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STUDY OF THE EFFECT OF MARTENSITIC TRANSFORMATION INDUCED BY PLASTIC DEFORMATION ON THE MECHANICAL PROPERTIES OF THE 304L STAINLESS STEEL.

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Abstract. *The 304L Stainless Steel has been very used in applications in chemical industry and equipments of pressure vessels, and pipelines in oil industry. When deformed, this material can undergo a solid state phase transformation also called transformation induced plasticity (TRIP) that is the transformation: austenite (CFC) → martensite (CCC). The 304L stainless steel is considered a high alloy TRIP steel and the transformation induced by the plastic deformation increase significantly its mechanical strength. In this work is investigated the effects of cold reduction in different amounts on the mechanical properties of the steel. It is going to be used the following techniques: tensile tests, Optical Microscopy, X ray diffraction, dilatometry and hardness tests.*

Keywords: *304L Stainless Steel, TRIP effect, martensitic transformation, α'-martensite, grain size.*

1. INTRODUCTION

The Austenitic Stainless Steels (ASS's) are the stainless steels with the largest production, worldwide. Due to a special combination of properties, as high corrosion resistance in a variety of corrosive environments, an excellent weldability, good toughness (even in low temperatures), good creep resistance, and others, these steels are used for industrial components of high responsibility.

However, these steels have a low yield strength in the annealed condition (about 200 MPa). They can not be hardened by heat treatment. Until cryogenic temperatures, they remain austenitic. However, they are indeed metastable austenitic alloys, since if they are deformed, they transform by a diffusionless transformation (martensitic), into another crystal structure. This transformation is easily remarkable, because the prior material is not magnetic, but when it is deformed, it becomes magnetic.

In order to increase the yield strength of this steel, the most common procedure is to reduce its grain size. Many authors (Forouzan *et al*., 2010) have suggested the procedure of deform the material by cold rolling for produce a martensitic structure (almost 100% of transformation) and after make the reversion of the structure obtained, by a treatment made in an appropriate temperature for avoid the grain growth. This procedure do not affect in a large scale the mechanical resistance.

In this work is investigated the effects of cold reduction in different amounts on the mechanical properties of the steel. It is going to be used the following techniques: Tensile Tests, Optical Microscopy, X ray diffraction, dilatometry and hardness tests.

2. MATERIALS AND METHODS

The material studied is a 304L austenitic stainless steel, produced in "Aperam Inox América do Sul S.A.", rolled in sheets of 6,0 mm of thickness. This material is an Fe-Cr-Ni alloy, with low content of carbon, for improve the corrosion resistance (sensitization resistance). The chemical composition is shown in the table 1.

The rolling was made in a two-high rolling mill installed in the department of Materials Engineering at "Instituto Militar de Engenharia". The samples were deformed by cold rolling, with a interpass cooling, using ice and water. Different thickness reductions of 10% and 20% were applied.

Table 1. Results of chemical composition of the material in the as received condition, made by the supplier.

Element	**C**	**Mn**	**Si**	**P**	**S**	**Cr**	**Ni**	**Mo**	**Al**
Mass%	0,018	1,27	0,479	0,0303	0,0015	18,36	8,02	0,026	0,0032
Element	**Cu**	**Co**	**V**	**Nb**	**Pb**	**B**	**Ti**	**Sn**	**W**
Mass%	0,043	0,102	0,042	0,0071	0,001	0,006	0,0018	0,0044	0,015

Table 2. Martensite transformation temperature, stacking fault energy, and ASTM grain size for AISI 304L stainless steel.

