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Magnetic and microstructural characterization of cold rolled UNS S31803duplex stainless steel

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Abstract

Duplex stainless steel presents an attractive combination of mechanical properties and resistance to corrosion. These properties rely on a two-phase mixture compound to produce a microstructure that is approximately equal for the ferritic and austenitic phases. The samples used in this study were cold rolled to degrees of deformation of 20 %, 40 %, 60 % and 80 % according to their thickness. The vibrating sample magnetometer (VSM), X-ray diffraction (XRD), and optical microscopy were used to characterize the rolled samples. It was observed that the cold rolled samples of 40 %, 60 % and 80 % showed an increase in saturation magnetization compared to the samples of as received and cold rolled of 20 %, and this probably occurred because of the formation of strain-induced martensite. It can be seen that the amount of the ferromagnetic phase calculated by ferritoscope tends to decrease with an increasing degree of deformation. The deformation caused by cold rolling resulted in a significant change in diffraction peaks and the mechanical and magnetic properties.

Keywords: duplex stainless steel; cold rolled; martensite; saturation magnetization; ferritoscope.

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1. Introduction

Duplex stainless steels with an attractive combination of mechanical properties, as well as resistance to corrosion, have been widely used in the marine and petrochemical industries [1]. Stainless steel duplex alloys are compounded by two phases (ferrite/austenite) in almost equal proportions which can combine the favorable properties of the ferritic and austenite phases [2-6].

Duplex stainless steel that undergoes cold plastic deformation may present a strain-induced martensitic phase from the austenitic phase [7,8]. As a result, the properties of this steel are modified. Plastic deformation is characterized mostly by the movement of dislocations.

During plastic deformation in duplex steels, an intense multiplication of dislocations occurs in both phases; however the austenite and ferrite phase may share the load and deformation differently. The ferrite (α, bcc) deforms by slip due to the high stacking fault energy (SFE) and the numerous slip systems. Austenite (fcc) can deform by different deformation modes, such as dislocation slip, mechanical twinning, or martensitic transformations [9].

During plastic deformation, the austenite and ferrite may share the load and deformation differently, occurring to a higher degree in the austenitic phase. The high work-hardening rate in the phase austenite has being associated the stacking fault energy present in the phase is lowered further by the presence of nitrogen, and also by the affinity with Cr induces short-range ordering. These effects hinder cross-slip and promote planar slip, increasing the strain - hardening rate in the austenitic phase [10].

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The martensitic transformation takes place due to a coordinated movement among the atoms. In his study, Bain (1924) reported the existence of a body-centered tetragonal structure in the interior of the two cells of the austenitic structure.

The proposed mechanism for the transformation of the martensitic structure has been described as a simple shear in the coordinated movement of the atoms, being able to convert the face centered cubic structure of the austenitic phase into a body centered cubic or body centered tetragonal by means of expansion or contraction the crystallographic axles [11,12]. The applied stress may be sufficiently large to introduce defects such as dislocations and shear bands in the austenite, which then contribute to the nucleation of the martensite [13].

It was observed that in the austenitic structure, two strain-induced martensitic structures can occur: structures ε and α. The ε phase, which forms close-packed (1 1 1) planes in the austenite, has hexagonal (hcp) and the α' phase forms a body-centered cubic crystal structure [14,15]. The ε phase has paramagnetic behavior, while the α' phase is ferromagnetic [7,14,16], but in the duplex steel, only the second martensitic structure, the structure of α', is observed. Lo & Lai, 2010 in their researches verified that 7MoPLUS stainless steels duplex not occurred transformation martensitic induced by deformation, the austenitic phase of 7MoPLUS was stable against martensitic transformation down to 4 K [17].

This work is an analysis of the influence of the plastic deformation on mechanical and magnetic properties of a duplex stainless steel UNS S31803. The ferromagnetic phase was measured through two techniques: ferritoscopy and saturation magnetization by a Vibrating Sample Magnetometer. The results will be discussed, taking into consideration the physical metallurgy of the steel before and after the process of cold rolling.

2. Experimental

Stainless steel duplex UNS S31803 with chemical composition shown in Table 1. Specimens with 110x 40x5.08 mm^3 were cut with the longitudinal direction parallel to the original rolling direction of the sheet. The samples were submitted to cold rolling with reductions of 20 %, 40 %, 60 % and 80 % in relation to their thickness. The deformation applied is shown in Table 2.

