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# STUDY OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF ABNT M2 STEEL THROUGH THE USE OF THERMAL AND CRYOGENIC TREATMENT

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***Abstract.*** *Recent studies about cryogenic treatment uses temperatures as low as -190°C to strengthen metals because it is in this range that the retained austenite transforms to martensite and the precipitation of fine carbides occurs. In this way, the combination of conventional heat treatments together with cryogenic treatment plays an important role in improving the quality of mechanical components. For this reason, the present work aims to compare the performance of the combination of quenching, tempering and cryogenic varying the parameters of time, temperature and quantity of treatments to investigate which aspects influence the mechanical and tribological properties of ABNT M2 steel The cryogenic treatment was performed before tempering and after quenching. The samples were austenized at 1180ºC and quenched in oil, cryogenictreated at -181ºC during 24h with a ramp of -0.5ºC/min and the tempering was done at 550ºC for 2h. After that, were done the mechanical tests of hardness, microhardness, charpy and abrasion and the microestrutural tests of optical microscopy and scanning electron microscopy to analyze the results. The steel that received double cryogenic treatment before double tempering obtained the best improvement in its mechanical properties.*

***Keywords:*** *cryogenics, heat treatment, high speed steel.*

## 1. INTRODUCTION

The studies on heat treatments in steels are of great importance for the evolution of the industries. The main idea is to improve the characteristics of the materials with regard to their microstructure and to optimize the mechanical and tribological properties. From this, the researches in the area of materials technology seek to use the theoretical and technical concepts in order to elevate the panorama of the industrial market.

For manufacturing processes of industrial instruments, high speed steels are constantly present because they have specific characteristics such as high shock resistance, high hardness and wear resistance. According to FANTINELLI (2015), steel ABNT M2 is well ordered because it has adequate toughness and high temperability, because the material has a high thermal stability martensitic structure coupled with the reinforcement of alloy carbides.

The cryogenic treatment, according to PODGORNIK (2015) is defined as an additional process to the conventional heat treatment, involving the cooling of the material to about -190 ° C for up to 40 hours and a controlled return of ambient temperature. According to BALDISSERA (2008), there are two types of cryogenics treatment and that are classified by the parameters of the cooling heating cycle. Cryogenic Shallow Treatment (SCT) where the samples are placed in a 193 K freezer and then exposed to room temperature; Deep Cryogenic Treatment (DCT) samples are slowly cooled to 77 K, held down for many hours to room temperature and exceeds the surface of the material and remains in the process for a longer time. The common tempering process in high speed steels does not suppress all the austenite present in the material, therefore, it is justified to apply the DCT. Heat treatments obtained after quenching must ensure a reduction or complete elimination of the retained austenite

According to TIER (1998), high speed steels at high working temperature can retain the hardness and remain in machining operation. Subzero or deep cryogenic treatments (DCT) may be performed during or after tempering and tempering. Many researches (BALDISSERA, 2008, AKHBARIZADEH, 2009, IDAYAN, 2014, PODGORNIK, 2015, FANTINELI, 2015) were approached in this area in order to know the routes and levels of ideal parameters such as temperature, treatment time and cooling rate to obtain a microstructure of high hardness and toughness.

Cryogenics is an extra procedure to conventional quenching and tempering heat treatments. It aims to suppress austenite retained in the quench and improve the mechanical and tribological properties of the material. In relation to the research concerning cryogenic treatment, PADMAKUMAR et al. (2018) explains that the studies are still in the initial stage, because the mechanisms behind the improvement in properties is still under debate, however, it is known that better wear rates and higher hardness values of the metal are achieved. For example, DA SILVA et al. (2006) used X-ray diffraction to evaluate the percentage of retained austenite. The samples not treated by DCT had a volume of 25% retained austenite, while in the treated samples the volume was close to 0%. According to LI et al. (2018), the cryogenic treatment can modify microstructures and mechanical properties of carbide cutting tools, improve the wear resistance of the tool and reduce the coefficient of friction.

