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Enhancement of ductility of work hardened strips in AISI 301 austenitic stainless steel

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During plastic deformation metastable austenitic stainless steels undergo a partial transformation from the initial face-centred-cubic austenite phase (γ) to the body-centred-tetragonal martensite (α′), commonly referred to as strain-induced martensite. The martensitic transformation enhances their work hardening capability and allows to obtain engineering materials with high strength and good corrosion resistance (typical of the austenitic stainless steels). These properties make them very attractive for a wide range of applications especially in the automotive and electronic sectors where the components are produced by forming and bending the strips into various shapes. For these specific applications a good ductility is also required besides the high mechanical properties. In this paper, the effect of chemical composition and processing conditions of a work-hardened stainless-steel strip of EN 1.4310 (AISI 301 type) has been studied. Each factor has been analysed both separately and in combination with the others. The role of chemical composition on the formation of strain-induced martensite formation has been evaluated by means of the M_{d30} temperature. Likewise, the effect of intermediate annealing treatments has been investigated by varying the temperature and the furnace atmosphere (N_2 or H_2). All the specimens have been qualified by tensile tests, measurement of the volume fraction of strain-induced martensite and bending tests. The most suitable and effective industrial production cycle for obtaining work hardened flat products with an aimed minimum ductility has been defined.

KEYWORDS: AUSTENITIC STAINLESS STEEL, WORK-HARDENED STATE, STRAIN-INDUCED MARTENSITE, DUCTILITY;

INTRODUCTION

Austenitic stainless steels are widely used in the automotive industry because they provide a combination of high mechanical strength and excellent formability, thus allowing a reduction of vehicle weight and consequently of CO_2 emissions.

Flat products made of austenitic steel EN 1.4310 (type AISI 301) are commonly requested in work-hardened condition. In fact, the chemical composition of this grade promotes the formation of strain-induced martensite during the cold working processes resulting in a consequent high increase of the mechanical properties. Therefore, strips of this material are obtained by a cold rolling process and used in a wide range of industry sectors because of their good corrosion resistance and mechanical characteristics. Most of these applications are related to the production of parts and components which need a high spring effect; for example, in the automotive industry the temper rolled strips are used for producing several items such as strip springs for instru-

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ments, car lighting components, hose clamps, anti-noise shims for the braking system and many others. They are also widely used in the electronic industry (electrical contacts, fine components etc…) as well as in the civil sectors (springs for furniture and windows, kitchen tools, paper clips etc..). However, in some of these cases, the steel strip is pressed and bent in very complicated shapes and its high strength must be combined also with a good ductility. For this reason, the manufacturers, beside the tensile strength, often require the bending resistance to avoid the cracks during forming.

This paper is focused on the production of cold rolled ultra thin precision strips in the thickness 0.25 mm. The process flow typically consists of a first rolling to an intermediate thickness, a bright annealing treatment and a final rolling to achieve the required thickness. The resulting mechanical properties of the work hardened product are affected by the chemical composition, as well as by the production process conditions: thickness reduction percentage, cold rolling practice and annealing parameters mainly.

The aim of this study is to evaluate not only the effect of each factor on the mechanical characteristics but also to define how they can be adjusted to obtain a material with same strength and enhanced ductility and, at the same time, to improve the bending resistance required in the production of specific components.

Mechanical properties of austenitic stainless steels depend strongly on the stability of the austenite matrix. High strength, ductility and toughness can be achieved via the TRIP (transformation induced plasticity) or TWIP (twinning induced plasticity) effects where, respectively, strain induced martensite and mechanical twinning form as a consequence of additional strain. The occurrence of these mechanisms depends on the stacking fault energy (γSFE), the initial microstructure and the deformation conditions [1,2]. The martensitic transformation from austenite (γ) to hexagonal ε martensite and/or α′ martensite occurs if γSFE is typically below 20 mJ/m^2, whereas mechanical twinning is promoted if the stacking fault energy lies between 15 and 30 mJ/m^2. It has been reported that these structures form simultaneously if γSFE lies between 15 and 20 mJ/m^2.

