# Improvement of mechanical properties on metastable stainless steels by reversion heat treatments



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# Improvement of mechanical properties on metastable stainless steels by reversion heat treatments

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**Abstract.** AISI 301LN is a metastable austenitic stainless steel that offers an excellent combination of high strength and ductility. This stainless grade is currently used in applications where severe forming operations are required, such as automotive bodies. When these metastable steels are plastically deformed at room temperature, for example by cold rolling, austenite transforms to martensite and, as a result, yield strength increases but ductility is reduced. Grain refinement is the only method that allows improving strength and ductility simultaneously. Several researchers have demonstrated that fine grain AISI 301LN can be obtained by heat treatment after cold rolling. This heat treatment is called reversion because it provokes the reversion of strain induced martensite to austenite. In the present work, sheets of AISI 301LN previously subjected to 20% of cold rolling reduction were treated and a refined grain austenitic microstructure was obtained. Mechanical properties, including fatigue limit, were determined and compared with those corresponding to the steel both before and after the cold rolling.

## 1. Introduction

Metastable steels are highly suitable for the fabrication of automotive components, since they have good formability and, after forming by processes like stamping, hydroforming or cold rolling, the components achieve a high strength, required to meet the safety standards regarding crash behavior. In the particular case of metastable austenitic stainless steels, the plastic deformation induces the transformation from austenite to martensite, which increases the hardening coefficient and gives the material an extraordinary energy absorption capacity [1]. Due to these outstanding characteristics, AISI 301 grade is widely used for construction of lightweight train structures [2], bus carriages [3], honeycomb structures and it is been introducing in the automotive industry for impact supporting frames such as the B-pillar [4].

Furthermore, various authors have shown that it is possible to simultaneously improve strength and ductility of metastable austenitic steels by means of heat treatments [1,5-7]. These treatments are applied to previously cold worked steels with the purpose of achieving recrystallization of the predeformed austenite and simultaneous reversion of the strain induced martensite, so that an austenitic microstructure of ultrafine [6] or even nanocrystalline [7] grain size can be obtained.

Based on the state of the art above described, the present work deals first with the determination of optimal conditions for reversion treatments on a stainless steel AISI 301LN after a 20% thickness reduction by cold rolling. Once these reversion treatment conditions were established, tensile properties, hardness and fatigue limits, were measured for the fine grain steel accordingly obtained.

## 2. Materials and Experimental Procedure

### *2.1. Material*

The stainless steel AISI 301LN, with the designation EN 1.4318 according to the European standard, was provided by OCAS NV, Arcelor-Mittal R&D Industry Gent (Belgium) as 1.5 mm thick sheets. Its chemical composition is presented in table 1.

**Table 1.** Chemical composition of the steel AISI 301LN (wt %).

| C | Cr | Ni | Mo | Mn | Si | N |
|---|---|---|---|---|---|---|
| 0.03 | 17.5 | 7.10 | 0.10 | 1.48 | 0.50 | 0.14 |

The material was provided in two conditions:

a) Cold rolled, with subsequent solution annealing (fast cooling from temperatures around 1050 ºC), a treatment aiming at a completely austenitic microstructure free of second phase precipitations.
b) As before, but subjected to a final cold rolling with a thickness reduction of 20%, process resulting in a microstructure consisting of an austenitic matrix besides deformation induced martensite plates.

### *2.2. Reversion treatments*

In agreement with literature [1,5-7] as well as with previous research carried out by the authors of this paper [8], the temperature was chosen in the range between 600 and 850 ºC. At lower temperatures martensite reversion cannot be totally achieved, while at higher temperatures the recrystallized austenite grains may grow too fast. The holding times were 1, 5 and 10 minutes, with exception of the test at the highest temperature, where the time was shortened to 30 s and 1 min.

Tensile tests were carried out with samples treated in an electric resistance oven. Samples were introduced into the oven once the specified holding temperature had stabilized, while the quenching was carried out by fast immersion into water.

