23rd ABCM International Congress of Mechanical Engineering
December 6-11, 2015, Rio de Janeiro, RJ, Brazil

EFFECTS OF CRYOGENIC TREATMENT ON STEEL TOOL ABNT M2

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Abstract. *The purpose of this study is to investigate the effect of cryogenic treatment on mechanical and tribological properties of ABNT M2 steel. Samples were tempered at 1170°C, 1200°C or 1230°C and subjected to double tempering prior to cryogenics, after cryogenics or one before and one after cryogenics. The cryogenic treatment was performed in a chamber with a controlled atmosphere of vaporized nitrogen. The steel was cooled at a 0.3°C/min rate until a temperature of -190 °C was reached, and it remained at this temperature for twenty-four hours. Heating the workpiece to ambient temperature was also carried out at a rate of 0.3°C/min. We conducted tests for hardness, microhardness, Charpy test and wear test and optical microscopy and scanning electronic microscopy analysis. It was observed that the use of cryogenics before tempering results in an increased hardness, microstructural homogeneity and higher toughness.*

Keywords: *Thermal treatment, cryogeny, mechanical proprieties.*

1. INTRODUCTION

In the mid-1930s, researches related to thermal treatments were carried out, and besides heating materials, they also cooled them. These studies were focused on the elimination of retained austenite and reached temperatures close to -80°C (Gulyaev, 1937). The process had already been used for years by Swiss watchmakers, where delicate metal parts were subjected to low temperatures in the Alps. So the watches had increased durability (Vales, 2011). Later on, NASA noticed an increased resistance on aircrafts that stayed longer periods of time in space, under the effect of low temperatures (SILVA, 1999).

More recent studies (Amini, 2012) related to this subject, show that submitting metals to temperatures close to liquid nitrogen's (-196 ° C) improve their mechanical properties. This thermal treatment known as Cryogenic treatment or Deep Cryogenic treatment (DCT) promotes austenite retinal transformation into martensite, resulting in an increased hardness (Zhirafar, 2007). It also occurs nano carbides precipitation, causing martensitic microstructure to possess a greater strength and toughness. (Meng, p.5, 1994).

According to (Amini, 2012) cryogenic treatment times above 36 hours make both the microhardness as the hardness progressively decrease due to coalescence of precipitated carbides.

The majority of studies in cryogenics do not investigate the hardening and tempering parameters concomitantly with cryogenics, which is the purpose of this study.

The objective of this study is to evaluate the mechanical and tribological properties of steel ABNT M2, varying the austenitizing temperature, tempering cycles and use or not of cryogenics. The hardness, microhardness, toughness and wear resistance of the samples were investigated.

2. MATERIALS AND METHODS

The steel chosen for this research was the rapid steel ABNT M2. Materials and Methods Its chemical composition is shown in Tab. 1.

Table 1. Chemical composition of steel ABNT M2 by Optical Emission Spectroscopy.

ELEMENT	C	Cr	V	W	Mo
% WEIGH	0,87	3,75	2,05	7,65	4,71

2.1 Thermal and cryogenic treatments

The hardening was carried out using the austenitizing temperatures of 1170°c, 1200°c or 1230°c. The cooling was done in a salt bath at a temperature of 500°c. For cryogenic treatment was used directly nebulization with a cooling rate of 0.3°c/minute up to ≈-190°c, where the material remained for 24 hours and returned to room temperature at a heating rate of 0.3°c/min. Double tempering was performed in an inti brand oven, qr 100-3, to 550 ° c for 120 minutes each, in the following sequence: before dct treatment, after the dct treatment or one tempering before and one after dct.

Table 2 shows the routes of heat treatments evaluated with the nomenclature of the respective samples.

Table 2. Cycles of thermal treatments.

