UNIVERSIDADE DE SÃO PAULO
ESCOLA DE ENGENHARIA DE LORENA

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Phase transformations in a high-Mn steel: strain hardening mechanisms, austenite reversion, and athermal martensitic transformation

Transformações de fase em um aço de alto Mn: mecanismos de encruamento, reversão da austenita e transformação martensítica atérmica

Lorena
2019

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Thesis presented at Escola de Engenharia de Lorena of Universidade de São Paulo to obtain the degree of Doctoral of Science issued by the Programa de Pós-Graduação em Engenharia de Materiais in the field of Conventional and Advanced Materials.

Advisor: Dr. Maria José Ramos Sandim

Original Version

Lorena

2019

Ficha catalográfica elaborada pelo Sistema Automatizado
da Escola de Engenharia de Lorena,
com os dados fornecidos pelo(a) autor(a)

Souza Filho, Isnaldi Rodrigues de
Phase transformations in a high-Mn steel: strain hardening mechanisms, austenite reversion, and athermal martensitic transformation / Isnaldi Rodrigues de Souza Filho; orientadora Maria José Ramos Sandim - Versão Original. - Lorena, 2019.
152 p.

Tese (Doutorado em Ciências - Programa de Pós Graduação em Engenharia de Materiais na Área de Materiais Convencionais e Avançados) - Escola de Engenharia de Lorena da Universidade de São Paulo. 2019

1. High-mn steel. 2. Strain-hardening mechanisms. 3. Austenite reversion. 4. Athermal martensitic transformation. 5. Magnetic properties. I. Título. II. Sandim, Maria José Ramos, orient.

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Tese apresentada à Escola de Engenharia de Lorena da Universidade de São Paulo para obtenção do título de Doutor em Ciências do Programa de Pós-Graduação em Engenharia de Materiais na área de Materiais Convencionais e Avançados.

Orientadora: Profa. Dra. Maria José Ramos Sandim

Versão Original

Lorena

2019

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1. Aços de alto mn. 2. Mecanismos de encruamento. 3. Reversão da austenita. 4. Transformação martensítica atérmica. 5. Propriedades magnéticas. I. Título. II. Sandim, Maria José Ramos, orient.

ABSTRACT

SOUZA FILHO, I. R. **Phase transformations in a high-Mn steel: strain hardening mechanisms, austenite reversion, and athermal martensitic transformation**. 2019. 152p. Thesis (Doctoral of Science) – Escola de Engenharia de Lorena, Universidade de São Paulo, Lorena, 2019.

In this work, phase transformations in a high-Mn steel containing 17.6 wt.% of Mn and belonging to the Fe-Mn-Al-Si-C-Ni system were investigated for a variety of states, including cold rolled and annealed ones. The strain hardening mechanisms of austenite, ε-, and α'-martensite were tracked during cold rolling. The complex superposition of several displacive reactions were revealed for each phase with the aid of the combinatorial use of XRD measurements (coupled with the software MAUD), ECCI-SEM, and EBSD. Dilatometry measurements revealed that the austenite reversion splits into two stages during continuous annealing. Such phenomenon is due to strong elemental partitioning between the growing austenite and the α'-matrix, as simulated using the software DICTRA and confirmed via near-atomic resolution APT. Results provided new insights that the successful austenite nucleation is preceded by long-range elemental partitioning. Besides, the growth of austenite is also given by strong elemental partitioning and solute redistribution within both austenite and α'-martensite. Magnetic properties were also investigated for several microstructures of the present steel, modified by means of straining and/or annealing. The formation of nano reversed γ-grains in the early stages of the austenite reversion is sufficient to induce strong magnetic shape anisotropy. Using *in-situ* magnetic measurements and thermodynamic modelling, the Curie temperature of the steel was evaluated, as well as the stability of austenite during controlled conditions of cooling. The influence of local changes in chemical composition on the magnetic properties was deeply investigated by means of magnetic measurements, thermodynamic simulations (Thermo-Calc), high-resolution microscopy, including STEM, and APT. The findings revealed that short- and long-range chemical fluctuations strongly affect the saturation magnetization of the steel and brought new insights on the use of magnetic probing as tool for quantification of phases in Mn-based steels.

Keywords: High-Mn steel. Strain-hardening mechanisms. Austenite reversion. Athermal martensitic transformation. Magnetic properties.

RESUMO

SOUZA FILHO, I. R. **Transformações de fase em um aço de alto Mn: mecanismos de encruamento, reversão da austenita e transformação martensítica atérmica**. 2019. 152p. Tese (Doutorado em Ciências) – Escola de Engenharia de Lorena, Universidade de São Paulo, Lorena, 2019.

