

## Determination of phases and residual stresses after martensitic transformation induced by rolling in 304L stainless steel

### Deteminação de fases e tensão residual após transformação martensítica induzida por laminação em aço inoxidável 304L

Juciane Maria Alves<sup>1,2</sup>, Luiz Paulo Brandao<sup>1</sup>,  
Andersan dos Santos Paula<sup>1</sup>

<sup>1</sup> Military Institute Engineering, Materials Engineering Section, Postgraduate Program in Materials Science, Rio de Janeiro, Rio de Janeiro, Brazil.

<sup>2</sup> Brazilian Center for Research in Physics, Laboratory of Applied Magnetism, Rio de Janeiro, Rio de Janeiro, Brazil.  
e-mail: juciane\_alves\_rj@yahoo.com.br, brandao@ime.eb.br, andersan@ime.eb.br

---

#### ABSTRACT

The 304L austenitic stainless steel is susceptible to deformation induced martensitic transformation. This phase transformation depends on the temperature as well as on the mode, rate and level of deformation. In this work the phases and residual stresses of a 304L TRIP steel where martensitic transformation was induced by cold rolling were investigated by X-ray diffraction XRD. The analyses were performed for different sample thicknesses. The results showed that the phase composition and the residual stresses are strongly dependent on sample thickness. All samples showed a compressive residual stress.

**Keywords:** 304L Stainless Steel, Martensitic Transformation, Residual Stress, Thickness variation.

---

#### RESUMO

O aço inoxidável austenítico 304L é suscetível à transformação martensítica induzida por deformação. Esta transformação de fases depende da temperatura, assim como do modo, taxa e nível de deformação. Neste trabalho as fases e tensões residuais de um aço TRIP 304L, em que a transformação martensítica foi induzida por laminação a frio, foram investigadas por difração de raios-X (DRX). As análises foram realizadas em diferentes espessuras das amostras. Os resultados mostraram que a composição de fases e as tensões residuais são fortemente dependentes da espessura das amostras. Todas as amostras apresentaram tensão residual compressiva.

**Palavras-chave:** Aço Inoxidável 304L, Transformação Martensítica, Tensão Residual, Variação de Espessura.

---

#### 1. INTRODUCTION

The transformation induced plasticity (TRIP) may occur in a wide temperature range and depends on several other parameters, such as the chemical composition of the alloy and the mode, rate and level of deformation. The products of the phase transformation austenite-martensite, in TRIP steel may be  $\epsilon$  and  $\alpha'$  martensite. The  $\epsilon$ -martensite nucleation occurs in the stacking faults intersections and  $\alpha'$ -martensite nucleation arises in  $\epsilon$ -martensite and the intersections among shear bands [1-3].

The chemical composition of the alloy influences important intrinsic features of phase transformation such as stacking fault energy (SFE), martensite formation starting temperature ( $M_s$ ) and  $M_{d30}$  temperature. The  $M_{d30}$  is the temperature where 30% of the strain induces 50% martensitic formation [4-6]. A lower percentage of alloying elements increases the austenite instability. Thus, the lower the number of elements in a TRIP steel and the lower the temperature during the deformation process, more intensity martensitic transformation [7]. The residual stress fields near dislocations influence the nucleation of martensite. Thus, the austenite-martensite transformation may occur heterogeneously [8].

Residual stresses are those that remain in a sample in the absence of external loads or temperature gradients. The residual stresses induced by austenite-martensite phase transformation arise mainly by volume expansion, elastic incompatibility and plastic mismatch between austenite and martensite [9]. The deformation induced martensitic transformation promotes local deformation fields causing stress variations in both austenite and martensite phases [9]. In general, the residual stresses are consequences of interactions between deformation, temperature and microstructure [9].

The purpose of the present work was to investigate the effect of the amount of thickness reduction on the properties of martensite induced by rolling in the 304L austenitic steel. The article results showed that the residual stress and quantitative phase analysis of the martensitic transformation evolved as a function of rolling reductions. There was variation in martensite content and residual stress gradient in analyses performed in different sample thicknesses. A compressive residual stress state was observed for all sample sections.

## 2. MATERIALS AND METHODS

The material under study consisted in 304L austenitic stainless-steel sheets, received in the condition of hot rolling, with a thickness of 6.37 mm. The chemical composition of the alloy is shown in Table 1.

