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Deformation induced martensitic transformation in 304 austenitic stainless steel: In-situ vs. ex-situ transmission electron microscopy characterization

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ABSTRACT

304 stainless steel is known to be metastable as the austenite phase can transform into martensite under deformation. In this work, both ex-situ and in-situ transmission electron microscopy (TEM) characterization were used to investigate the mechanisms of the deformation-induced transformation at room temperature. The ex-situ tensile tests were conducted at a strain rate of $10^{-3}\,s^{-1}$ until rupture, followed by TEM and X-Ray Diffraction (XRD). Samples were also interrupted at strains of 7%, 18%, and 30% with the goal of investigating the intermediate microstructure. In addition, tensile tests were conducted in-situ in a TEM at 25 °C using a special straining-stage with the goal of capturing the nucleation and growth of the martensitic phase as it develops under deformation. The formation of stacking faults and the subsequent formation of ε-martensite (hcp) through their overlapping/bundling was captured in-situ, confirming the role played by Stacking Faults (SFs) as intermediate step during the transformation from γ-austenite to ε-martensite. Direct transformation of γ-austenite (fcc) to α'-martensite (bcc) was also captured upon straining and characterized. Such unique in-situ observations showcase how in-situ straining in a TEM, as a small scale tensile technique, is a powerful technique to visualize and investigate the mechanisms of deformation induced phase transformations.

1. Introduction

Thanks to its good mechanical properties, chemical corrosion resistance, and low cost, 304 stainless steel (and its low carbon variant 304L) is widely used in engineering applications. It is known to be metastable as the austenite phase can transform into martensite under deformation. Although the martensitic transformation in general has been the object of many studies over several decades and models have been proposed to explain possible mechanisms of the transformation, one limitation in the study of the deformation induced martensitic transformation, as is the case for such deformation induced phase transformations in general, has been the lack of in-situ direct observation to validate underlying hypothesis or to discover the microstructural processes at play, particularly in the nucleation process. Usually tensile experiments are done on bulk samples and the microstructure is characterized after the experiment to look for clues on how the transformation may have happened. Hence, the usefulness of developing in-situ test capabilities. In this context, tensile tests were conducted in-situ in a TEM at 25 °C using a special straining-stage with the goal of capturing the nucleation and growth of the martensitic phase as it develops under deformation in complement to bulk tensile experiments (carried out to rupture or interrupted).

2. Materials and experiments

2.1. Materials

Different grades of this alloy exist, 304, 304L and 304H. The main difference between these three grades is the carbon content with nominal content ranges of [0.04–0.1 wt%] in 304 H, [0.03–0.07 wt%] in regular 304SS, and less than 0.03 wt% in 304L. 304 and 304L grades stainless steels were used in the current research. The material was obtained in the shape of a sheet of 304 SS thickness of 0.51 mm and a 304L bar with 3.2 mm radius. The chemical composition (in wt%) of the materials is shown in Table 1.

The microstructure of the as-received 304 sheet and 304L bar was characterized by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). The SEM micrographs shown in Fig. 1 indicate that the microstructure of 304 and 304L SS consists of polygonal grains of austenite with twins interspersed in some grains. The 304 sheet showed a fraction of martensitic phase originating from the cold-rolling process; (the pre-existing martensite is marked by the white arrows on Fig. 1a.).

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Table 1
Materials chemical composition (wt%).

	C	Mn	P	S	Si	Cr	Ni	N	Mo	Cu	Fe
304 sheet	0.042	0.84	0.032	0.003	0.39	18.23	8.09	0.053	0	0	bal.
304L bar	0.03	1.82	0.03	0.04	0.31	18.08	8.02	0.08	0.33	0.41	bal.

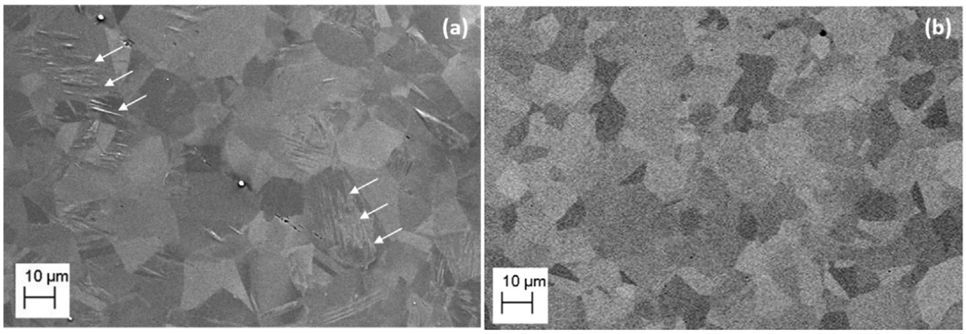

Fig. 1. SEM characterization of as-received 304 sheet and 304L bar stainless steel sample.