CYCLES OF THERMAL TREATMENT	AUSTENITIZING TEMPERATURE [°C]	DEEP CRYOGENIC TREATMENT (DCT)	DOUBLE TEMPERING AFTER HARDENING	DOUBLE TEMPERING AFTER DCT	TEMPERING BEFORE AND AFTER DCT
1170/2T	1170	NO	YES	NO	NO
1170/2T/DCT	1170	YES	YES	NO	NO
1170/1T/DCT/1T	1170	YES	NO	NO	YES
1170/DCT/2T	1170	YES	NO	YES	NO
1200/2T	1200	NO	YES	NO	NO
1200/2T/DCT	1200	YES	YES	NO	NO
1200/1T/DCT/1T	1200	YES	NO	NO	YES
1200/DCT/2T	1200	YES	NO	YES	NO
1230/2T	1230	NO	YES	NO	NO
1230/2T/DCT	1230	YES	YES	NO	NO
1230/1T/DCT/1T	1230	YES	NO	NO	YES
1230/DCT/2T	1230	YES	NO	YES	NO

2.2 Charpy Test

To carry out the Charpy test was used RMU equipment with 150J hammer.

Previous Charpy tests were conducted on tempered (austenitizing at 1200°C) and tempering (550°C for 2 hours) samples using U and V-notches.

The average results with the V-notch were 1.5 J, and the average with U-notch was 2J. So we chose not to use notches for this test because the average toughness was very small, basically the minimum measurement scale from the equipment. Thus were prepared four Charpy samples without notches for each route of treatment.

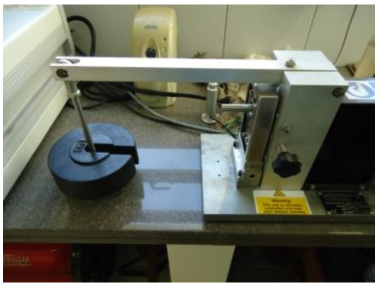
2.3 Hardness and Microhardness

Hardness measurements were done at a durometer of Instrutherm brand, DB model - 300 on the scale Rockwell C (HRC).

Microhardness measurement was performed in a Buehler Micromet 6010 Micro durometer, using Vickers (HV) and Knoop (HK) scales, with loads of 500g, 200g, 100g, 50g and 25g. Twelve measurements were carried out for each load/scale.

2.4 Wear Test

To perform the abrasive wear test samples were cut to a length of 12.7 mm. The test was conducted in a Phoenix Tribology equipment, model SLIM TE 53, as shown in Picture 1. This equipment uses the "Block on Ring" principle, where the sample is rubbed against a disc. On this disc is placed a granulometry sandpaper of 120 to obtain a measurable wear.



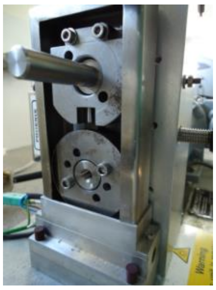


Figure 1. Equipment conducting the wear test.

A load of 5 Kgf was used, and speed of 50 RPM cycles of 25, 50, 100, 200 and 300 revolutions. The wear was measured by mass loss percentage, on a Shimadzu analytical balance with a resolution of 0,001g.

Wear was calculated using eq. 1.

$$\text{Mass low} = [(mi - mo) / mi] \times 100 \quad (1)$$

Where: mo= initial mass
mi= Mass after the wear test

2.5 Microstructural Analysis

2.5.1 Optical Microscopy (OM)

A analysis using an optical microscope was performed using a Diag Tech, model XJM104 microscope. Grain size and carbides distribution in the microstructure of the tested samples were analysed.

Before the samples suffer microscopic analysis, they have been embedded, sanded on granulometry sandpaper of 120 to 2000 grains/in2, and finished with a polishing diamond paste of 9µm. Then chemical etching was performed in 3% Nital reagent for 60 seconds.

2.5.2 SCANNING ELECTRONIC MICROSCOPY (SEM)

It was used a scanning microscope EVO MA10 of Zeis brand to perform the scanning electronic microscopy, where fracture mechanisms were investigated.

3. RESULTS AND DISCUSSION

3.1 Hardness

Table 3 presents the averages and standard deviations from the HRC hardness measurements.

Table 3. Average HR hardness.

