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QUANTIFICATION OF STRAIN-INDUCED MARTENSITE IN 304L STAINLESS STEEL IN THE PRESENCE OF STRESS CONCENTRATION

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Abstract. *The high corrosion resistance, high mechanical resistance and toughness are some positive factors of the stainless steels. Linked primarily by iron, carbon and chromium, they can also contain other elements such as nickel and molybdenum. Among the various classifications of stainless steels, austenitic steels type will be tackled harder. The addition of nickel as an alloying element in certain quantities, causes the ferritic structure to become into austenitic, generating excellent corrosion resistance, ductility and weldability. Because they contain several advantageous features, the austenitic stainless steels are widely used in various areas, such as in the oil and gas industry, petrochemical, aeronautics, among others. This work will propose the use of 304L austenitic stainless steel as an object of study; specimens with notches were produced and tested in tensile tests in order to observe and measure the transformation of austenite into martensite in a particular region with stress concentrators. The improvement of the mechanical resistance of the material is one of the benefits of this transformation, which is also a motive for more studies and research on this proposed topic.*

Keywords: *Stainless steel, austenitic stainless steel, martensitc transformation, stain-induced martensite.*

1. INTRODUCTION

Stainless steels are iron-carbon alloys with chromium above 10.5% by weight. Other alloying elements comprise material, such as nickel, molybdenum, manganese, silicon, copper, aluminum and nitrogen. (Padilha, 1994) The austenitic series steels have structure obtained due to the addition of elements such as nickel, manganese and nitrogen, which stabilizes the austenite in temperature environment. These steels have CFC structure, are non-magnetic in the annealed condition and can be hardened by cold forming mainly. (Davis, 1994)

The austenitic stainless steels are increasingly being studied because of its good features, such as good resistance, good resistance corrosion, high ductility and excellent machinability. However, the austenitic stainless steels have high ductility, are significantly affected by strain-induced martensitic transformation. Many stainless steels are used in various mechanical processes that result in plastic strain, such as stretching, drawing and bending. Okayasu *et al*, 2013

The martensitic transformation does not happen by diffusion, in which the atoms move in a cooperative and coordinated way by a shear mechanism, and this change brings about a substantial increase in the density of crystal defects, which produces a microstructure with high mechanical strength (Rios, 2007). After cold deformation, approximately 10% of the energy used in the deformation is stored in the material (Padilha, 1994). The transformation martensite in austenitic stainless steel is influenced by the chemical composition of the steel and the variables related to cold working, such as deformation, strain rate, stress state, and deformation temperature (Padilha, 2003)

Dan *et al*, 2007, observed that the gradual transformation of austenite into martensite induced deformation increases the resistance of these steels. Reason that motivates increasing amount of research and utilities on the material. It is already widely used in various sectors, such as: construction, oil industry, pulp and paper, household, industrial, and in aggressive environments in general, as tanks for chlorine reservoir, nuclear waste, chemicals, etc.

This work will propose the study of 304 L austenitic stainless steel. Specimens with notches were produced and tested in tensile tests in order to observe and measure the transformation of austenite into martensite in a particular region with stress concentrators.

2. MATERIALS AND METHODS

The material used in this work, was an austenitic stainless steel 304L provided by APERAM Inox América do Sul S/A in rectangular plates of dimensions 150 X 300 mm, whose chemical composition is shown in Table 1.

Table 1. Chemical composition of 304L austenitic stainless steel (weight percent)

C	Mn	Si	P	S	Cr	Ni	Mo	Al
0.0192	1.2686	0.377	0.0352	0.0026	18.0722	8.0082	0.0958	0.0032
Cu	Co	V	Nb	Pb	B	Ti	Sn	W
0.1557	0.1712	0.0802	0.0056	0.001	0.0006	0.006	0.0062	0.0197

The samples were manufactured in the direction of lamination with two notches and dimensions as shown in Fig. 1 through the wire eletrical discharge machining process (Okayasu *et al*, 2013). The reason the notches is the aim of measuring the martensitic transformation in a region with concentrator tensions.