Neste trabalho, transformações de fases foram estudadas em um aço contento 17,6 % em massa de Mn e pertencente ao sistema Fe-Mn-Al-Si-C-Ni. Os mecanismos de encruamento na austenita, martensitas ε e α’ foram monitorados durante a laminação a frio. A complexa superposição de várias transformações adifusionais foram reveladas para cada fase com auxílio de medidas de difração de raios X (acopladas ao uso do *software* MAUD), ECCI e EBSD. Medidas de dilatometria revelaram que a reversão da austenita ocorre em dois estágios durante recozimentos contínuos. Esse fenômeno é devido à pronunciada partição de elementos entre a austenita revertida e a matriz martensítica, como mostrado por meio de simulações utilizando-se o *software* DICTRA e confirmado via APT. Os resultados obtidos evidenciaram que a nucleação bem-sucedida da austenita é precedida da partição de longo alcance de elementos químicos. Além disso, o crescimento da austenita revertida também é acompanhado de partição de longo alcance e redistribuição de solutos entre as fases γ e α’. As propriedades magnéticas do presente aço também foram investigadas para uma ampla variedade de microestruturas, modificadas por deformação e/ou recozimento. Foi observado que a formação de grãos austeníticos de ordem nanométrica no início da reversão é suficiente para induzir pronunciada anisotropia magnética de forma. Por meio de medidas de magnetização *in-situ* e modelamento termodinâmico, a temperatura de Curie foi avaliada como também a estabilidade da austenita frente ao resfriamento controlado. A influência de mudanças composicionais sobre as propriedades magnéticas foi detalhadamente investigada com auxílio de medidas magnéticas, simulações termodinâmicas (Thermo-Calc), microscopia de alta resolução, incluindo microscopia eletrônica de transmissão (MET), e APT. Os resultados mostraram que flutuações químicas de curto e longo alcance afetam fortemente a magnetização de saturação do material. Além disso, este trabalho trouxe novos *insights* acerca do uso de medidas de magnetização como uma ferramenta para quantificação de fases em aços a base de Mn.

Palavras-chave: Aços de alto Mn. Mecanismos de encruamento. Reversão da austenita. Transformação martensítica atérmica. Propriedades magnéticas.

Chapter 1

Introduction

In the last decades, the rising interest in Mn-based steels for automotive industry applications is due to their outstanding mechanical properties (FROMMEYER; BRÜX, 2006; DE COOMAN; CHIN; KIM, 2011; KIM; SUH; KIM, 2013; RAABE et al., 2014; ZHANG; RAABE; TASAN, 2017). These materials are known as advanced high strength steels (AHSS) and combine high strength with ductility without sacrificing the crashworthy properties. Depending on several metallurgical aspects, especially the chemical composition in terms of Mn, metastable austenite (γ) in these steels may accommodate deformation by means of different and complex strain hardening mechanisms. Among them, one can cite the transformation-induced plasticity (TRIP effect) where fcc-γ transforms into bcc α'-martensite with or without the intermediate presence of the hcp ε-phase (REMY; PINEAU, 1977; LEE; CHOI, 2000; ALLAIN et al., 2004; BRACKE; KESTENS; PENNING, 2007; SAEED-AKBARI et al., 2009; DING et al., 2011; STEINMETZ et al., 2013; PIERCE et al., 2014; WONG et al., 2016). Another important mechanism for strain accommodation is the twinning-induced plasticity (TWIP effect), where the profuse formation of nano-twins subdivides the original γ-grains into smaller units and consequently diminishes the mean free path for dislocation motion (GRÄSSEL et al., 2000; DE COOMAN; CHIN; KIM, 2011). In terms of Mn, the TRIP effect is generally observed for steels with Mn content lower than 15 wt.%. For Mn compositions higher than 25 wt.%, the TWIP effect is predominant. Finally, for Mn contents ranging between 15 and 25 wt.%, both TRIP and TWIP mechanisms may take place simultaneously (GAZDER et al., 2015).

Recently, Mn-based steels have been designed by means of precipitation reactions, segregation engineering (RAABE et al., 2013), and grain refinement (ZHANG; RAABE; TASAN, 2017). Reduction of anisotropy in mechanical properties is one of the most important advantages of strengthening by means of grain refinement (ESCOBAR; DAFÉ; SANTOS, 2015). This can be achieved by the transformation of α'-martensite back into austenite using the so-called austenite reversion treatments (α' → γ) (DASTUR et al., 2017). In this context, the elemental redistribution (especially Mn) between parent α' and growing γ plays a key role on the final microstructure and mechanical properties of the steel. In fact, Mn possesses very low diffusivity in austenite. Contrastingly, its high diffusivity in α'-martensite promotes Mn fluxes towards the growing austenite, leading to strong partitioning and segregation at α'/γ interfaces and grain boundaries (DMITRIEVA et al., 2011). In this scenario, the segregation tendency is also observed to be enhanced by high Mn contents.

