode, it is assumed that the free energy of transformation is dissipated by interface migration only. For more detail about the interface-controlled model, see e.g. Ref. [12]. In the diffusion-controlled mode, the free energy dissipated by interface migration is negligible, and the kinetics of phase transformations is governed by diffusion processes only. The first, and now classic, diffusion-controlled model for describing the growth kinetics of binary alloys is due to Zener [16] and is based on the local equilibrium at the moving interface. For ternary alloys Fe–C–M (M = Mn, Ni, Cr, Mo, ...), the formation of full local equilibrium at the moving interface is much more complicated than for simple binary alloys due to the addition of the substitutional element M [17]. Based on different assumptions for the partitioning mode of substitutional elements, two models have been proposed to describe the phase transformation kinetics in ternary Fe–C–M alloys: (i) the local equilibrium (LE) model [17,18], in which the interface is assumed to migrate under full local equilibrium with the partitioning of both C and M. Depending on the alloy composition and temperature, the transformation rate is determined either by carbon diffusion or M diffusion. (ii) The paraequilibrium model (PE) [17], which relies on constrained equilibrium: it indeed assumes that the phase transformation in Fe–C–M alloys can proceed without any redistribution of M and that the chemical potential of carbon across the interface should be constant. Hence, the transformation rate is only determined by carbon diffusion. In the last decades, these two models have been widely applied to describe the growth kinetics of partitioning phase transformations in Fe–C–M alloys, and their respective relevance has been discussed at length [19–22]. Although much effort has been expended to address this issue, there are still many uncertainties about the growth mode of partitioning phase transformations in Fe–C–M alloys [17].

Recently, a cyclic phase transformation concept [23] has been proposed to investigate the growth kinetics of the $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations. The main advantages of the cyclic phase transformations are: (i) the experimental and modeling uncertainties as a result of nucleation being an integral part of the transformation are avoided; and (ii) pearlite dissolution, leading to a compositionally inhomogeneous initial state and affecting the subsequent $\alpha \rightarrow \gamma$ phase transformation kinetics, is also avoided. In the cyclic phase transformation approach employed here, a simple starting stage will be built for the $\alpha \rightarrow \gamma$ phase transformation, which results in a more precise determination of the interfacial conditions and a more accurate analysis of the $\alpha \rightarrow \gamma$ phase transformation kinetics.

In this work, a series of cyclic phase transformation experiments in the $\gamma + \alpha$ two-phase region of a lean Fe–Mn–C alloy are performed using dilatometry. The corresponding cyclic phase transformation experiments are also simulated by LE and PE models. A detailed comparison between the experiments and simulation is made. The growth mode transitions during the $\gamma \rightarrow \alpha$ and $\alpha \rightarrow \gamma$ transformations are discussed, and some suggestions for various improvements to the growth models are made.

2. The cyclic heat treatments

The material investigated here is a high-purity Fe–0.17Mn–0.023C (wt.%) alloy with impurities 0.009 wt.% Si , 0.006 wt.% Ni and 0.008wt.% Cu. A Bähr 805A dilatometer is used to measure the dilation of the specimen (10 mm in length and 5 mm in diameter) during the cyclic experiments. Two thermocouples, spaced 4 mm apart, were spot-welded to the sample to obtain accurate temperature measurements and to check for the absence of a significant temperature gradient along the sample. The measured temperature gradient along the sample was always smaller than 5 K. The heat treatment procedures for the cyclic experiments in the Fe–0.17Mn–0.023C (wt.%) alloy can be divided into type I (immediate) and type H (holding) experiments, as shown in Fig. 1a,b. In both experiments, the as-received material is first fully austenized at 1000 °C and then cooled down to T_1 for 20 min isothermal holding to create a mixed ferrite–austenite microstructure with minimal compositional gradients. In type I experiments, the temperature is cycled between T_1 and T_2 without any isothermal holding at the two heating–cooling inversion temperatures. In type H experiments, the temperature is also cycled between T_1 and T_2 but with isothermal holding ($t = 20$ min) at both temperatures. Both T_1 and T_2 are located in the $\alpha + \gamma$ two-phase field in the phase diagram.

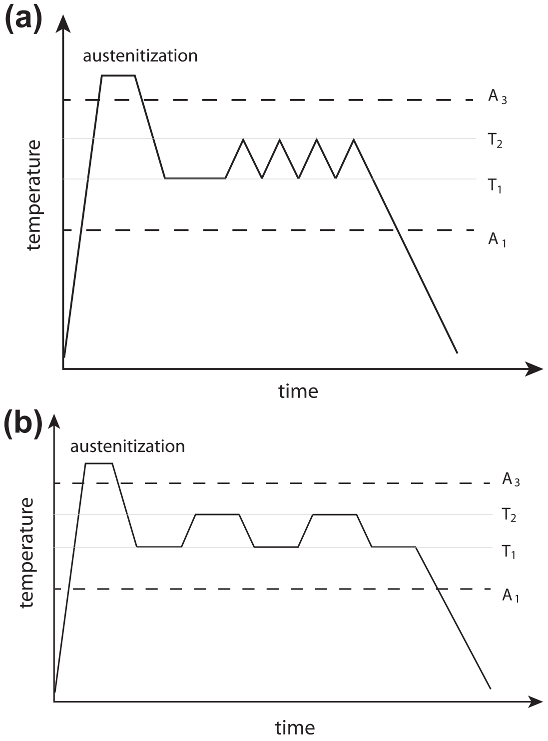

Fig. 1. The heat treatment procedures for (a) type I and (b) type H cyclic experiments.