Martensite quantification, mechanical properties and cold rolling in AISI 301 Austenitic Stainless Steel

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Austenitic Stainless steel known as AISI 301 are often used as cold rolled foil with very thin thickness (down to 0.2 mm). Such materials are further shaped by bending or stamping and need to show a suitable deformability even at the cold hardened stage as is the case of the commercialized product. The quantification of the martensite, precipitated as main by product of the cold deformation of the metastable austenitic microstructure, is considered of dramatic importance to predict the toughness of this alloy. In this paper an original method to quantify the martensite by using the magnetic susceptibility (SQUID) is presented and the combination of the so collected data with stress-strain curves on differently deformed samples are discussed. The conclusions introduce the role of cold hardening in the mechanical properties of cold deformed AISI 301 beside the effect of the martensite volume fraction.

KEYWORDS: AUSTENITIC STAINLESS STEEL, MARTENSITE, COLD HARDENING, SQUID, STRESS-STRAIN CURVES, MECHANICAL PROPERTIES

INTRODUCTION

Austenitic stainless steels are interesting engineering materials, due to their high corrosion resistance and versatile mechanical properties. They have excellent ductility and toughness in annealed condition. Furthermore their tensile strength can be greatly increased by cold working due to their metastable nature. Such a skill is mainly related to the formation of martensite microphases dispersed into the austenitic matrix as a consequence of the energy absorbed during the cold deformation or the mechanical stress in general. The martensite is stronger and harder than the austenitic structure, causing a sort of precipitation strengthening effect and thus a high strain effect. This known phenomenon [1] is on one side explained by the natural evolution of the metastable austenite into a more stable lattice at room temperature but on the other side there are still kinetic and thermodynamic aspects to be investigated and understood in order to model the martensite transformation and to make it profitable for technical applications [2]. The thermal treatment between the cold rolling stages seems also to play a role by its efficacy in recovering deformability and, at the same time, dissolving the martensite precipitates. The final state is often the cold rolled one and thus cold hardened with an amount of martensite which needs to be quantified in order to predict the basic mechanical properties of the metal sheet.

Due to their expected excellent ductility austenitic stainless steels find applications in those fields where severe forming operations are required [3]. They are also gaining more interest for their combination of formability and high strength after forming. An annealed sheet can easily be formed to complex shapes but this skill is also required finished cold rolled sheets. The combination of cold rolling steps and recovery thermal treatments become thus strategic in order to offer the lowest risk of failure. At the same time a reliable prediction of the mechanical properties under stress would make such steels even more attractive for specific applications. To reach such a goal a reliable tool to quantify martensite is therefore demanded as well as a deeper understanding of its formation beside other transformation observed

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during the manufacturing process.
The deformation induced martensitic transformation is a complex transformation and the theory on martensitic transformation remains vague. This issue depends on many factors both practical and analytical. On one hand there is the lack of profound understanding on how parameters such as chemical composition, temperature, strain rate, grain size, deformation mode affect the transformation. On the other hand the is the need to measure the amount of martensite even at very low concentration and to relate its volume fraction to the manufacturing parameters and to the mechanical properties. It is known that the presence of martensite is changing the magnetic properties of the austenitic stainless steel [4] and thus this can be used as an alternative to the classical XRD method which is not enough sensitive to low martensite volume fraction and to the expensive and too local TEM investigation.
In this paper the SQUID measuring system, based on the quantification of the martensite volume fraction by its magnetic properties, is used on industrial products obtained by standard manufacturing. The same materials are also mechanically characterized by stress-strain experiments and the mechanical properties are related to the microstructural features.

MATERIALS AND METHODS

The compositional data reported in table 1 are referred to the rolled stainless steel AISI 301 that has been studied in this research.