For example, Shen et al. have reported that in the austenitic grades 304 (γSFE=18 mJ/m^2) and 301 (γSFE=14.7 mJ/m^2), ε, α′ and twinning operate at different strains. In particuar, ε martensite and twinning act as intermediate phases in the transformation from γ to α′ martensite [1, 2].

Besides the enhancement of tensile properties, it has to be considered that the existence of α′ martensite is closely related to the hydrogen induced embrittlement of metastable CrNi austenitic steels. The formation of α′ martensite is accompanied by a change of the diffusion conditions of hydrogen atoms within the body-centred cubic (bcc) lattice structure [3]. Hydrogen embrittlement is a knwown phenomenon in materials with high mechanical properties and can cause fracture initiation that is responsible of various macroscopic consequences and in particular of the reduction of ductility [4].

The tendency towards the formation of the strain induced α′ martensite is mainly dependent on chemical composition and deformation temperature. Other factors that can affect the extent of transformation are plastic strain, strain rate, stress state, and grain size. The transformation results in an increased strength, because martensite is stronger than austenite and, at the same time, it can increase the ductility if it occurs rapidly, just prior to the formation of severe strain accumulation. Transformation from austenite to martensite can also occur by two other mechanisms such as spontaneous transformation at low temperatures and stress-assisted nucleation.

Spontaneous transformation occurs when the material is cooled below the martensite start temperature (M_s) as in conventional steels. Slightly above the M_s, stress-assisted martensite can form in response to an applied elastic stress. The temperature above which strain-induced martensite is not produced by plastic deformation is referred to as M_d. The susceptibility of an alloy to strain-induced transformation depends on the stability of austenite.

Nohara et al. (1977) proposed to describe the Md30 temperature of Cr-Ni steels by the following equation:

$$M_{d30}(°C)=551-462\cdot(C+N)-9.2\cdot Si-8.1\cdot Mn-13.7\cdot Cr-29\cdot(Ni+Cu)-18.5\cdot Mo-68\cdot Nb \qquad [1]$$

where all the concentrations are expressed in mass percent. The M_{d30} is defined as the temperature at which an amount of 50% austenite is transformed into martensite by a cold-deformation with true strain equal to 0.30. Equation [1] indicates that interstitially dissolved elements, such as C and N, are particularly strong austenite stabilizers.

A reduction of the temperature increases the thermodynamic driving force for the martensite formation and leads to a higher martensite transformation rate during shaping. Moreover, as temperature decreases, the stacking fault energy reduces, thus promoting a higher martensite formation.

Generally speaking, the austenitic grades with lower alloy content are found to be less stable. For example, 301 and 302 are less stable than 304, 305, and 309 [5-9].

MATERIAL AND METHODS

In this work, a selected set of specimens of the austenitic grade EN 1.4310 (see Tab.1), has been analyzed in order to:

- assess the effect of chemical composition, intermediate annealing parameters and final cold rolling reduction on mechanical properties of the steel in the work hardened state;
- define the required conditions for the production of sheets combining a tensile strength complying with the C1300 level (1300 MPa<Rm<1500 MPa) and a good ductility.

Tab.1 – Chemical composition of the austentic grade EN 1.4310 according to EN 10088-2 standard.

Steel Grade		C (%)	Mn (%)	Cr (%)	Ni (%)	N (%)
EN 1.4310	Min	0.05		16.0	6.0	
	Max	0.15	2.0	19.0	9.5	0.10

The list of the analysed specimens together with the corresponding Md30 temperature and processing conditions is reported in Tab. 2.

Tab. 2 - Analysed specimens.

