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MECHANICAL PROPERTIES IN FUNCTION OF STRAIN RATES OF AISI 304 AUSTENITIC STAINLESS STEELS: A COMPARATIVE STUDY AT ROOM AND CRYOGENIC TEMPERATURES

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Abstract. *Remarkable mechanical behaviors are observed in austenitic stainless steel AISI 304 when subject to deformation at cryogenic temperatures (CT approx. -180 ºC) compared to room temperature (RT approx. 25 ºC). In this research, specimens of AISI 304 austenitic stainless steel were subjected to tensile test at RT and CT, at different strain rates of* $1.2\times10^{-3}\ s^{-1}$; $2.4\times10^{-4}\ s^{-1}$; $6\times10^{-5}\ s^{-1}$ *in order to identify the provable combination between strain rate and temperature to improve ultimate tensile strength (UTS) and elongation. It is known that higher strain rates and lower temperatures induce a decrease of Stacking Fault Energy (SFE) and a suppression of dynamic recovery. This effect induces the change of deformation mechanisms, which can be twinning over slip. In addition, the microstructure and fracture surfaces were analyzed by optical microscopy and scanning electron microscopy with Field Emission Gun (SEM-FEG). The fractographic analysis showed several morphology, which can help to explain the microstructural evolution. It was investigated the connection between strain rate with mechanical properties and fracture morphology, at RT and CT.*

Keywords: *Cryogenic Deformation, Austenitic Stainless Steels, Strain Rate, Martensitic Transformation, Fractography.*

1. INTRODUCTION

Extensive studies have been developed about influence of temperature in processing of stainless steels, as tensile deformation of metals (Behjati, 2011). It was observed that in addition to the temperature influence, exist also the strain rate influence in mechanical behavior of this class of steels and both parameters were severely investigated (Huang *et al.*, 1989). It is known that 304 stainless steels is sensible to these factors. However, of according of literature (Michael *et al.*, 2016) (Behjati, 2011) the strain rate and temperature are not only parameters that cause changes in deformation and fracture mechanisms, variables as chemical composition and SFE; can, in addition, also to induce the material to present phase transformation. The Deformation – Induced Martensitic Transformation (DIMT) is an example of phase transformation, which can form ε-martensite, and α`-martensite by means different transformation mechanisms and the low SFE of AISI 304 makes it passive of these transformations. The type of structures resulting of DIMT formulates the mechanical behavior of material (Amar, 2004) (Hecker *et al.*, 1982).

Metals, when tested at low temperature or high strain rate presents increase of Ultimate Tensile Strength (UTS) and consequently increase their hardness (Padilha, 2005). According to some studies, (Martin *et al.*, 2016) (Shanmugasundaram *et al.*, 2006) the behavior conferred on materials that was tested at CT may occurs due to inhibition partial of recovery dynamic. Therefore, there will be significant growth in amount of defects, comparing with the tests performed at RT. Recovery is a phenomenon that occurs in metallic materials deformed and consist of the change of dislocations arrangement, afterward the recovery; the metal recovers partially their initial values of properties. In some cases (especially at RT) may occurs significant dynamic recovery while the material is deformed (Humphreys, 2004).

This paper reported the influence of strain rate in deformation mechanisms and the correlations has been made with the fracture morphology to understand the mechanical behavior presented by material. The analyses were made in both temperatures (RT and CT) varying the strain rates; the strain rates utilized to perform the tensile test were 1.9×10^{-3} s^{-1}; 2.45×10^{-4} s^{-1}; 6×10^{-5} s^{-1}.

2. EXPERIMENTAL PROCEDURE

The samples were rolled and machined to obtain sheets of 1.5 mm of thickness with final dimensions comparable to subsize. The samples has shape and dimensions as shown in Fig.1.

