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INFLUENCE OF COLLING/HEATING RATE ON AISI M2 TOOL STEEL SUBJECTED TO DEEP CRYOGENIC TREATMENT

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Abstract. The present research focuses on the optimization of the AISI M2 tool steel after deep cryogenic treatment. The cryogenic treatment was performed varying the rate of cooling and the rate of return to the ambient temperature during cryogenics. The samples were austenitized at 1200 ºC and quenched. After this, deep cryogenic treatment was performed in four different rates. The samples from Group A were subjected to a cooling to -181 ±1 °C rate and heating up to room temperature of 0.5 °C / min; The samples from Group B were cooled and heated at a rate of approximately 1.68 °C / min; The samples from Group C were directly immersed in liquid nitrogen at -196 °C while samples from Group D were not subjected to DCT treatment. After the deep cryogenic treatment, all samples were double tempered at 550 °C during 2h each. HRC hardness test, Charpy and wear tests were performed. Statistical analysis of the results showed that deep cryogenic treatment resulted in an increase of 57.55% in toughness obtained in the Charpy impact test and 72.58% increase in the hardness. Also the deep cryogenic treatment represents a 23.09% increase in the wear resistance, these results are in relation to the samples without cryogenic treatment.

Keywords: wear, secondary carbide, hardness, toughness.

1. INTRODUCTION

The AISI M2 tool steel has a high hardenability, high wear resistance and good toughness. In addition, has a martensitic structure of high thermal stability. In addition, has an alloy reinforced with carbides such as chromium, molybdenum, tungsten and vanadium. These steels are widely used in the industry for manufacturing tools (FANTINELI, 2015). Literature shows that the deep cryogenic treatment (DCT), because it is performed at extremely low temperatures compared to the temperatures used for the conventional quenching treatment, provides greater efficiency in the transformation of the retained austenite to martensite and the formation of precipitated fine carbides, resulting in increased performance and durability of the components subjected to this type of heat treatment (GONÇALVES et al., 2019; KUMAR; THIRUMURUGAN; VISWANATH, 2017; VILLA; HANSEN; SOMERS, 2017).

Most tool steels have a martensite start temperature below 0 ° C, all austenite is not transformed into martensite and this retained austenite represents a hardness loss problem (DAVIS, 1995). Cryogenic treatment converts this retained austenite to martensite once the sub-zero temperature is reached and during this treatment compressive residual stresses play an important role in carbide precipitation (SHINDE, 2019). Cryogenics is an extra procedure to conventional quenching and tempering heat treatments and it aims to suppress austenite retained in the quench and improve the mechanical and tribological properties of the material (PADMAKUMAR et al., 2018).

In this study, the DCT-treated samples were analyzed in relation to samples that were submitted to the same heat treatments with different cooling rates. Thus, the present work focuses on finding a logical correlation between the mechanical properties and the influence of the temperature gradient in which the samples were submitted during cryogenic treatment.

2. EXPERIMENTAL

2.1 Materials and Treatments

In this study was investigated the influence of thermal shock in the AISI M2 tool steel submitted to the deep cryogenic treatment (DCT), whose chemical compositions are presented in Tab. 1.

Figure 1 shows how the cryogenic cycles were used for this work and how the heat treatments were distributed. All the samples were austenitized at 1200 °C (performed by– Hurth Infer Machinery and Tools Industry, Sorocaba SP). The DCT were performed using a cryogenic chamber in which the temperature is ramped down to -181 ± 1 °C for 24 hours and then the temperature is ramped up to atmospheric temperature. The cryogenic treatment was carried out in the laboratory of UNIPAMPA. The cryogenic chamber prototype consists of a capsule made of PVC and polyurethane to be used as a sample container. The idea is making the capsule collaborates for a slow cooling transition in the samples.

The influence of thermal shock was investigated by using four different groups of heat treatment, where the most critical parameters were the rate of cooling and the rate of heating during the DCT cycles. The samples were divided in:

- Cycle A: Quenching + DCT for 24 hrs at a cooling rate of 0.50 °C/min + 2 Tempering.
- Cycle B: Quenching + DCT for 24 hrs at an approximate cooling rate of 1.68°C/min + 2 Tempering.
- Cycle C: Quenching + DCT by direct immersion in liquid nitrogen for 24 hrs + 2 Tempering.
- Cycle D: Quenching + 2 Tempering.