Steel	**M_s (°C) (Eichelman and Hull)**	**M_{d30} (°C) (Angel)**	**SFE (mJ/m^2) (Schramm and Reed)**	**ASTM grain size**
304L	-117	40,0	14,3	7,2

Where M_s is the temperature of start of martensitic transformation. M_{d30} is the temperature in which 50% of the austenite transforms into martensite with 30% of reduction. SFE means Stacking Fault Energy. And they were calculated by equations (Padilha *et al.*, 2004):

$$M_s = 1305 - (1667. (\%C+\%N) +28. \%Si+33. \%Mn+42. \%Cr+61. \%Ni) \quad (1)$$

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

$$SFE = - 53 + 6.2. \%Ni + 0.7. \%Cr + 3.2. \%Mn + 9.3. \%Mo \quad (3)$$

After cutting the sheets, the samples were prepared by ordinary metallographic procedure. The metallography was made in the conditions: as received and as rolled (in the different amounts of reduction). The samples were ground with SiC emery papers with roughness until 2400. The samples were polished in felt cloth with alumina 1,0 , 0,3 and 0,05 µm. After, eletrolytic etching with a mixture of 60 mL nitric acid and 40 mL distilled water was utilized to reveal the austenite grain boundaries. This etching was carried out with the sample inside the solution for 8 minutes, and at 1.0 V (Forouzan *et al.*, 2010). And for reveal the martensite phase, a reagent that is a mixture of two solutions: 0,15 g of sodium metabisulfite diluted in 100 mL of distilled water and 10 mL of hydrochloric acid diluted in 100 mL of distilled water was utilized (Talonen *et al.*, 2007). The microstructures were observed in an optical microscope Nikon Eclipse LV150, with a camera DS-Fi1 and analyzer software Nis – Elements 3 D.0. For the measurements of grain size, it was used in this work the lineal intercept method (Heyn method), which is described in detail in section 13 of ASTM standard E 112-96. From this standard, the equation to obtain the ASTM grain size based on the average length of intercept is:

$$G = (-6.643856. \log_{10} \lambda) - 3.288 \quad (4)$$

For the measurements of amount of martensite present in the samples, it was used a Ferritescope® Fischer FMP30. It has a inferior limit of detection of 0,3% of martensite (minimum fraction). The device was calibrated with δ-ferrite standard samples. After, in specimens as received and in the specimens rolled, it was made XRD measurements for assessment of the present phases, using a diffractometer XRD-6000 Shimadzu, with incident radiation of Copper.

For the hardness measurements for assess the changes in the mechanical properties of the samples, were utilized the Vickers hardness tests with a load of 10 kg. And in the tensile tests, one machine EMIC GR 044, with maximum capacity of 20 kN, was utilized, with a deformation rate of 1.71 mm/min, with the tensile direction parallel to the rolling direction. The deformation was measured by a extensometer with 50.0 mm of gauge length. Finally, for the dilatometric tests a dilatometer NETZCH, model DIL 402C, with range of temperature from 25°C until 1600°C, with thermal bath and flowmeter to measure the output of cooling gas, was utilized to obtain the range of temperature that occur the reversion of martensite for austenite.