For the microstructural analyses, the samples were ground, polished and embedded.

Table 1. Chemical composition for stainless steel duplex UNS S31803.

Chemical Composition	%
C	0.018
Cr	22.2
Mn	1.48
Ni	5.59
Mo	3.08
Si	0.45
Co	0.13
Cu	0.28
Fe	66.5

Table 2. Specimens produced by cold rolling.

Specimen cold rolling	Thickness (mm)	True deformation ε
20%	4.12	-0.229
40%	3.18	-0.468
60%	2.16	-0.855
80%	1.08	-1.548

Chemical etching was performed using Behara's solution for reaction times between 15 s and 25 s to be able to visualize the microstructure. The metallographic experiments were performed using optical microscopy. The technique of diffraction of X-rays was used for the identification of the phases in the microstructure before and after the process of cold rolling. The difratometer used was the X-PERT Pro model, with CuK$_\alpha$ radiation, a wavelength of 1.54 nm (40 kV and 40 mA), sweeping angle of 30^o to 110^o, and a step of 0.02^o.

The measurement of magnetic properties was achieved with discs of approximately 3 mm in diameter and 1 mm of thickness. In the sequence, these samples had their mass gauged on a Bel Engineering precision balance and afterwards, they were submitted to a Lake Shore, model VSM7404 vibrating samples magnetometer, by for the magnetic characterization.

A correction on the values of the magnetic field was applied to the hysteresis curves, depending on the dimensions and deformity of the samples, and such a correction corresponded to the value of the demagnetization fields (H_d) which were at the edges of the samples [18].

For the measurement of microhardness, a Digimess Micro Hardness HV-1000 was used. Ten measurements along the surface of each sample were performed, by using a charge of 0.2 kgf. For the calculation of the quantity of ferromagnetic phases a Fischer model FMP30, version 1.2 ferritoscope was used, and this value was compared with the calculation of the quantity of ferromagnetic phase described by the saturation magnetization formula, developed by Tavares [8]. The steel UNS S31803 had its percentage of ferromagnetic phase determined by eq. 1 [8],

$$C_\alpha = M_s/133 \quad (1)$$

where C_α is the quantity of ferrite, M_s is the saturation magnetization.

3. Results and discussion

Figs. 1, 2, 3, and 4 show the microstructures of materials as received and cold rolled at the reduction rate of 40 %, 60 % and 80 % of the UNS S31803 duplex stainless steel, respectively. Fig. 1 shows the microstructure of the material without the effect of cold rolling, where there is the presence only of the ferritic phase (dark region) and the austenitic phase (clear region).

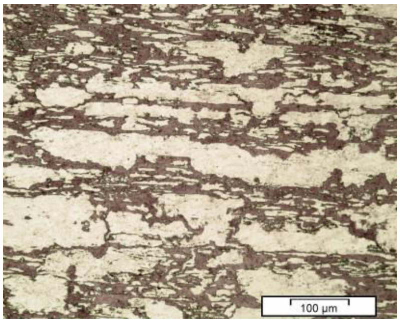

Fig. 1. Sample as received with etching Behara's about 25 s.

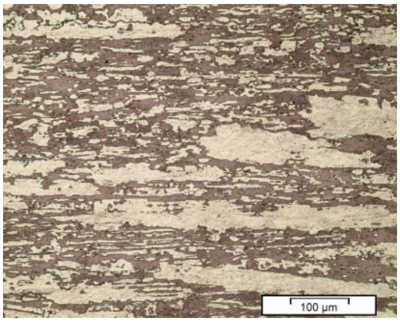

Fig. 2. Sample cold rolled 40 with etching Behara's about 15 s.

The microstructure changes with cold rolling; the deformed grains become and elongated acquire a preferred crystallographic orientation according to the direction of the forming process. The cold plastic deformation produces a refinement in the microstructure of the cold rolled steel samples of 40 %, 60 % and 80 % reduction in relation to the sample as received.

The ferrite phase has numerous slip systems, high stacking fault energy, while the austenitic phase has a smaller number of slip systems and low stacking fault energy [19,20]. For the two phases to undergo the same plastic deformation, the dislocation density in the austenite must be greater.

