

STUDY OF THE INFLUENCE OF LOW TEMPERATURE TREATMENTS IN THE AISI H13 STEEL

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Abstract. *Several studies of the influence of the parameters of heat treatment have been developed to obtain properties that could meet the requirements of work and tools that generate useful life extended. In this context, the use of cryogenic treatment in steel led to a significant improvement in their properties, with regard to increased wear resistance and toughness. These results are due to three factors: the transformation of residual austenite, which brings an improvement in dimensional piece, the standardization of fine carbides and reduce the residual stress. These benefits are dependent on values of low temperature used and length of stay in these temperatures, a fact inconsistent between literatures.*

It's important to check the effectiveness of cryogenic treatment on increasing the lifetime of the tool because the maintenance of these baths is expensive and makes sense only if the gain exceeds the spending on properties with treatment. Information such as these is of fundamental importance for the companies providing such service.

Present study intend to show AISI H13 steel performance in terms of Impact Charpy Energy and hardness under different heat treatment conditions. Cryogenic bath (-196°C) is carried out after and before tempering and different holding times in the cryogenic bath were used.

Conventional tempering and quenching heat treatment for AISI H13 was used the baseline.

Results were compared among those different conditions and the best performance in impact Charpy energy was obtained for the cryogenic bath (1 hour) carried out before tempering. Hardness results did not presented great changes with the heat treatment conditions.

Keywords: Heat Treatment, AISI H13 steel, Cryogenic.

1. INTRODUCTION

The interest on the effect of low temperatures cycles treatments in the steels is not new. As early as the 1930's, studies were made to improve the steels properties using sub zero treatment. Tests were conducted at temperatures of about -80°C (using methanol, dry ice or freon), the purpose of the process was to transform residual austenite, retained after initial hardening to martensite, in order to stabilise the tempered structure. In the 1970s, the development of low-temperature technology had extended the cryogenic treatment temperature down to -196°C (about liquid-nitrogen temperature (Yun *et. al*, 1998)).

Cryogenic treatments of alloy steels have been claimed to increase significant wear resistance and toughness through the interplay of three effects: completing martensitic transformation, promoting uniform precipitation of fine carbides and imparting residual stresses (Zurecki, 2005).

Collins and Dormer (1997) claimed that the process of "low temperature conditioning" of martensite, normally at liquid-nitrogen temperatures, increases both toughness and wear resistance. During this process it's initiated the nucleation of sites for subsequent precipitation of fine carbides particles in the tempered microstructure. Properties improvement are influenced by the time and temperature adopted in the low temperatures treatment. Literature shows different opinions about this subject. These benefits are dependent on values of low temperature used and the holding time in the low temperature bath, a fact inconsistent between literatures (Collins, 1996).

The purpose of this work is to study the characteristics of AISI H13 tool steel heat treated using the conventional tempering and hardening treatments with cryogenic bath. In some samples the cryogenic bath was done before the tempering and, in other samples, after the tempering. The time of soaking utilized was: 1hour, 24hours, and 48hours. Results of Charpy impact test and hardness test was compared with those obtained from conventional tempering and hardening.

2. LOW TEMPERATURE TREATMENTS IN THE AISI H13 STEEL

The AISI H13 tool steel has the composition (NADCA, 1997) described in the Tab. 1, It has excellent resistance high-temperature impact loading, resistance to softening during high-temperature exposure, and very good performance in terms of thermal fatigue.

The composition medium-carbon content promotes toughness by limiting the carbon concentration of the martensite and by limiting the size of alloy carbides particules. Good high-temperature strength is achieved by tempering at high temperatures where fine and stable dispersions of chromium and vanadium alloy carbides precipitate.

These carbides coarsen only in service. The high- alloy content of the H steels also provides excellent hardenability and permits the hardening of heavy sections by air cooling (Roberts *et.al*, 1998).

