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Effect of sub-zero deformation temperatures and rolling directions on the formability of AISI 316 stainless steel sheets

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Abstract. Sheet forming carried out at sub-zero temperatures is gaining more and more interest for deforming metal sheets as it can represent an alternative to room temperature forming to increase the sheet metal formability and to high-temperature forming to avoid the need for subsequent heat treatments aimed at restoring the alloy microstructural characteristics. In this framework, the present work deals with mechanical testing of AISI 316 stainless steel sheets in a wide range of temperatures, namely from -100°C to 700°C, and rolling directions. After mechanical testing, the strain at ultimate tensile strength, identified as an indicator of steel formability, was measured at varying process conditions. Then, the samples were analyzed in terms of microstructural features, micro-hardness, and martensite formation. The obtained results showed that deforming at -50°C induced a substantial increase in formability preserving the hardness, compared to testing at higher temperatures.

Introduction

Austenitic stainless steels are used in several industrial sectors thanks to some characteristics they offer, such as high corrosion resistance, good ductility, and reasonable cost, even if their mechanical strength is quite low due to the face-centered cubic (FCC) austenite phase they present. During cold deformation processes, the phenomenon called strain-induced martensitic transformation (SIMT) may occur, which leads to the steel mechanical resistance increase but also reduces its ductility and corrosion resistance. When forming corrosion-resistant parts, SIMT needs to be decreased, which can be fulfilled through forming in the warm temperature range, which includes temperatures below the steel recrystallization one. For example, in [1], it was proved that forming AISI 304 stainless steel sheets at temperatures lower than 150 °C can help in SIMT suppression and formability enhancement. However, it is well known that warm forming decreases the work hardening and, therefore, the part's final strength. To this regard, in very recent years, sheet forming processes at temperatures below room one have been investigated with two main aims, namely i) to delay the necking onset in view of enhancing the sheet uniform elongation characteristics, and ii) to avoid possible heat treatments that can become mandatory when forming at elevated temperatures in order to recover the sheet initial mechanical and microstructural characteristics.

Literature records are available describing the application of these forming processes to mainly aluminum alloy sheets. AA7075 aluminium alloy sheets, deformed in [2] at varying stress triaxiality and temperature ranging from -100° and 400 °C, showing that the mechanical characteristics at necking were improved when deforming below the room temperature compared to high temperature testing, as a consequence of the formation of a higher amount of precipitates and suppression of dynamic recovery. AA7075 sheets were deformed in [3] to analyze the damage and fracture behaviour at cryogenic temperatures making use of a phenomenological fracture

model; the validation of the latter through experimental trials carried out on U-shaped parts proved lower damage values in the part when cryogenic formed.

On the contrary, the performances of sheets other than those made of aluminum alloys under cryogenic temperatures have been scarcely investigated. In addition, just metals with a FCC structure have been addressed, pointing out a significant sensitivity of this crystal lattice to sub-zero temperature forming.

In this view, to enlarge the knowledge about the metal sheet response to sub-zero temperatures, the paper focuses on the effect of sub-zero deformation temperatures on the mechanical behaviour of austenitic stainless steel sheets. In particular, the AISI 316 sheets were strained under uni-axial tensile testing conditions in a wide range of temperatures, spanning from -100 °C to 700 °C and their response in terms of stress and deformation at necking as well at fracture was evaluated together with the post-deformation micro-hardness. The effect of the rolling direction was taken into account as well. The sheet microstructural features were analyzed and correlated to the mechanical response in order to explain the different behavior at varying testing temperatures.

Experimental procedures

Material

The material under investigation was the AISI 316 stainless steel supplied in form of sheets of 1 mm thickness. Its nominal chemical composition in the as-received condition is reported in Table 1.

Table 1. Chemical composition of the AISI 316 sheets (weight %) [4].

Fe	Cr	Ni	Mo	Mn	Si	C
67.69	16.63	10.85	2.42	0.38	1.28	0.018

Uniaxial tensile tests

The universal test frame (MTS^{TM} 322) was used to perform uniaxial tensile tests at different temperatures. Mechanical tests were conducted at temperatures ranging from 700 to −100 °C (including 25, -50, and 300 °C) at a fixed strain rate of 0.1 s^{-1}.

