

## The study of TRIP effect in an austenitic stainless steel AISI 304

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### ABSTRACT

**Purpose:** This study investigated the deformation effect in variation of  $\alpha'$ -martensite volume fraction and its microstructural characteristics in an austenitic stainless steel AISI 304.

**Design/methodology/approach:** The study was fulfilled through magnetic analysis with Vibrating Sample Magnetometer (VSM) and transmission electron microscopy (TEM).

**Findings:** The presence of austenite,  $\epsilon$ -martensite and  $\alpha'$ , and that the nucleation of  $\epsilon$ -martensite occurs in stacking faults, grain boundaries and accumulated dislocations in grain boundaries. Already  $\alpha'$ -martensite nucleates in stacking faults, twinning edges, deformation bands and mechanical twinning. In addition, the magnetic martensite takes shape directly from the austenite.

**Research limitations/implications:** A suggestion for future research is to increase the number of samples for deformations larger than 0.30 equivalent strain, due to the significative percentage of  $\alpha'$ -martensite from this strain.

**Practical implications:** A crescent parabolic behavior in relation between percentage of  $\alpha'$ -martensite and equivalent deformation was observed by VSM. The increase of equivalent deformation increases the austenite transformation in  $\alpha'$ -martensite.

**Originality/value:** The behavior in relation between percentage of  $\alpha'$ -martensite and equivalent deformation, and the study of  $\alpha'$ -martensite nucleation.

**Keywords:** AISI 304; Austenitic stainless steel; TRIP effect; Martensite, Formability

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### PROPERTIES

## 1. Introduction

Metastable austenitic stainless steels are susceptible to martensitic transformations induced by deformation, which changes their mechanical properties. The formation of martensite during deformation is one of the most responsible

factors by its high conformability. This plasticity increase of the material due to austenite transformation in martensite is named TRIP effect (Transformation Induced Plasticity). In the metastable austenitic stainless steels can constitute  $\epsilon$ -martensite, a paramagnetic phase (HC) and  $\alpha'$ -martensite, a ferromagnetic phase (CCC), induced by deformation [1].

According to Petit [2], the nucleation of  $\alpha'$ -martensite occurs in the  $\varepsilon$ -martensite interface with the  $\gamma$ -austenite. The volume fraction of martensites can be controlled through the steel chemical composition, of plastic strain quantity and temperature [3 - 5]. The chemical composition and temperature also affect the stacking fault energy (EFE). The lower the stacking faults energy, the higher the exponential value of hardening and the higher the susceptibility to martensite formation [6, 7]. Murr et al [8] noticed that the more intersections between shear bands (stacking faults, twinning and  $\varepsilon$ -martensite), the higher  $\alpha'$ -martensite volume fraction. Therefore, this work had aimed to study the deformation effect in the  $\alpha'$ -martensite volume fraction in an austenitic stainless steel AISI 304, through magnetic analysis and to identify, through microstructural analysis, the present phases in the different conditions of deformation and the nucleation sites of  $\varepsilon$ -martensite and  $\alpha'$ .

## 2. Experimental Procedure

### 2.1. Material

Steel sheet used in this study had 0.5 mm thickness and 1.2 m width whose composition is shown in Table 1.

### 2.2. Nakazima Test

The Nakazima test was performed according to ASTM E2218-02. In this work was used the  $\beta = 1$  way of deformation, where:  $\beta = \varepsilon_2 / \varepsilon_1$  obtained by Gilapa et al [9]. Next, the equivalent deformations were calculated using the equation 1.

$$\varepsilon_{eq} = \sqrt{\frac{2}{9} \{(\varepsilon_1 - \varepsilon_2)^2 + (\varepsilon_2 - \varepsilon_3)^2 + (\varepsilon_3 - \varepsilon_1)^2\}} \quad (1)$$

### 2.3. Magnetic measurements

The tests were accomplished in Magnetic Materials Characterization Lab (UFSC) in the Vibrating Sample Magnetometer (VSM) – EV9 model - Microsense manufacturer. The samples obtained from Nakazima test were cleaned with alcohol and set (one sample per measurement) – in a glass rod laboratory apparatus with spherical and perpendicular tip 8mm - with silicone and wrapped up with white tape thread sealing. Next, they were inserted into the VSM equipment and subjected to maximum

inductor magnetic field of 2,1T, aiming to get magnetization of saturation for each sample. To determination of  $\alpha'$ -martensite volume fraction,  $V_{\alpha'}$  (%), from the results obtained in the magnetic characterization was used the equation (2) suggested by Shimozono et al [10]:

$$V_{\alpha'}(\%) = (4 \cdot \pi \cdot \sigma'_s) \cdot 100 / 10^4 \sigma_s \quad (2)$$

Where:  $\sigma'_s$  is the magnetic saturation of the tested sample in the VSM and  $\sigma_s$  is the magnetic saturation of the sample considering its total transformation to  $\alpha'$ -martensite.  $\sigma_s$  was calculated using the (3) equation obtained from Slater-Pauling curve [10].

$$\sigma_s = \{2,2 \cdot (1 - x - y) + 0,6x\} \cdot 1.003 \quad (3)$$

Where: x and y are the molar fractions for Ni and Cr, respectively.