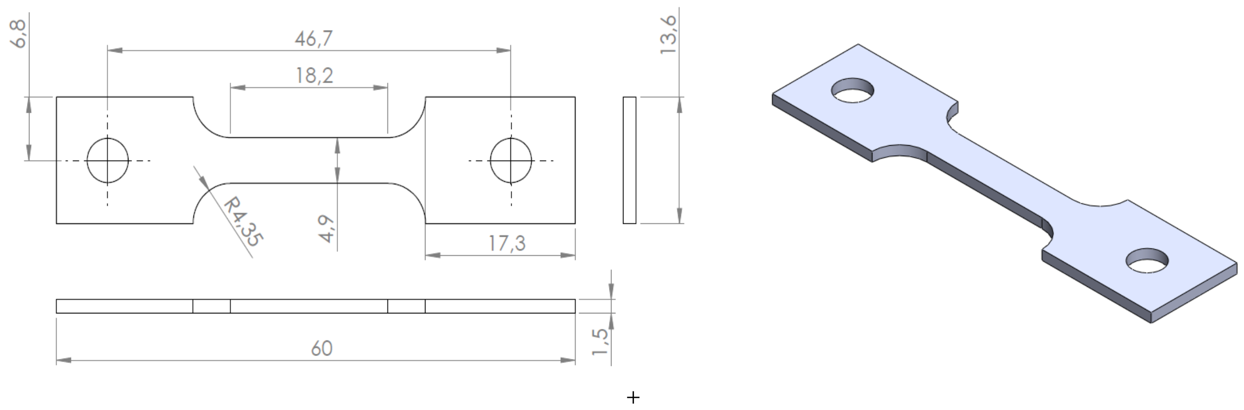

Figure 1. Dimensions and final form of subsize specimen

The samples were annealed at 1010°C during 15 min and furnace cooling, afterwards was realized the conventional metallographic procedure using the electropolishing and the step of chemical etchings was performed with Beraha`s II during 30 seconds. Images of microstructures were characterized by optical microscopy, utilizing the Olympus microscope model BX-51M.

The tensile tests were performed utilizing the universal testing machine SHIMADZU model AGS-X Series 300KN; the tests were initialized from strain rate of 1.2×10^{-3} s^{-1}; 2.4×10^{-4} s^{-1} and $6\text{x}10^{-5}$ s^{-1}, the all tests were performed at RT and CT. In order to keep the samples tested at CT at low temperatures, were immersed in a dewar containing liquid nitrogen; this container is coupled to the testing machine and was developed exclusively to perform these tests.

The fracture surface of each specimen tested in both conditions were analyzed using a Scanning Electron Microscope with Field Emission Gun (SEM-FEG) of Tescan.

Additionally microhardness tests were performed before and after tensile tests for comparison purposes of values. For hardness tests, it was used the load of 980.7 mN and indentation time kept at 12 seconds.

3. RESULTS AND DISCUSSION

3.1 Microstructure Analysis

The first characterization performed was the revealing of microstructure in longitudinal and cross section to verify the microstructural condition of samples that were annealed. The microstructure observed in Fig. 2 and Fig. 3, are presented with coarse and equiaxed grains in both sections and this morphology is characteristic of austenitic microstructure. In function the step of rolling and heat treatment, it is observed the presence of annealing twins and deformation twins. The arrows in figures of microstructure are to indicate the rolling direction.