ZHOU et al. (2018) showed the importance of using the cryogenic treatment, the authors made the treatment in a TC6 titanium alloy with an immersion time of 18 hours and had optimization results, such as surface microhardness and tensile strength of samples increased by 28.11% and 28.71%, respectively, and better mechanical properties. The cryogenics performed by IDAYAN et al. (2014) had better hardness results, was increased by 7% when submitted to DCT and the percentages of austenite retained from these samples were 29%, 8% and 5.7%. VILLA (2017) clarifies that the cryogenic treatment is an intermediate step in the hardening of carbon steels, but that it can be further optimized by adapting the temperature program and exerting more control of the cooling parameters.

According to PADMAKUMAR (2018), cryogenic research is in an early stage, because the mechanisms behind the improvements in properties are still under discussion, which the author affirms is the improvement of the rate of wear and the increase of the hardness of the material.

Thus, the purpose of this work is to evaluate the effect of cryogenic treatment on ABNT M2 high speed steel in conjunction with conventional heat treatments. For this, a system with control of the rate of cooling and heating was used for the application of the DCT. This study aims to answer the following question; does the use of deep cryogenic treatment in ABNT M2 high speed steel contribute to improve the mechanical and tribological properties of the material?

The general objective of this study is to compare the performance of cryogenics in ABNT M2 fast steel combined with quenching and tempering treatments in relation to steel only with conventional treatments. The specific objectives are to analyze the tribological properties and the toughness of the M2 steel in samples without DCT, double DCT and simple DCT through the tests of abrasion and Charpy, evaluate the resulting microstructural divergences and check the optimization of the material parameters after all the tests.

The initial hypotheses for the present work are: The combination of the thermal and cryogenic treatments optimizes the structural characteristics of ABNT M2 steel; Dual deep cryogenic treatment achieves better properties than just conventional treatments; the microstructure of the ABNT M2 steel after the treatments shows elimination of retained austenite, total transformation of martensite and homogeneous carbides.

## 2. MATERIALS AND METHODS

In this stage, the methods and experiments will be presented for the development of the work. Experimental research is the ideal way to achieve the proposed objectives. Articles, theses and books that mention heat treatments, cryogenics and high speed steels were studied, the data were obtained from an experimental research. The aim of the present work is to analyze how the variation of thermal treatment routes will influence the mechanical and tribological properties of ABNT M2 steel. Thirty samples of ABNT M2 steel were cut in circular bars of 15mm diameter. Samples were used for the standard square format 10x10mm with length of 55mm. The machining process was executed at UNIPAMPA. After this, the samples were submitted to tempering, with austenitization temperature of 1180°C. To understand the sequence of experiments performed, look at Fig. 1.

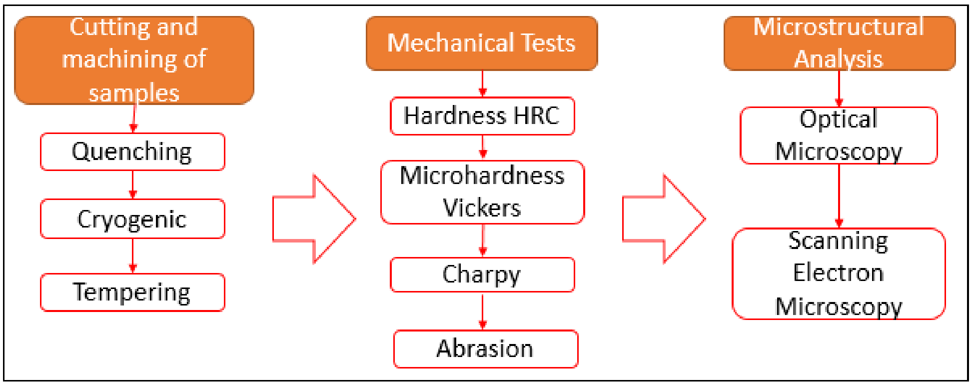



Figure 1. Sequence of experiments.

After the pieces were austenitized and hardened, the cryogenic treatment was carried out in the university laboratory, where the temperature reached -181ºC during 24 hours. The cooling rate will be - 0.5 ° C / min, preventing thermal shock in the samples. To achieve this temperature control in the descent and rise, the cryogenic treatment was carried out in the laboratory of UNIPAMPA, the samples were placed inside a capsule developed by the project team and inserted inside the nitrogen tank. The prototype consists of a capsule made of PVC and polyurethane to be used as a sample container, the idea is for the capsule to collaborate for a slow cooling transition in the samples. After that, In a later stage, double and single tempering were performed, depending on the route chosen, with a temperature of 550ºC and with air cooling.