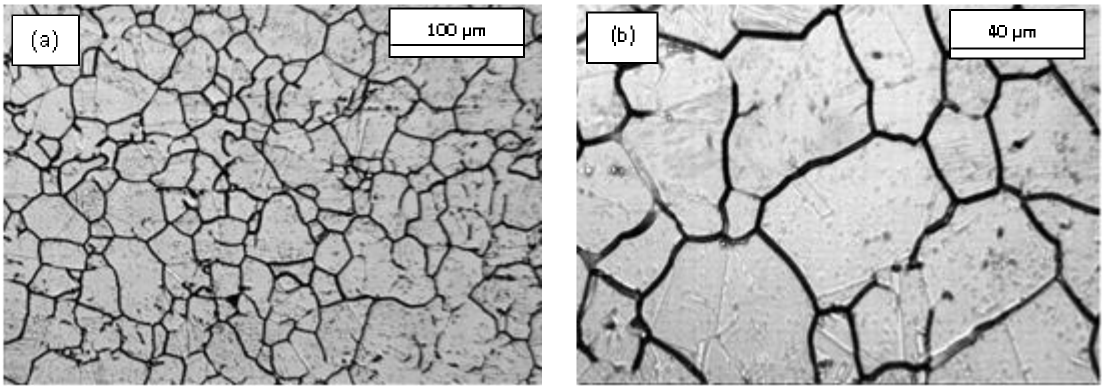
3. RESULTS AND DISCUSSION

Following will be shown results by sample As Received (AR), with 10% Cold Rolling (CR) and 20% Cold Rolling (CR). The results consist in Optical Microscopy, X ray diffraction, Ferritescope, hardness and Tensile tests for AR. For 10%CR and 20%CR the techniques are same without tensile test. At the end, the dilatometry is shown.

3.1 AR

3.1.1 Optical Microscopy

Figure 1 shows the structure of the samples in the as received condition. It may be noted the polygonal structure, also called equiaxial, common in austenitic steels.



Figures 1a,b. Pictures showing the grains of the samples as-received, in different magnifications.

The lineal intercept method gives an average length of intercept of 0.026 mm or 26 µm. The ASTM grain size is therefore 7.2. The grain size can be computed by the equation (4), based on the average intercept (λ).

3.1.2 X ray diffraction and ferristescope

The diffractometric measurements were made in the as received samples in two conditions: the first with the sample submitted to electrolytic etching and the other sample was not submitted to electrolytic etching. As suggested by a recent work (Talonen *et al.*, 2007), the etching can reduce the amount of α'-martensite, probably the one that was induced by the grinding. The Fig. 2 shows the diffractograms obtained. One can note the presence of one peak of α'-martensite (111) that is not present in the other diffractogram (etched sample). The ferritescope measurements gave the average result of amount of martensite equal to 0.53% ± 0.10%.

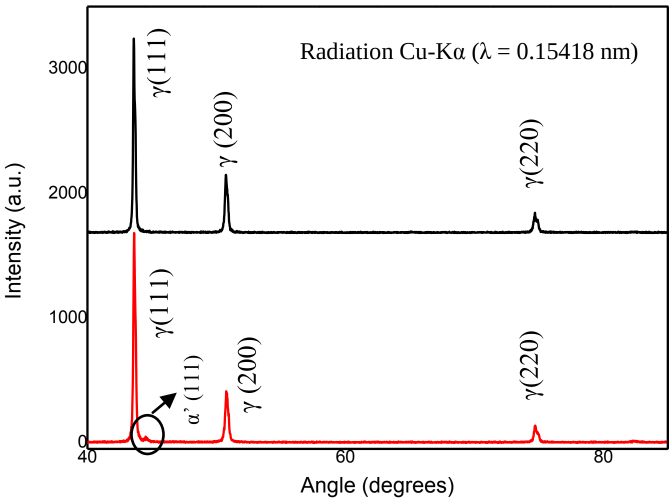


Figure 2. Diffractometer of the sample as received in two conditions, with electrolytic etching and without electrolytic etching.

3.1.3 Hardness and Tensile Test

The hardness measurements were made on the surface of the sample, in the normal direction. It was measured thirty prints, given the average value of (210 ± 9) HV.

The specimen for the tensile test was made with size and shape within the requirements of ASTM standard of tensile tests at room temperature. Figure 3 shows the curve obtained in the test. It may be noted the high level of strain hardening the steel can display. About 10% is noticed that there is an acceleration in the increase of resistance. This is due to the hardening caused by the martensitic phase. The offset method gives yield strength at 0.2% of 300 MPa. The tensile strength is approximately 712 MPa. The test was stopped in a 48% strain level because of the aperture limit of the extensometer utilized. A ferritescope was attached to the specimen to measure *in loco* the formation of martensite during the test. It can be noted the transformation is occurring. This test was made with the sample at room temperature, what reduces the transformation, because the SFE increases, but is possible to see the transformation occurring even in the room temperature, and also, this effect on the stress-strain curve (hardener effect)