Tab.1 - Steel composition (AISI 301)

Element	C	Si	Mn	P	S	Cu	Ni	Cr	Mg	N
Wt.%	0.10	1.11	1.20	0.024	<0.01	0.13	6.4	16.9	0.68	0.059

To reach the final thickness of the metal sheet several sessions of cold rolling and recovery bright annealing were applied using industrial parameters on an unidirectional mill. Table 2 reports the thermomechanical treatment details.

Tab. 2 - Thermomechanical parameters

Cold Rolling Cycle	initial - final thickness (mm)	No. of steps	% reduction	Bright Annealing
1	0.90 - 0.36	6	60	1150°C in N_2
2	0.36 - 0.25	8	30.5	-

With the purpose to quantify the martensite volume fraction and to determine the mechanical properties a sample from each step of the second cold rolling cycle was acquired and investigated, table 3 reports the complete list of samples.

Tab. 3 - List of samples

Sample Name	Step	Thickness (mm)	Def. rate (%)	$\Delta_{(def.rate)}$
0.360	ANNEALED	0.36	0	0
0.330	1	0.33	8.3	8.3
0.296	2	0.295	17.8	9.5
0.285	3	0.285	20.8	2.7
0.280	4	0.28	22.2	1.4
0.276	5	0.276	23.3	1.1
0.261	6	0.261	27.5	4.2
0.254	7	0.254	29.4	1.9
0.252	8	0.252	30	0.6
0.250	9	0.25	30.5	0.5

The $\Delta_{(def.rate)}$ was calculated by subtracting the deformation rate of previous sample to the deformation rate of the considered sample (as shown in equation 1): is a worth information to easily evaluate the strain entities to single step.

$$\Delta_{(def.rate)} = [Deformation\ Rate_{(sample\ x)}] - [Deformation\ Rate_{(sample\ x-1)}] \qquad (1)$$

The investigation methods applied to all samples are:
1. Tensile test
2. Magnetometer SQUID M vs H measure
3. Optical Microscope (LOM) metallography
4. Scanning Electron Microscope (SEM) fractographic analysis

Only results statistically representative of a group of samples and meaningful for the discussion will be presented in the following paragraphs.

Investigations at points 1 and 2 were made on the sampled specimen without further preparation but simply sizing the fragment according to the analytical method while samples for characterization methods 3 and 4 were hot mounted in resin and metallographically polished up to 250 nm diamond grain size in order to show a longitudinal cross section. The microstructural features were highlighted by etching the polished sample with a water diluted aqua regia (H2O, HCl, HNO3, vol:vol 35:35:1). The fracture of the samples generated during the tensile test where characterized by SEM (point 4).

RESULTS AND DISCUSSION

OPTICAL MICROSCOPY

The optical metallography helps to define the matrix microstructure and to detect features directly related to the manufacturing process such as mechanical twins and slip bands. The concentration of a deformed matrix observable in the center of the most rolled samples was also observed. It was decided to not quantitatively asses the grain size due to strong microstuctural distortion. Figure 1 shows the most significant microstructures according to the rolling step.