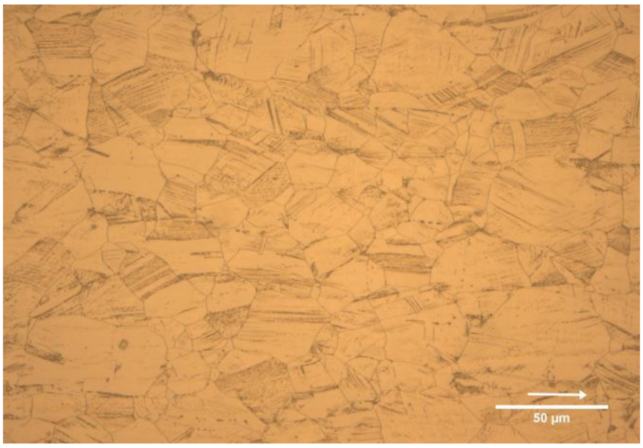

Figure 2. Microstructure of AISI 304 observed in longitudinal section

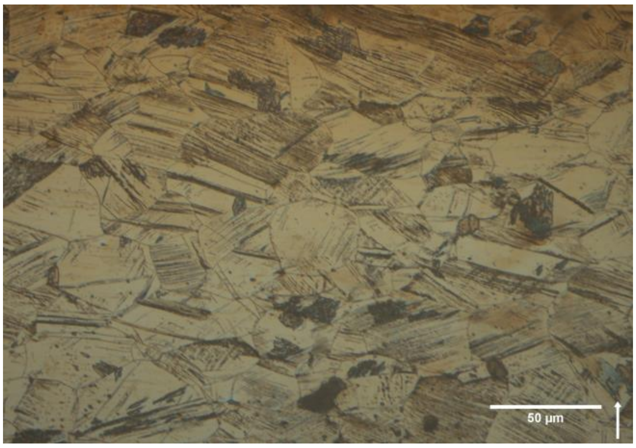

Figure 3. Microstructure of AISI 304 observed in cross section

3.2 Mechanical Properties Analysis

Results of tensile tests are presented in Fig. 4 as engineering curves, it is possible observes that highest values of UTS are correspondent to tests executed at CT, while the larger capacity of elongation are exhibited by tests executed at RT.

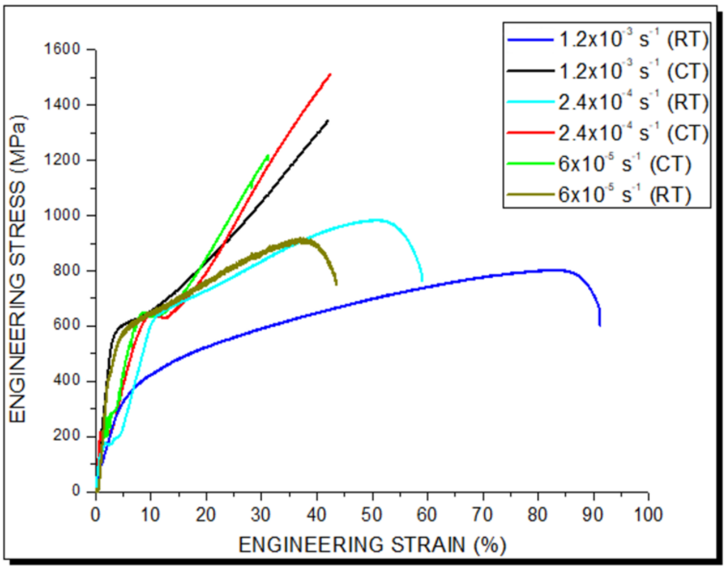

Figure 4. Engineering stress-stains curves presented in both temperatures conditions

The engineering stress-strain curves that represent the tests performed at CT, shown that there were not reduction of stress (MPa) after the material attain the UTS, the ductility of samples tested under these conditions underwent decrease comparing with results of tests at RT and can be observed that there is and inflection point. The inflection of curve and the slope change are characteristics that may be due to DIMT in 304 stainless steels (Huang *et al.*, 1989). The alteration slope of some curves occurs in both conditions CT and RT, the plateau in some curves are begin at low deformation levels (approx. 5% elongation) with the exception of the test realized at RT with strain rate of 2.4×10^{-4} s^{-1} that bestowed the highest plateau in terms of elongation (approx. 11%) (Hecker *et al.*, 1982).

The Table 1 indicates the values about mechanic properties obtained by tensile tests, were performed some comparisons between the gain of strength at CT and the elongation to failure in percentage.

