

## The Influence of Sample Preparation on the Quantitative Analysis of the Volume Fraction of Martensite Formed in a 304L Trip Steel

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The metastable 304L austenitic stainless steels are susceptible to phase transformation induced by plastic deformation. The traditional metallographic preparation, via mechanical methods, introduces superficial plastic deformation from grinding and polishing. It is important to evaluate this effect on the results of quantitative analysis of martensite formed in 304L steels and similar alloys. In this work, phase transformation was induced by a tensile test at room temperature with two different plastic deformation rates in order to provide distinct structural conditions for the proposed analysis. X-ray Diffraction (XRD) and Ferritescopy were used to determine the volume fraction of martensite in samples with and without metallographic preparation. The results show that the analyses quantitative were not affected by metallographic preparation.

**Keywords:** TRIP effect, XRD analysis, ferritescopy probe, metallographic preparation

### 1. Introduction

Metastable austenitic steels, including the 304L steel, are widely used in various applications due to characteristics such as high formability and corrosion resistance, excellent mechanical strength and ductility and good weldability. These materials have low yield strength, but deformation-induced phase transformation may introduce a significant volume fraction of martensite, which is more resistant than austenite. Therefore, improvement of the mechanical properties of 304L steel can be achieved through the TRIP effect (Transformation Induced Plasticity).

The austenite-martensite transformation ( $\gamma \rightarrow \alpha'$ ) induced by plastic deformation occurs in the range between slightly above room temperature and cryogenic temperatures<sup>1,2</sup>. External stress has the effect of increasing the critical ( $M_s$ ) temperature<sup>2</sup>. The temperature range where this type of transformation occurs is between ( $M_s$ ) and ( $M_d$ ), where austenite is thermodynamically unstable in presence of stress<sup>1</sup>. This reaction has athermal and adifusional characteristics, and the applied mechanical force increases the Gibbs free energy.

The parameters that affect this kind of phase transformation are temperature, chemical composition, strain rate, and strain level and the kind of mechanical stress<sup>3</sup>.

Because phase transformation is induced by plastic deformation, in this article was also addressed the influence of metallographic preparation in quantitative analysis.

Several methods are used to evaluate the volume fraction of martensite. Two of the commonest are magnetic analysis, using a ferritescope probe and X-Ray Diffraction. Magnetic analysis is based on the fact that the martensitic phase be strongly magnetic while the austenite phase is

paramagnetic<sup>4</sup>. In this method, a coil is used to apply to the sample an alternate magnetic field that interacts with the martensite grains, inducing a voltage proportional to the volume fraction of martensite in a second coil. The final result takes into account correction factors as shown in the Equation 1<sup>5</sup>.

$$Fe_w = Fe_m f \quad (1)$$

where  $Fe_w$  is the real volume fraction of the ferromagnetic phase,  $Fe_m$  is the result of the experiment and  $f$  is a correction factor for errors due to curvature, thickness of the material and surface roughness.

Analysis of XRD patterns, using standard refinement methods, yields meaningful volume fraction data because there is a significant difference between the crystal structures of the phases involved (austenite and martensite)<sup>6</sup>. X-ray diffraction occurs only to the next material surface with a depth of only a few micrometers which depends on the radiation used, which is 6.2  $\mu\text{m}$  for  $\text{CoK}_\alpha$ <sup>[4]</sup>. Because of this, it is called superficial analysis when compared to other techniques such as neutron diffraction and gamma radiation. Rietveld refinement is based on achieving a fit between the theoretical diffraction pattern calculated from crystallographic information and the diffraction pattern measured experimentally using the mathematical method of least squares. The volume fraction after refinement is estimated from the area under the diffraction peaks. The Equation 2 calculates the quality of the fit (GOF) which indicates how the analysis is satisfactory.  $R_{wp}$  is a parameter that best shows the evolution of refinement and  $R_{exp}$  refers to the quality of the diffraction pattern<sup>7</sup>.