	AVARAGE	SD
1170/0T	64,0	0,5
1170/2T	63,9	1,0
1170/2T/DCT	63,7	0,8
1170/1T/DCT/1T	64,2	0,6
1170/DCT/2T	**65,2**	**0,5**
1200/0T	63,2	1,0
1200/2T	64,2	1,0
1200/2T/DCT	64,6	1,1
1200/1T/DCT/1T	64,5	0,6
1200/DCT/2T	**65,4**	**0,6**
1230/0T	64,0	0,6
1230/2T	65,3	0,6
1230/2T/DCT	64,6	0,7
1230/1T/DCT/1T	65,4	0,6
1230/DCT/2T	**66,0**	**0,6**

Figure 2 shows the results in a graphic.

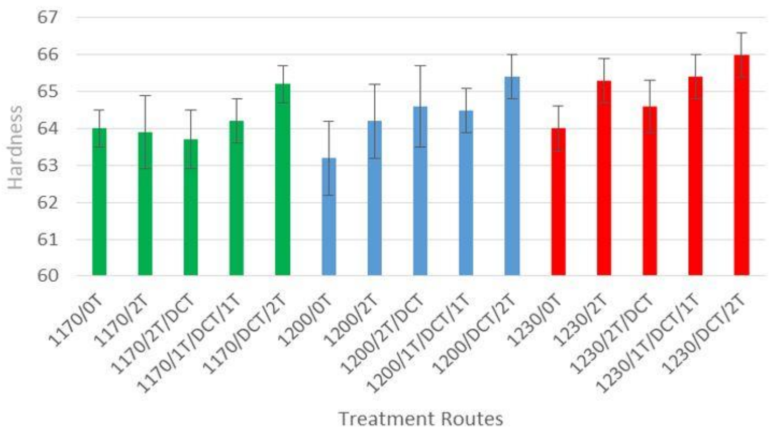


Figure 2. HRC Hardness after different routes of thermal treatments.

Based on these results it can be seen that, for each austenitizing temperature cycles, TEMP/DCT/2T showed the highest hardness and the lowest standard deviation. The smallest standard deviation of the measurements indicates a more homogeneous microstructure.

When tempering is applied before and after the first cryogenic treatment, the hardness does not vary substantially in comparison to the TEMP/2T cycle.

On Table 4 the HRC hardness values for TEMP/2T and TEMP/DCT/2T samples are compared. Based on this table it can be stated that there was an increase in hardness after cryogenic route since the hardness variation is greater than the variation coefficient on the results.

Table 4. Hardness comparison of cycles TEMP/2T and TEMP/DCT/2T.

TREATMENT CYCLE	HARDNESS ARITHMETIC AVERAGE	STANDARD DEVIATION	VARIATION COEFFICIENT	% OF HARDNESS INCREASE
1170/2T	63,9	1,0	1,58%	2,05
1170/DCT/2T	65,2	0,5	0,83%	
1200/2T	64,2	1,0	1,60%	1,85
1200/DCT/2T	65,4	0,6	0,90%	
1230/2T	65,3	0,6	0,91%	1,14
1230/DCT/2T	66,0	0,6	0,85%	

3.2 Charpy Test

Using samples without notches for performing the Charpy test, it was found that the standard deviation and variation coefficient are high.

Table 5. Average absorbed energy.

THERMAL TREATMENT	ARITHMETIC AVERAGE	STANDARD DEVIATION	VARIATION COEFFICIENT	INCREASE IN COMPARISON TO STANDART THERMAL TREATMENT
1170/2T	**14,4**	**-**	**-**	-
1170/2T/DCT	17,8	4,9	28%	23%
1170/1T/DCT/1T	17,8	6,7	38%	23%
1170/DCT/2T	16,9	2,2	13%	17%
1200/2T	**14,6**	**-**	**-**	-
1200/2T/DCT	14,1	2,9	21%	-3%

1200/1T/DCT/1T	17,6	1,5	8%	21%
1200/DCT/2T	19,3	1,9	10%	32%
1230/2T	**17,4**	**-**	**-**	-
1230/2T/DCT	14,3	3,5	25%	-18%
1230/1T/DCT/1T	14,6	1,9	13%	-16%
1230/DCT/2T	14	1,7	12%	-19%

In examining Table 5, it can be concluded that for austenitization temperatures of 1170°C and 1200°C, there is an increase in toughness when the DCT treatment was performed after tempering.