Table 1. Experimental results including the tests conditions.

Temperature(1)/Stain Rate (s^{-1})	UTS (MPa)	UTS Increase (%)	Elongation to Failure (%)
RT/$1.2x10^{-3}$	803	-	91.2
CT/$1.2x10^{-3}$	1342	67	42
RT/$2.4x10^{-4}$	983	-	59
CT/$2.4x10^{-4}$	1512	53	42.4
RT/$6x10^{-5}$	920	-	43.4
CT/$6x10^{-5}$	1218	32	31.1

(1) RT is approx. 25°C and CT is approx. -180ºC

The increase of strength is accompanied by the reduction of capacity of elongation, this relation may be due to occurrence of DIMT. It is known that the martensite fraction increase with the diminution of temperature (Talonen *et al.*, 2005), this explains the high UTS in CT as also the reduction of elongation to failure on account of mechanical properties of martensite.

Table 2. Values of Hardness Vickers in both temperature conditions

Strain rate (s^{-1})	1.2×10^{-3} s^{-1}		2.4×10^{-4} s^{-1}		$6x10^{-5}$ s^{-1}		-
Temperature	CT	RT	CT	RT	CT	RT	Bt[1]
Hardness Vickers (HV)	437	464	432	348	410	370	327
	392	492	429	359	394	366	325
	413	486	448	353	456	372	316
	417	463	454	338	426	354	322
	431	465	423	353	415	372	320
	409	461	432	362	422	384	332
Average	416.5	471.8	436.3	352.1	420.5	369.6	323.7
Increase (%)[2]	13		23		13		-
Standard deviation	14.7	12.3	11	7.7	20.6	9.75	5.1

(1) Bt - Before tests, indicates that hardness test was performed before the tensile test
(2) Increase of hardness when comparison samples tested at RT with samples tested at CT

The values contained in Table 2 clearly indicates that highest HV belong to samples tested at CT, which proves the occurrence of martensite formation as also the larger fraction martensite formed by DIMT. The increase of hardness occurs due to mechanical behavior of martensite.

3.3 Fractography Analisys

It is clearly visible in Fig. 5 and Fig. 6 the difference that exist between the fracture morphologies, being predominantly, in Fig. 5, the event of ductile fracture, due to presence innumerable dimples dispersed throughout the fracture surface. The Fig. 6 in turn, indicates different fracture morphologies in same specimen, this implies the possibility of different deformation mechanisms occurring simultaneously. This anomalous phenomenon may occur due to decreases of SFE with the reduction of temperature, which leads on changing of these mechanisms and consequently of fracture mechanism. (Michael, *et al.*, 2016)

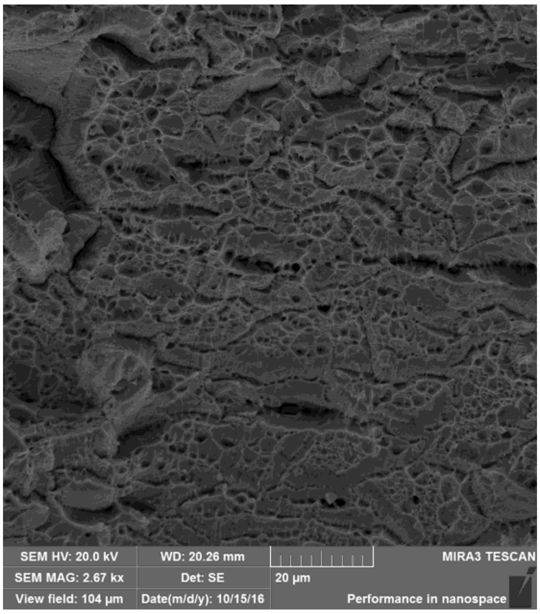

Figure 5. SEM-FEG fractography of sample tested at RT with strain rate of $1.2x10^{-3}$ s^{-1}