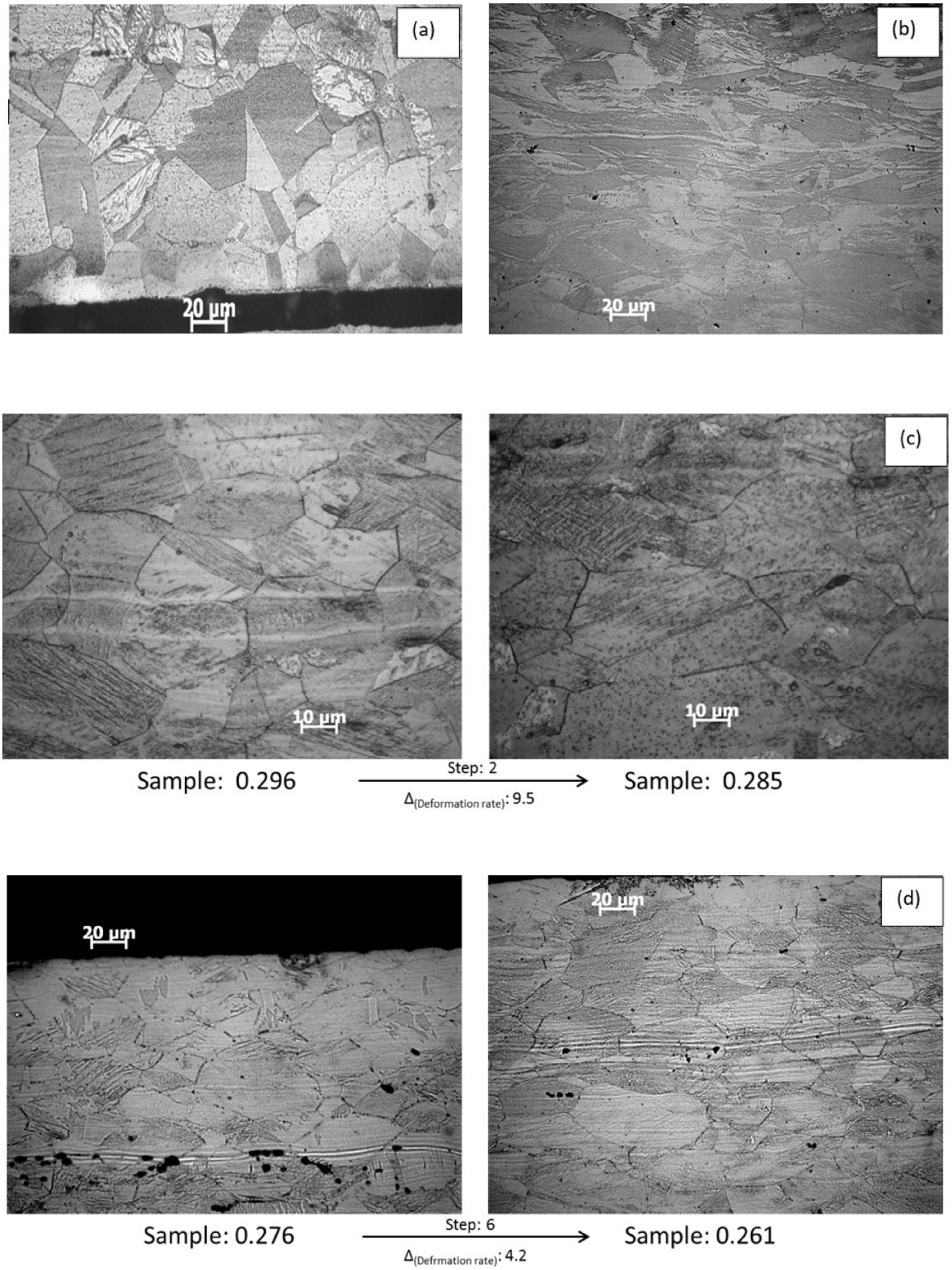

Fig. 1 - Metallography, magnifying 500x. Sample: 0.360annealed (a), 0.250 (b), 0.296 (c left), 0.285 (c right), 0.276 (d left), 0.261 (d right)

The pictures (a) and (b) relatively show the microstructure of the as bright annealed (a, 0.360mm) and the final cold rolled product (b, 0.250mm). The metallography couples (c) and (d) are the most significant, since they correspond to steps 2 and 6, which have a larger value of Δ(def.rate), which entails a greater structural deformation.
Especially sample 0.285 compared to sample 0.296 shows a greater amount of alteration with an higher concentration of slip bands and distorted grains. Sample 0.261 compared to sample 0.276 presents areas looking like a coalescence of darker phases (possibly martensite) and much more grains elongated in the rolling direction.