Specimen ID	Analysed parameter	M_{d30} (°C)	Intermediate annealing treatment			Cold Rolling reduction (%)
			Atmosphere	Temperature (°C)	Time (s)	
1	Chemical composition	29	N_2	1060	39	22
2		21	N_2	1060	39	22
3	Annealing time	30	N_2	1060	26	22
4		30	N_2	1060	39	22
5	Annealing atmosphere	30	N_2	1100	39	22
6		30	N_2	1100	39	22
7	Post treatment (after intermediate annealing)	30	N_2	1060	39	22
8	Post treatment (after final cold rolling)	30	N_2	1060	39	22
9	Cold Reduction	30	N_2	1060	39	26
10		28	N_2	1060	39	19
11	Temperature+Cold Reduction +Annealing time	30	N_2	1040	32	19
12		30	N_2	1040	54	29

All samples have been analysed in the work-hardened state and qualified by means of:

- Tensile tests;
- Measurement of the volume fraction of martensite (ferritoscope);
- Bending tests.

Tensile tests have been carried out according to ISO 6892-1 standard by means of Zwick Roell Z050 testing machine using dog bone specimens, taken parallel to the rolling direction.

Contact bending tests at 180° have been performed on specimens taken parallel to the rolling direction. Bent specimens have been observed by a stereomicroscope (50X). The test is considered passed if no heavy cracks are present. The acceptance criterion is based on the comparison of the specimen surface after bending with the reference images shown in Fig. 1.

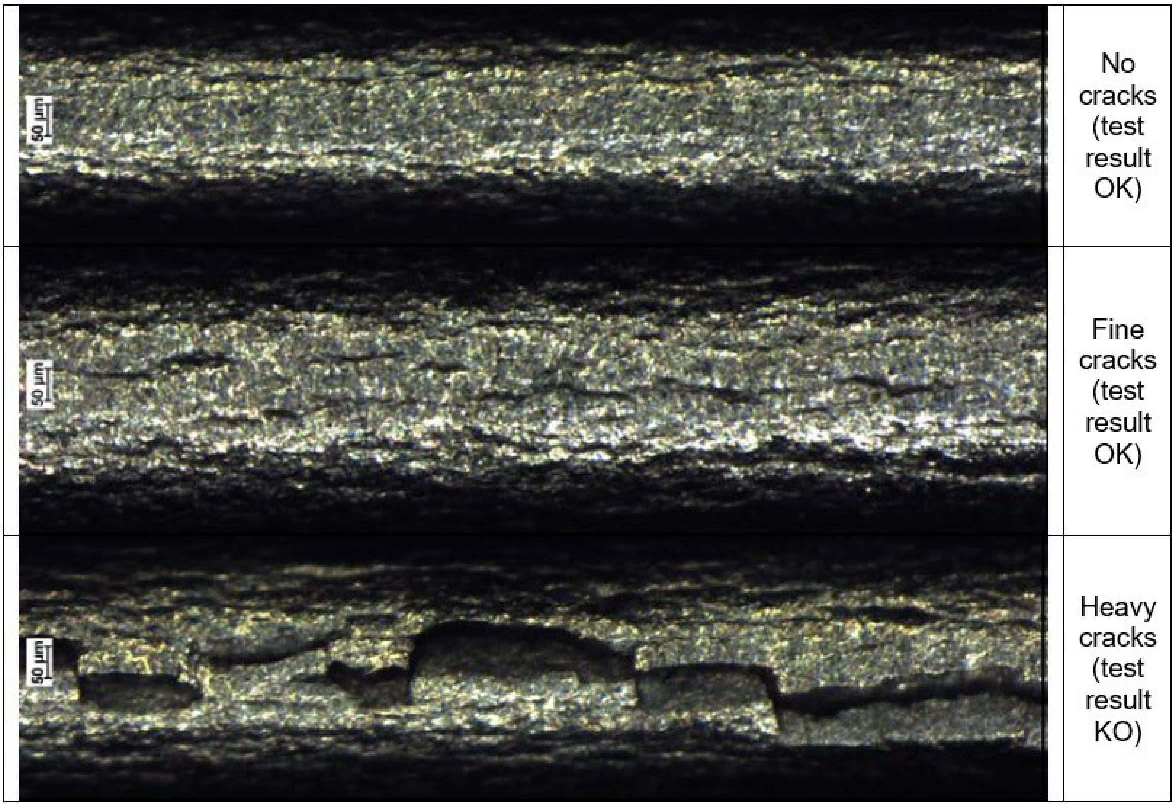


Fig.1 – Reference images for bending test evaluation (stereomicroscope, 50X magnification).

