MATERIALS CHARACTERIZATION 59 (2008) 901–904

Magnetic properties and α′ martensite quantification in an AISI 301LN stainless steel deformed by cold rolling

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ARTICLE INFO

Article history:
Received 26 June 2007
Accepted 11 July 2007

Keywords:
AISI 301LN steel
Ferromagnetic properties

ABSTRACT

Ferromagnetic properties of cold worked AISI 304 and 304L steels have been studied by many authors. The AISI 301LN steel has lower chromium and nickel contents than AISI 304L and 0.1–0.15%N addition. This steel presents a higher work hardening rate due to its higher susceptibility to martensite formation during cold deformation. In the present work the magnetic properties (magnetization saturation, residual induction and coercive force) of cold rolled AISI 301LN steel were investigated. Different degrees of deformation were applied. The dependence of the saturation magnetization on the amount of deformation is modeled by an exponential decay law. An increase in the amount of deformation also promotes a decrease of coercive force. Heat treatments at 350 °C and 400 °C after deformation were also applied and found to increase the magnetization saturation values.

1. Introduction

Conventional austenitic stainless steels (AISI 304, 316, 301, 302) are susceptible to martensitic transformations induced by cold deformation. Two types of martensite are often obtained by this fashion: ε and α′. Phase ε is hcp and paramagnetic, and α′ martensite is bcc and ferromagnetic. According to Mangonon and Thomas [1], ε martensite is formed in a 304 stainless steel deformed by tension at −196 °C at the beginning of the straining and reaches a maximum amount (10%) at about 5% deformation. A further increase of deformation leads to a decrease in the amount of ε and a continuous increase of α′. The behavior of AISI 316 is very similar, but the maximum amount of martensite ε is attained at 13% deformation [2]. Based on these results the sequence of transformation $\gamma \rightarrow \varepsilon + \alpha'$ was suggested. However, the direct transformation $\gamma \rightarrow \alpha'$ via dislocation reactions is also possible, as suggested by Bogers and Burguers [3].

Previous works [1,4–6] report an increase in the amount of α' martensite in AISI 304 heat treated at 400 °C after deformation. The explanation for this increase is still under discussion. The initial theory was based on the precipitation of carbides, which would locally increase the martensite start (M_s) temperature. However, the absence of experimental observation of chromium carbide precipitation at 400 °C does not support this theory. On the other hand, Guy et al. [6] proposed that the growth of existing α′ laths at 400 °C occurs as the defect structure in the austenite recovers, allowing the relaxation of the γ/α′ interfaces. Gauzzi et al. [5] showed by X-ray diffraction analysis that the increase of martensite content in a AISI 304 steel deformed and treated at 400 °C is accompanied by recovery of the austenite phase.

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doi:10.1016/j.matchar.2007.07.007

Table 1 – Chemical composition of the AISI 301LN steel

Cr	Ni	Mo	C	N	Mn	Cu	Si	S	Fe
17.650	6.616	0.168	0.023	0.105	1.687	0.169	0.530	0.004	Bal

While the magnetic properties of cold rolled AISI 304 and 304L steels have been studied in previous work [7,8], no such studies have been reported on AISI 301LN steel, to our knowledge. In this paper, the magnetic properties of the AISI 301LN are reported as a function of the amount of deformation. The effect of post-deformation heat treatments at 350 °C and 400 °C were also investigated.

2. Experimental

A 1.9 mm thick sheet of AISI 301LN steel (nominal composition shown in Table 1) was solution treated at 1050 °C and cold rolled to 1.43 mm, 1.0 mm, 0.5 mm, 0.27 mm and 0.16 mm. Table 2 shows the true strain and the effective strain in each condition. After deformation small samples were cut for magnetic measurements. Deformed samples were heat treated at 350 °C and 400 °C for 1 hour to produce samples A2, A3, B2, B3, C2, C3, D2, D3, E2 and E3 (see Table 2). Vickers microhardness measurements were performed for all conditions.

Magnetic properties of the deformed AISI 301LN steel were measured at room temperature in a vibrating sample magnetometer EGG-PAR model 4500, with maximum applied field of 6.30 kOe, a sweep time of 1 ms and total measurement time of 30 min. Measurements reported for AISI 304L and a duplex steel UNS S31803 were considered, for comparison.