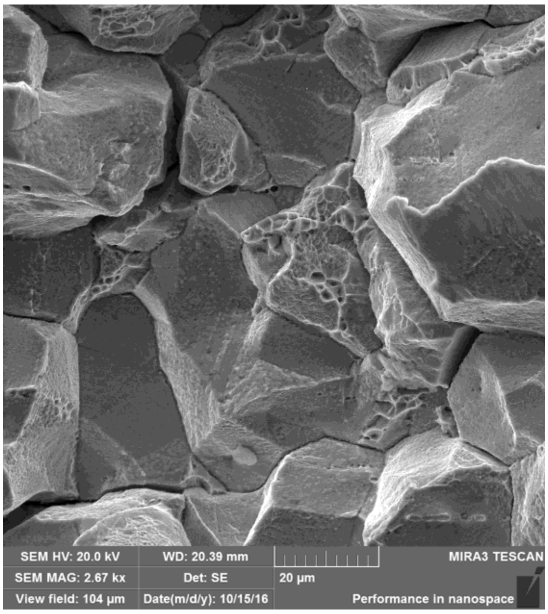

Figure 6. SEM-FEG fractography of sample tested at CT with strain rate of $1.2x10^{-3}$ s^{-1}

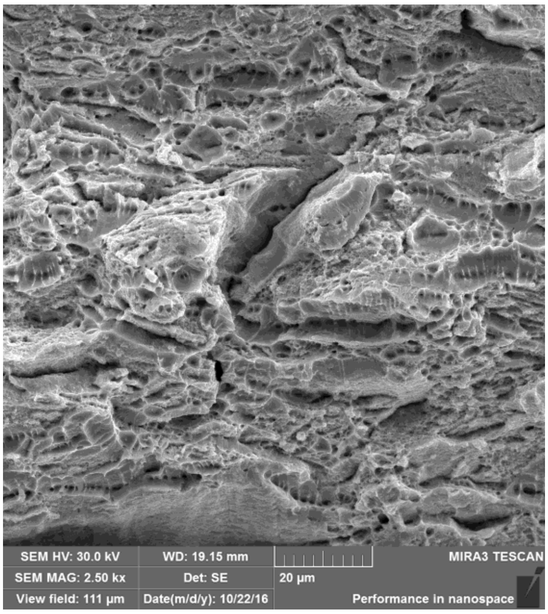

Figure 7. SEM-FEG fractography of sample tested at RT with strain rate of $2.4x10^{-4}$ s^{-1}

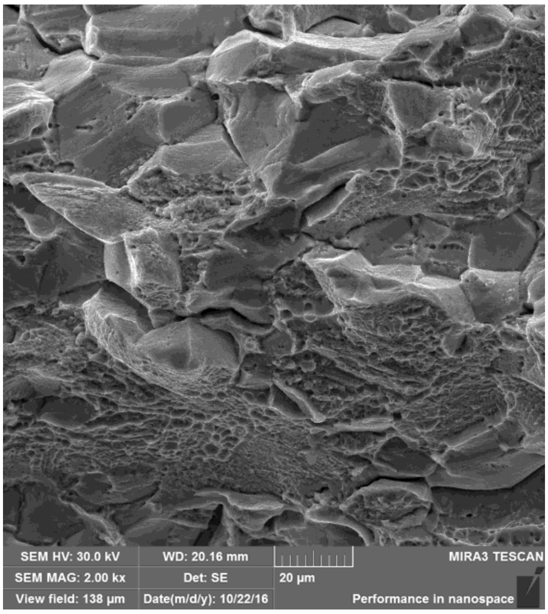

Figure 8. SEM-FEG fractography of sample tested at CT with strain rate of $2.4x10^{-4}$ s^{-1}

Observing the fracture surfaces on Fig. 7 and Fig. 8, can witness that the sample tested at CT present, as well as in Fig. 6, intergranular fracture with dimples and cleavage. The areas of brittle fracture observed on Fig. 8 has the less size in comparison with the Fig. 6 and apparently the intergranular reveal grains that has minors size, may be indicate the refined of microstructure. The comparison of their engineering stress-strain curves shown that the sample tested with strain rate of $2.4x10^{-4}$ s^{-1} has highest UTS, indicating again the provable refine of microstructure.