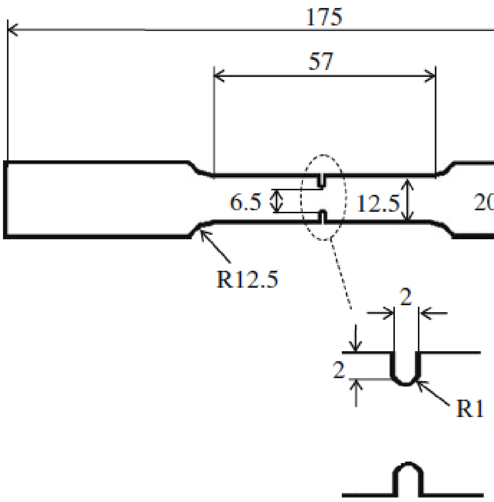

Fig 1. Schematic illustration of the test specimen (adapted of Okayasu *et al*, 2013)

The Fig. 2 shows a photo of specimens used in the tensile test. A specimens without notches was used to obtain the normal properties of the material and to observe the difference in the amount of martensite transformed between the two specimens.

Fig. 2. Specimens used in tensile tests

2.1. Tensile Test

The specimens were tested in tension in EMIC DL60000 machine with capacity up to 600kN. To obtain more precise results of the specimen elongation during the test it was used an extensometer EMIC EE09. The test was performed at room temperature with a speed of 1.71 mm / min. As shown in Fig 3, was fixed to the probe of the feritscope Fischer FMP30 between the two notches of the sample to be measured martensitic transformation during the test, and not only at the end. The test ended after fracture of the specimen, with the data obtained it was possible to obtain a stress x strain curve and also a martensite transformation curve as a function of test time.

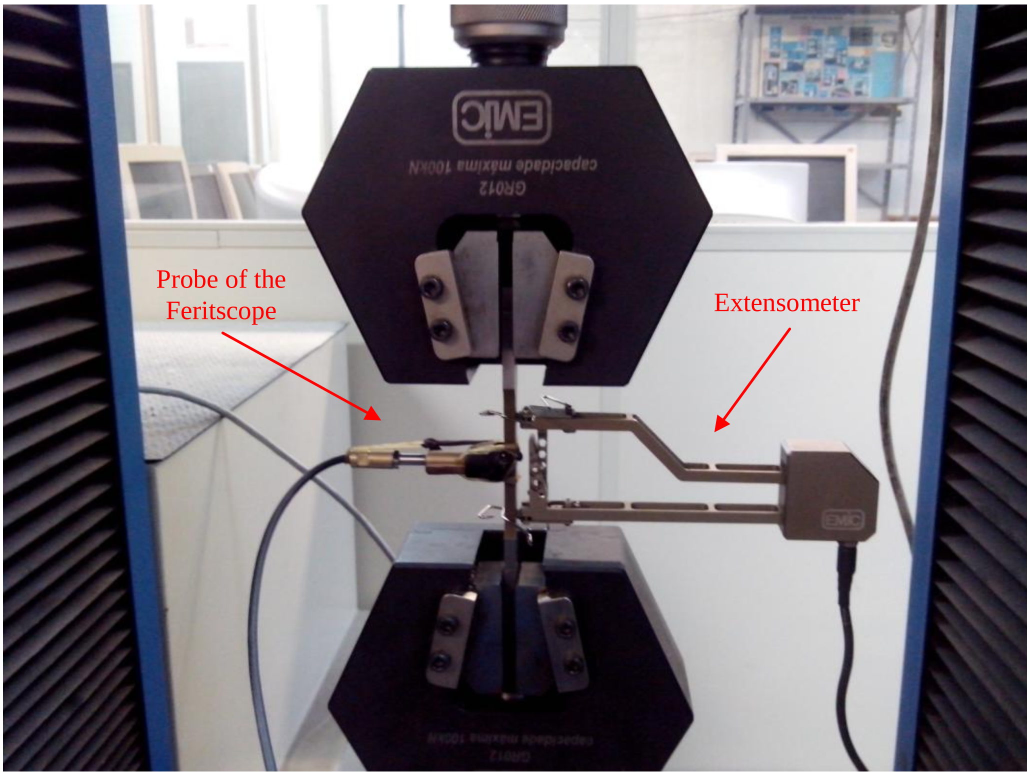

Fig. 3. Machine testing with feritscope and extensometer fixed to specimen.

