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Mechanisms of ultrafine-grained austenite formation under different isochronal conditions in a cold-rolled metastable stainless steel

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ABSTRACT

The primary objective of this work is to obtain fundamental insights on phase transformations, with focus on the reaustenitization process (α′→γ transformation), of a cold-rolled (CR) semi-austenitic metastable stainless steel upon different isochronal conditions (0.1, 1, 10 and 100 °C/s). For this purpose, an exhaustive microstructural characterization has been performed by using complementary experimental such as scanning and transmission electron microscopy (SEM and TEM), electron backscattered diffraction (EBSD), electron probe microanalysis (EPMA), micro-hardness Vickers and magnetization measurements. It has been detected that all microstructural changes shift to higher temperatures as the heating rate increases. The reaustenitization occurs in two-steps for all heating rates, which is attributed to the chemical banding present in the CR state. The α′→γ transformation is controlled by the migration of substitutional alloying elements across the austenite/martensite (γ/α′) interface, which finally leads to ultrafine-grained reaustenitized microstructures (440–280 nm). The morphology of the martensite phase in the CR state has been found to be the responsible for such a grain refinement, along with the presence of χ-phase and nanometric $Ni_3(Ti,Al)$ precipitates that pin the austenite grain growth, especially upon slowly heating at 0.1 °C/s.

1. Introduction

Metastable austenitic stainless steels (MASSs) with ultra-high strength are well known due to their good combination of corrosion resistance, moderate ductility and strength, which make them competitive for a number of applications [1,2] and put them on the spot for several structural applications in a near future [3]. With a carefully balanced addition of austenite and ferrite stabilizers, it is possible to retain the austenite (γ) phase at room temperature with an adequate metastablity. Under certain conditions and processes, such as the application of stresses/strains [4–7] or cryogenic treatments [8], this metastable austenite transforms into martensite (α′) phase, enhancing the yield and tensile strength of the material. Additionally, these steels contain alloying additions that might form strengthening particles within the martensite upon the adequate aging treatment, raising the mechanical properties to ultra-high-strength (UHS) values [9–13]. One of the key microstructural parameters to improve the strength without compromising much the ductility is the austenite grain size (AGS). It is clear from the literature that the application of advanced thermo-mechanical processing treatments to MASSs, as the severe plastic cold-deformation followed by a controlled annealing, leads to nano/ultra-fine-grained (NG/UFG) microstructures with improved mechanical properties, compared to their coarse-grained counterparts [14–21]. To obtain the desire microstructural refinement, the metastable austenite should be completely transformed into martensite during the cold deformation, for which a great percentage of deformation is required [5,6,20–24]. It appears that cold deformations beyond the level needed to form lath-type martensite result in a crystallographic distortion of this morphology, leading to a dislocation-cell-type of martensite, with an increase in the dislocation density and, thus, in the number of nucleation sites. A carefully controlled annealing treatment ensures the grain refinement of the final austenitic microstructure [6,14–21]. In this manner, Forouzan et al. [16] attained grain sizes of 330 nm in an AISI 304 L; Misra and co-authors obtained UFG (100–500 nm) austenitic structures in 17Cr-7Ni-N [6] and in an AISI 301LN [17] and Weidner et al. [21] obtained an AGS below 1 μm after annealing a 90% cold-deformed high-alloyed austenitic TRIP steel. All of these microstructures exhibited ultimate tensile strengths of about 1100 MPa and elongations in the range 30–40%. The outstanding mechanical properties of NG/UFG MASSs stem from the instability of the austenite phase, which can easily transform into strain-induced martensite during plastic deformation due to the Transformation Induced Plasticity (TRIP) effect [25].

The α′→γ transformation has been extensively studied due to its role in the grain refinement of austenitic microstructures [9,26–30].

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These works refer to investigations on phase transformations upon anisothermal conditions and point out the effect of the heating rate on processes such as precipitation and reversion ($\alpha'\rightarrow\gamma$) is pointed out. Phase transformations can occur in different ways, i.e. by a reconstructive or displacive mechanism, long- or short-range diffusion-controlled of substitutional or interstitial elements. The main mechanism prevailing during the transformation depends on the driving force and the heating rate (the time available for a transformation). Besides, any process occurring before the $\alpha'\rightarrow\gamma$ transformation (recovery, clustering, precipitation) give rise to the redistribution of alloying elements, which affects the reversion. For instance, by using atom probe tomography (APT) and dilatometry, Moszner and co-authors [30] studied the effect of prior precipitation in the reverse transformation of Fe-Mn-Pd maraging-type alloys and found that the partitioning of the Mn in the matrix leads to a change in the dominating transformation mechanism, from long-range diffusion to interface-controlled. In the same manner, this phenomenon as well as the $\alpha'\rightarrow\gamma$ transformation mechanism have been studied in other precipitation hardenable steels such as PH13-8 Mo, 17-4 PH or maraging steels [9,31]. Apart from to the above mentioned factors, additional challenges arise in the study of the $\alpha'\rightarrow\gamma$ transformation when the steel under consideration exhibits a strong texture and a pronounced segregation as a consequence of severe deformation [32–34]. To obtain austenitic microstructures with the targeted mechanical properties in a MASS requires the control of the stability of the austenite phase. This stability is closely bounded to the control of the thermo-mechanical processing, which requires the fundamental knowledge on solid-solid phase transformations in this type of steels.