In light of the above observations, the high-quality performance of Mn-based steels lies on the complexity of their microstructural aspects. In this Doctorate work phase transformations driven either by diffusionless or diffusion mechanisms were carefully investigated for a high-Mn steel belonging to the Fe-Mn-Al-Si-C-Ni system and containing 17.6 wt.% of Mn. The present steel possesses a stacking fault energy (SFE) of ~ 8.1 mJ/m^2 and therefore the formation of α'-martensite is preceded by the intermediate formation of the ε-phase (SOUZA FILHO et al., 2019a). The complex superposition of several displacive (diffusionless) and incommensurate transformation reactions occurring concomitantly in γ, ε - and α'-martensite were revealed for a broad range of strain imposed by rolling (SOUZA FILHO et al., 2019b). With regard to the diffusion reactions, austenite reversion was tracked for continuous and isothermal conditions of annealing. Relevant and new results regarding austenite nucleation and growth, as well as the corresponding elemental redistribution during the occurrence of the α' → γ reaction threw lights on the thermo-kinetics of the austenite reversion in Mn-based steels (SOUZA FILHO et al., 2019c). Starting with fully austenitic microstructures at high temperatures (viz. 800°C), the stability of austenite faced to the athermal martensitic transformation was also investigated by means of *in-situ* magnetic measurements for controlled conditions of cooling and thermodynamic modelling (SOUZA FILHO et al., 2019a). Last but not least, the changes in magnetic properties (especially regarding the coercive field and saturation magnetization) were evaluated for a variety of microstructures modified by straining and/or annealing. In this work, the strong influence of chemical composition fluctuations on the magnetization was clearly revealed for the Mn-based steels variants containing the ferromagnetic α'-martensite. The systematic investigation conducted in this work brings new insights about the use of the magnetization saturation as a probe for quantifying ferromagnetic phases in steels.

This thesis is organized in seven chapters as follows:

Chapter 1: Introduction

Chapter 2: Experimental

Chapter 3 Thermodynamic assessment

Chapter 4: Strain hardening mechanisms during cold rolling: interplay between submicron defects and microtexture

This Chapter is a modified version of the present author's publication: **I.R. Souza Filho**, M.J.R. Sandim, D. Ponge, H.R.Z. Sandim, D. Raabe. Strain hardening mechanisms during cold rolling of a high-Mn steel: Interplay between submicron defects and microtexture. Materials Science & Engineering A, v.754, p.636-649, 2019.

Chapter 5: Martensite to austenite reversion: partitioning-dependent two-stage kinetics revealed by atom probe tomography, in-situ magnetic measurements and simulation

This Chapter is a modified version of the present author's publication: **I.R. Souza Filho,** A. Kwiatkowski da Silva, M.J.R. Sandim, D. Ponge, B. Gault, H.R.Z. Sandim, D. Raabe. Martensite to austenite reversion in a high-Mn steel: Partitioning dependent two-stage kinetics revealed by atom probe tomography, in-situ magnetic measurements and simulation. Acta Materialia, v.166, p.178-191, 2019.

Chapter 6: Magnetic properties: study of strain-induced martensite formation, austenite reversion, and athermal α'-formation

This Chapter is a modified version of the present author's publication: **I.R. Souza Filho**, M.J.R. Sandim, R. Cohen, L.C.C.M. Nagamine, H.R.Z. Sandim, D. Raabe. Magnetic properties of a 17.6 Mn-TRIP steel: Study of strain-induced martensite formation, austenite reversion, and athermal α′-formation. Journal of Magnetism and Magnetic Materials, v.473, p.109-118, 2019.

Chapter 7: Revealing the anomalous increase in magnetization of α'-martensite at low temperatures

8. Final Remarks

Since the high-quality performance of Mn-based steels is closely related to microstructural aspects and in order to design high-strength variants of such materials, it is imperative to deeply understand their complex superposition of several strain hardening mechanisms, diffusion-controlled reactions, as well as their influence on mechanical properties. In this work, a systematic characterization of a high-Mn steel containing 17.6 wt. % of Mn and belonging to the Fe-Mn-Al-Si-C-Ni system was conducted for a variety of deformed and annealed conditions. Starting with a fully recrystallized microstructure, the combinatorial use of XRD, ECCI-SEM, and EBSD allowed revealing the occurrence of several displacive and important reactions in γ, ε - and α'-martensite, including the formation of sub-micron structural defects and the martensitic transformation induced by deformation. With regard to the austenite reversion, near-atomic resolution APT combined with thermodynamic and thermo-kinetic simulations, and dilatometry measurements brought new information about the challenging observation of the γ-nucleation in high-Mn steels. This work also contributes to add new evidences to the literature that the successful γ-nucleation events are preceded by strong long-range elemental partitioning, especially in terms of Mn. Besides, the growth of reversed austenite is also controlled by pronounced elemental partitioning and solute redistribution, as revealed by thermo-kinetic simulations and confirmed via APT. Magnetic measurements are considered as one of the most powerful tools for investigating phase transformations in steels. By using *ex-situ* and *in-situ* magnetic measurements, the stability of austenite faced the martensitic transformation upon cooling was studied and the findings of this work confirmed that the ε-martensite is an intermediate phase for α'-formation. Regarding isothermal annealings, the formation of nano reversed γ-grains in the early stages of the austenite reversion is sufficient to induce strong magnetic shape anisotropy. Finally, the strong influence of short and long-range chemical fluctuations on the magnetic properties was studied using APT, high-resolution microscopy, thermodynamic simulations, and magnetic measurements. It was clearly demonstrated that the Mn-based steels variants containing the ferromagnetic α'-martensite are very sensitive to local changes in chemical composition. The results reported in this thesis throw new insights regarding the use of the magnetization saturation as a probe for quantifying ferromagnetic phases in steels.

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