Table 1. Chemical composition of the tool steel (AISI M2).

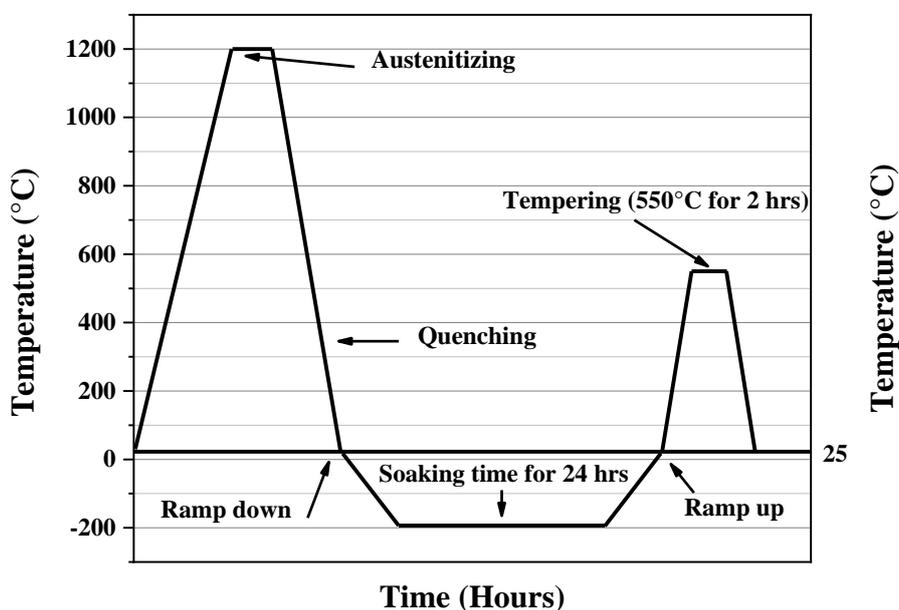| Element	Current weight %	Standard weight %
C	0.81	0.85-1.05
Cr	3.90	3.75-4.50
V	1.83	1.60-2.20
W	7.84	5.50-6.75
Mo	4.72	4.50-5.50

Figure 1. Cryogenic cycle graph and thermal treatments.

2.2 Impact Test

The impact tests were performed according to ASTM E23. No notch was done in the samples used in the Charpy impact test. The Charpy test was performed at the mechanical engineering laboratory of UFSM, where it was used the German manufacture machine of the company VEB Werkstoffprüfmaschinen Leipzig. The value of Charpy impact toughness was measured by using five Charpy impact tests for conventional and varied cryogenically treatment samples and energy was recorded according to Eq. (1).

$$E(J) = E(kg.f.m) \: x \: 9.807 \tag{1}$$

2.3 Hardness Test

Hardness test was performed on a Rockwell-C hardness (HRC), Model: TH500, Make: TIME, using diamond spheroconical indenter 120° with an applied indentation load of 150 kgf and dwell time of 10 s. Five hardness readings were taken at different points to estimate the average value of hardness for each sample.

2.4 Wear Resistance

For the wear test was used the equipment Phoenix Tribology, model TE 53 SLIM with the parameters: 400 cycles; sandpaper 120 grains/in²; 100 rpm of angular speed; on the tests were applied loads of 5 kgf and by using the multiplication system of the equipment itself, applied 287 N load on the sample. The rehearsal was Block type on ring with samples being frictioned against a moving cylinder as shown in the Fig. 2, samples were cut to 12.7 mm length and subjected to the abrasive wear test. Abrasion tests were carried out in the laboratory of UNIPAMPA. The wear rate was determined as the mass loss of the specimen material by electronic weighing precision balance of Schimadzu brand with 0.1 mg resolution. Mass loss was obtained according to Eq. 2.
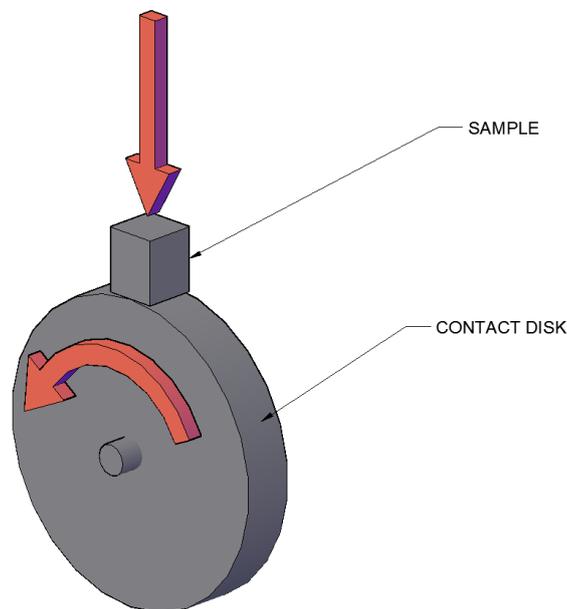