X-ray diffraction (XRD) measurements were carried out with a diffractometer PHILIPS®, model X'Pert Pro, in the step-scan mode with a step size of 0.02° and time per step of 3 s. CuKα (0.154056 nm) radiation at 40 kV and 40 mA was used. The precise lattice parameters were determined by plotting $d_{hkl} x \cot\theta \cos\theta$ and taking the value at $\theta = 0$.

3. Results

Fig. 1 compares the increase of saturation magnetization (m_s) with the amount of true deformation in samples deformed and treated at 350 °C. Fig. 2 shows similar data for samples treated at 400 °C. The m_s values increase with deformation and exhibit a slight additional increase after the heat treatments at 350 °C and 400 °C. The experimental points can be fitted by a first order exponential law:

$$M_s = 153.2 - 153.1 \cdot \exp(-2.417\varepsilon) (\text{asdeformed}) \quad (1)$$

$$M_s = 153.9 - 154.8 \cdot \exp(-2.575\varepsilon) (\text{after heat treatment at350°C}) \quad (2)$$

$$M_s = 157.1 - 157.5 \cdot \exp(-2.506\varepsilon) (\text{after heat treatment at400°C}) \quad (3)$$

Fig. 3 presents the X-ray diffractograms of the as-deformed samples D1 and E1, with the highest amount of deformation

Table 2 – Deformation levels applied in this work

Samples	Final width	Reduction	True strain, ε_1	Effective strain, ε_{eff}
A1–A2–A3	1.41	26%	0.314	0.363
B1–B2–B3	1.00	47%	0.642	0.741
C1–C2–C3	0.50	74%	1.335	1.542
D1–D2–D3	0.27	86%	1.951	2.253
E1–E2–E3	0.16	92%	2.474	2.857

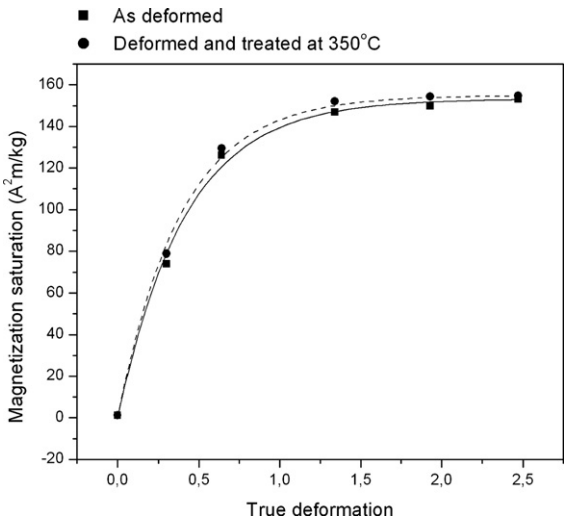

Fig. 1 – Curves of saturation magnetization *versus* true deformation for samples deformed and heat treated at 350 °C after deformation.

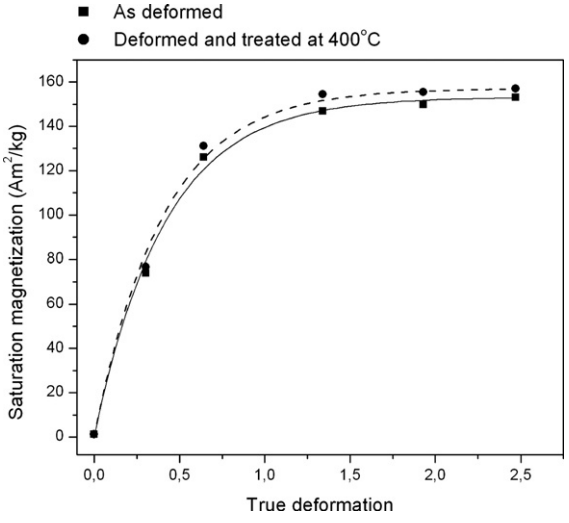

Fig. 2 – Curves of saturation magnetization *versus* true deformation for samples deformed and heat treated at 350 °C after deformation.

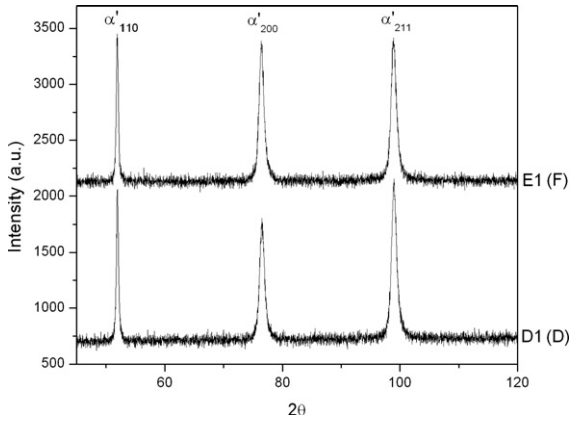

Fig. 3 – X-ray diffractograms of samples D1 and E1.