2.2. Optical Microscopy

After the tensile test, the specimen was cut at a distance of 30 mm of the rupture in order to analyze with the help of a microscope the microstructure. The sample was ground with emery paper to 1200 mesh. After this, the sample was polished in polishing cloths with 1µm and 0.05µm respectively with aqueous alumina 1µm and 0.05µm concerning each cloth and distilled water. Each process step was carried out with due care and cleaning of the sample in order to remove excess residues and no contamination occurs in the next step.

Based on studies Forouzan, *et. al.*, 2010, a mixture of 60 ml nitric acid and 40 ml of distilled water was prepared in order to cause electrolytic etching in the sample to be revealed to the grain boundaries of the material. The sample was submerged 1v for 8 min. After the electrolytic etching, it was allowed to observe through Olympus Optical Microscope BX51M with the help of image capture software Olympus Stream Basic 1.9 the material grain boundaries. To reveal the martensite phase of the material was used etching like a recently work (Talonen 2007), where dilute 0.15g of sodium metabisulfite in 100 ml of distilled water, mixed with 10 ml of hydrochloric acid with 100 ml of distilled water. The sample was submerged in the solution for 160s.

2.3. X-ray Diffraction

The same sample was prepared for the realization of optical microscopy was also used for the realization the x-ray diffraction test. It used the diffractometer Shimadzu model XRD-6000. The incident radiation was Cu Kα type 30 kV voltage and 30 mA current.

3. RESULTS AND DISCUSSION

The tensile tests provided the data that has been generated engineering stress-strain curves as shown in Fig. 4. In Fig. 4 (A) shows the graph specimen without notches. Because of the extensive specimen of stretching, the test was terminated when it reached the extensometer limit. In Fig. 4 (B) shows the graph referring to the specimen with notch where was found some important material properties such as ultimate tensile strength, σ_{UTS} = 800 MPa and the yield strength, $\sigma_{0.2}$ = 510 MPa. These values are equivalent to that particular specimen, as shown in Okayasu *et al*, 2013. For better visualization, the Fig. 5 shows the comparison between samples with and without notch.

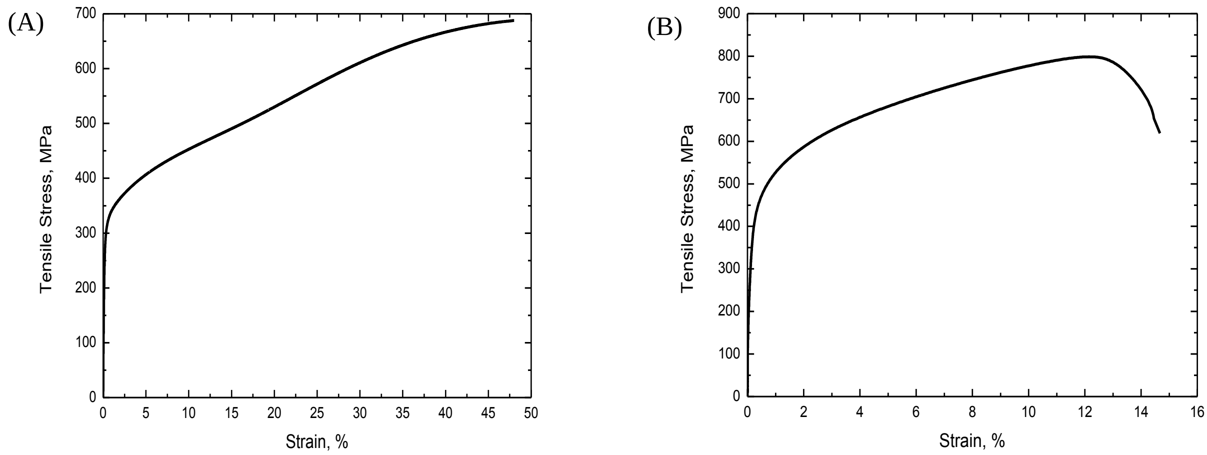

Fig 4. (A) Tensile stress-strain curve for the 304L stainless steel without notches and (B) Tensile stress-strain curve for the 304L stainless steel with notches.