Figure 2. Wear test. Adapted from FANTINELI (2015).

$$weight \: loss = \left(\frac{m_i - m_f}{m_i} \right) \times 100 \tag{2}$$

At where:

m_i - Initial mass;

m_f - Mass of samples after the test.

2.5 Statistical Analysis

Thirty samples were prepared, where: 12 for cycle A; 6 for cycle B; 6 for cycle C and 6 for cycle D. The mean, the standard deviation and coefficient of variation for Charpy test, HRC hardness test and wear test were verified. It was also checked if the samples had normal distribution; therefore, were performed the ANOVA analysis in all the assays and Tukey test in cases where there was significant difference between cycles.

3. RESULTS AND DISCUSSIONS

3.1 Impact Toughness

The impact energy for all the samples is tabulated in Tab. 2 and Fig. 3 shows the toughness values of the base material with DCT after the Charpy impact test. In this analysis, it was possible to observe that the control of the cooling rate and the heating rate during the DCT are extremely important factors, as well as described by Molinari et al. (2001).

The specimens that were submitted to a route with a lower temperature gradient, such as cycles A and B, showed an improvement in the energy absorption during the fracture, what is an increase in the tenacity. According to HUANG et al. (2003) the probable cause of the increase of tenacity is due to the fact that the precipitation of carbides during the application of the DCT reduces the amount of carbon in the metal matrix, which leads to increase in matrix toughness (FANTINELI, 2015).

Table 2. Data from impact Charpy test.

Routes	Average (J)	Standart deviation	Coefficient of variation	Increase compared samples without DCT
Cycle A	37.07	6.26	16.88%	32.63%
Cycle B	34.52	6.80	19.70%	23.51%
Cycle C	23.11	2.97	12.85%	-17.33%
Cycle D	27.95	4.10	14.68%	-

However, the data showed a normal distribution. So, in this study, from the statistical point of view, the thermal treatments present a significant difference in at least one of the cases. This was verified by using the analysis of variance (ANOVA), where the Fisher test presented a probability of significance lower than 5%.

Table 3. ANOVA of Charpy test.

Source	Degrees of freedom	Sum of squares	Mean square	F	P
Cycle A Cycle B Cycle C Cycle D	3	602.05	200.68	7.23	0.27%
Error	16	444.13	27.76		
Total	19	1046.18			

According to the Tukey test, there is a significant difference between cycles A and C and cycles B and C. These data can be verified in Tab. 3 and Tab. 4. The results show that deep cryogenic treatment represents a 57.55% increase in the toughness of AISI M2 steel.

Table 4. Tukey of Charpy test.