SCANNING ELECTRON MICROSCOPE FRACTOGRAPHIC ANALYSIS

Fractures produced by tensile tests was observed by Scanning Electron Microscope (SEM). The goal of this analysis is to observe the possible presence of fracture surface alteration and austenite according to the different fracture type. Figure 2 shows representative examples of fractography showing evidences of alteration of the classical micro-dimples ductile fracture surface which is typical of the austenitic stainless steels.

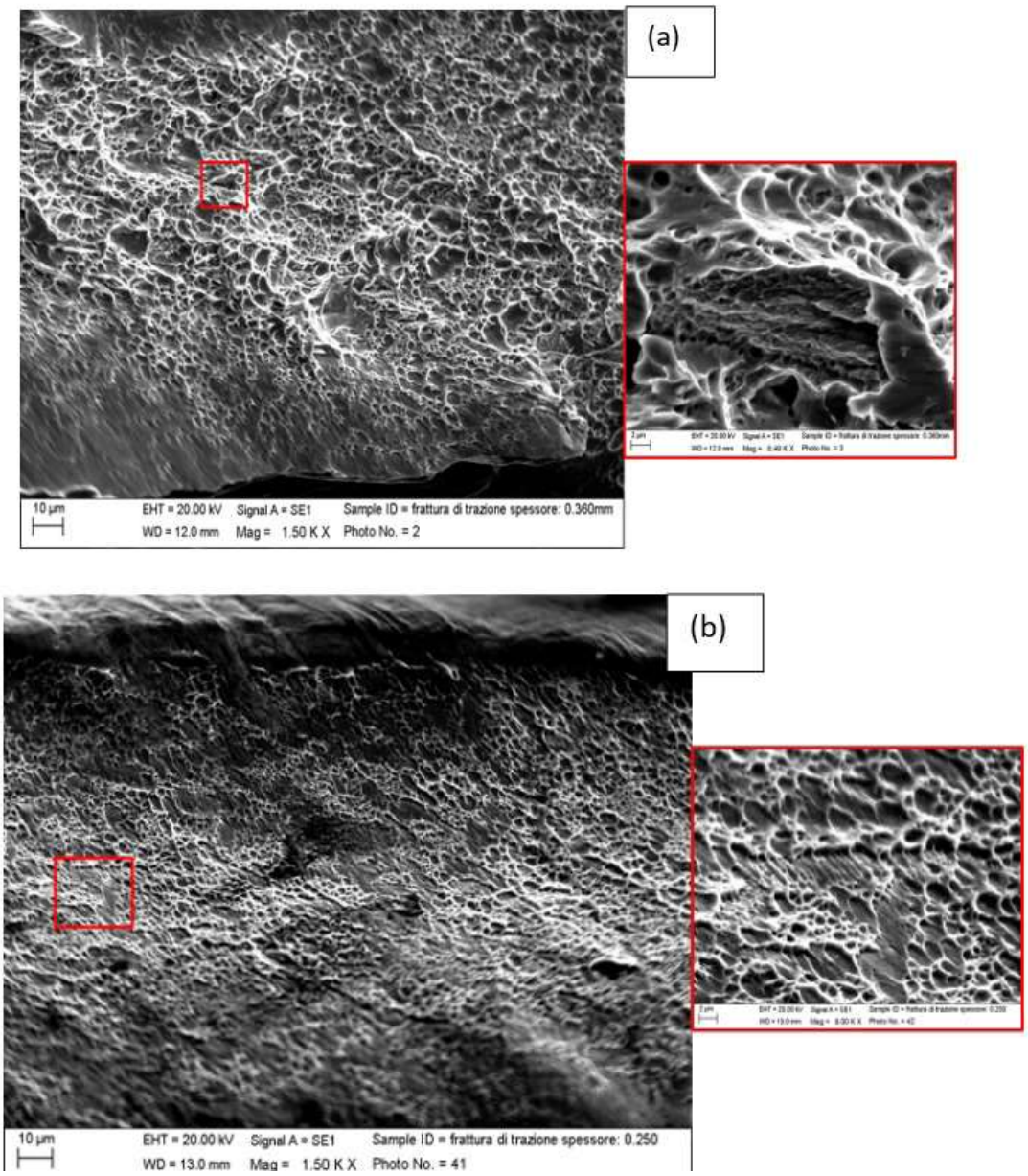

Fig. 2 - Frattographies, SEM-SE. Sample: 0.360annealed (a), 0.250 (b)

As shwon in the pictures of figure 2, all cold rolled samples froom the annealed to the final product show a mostly ductile fracture surface regardless of the deformation rate.