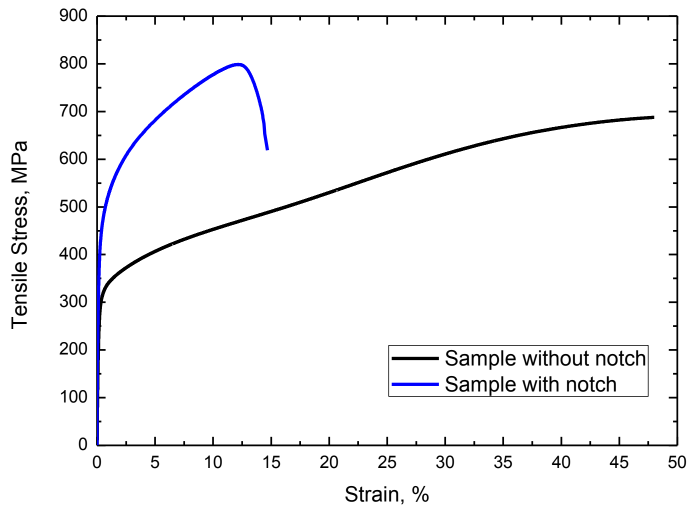

Fig. 5. Tensile stress-strain curve comparison of the material with and without notch.

The specimen before being test showed martensite fraction of less than 1% in all regions. The Fig. 6 shows the growth of martensitic fraction in the sample without notch during the tensile test. It was observed that until the specimen did not suffer plastic deformation, the martensitic transformation is practically zero.

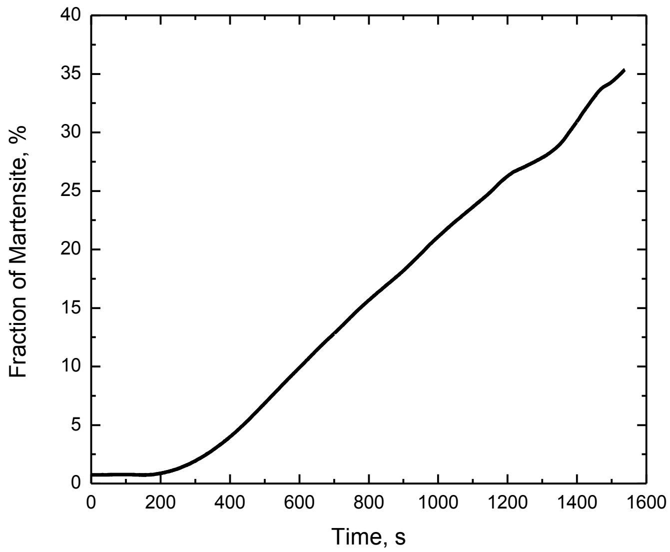

Fig. 6. Fraction of martensite-time curve in the 304L stainless steel (sample without notch)

At the moment of rupture of the specimen, the transformed martensite fraction was approximately 35%, however, measurements made after the test, detected that the martensite fraction increased, to read about 50%. This growth is due to spring back of the material. That generated residual stress and so increased the amount of martensite.

The same test was performed for the sample with notch, as shows the Fig. 7. The test ended after the break the specimen. It was obtained a martensite transformation of approximately 47%. The specimen region where there were no notch, the martensitic transformation was very small, around 1%, reason caused by the concentration of tension between the notches of the specimen. The spring back was not detected in the specimen with notch, that is, the fraction transformed at the moment of break of specimen has not changed.

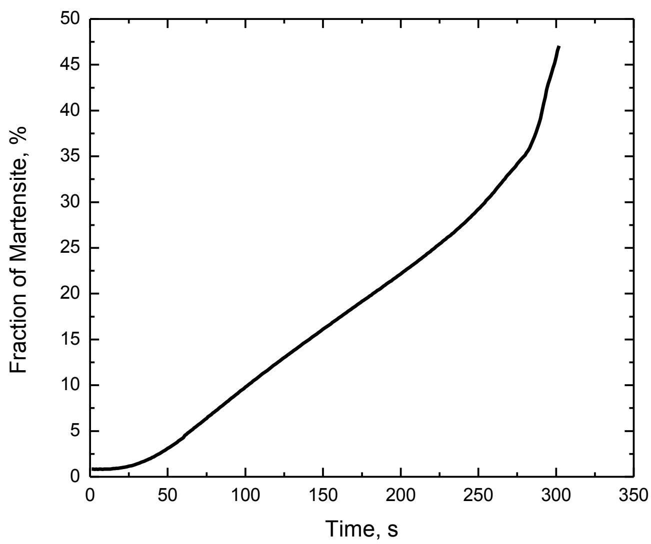

Fig. 7. Fraction of martensite-time curve in the 304L stainless steel (sample with notch)

The Fig. 8 shows a comparison between the results of feritscope of the sample with and without notch.