Table 1. Chemistry composition of AISI H13 steel

Composition % weight	C	Mn	Si	Cr	V	Mo
Minimum	0,37	0,20	0,80	5,00	0,80	1,20
Maximum	0,42	0,50	1,20	5,50	1,20	1,75

In the heat treatment of steels is important to prevent surface decarburization and oxidation of component. And vacuum furnaces which also that increases uniform cooling is recommended. Heat treatment parameters of heat (austenitizing temperature, rate of heating and cooling, difference between surface and nucleus in the heating and cooling) follow the regulation of NADCA (2003). The Figure 1 shows the cycle of heat treatment for AISI H13 tool steels.

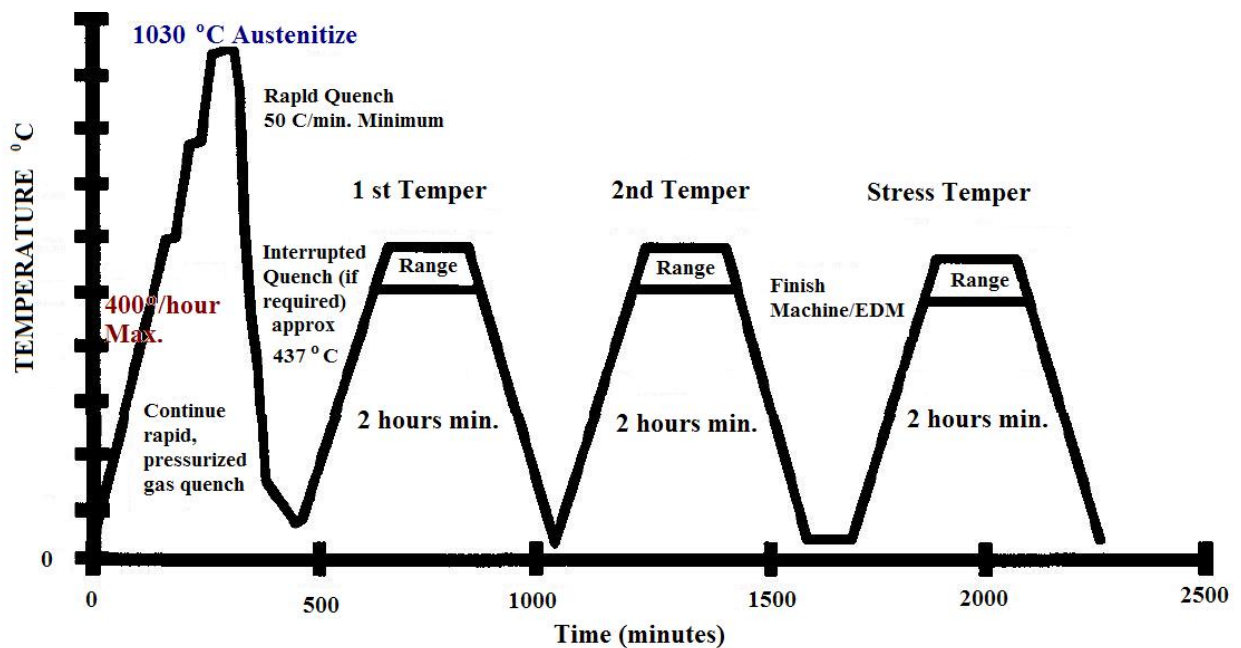


Figure 1. Schematic heat treatment Cycles - adapted

The main constituents of heat treating a tool steels, retained austenite, martensite and carbides, can have been changes in the quantity, dimension and distribution.

Since the late 19th, problems due to excessive retained austenite had plagued the tool-steel industry; excessive retained austenite is universally considered to be detrimental (Voort, 2009). The amount of retained austenite can increase due to various factors, increasing austenite temperature that increases austenite grain size, and also alloy and carbon dissolution, further reducing M_s . In plain-carbon and low-alloy steels, retained austenite transforms to bainite, or is stabilized, at relatively low tempering temperatures.

In high-alloy steels, austenite remains untransformed at tempering temperatures up to about 450°C or higher, at which temperature it becomes “conditioned” by carbide precipitation, transforming to martensite (of lower carbon and alloy content) on cooling back to room temperature (Collins, 1996).

Martensite is supersaturated with carbon which, during tempering, precipitates out as carbides, depending on alloy content and tempering temperature. The instability of martensite is associated with the strain energy relating to its dislocation/twin lath boundaries and martensite/retained austenite boundaries. At very low temperatures, the activation energy for carbon diffusion (and alloy diffusion) is too high to permit formation of carbide precipitates as in the final stages of tempering.