After all completed routes, the mechanical and tribological tests were implemented. The test of Rockwell C hardness (HRC) was made in the equipment Beijing TIME High Technology, model TH500. The Vickers microhardness (HV) was done in the Buehler microdurometer, model Micromet 6010 with a load of 1.0kg.

For the wear test it was used the equipment Phoenix Tribology, model TE 53 SLIM with the parameters: 300 cycles; sandpaper 120 grains/in²; 50rpm of angular speed; Charge 49 N (5.0 kg). The test used the block-on-ring configuration, where the piece forces a disk, generating wear. The sample being rubbed against a moving cylinder and the ring was coated with a sandpaper of particle size 120. The characterization of the wear was performed by the weight loss of the sample by a balance with a resolution of 0.0001g.

Hardness, Microhardness and Abrasion tests were carried out in the laboratory of UNIPAMPA. However, the charpy test was performed at the mechanical engineering laboratory of UFSM, was used the machine of German manufacture of the company VEB Werkstoffprüfmaschinen Leipzig.

In a later stage, the microstructural analysis was made through by optical microscopy (OM) and by scanning electron microscopy (SEM). The microstructure can be visualized in the optical microscope Kontrol, model IM713. The fracture mechanisms were analyzed using the SEM of the brand ZEISS, model EVO MA10, from the electron microscopy laboratory of Unipampa.

The treatments are abbreviated as Quenching (Q), Cryogenic (DCT) and Tempering (T). Table 1 shows the routes chosen for this work and how the treatments are distributed. Each route was composed of five samples and the average of the hardness test was obtained in five points of each sample.

Table 1. Routes

| ROUTE | TREATMENT | CODE |
|---|---|---|
| 1 | Quenching + Tempering | Q/T |
| 2 | Quenching + Tempering + Tempering | Q/2T |
| 3 | Quenching + Cryogenic + Tempering | Q/DCT/T |
| 4 | Quenching + Cryogenic + Tempering + Tempering | Q/DCT/2T |
| 5 | Quenching + Cryogenic + Cryogenic + Tempering | Q/2DCT/T |
| 6 | Quenching + Cryogenic + Cryogenic + Tempering + Tempering | Q/2DCT/2T |

## 3. RESULTS AND DISCUSSIONS

In this chapter the results of the mechanical tests of hardness, microhardness, charpy and wear tests and of the microscopical tests were presented through the microstructural analysis in OM and fracture in SEM, achieved according to the experimental method described in Chapter 2.

### 3.1 Hardness and Microhardness Tests

The results of the averages of the hardness and microhardness tests are shown in Tab. 2. Each route had five samples and each sample was measured five times. The mean of each sample was obtained, and then the final average for each route. Fig. 2 and Fig. 3 show the results in graphic form.

Table 2. Hardness and Microhardness Test

| ROUTE | AH (HRC) | SD | CV (%) | Increase of Hardness (%) in relation to Q/2T | AM (HRV) | SD | CV (%) | Increase of Microhardness (%) in relation to Q/2T |
|---|---|---|---|---|---|---|---|---|
| Q/T | 56.08 | 1.30 | 2.32 | 0.36 | 522.07 | 73.58 | 52.04 | -19.40 |
| Q/2T | 55.88 | 2.17 | 3.88 | -- | 647.753 | 38.64 | 28.13 | -- |
| Q/DCT/T | 57.20 | 1.32 | 2.31 | 2.36 | 653.56 | 49.76 | 39.44 | 1.12 |
| Q/DCT/2T | 56.51 | 1.47 | 2.60 | 1.13 | 655.02 | 38.17 | 28.47 | 0.90 |
| Q/2DCT/T | 57.36 | 0.55 | 0.97 | 2.65 | 687.2 | 41.67 | 29.68 | 6.09 |
| Q/2DCT/2T | 59.00 | 0.60 | 1.02 | 5.58 | 734.353 | 54.93 | 29.68 | 13.37 |

AH: Average of Hardness; SD: Standard Deviation; CV: Coefficient Variation; AM: Average of Microhardness.