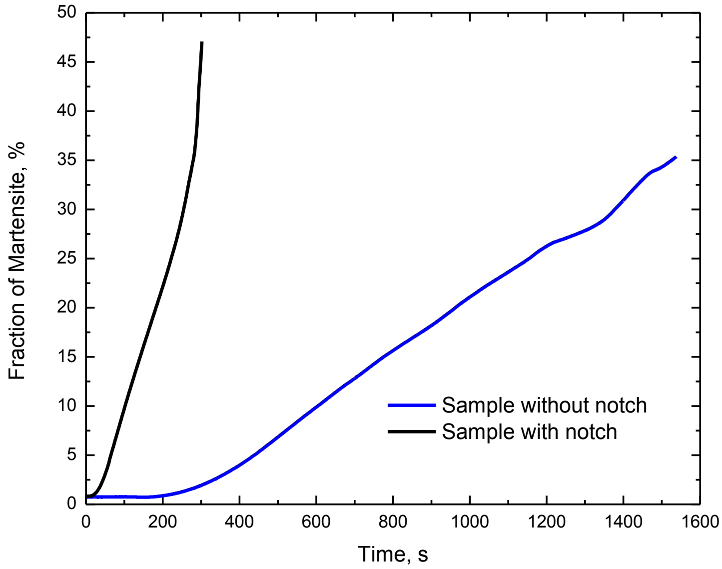

Fig. 8. Fraction of martensite-time curve between the sample with and without notch.

In the notched region, the only method used to quantify the martensitic transformation was the feritscope because the notched region was not possible to ground and polish the sample for quantification were performed by other methods.

With the sample prepared as described in topic 2.2, the microstructural characterization of the material was performed by optical microscopy we can see the material as received and after the tensile test as shown in Fig. 9.

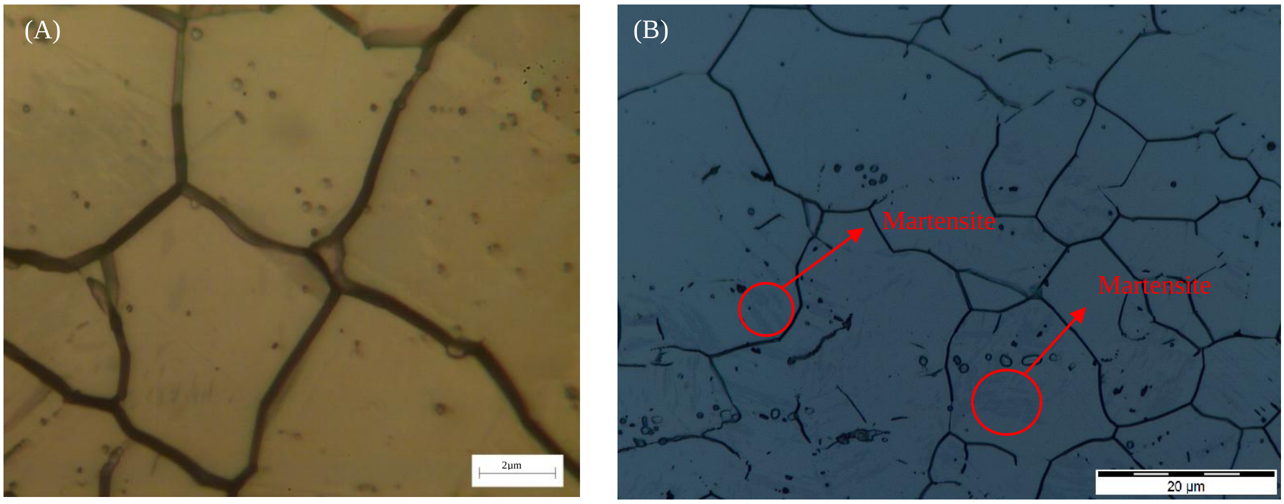

Fig. 9. (A) Microstructure of the material as received (B) Microstructure of the material after tensile test

The Fig. 9 (A) shows the material as received, we observe the grain boundaries and a fully austenitic microstructure. In Fig. 9 (B) already the material tested in tension, we also observed the grain boundaries and the start of martensite formation. In Fig. 10 difratograms for the sample as received and in Fig. 11 after the tensile test are presented. The tests x-rays diffraction had the objective identify the phases present in the conditions and check the evolution of martensitic transformation through the diffraction peaks of each of the phases associated with the orientations and plans of the grain parallel to the plate surface that are consumed (austenite) and formed (martensite).

