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Meta-equilibrium transition microstructure for maximum austenite stability and minimum hardness in a Ti-stabilized supermartensitic stainless steel^{*}



J.D. Escobar ^{a,*}, J.P. Oliveira ^b, C.A.F. Salvador ^a, G.A. Faria ^c, J.D. Poplawsky ^d, J. Rodriguez ^e, P.R. Mei ^a, S.S. Babu ^{f,g}, A.J. Ramirez ^c

^a College of Mechanical Engineering, University of Campinas – FEM - Unicamp, Campinas, SP 13083-860, Brazil

^b UNIDEMI, Department of Mechanical and Industrial Engineering, NOVA School of Science and Technology, NOVA University Lisbon, 2829-516 Caparica, Portugal

^c Welding Engineering, Department of Materials Science and Engineering, The Ohio State University, 1248 Arthur E. Adams Drive, Columbus, OH 43221, USA

^d The Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, 37831, USA

^e Mechanical Engineering Department, EIA University, Envigado, Colombia

^f Mechanical, Aerospace and Biomedical Engineering, University of Tennessee, Knoxville, TN 37996-2210, USA

^g Manufacturing Demonstration Facility, Oak Ridge National Laboratory, Knoxville, TN, USA

HIGHLIGHTS

GRAPHICAL ABSTRACT

- Compositional gradients after single tempering forced a meta-equilibrium during the second and third tempering cycles.
- Nucleation at the Ni-poor tempered matrix was suppressed.
- Site-specific austenite reversion occurred at the Ni-rich fresh martensite laths.
- The softening mechanism was insensitive to the maximization of austenite after double and triple tempering.

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ABSTRACT

The maximization of stable reverted austenite at room temperature through inter-critical tempering is a widely used method to reduce hardness in supermartensitic stainless steels. Nevertheless, partial martensitic transformation might occur due to insufficient compositional stabilization. In this work, we conducted a time-resolved triple-step inter-critical tempering, specially designed to obtain maximum austenite stability and minimum hardness through the progressive suppression of the martensitic transformation. The mechanism behind the progressive increase in stable reverted austenite was the generation of a meta-equilibrium state, which imposed a

E-mail addresses: juliandescobar@gmail.com jdescoba01@fem.unicamp.br (J.D. Escobar), jp.oliveira@campus.fct.unl.pt (J.P. Oliveira), csalvador@fem.unicamp.br (C.A.F. Salvador), abreufaria.1@buckeyemail.osu.edu (G.A. Faria), poplawskyjd@ornl.gov (J.D. Poplawsky), johnnatan.rodriguez@eia.edu.co (J. Rodriguez), pmei@fem.unicamp.br (P.R. Mei), sbabu@utk.edu (S.S. Babu), ramirezlondono.1@osu.edu (A.J. Ramirez).

Abbreviations: α_{eq} . ferrite in thermodynamic equilibrium; α'_{f} , fresh martensite; α'_{t} , tempered martensite; γ , used to generically refer to austenite; γ_{eq} , austenite in thermodynamic equilibrium; γ_{r} , reverted austenite stabilized at room temperature; APT, Atom Probe Tomography; EDS, Energy Dispersive X-ray Spectroscopy; ICT, inter-critical tempering; SMSS, supermartensitic stainless steels; SXRD, synchrotron X-ray diffraction.

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Corresponding author.

Keywords: Atom Probe Tomography Austenite reversion Isothermal tempering treatments Synchrotron diffraction limit in both high temperature austenite reversion and room temperature austenite stabilization. Such limit corresponded to the high temperature volume fraction of austenite, obtained right before cooling from the first cycle. This effect was associated to the Ni-rich fresh martensite laths acting as local Ni compositional pockets, providing site-specific austenite reversion; and to the suppression of any additional nucleation at the Ni-poor matrix as the T0 temperature for austenite reversion was strongly increased. The softening mechanism was mainly controlled by the carbon arrest effect by the precipitation of Ti (C, N), which was completed after the first tempering cycle. Nevertheless, maximizing reverted austenite and suppressing fresh martensite at room temperature did not result in additional hardness reductions.

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1. Introduction

The isothermal reversion of nano-sized austenite (γ_r) in tempered martensitic matrixes (α'_t) has regained interest in the past decade as a methodology for the design, control and optimization of the mechanical properties of martensitic steels [1-4]. As an example, the austenite reversion has been used for tailoring the mechanical properties of supermartensitic stainless steels (SMSS) pipelines in the oil and gas industry [5-9]. The maximization of the γ_r volume fraction at room temperature after inter-critical tempering (ICT) has been directly associated with the simultaneous maximization of the impact toughness and minimization of hardness [6,9-11]. The latter is especially important in order to satisfy the maximum hardness requirement of 253 HV for field applications in sour service, commonly adopted by the oil and gas industry [12]. Nevertheless, this has proven to be challenging due to the relatively small changes in hardness after tempering cycles [13,14]. Thus, a variety of methodologies for hardness minimization have been proposed, such as the reduction of C and N from the nominal composition [13] and the addition of stabilizing elements such as Ti, Nb and V to arrest the interstitial elements from the solid solution [15].