The volume fraction of strain induced martensite has been evaluated by means of a Helmut-Fischer ferritscope, which measures the content of magnetic phases (δ-ferrite and strain-induced martensite) in austenitic and duplex steels according to the magnetic induction method.

In order to asses the effectiveness of the intermediate annealing, selected specimens have been characterised in the annealed state by optical microscope (OM) analysis. The microstructure analysis has been carried out according to ASTM E3 standard by means of a Nikon Metaphot Optical Microscope. Specimens have been observed in longitudinal sections, mechanically polished and electrolytically etched in a nitric-hydrochloric acid solution.

RESULTS

The results of tensile tests, martensite fraction and bending tests of the studied samples are shown in Tab. 3, together with the corresponding values of the calculated M_{d30} temperature.

Tab. 3 - Mechanical properties, volume fraction of martensite and bending test of the analysed specimens.

Specimen ID	M_{d30} (°C)	Intermediate annealing treatment			Cold Rolling reduction (%)	$R_{p0.2}$ (MPa)	R_m (MPa)	Strain induced Martensite (vol.%)	Bending test
		Atmosphere	Temperature (°C)	Time (s)					
1	29	N_2	1060	39	22	1078	1368	5.9	Fine cracks
2	21	N_2	1060	39		1141	1360	6.4	Heavy cracks
3	30	N_2	1060	26	22	1144	1417	10.1	Heavy cracks
4	30	N_2	1060	39		1036	1340	5.2	Heavy cracks
5	30	N_2	1100	39	22	951	1326	4.7	Heavy cracks
6	30	N_2	1100	39	22	1037	1383	5.2	No cracks
7	30	N_2	1060	39	22	965	1408	8.7	Heavy cracks
8	30	N_2	1060	39	22	1073	1379	6.0	Heavy cracks
9	30	N_2	1060	39	26	1111	1435	8.0	Heavy cracks
10	28	N_2	1060	39	19	1017	1356	5.3	Fine cracks
11	30	N_2	1040	32	19	1133	1416	8.8	Heavy cracks
12	30	N_2	1040	54	29	1092	1411	5.7	Heavy cracks

It can be observed that all the specimens are compliant with the C1300 level but the bending resistance is affected by the process parameters.

Samples 1 and 2, produced with same annealing treatment conditions and cold rolling final reduction percentage, show that the effect of chemical composition on the amount of strain-induced martensite is not of primary importance provided that the M_{d30} temperature lies in the range between about 20 and 30°C (considering that the strain induced martensite fractions for both samples are within a strict range of variation and the tensile strength values are within the measurement uncertainty).
It is apparent that the yield strength increases as the martensite fraction increases. In the present case indeed, sample 1 has passed the bending test (Fig.2), whereas sample 2, which exhibits a lower bending resistance and a higher yield strength, failed the test (Fig. 3).

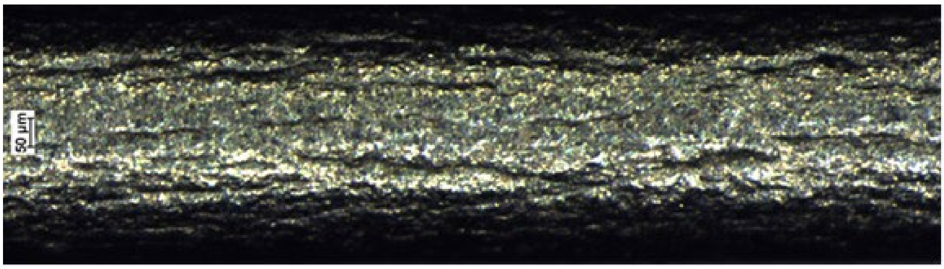


Fig.2 – Stereomicroscopic observation of sample 1 after bending test: OK (fine cracks).