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$$GOF = \sqrt{\frac{R_{wp}}{R_{exp}}} \quad (2)$$

This study aimed to evaluate the influence of metallographic preparation in quantitative analysis of martensite formed in 304L TRIP steel, subjected to tensile tests for two different plastic deformation rates in order to provide distinct structural conditions for the study.

## 2. Experimental Procedure

The material used in this study consisted of 304L austenitic stainless steel sheets. According to supplier, this material was hot and cold rolled to a final thickness of 1.0 mm and annealed at 1060 °C. At end of processing, the material was submitted to one step of skin pass mill. Its chemical composition is shown in Table 1.

Uniaxial tensile tests were carried out in a first group of samples (IN-L2 to IN-L11) at room temperature (close to 20 °C) using two different strain rates ( $5.55 \times 10^{-4}$  and  $5.55 \times 10^{-3} \text{ s}^{-1}$ ) with strain speeds of 30 and 3 mm/min, respectively. Plastic deformation was defined as the percentage of stop condition intervals between values of the yield strength (YS) and the tensile strength (TS) (10, 25, 50, 75 and 90%), according to Equation 3.

$$\text{Stop Condition (X\%)} = YS + [(TS - YS) \cdot X] \quad (3)$$

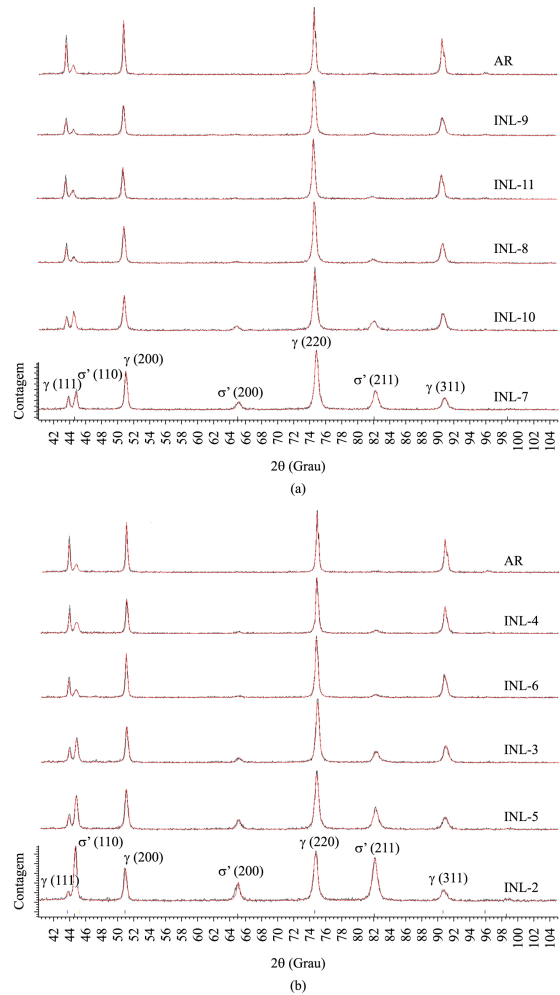
where X the percentage of the stop condition between YS and TS (0.10; 0.25; 0.50; 0.75 and 0.90), YS and TS are yield strength and to tensile strength, respectively.

Tensile tests were also performed in a second group of samples (IN-L12 and IN-L13) in order to investigate the influence of sample preparation on the measurements of phase transformation. The conditions chosen were 70 and 95% deformation, calculated according to Equation 3, and a strain rate of  $1.00 \times 10^{-2} \text{ s}^{-1}$  with a speed of 30 mm/min at room temperature (close to 22 °C). The rate of plastic deformation differed from the first group of samples because we chose to keep the same strain rate and use different gauge lengths for in the two essays. The gauge length was 90 mm for the first group and 50 mm for the second group.