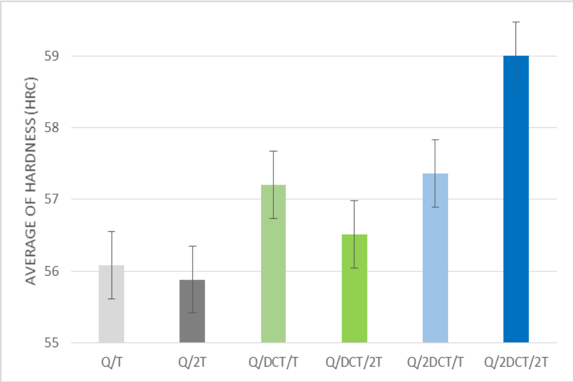



Figure 2. Hardness test.

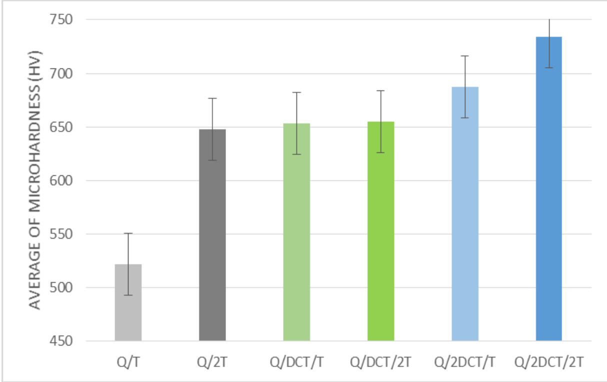



Figure 3. Microhardness test.

The highest hardness increase with the use of cryogenic treatment occurred in the Q/2DCT/2T route. It is possible to observe a reduction of the standard deviation in the routes with double DCT, indicates that the material is more homogeneous. For the microhardness test, it can be observed that the values had a greater variation, but the highest value still occurred in the route Q/2DCT/ 2T.

To understand the values obtained, a statistical tool was applied to the data, an analysis of variance (ANOVA) of hardness and microhardness data was executed. The results of hardness test are shown in Tab. 3.

Table 3. ANOVA of Hardness Test

| *Effect* | *QS* | *FD* | *QA* | *F* | *Critical F* |
|---|---|---|---|---|---|
| Tempering (T) | 3.7808 | 1 | 3.7808 | 2.063634 | 4.259677 |
| Cryogenic (DCT) | 24.5375 | 2 | 12.2688 | 6.696611 | 3.402826 |
| TxDCT interaction | 4.2335 | 2 | 2.1168 | 1.155379 | 3.402826 |
| Error | 43.9700 | 24 | 1.8321 | | |
| Total | 76.52175 | 29 | | | |

QS: Quadratic Sum; FD: Freedom Degrees; QA: Quadratic Average

The results of probability for the DCT effect indicate that there is a statistically significant difference between the parameters analyzed, because F> Critical F. After this, Fisher's method, also known as Least Square Difference (LSD), was used. The results expose that there is a significant difference in the use of double DCT, because Average>LSD.

The results of microhardness test are shown in Tab. 4. The data obtained from indicate that there is statistically significant difference in Tempering, DCT and TxDCT interaction, because F> Critical F in the three effects studied. However, Fisher's method in the microhardness test suggests that there is no significant difference in any case, since the Average is greater than LSD.

Table 4. ANOVA of Microhardness Test

| *Effect* | *QS* | *FD* | *QA* | *F* | *Critical F* |
|---|---|---|---|---|---|
| Tempering (T) | 25310.68 | 1 | 25310.68 | 16.76795 | 4.259677 |
| Cryogenic (DCT) | 79486.51 | 2 | 39743.26 | 26.32931 | 3.402826 |
| TxDCT interaction | 19741.48 | 2 | 9870.738 | 6.539216 | 3.402826 |
| Error | 36227.24 | 24 | 1509.468 | | |
| Total | 160765.9 | 29 | | | |

QS: Quadratic Sum; FD: Freedom Degrees; QA: Quadratic Average

### 3.2 Charpy and Abrasion Test

The tenacity results obtained from the Charpy test and the wear test results are shown in Tab. 5. In graphic form can see the results in Fig. 4 and Fig. 5.