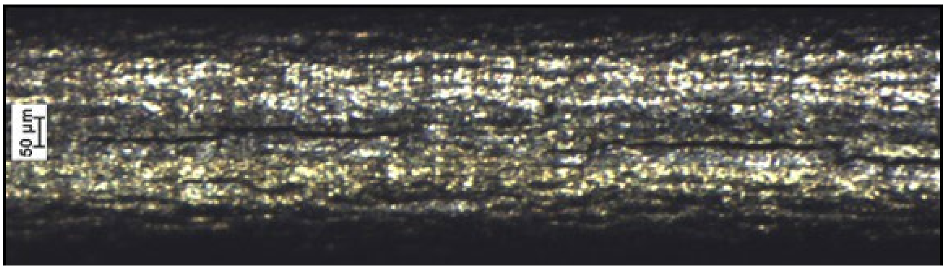


Fig.3 – Observation at stereomicroscope of sample 2 after bending test: KO (heavy cracks).

Qualification of samples 3, 4 and 5 has shown that, if the intermediate annealing is performed in hydrogen atmosphere, the resistance to bending of the final product is impaired whereas changes in time/temperature are not effective. Indeed, only sample 6, annealed in a N2 atmosphere, has passed the bending test.

Qualification of samples 7 (hydrogen desorption treatment after the intermediate annealing) and 8 (hydrogen desorption treatment after cold rolling) has shown that this treatment is effective in promoting the hydrogen desorption since a recovery of the ductility in terms of elongation to rupture (tab. 4) is observed. On the other hand, the resistance to bending still remains poor. Moreover, it is noticed that the post treatment is more effective if it is carried out on the final product because it improves the resistance to bending although, in this case, it is shifted towards the lower limit of acceptance.

The qualification of samples 9 and 10 has shown that a decrease of the cold rolling reduction down to 19% enhances the resistance to bending but tensile strength is also reduced, although still being within the limits of the C1300 level. If the cold rolling rate is increased to 26%, the highest tensile strength is achieved but with poor ductility in terms of resistance to bending and a higher volume fraction of strain induced martensite.

The combination of a lower annealing temperature and cold rolling rate realized on samples 11 and 12 appears to have detrimental effect on ductility since it induces a poor resistance to bending.

Tab. 4 - Comparison of mechanical properties (tensile strength and elongation to rupture) of the specimens annealed in H_2 before/after post treatmen(200°C x 2h).

Specimen ID	Intermediate annealing treatment			$R_{p0.2}$ (MPa)	R_m (MPa)	A (%)	Bending test
	Atmosphere	Temperature (°C)	Time (s)				
7	N_2	1060	39	965	1408	16.4	Heavy cracks
7P (post treatment after intermediate annealing)				920	1367	18.8	Heavy cracks
8	N_2	1060	39	1073	1379	16.8	Heavy cracks
8P (post treatment after cold rolling)				1134	1330	22.0	Fine cracks

DISCUSSION

The matrix of experimental tests has been designed in such a way to isolate the effect of a single compositional or processing parameter to permit a straightforward analysis of the results.

Samples 1 and 2 differ for the M_{d30} temperature and have been manufactured with the same processing conditions. To assess the effect of a single processing parameter such as the annealing atmosphere, the temperature and time of the intermediate annealing treatment and the final cold rolling reduction, specimens having similar M_{d30} and different process parameters have been selected (samples 3 to 12).