Type of carbide formed during tempering depends mainly on alloy content and tempering temperature. The nucleation of carbide and growth are time-dependent. Some carbide in the final microstructure will be those that remained undissolved during the austenitizing treatment.

Low tempering is widely used for high precision parts and components, since it enhances the transformation of austenite to martensite, also stabilises the dimensions of the piece. This effect is largely complete for most steels at

temperatures of between -80 and -110°C, and it has been claimed it to increase wear resistance, hardness, dimensional stability and reduction in toughness.

Cryogenic bath transforms residual austenite into martensite and alters the morphology of martensite and precipitates out ultrafine carbides (Collins And Dormer, 1997). If martensite holding enough time at the low temperature it will be conditioned, due to crystalline lattice tends to decrease, the crystal deformation of martensite in the supersaturated solid solution tends to increase, and martensite is in a more unstable thermodynamic state. As a result, martensite decomposes, precipitating out carbon atoms and increasing the thermodynamic driving force (Yun *et. al.* 1998).

On subsequent heating up to or above room temperature, these sites act as nuclei for the formation of the fine carbide particles observed in cryogenic treated steels. The time at the cryogenic bath is contentious.

However, few authors claimed that no secondary hardening occurs if steel is previously tempered in the normal secondary-hardening temperature range (Collins 1996). In literature there aren't agreements about the parameters of cryogenic treatments, and the results of hardness, toughness and wear resistance can be different (Molinari *et. al.*, 2001)

Although some researches conclude relationship between these parameters, there are results showing that the parameters relation cannot exist (Zurecki, 2005).

3. EXPERIMENTAL PROCEDURES

3.1. Heat Treatment

Same austenitizing time and temperature and same oil quenching conditions were used in all the six set of experiments conditions (total of 18 samples). Test matrix, Fig. 2, shows six different heat treatment conditions, and there were three sets of AISI H13 steel samples of each type treated within each condition in order to produce statistically valid averages. Heat parameters for vacuum furnaces were:

- Austenitizing: 1040°C for half hour
- Cooling: 5 bar of N₂
- Tempering: 540°C - 2 hours

3.2. Cryogenic Treatment

Cryogenic quenching and different holding time bath were holding was realized in liquid nitrogen bath (-196°C) for conditions C2 to C6. Condition C1 was the baseline or the only condition within the entire test matrix that did not involve cryogenic.