The samples were ground until grit sizes of 1200 and 2000 mesh and then polished with diamond of 6, 3, 1 and 0.25  $\mu\text{m}$ . The polishing was manually done with speed control. During both grinding and polishing was applied the least force that was possible in order to ensure that the sample surface layer, deformed in grinding step, were mainly removed in the polishing, in an attempting to non-insert a surface layer of martensite. The lubrication/cooling during the polishing was performed with hydrated ethyl alcohol.

The volume fraction of martensite was estimated using ferritescopy and XRD. Ferritescopy measurements were performed using a Fischer Ferritescope FMP30. Before the measurements, the instrument was calibrated with ferrite samples the even can detect ferromagnetic phases in the range of 0.1 to 80%. The tests were performed with five measurements at six different points of each sample flat section totaling 30 measurements, the method requires at least five equidistant measurements.

Diffraction measurements were performed using a PANalytical diffractometer X'Pert PRO MPD. The measurement parameters were: initial and final  $2\theta$  angles of 40° and 105°, respectively, unbound  $\theta/2\theta$ , step 0.05° and time by step of



**Figure 1.** Evolution of martensitic transformation in different stop conditions with strain rates (a)  $5.55 \times 10^{-3} \text{ s}^{-1}$  (b)  $5.55 \times 10^{-4} \text{ s}^{-1}$ . Samples with grinding/polishing.

**Table 1.** Chemical composition of the material.

Element (weight, %)								
C	Mn	Si	P	S	Cr	Ni	Mo	Al
0.018	1.2693	0.4786	0.0303	0.0015	18.3639	8.0221	0.0261	0.0032
Cu	Co	V	Nb	Pb	B	Ti	Sn	W
0.0428	0.1015	0.0418	0.0071	0.001	0.006	0.0018	0.0044	0.0146

50 s, X'Celerator detector, copper tube ( $\text{Cu K}_\alpha$  1.540Å) and nickel filter. The diffraction peaks associated with austenitic (fcc) planes (111), (200), (220) and (311) and martensitic (bcc) planes (110), (200) and (211) were analyzed (Figure 1). After obtaining the XRD patterns, the Rietveld refinement process was performed in order to quantify the transformed phase, using the TOPAS academic software version 4.1. In the Rietveld refinement the fit between experimental and calculated peak, was performed using crystallographic information. After refinement, the results were converted to volume fraction for a comparison with ferritescopy data according to Equation 4.

$$\%V\alpha = \frac{\frac{W\alpha}{\rho\alpha}}{\frac{W\alpha}{\rho\alpha} + \frac{W\gamma}{\rho\gamma}} \times 100 \quad (4)$$

where  $V$  e  $W$  are volume and mass fractions, respectively,  $\rho$  is density and  $\alpha$  and  $\gamma$  mean martensite and austenite, respectively.

The hardness tests were performed in microdurometer Buehler - Micromet 2003 with a maximum load of 220 g, using a pyramidal diamond indenter with a square base and 15 s of creep time.

### 3. Results and Discussion

The ferritescopy results showed that phase transformation increases with plastic deformation (Figure 2). Samples with the largest volume fraction of  $\alpha'$  were those deformed at the lower strain rate ( $5.55 \times 10^{-4} \text{ s}^{-1}$ ). This is in agreement with the work of Arpan et al.<sup>8</sup>, and is attributed to the fact that a low strain rate leads to a smaller increase in the internal temperature during deformation. The large amount of phase transformation was also favored by a nickel content close to 8%, since Ni addition decreases temperatures  $M_s$  and  $M_d$ .

The XRD results are shown in (Figure 3). One can see that samples deformed at the smaller strain rate ( $5.55 \times 10^{-4} \text{ s}^{-1}$ ) had a larger volume fraction of martensite than samples

deformed at the larger strain rate ( $5.55 \times 10^{-3} \text{ s}^{-1}$ ). These results are consistent with those obtained by ferritescopy (Figure 2).