In particular, samples 3, 4, 5 and 6 differ in the time of heat treatment and annealing atmosphere. Samples 7 and 8, annealed in H_2 atmosphere, have been submitted to a heat treatment at 200°C for 2 hours to promote hydrogen desorption. The heat treatment has been performed after the intermediate annealing in sample 7 and after the final cold rolling in sample 8. In case of sample 7 the final cold rolling has been performed in the laboratory.

Samples 9 and 10 differ only for the final cold reduction rates. Samples 11 and 12 differ from the former ones for the temperature and duration of the intermediate annealing treatment and, among them, also for the final cold rolling reduction.

The experimental activity has shown that an intermediate annealing in hydrogen atmosphere impairs the ductility of the material, probably because the strain-induced martensite interfaces formed during the first cold rolling step act as trapping sites for hydrogen.

The bending resistance of specimens annealed in N_2 atmosphere is shown in the histrogram of Fig. 4 as a function of the processing parameters, while the tensile strength is represented in Fig. 5. It can be observed that, if the annealing treatment is carried out at the lowest temperature 1040°C, a poor resistance to bending is obtained even when a longer annealing time is used (54 s instead of 32 s) and the R_m value is close to the upper threshold for the C1300 level. When the annealing treatment is performed at 1060°C, the resistance to bending is close to the acceptance threshold provided that the cold rolling reduction is in the range between 19% and 22%. In fact, a poor bending resistance and high R_m value is found when the cold rolling reduction is raised to 26%. The highest resistance to bending combined with good tensile strength is observed when the annealing treatment is performed at 1100°C and the cold rolling rate is 22%.

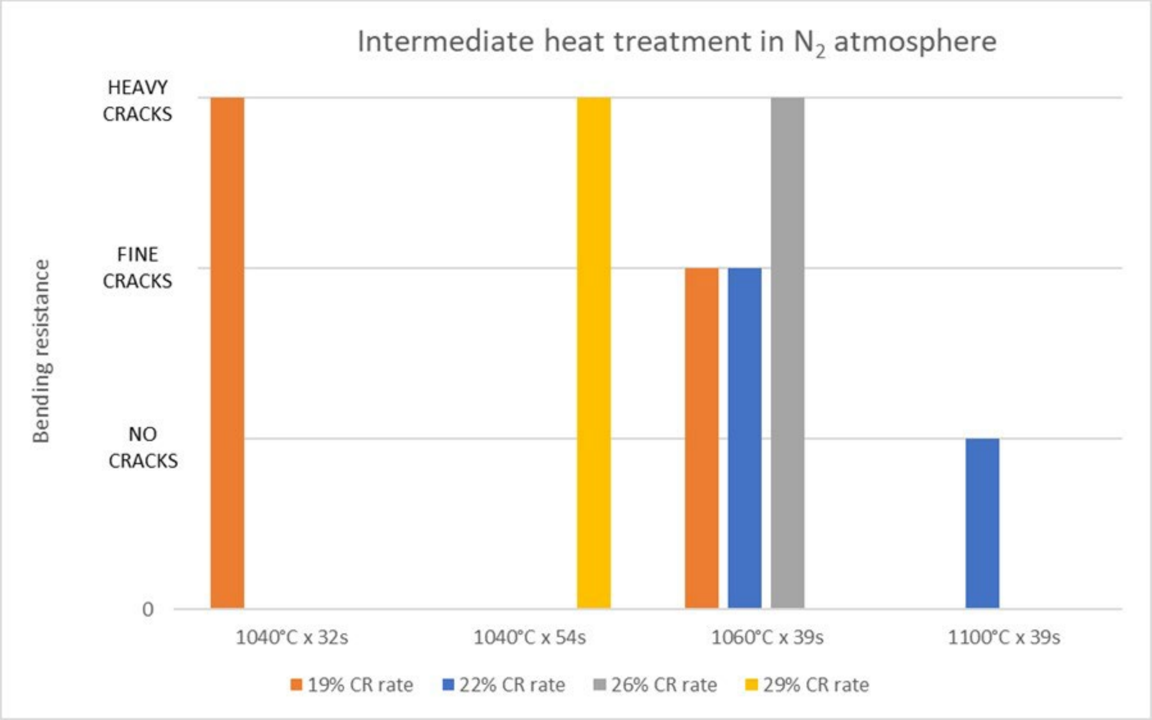


Fig.4 – Resistance to bending as a function of the processing parameters.