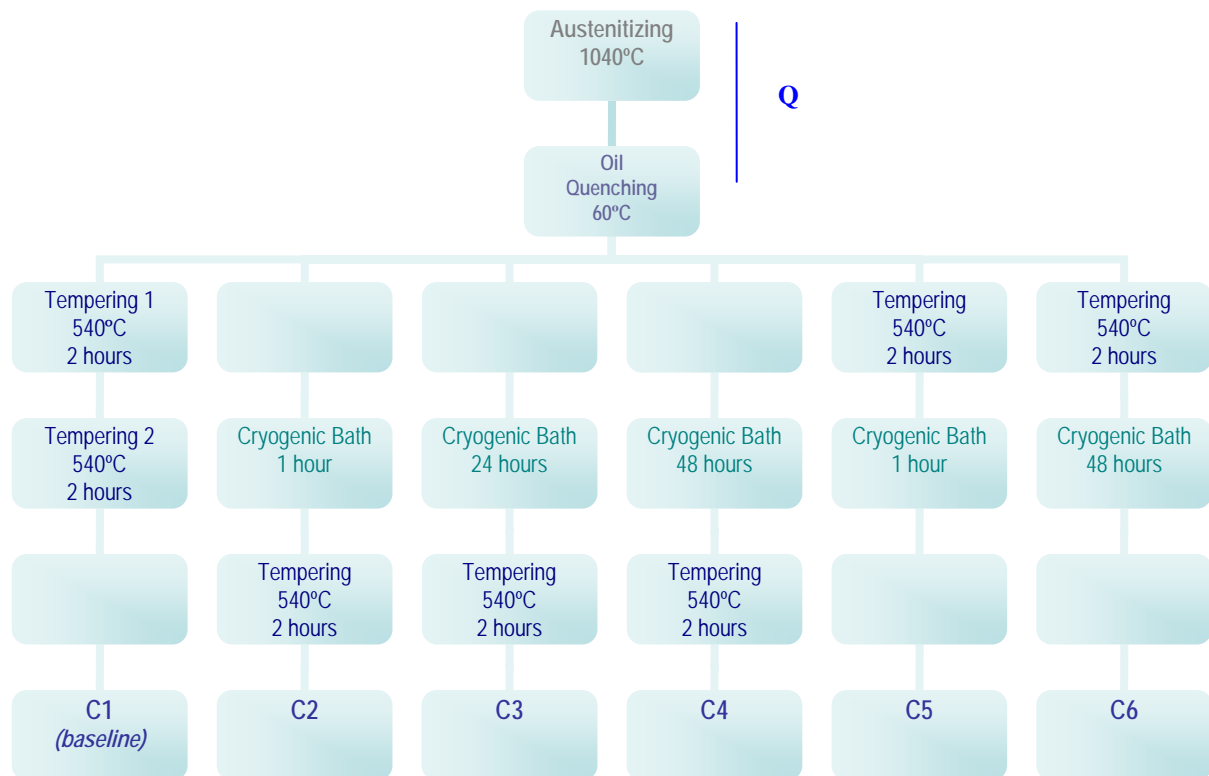


Figure 2 – Heat treatment test matrix for Cryogenic Treatment – (Q) means quenched, (T) tempered, and (Cryo) Cryogenic bath.

3.3. Mechanical Tests

Charpy V-notch instrumented impact testing was performed in a 300J impact pendulum manufactured by Instron Wolpert PW30.

The testing was performed according to the ASTM E23 (2007), sample dimension are showed in the Fig. 3. There were cut in the short transverse direction of the bar. The Rockwell-C- hardness was collected using a LECORT-240 durometer, with load 150kgf, and results are the average of 4 measurements. Conventional techniques were used to preparative samples to optical microscopy.

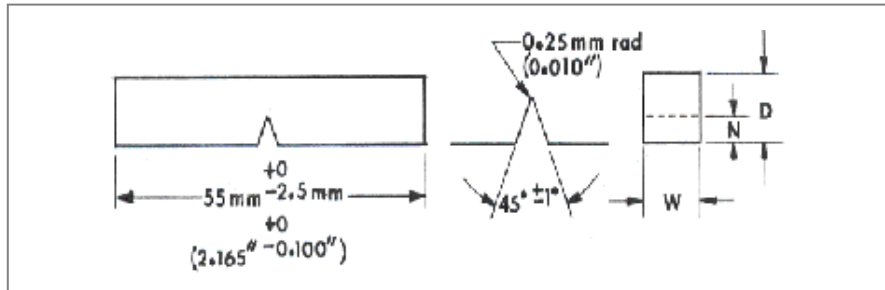


Figure 3- Dimension Charpy sample

4. RESULTS AND DISCUSSION

The condition 1 is the baseline, and the routes followed the schedules described in fig 2. The table 2 shows the Energy Impact tests and Hardness test for the conditions C1- C6.

Table 2. Energy Impact tests and Hardness test for the conditions

Conditions	Impact Energy	Hardness
C1	70	53
C2	82	53
C3	70	53
C4	66	51
C5	78	53
C6	77	52

In order to know how much better the conditions are C2- C6 were compared to the baseline condition results and they are shown in figure 5.

Regarding to hardness values, all the used heat treatment conditions promoted similar values for this property. A decrease of about 1 point in HRC was only observed to C4 condition (Q+Cryo 48h+T). Studies performed by Yun *et al.* (1998) suggested that higher hardness is obtained when tempering is performed after cryogenic bath. According them if the tempering is performed before cryogenic bath, an ageing process will occur, decreasing the effective ageing during the cryogenic bath.