The sub-zero temperature tests were carried out by using an environmental chamber connected to a liquid nitrogen (LN_2) tank, as visible in Fig. 1a. Fig. 1b offers a magnified view of the dog-bone sample tightly fixed to the gauges. Liquid nitrogen was used as a refrigerant and its flow rate was digitally controlled to maintain the testing temperature with an accuracy of ± 2 °C. Uniaxial tests at elevated temperatures were performed with the same testing apparatus using a resistance heating system to heat the sample to the testing temperature.

The tensile tests were carried out using dog-bone-shaped samples, characterized by a nominal gauge length of 65 ± 0.1 mm and a nominal gauge width of 12 ± 0.05 mm, as prescribed in the ISO 6892 standard [5]. The samples were water-jet cut from the rolled sheets along the sheet rolling direction and the orthogonal one. The former samples will be hereinafter called *0deg* samples whereas the latter *90deg* samples.

In Fig. 1c the rolling direction (RD) refers to the direction of motion of the rolling plate, the transverse direction (TD) refers to the direction that parallel the rolling plate, and the normal direction (ND) refers to the direction that is perpendicular to the rolling plate. Given that, under uniaxial tensile testing, RD and TD represent the resistant section of the *0deg* and *90deg* samples, respectively.

At the industrial level, uniform elongation is the usual indicator of metal sheet formability. For this reason, for each testing condition, two sets of experiments were carried out, namely one at fracture to provide the whole engineering stress-strain curve and, thus, identify the ultimate tensile strength (UTS) for each testing temperature, and the other at UTS to assess the material characteristics at the necking occurrence.

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To have insights into the overall sheet ductility, the fracture surfaces were inspected using FEI™ QUANTA 450 Scanning Electron Microscope (SEM) with the Secondary Electron (SE) probe. An image at 100X magnification was acquired for each sample to evaluate the deformation at fracture.

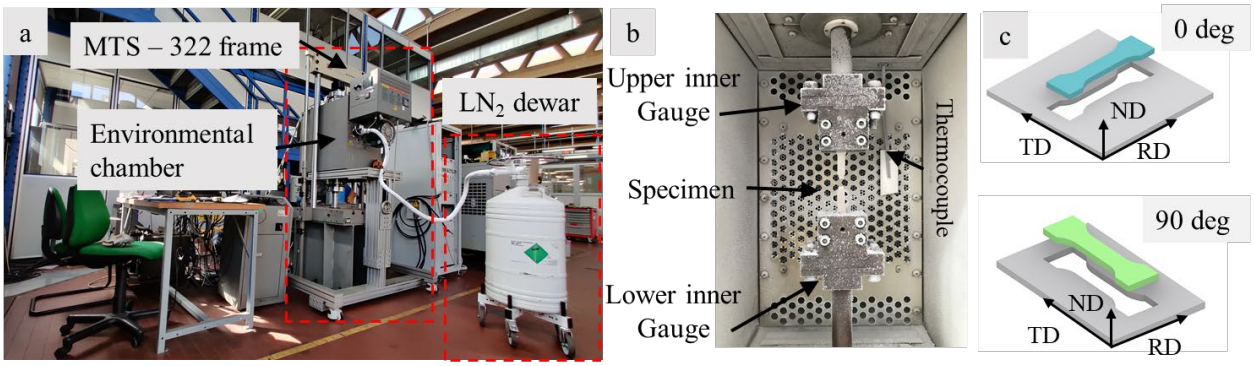

Figure 1. (a) Uni-axial tensile test equipment for sub-zero and elevated temperature testing, (b) zoom of the specimen fixed on the machine gauges, and (c) geometries of the samples.

Characterization methods after tensile testing

The samples strained at UTS were further characterized to gain more information about the formability of the AISI 316 at varying temperatures.

Firstly, the samples were cut, hot-mounted, and then ground with silicon carbide abrasive papers P320, P500, P800, P1200, P2400, P4000. Afterward, a finishing polishing pass was performed with a napped cloth using a diamond slurry. The polished samples were then etched twice to evidence: (i) the austenite grain boundaries, and ii) the possible presence of martensite. The first etching was electrolytic with a solution of 65% nitric acid (HNO_3) in water at 1 V for 10 s while the second one used Behara's solution (100 ml H_2O, 20 ml HCl, 1 g $K_2S_2O_5$) for 10 s.

The etched samples were observed through an optical microscope (Leica DMRE™) and their grain size was determined using the linear intercept method according to ASTM E112 [6].