Therefore, the goal of this work is to examine the evolution of the microstructure upon continuous heating and, thus, the processes that lead to UFG microstructures in a MASS. For this purpose, four isochronal conditions (0.1–100 °C/s) have been selected and their influence on the recovery, precipitation and $\alpha'\rightarrow\gamma$ transformation has been investigated. In addition to understanding of the mechanisms behind phase these phase transformations upon continuous heating in this kind of steels, these results will give valuable information to exert control over the microstructural design.

2. Material and experimental procedure

2.1. Material and heat-treatments

The MASS investigated in this work presents the chemical composition shown in Table 1. The material has been delivered in the form of cold-rolled (CR) sheets of 300 mm in width and 0.45 mm in thickness after being manufactured by continuous casting followed by several hot- and cold-rolling steps. During the final cold-deformation steps the material is subjected to a 92.5% cold-reduction, which causes the almost complete transformation of the metastable austenite (γ) phase into heavily deformed martensite (α') phase. The CR microstructure, which has been characterized in a previous work [33], consists of heavily deformed martensite phase ($f_{\alpha'}\sim 0.97$) with small volume fractions of the intermetallic χ-phase ($Fe_{36}Cr_{12}Mo_{10}$) ($f_{\chi}\sim 0.02$) and retained phases such as austenite and δ-ferrite ($f_{\gamma+\delta}\sim 0.01$) and few titanium nitrides.

Specimens of 12 mm in length and 4 mm in width have been cut with their axial length perpendicular to the steel-sheet rolling direction and have been subjected to interrupted heating by quenching (~300 °C/s) at 0.1, 1, 10 and 100 °C/s up to different temperatures. Heat-treatments, schematically summarized in Fig. 1, have been performed using the high precision furnace of a high-resolution dilatometer Adamel Lomargy DT1000, which operates in a vacuum atmosphere of 10^{-1} mbar. In Fig. 1, A_S and A_F stand for the starting and finishing $\alpha'\rightarrow\gamma$ transformation temperatures.

2.2. Experimental limitations

It is important to mention that some inherent features of the material investigated in this work, i.e. the chemical banding along the steel sheet cross-section, the stability of the sheet or the small sheet thickness, have precluded the use of some experimental techniques. For instance, phase transformations take place inhomogeneously due to the pronounced chemical banding present in the CR state. Therefore, to obtain representative results of the whole material, it is mandatory to perform the microstructural characterization on the cross-section rather than on the surface perpendicular to the normal direction. On the other hand, the low sheet thickness (0.45 mm) makes it difficult to perform standard laboratory X-ray diffraction on the cross-sectional area since a minimum surface of 1 cm^2 is recommended. When X-ray diffraction measurements are performed on the normal section, the results may be unreliable since they would be dependent on the chemical banding. In a similar way, due to the small sheet thickness, TEM thin foils have to be prepared parallel to the sheet longitudinal section, being thus also dependent on the chemical banding. Besides, the metastability of the austenite phase also complicates the preparation of TEM thin foils and surfaces for EBSD, since it may transform into martensite during grinding and electropolishing. Therefore, the higher the volume fraction of austenite, the more tedious the thin foil preparation is. Moreover, the martensitic transformation is enhanced as the thickness of the samples is reduced.

Finally, it has been found in a previous work that high-resolution dilatometry, generally used to characterize phase transformations in steels upon continuous heating [35,36], is not able to detect the whole contraction associated to the $\alpha'\rightarrow\gamma$ transformation [34]. As a consequence of the above mentioned problems, bulk-volume measurements techniques, such as magnetization or micro-hardness Vickers measurements, have been identified as the most suitable for characterization in this research. In addition, other complementary experimental techniques have been employed to aid in the understanding of the results.

2.3. Characterization techniques

2.3.1. Micro-hardness Vickers tests

Micro-hardness Vickers tests are rapid, easy-to-use and cheap bulk-measurements that give valuable ex-situ understanding of the microstructural evolution upon continuous heating. For this reason, indentations have been performed along the polished cross-section of CR state and on interrupted heated by quenching samples for all heating rates. A WILSON WOLPER 401 MVA equipment, using a load of 1 kg with an indentation time of 15 s has been used. Hardness is reported as the mean value and its corresponding standard deviation obtained after the measurement of at least five indentations.

2.3.2. Magnetization measurements

A quantum design MPMS-XL SQUID magnetometer has been employed to characterize ex-situ, on heat treated samples, the kinetics of the $\alpha'\rightarrow\gamma$ transformation for all heating rates. By measuring the magnetization saturation (M_{Sat}) of a sample, its martensite volume fraction ($f_{\alpha'}$) can be calculated through: $f_{\alpha'}=M_{Sat}/M_{Sat}^{\alpha'}$; where $M_{Sat}^{\alpha'}$ is the saturation magnetization of a sample containing 100% of martensite

Table 1
Chemical composition of the steel under investigation in wt.% with balanced Fe.

Cr	Ni	Mo	Cu	Ti	Al	Si	Mn	C+N
12	8.87	4.05	1.91	1.35	0.39	0.36	0.33	<0.01