Table 5. Charpy and Abrasion Test

| ROUTE | AC (J) | SD | CV (%) | Increase of Tenacity (%) in relation to Q/2T | AA (mg) | SD | CV (%) | Increase of Wear (%) in relation to Q/2T |
|---|---|---|---|---|---|---|---|---|
| Q/T | 3.97 | 0.15 | 3.85 | -5.92 | 5.5650 | 0.0085 | 0.1527 | -27.44 |
| Q/2T | 4.22 | 1.14 | 27.05 | -- | 7.6700 | 0.0587 | 0.7649 | -- |
| Q/DCT/T | 2.12 | 0.43 | 20.09 | -49.76 | 4.0400 | 0.0238 | 0.5881 | -47.33 |
| Q/DCT/2T | 5.70 | 2.69 | 47.14 | 35.07 | 2.4870 | 0.0216 | 0.8685 | -67.57 |
| Q/2DCT/T | 4.27 | 1.17 | 27.49 | 1.18 | 3.6200 | 0.0389 | 1.0743 | -52.80 |
| Q/2DCT/2T | 3.73 | 0.12 | 3.09 | -11.61 | 1.4330 | 0.0034 | 0.2387 | -81.32 |

AC: Average of Charpy; SD: Standard Deviation; CV: Coefficient Variation; AA: Average of Abrasion.

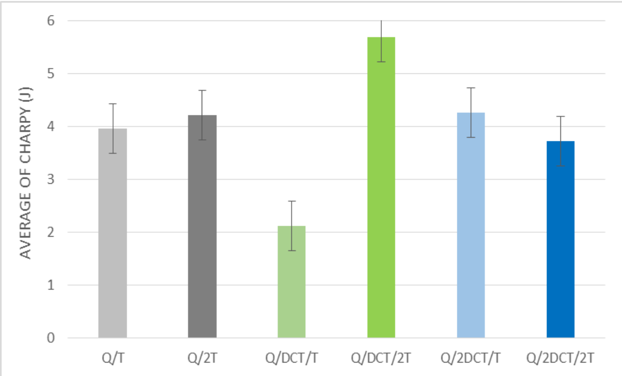



Figure 4. Charpy Energy test.

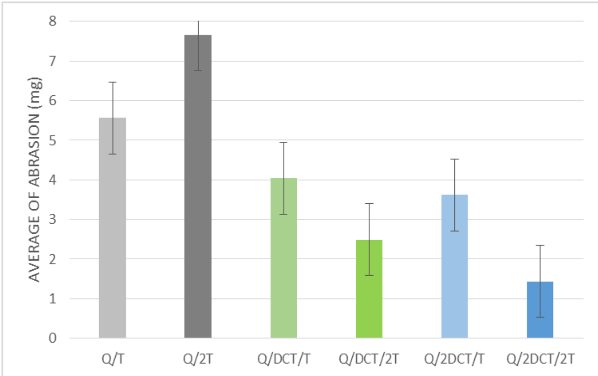



Figure 5. Weight Loss test.

In the charpy test, the results suggest an increase in toughness with the use of cryogenics for the Q/DCT/2T and Q/2DCT/T routes. However, it is not possible to say with certainty that the increase values are smaller than the respective coefficients of variation. It can be observed from these results that, unlike hardness and microhardness tests, the highest absorbed energy value is in the Q/DCT/2T route, with 35.07% in relation to the Q/2T route. The increase of the impact strength in the material, which occurs after the combination of the treatments, may have been an effect of the reduction of martensite tetragonality next to the precipitation of carbides. Routes with values smaller than the reference route of comparison (Q/2T) were obtained. We hypothesize that these divergences may be linked to the instability of the equipment. For this reason, ANOVA and Fisher test were not implemented for the charpy assay.

In the abrasion test, it can be seen that Q/2DCT/2T had the smallest mass reduction within the routes tested. The comparison of mass loss reduction can be seen by the percentage of less mass loss, where the double DCT samples had a decrease of 52.80% and 81.32%. It is more evident how much the loss of mass with the application of the cryogenic treatment decreases when compared with the routes with only conventional treatment, including with a great percentage variation.