TENSILE TEST

This test represents the routine qualification essay to be made on the final product in order to define its mechanical-strength properties. In the specific case of this research tensile tests were carried out on all intermediate cold rolling steps in order to appreciate the variation of resistance properties at each rolling step. All acquired profiles are shown in Figure 3. The curves are clearly showing a decrease of toughness coherently with the cold rolling progression. The UTS is more and more corresponding to the breaking position and the YS (measured at 0.2 of permanent deformation) is also increasing with a limited variation of the Young modulus.

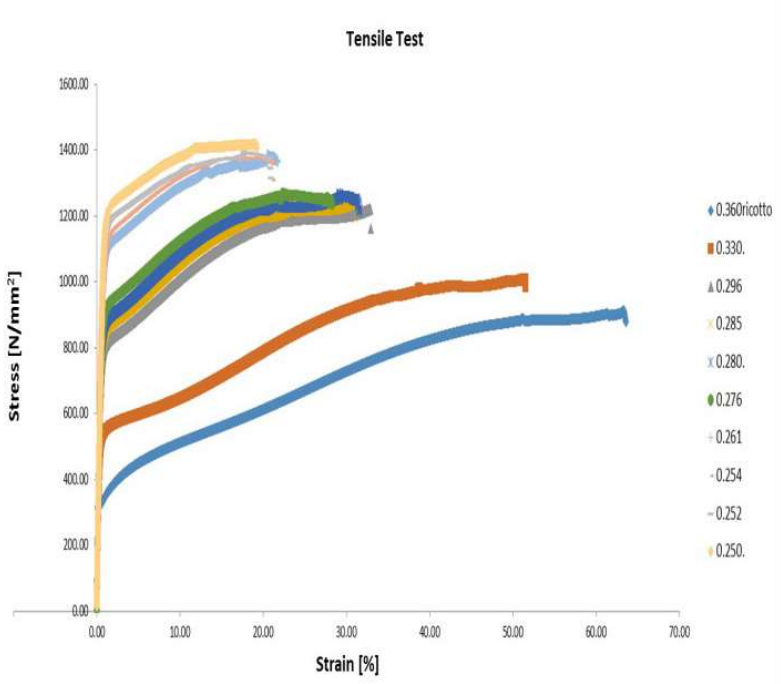

Fig. 3 - Tensile curves of each rolling step of the second cycle

MAGNETOMETER SQUID M VS H MEASURE

Magnetic measurements with dc-SQUID instrument were carried out in order to measure amount (volume fraction) of the magnetic phase (i.e. martensite) in the austenitic matrix. The ferromagnetic phase quantification was performed using a theoretical $M_{saturation}$ value of a standard AISI 301 steel, theoretically containing 100% martensite. To quantify martensite is necessary to assess the sample saturation magnetization. However, the small contribution of austenitic phase magnetic response (the paramagnetic phase) is to be taken into account: the value of austenitic magnetization must be eliminated. For this purpose the linear portion of the sample chart with lower $M_{saturation}$ is plotted, i.e. the sample containing the greater austenite amount (paramagnetic phase). This value was subtracted from each sample, so that the obtained profile is solely referred to martensite magnetization. The martensite value % is calculated by $M_{saturation}$, as shown in table 4.

Tab. 4 - $M_{saturazion}$ graphic interpolation; infinite magnetic field applied.