**Table 1:** Chemical composition of 304L stainless steel (weight %).

C	Mn	Si	Cr	Ni	Mo	N
0.0196	1.262	0.491	18.036	8.084	0.135	0.051

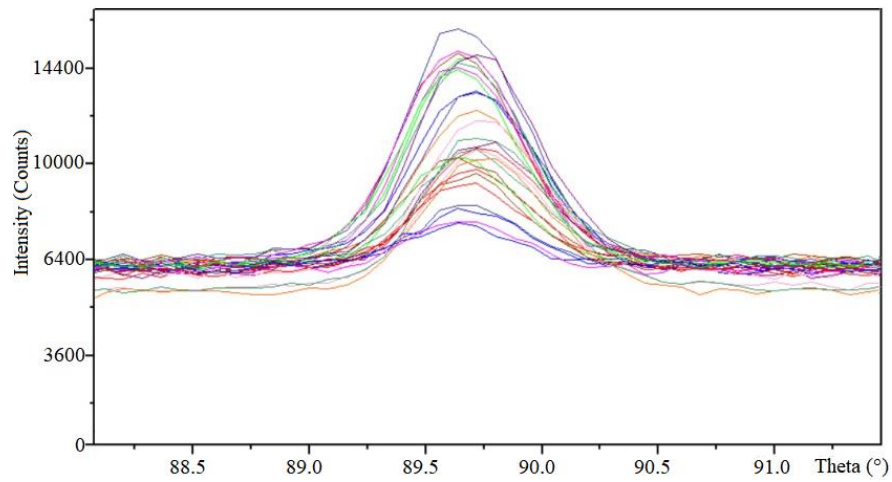
The martensitic transformation was induced by rolling at room temperature (25 °C) with 20% and 80% reductions. For 20% reduction, 2 passes were used, and for 80% reduction, 15 passes were used, with approximately 10% reduction per pass. The deformation average rate was  $9.8 \times 10^{-1} (\pm 1.6)$ .

After rolling, the material was cut into samples with an area of 20 × 20 mm. The samples were ground with grit sizes of 200, 400, 600, 800 and 1200 mesh. Then, electrolytic polishing using a solution of 20% perchloric acid in ethanol. Only the front surface of the sample was placed in contact with the electrolytic solution; an electrical tape was used to cover the sides and the back surface of the sample, since it was observed that these areas, when exposed, decreased the etching speed.

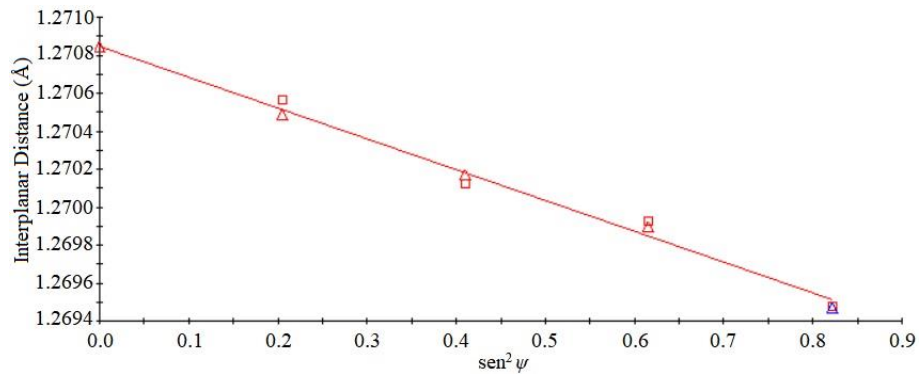
The phase analyses were performed by XRD using a PANalytical X'PERT PRO MRD diffractometer. The measurement parameters were initial and final angles of 45 and 130°, step size of 0.030° and time per step of 200 s. The peaks associated with austenite and martensite were analyzed using ICDD crystallographic data ( $\gamma$ -phase 00-047-1417 and  $\alpha'$ -phase 03-065-7753).

The residual stress measurements were performed by the  $\psi$  geometry method with multidirectional analysis, using several tilt angles in  $\psi$  such as 26.95, 39.86, 51.71, 65.00, 0.00, 26.95, 39.86, 51.71 and 65.00° and, for each  $\psi$ ,  $\phi$  rotations of 0, 45 and 90°. Thus, each measurement was produced through 27 scans resulting from a combination of 9 tilts in  $\psi$  with 3 directions in  $\phi$  for each value of  $\psi$  (Figure 1). These points were used to determine, with good accuracy, the interplanar distance versus  $\sin^2\psi$  graph shown in Figure 2.