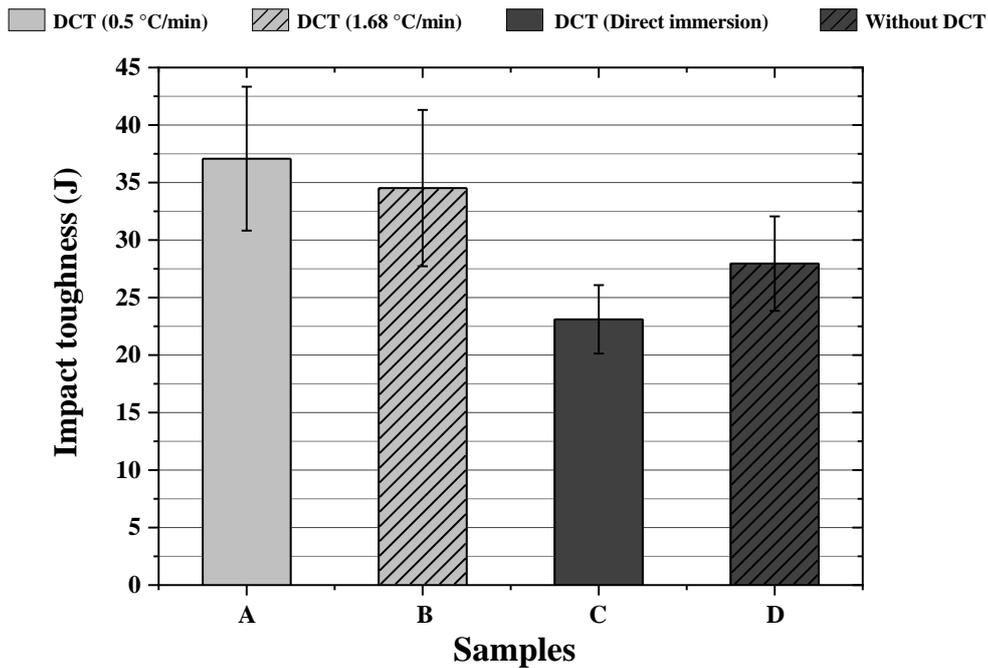|	Cycle A	Cycle B	Cycle C	Cycle D
Cycle A	-	0.8688	0.0035	0.0633
Cycle B	1.0820	-	0.0164	0.2391
Cycle C	5.9270	4.8450	-	0.4862
Cycle D	3.8710	2.7890	2.0560	-

Figure 3. Charpy test.

Table 5 shows that greater control of thermal shock represents 59.48% of the improvement in the results observed in this study. These data were verified in relation to cycles A, B and C as these cycles were submitted to DCT.

Table 5. ANOVA of Charpy test subject DCT.

Source	Degrees of freedom	Sum of squares	Mean square	F	P
Cycle A Cycle B Cycle C	2	553.03	276.52	8.81	0.44%
Error	12	376.81	31.40		
Total	14	929.84			

3.2 Hardness

The hardness was measured on the different specimens and the results are reported in Tab. 6 and Fig. 4. The hardness of the DCT samples showed better results than samples that were not submitted to the deep cryogenic treatment.

The reduction in standard deviation after DCT application indicates that there was a greater homogenization of the microstructure. According to HUANG et al. (2003) cryogenic treatment not only facilitates the formation of carbides, but also makes them have more homogeneous distribution. It is noticed that the results of hardness increment is greater than the coefficient of variation, these results were also described by FANTINELI (2015).

Table 6. Data from impact Hardness test.

Routes	Average (HRC)	Standart deviation	Coefficient of variation	Increase compared samples without DCT
Cycle A	63.59	1.0041	1.57%	5.40%
Cycle B	62.84	0.8513	1.35%	4.17%
Cycle C	63.03	0.4734	0.75%	4.48%
Cycle D	60.33	1.0065	1.66%	-

The data showed a normal distribution for Hardness test. So, according to Tab. 7, from the statistical point of view, the thermal treatments present a significant difference in at least one of the cases. This was verified by using the analysis of variance (ANOVA), where the Fisher test presented a probability of significance lower than 5%.

The results show that deep cryogenic treatment represents a 72.58% increase in the hardness of AISI M2 steel. However, the results of samples submitted to DCT did not have significant influence between cycles A, B and C.

Table 7. ANOVA of Hardness test.

Source	Degrees of freedom	Sum of squares	Mean square	F	P
Cycle A Cycle B Cycle C Cycle D	3	31.44	10.48	14.11	0.0092%
Error	16	11.89	0.74		
Total	19	43.32			

According to the Tukey test, there is a significant difference between all cycles in relation to cycle D. These data can be verified in Tab. 8.

Table 8. Tukey of Hardness test.

