



STUDY OF INFLUENCE OF CRYOGENIC BATH IN THE AISI H13 STEELS PROPERTIES¹

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Abstract

The present work is to study the influence of cryogenic treatment on AISI H13 hot work steel. To this end several cycles of heat treatments were performed with conventional quenching and tempering and cryogenic treatment. In some samples the cryogenic treatment is done before tempering, in others after, with soaking times of 1 hour, 24 hours and 48 hours. To evaluate the efficiency of cryogenic treatment on AISI H13 steel, the hardness, Charpy impact and wear resistance were compared with those from heat-treated AISI H13 conventionally. The results were analyzed, compared and discussed. We conclude that the cryogenic bath did not add improvements over hardness values obtained with conventional treatment. The cryogenic treatment performed in conjunction with heat treatments of quenching and tempered may increase about 20% the energy of impact, and the soaking time of the bath cryogenic not presented positively influence this process, because the best value of impact energy wascryogenic bath was lasting an hour. The best wear resistance was presented by the stay of 48 hours in the bath after cryogenic tempering.

Key words: Heat Treatment; Steel H13; Cryogenic.

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1 INTRODUCTION

The interest in heat treatments on steels at low temperature is not new. Since 1930, studies show an improvement in the properties of steels exposed to low temperature. The treatments were originally performed in the range of -80° C, known today as a sub-zero treatment (with the use of methanol, dry gel or freon), with the goal of transforming the retained austenite after quenching and improve the stabilization of martensite.^[1]

Starting in 1970 with the development of the low temperature technology, initiated the use of cryogenic treatment with temperatures ranging from -196 °C using liquid nitrogen, promoting the precipitation of fine carbides, and an increase in toughness and wear resistance.^[2] These benefits are dependent on the values of low temperature and also used the time spent at these temperatures.^[3]

However there is no agreement about the real benefits of this treatment.^[4] This debate has been extended for years and many professionals in the metallurgical industry have serious restrictions on the use of treatments and sub-zero cryogenic attributed to lack of knowledge technology, as well as the absence of generally accepted procedures for applying it.^[5]

The aim of this work is to study the influence of cryogenic treatment on hot work steel AISI H13, widely used in industry for use in extrusion and injection molds, hot forming presses and hammers. To that end several cycles of heat treatments were performed with conventional guenching and tempering and cryogenic treatment.

In some samples the cryogenic treatment is done before tempering, in others after, with soaking times of 1 hour, 24 hours, and 48 hours.

To evaluate the effectiveness of cryogenic treatment on AISI H13 steel, the hardness, toughness and wear resistance, were compared with those from heattreated AISI H13 conventionally. This will allow it to generate their own results, analyzing the advantages and disadvantages of using these processes in the industry, enabling an analysis of the feasibility of its application.

The steel classified as Premium AISI H13, has the chemical composition described in Table 1 according to the specifications of the North American Die Casting Association NADCA,^[6] which gives it properties: good hardenability, high resistance to softening by heat, good wear resistance at elevated temperatures, excellent toughness, good machinability in the category of tool steels and excellent resistance to thermal shock due to continuous heating and cooling, causing the appearance of thermal cracks is reduced.

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

Table 1. Chemical composition in weigth % of AISI H13

Search: ASM Metals Handbook^{1/1}

Several factors affect the final results of a temper and, therefore, the treatment parameters should be well defined so that it obtain the required properties for the component, it is also important to avoid decarburization and surface component.

In the treatment of steels for hot working, the austenitizing temperature and time of austenitizing temperature are well known. Usually makes two tempered, the third

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being optional.^[2] The parameters of heat treatment (austenitizing temperature, cooling and heating rates and differences between surface and core in heating and cooling) are recommended by manufacturers of steel, as well as by NADCA standards such as,^[6] which recommends specific cycles of quenching and tempering.

The tempering is done with pre-heating temperature from 600°C and 850°C and austenitizing temperature between 980 ° C and 1040 ° C with residence time at temperature between 30 and 60 minutes. After the soaking time elapsed between 30 and 45 minutes, there is cooling, which is a difficult operation and directly responsible for the final results of treatment. Thus, tool steels are normally used in vacuum furnaces for heat treatment,^[8] which aims at removing the elements that compose the atmosphere tempering avoiding, among other things, the oxidation and decarburization of the material treated, and additionally offers excellent uniformity of cooling, it is possible to control the cooling speed of the play and getting to the desired properties in tempera.

Temper has been the formation of martensite microstructure and retained austenite and carbides (which are not transformed during quenching) may have changes in their quantity, size, and distribution.

During tempering, the martensite becomes saturated with carbon, which precipitates in the form of carbides, varying according to the composition of the steel. The type of carbide formed is dependent on alloy composition and the tempering temperature may, in tool steels, promote secondary hardening, or the maintenance of hardness with increasing tempering temperature, as shown in Figure 1.



Figure 1. Hardness variation with Tempering temperature for AISI H13 steel.