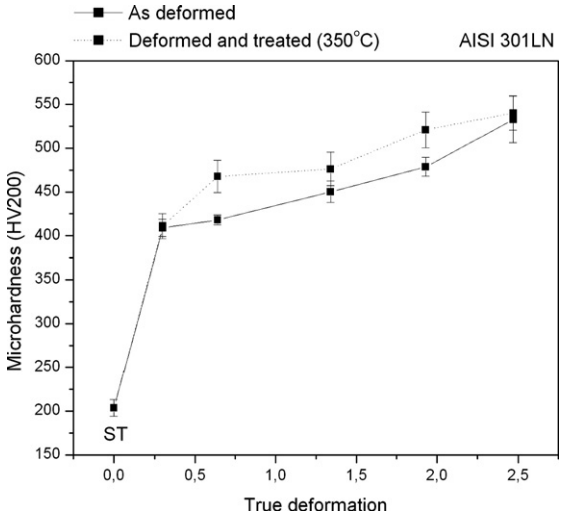

Fig. 4 – Microhardness against true deformation for samples deformed and heat treated at 350 °C after deformation.

(ε=1.93 and 2.47, respectively). The diffractograms reveal the presence of α′ martensite as the sole phase. The lattice parameters are 0.2887 nm and 0.2884 nm, respectively, and are found to be larger than that of α′ martensite in AISI 304 steel due to the interstitial nitrogen atoms. XRD is one of the methods most often used to perform phase quantification, in particular austenite quantification in martensitic steels. However, in the case of the as-deformed and heat treated sample D2 the small quantities of austenite still present could not be quantified by XRD.

Table 3 shows the X-ray diffraction data collected from the diffractograms of samples A1 (as-deformed) and A2 (as-deformed plus 350 °C heat treatment). The phase quantifications were performed using the direct comparison method described by Cullity [9] assuming the same chemical composition for both phases. The heat treatment at 350 °C causes a decrease in the half height peak width in austenite and martensite, which suggests that both phases undergo recovery processes at 350 °C.

Despite the recovery effects, the heat treatment at 350 °C promotes an increase of the microhardness in the conditions B, C and D (true strains from 0.6 to 1.95), as shown in Fig. 4. This hardening effect can be attributed to the strain aging of the martensite [10] and also to the increase in martensite volume fraction. The increase of hardness with treatment at 350 °C is almost nil in the least and highest deformed samples. This suggests that at the highest level of deformation (Sample E; ε_1=2.474) the recovery process may counterbalance the hardening effects. In addition, the increase in martensite volume fraction is less important at this condition, as will be shown later.

Table 3 – X-ray diffraction data collected from the diffractograms of samples A1 and A2

Sample	Volume fractions (XRD quantification)		Half height width (β_{hkl})				
			Martensite α′			Austenite γ	
	f_γ	f_α	β_{110}	β_{200}	β_{211}	β_{111}	β_{220}
A1	0.217	0.783	0.319	0.795	0.739	0.225	0.597
A2	0.143	0.857	0.304	0.760	0.696	0.206	0.566

Fig. 5 shows that the coercive force tends to decrease with the amount of deformation and, consequently, with an increase in martensite volume fraction. This trend was also observed previously in AISI 304 steel [8]. The effect of the heat treatments at 350 °C and 400°C on the coercive force was unexpected. Based on the results for the AISI 304 steel [8] the coercive force was expected to decrease with the heat treatment. Although a modest change was observed, this behavior was not prominent for the AISI 301LN.

Eq. (3) suggests that the intrinsic magnetization saturation of the martensite phase is 157.1 emu/g. Consequently, the quantification of the martensite α′ can be calculated from the following equation:

$$\mathrm{MVF} = \frac{m_s}{157.1} \tag{4}$$

where MVF is the martensite volume fraction and m_s is the magnetization saturation of the analyzed sample in Am^2/kg.