Molinari *et al.* (2001) working with AISI H13 steel obtained a small increase in hardness for cryogenic bath performed before tempering. According them when the cryogenic treatments is carried out after quenching and followed by the usual tempering cycle, its influence on the properties of steels is negligible, including hardness. In the present work, same conclusion was obtained, once under these heat treatment conditions, hardness values were close. Regarding to Impact energy (E), C3 (Q+Cryo24h+T) and C4 (Q+Cryo48h+T) present a very little difference compared to the C1 (baseline). However C2 (Q+Cryo1h+T) condition shown best performance. Conditions C5 (Q+T+Cryo1h) and C6 (Q+T+Cryo48) shown similar performance.

For Impact energy, tempering before or after cryogenic bath has influence in the obtained results. In the C2 condition, cryogenic bath was performed before tempering and best Impact energy result was obtained. Therefore, as mentioned before, Molinari *et al.* (2001) found negligible improve when cryogenic bath is performed before tempering. However this work is in agreement with Molinari *et al.* (2001), Collins (1996), Meng (1994), since cryogenic bath is useful to increase the impact energy and so toughness.

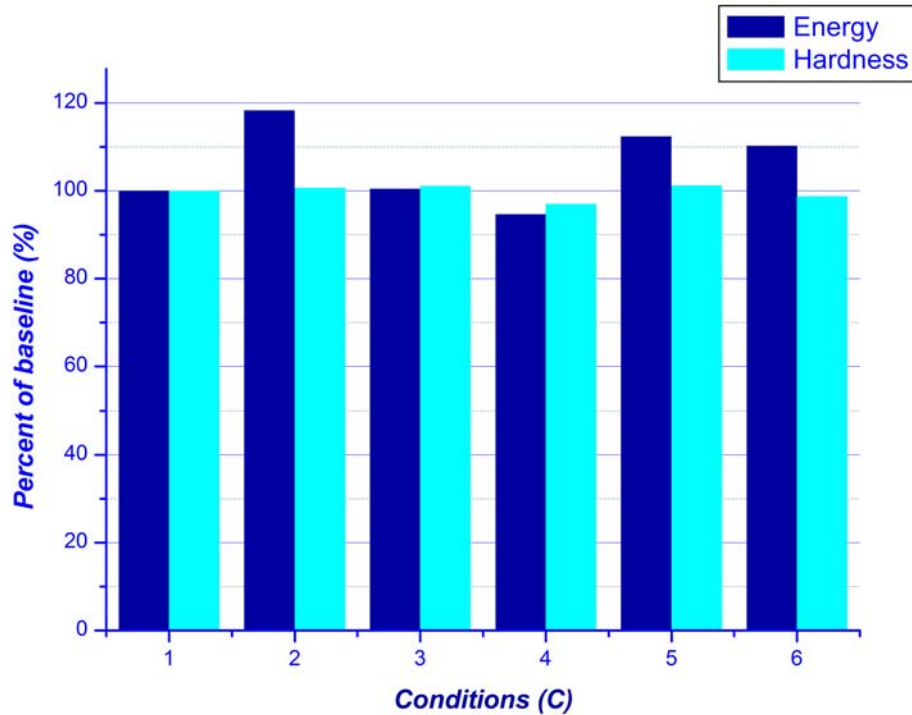


Figure 5 – Average values of impact energy and hardness measured for conditions C1-C6 in %.

Microstructure definition using optical microscopy did not show significant differences among the samples and Fig. 6 is all samples representative through SEM and TEM analyses it hopes to see differences among the samples.