Residual stress measurement and phase analysis for austenite and martensite were performed for the sample surface and when the sample thickness was reduced to  $\frac{1}{4}$  and  $\frac{1}{2}$  the initial thickness using mechanical grinding.



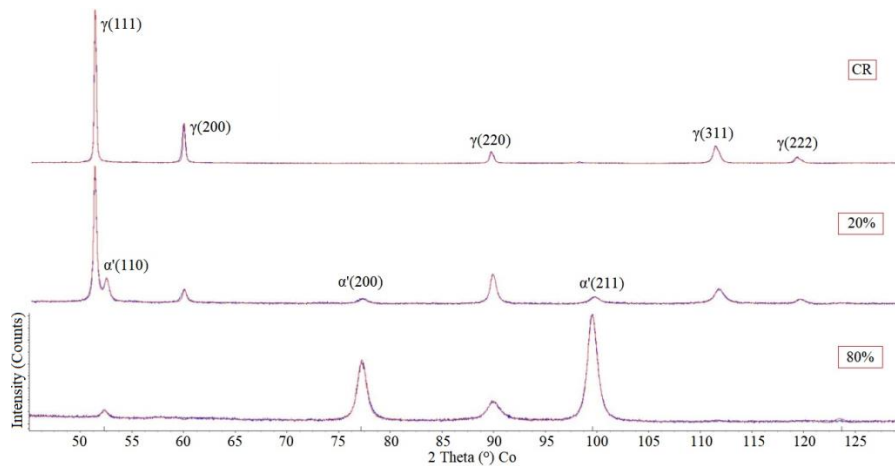
**Figure 1:** Multidirectional analysis of residual stress by XRD (27 scans).



**Figure 2:** Interplanar distance versus  $\text{sen}^2 \psi$ . Squares refer to measurements for psi angles positive and the triangles to the measurements for negative psi angles.

### 3. RESULTS AND DISCUSSION

The diffractograms showed that as the sample thickness was reduced by rolling, the intensity of austenite peaks decreased and the intensity of the martensite peaks increased. The diffraction peaks associated with austenite and martensite were, respectively,  $\gamma$  [(111), (200), (220), (311), (222)] and  $\alpha'$  [(110), (200), (211)], as shown in Figure 3.



**Figure 3:** XRD of rolled samples at room temperature with 20 and 80% reduction.

The quantitative results, obtained by the Rietveld method, showed 35% martensite for 20% of rolling reduction and 96% martensite phase for 80% reduction. The volume fraction of martensite was different at the surface and when the sample thickness was reduced to ¼ and ½ the initial thickness (Table 2). This is probably due to the heterogeneity of martensite nucleation, as reported by FISHER [8]. It was observed that martensite volumetric variation in sample with 20% reduction in thickness was not very significant for the analyzes performed between sample surface and the ¼ thickness (35 and 32% martensite), respectively, but they were meaningful on the comparison to the 24% of volume fraction observed on ½ thickness.

The analysis of the sample with 80% thickness reduction showed 96% martensite on surface and 88% and 79% on ¼ and ½ thickness, respectively. The sample as received (AS) showed no significant martensite variation between plane and half thickness, a martensite percentage obtained was 0.8 and 0.9%, respectively, close to the technique detection limit (Table 2).

Residual stress measurements of austenite and martensite were performed as well. For the sample as received and for a 20% reduction, the volume fraction of martensite is so low that the measurement is assumed to be the residual stress of austenite. For the sample with 80% reduction, the volume fraction of austenite is so low that the measurement is assumed to be the residual stress of martensite (Table 2).