The evolution of the phase transformation evaluated by Rietveld method, referring to Figures 1 and 2 is shown from the XRD patterns of each of the samples, after the metallographic preparation. As can be seen, from the stop condition of 75% occurred a considerable increase in the peak intensity of  $\alpha'$  ((110), (200) and (211)) of the deformed samples  $5.55 \times 10^{-3} \text{ s}^{-1}$  (Figure 1a). Since the XRD patterns of (Figure 1b) show that as the stop condition 50% of the deformed samples  $5.55 \times 10^{-4} \text{ s}^{-1}$  there is an increase in the intensity of the peaks  $\alpha'$  ((200) and (211)), and from 75% stop condition the intensity of these peaks become more pronounced including the peak (110).

Figure 4 shows the results of the second group of the tensile test specimens with strain rate from  $1.00 \times 10^{-2} \text{ s}^{-1}$ . These samples were analyzed by XRD and ferritescopy with and without metallographic preparation, whose objective was to evaluate the possible influence of mechanical preparation in quantitative analysis. The ferritescopy and XRD results show a smaller volume fraction of martensite after metallographic preparation. This shows that the metallographic preparation did not induce phase transformation and showed to be efficient to reduce the fraction of martensite introduced.

Both ferritescopy and XRD were able to detect the increase in  $\alpha'$  volume fraction induced by plastic deformation (Figures 2 and 3). The estimated standard deviation was between 0.02 and 0.48 for ferritescopy and the goodness of fit (GOF) was between 1.228 and 1.378 for XRD. These ranges are considered adequate. According to Talonen et al.<sup>4</sup>, since the XRD technique explores a thin layer (less than 10  $\mu\text{m}$  thick) near the surface of the material, the XRD results cannot be directly compared with techniques involving a larger volume, such as ferritescopy, unless the martensitic phase are homogeneously distributed in the sample. The fraction of martensite measured by ferritescopy was smaller than the fraction measured by XRD. This is attributed to the fact that the XRD analysis has focused on the layer of material that was possibly modified by the final step of crossing the skin.

As can be seen in Figure 5, in the 10% stop condition, the Vickers hardness was larger in the sample deformed at the

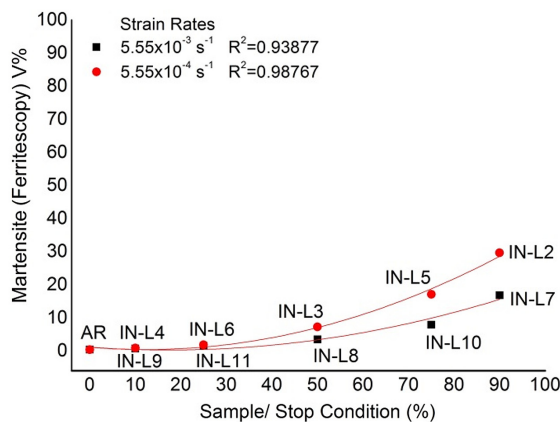


Figure 2. Volume fraction of deformation induced martensite in samples of the first group for two different strain rates, measured by ferritescopy. Samples with grinding/polishing