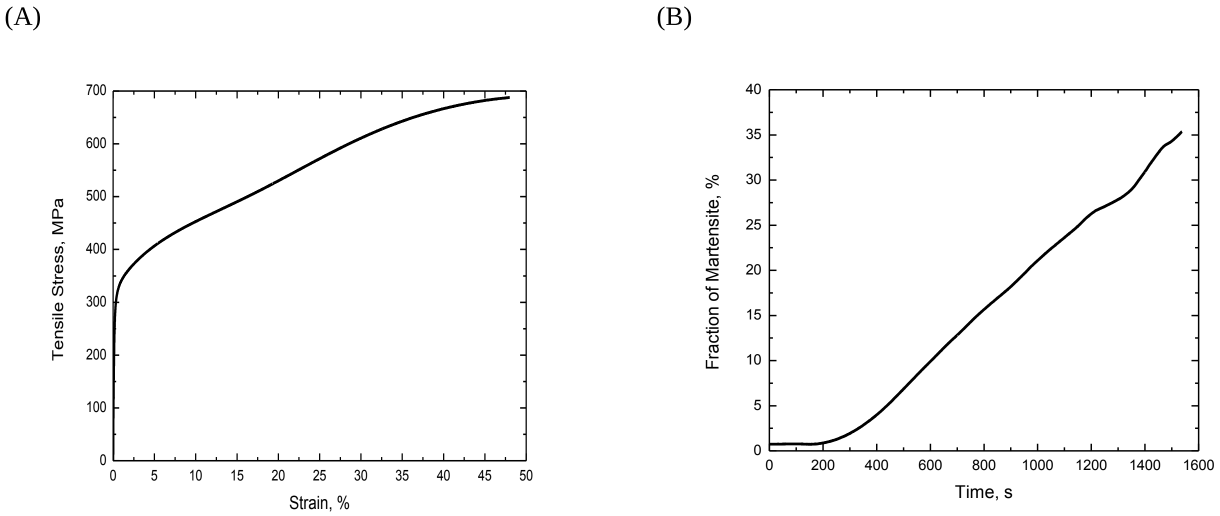


Figure 3. (A) Stress-Strain Curve of the as received sample; (B) Formation of martensite during the test.

3.2 10% CR

The following are the results of samples that have been deformed by rolling at pilot mill at 0 ° C (with interpass cooling).

3.2.1 Optical Microscopy

The etching used to reveal martensite gave satisfactory results, as can be seen in micrographs in the Fig. 4. It may be noted the needles of martensite, with several intersections among them and the prior austenitic grain boundaries. Several researchers have noted that the transformation begins at these intersections, of shear bands. The lineal intercept method gives an average length of intercept of 0.032 mm or 32 µm. The equivalent ASTM grain size is 6.6.

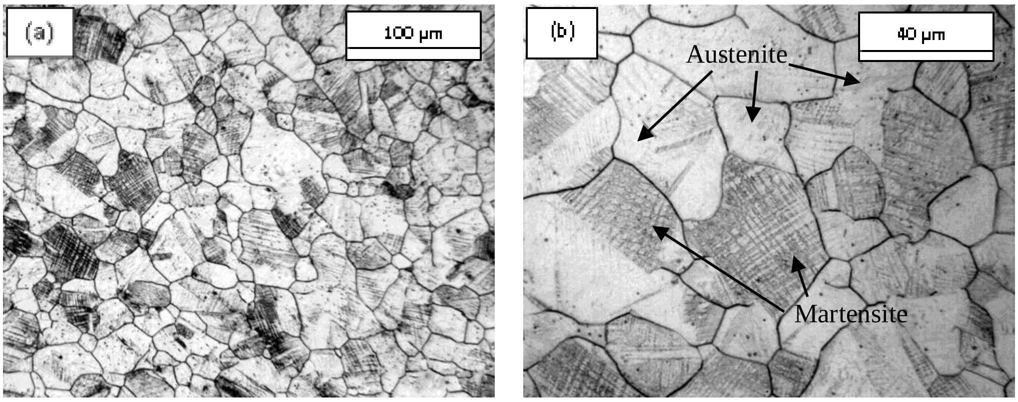


Figure 4. Micrographs showing the samples with 10% of reduction, in different magnifications.