Sample	$M_{sat.}$(A/m)	Q.$ty_{mart.}$(%)	Msat. 100%martensite (A/m)
0.360	3.07E+04	2.3	1.31E+06
0.330	3.51E+04	2.7	
0.295	1.28E+05	9.7	
0.285	2.02E+05	15.4	
0.280	2.54E+05	19.4	
0.276	3.18E+05	24.2	
0.261	2.96E+05	22.6	
0.254	3.71E+05	28.3	
0.252	4.17E+05	31.8	
0.250	4.68E+05	35.6	

Comparing the estimated martensite volume fraction with the deformation rate (fig. 4) and then with the data acquired by the tensile tests (fig. 5) it is possible to draw some interesting suggestions for the discussion. Figures 4 and 5 graphically describe a tentative combination of data.

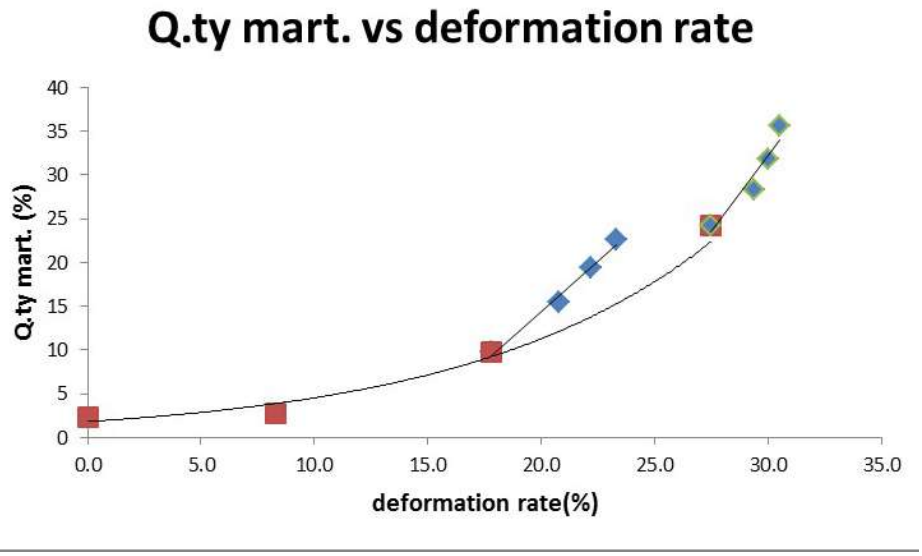

Fig. 4 - Correlation between martensite amount and strain rate; red dots = high $\Delta_{(def.rate)}$; blue dots = low $\Delta_{(def.rate)}$.

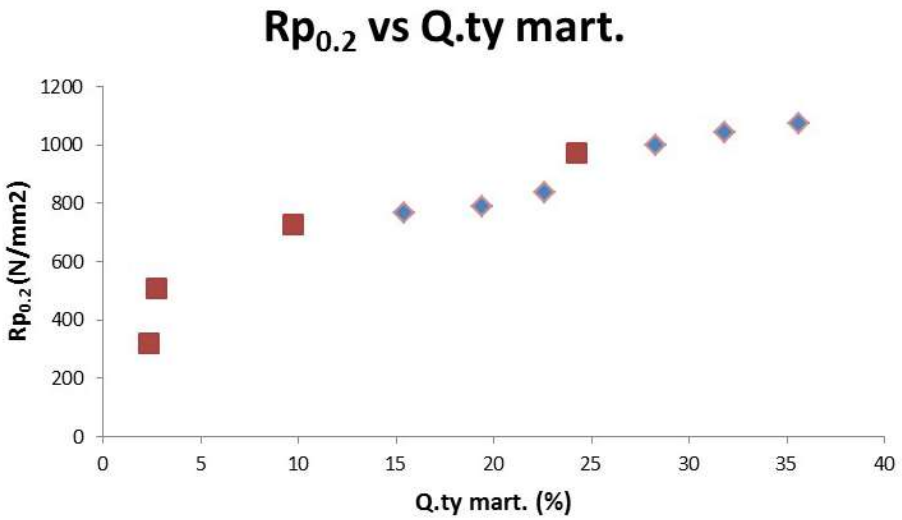

Fig. 5 - YS ($Rp_{0.2}$) and martensite amount correlation; punti red dots = high $\Delta_{(def.rate)}$; blue dots = low $\Delta_{(def.rate)}$.