Additionally, complex multiple ICT cycles have been performed as an effort to increase the volume fraction of stable γ_r at room temperature, from 0.05 to 0.2 after single ICT cycles [16–19] up to 0.4 [6,10,20]. The increased reversion efficiency obtained by the use of multiple ICT cycles has been related to a wide variety of microstructural effects, such as the presence of retained austenite [4,16,21,22], reverted austenite and fresh martensite after a single ICT cycle [10,16,23], the precipitation of carbides [11,24] and even to compositional segregations after the controlled dissolution of carbides [2,25]. Interpreting the austenite reversion mechanism through multiple tempering cycles can be rather complex due to the aforementioned competitive microstructural effects. However, most of these can be avoided or isolated under controlled conditions in Ti-stabilized SMSS.

This work aims to clarify the kinetic and compositional aspects behind the austenite nucleation and stabilization mechanisms throughout the execution of a triple ICT cycle, designed to achieve maximum austenite stability and minimum hardness. Before tempering, samples were fully austenitized to provide a well-known starting microstructure, consisting of compositionally homogeneous fresh martensite and stable titanium carbo-nitrides, Ti (C, N), in the absence of retained austenite. Then, the samples were subjected to controlled single, double and triple ICT cycles. The evolution of the microstructure was studied by time-resolved synchrotron X-ray diffraction throughout the thermo-mechanical simulation of the multiple ICT heat treatment. Furthermore, samples were extracted after each step of the triple ICT heat-treatment in order to be further characterized using Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDS) in Scanning Transmission Electron Microscopy (STEM) and Atom Probe Tomography (APT). The maximization of γ_r and the carbon arrest from the solid solution were directly related to the resultant hardness modifications. Results were interpreted with the aid of equilibrium and kinetic calculations.

2. Experimental

2.1. Time-resolved synchrotron X-ray diffraction during thermomechanical simulation

A commercial hot rolled and homogenized Ti-Stabilized SMSS with a nominal composition shown in Table 1 was used. Dog-bone type samples with 2 mm thick, 5 mm wide and 20 mm long reduced section were machined and then homogenized at 950 °C during 20 min, followed by furnace cooling to room temperature. A constant flux of Argon was used to protect the samples from decarburization and oxidation. A fully martensitic microstructure was obtained [17].

Time-resolved synchrotron X-ray diffraction (SXRD) experiments were conducted in order to study the austenite transformation kinetics during the heating, isothermal and cooling stages throughout the multiple ICT heat treatment. This analysis was performed at the X-ray scattering and thermo-mechanical simulation experimental station (XTMS), at the Brazilian National Synchrotron Source (LNLS). Incident beam energy of 12 keV was used. A thermo-mechanical simulator (Gleeble® 3S50) coupled to the synchrotron light source was used to perform the heat treatments. The first, second and third ICT cycles were conducted at 625 °C during 2.5 h, 600 °C during 6 h, and 580 °C during 6 h, respectively. A final austenitization cycle was conducted at 950 °C for 5 min, followed by cooling to room temperature. All tempering cycles were performed using the direct resistive heating method, controlled by type K thermocouples welded to the center of the reduced section of the samples. The typical temperature uncertainty was ± 1 °C. In all cases, heating and cooling rates were fixed to 0.166 $^{\circ}C \cdot s^{-1}$ and 5 $^{\circ}C \cdot s^{-1}$, respectively. The transformation temperatures at the surface and bulk of the samples were indirectly measured by X-ray diffraction and laser dilatometry data analysis, respectively. A graphical representation of the aforementioned four heat treatment cycles is shown in Fig. 1.

The quantification of the X-ray diffraction data was performed for the austenite using Eq. (1) [26,27], which establishes a relationship between the measured peak areas and the corresponding phase fractions. In this equation, F_p is the fraction of phase p, n_p is the number of peaks from phase p, K represents a given {*hkl*} family, I_{pK} is the area under a peak of the family K on phase p and R_{pK} is a dimensionless scalar containing the effects of the form factor and multiplicity of each {*hkl*} family for each phase, as well as the influence of the lattice parameters. Such quantification is relative to the surface since the experiment was set for X-ray reflection configuration obtained at 12 keV. The timeresolved austenite quantification was conducted using {111} γ , {200} γ peaks and {110} α' peak. Also, a wider spectra, including {111} γ ,

 Table 1

 Nominal composition of a Ti-stabilized SMSS.

	С	Ν	Si	Mn	Ni	Cr	Мо	Cu	Ti	V
wt%	0.024	0.0129	0.260	0.480	5.90	12.02	1.93	0.09	0.130	0.040
at.%	0.111	0.0513	0.516	0.487	5.60	12.88	1.13	0.08	0.153	0.044

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