3.2.2 X ray diffraction and ferritescope

The diffractometric measurements in the 10% deformed samples were made just with that submitted to electrolytic etching. The Fig. 5 shows the diffractogram obtained. The ferritescope measurements gave the average result of martensite quantity of 6.97% ± 0.37%.

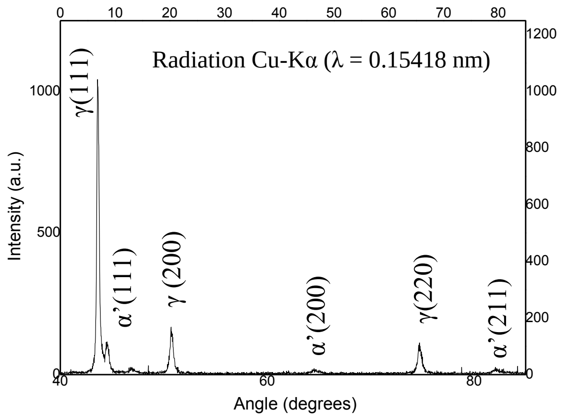


Figure 5. Diffractogram showing the peaks of austenite and martensite phases. Specimen with 10% reduction.

3.2.3 Hardness

The hardness measurements were made on the surface of the sample, in the normal direction. It was measured thirty prints, given the average value of (325 ± 15) HV, which represents almost 55% increase in the hardness of the sample with only 10% of deformation.

3.3 20% CR

The following are the results of sample that have been deformed by rolling at pilot mill at 0 ° C (with interpass cooling).

3.3.1 Optical Microscopy

The Fig. 6 shows the microstructure of the specimen submitted to 20% of deformation by rolling. It is shown micrographs in which the austenitic grain boundaries are not revealed and other pictures with the grain boundaries revealead. It may be noted bundles of needles of martensite. The average length of intercept was found to be 0.037 mm or 37 μm that corresponds to a ASTM grain size of 7