### *2.3. Microstructural characterization*

In case of metastable steels grinding and mechanical polishing can induce martensite formation at the sample surface. In order to avoid this effect, electropolishing in a nitric acid solution was performed. It was not possible to reveal simultaneously austenite and martensite. Thus, the same etching solution was used for electrolytic etching to visualize austenite grains, while martensite was revealed by immersion in Beraha solution [9].

Since micrographic quantification of the martensite fraction induced by cold rolling is little reliable, X-ray diffraction was applied, by using the copper radiation, and the phase fractions were calculated with the RIR method (*Reference Intensity Ratio*), as explained in detail in reference [10].

### *2.4. Mechanical properties*

Vickers hardness testing with a load of 100 g was performed. A minimum of six indentations for each material condition were done. Subsequently, tensile tests were carried out with three specimens per condition. All tensile specimens were oriented longitudinally, that is with the tensile axis parallel to the cold rolling direction. Test rate was of 3 mm/min.

Considering the tensile strength, a testing procedure based on the staircase (or up-and-down) method was applied to determine the fatigue limits of AISI 301LN in the different material conditions [11]. The first step consisted in applying a maximum load ($\sigma_{max}$) of around 55% of the ultimate tensile strength of the corresponding steel condition. If the specimen failed before infinite life (that is $2x10^6$

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cycles), the next specimen had to be tested at a 5% lower stress level. If the specimen did not fail, the next test was run at a higher stress level with the same specimen, and so on until fracture. Despite it is recommended to run the staircase with at least 15 specimens, a more reduced number (at least 8 per condition) were used for this research. Tests were conducted in a resonant testing machine at frequencies around 150 Hz with a stress ratio ($R= \sigma_{min}/\sigma_{max}$) of 0.1. Fatigue specimens were cut by laser from the sheets. Laser cutting is widely used in the metal industry, particularly when components of high strength steel sheets are produced. On the other hand, the roughness of cut edges produced by laser differs from that obtained by mechanical cutting, and this may influence the fatigue performance. Therefore, fatigue specimens were carefully polished up to $R_a$ values under 1 μm before the tests.

## 3. Results and Discussion

### *3.1. Microstructural characterization*

Figure 1 shows a micrograph of the sheet surface in annealed condition. The microstructure is almost completely austenitic, with equiaxial grains of 11.7 ± 4 μm mean size and abundant presence of twins. After cold rolling the microstructure remained similar with respect to grain size, but the martensite volume fraction determined from X-ray diffractograms was 28%.