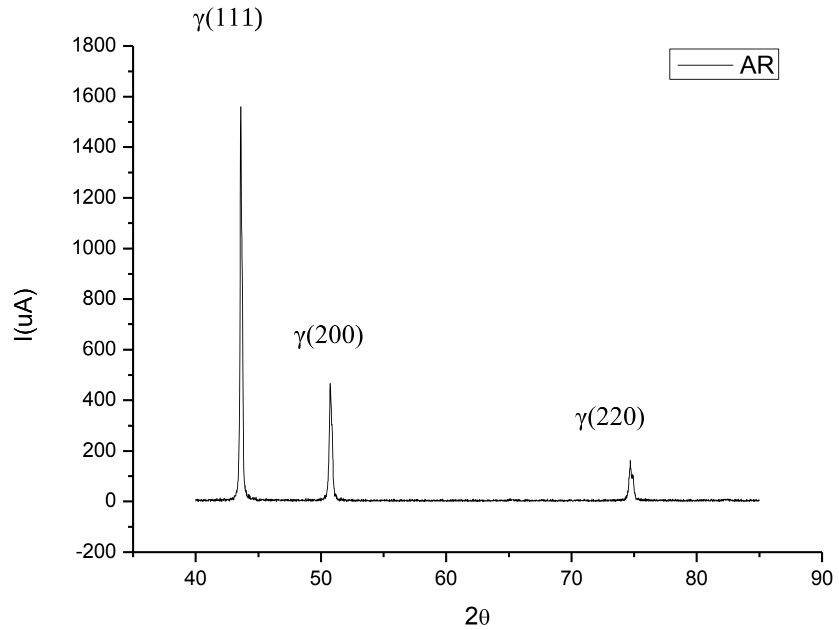

Fig. 10. X-ray diffraction of the material as received

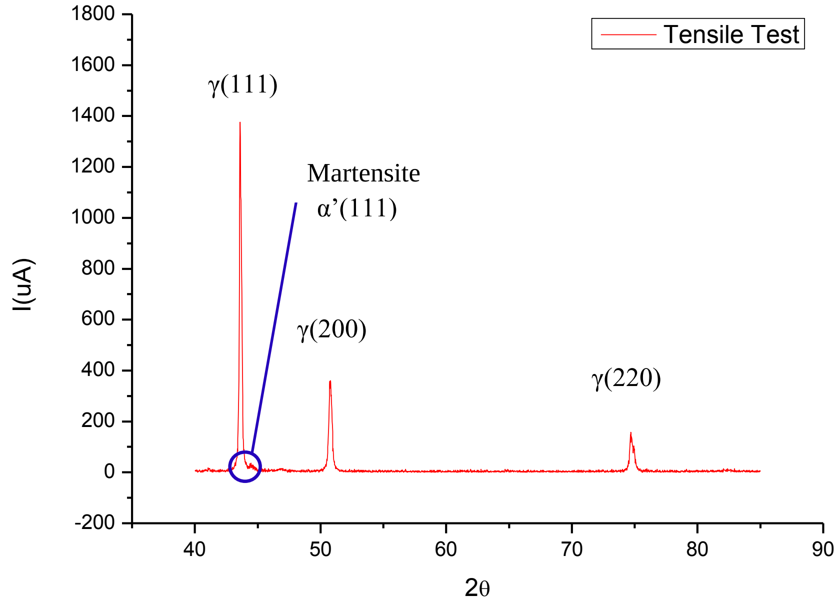

Fig. 11. X-ray diffraction of the material after tensile test

For better visualization this transformation, the Fig. 12 shows a difractogram with a comparison between the material before and after tested in tension. Note that the transformation is small, as shown in the optical microscope images.