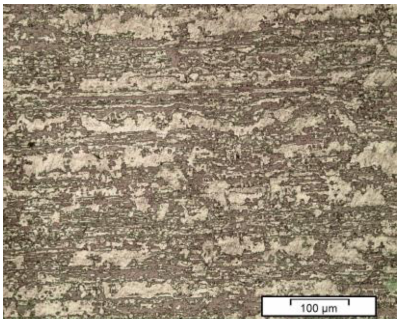

Fig. 3. Sample cold rolled 60 % with etching Behara's about 15 s.

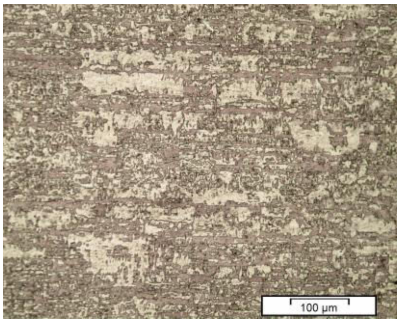

Fig. 4. Cold rolled 80 % with etching Behara's about 15 s.

The presence of the martensitic phase cannot be detected by optical microscopy because the etching with Behara’s solution can distinguish only the ferritic and austenitic phases. The martensitic phase has a very fine structure, and its observation can be made only by using the transmission electron microscope.

Fig. 5 shows microhardness graph as a function of percentage of cold rolled. The maximum microhardness was obtained in the specimen with 80 % reduction (405 HV0.2). In the range analyzed, the hardness increased almost linearly with the amount of cold reduction by deformation.

The same linear behavior of variable hardness was observed by Baldo & Mészáros [7] in their study of the cold rolling effects on the microstructure and magnetic properties of a lean duplex stainless steel. Analyzing only the variation of microhardness of cold rolled duplex steel UNS S31803, there is no guarantee that a strain-induced martensitic phase was obtained by the deformation.

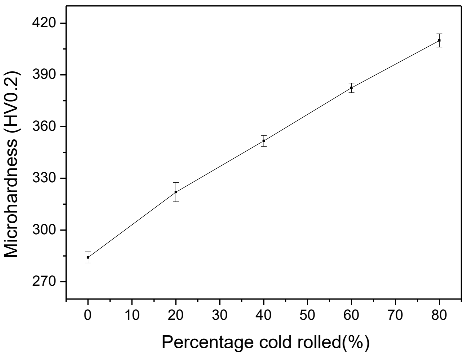

Fig. 5. Graphic of Microhardness of the specimens cold rolled with a percentage of 20 %, 40 %, 60 % and 80 % reduction and also the material as received (0 % cold rolled).

The increase in hardness may also be due to the phenomenon of strain hardening which leads to an increased mechanical strength of the material. Other techniques must be used to verify the appearance of the deformed martensitic phase in a sample, such as a study of the saturation magnetization and an X-ray diffraction analysis of the material before and after the process of cold rolling.

Fig. 6 shows the magnetic behavior of the duplex stainless steel of the rolled samples at 20 %, 40 %, 60 %, and 80 % reduction as well as the material as received (0 % cold rolled). It is observed that the samples rolled at 60 % and 80 % reduction showed an increase in saturation magnetization compared to the cold rolled 20 % reduction sample and the as received sample. It is reported that saturation magnetization depends only on the volume fraction of the magnetic phase which is present in the samples, thus these data show strong agreement with previous studies of martensitic transformation induced by plastic deformation [8].

It can be seen that up to 40% of rolling deformation, the magnetization just increased a little bit, meaning that the austenite phase of UNS 31803 is actually relatively stable against martensitic transformation. Lo e Lai, 2010 showed through of magnetic hysteresis loops and X-ray analysis that the austenite phase of 7MoPLUS did not transform martensitic after cold-rolling reduction of 50% thickness at room temperature [17].

In their studies, Baldo and Mészáros [7] have reported that for lean duplex stainless steel, the saturation magnetization does not change for the samples deformed to a value less than 30 %. It is observed increase of values for saturation magnetization upon cold deformation with more than 60 % reduction, thereby reporting the possible formation of martensitic structures induced by deformation.