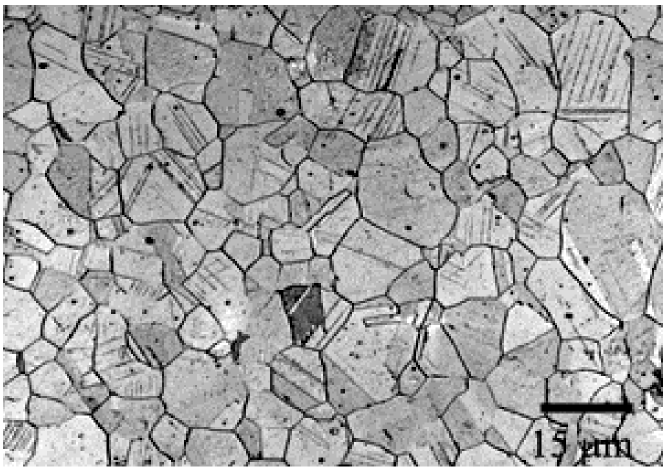



**Figure 1**. Micrograph of the AISI 301LN steel in annealed condition.

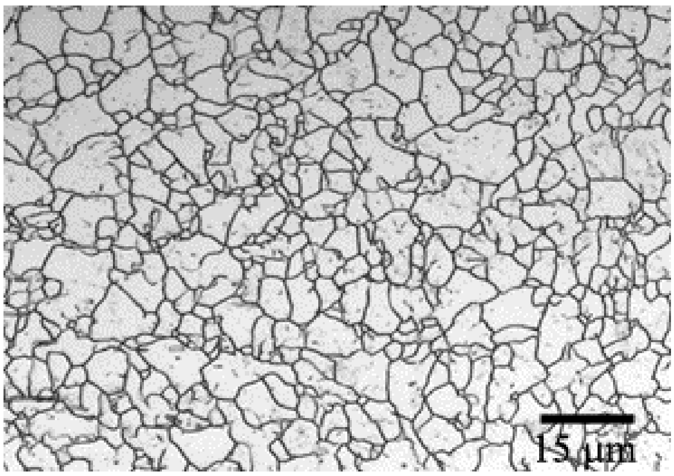



**Figure 2**. Micrograph of the AISI 301LN after a reversion treatment of 10 minutes at 750 ºC.

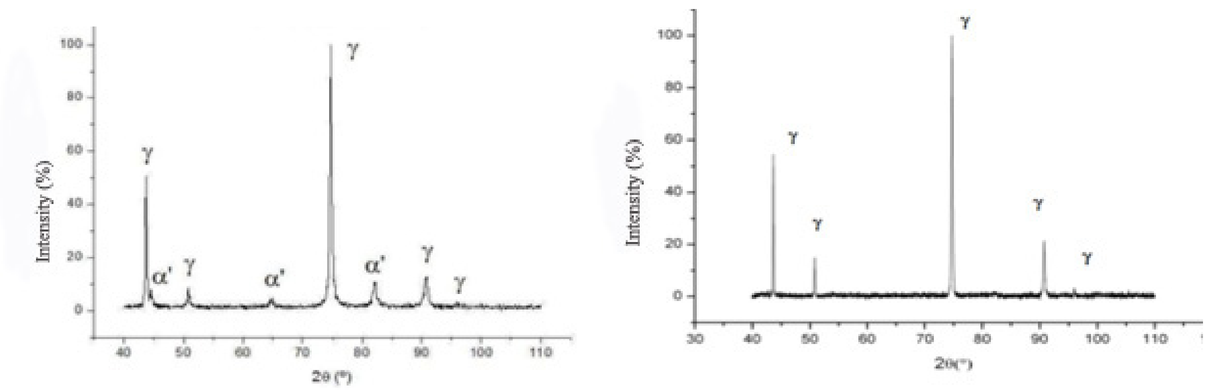
The respective values of the achieved grain sizes after reversion heat treatments are given in table 2. From these results, it is evident that all the treatments produce a decrease in grain size. However, the best grain refining was achieved at 750 ºC and a holding time of 10 minutes. Therefore, these were the heat treatment conditions selected to perform the mechanical characterization. At this temperature there is no risk of growing for the recrystallized austenitic grain, since Di Schino et al. [5] showed that this event will take place only at temperatures above 900 ºC.

Figure 2 shows the microstructure after reversion at the optimal conditions and allows comparing it with the annealed steel at the same magnification (Figure 1). Grain size distribution indicates a predominance of ultrafine grains (size between 0.5 and 1.5 µm), but coexisting with bigger grains (probably austenitic grains which did not recrystallize during the reversion treatment). This heterogeneous distribution is reflected in the dispersion level, which is almost of the same value as the average grain size: 2.9 ± 2.8 µm.

**Table 2.** Average austenitic grain sizes (in µm) resulting from reversion heat treatments.

| t (min) / T (ºC) | 600 | 700 | 750 | 850 |
|---|---|---|---|---|
| **0.5** | - | - | - | 4.6 |
| **1** | 7.8 | 7.8 | 4.0 | 4.9 |
| **5** | 7.7 | 8.7 | 3.7 | - |
| **10** | 7.5 | 7.5 | 2.9 | - |

Concerning martensite dissolution, neither α'-martensite phase nor ε–martensite were observed after the reversion treatment. Figure 3 compares the XRD spectrum corresponding to the cold rolled condition, where α' peaks are present, with the one for the steel after reversion where only austenite peaks appear.