3.3 Microhardness

When performing microhardness tests we used Vickers (HV) and Knoop (HK) scales and loads ranging from 500g to 25g. Table 6 shows the standard deviations according to the scale and load employed.

Table 6. Average standard deviations.

	500g	200g	100g	50g	25g
VICKERS	32	43	50	80	106
KNOOP	64	73	100	139	185

Prior to performed tests, it was expected that by using the HK scale would have resulted in a smaller standard deviation since there is a long diagonal to be measured. However, in HK scale the standard deviation was higher than in HV scale.

Thus, for the analysis were used the results of measurements with HV 500g loads where the lowest standard deviation values were found, as is shown in Table 7.

It was observed that the microhardness variation with the use of DCT was lower than the variation coefficient. Thus, it was not possible to conclude that there was a microhardness HV increase with the use of cryogenics, although it showed a tendency to increase.

Table 7. Analysis of microhardness values with 0.5kg on HV scale.

TREATMENT ROUTES	AVERAGE HARDNESS VICKERS 0.5	STANDARD DEVIATION	VARIATION COEFFICIENT [%]	MICROHARDNESS INCREASE VICKERS
1170/2T	833,5	33,8	4,1	0,0
1170/2T/DCT	831	38,8	4,7	-0,3
1170/1T/DCT/1T	839,6	39,9	4,8	0,7
1170/DCT/2T	857,9	30,3	3,5	2,9
1200/2T	859,4	34,6	4	0,0
1200/2T/DCT	885,3	40,6	4,6	3,0
1200/1T/DCT/1T	862,6	36,1	4,2	0,4
1200/DCT/2T	880,9	29,6	3,4	2,5
1230/2T	899,3	33,1	3,7	0,0
1230/2T/DCT	871,9	17,2	2	-3,0
1230/1T/DCT/1T	874,3	27,4	3,1	-2,8
1230/DCT/2T	879,7	26,5	3	-2,2

Table 8 shows a comparison between standard deviations results in the cycles with and without cryogenics and through it can be concluded that the standard deviation measured on the microhardness HV scale decreases with cryogenic treatment. This indicates a greater microstructural homogeneity.

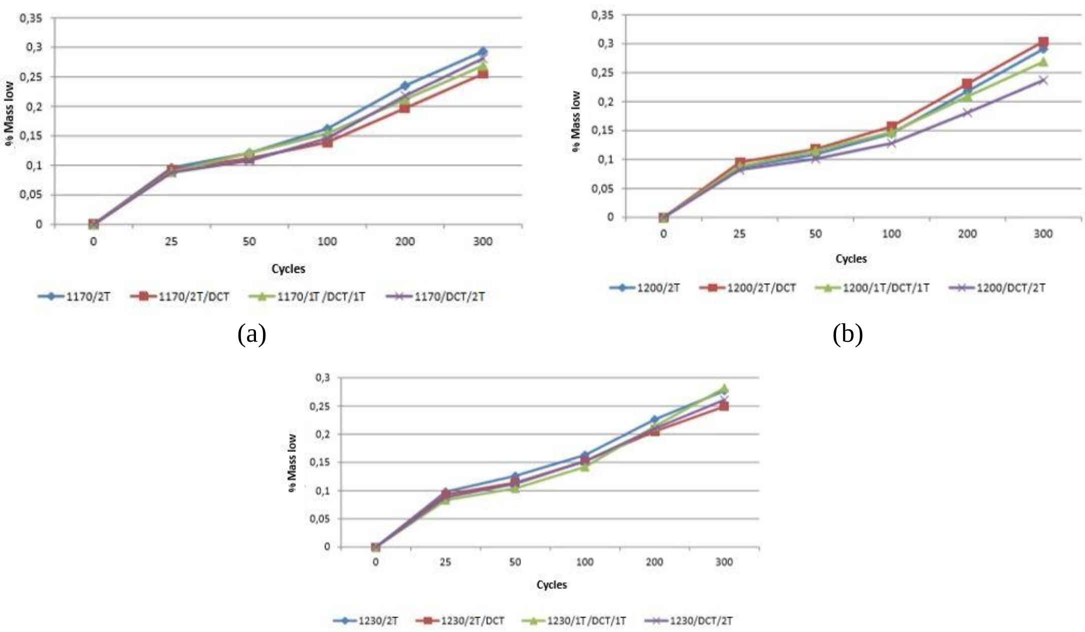
Table 8. Percentage reduction in the standard deviation.