An analysis of variance (ANOVA) of abrasion test was performed. The results are shown in Tab. 6.

Table 6. ANOVA of Abrasion Test

| *Effect* | *QS* | *FD* | *QA* | *F* | *Critical F* |
|---|---|---|---|---|---|
| Tempering (T) | 0.000374 | 1 | 0.000374 | 0.449776 | 4.747225 |
| Cryogenic (DCT) | 0.00571 | 2 | 0.002855 | 3.429069 | 3.885294 |
| TxDCT interaction | 0.001368 | 2 | 0.000684 | 0.821784 | 3.885294 |
| Error | 0.009991 | 12 | 0.000833 | | |
| Total | 0.017443 | 17 | | | |

QS: Quadratic Sum; FD: Freedom Degrees; QA: Quadratic Average

The results of probability for the DCT effect indicate that there is not a statistically significant difference between the parameters analyzed, because F<Critical F in the three effects studied. After this, Fisher's method, also known as Least Square Difference (LSD), was used. This method in the abrasion test suggests that there is significant difference in any case, since the LSD is greater than Average.

### 3.3 Comparisons of mechanical tests

For a better understanding of mechanical test data, it is relevant to analyze the wear behavior versus hardness and the wear versus charpy. Fig. 6(a) and Fig. 6(b) show these relationships.

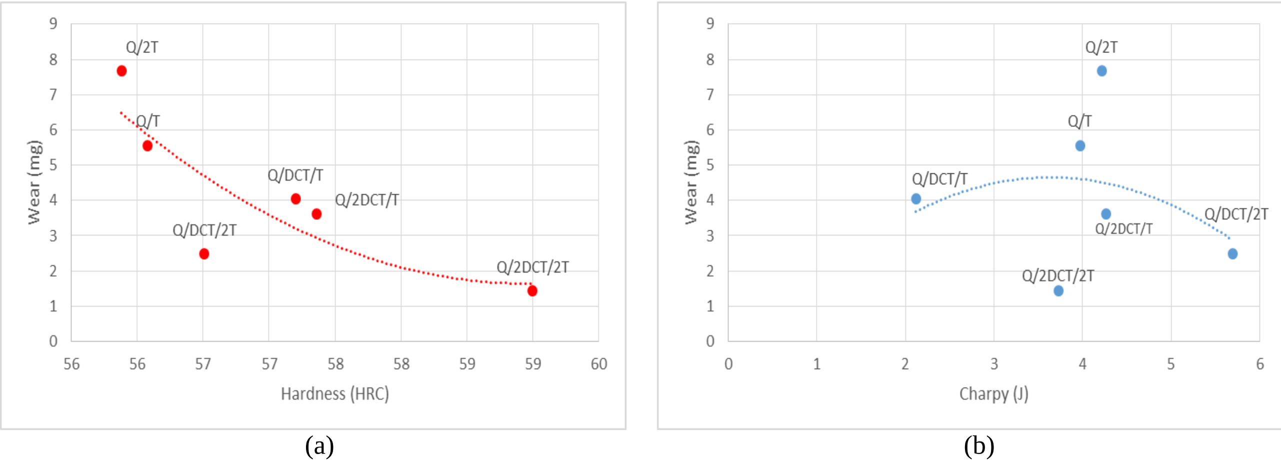



Figure 6. (a) Wear x Hardness (b) Wear x Charpy

According to the graph in Fig. 6 (a) it is possible to identify the improvement of mechanical and tribological properties of ABNT M2 steel from the application of the double DCT, as it can be seen the reduction of wear and the increase of hardness. In the trend line, it is possible to observe the influence of cryogenic treatment, since each of the route improves the behavior of steel.

Fig. 6 (b) helps to identify the relationship of Charpy energy absorption and wear. It is possible to observe that the simple and double DCT resulted in the better results for the relationship lower wear/higher charpy energy.

### 3.4 Microestrutural Analysis

Fig. 7 show the microstructure of ABNT M2 steel to Q/2DCT/2T. This route got the best test results. In the optical microscopy (OM) analysis, the carbide precipitation can be seen in the microstructure due to cryogenics and the grain outline. From this, one can calculate the average grain size; the value found was 6.69 µm.