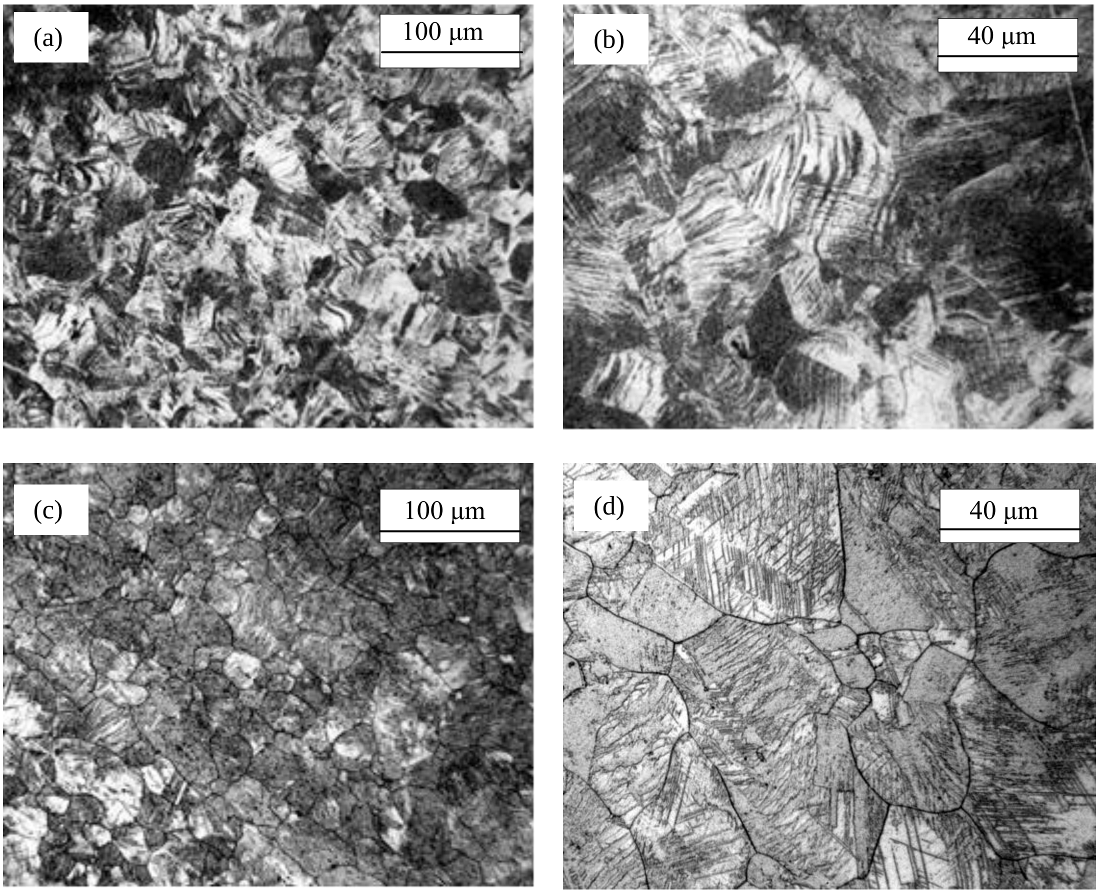


Figure 6. Micrographs of specimen deformed 20%. (A) and (b) images are of the specimen not etched to show austenitic grain boundaries, (c) and (d) images the specimen was etched (eletrolytically).

3.3.2 X ray diffraction and ferritescope

The diffractometric measurements in the 20% deformed samples were made just with that submitted to electrolytic etching. The Fig. 7 shows the diffractogram obtained. The ferritescope measurements gave the average result of amount of martensite equal to 25.5% ± 1.5%. It may be noted the peaks corresponding to α'-martensite planes, i.e. (111), (200) and (211) planes, their intensity was significantly increased in relation to the sample deformed 10%. The peaks have increased more in the interval from 10 to 20% than from 0 to 10%. The more the intensity of the peaks, the more the volume fraction of the respective phase.

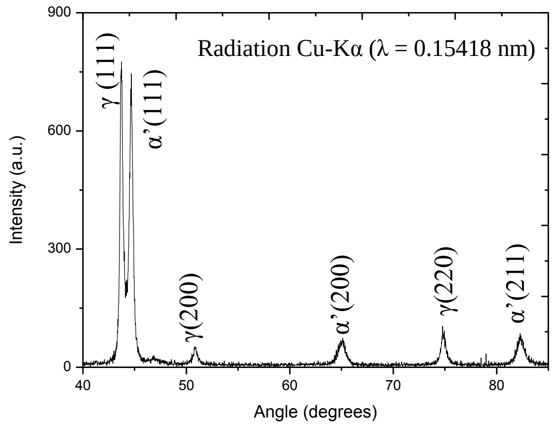


Figure 7 – Diffractogram of the sample 20% Cold Rolling

3.3.3 Hardness

The hardness measurements were made on the surface of the sample, in the normal direction. It was measured thirty prints, given the average value of (398 ± 15) HV.

3.4 Comparison between AR, 10%CR, 20%CR by XRD

In the Figure 8, is shown the XRD for all samples. Is noted that the austenitic peaks disappear and Martensitic peaks appear.