THERMAL TREATMENT CYCLE	STANDARD DEVIATION	REDUCTION

1170/2T	33,8	10%
1170/DCT/2T	30,3	
1200/2T	34,6	15%
1200/DCT/2T	29,6	
1230/2T	33,1	20%
1230/DCT/2T	26,5	

3.4 Wear test

Figure 3 shows the results of the abrasive wear test, which are shown in percentage of mass loss.
For the austenitizing temperature of 1170°C (a) less wear was found in cycles 1170°C/2T/DCT and 1170°C/DCT/2T. In the temperature 1200°C (b) less wear was found in cycle 1200°C/DCT/2T followed by 1200°C/1T/DCT/1T. And in temperature 1230°C (c) reduced wear was observed in cycle 1230°C/2T/DCT.



(a) (b) (c)

Figure 3. Abrasive wear to temperatures (a) 1170°C (b) 1200°C (c) 1230°C.

Table 9 shows the weight loss values for 300 revolutions.

Table 9 - Abrasive wear test comparison for 300 cycles.

THERMAL TREATMENT	AVERAGE % IN MASS LOSS	STANDARD DEVIATION	VARIATION COEFFICIENT	% IN COMPARISON
1170/2T	0,293	0,033	-	-
1170/2T/DCT	0,255	0,022	9%	15%
1170/1T/DCT/1T	0,269	0,050	18%	9%
1170/DCT/2T	0,281	0,038	14%	4%
1200/2T	0,291	0,018	-	-
1200/2T/DCT	0,304	0,022	7%	-4%
1200/1T/DCT/1T	0,269	0,039	14%	8%
1200/DCT/2T	**0,237**	**0,030**	**13%**	**23%**
1230/2T	0,277	0,041	-	-
1230/2T/DCT	0,249	0,056	22%	11%
1230/1T/DCT/1T	0,282	0,029	10%	-2%

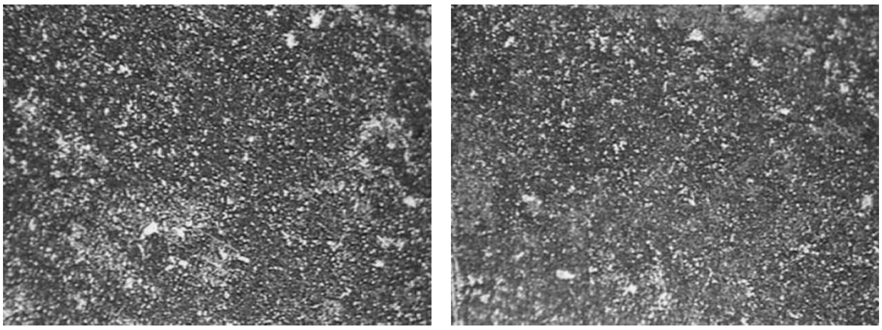
1230/DCT/2T	0,261	0,045	17%	6%

According to tab. 9, at 300 revolutions cycle for the 1200°C/DCT/2T cycle the wear percentage reduction as compared to the standard treatment sample comes up to 23%, whereas the variation coefficient between measurements was 13%. Confirming the improvement in wear resistance with the use of DCT.

3.5 Microstructural Analysis

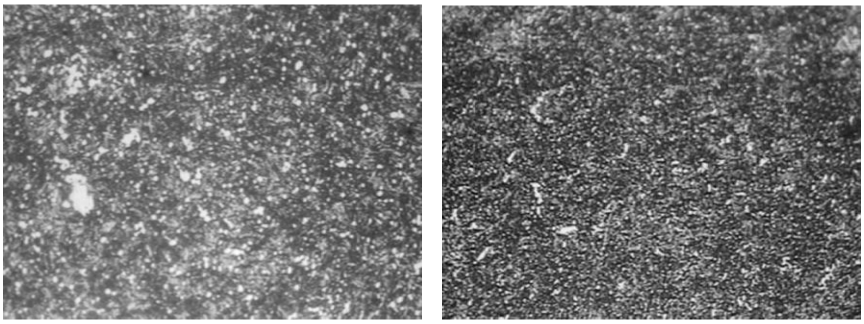
3.5.1 Optical Microscopy (OM)

Figure 4 and 5 indicate a more refined and homogeneous carbide distribution after DCT (right images).