Later on, an X-ray diffractometer (Siemens™ D500), equipped with a monochromator on the detector side and a Cu radiation tube, was used to quantify the martensitic phase percentage in the samples strained at UTS. The 2Θ = 40–100 ° angular range was investigated with a step scan of 0.04 ° and a counting time of 8 s.

The Vickers micro-hardness of the samples strained at UTS was measured using a Leitz Durimet™ micro-hardness tester with a load of 50 gr for 30 s; five values for each section were recorded and then the average value was calculated.

Results & Discussion

Mechanical properties

The engineering tensile curves of the AISI 316 samples are shown in Fig. 2 at varying temperatures and rolling directions. All the curves exhibited the same trend, except for the -100 °C curve presenting an S-shaped, more evident in the case of the 90 deg sample. Such peculiar behavior can be attributed to the SIMT effect that occurs at sub-zero temperatures.

A parameter conventionally used to predict the stability of the austenite phase with respect to martensite one is M_{d30}, namely the temperature at which 50 % of martensite is formed at a true strain of 0.3 [7]. The calculated M_{d30} of the AISI 316 settles to -83 °C, meaning that a fully austenitic structure cannot be kept at -100 °C during plastic deformation, which is consistent with the S-shape of the AISI 316 curves at -100 °C.

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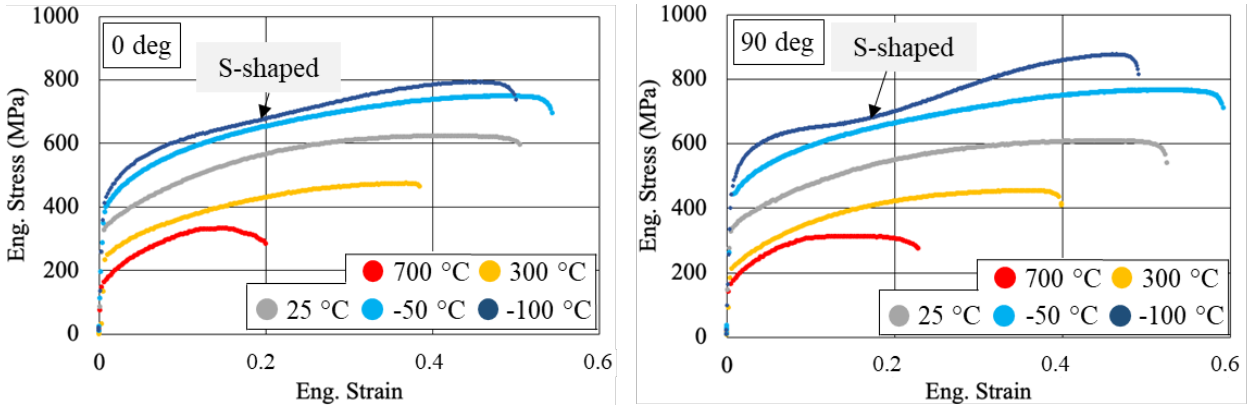

Figure 2. Tensile curves at varying temperatures and rolling directions.

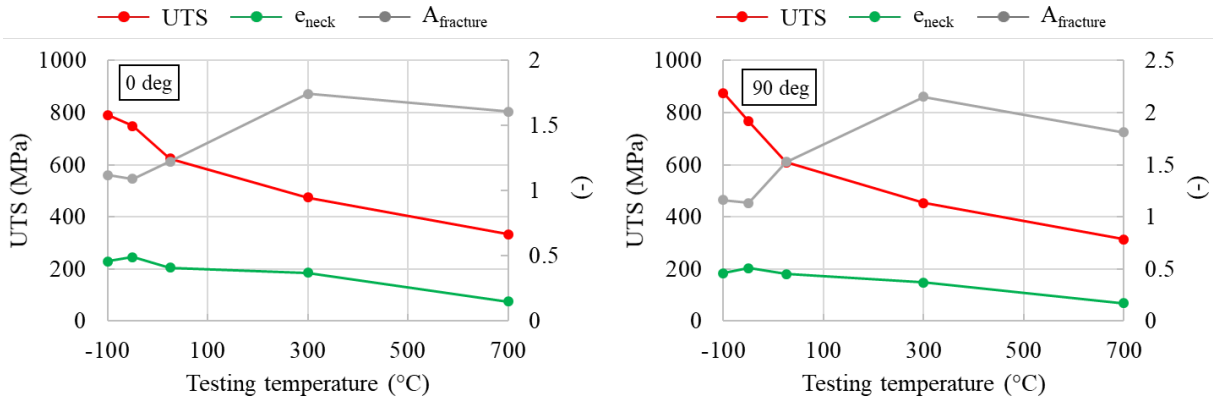

Figure 3. UTS, e_{neck} and $e_{fracture}$ at varying temperatures and rolling directions.