## 2.4. Characterization by Transmission Electron Microscope (TEM)

Two samples were selected that showed 0.18 and 0.28 values of equivalent deformation, in other words, samples that showed different volume fractions of  $\alpha'$ -martensite. The samples were transformed from 6 mm in diameter and 0.5 mm thickness to samples of 3 mm and thickness of 80  $\mu$ m approximately. Samples were manually sanded using a "disc grinder", in other words, a dresser where fits the sample and makes the trimming of the same, having the reduction control in thickness through a micrometer. The sample was trimmed with 2000-mesh sandpaper until the thickness of 80  $\mu$ m and further polished with 1  $\mu$ m alumina slurry on both sides. Finally, the samples were electrochemically polished using a twin jet electro-polishing (Tenupol) system. The electrolyte used was 1: 3 nitric acid ( $\text{HNO}_3$ ) volume and methanol ( $\text{CH}_3 \text{OH}$ ) with a voltage of 15V. The samples, after being removed from the electrolyte, were washed in distilled water.

## 3. Results

### 3.1. Magnetic Analysis

Figure 1 shows the percentage variation of  $\alpha'$ -martensite in function of equivalent deformation in the 304 austenitic stainless steel using the magnetic measurement method VSM. It can be noticed that the percentage of  $\alpha'$ -martensite increases with the equivalent deformation, in other words, there is an austenite transformation in martensite during deformation.

Table 1.

Chemical composition of AISI 304 austenitic stainless steel used

Steel	C	Mn	Si	P	S	Cr	Ni	Mo	Al	Cu	Co	V	Nb	Ti	N <sub>2</sub>
AISI 304	0.063	1.007	0.48	0.03	0.03	18.270	8.1	0.057	0.0029	0.095	0.048	0.046	0.011	0.0065	0.0531

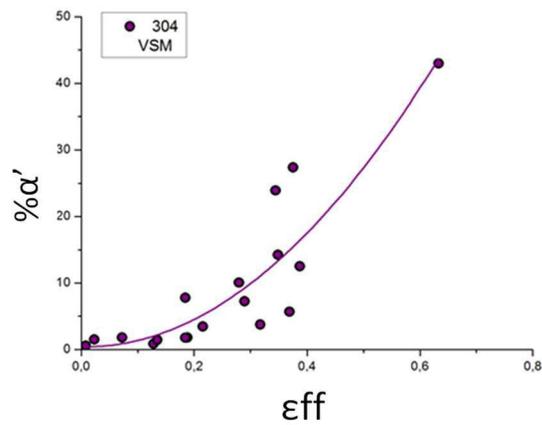


Fig. 1. Percentage variation of  $\alpha'$ -martensite in function of equivalent deformation of 304 austenitic stainless steel obtained by VSM

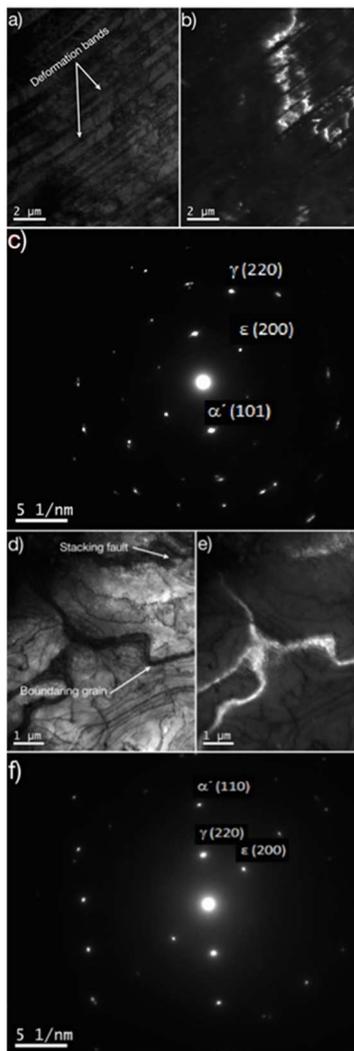


Fig. 2. Steel microstructure of AISI 304 with deformation of 0.28. Bright field (a) and (d), dark field of  $\alpha'$ -martensite (b) and (c) its diffraction matrix. Dark field of  $\epsilon$ -martensite (e) and its diffraction matrix (f).