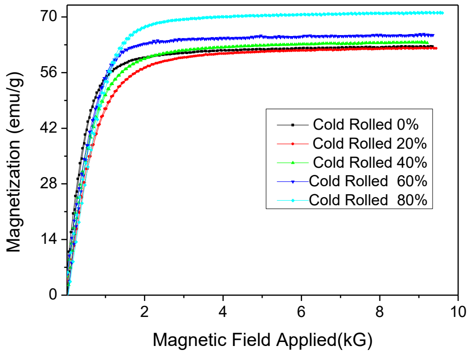

Fig. 6. Curve of Magnetization *vs.* Magnetic Field Applied of samples.

Table 3 shows the values of saturation magnetization, percentage of ferromagnetic phase and of cold rolled samples. Also included are the percentages of ferromagnetic phase developed by Tavares [8] by Eq. 1. Table 4 shows the coercive field and remanent magnetization of UNS S31803 duplex stainless steel; of the material as received, and samples cold rolled at 20 %, 40 %, 60 % and 80 % reductions. It can be seen that both values, the remanent magnetization and the coercive field, increase with the rate of deformation. These materials have the behavior of magnetically soft material.

Table 3. Magnetic Parameters: saturation magnetization (M_S), the percentage of ferromagnetic phase and the number of ferrite (FN).

Percentage of cold rolled	Saturation Magnetization (emu/g)	Percentage of Ferromagnetic phase (%)	Number of ferrite (FN)
0	62.12±0.30	46.71±0.22	44.60±1.90
20	62.29±0.20	46.84±0.15	37.31±0.57
40	63.48±0.46	47.73±0.34	35.16±0.48
60	65.23±0.21	49.04±0.16	32.79±0.48
80	70.18±0.59	52.76±0.44	33.74±0.25

Table 4. Magnetic Parameters: Coercive Field (G) and remanent Magnetization (M_R).

Percentage of cold rolled	Coercive Field (G)	Remanent Magnetization (emu/g)
0	22.5±1.00	2.05±0.14
20	31.00±0.20	2.19±0.20
40	35.38±0.59	2.84±0.14
60	40.40±0.56	3.40±0.28
80	51.07±0.83	4.04±0.16

Note that both values of ferrite calculated by the ferritoscope tend to decrease with the increase in deformation. Also, observe the increase of ferrite content in the sample cold rolled at 80 % reduction relative to the sample cold rolled at 60 % reduction. This decrease in the amount of ferromagnetic phase in cold rolled samples, as obtained by the ferritoscope, may be due to the fact that the operation of this equipment is based on the magnetic permeability of the material.

The deformation generated led to changes in the texture of the material, resulting in errors in the readings. The magnetic permeability of α' martensitic structures is affected by the deformation process [21]. Escriba [22] report that the magnetic measurements obtained by the ferritoscope are strongly influenced by the crystallographic texture of the cold rolled sample.

The ferritoscope could not identify the formation the strain-induced martensitic phase the austenite, because as the martensitic phase is a magnetic phase, while the austenite is nonmagnetic, thus, the magnetic signal tends to increase, which was not observed in use of ferritoscope.

Fig. 7 shows the X-ray diffraction of UNS S31803 stainless steel of the cold rolled samples, as well the identification of the austenite and ferrite phases. It may be noted that the deformation caused by the cold rolled process resulted in a significant change in peaks of the phase austenite and ferrite. It can be seen that the peak of the austenitic phase (111) became the highest after 80% deformation, possibly due to the change arising from the texture of the rolling process.

In the cold rolled samples, the peaks of ferrite and martensite cannot be distinguished, because both phases have the same reflection.

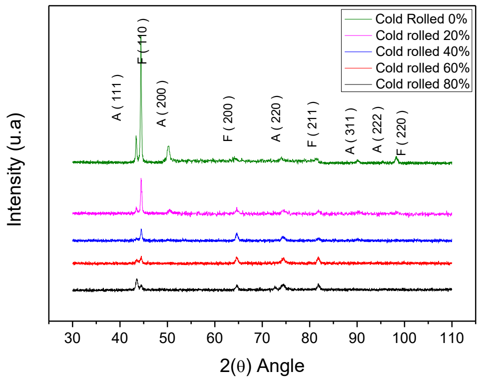

Fig. 7. Diffractions X- rays of samples with: A= Austenite phase and F = Ferrite phase.