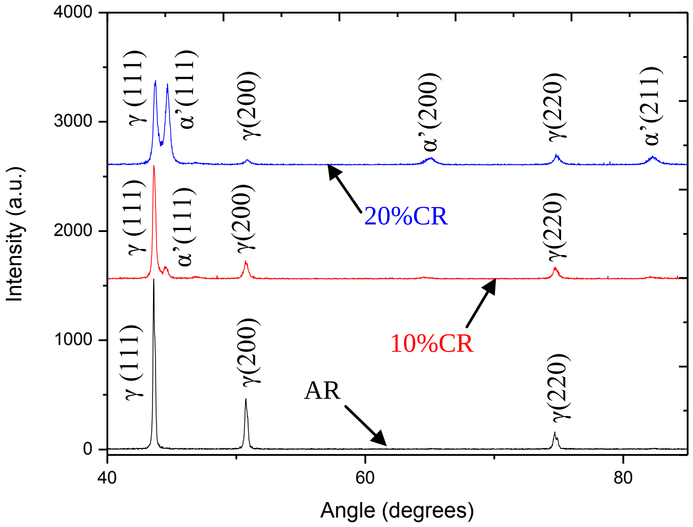


Figure 8: Comparison of AR, 10CR and 20CR diffractograms.

This is comproved by Ferritoscope measuremens, Micrographs and Hardness presented in this work.

3.5 Dilatometry for finding the temperature of martensite reversion

In order to find the temperature of reversion of austenite the dilatometric curve of a sample with 1.5% of martensite induced was performed. The heating rate that was applied was 20°C/min, and the chamber of heating was in a protected atmosphere of nitrogen. The Fig. 8 shows the curve obtained in the dilatometer. It can be concluded that the reversion starts at approximately 600 ° C, going up to 800 ° C. It is known that the reversion temperature is not dependent on the amount of martensite, it is dependent of martensite type only (if α'-martensite or ε-martensite).

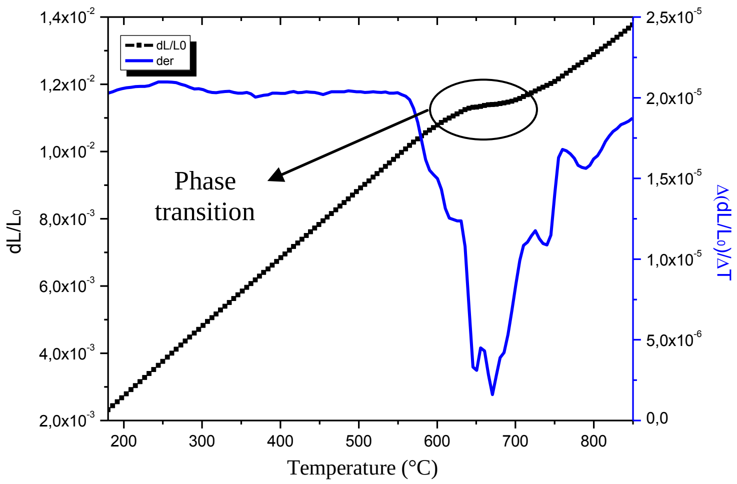


Figure 9. Dilatometric Curve with respective of a sample with 1.5% of martensite, showing the transition of α' to γ between 600 and 800°C. The blue curve is the rate of change in the length of the specimen.

4. CONCLUSION

The samples showed transformation induced plasticity. In fact the deformation caused the austenite to transform for martensite, and consequently hardened the material, from a initial hardness of 210 HV to 325 HV with 10% CR and almost 400 HV with 20%CR. The hardening can be explained by the precipitation of martensite, in such a way that end up hardening the austenitic matrix. And this can be easily verified, by different techniques, as done in this work. At the end, can be concluded also that the martensite can reverse for austenite, just is necessary a heating until a temperature between 600 and 800°C. The effect of this reversion on the mechanical properties is going to be studied.

5. ACKNOWLEDGEMENTS

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