Fig. 3 shows the UTS, e_{neck}, and $A_{fracture}$ at varying temperatures and rolling directions Compared to room temperature, UTS increased on average by 23 % and 35 % for the samples tested at -50 °C and -100 °C, respectively. Interestingly, such increase in strength did not affect the uniform elongation. In fact, the e_{neck} of the sample tested at sub-zero temperatures is higher than that at room temperature, increasing on average of 16 % at -50 °C and of 7 % at -100 °C. On the contrary, $A_{fracture}$ decreases on average of 18 % at -50 °C and of 16 % at -100 °C, respectively.

It is worth noting that the highest values of e_{neck} as well as the lowest value of $A_{fracture}$ were registered when testing at -50 °C.

A completely different scenario is observed when considering testing at temperatures over the room one. In fact, both UTS and e_{neck} decrease while $A_{fracture}$ drastically increases. A 48 % and 25 % reduction in UTS was registered for samples tested at 700 °C and 300 °C, respectively. For the same testing temperatures, a reduction of 63 % and 14 % was achieved in e_{neck} while an increase of 25 % and 42 % in $A_{fracture}$.

The uniform elongation increase at decreasing testing temperature can be attributed to the face centered cubic (FCC) crystalline structure that characterizes stainless steels with quite elevated stacking fault energy (SFE). The SFE effectively affects the deformation mechanisms and the mechanical behavior of metals through its influence on the dislocations mobility: the higher the SFE the easier the dislocation movement as well as the dynamic recovery. On the other hand, the SFE is drastically influenced by the temperature, namely as the temperature increases the SFE increases as well. The application of sub-zero temperatures leads to the effective suppression of dynamic recovery, like thermal-induced cross slip and climb movements of dislocations,

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increasing the work hardening and postponing the formation of geometric instabilities to higher values of strain.
The influence of the rolling direction is notable only when considering the strain at fracture, which was always higher for the *90deg* samples, regardless of the testing temperature.

Characteristics after tensile testing
Fig. 4 shows the microstructure of the samples strained at UTS at varying temperature and rolling direction. The microstructures of the *0deg* and *90deg* samples deformed at 25 °C and 300 °C are very similar, indicating no thermally-induced microstructural phenomena. The same was found for the *0deg* and *90deg* samples deformed at -50 °C and -100 °C, here not reported for sake of brevity.

On the contrary, the *0deg* and *90deg* samples deformed at 700 °C present partially recrystallized austenite grains with annealing twins, with an average size slightly lower than the one of the samples deformed at lower temperatures.