(a) (b)

Figure 4 - 800X micrograph (a) 1170/2T (b) 1170/DCT/2T

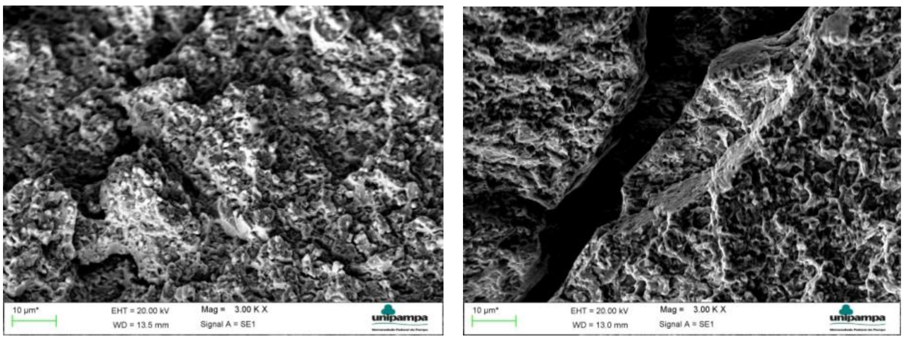


(a) (b)

Figure 5 - 800X micrograph (a) 1200/2T (b) 1200/DCT/2T.

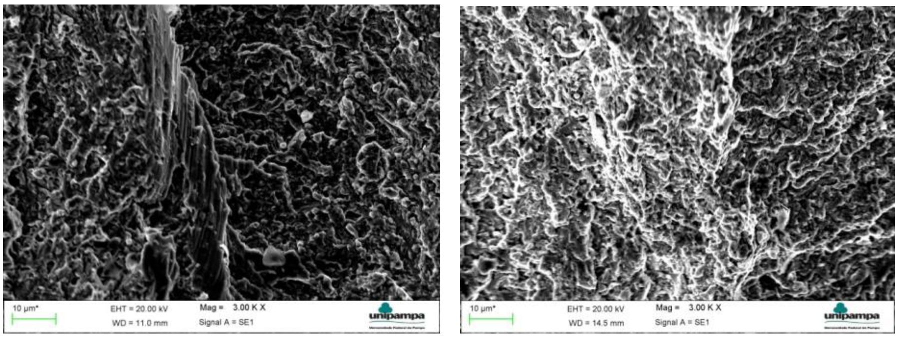
3.5.2 Scanning Electronic Microscopy (SEM)

The analysis made in SEM show that the samples subjected to austenitizing temperatures of 1170°C and 1230°C showed large cracks in their microstructures, as illustrated in Fig. 6.



(a) (b)

Figure 6 - SEM 3000X (a) 1170/2T (b) 1170/DCT/2T.



(a) (b)

Figure 7 - SEM 3000X (a) 1200/2T (b) 1200/DCT/2T.

According to Fig. 7, samples austenitized at 1200°C also showed cracks, but on a much smaller scale than in the other temperatures, and much more superficial than in the other temperatures.

4. CONCLUSIONS

- It was found that with the use of cryogenic treatment, material hardness is increased. It is likely that this variation in hardness is associated with the presence of fine and homogeneous carbides in the martensitic microstructure, where cryogenics causes fine carbides to precipitate during tempering.
- The cryogenic treatment reduces the standard deviation of microhardness HV, indicating a more homogeneous microstructure.
- The smallest wear was observed in 1200°C/DCT/2T cycle.
- The optimal parameters of hardening, tempering and cryogenics for the conditions tested are: austenitization 1200°C, use of DCT treatment after hardening and double tempering the end of the process: 1200°C/DCT/2T.

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