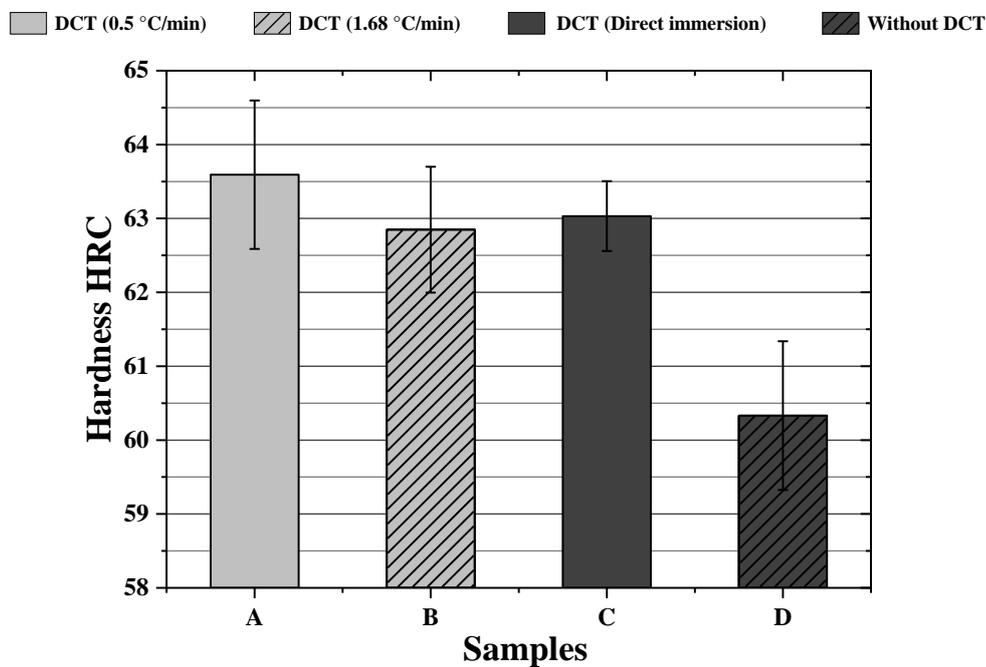|	Cycle A	Cycle B	Cycle C	Cycle D
Cycle A	-	0.5375	0.7362	0.0001
Cycle B	1.9310	-	0.9863	0.0014
Cycle C	1.4530	0.4775	-	0.0007
Cycle D	8.4590	6.5290	7.0060	-

Figure 4. Hardness test.

3.3 Abrasion Test

It is observed that cycle A presented less mass loss compared to the other cycles in the wear test. However, cycles B and C also had a good result in relation to cycle D. These data can be viewed in Tab. 9 and Fig. 5. The results show that deep cryogenic treatment represents a 23.09% increase in the resistance in to wear and that the greater control of thermal shock represents 15.76% in relation to cycles A, B and C as these cycles were submitted to DCT.

Table 9. Data from Abrasion test.

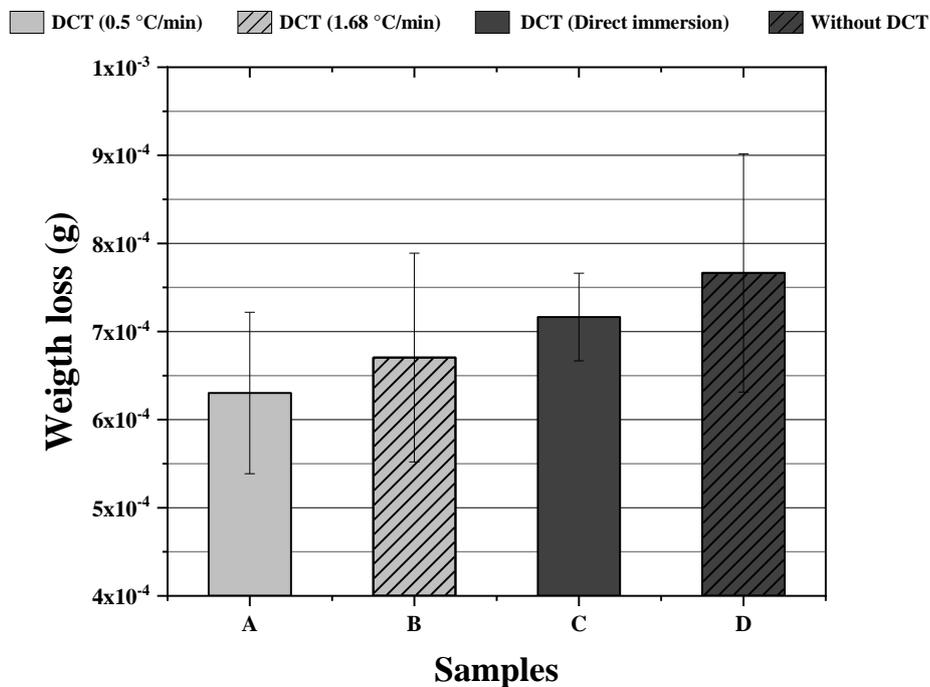| Routes	Average (mg)	Standart deviation	Coefficient of variation	Increase compared samples without DCT
Cycle A	0.63	9.16×10^{-5}	14.53	-17.78%
Cycle B	0.67	1.18×10^{-4}	17.66	-12.53%
Cycle C	0.71	4.97×10^{-5}	6.94	-6.53%
Cycle D	0.76	1.35×10^{-4}	17.64	-