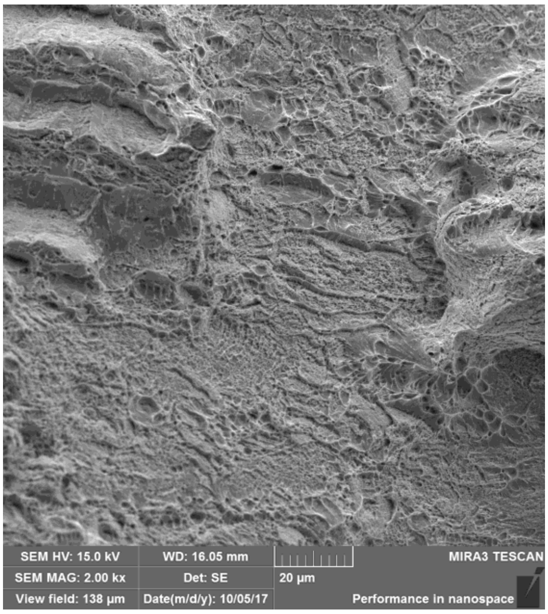

Figure 9. SEM-FEG fractography of sample tested at RT with strain rate of $6x10^{-5}$ s^{-1}

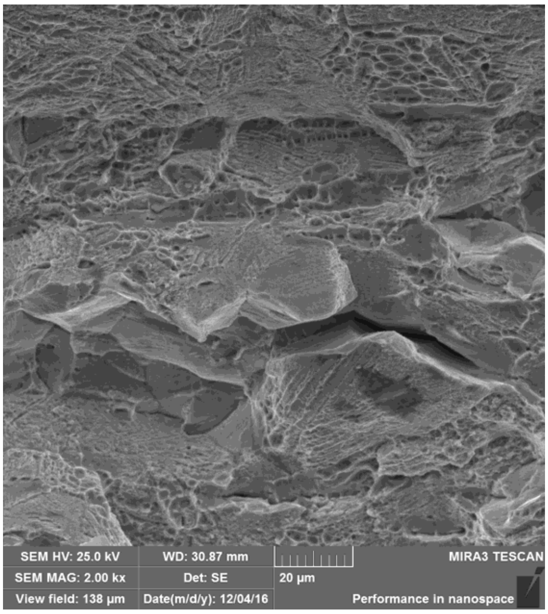

Figure 9. SEM-FEG fractography of sample tested at CT with strain rate of $6x10^{-5}$ s^{-1}

Analyzing overall the figures of fracture surface, can detect that the surface of specimens tested at CT has major areas with characteristics of brittle fracture as cleavage. The difference of morphologies is perceptible between RT and CT, where the amount of martensite transformed will depend of temperature, which at the test was realized (Huang *et al.*, 1989)

4. CONCLUSIONS

The initial proposal about the increase of strength was achieved by means tests are realized at CT and can be correlated by means of engineering stress-strain curves contained in Fig.4. The methodology used to performed tests at CT can be applied in other correlate studies, because the results achieved are promising to improvement of mechanical properties utilizing conventional method as the aborted in this paper. It is explicated by the values of strength shown in Table 1 and Fig. 4.

The samples tested at strain rate of $2.4x10^{-4}$ s^{-1} presented the highest strength among other strain rates used in this paper.

The size of regions that have brittle morphology change to according with strain rate used to perform the tensile tests.

The elongation decrease and gain strength may be influence underwent of DIMT.

5. ACKONOWLEDGMENTS

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