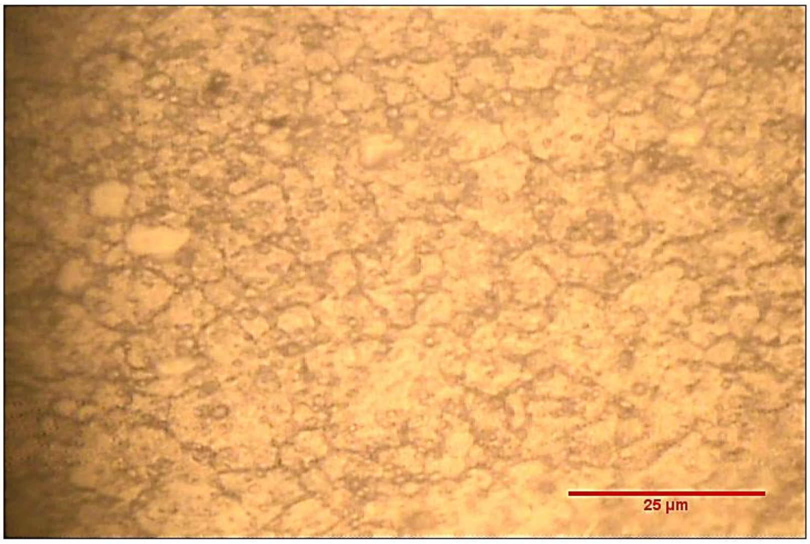
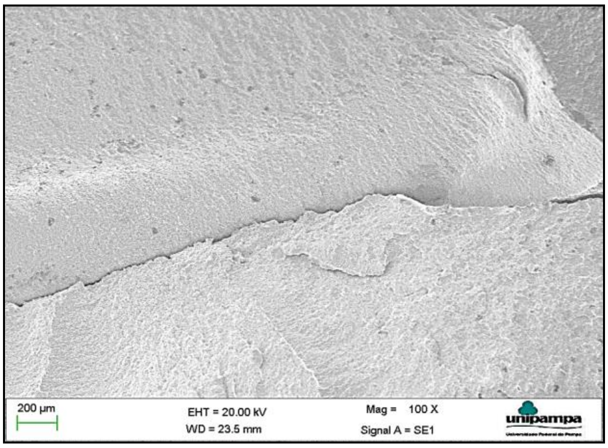


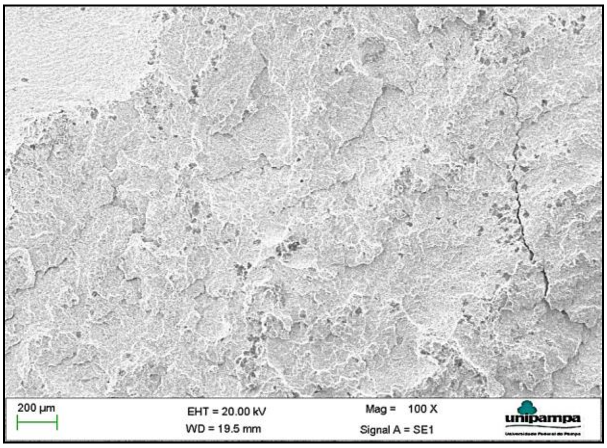

Figure 7. OM – Route Q/2DCT/2R (magnification 800x).

### 3.5 SEM Analysis

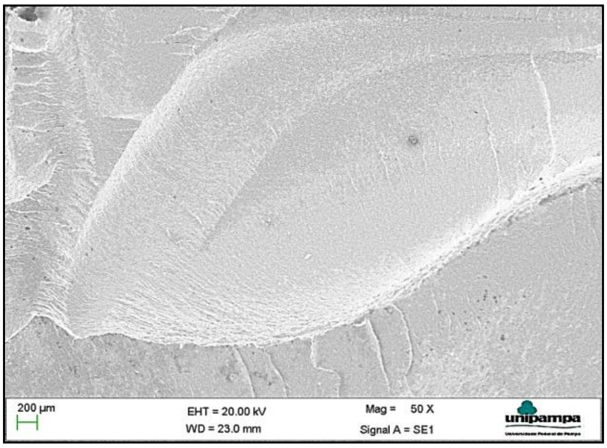
The analyzes of the SEM were made after the Charpy test, the cracks and deformations of the microstructure were evaluated and how the fracture behaved after the impact. For the routes that were submitted to double cryogenics, in the combination with simple tempering (Q/2DCT/T), the cracks appear with greater prominence; this can be analyzed in Fig. 8 (a).