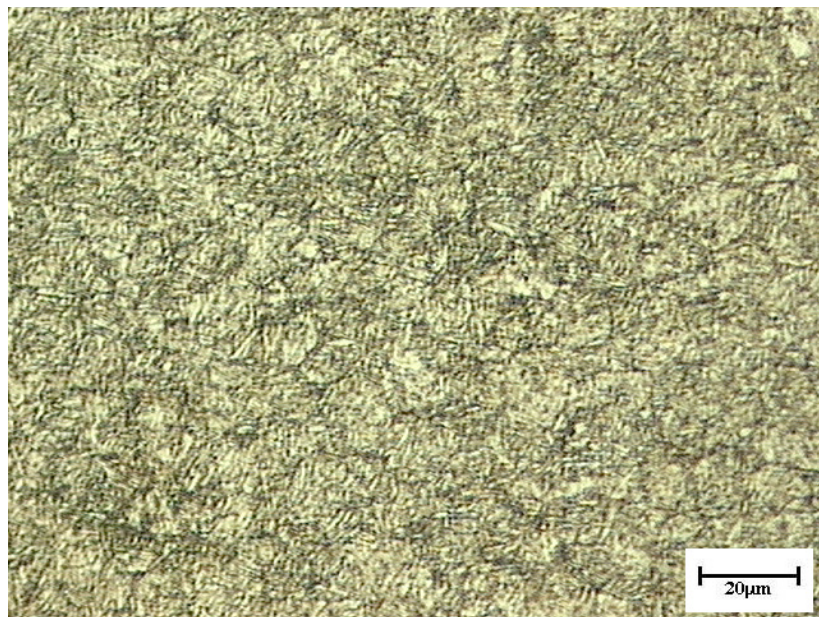


Figure 6 – Microstructure of H13 (C₂).

Results showed that holding time in the cryogenic bath doesn't directly affect the physical properties, due to the fact that the samples with larger holding time don't show the best results.

5. CONCLUSIONS

Cryogenic bath of tempering and quenched AISI H13 steel improves about 20% the impact energy. This improvement was obtained when cryogenic bath was carried out after quenching and before tempering. Lower improvement was observed when cryogenic bath was performed after tempering and quenching. The rise of holding

time in the cryogenic bath had no positive influence in this process. One hour at the cryogenic bath promoted best impact energy value.

Cryogenic bath had no influence in the hardness values compared to the baseline condition (C1).

Results obtained are not exhaustive and TEM analyses are being performed in order to get a better understanding of these processes.

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7. REFERENCES

ASTM E23, 2007, "Standard Test Methods for Notched Bar Impact Testing of Metallic Materials".

Collins, D. N., 1996, "Deep Cryogenic Treatment of tool steels: a review", Heat Treatment of Metals. Vol.2, pp 40-42.

Collins D. N., and Dormer J., 1997, "Deep Cryogenic Treatment of a D2 Cold-work Tool Steel", Heat treatments of Metals, Vol.3, pp 71-74.

Jespersion, H., 2005, "Influence of the heat treatment on the toughness of some hot-work tool steel grades", TRATTAMENTI TERMICI 2/2006; Paper presented at the 2nd International Conference HEAT TREATMENT AND SURFACE ENGINEERING IN AUTOMOTIVE APPLICATIONS, Riva del Garda, 20-22 June 2005.

Meng, F., Tagashira, K., Azuma, R., and Sohma, H., 1994, "Role of Eta-carbide Precipitations in the Wear Resistance Improvements of Fe-12-Cv-Mo-V-1.4C Tool Steel by Cryogenic Treatment", ISIJ International, Vol.34, No.2, pp 205-210.

Molinari, A., Pellizzari, M., Gialanella, S., Straffelini, G., and Atiasny, K. H., 2001, "Effect of Deep Cryogenic Treatment on the Mechanical Properties of Tool Steels", Journal of Materials Processing Technology Vol. 118 pp 350-355. Brunico, Italy.

NADCA, 1997, "Recommended Procedures, H-13 Tool Steel". USA.

NADCA, 2003, "Premium and Superior Quality H13 Steel and Heat Treatment Acceptance Criteria for Pressure Die Casting Dies" NADCA #207-2003.

Roberts, G., Krauss, G., and Kennedy, R., 1998, "Tool Steels", 5th edition, ASM International, USA, pp. 14-15.

Voort, G. F. V., 2009. "Martensite and Retained Austenite", Industrial Heating.com. April 2009. <<http://www.industrialheating.com>>.

Yun, D., Xiaoping, L., and Hongshen, X., 1998, "Deep Cryogenic Treatment of High-speed Steel and its Mechanism", Heat Treatment of Metals, Vol.3 pp 55-59.

Zurecki, Z., 2005, "Cryogenic Quenching of Steel Revisited", Air Products and Chemicals, Inc., Pennsylvania, USA.

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