The intrinsic magnetization saturation of the ferromagnetic phase is a composition-sensitive property. The value

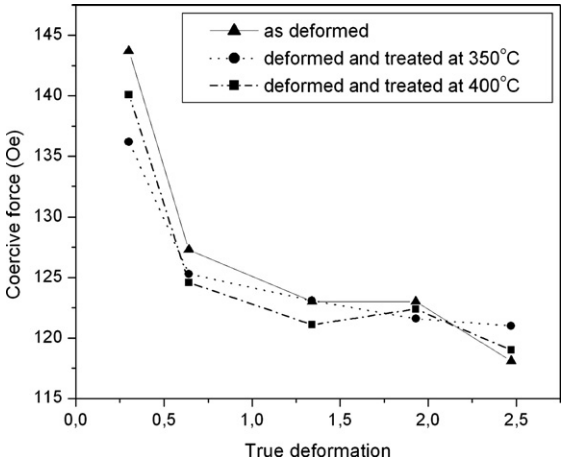

Fig. 5 – Coercive force against true deformation for samples deformed and heat treated at 350 °C after deformation.

Table 4 – Magnetization saturation values of the ferromagnetic α′ martensite phase used for magnetic phase quantification

Material	Magnetization of α′ martensite	Reference
AISI 304	160.4	[1]
AISI 301LN	157.1	This work
UNS S31803 (duplex)	133.0	[12]

157.1 Am^2/kg found in this work can be compared to other published values for AISI 304 [1] and UNS S318003 duplex steel [13], as shown in Table 4.

Fig. 6 compares the martensite volume fractions (MFV) of AISI 301LN, AISI 304 and duplex UNS S31803 steels as a function of true deformation. The MFV were quantified by the magnetic method. The susceptibility to martensite-induced transformation of austenitic steels is generally related to the stacking fault energy (s.f.e.), which is also a composition-sensitive parameter. Using the equation proposed by Schamm and Reed [11] the s.f.e. of the AISI 301LN studied is 6.0 mJ/m^2, which is much lower than published values for AISI 304 steel (~18 mJ/m^2 [2]). This explains the faster kinetics of the $\gamma \rightarrow \alpha'$ transformation with deformation for the AISI 301LN steel than for the AISI 304, Fig. 6. In these austenitic steels the $\gamma \rightarrow \alpha'$ transformation rate decreases with the amount of deformation. Analyzing the behavior of the duplex UNS S31803 stainless steel, despite the low stacking fault energy reported by Reick et al. [12] for this alloy (12.0 mJ/m^2), the susceptibility to martensite formation is much lower than in the austenitic grades, and the rate of transformation increases slowly with an increase of true deformation, as shown in Fig. 6.

4. Conclusions

The deformation-induced martensite volume fraction in an austenitic AISI 301LN steel can be quantified by magnetization saturation measurements. The intrinsic magnetization saturation of the martensite phase in this steel is 157.1 Am^2/kg. The coercive force tends to decrease with true deformation, especially in the initial stage of deformation. Heat treatments at 350 °C and 400 °C after deformation promote an increase of magnetization saturation, which is attributed to additional martensite formation. X-ray diffraction measurements confirm the increase of the α′martensite phase and also show that the material undergoes a recovery process during the re-heating at 350 °C. Despite this, the 350 °C treatment modestly increases the hardening due to the additional martensite formation and strain aging effect.

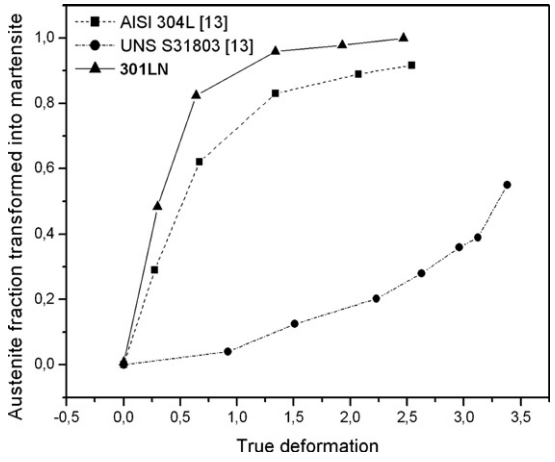

Fig. 6 – Comparison of martensite volume fractions against true deformation curves for AISI 301LN, AISI 304 and duplex UNS S31803 steels.

Acknowledgments

The authors acknowledge Brazilian research agencies (CAPES, FAPERJ and CNPq) for financial support.

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