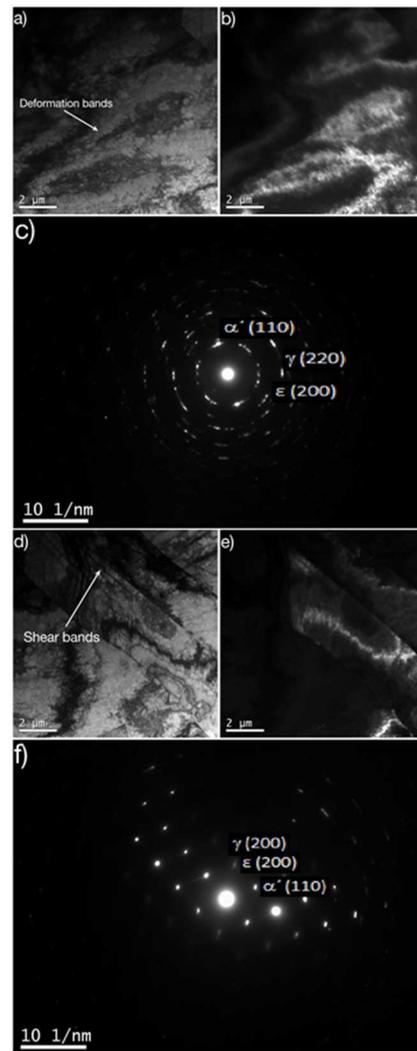


Fig. 3. Steel microstructure of AISI 304 with deformation of 0.18. Bright field, Fig. 3 (a) and (d). Dark field of  $\alpha'$ -martensite (b) and (c) its diffraction matrix. Dark field of  $\epsilon$ -martensite (e) and its diffraction matrix (f).

### 3.2. Transmission Electron Microscope (TEM)

Figure 2 shows the microstructure of AISI 304 steel with equivalent deformation of 0.28. It can be observed in Fig. 2 (a and d) deformation bands, stacking faults, dislocations and shear bands. The dark field of diffraction spot (202) of  $\alpha'$ -martensite (Fig. 2 b) shows its formation in deformation bands and the mechanical twinning. The  $\varepsilon$ -martensite formation can be observed in the stacking faults, grain boundaries of the dark field image (Fig. 2 e), obtained from the diffraction spot (200) of its diffraction matrix (Fig. 2 f). Figure 3 shows the bright field, dark field and the 304 steel electrons diffraction with equivalent deformation of 0.18. It can realize the presence of deformation bands, shear bands, deformation twinning and dislocations (Fig. 3 a and d). The dark field of  $\alpha'$ -martensite (Fig. 3 b), indicates its nucleation in dislocations intersection and in mechanical twinning contour. Already the  $\varepsilon$ -martensite nucleates in deformation bands and inside the twinning, as it can be seen in its dark field (Fig. 3 e). The dark fields of  $\alpha'$ -martensite and  $\varepsilon$ -martensite were obtained from the (110) and (200) planes, respectively, from their diffraction matrix (Fig. 3 c and f).

The obtained results indicate that with low deformation the  $\varepsilon$ -martensite is formed in stacking faults and in the accumulated dislocations in grain boundaries and with greater deformation, the  $\alpha'$ -martensite nucleates in stacking faults. The nucleation of  $\alpha'$ -martensite in these sites were also observed by various authors [7, 11 - 14]. In metastable steels, the formation sequence of martensite induced by deformation is reported as austenite ( $\gamma$ )  $\rightarrow$  martensite ( $\varepsilon$ )  $\rightarrow$  martensite ( $\alpha'$ ) [1], however, as it can be seen in the analysis by transmission electron microscopy, the magnetic martensite takes shape directly from the austenite, in other words:  $\gamma \rightarrow \alpha'$ . The same sequence of formation was also observed by Gilapa et al. [9].

### 4. Conclusions

The magnetic measures have showed that there is an increasing parabolic behavior in the relation between percentage of  $\alpha'$ -martensite and equivalent deformation, in other words, with the increasing of equivalent deformation there is an increasing in the austenite transformation in  $\alpha'$ -martensite.

The analysis accomplished by transmission electronic microscope (TEM) have showed the presence of austenite,  $\varepsilon$ -martensite and  $\alpha'$ , and shows that the  $\varepsilon$ -martensite nucleation occurs in the stacking faults, grain boundaries and in the accumulated dislocations in the grain boundaries. Already  $\alpha'$ -martensite nucleates in stacking faults, twinning edges,

deformation bands and mechanical twinning. As it can be seen in the analysis by transmission electron microscopy, the magnetic martensite takes shape directly from the austenite.

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