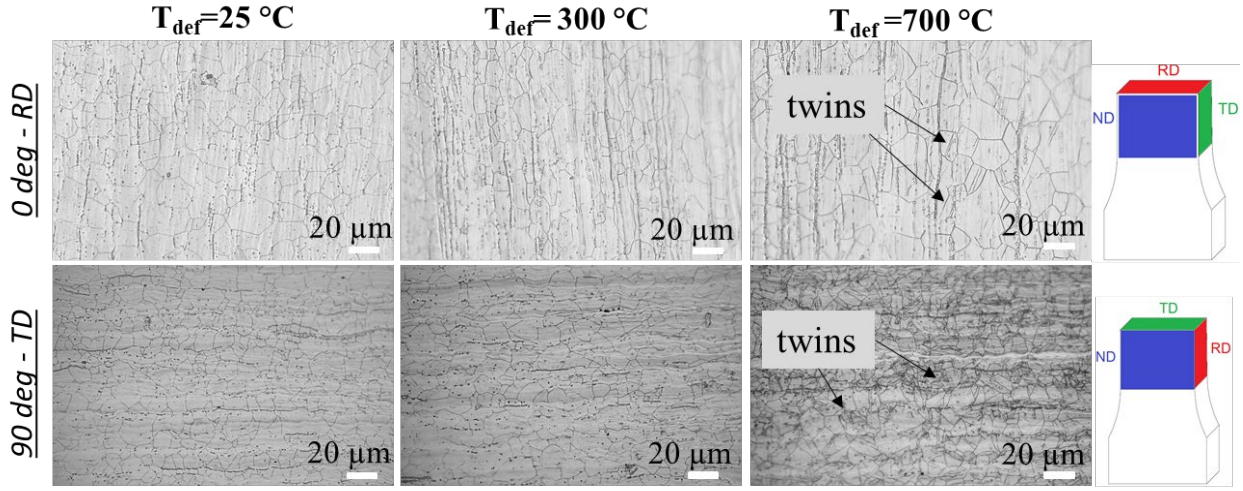

Figure 4. Microstructure of the samples strained at UTS at room and elevated temperatures.

The Beraha's etchant was then used on the same samples as indicated in the description of the experimental methods. Fig. 5 shows the microstructure of the *0deg* and *90deg* samples strained at UTS at room and sub-zero temperatures and varying rolling directions.

The deformation behavior of the samples tested at -50 °C and -100 °C is predominantly governed by SIMT, involving the deformation-induced transformation of austenite into martensite. On the contrary, the samples deformed at 25 °C, 300 °C and 700 °C (here not reported for sake of brevity) barely show the presence of the martensite phase.

SIMT of austenitic stainless steels deformed at cryogenic temperatures is a well-known phenomenon [8, 9]. The SIMT phenomenon is induced by both mechanical and thermodynamic driving forces. Specifically, the lower the temperature, the higher the thermodynamic driving force. This is due to the fact that lower temperatures impair the atomic diffusion ability suppressing the dynamic recovery. At the same time, sub-zero deformation temperatures produce a large number of defects and dislocations, which is conducive to martensite nucleation and growth.

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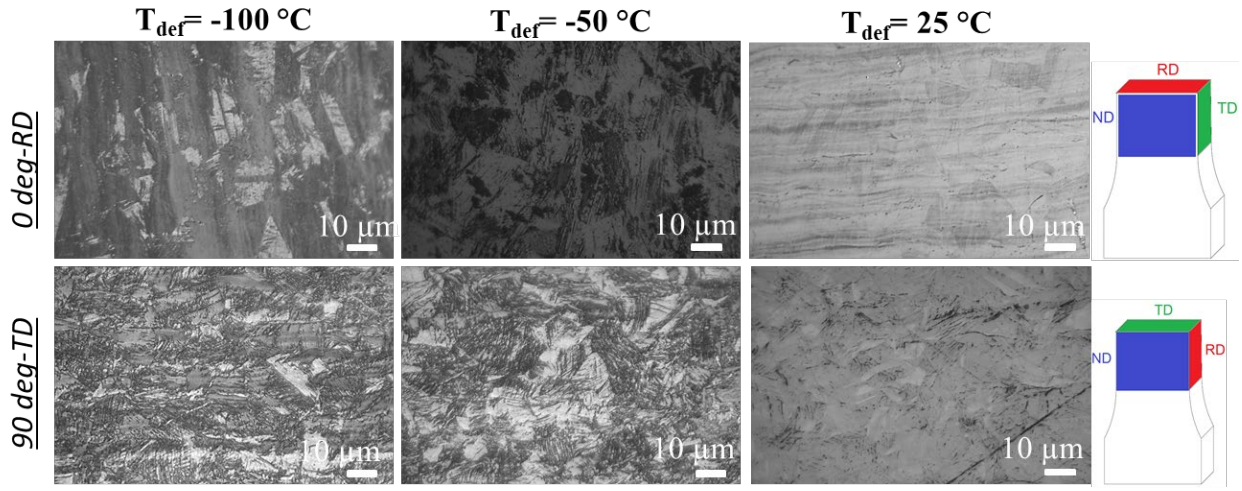

Figure 5. Microstructure of the samples strained at UTS at room and sub-zero temperatures.

XRD analyses were performed on the samples strained at UTS to investigate the microstructural evolution of the material, and the results are shown in Fig. 6. It can be seen that only the peaks related to the face-centred cubic (FCC) γ-austenite phase are visible at 25°C, regardless of the rolling directions. With the temperature decrease, peaks related to the body-centred cubic (BCC) martensite phase are evident at both -50 °C and -100 °C.