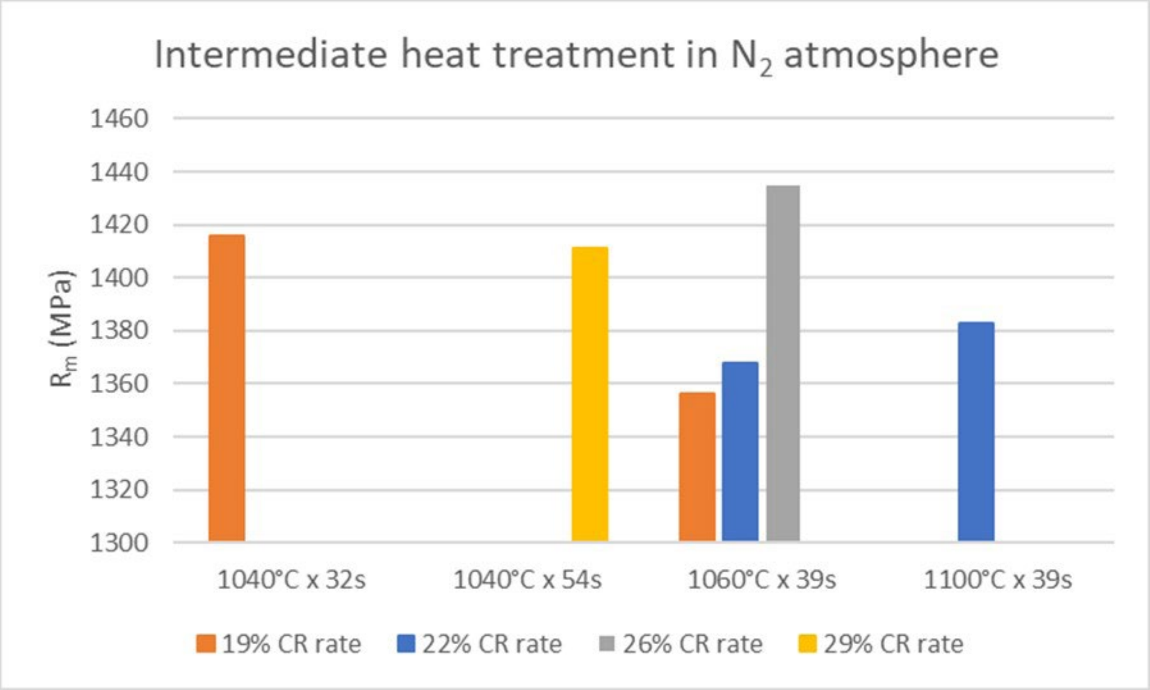
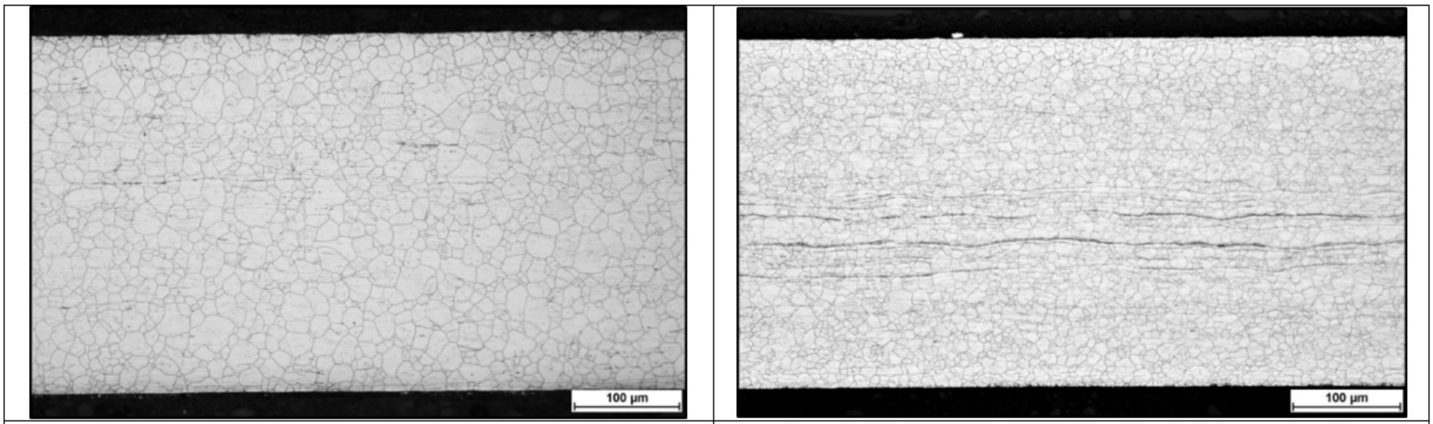