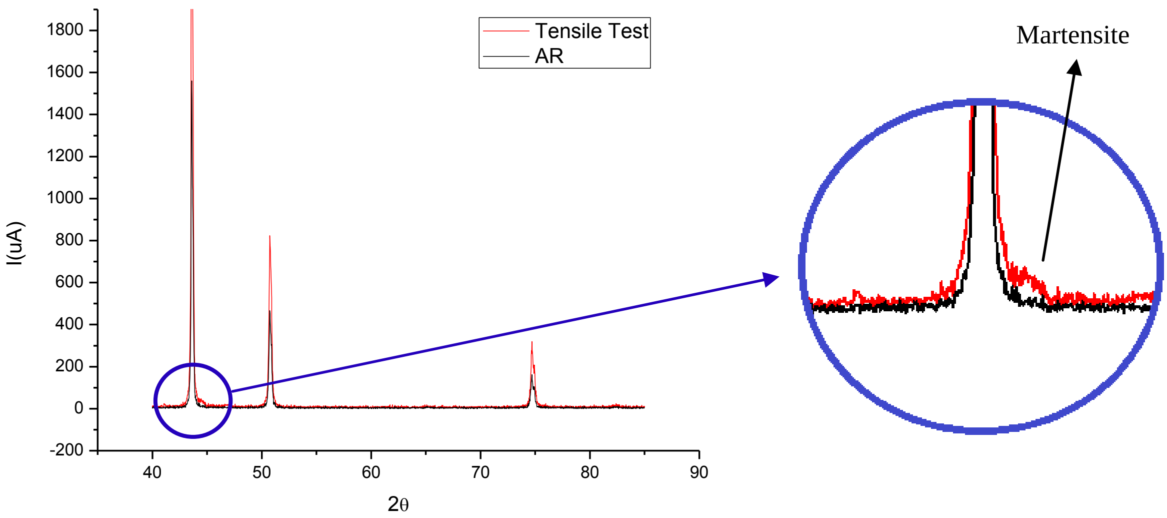

Fig. 12. X-ray diffraction comparison of the material as received and material after the tensile test.

4. CONCLUSIONS

In this work, the martensitic transformation strain-induced was characterized by feritscope methods, optical microscopy and x-ray diffraction. Through the results of each method, we obtained the following conclusions:

1) The assembly of feritscope equipment worked perfectly, and we were able to measure the martensitic transformation during the tensile test.

2) The martensitic transformation increases faster in the presence of the notch, namely in regions with tension concentrators.

3) It was not possible to view the martensitic transformation by optic microscopy in the notch region, but the measurement made 1 mm to notch (without concentrators tension region), showed very little martensitic transformation.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

DAN, W. J., ZHANG, W. G., LI, S. H., LIN, Z. Q., 2007. "A model for strain-induced martensitic transformation of TRIP steel with strain rate". *Computational Material Science*, Vol. 40, p. 101-107.

Davis, J. R., 1994. *ASM Specialty Handbook: Stainless Steel.* Ed. ASM International.

FOROUZAN, F., NAJAFIZADEH, A., KERMANPUR, A., HEDAYATI, A., SURKIALIABAD, R., 2010. "Production of nano/submicron grained AISI 304L stainless steel through the martensite reversion process". *Material Science Engineering A*, Vol. 527, p. 7334-7339.

OKAYASU, M., FUKUI, H., OHFUJI, H., SHIRAISHI, T., 2013. "Strain-induced martensite formation in austenitic stainless steel". *J Mater Sci*, Vol. 48, p. 6157-6166.

Padilha, A. F., Guedes, L. C., 1994. *Aços Inoxidáveis Austeníticos: Microestrutura e Propriedades*. Ed. Hemus, São Paulo.

PADILHA, A. F., PLAUT, R. L., RIOS, P. R., 2003. "Annealing of col-worked austenitic stainless steels". *ISIJ International*, Vol. 43, p. 135-143.

Rios, P. R., Padilha, A. F., 2007. *Transformações de Fase.* Ed. Artliber, São Paulo.

TALONEN, J., 2007. *Effect of strain-induced α'-martensite transformation on mechanical properties of metastable austenitic stainless steels.* Doctorate thesis, Helsinki University of Technology, Helsinki.

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