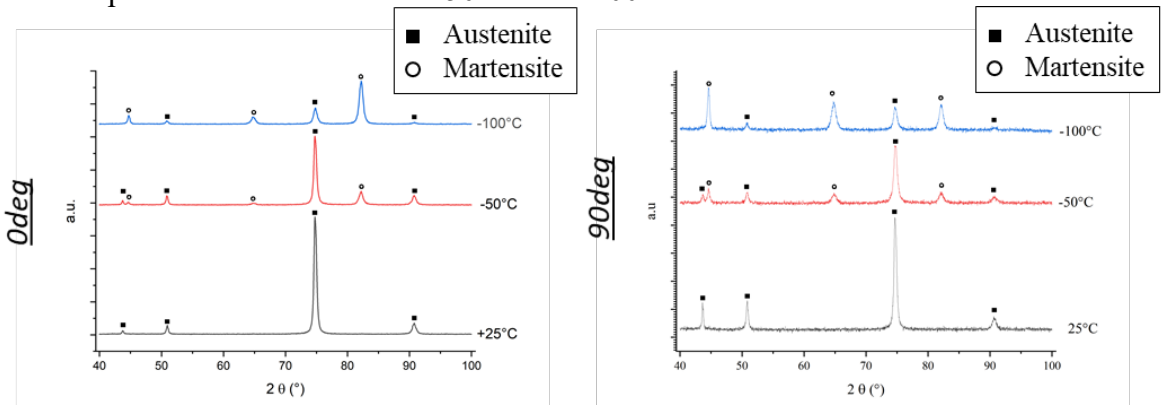

Figure 6. XRD spectra of the samples strained at UTS at room and sub-zero temperatures.

The XRD spectra give the chance to quantify the martensite percentages that are reported in Fig. 7a. At -100 °C, 78 % and 72 % of martensite formed in the *0deg* and *90deg* samples deformed at UTS. These results are in accordance with the previous considerations on M_{d30} temperature. At -50 °C, the martensite content decreased to 25 % and to 35 % in the *0deg* and *90deg* samples deformed at UTS, respectively. On the contrary, a fully austenitic structure was detected in the samples tested at 25 °C.

Fig. 7b reports the hardness values at varying testing temperatures and rolling directions calculated with respect to the baseline value obtained at room temperature. The highest hardness increase, namely 15 % on average, compared to the room temperature baseline, was found in the samples deformed at the lowest temperature. These outcomes are in accordance with the microstructures reported in Fig. 5, which shows the highest amount of martensite content when the material was deformed at -100 °C, and, therefore, the highest hardness.

On the contrary, a drastic decrease in hardness was visible for the samples tested at temperatures higher than room one. Specifically, the hardness of the samples deformed at 300 °C ad 700 °C

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decreased by 14 % and 25 %, respectively, with respect to one of the samples deformed at room temperature.

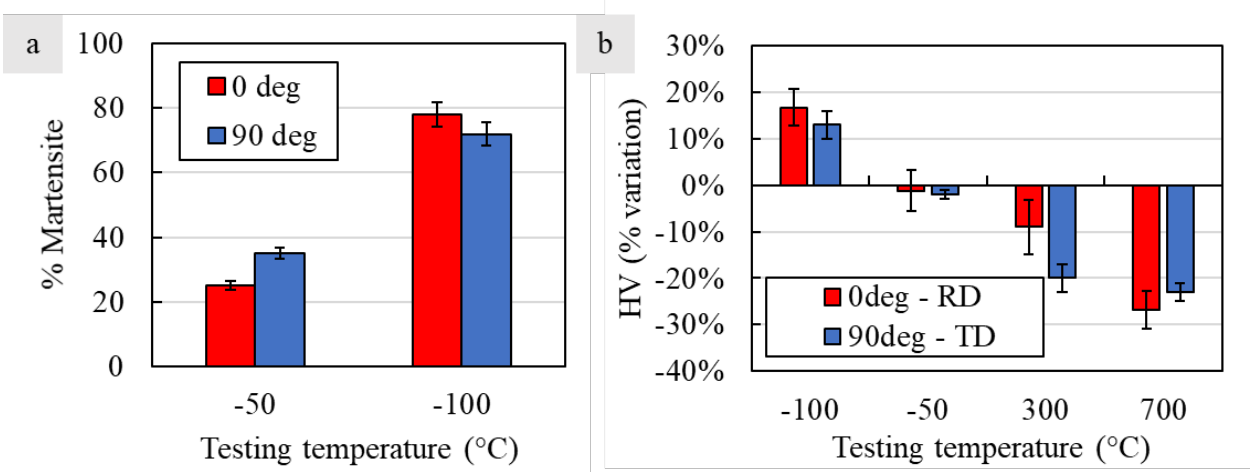

Figure 7. (a) Percentage of martensite calculated from the XRD spectra of Fig. 6 and (b) percentage variation of hardness at varying testing temperature and rolling direction.