**a)** **b)**

**Figure 3**. X-ray diffraction spectra of AISI 301LN steel: a) Cold rolled; b) After reversion treatment.

*3.2. Mechanical characterization*

Table 3 compares the values of hardness, yield strength, ultimate tensile strength and elongation for AISI 301LN in the two initial conditions, as well as after reversion treatment of the cold rolled material.

Values in table 3 show the excellent combination of static properties exhibited by the 301LN steel after the reversion treatment. Especially outstanding is its yield strength which more than doubles the value corresponding to the annealed condition. On the other hand, the hardness and grain size values of the completely austenitic microstructures, i.e. annealed and reversion conditions, are in good agreement with Di Schino et al. [5], who reported the hardness of AISI 301 steels to depend on the grain size according to the Hall-Petch relationship for grains larger than 3 μm. For finer grains a saturation value around 330 HV0.1 was found.

Table 4 gives the fatigue limits obtained after statistical calculations based on the staircase tests performed. Results are indicated both in terms of stress amplitude, as well as maximum applied stress. After reversion the steel exhibits an improvement of almost 100 MPa (in terms of $\Delta\sigma/2$), that represents a degree of 36%, as compared to the annealed condition. This substantial improvement of fatigue resistance by grain size refinement is consistent with earlier investigations [12]. The ratios between maximum stress in fatigue to yield stress and also to ultimate tensile strength are indicated in table 4 as well. Cold rolled and reversion conditions exhibit similar ratios, whereas annealed steel has an elevated ratio when yield stress is considered, but low for the relationship $\sigma_{max}/\sigma_{uts}$. This fact is associated with the very high strain hardening coefficient *n* in the annealed condition, which has an average value of 0.55, but reaches instantaneous values close to 0.80, as determined by the authors in [13].

**Table 3**. Hardness and tensile properties of AISI 301LN in the different conditions.

| Condition | Hardness (HV0.1) | $\sigma_{ys}$ (MPa) | $\sigma_{uts}$ (MPa) | Elongation (%) |
|---|---|---|---|---|
| **Annealed** | 246 ± 12 | 360 | 902 | 40 |
| **Cold rolled** | 408 ± 30 | 926 | 1113 | 24 |
| **Reversion** | 314 ± 11 | 749 | 1010 | 33 |

**Table 4**. Fatigue limit values of AISI 301LN in the different conditions.

| Condition | Fatigue limit, $\Delta\sigma/2$ (MPa) | Fatigue limit, $\sigma_{max}$ (MPa) | $\sigma_{max}$ / $\sigma_{ys}$ (%) | $\sigma_{max}$ / $\sigma_{uts}$ (%) |
|---|---|---|---|---|
| **Annealed** | 256 ± 25 | 570 ± 56 | 1.58 | 0.63 |
| **Cold rolled** | 403 ± 43 | 895 ± 95 | 0.97 | 0.80 |
| **Reversion** | 349 ± 25 | 775 ± 56 | 1.03 | 0.77 |

## 4. Conclusions

The results of this work show the viability of reversion heat treatments as a method to achieve a grain refinement in metastable stainless steels. In the present case, cold rolled AISI 301LN steel with a martensite fraction of 28% and an initial austenitic grain size of 12 μm was studied. The first part of the research pointed out that the optimal reversion treatment conditions were 10 minutes at 750 °C. This reversion treatment led to an austenitic mean grain size below 3 μm, although with a very heterogeneous distribution, and also to a complete disappearance of the martensite. The second part of the study proved that this fine grain steel has an excellent combination of mechanical properties, featuring a yield strength which doubles that of the annealed condition, and also a fatigue limit improvement of 36%.

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