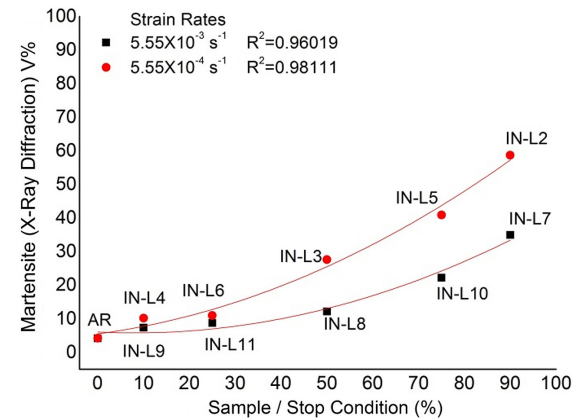
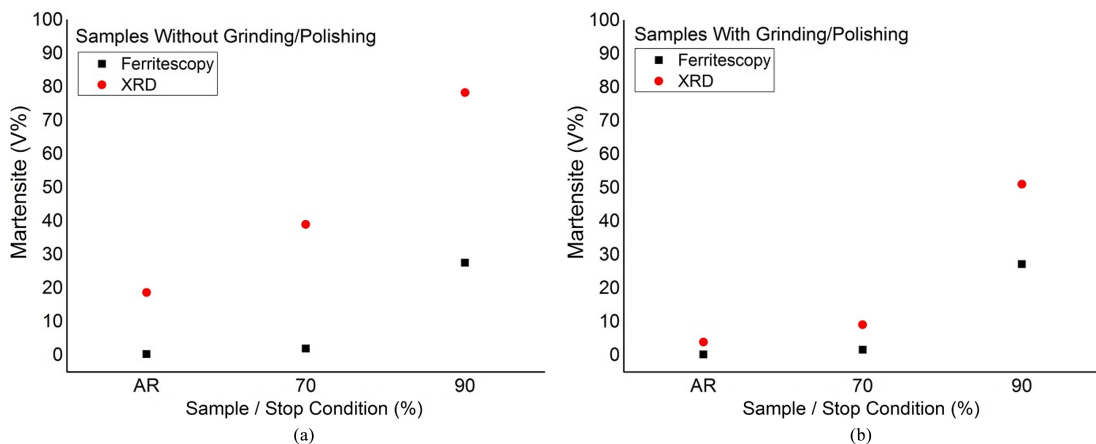
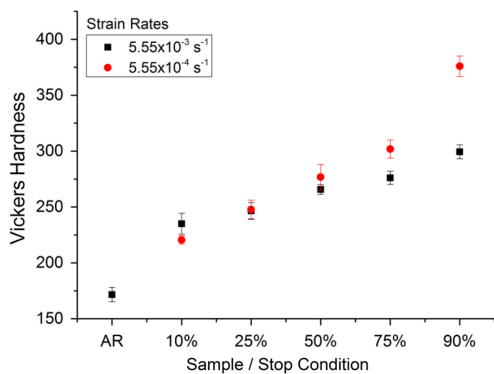


Figure 3. Volume fraction of deformation induced martensite in samples of the first group for two different strain rates, measured by X-ray diffraction. Samples with grinding/polishing..



**Figure 4.** Volume fraction of martensite by ferritescopy and diffraction X-ray in samples without and with metallographic preparation. Plastic strain ratio  $1.00 \times 10^{-2} \text{ s}^{-1}$ .



**Figure 5.** Vickers hardness as a function of stop condition, for samples deformed at two different strain rates.

higher strain rate ( $5.55 \times 10^{-3} \text{ s}^{-1}$ ). In the 25% stop condition, the hardness was approximately equal for both strain rates,  $5.55 \times 10^{-3}$  and  $5.55 \times 10^{-4} \text{ s}^{-1}$ ; and beyond these stop conditions, the hardness was larger in the sample deformed at the lower strain rate. This suggests that austenitic microstructure was reinforced by the martensitic phase, whose volume fraction increases progressively with plastic deformation.

## 4. Conclusions

The sample as received had volume fraction of martensite close to the surface for samples with or without metallographic preparation, which was possibly generated during the final step of skin pass mill.

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The volume fraction of martensite increased with plastic deformation.

Through the metallographic preparation method and the chosen quantitative techniques it was possible to detect microstructural changes.

The ferritescopy and XRD techniques can be used to follow the process of phase transformation, but cannot be directly compared unless it is assumed that the  $\alpha'$  is uniformly distributed over the sample, which is not always the case. Moreover, the techniques covers different parts of the sample; ferritescopy is a volume analysis that involves a larger volume of the material while XRD covers a small surface layer.

Mechanical preparation for metallographic analysis did not induce significant phase transformation and was efficient to reduce the fraction of superficial martensite introduced during step skin pass mill.

Hardness measurements showed that the material became strengthener with the increasing of martensitic fraction and strain hardening.

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