Summary

In this paper, AISI 316 stainless steel samples were subjected to uniaxial tensile testing in a wide range of temperatures, from -100 °C to 700 °C. Their strain at UTS, namely the maximum strain for uniform elongation, was considered as a measure of the steel ductility at varying temperatures and rolling directions. The samples strained at UTS were also analyzed in terms of microstructural features, phase constituents and micro-hardness.

The main following conclusions can be drawn:

- All the tensile curves exhibited an approximately parabolic shape, except for the -100 °C curves that were S-shaped, especially in case of the 90 deg sample. Such peculiar behavior can be attributed to the SIMT effect that occurs at sub-zero temperatures.
- Regardless of the rolling direction, UTS monotonically decreased at increasing temperature. The same trend was registered for e_{neck}, which, however, showed the maximum value at T_{def}=-50 °C. Finally, $A_{fracture}$ increased at increasing temperature, presenting the minimum value at T_{def} =-50 °C.
- Regardless of the rolling direction, partially recrystallized austenite grains with annealing twins were evidenced only in the samples deformed at 700 °C. On the contrary, a certain amount of martensite was registered in the samples deformed at sub-zero temperatures. The outcomes of the microstructural analysis were also confirmed by the XRD measurements.
- Regardless of the considered section with respect to the rolling direction, the hardness of the sample strained at UTS at -100 °C was the highest, as a consequence of the most significant SIMT effect, with a less significant influence in the case of the sample strained at UTS at -50 °C. The opposite situation was registered for the samples deformed within the warm forming range, whose hardness was reduced as expected.

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References

[1] H. Takuda, K. Mori, T. Masachika, E. Yamazaki, Y. Watanabe, Finite element analysis of the formability of an austenitic stainless steel sheet in warm deep drawing. J. Mat. Proc. Tech. 143-144 (2003) 242-248. https://doi.org/10.1016/S0924-0136(03)00348-0

[2] E. Simonetto, R. Bertolini, A. Ghiotti, S. Bruschi, Mechanical and microstructural behaviour of AA7075 aluminium alloy for sub-zero temperature sheet stamping process. Int. J. Mech. Sci. 187 (2020) 105919. https://doi.org/10.1016/j.ijmecsci.2020.105919

[3] W. Liu, H. Hao, Damage and fracture prediction of 7075 high-strength aluminum alloy during cryogenic stamping process. Mech. Mater. 163 (2021) 104080. https://doi.org/10.1016/j.mechmat.2021.104080

[4] S.M. Hussaini, S.K. Singh, A.K. Gupta, Formability and fracture studies of austenitic stainless steel 316 at different temperatures. J King Saud Univ-Eng Sci 26(2) (2014)184-190. https://doi.org/10.1016/j.jksues.2013.05.001

[5] J. Aegerter, H.J. Kühn, H. Frenz, C. Weißmüller. EN ISO 6892-1: 2009 tensile testing: Initial experience from the practical implementation of the new standard. Materials Testing (2011) https://doi.org/10.3139/120.110269

[6] ASTM E 112-96 Standard Test Methods for Determining Average Grain Size.

[7] K. Couturier, S. Sgobba, Phase stability of high manganese austenitic steels for cryogenic applications (No. CERN-EST-2000-006-SM) (2000).

[8] G.M. Karthi, E.S. Kim, P. Sathiyamoorthi, A. Zargaran, S.G. Jeong, R. Xiong, H.S. Kim, Delayed deformation-induced martensite transformation and enhanced cryogenic tensile properties in laser additive manufactured 316L austenitic stainless steel. Additive Manufacturing, 47 (2021) 102314. https://doi.org/10.1016/j.addma.2021.102314

[9] X. Li, Z. Wei, X. Wang, L. Yang, X. Hao, M. Wang, J. Guo, Effect of cryogenic temperatures on the mechanical behavior and deformation mechanism of AISI 316H stainless steel. J. Mater. Res. Technol. 22 (2023) 3375-86. https://doi.org/10.1016/j.jmrt.2022.12.190