These changes in the appearance of the diffraction patterns can be caused by a change occurring in the texture of the material, together with a possible phase transformation induced by the deformation

4. Conclusions

The cold rolled process caused changes in the microstructure and properties of duplex stainless steel UNS S31803. The cold rolling produces a refinement in the microstructure of the duplex stainless steel. It was verified that there was a linear increase in microhardness with the plastic deformation.

For the samples cold rolled at 40 %, 60 % and 80 % reductions, an increase in the saturation magnetization was noted, which may indicate the formation of strain-induced martensite. While the cold rolled sample with a reduction of 20 % shows the same value for the magnetization of saturation as the sample as received; it is possible that martensite was not formed in the deformation.

A decrease of the values obtained by the ferritoscope was observed. The ferritoscope could not identify the strain-induced martensitic phase. The values obtained by the ferritoscope decreased due to the change in the texture of the material caused by the cold rolling. Changes in the appearance of the diffraction patterns of the cold rolled samples were observed.

These changes were observed in the magnetic properties, where the diffraction peaks can indicate a possible transformation of the strain-induced martensitic phase generated by the cold deformation process.

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References

[1] T.H Chen, J.R Yang, Mater. Sci. Eng. A311 (2001) 28.
[2] R.N. Gunn, Duplex stainless steels- Microstructures, properties and applications, ed., Abington Publishing, Cambridge England, 2003.
[3] J.M. Cabrera, A. Mateo, L. Llanes, J.M. Prado, M. Anglada, J. Mater. Process. Technol. 143 (2004) 321.
[4] A.L.V.C. Silva, P.R Mei, Aços e ligas especiais, ed., Edgard Blucher, São Paulo, 2006.
[5] S.K. Ghosh, D. Mahata, R. Roychaudhuri, R. Mondal, Bull. Mater. Sci. 35 (2012) 839.

[6] S. Fréchard, F. Martin, C. Clément, J. Cousty, Mater. Sci. Eng. A418 (2006) 312.
[7] B. Silva, I. Mészáros, J. Mater. Sci. 45 (2010) 5339.
[8] S.S.M. Tavares, M.R. da Silva, J.M. Pardal, H.F.G. Abreu, A.M. Gomes, J. Mater. Process. Technol. 180 (2006) 318.
[9] C. Herrera, D. Ponge, D. Raabe, Acta Mater. 59 (2011) 4653.
[10] J. Johansson, M. Odén, Metall. Mater. Trans. A. 31(6) (200) 1557.
[11] C.M. Wayman, Metall. Mater. Trans. A 25 1787 (1994) 1787.
[12] H.K.D.H, Bhadeshia, Martensitic Transformation, Encyclopedia of Materials: Science and Technology, 2001.
[13] A. Das, P. C. Chakraborti, S. Tarafder, H.K.D.H. Bhadeshia, Mater. Sci. Technol. 27 366 (2011) 366.
[14] S. Mészáros, J. Prohászka, J. Mater. Process. Technol. 161 (2005) 162.
[15] J. Talonen, H. Hãnninen, Acta Mater. 55 (2007) 6108.
[16] S.S.M. Tavares, D. Fruchart, S. Miraglia, J. Alloys Compd. 307 (2000) 311.
[17] K.H. Lo, J.K.L. Lai. J. Magn. Magn. Mater. 322 (2010) 2335.
[18] S. Chikazumi, Physics of Magnetism, John Wiley, 1964.
[19] A.F. Padilha, R.L. Plaut, Phase transformation and microstructure. In: Iris Alvarez-Armas; Suzanne Degallaix-Moreuil. (Org.). Duplex Stainless Steels, ISTE Ltd and John Wiley \& Sons, 2009.
[20] W. Reick, M. Pohl, A.F. Padilha, ISIJ international, 38 (1998) 567.
[21] J. Talonen, P. Aspegren, H. Hänninen, Mater. Sci. Technol. 20 (2004) 1506.
[22] D.M. Escriba, E. Materna-Morris, R.L. Plaut, A.F. Padilha, Mater. Charact. 60 (2009) 1214.