During the cold deformation the austenitic lattice has two main microstructural modifications: the formation of martensite and the hardening process (e.g. stacking faults, mechanical twins, slip bands). The importance, in terms of how the metal is affected, of the two phenomena is strongly depending from the way the stress is applied on the metal, in other words, from the experimental conditions (eq. 2)

$$E_{Deformation} = E_{Q} + E_{Hardening} + E_{Martensite} + Deformation \qquad (2)$$

where: $E_{Deformation}$ = Energy released during cold rolling; E_{Q} = Energy lost as heat; $E_{Hardening}$ = Energy absorbed as work hardening; $E_{Martensite}$ = Energy used to produce martensite; Deformation = plastic deformation.

The martensite amount exponentially increases as a deformation rate function. In particular high $\Delta_{(def.rate)}$ steps are part of the exponential curve, while low $\Delta_{(def.rate)}$ steps deviate from the aforementioned trend. This leads to the hypothesis that small $\Delta_{(def.rate)}$ involves a deformation mechanism other than the one dominating when there is a large $\Delta_{(def.rate)}$.

The usage of the quantitative analysis results gathered by the SQUID magnetometer it was possible to express the YS can be expressed as martensite amount function, as shown in Figure 5, and thus to estimate if a deviation similar to that of figure 4 was reported. It results that YS, which roughly indicates the elastic limit for these steels, does not vary linearly as a martensite function, as should be expected, but is affected indeed by by a combination of martensite and a secondary effect related to the cold rolling process. In this case it is coherent to suggest that the energy accumulated in the lattice as defects and usually defined under the generic term of work hardening contributes to the definition of the mechanical properties of the steel. This leads to the formulation of the hypothesis that would explain the different transformation involving small $\Delta_{(def.rate)}$ rather than high in the martensitic contribution to the YS: at low $\Delta_{(def.rate)}$ corresponds YS increase.

Equation (2) defines how the energy applied on the metal by cold rolling is handled by the microstructure, the graphs of figures 4 and 5 how the microstructural and mechanical properties are affected by it. The fact that the strain density (amount of deformation applied per rolling step) has to be seriously taken into account becomes thus a key aspect for further interpretation. As one of the main issues of this research work there is the evidence that martensite and work-hardening results from a distribution of the absorbed energy strictly related to the application rate. In particular it has been experimentally noticed that:

- Low $\Delta_{(def.rate)}$ are prone to generate a greater martensite amount, which is formed on energy costs, accumulated during hardening. So the martensite increment is balanced by an energy decrease from work hardening, resulting in only a modest increase in YS.
- High $\Delta_{(deformation\ rate)}$ produce both martensite and hardening, such that the martensite formation is not able to balance the work hardening, producing a steady increase of YS.

Conclusion

The experimental sessions and data gathered during the present research generated the awareness that not enough attention was previously paid to the work hardening of the austenite by accumulation of lattice defects during the manufacturing process. The formation of martensite confirmed to be the most important and evident phenomenon triggered by mechanical stress of the metastable austenitic matrix however the tensile curves measured on several samples issued by the cold rolling process have shown that the martensite volume fraction has to be considered beside other matrix strengthening processes in order to justify the changes in YS.

Further researches should be made in this direction in order to offer a sound contribute to reach the mature knowledge needed to refine the predictive models of Austenitic stainless steels behaviour under stress.

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