The size and distribution of the carbide precipitates out of the martensite are dependent on the nucleation, the thermal history of material and austenitizing temperature. Treatment sub-zero and has been used in cryogenic steels with high amount of retained austenite, using various liquefied gases to obtain the desired temperature.^[10] This treatment performed on steels highly connected, can cause two distinct phenomena, which will cause different effects:

• Temperature of 110°C-80°C to-: the transformation of retained austenite. In many steels has not yet been the stabilization of retained austenite may occur on a





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large scale. In this case, the effects achieved are: increased hardness, reduction in toughness, slightly improved performance in wear and dimensional stability, and

• In the range of liquid nitrogen: when the component stays long enough at this low temperature, precipitates of fine carbides (depending on the alloying of steel) that add to carbides present before cryogenic treatment. The result is that there is an improvement in wear resistance, toughness gain with a slight increase (or not) of hardness.^[11]

What happens in martensite is that, if given sufficient time at low temperature, there is an instability of martensite, and C atoms can migrate forming sites. There is a "conditioning" of martensite. On subsequent heating to room temperature, these sites act as nuclei for the formation of fine particles of carbide precipitates.

However, according to some authors, this secondary hardening effect will not occur if it gets guenched prior to the cryogenic process in temperature ranges that promote secondary hardening during tempering. The literature is also controversial on the issue of residence time (at extremely low temperatures) required for the phenomenon of "aging" occurs. There are studies arguing that two hours is sufficient and other times calling for as long as 72 hours were required for this phenomenon,^[5] the treatment and sub-zero cryogenic aspect in microstructural differ basically by the fact that apart from stepping up the transformation of retained austenite into martensite (happens on both), the cryogenic treatment promotes the precipitation of carbides.[12]`

Although some results have been presented, there is some disagreement in the literature with respect to some parameters of cryogenic treatments, as already discussed issue related to length of stay. Another discrepancy concerns the influence of cooling rate to reach the cryogenic temperature, the properties obtained. There are important works that probe the low cooling rates so that they obtain the best properties and others that simply drop it, dipping the part directly in the cryogenic temperature from room temperature.^[2] The results of hardness and toughness can also contrast because although some studies conclude a direct relationship between these parameters, there are results showing that there is not always such a relationship.^[12]

Given the lack of definitive results for various treatment parameters such as time and temperature display, and the results are not strong and contrasting for testing the hardness and impact, this work will bring an important contribution. The main controversial issues will be studied, which are: Water temperature and length of stay.

2 MATERIAL AND METHODS

2.1 Heat Treatments

The parameters of heat treatment used in the vacuum furnace were:

- Austenitizing: 1040 °C (30 minutes);
- Cooling: pressure of 5 bar N₂ nitrogen gas;
- Double tempering; •
- Tempering: 540 °C 2 h.

2.2 Criogenic Treatment

After quenching, as specified above, in some samples the cryogenic treatment (C-196th) is done before tempering, after other, with soaking time of 1, 24, and





48 hours. The condition C1 is the basis and follows the conventional heat treatment.

 Table 1. Cryogenic Heat Treatment Cycles AISI H13 (Cryo - Cryogenic bath, T – Tempering, C - Conditions)

Austenitizition 1040°C										
Hardening (vacuum - N ₂ gas)										
T ₁				T ₁	T ₁					
T ₂	Cryo 1h	Cryo 24h	Cryo 48h	Cryo 1h	Cryo 48h					
	T ₁	T ₁	T ₁							
C1*	C2	C3	C4	C5	C6					

2.3 Dimensions of the Samples for Charpy and Abrasion Tests

The Charpy specimens followed the ABNT and the specimens for testing abrasion resistance are obtained from the fractured Charpy specimens, as showed in Figure 3.



Figure 3. Dimensions of the Charpy specimen, according ABNT.

2.4 Description Tests

Altogether 18 samples were tested in relation to:

• Hardness / microhardness (150 kgf Vickers); made in durometer ECORT - 240;

• Charpy; in equipment Instron Wolpert PW30;

• 2-body abrasion (gravimetric method of assessment) in equipment pin on sandpaper.

3 RESULTS AND DISCUSION

The following graphs the results of tests of hardness, Charpy impact and wear.





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Figure 1. Average values of Charpy impact and hardness to the conditions C1-C6%.



Figure 2. Average values of wear for the conditions C1-C6%.

In this paper there was an increase in hardness in all conditions of stay in the cryogenic bath, which was observed by Molinari et al.^[13] and Yun et al.^[14] for the conditions under which comes after the tempering treatment cryogenic. The values of impact, C3 and C4 show a small difference compared with the base C1. However the best performance was obtained in condition C2. The conditions C5 and C6 showed a similar behavior.

The absorption of energy, Charpy impact, temper before or after the cryogenic bath influences the results. In condition C2, the bath was held before the cryogenic tempering and presented the best result. However, this work is consistent with Molinari et al.,^[13] Collins,^[11] Meng,^[15] and the cryogenic bath effective to increase the absorption of impact energy and therefore tenacity. It appears that all the conditions subjected to cryogenic treatment showed an increase in wear resistance, and the





condition C5 (Q + Rev + Cryo48h) was the best performance and is consistent with the work of Mohan Lal.^[16]

5 CONCLUSIONS

The cryogenic treatment done in conjunction with thermal treatments of quenching and tempering may increase about 20% of the impact energy (Figure 1). This increase is obtained when the cryogenic treatment was performed after quenching and before tempering.

A slight improvement was shown when the bath was held after the cryogenic temperature. The residence time of the cryogenic bath did not show a positive influence this process, as the best value of impact energy was staying with cryogenic bath an hour.

The cryogenic bath did not add improvements in relation to hardness values in C1, basis.

A significant increase in wear resistance in all conditions subject to the cryogenic bath regardless of length of stay.

The results are not exhaustive and TEM analysis is being undertaken to gain a better understanding of these processes.

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