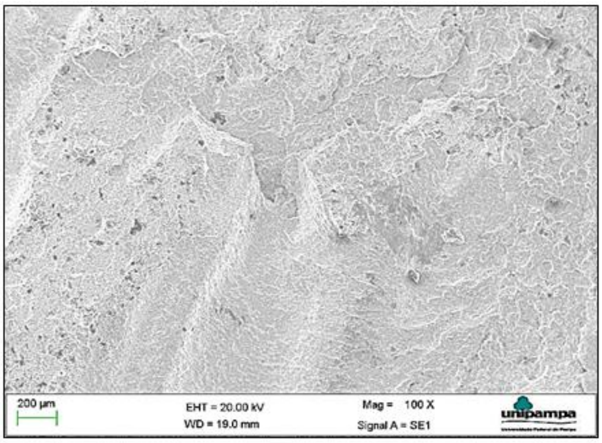

(a)



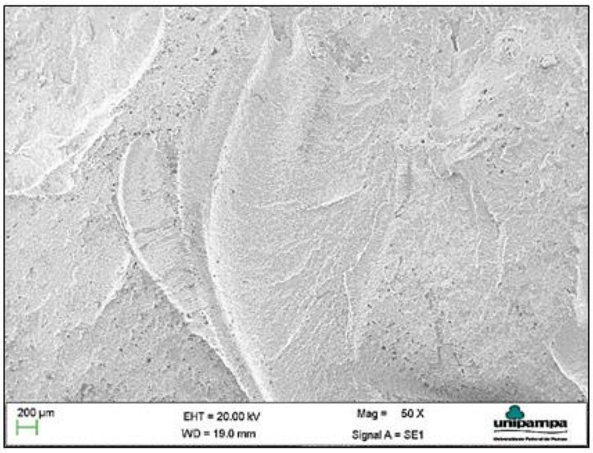

(b)




(c)




(d)




(e)

Figure 8. (a) Route Q/2DCT/T (b) Route Q/DCT/T (c) Route Q/DCT/T (d) Q/2DCT/2T (e) Q/2DCT/2T.

Samples with simple DCT had more evident cracks, which can be observed by the streaks that identify the position of the crack tip. Fig.8 (b) and Fig. 8 (c) shows the analysis of the Q/DCT/T route. It is possible to see a more ductile fracture due to the application of cryogenics, indicating a material with greater tenacity and less fragile in comparison to the other routes.

The Q/2DCT/2T route proved the most efficient in the fracture process, because no cracks were found, fractures only pointed to cavities, which resulted in greater energy absorption, cab bee see in Fig. 8 (d) and Fig. 8 (e).

The fractures shown in the SEM analysis are related to the charpy test. Q/2DCT/T and Q/DCT/T routes had the lowest energy absorption values, for this reason, the fractures in Fig 8 (a) and Fig. 8 (b) are more evident.

## 4. CONCLUSIONS

• The experience of the application of the double cryogen before tempering showed the improvement of the mechanical and tribological properties of ABNT M2 fast steel;

• In the hardness and microhardness tests the best averages obtained were in the route Q/2DCT/2T;

• For the charpy assay, the highest energy absorption average was in the simple cryogenic Q/DCT/2T route;

• The application of the statistical tool ANOVA showed that the cryogenic influences the properties of the material;

• In the SEM analysis, the Q/2DCT /2T route presented a surface with no cracks and a tendency to a ductile fracture in relation to the samples with only conventional treatment;

• The tribological properties improved after the use of the cryogenic treatment, a fact that can be proven through the wear test;

• The fracture surfaces of the 2DCT routes presented evenly than the surfaces of the DCT routes. This indicates that the double cryogen resulted in a more resistant material.

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## 6. RESPONSIBILITY NOTICE

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