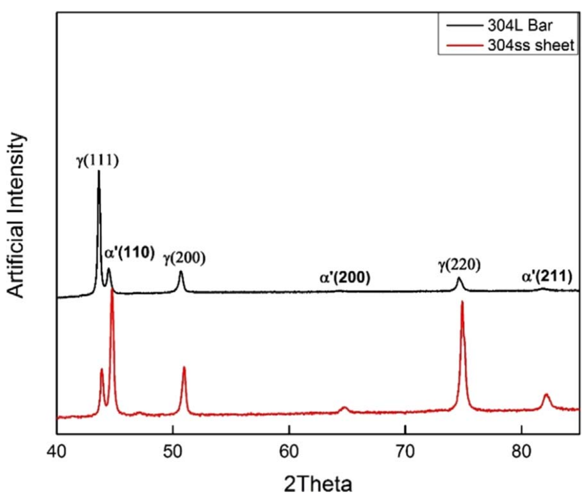

Fig. 2. XRD patterns of as-received 304 sheet and 304L bar.

The content of martensite was further confirmed with XRD (Fig. 2). The 304L bar showed much less initial martensite content.

The size of the austenitic grains was determined using Image J software. Austenitic grains shown in Fig. 1 were outlined and scanned into Image J. The software was used to measure the area of each grain automatically. Then an equivalent diameter of each grain could be estimated. The average grain size (equivalent diameter) of 304 sheet and 304L bar was thus found to be about 21 µm and 24 µm respectively. The inclusions found in the 304 sheet were essentially rich in Cr and Mn.

One important parameter used to describe Deformation induced martensite (DIM) is M_{d30} which is the temperature for which 50% of martensite has formed at 30% true strain. This temperature is a good measure of the "stability" of the metastable stainless steels [1]. Multiple empirical formula for calculating M_S and M_{d30} were summarized by Hahnenberger [2]. In this work, Pickering and Angel's formula were chosen to calculate M_S and M_{d30} [3,4].

$$M_{S,Pickering} = 502 - 810C - 1230N - 13Mn - 30Ni - 12Cr - 54Cu - 46Mo \quad (1)$$

$$M_{d30,Angel} = 413 - 462(C + N) - 9.2Si - 8.1Mn - 13.7Cr - 9.5Ni - 18.5Mo \quad (2)$$

The Stacking Fault Energy (SFE) is also an important parameter for determining how easy stacking fault can form and subsequently hcp martensite. For the record, twinning is reported to occur at SFE at a range of 18–45 mJ/m^2 [5] and SFs occur more easily when the SFE is lower. Eq. (3) (below) was used to calculate SFE [6]:

$$SFE\,(mJ/m^2) = -53 + 6.2 \times Ni + 0.7 \times Cr + 3.2 \times Mn + 9.3 \times Mo \quad (3)$$

where the alloying elements are in weight percentage. M_s, M_{d30}, and the SFE values calculated from Eqs. (1)–(3) are reported in Table 2.

2.2. Tensile experiments

Tensile specimens were machined from the 304 sheet with a dog-bone geometry with dimensions of 0.6 mm thickness, 6.35 mm gage width and a 31.75 mm gage length. Uniaxial tensile tests were conducted at 25 °C under a strain rate of $10^{-3}\,s^{-1}$ until rupture in an Instron 5984 machine in laboratory air. The fractured area was examined under TEM. Three tensile samples were interrupted after reaching a strain of 7%, 18% and 30% respectively with the goal of investigating the intermediate microstructure.

In-situ tensile tests were conducted at 25 °C using a special straining-stage with the goal of capturing on video the nucleation process of the martensitic phase transformation as well as the mechanisms as it develops under deformation. The samples for the in-situ experiments had dimensions of 2.5 mm × 11.43 mm. The samples where thinned down to 100 µm and electropolished in the middle section with a solution of 5% perchloric acid and 95% methanol solution. During the in-situ experiments, the observations in Bright Field and Dark Field imaging modes were recorded as videos. Adobe Premier Pro CS4 was used to extract frames from the videos.

3. Results and discussion

3.1. Stress strain curve

The engineering stress-strain (S-S) curve of the 304 SS sheet at 25 °C showed a sigmoidal shape as seen on Fig. 3. The inflection point in the curve marks an acceleration of the strain hardening rate (typical of

Table 2
Ms, M_{d30} and SFE of materials.

	Ms (°C)	M_{d30} (°C)	SFE (mJ/m^{-2})
304 sheet	−69.6	32.1	12.6
304 L bar	−139.2	14.6	18.3