Figure 5. Abrasion test.

Figure 6 shows the radar graph type, comparing the averages performed for all tests. It is noticed that cycle A presents the best results in all aspects observed in this study.

Prior research has shown that deep cryogenic treatment refines secondary carbides, increases their amount and population density, and leads to more uniform distribution by categorization of secondary carbides. Therefore, according to Fig. 6, where the energy absorption, hardness and wear tests for all cycles presented in this study were related, it is clear that DCT with a better cooling rate control promotes a more homogeneous distribution in carbides precipitation, resulting in the improvement of the mechanical properties of the material (LI et al., 2013).
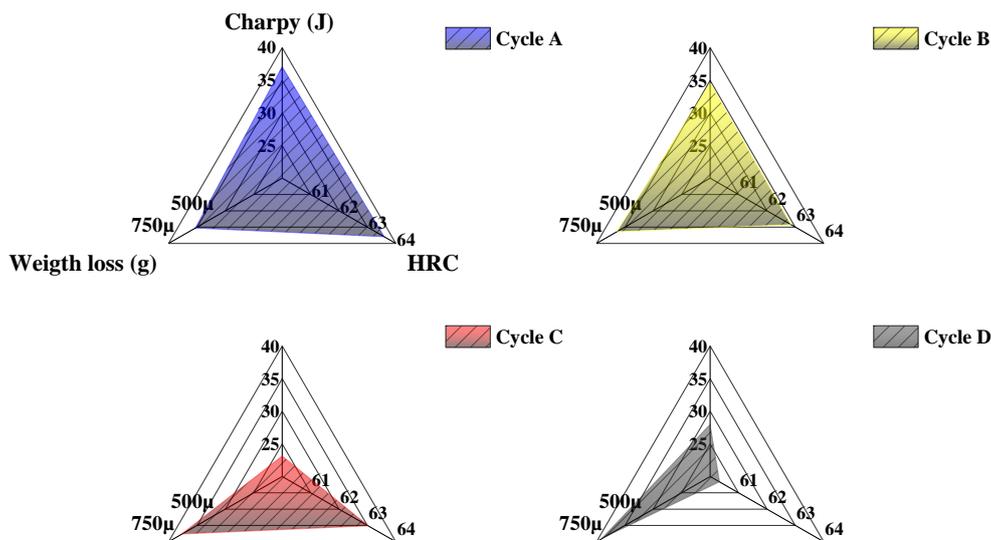
Figure 6. Analysis comparisons.

4. CONCLUSIONS

- Samples cryogenic treated at cooling/heating rate of 0.5 °C / min presented an improvement in the toughness of 32.63%, while samples cryogenic treated at cooling/heating rate of 1.68°C / min presented an improvement of 23.51%. Samples cooled direct in liquid nitrogen reduced their toughness compared with samples without cryogenic treatment;
- DCT promotes carbide homogenization in the metal matrix, reducing the standard deviation in hardness tests;
- The deep cryogenic treatment represents a 72.58% increase in the hardness test;
- Samples cryogenic treated at cooling/heating rate of 0.5 °C / min presented an improvement in the hardness test of 5.40%;
- The deep cryogenic treatment represents a 23.09% increase in the resistance in to wear;
- DCT at cooling/heating rate of 0.5 °C / min resulted in an increase of 17.78% in the wear resistance compared to sample without DCT;
- DCT reduced the mass loss for all cycles investigated;
- DCT at cooling/heating rate of 0.5 °C / min presented the best results in toughness, wear resistance and hardness test. The improvement is associated with the more uniform homogenization of the carbides after the DCT submitted to the lowest thermal shock.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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