**Table 2:** Phases and residual stress quantification after martensitic transformation of 304L stainless steel.

SAMPLE	VOLUME α'(V%)	RESIDUAL STRESS γ(MPa)	STANDARD DEVIATION (MPa)
AR_Surface	0.8	-184	8
AR_¼	0.8	-153	17
AR_½	0.9	-98	14
SAMPLE	VOLUME α'(V%)	RESIDUAL STRESS γ(MPa)	STANDARD DEVIATION (MPa)
20_Surface	35	-50	23
20_¼	32	-78	22
20_½	24	-113	23
SAMPLE	VOLUME α'(V%)	RESIDUAL STRESS γ(MPa)	STANDARD DEVIATION (MPa)
80_Surface	96	-180	50
80_¼	88	-184	55
80_½	79	-209	59

The residual stress analyses show a compressive stress gradient from the surface to half thickness. The sample as received had a larger residual stress at the surface, while those with 20 and 80% reduction had a larger compressive stress at half thickness. The residual stress of the as received sample is attributed to the previous thermomechanical processing, hot rolling.

In the case of samples submitted to martensitic transformation induction by rolling, with 20 and 80% reductions, a larger compressive residual stress was observed at half thickness. The largest stress (-209 MPa) was observed in sample with highest reduction, 80%, and, consequently, the largest volume fraction of martensite. The residual stresses in rolled samples are due consequences to phase transformation and of the

interaction between stresses arising phase change with those promoted material plastic deformation in the rolling process. It is difficult to separate the two contributions, since the phase transformation occurred by plastic deformation.

#### 4. CONCLUSION

The present results show that martensitic transformation can be achieved by cold rolling in 304L stainless steel. There was a gradient of the volume fraction of martensite from the surface to half sample thickness. There was also a gradient of compressive residual stress from the surface to half sample thickness. The residual stresses are attributed the interaction between phase transformation and plastic deformation in the rolling process.

#### 5. ACKNOWLEDGMENTS

The authors thank Aperam South America for supplying the material, FAPERJ for a Doctorate scholarship and CNPq for a research productivity grant (PQ-2), process 307798 / 2015-1.

#### 6. BIBLIOGRAPHY

- [1] TALONEN, J., HANNINEN H, "Formation of Shear Bands and Strain-Induced Martensite During Plastic Deformation of Metastable Austenitic Stainless Steels", *Acta Materialia*, v. 55, pp. 6108-6118, 2007.
- [2] SHEN, Y.S., LI, X.X., SUN, X., *et al.*, "Twinning and Martensite in A 304 Austenitic Stainless Steel", *Materials Science and Engineering A*, v. 552, pp. 514-522, 2012.
- [3] OKAYASU, M., TOMIDA, S., "Phase Transformation System of Austenitic Stainless Steels Obtained by Permanent Compressive Strain", *Materials Science and Engineering A*, v. 684, pp. 712-725, 2017.
- [4] CINA, B., "Effect of Cold Work on the Gamma-Alpha Transformation in Some Fe-Ni-Cr Alloys", *Journal of the Iron and Steel Institute*, v. 177, pp. 406-422, 1954.
- [5] ANGEL, T., "Formation of Martensite in Austenitic Steels", *Journal of the Iron and Steel Institute*, v. 177, pp. 165-174, 1954.
- [6] HEDAYATI, A., NAJAFIZADEH, A., KERMANPUR, A. *et al.*, "The Effect of Cold Rolling Regime on Microstructure and Mechanical Properties of AISI 304L Stainless Steel", *Journal of Materials Processing Technology*, v. 210, pp. 1017-1022, 2010.
- [7] ANTUNES, A.E.B., ANTUNES, L.M.D., SAMPAIO, M., "Comportamento Plástico no Escoamento de Aços Inoxidáveis Austeníticos Metaestáveis", *Revista Brasileira de Aplicações de Vácuo*, v. 30 n.1-2, pp. 18-23, 2011.
- [8] FISHER, J.C., TURNBULL, D., "Influence of Stress on Martensite Nucleation", *Acta Metallurgica*, v. 1, v.5, pp. 310-314, 1953.
- [9] PAPULA, S., TALONEN, J., HANNINEN, H., "Effect of Residual Stress and Strain-Induced A'-Martensite on Delayed Cracking of Metastable Austenitic Stainless Steels", *Metallurgical and Materials Transactions*, v. 45a, pp. 1238-1246, 2014.

#### ORCID

Juciane Maria Alves <https://orcid.org/0000-0001-7433-8838>  
Luiz Paulo Brandao <https://orcid.org/0000-0002-2623-648X>  
Andersan dos Santos Paula <https://orcid.org/0000-0002-0904-4240>