Fig.5 – Tensile strength as a function of the processing parameters.

It has been noticed that the volume fraction of strain-induced martensite is always higher in all the samples exhibiting a poor bending resistance than that formed in those passing the test. This is probably due to a low effectiveness of the intermediate annealing treatment in reverting it back to austenite.

Moreover, as shown in Fig. 6, the recrystallised structure of sample 6, annealed at a higher temperature, is characterised by a larger average grain size (9.5 ASTM) and by a lower average through-thickness hardness of 196 HV than specimen 12 annealed at lower temperature (10.5-11.0 ASTM) and whose average through-thickness hardness is 215 HV. The former has passed the bending test, whereas the latter has failed it.

This supports the hypothesis that a more effective intermediate annealing favours the ductility of the material in the work hardened state and therefore its ability to resist bending.



Sample 6 (1100°C x 38s): Bending test: no cracks ASTM average grain size: 9.5 Hardness: 196Vickers (average through-thickness)	Sample 12 (1040°C x 32s): Bending test: heavy cracks ASTM average grain size: 10.5/11 Hardness: 215Vickers (average through-thickness)

Fig.6 – Microstructure of two specimens annealed at different times/temperatures (Optical Microscope).

CONCLUSION

Flat products in austenitic steel EN 1.4310 (type AISI 301) are commonly manufactured in the work hardened condition because the strain-induced martensite formed during cold working allows to obtain a high increase of the mechanical properties. They are used in a wide range of industry sectors thanks to the combination of good corrosion resistance and mechanical strength. Most of the applications are in the automotive industry where the sheets are cold formed in very complex shapes, and this requires the high mechanical strength to be associated with good ductility.

In this study, the effect of chemical composition, intermediate annealing and final cold rolling reduction on the mechanical properties of this steel in the work hardened state have been analysed.

Laboratory investigations have shown that the atmosphere of the intermediate annealing treatment is of primary importance for the production of material compliant with the required high mechanical properties while maintaining a good ductility and resistance to bending. It has been observed that the annealing in hydrogen atmosphere has a detrimental effect on the bending resistance of the final product because the strain-induced martensite interfaces, formed during the first cold rolling step, act as trapping sites for hydrogen thus promoting the formation of cracks during bending.

In order to obtain a material compliant with the C1300 level while maintaining a good ductility and resistance to bending test, the following conditions should be fulfilled:

- the chemical composition should be balanced so that an M_{d30} of 28-30°C is obtained;
- the intermediate annealing should be carried out in N_2 atmosphere;
- the minimum recrystallization annealing temperature is 1060°C;
- the final cold rolling reduction should be in the range between 19% and 22%.

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