CRYOGENIC TREATMENT AND IT'S EFFECT ON TOOL STEEL

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Abstract Tools for presswork, powder compaction, seamless tube pilgering, extrusion and metal cutting and metal sponge cutting (chisel) were subjected to cryogenic treatment. Each type of the above tool was studied in detail about its performance versus the hardened and tempered tools. Then wherever the function of the tools demanded high surface finish criterion, those tools were subjected to hardening and tempering followed by surface coating method, plasma ion deposition method, and nitriding by impregnation methods etc.

Keywords: Tools steel, nitriding, cryogenic treatment

PROCESS

The process typically involves slowly cooling a mass of parts to $-196\,^{\circ}C$, holding them at this temperature for 30 h or more, then slowly heating them back to ambient temperature. In case of steels, the benefits are usually

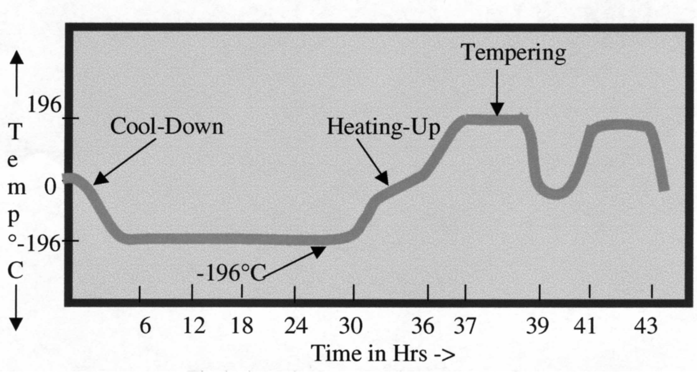

Figure 1. A typical cryogenic treatment for tool steels.

attributed to the reduction or elimination of retained austenite from hardened steel and accompanied by the precipitation of small finely dispersed carbides (η-carbides) in the martensite. Figure 1 shows a typical cryogenic treatment temperature sequence for tool steels.

PRESENCE OF RETAINED AUSTENITE IN TOOL STEELS

Through hardening of steel involves heating the steel to a temperature at which it becomes austenite and then cooling rapidly enough to produce martensite, a hard and strong, but brittle structure. Tempering at moderately elevated temperatures reduces this brittleness. Generally austenite phase may be retained in small amounts in low-alloy steels and in appreciable amounts in high-alloy steels, because of the austenite stabilizing effect of various alloying elements. The Fig. 2 shows the steel portion of the iron-carbon diagram which describes how the room temperature structure of the steel changes to austenite and the different critical temperature points where these structural changes takes place. The TTT curve, Fig. 3, describes how the austenite upon cooling is transformed into various phases like pearlite, bainite, martensite and retained austenite at different cooling rates. The first phase undergoes diffusion type transformation, the martensite phase undergoes diffusionless transformation, while the bainite phase undergoes both diffusion and diffusion less transformations. As shown in Fig. 3, the curve 1

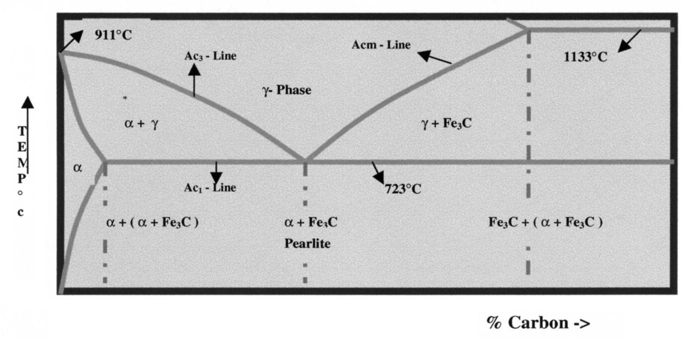

Figure 2. Steel Portion of Iron-Carbon Diagram.

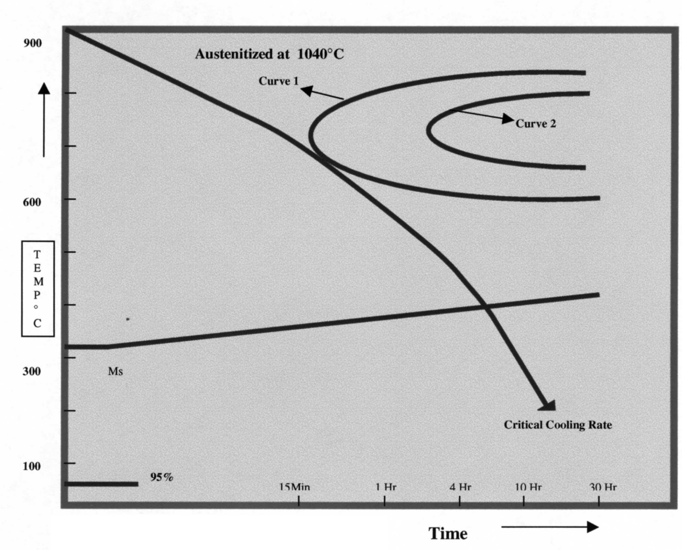

Figure 3. T.T.T. Diagram For AISI H-12.

gives the starting points of transformation while curve 2 gives the end points of transformations at different temperatures. In view of the cryogenic treatment it is more important to study the bottom portion of the TTT curve, to have an idea about where the martensite transformation starts and ends. This will in turn enable us to know how the austenite is retained. In general, the martensite starting point is slightly above room temperature in most of the tool steels. The transformation end point in some tool steels is well below the room temperature, which leads to retaining some amount of austenite, The retained austenite percentage depends on the chemical composition of the tool steel and its hardening and quenching procedure. To arrest the retained austenite transformation it is necessary to quench the tool steel not less than its critical cooling velocity, and allow the tool steel to cool down to temperature well below the martensite transformation end point. Here, practically all the austenite will be transformed into martensite. Some times this transformation is not complete, because the velocity of the tool steel quench is lower than the required, or the temperature to which the steel is cooled is well above the martensite transformation end point. treatment to optimize its service quality, it should be comparable with the tool in which the transformation of martensite is complete. The Ms and Mf temperatures for various steels can be found out by using empirical formulas. Some manufacturers prescribes the sub-zero treatment (not necessarily at cryogenic temperatures) between quenching from austenitizing temperatures and tempering. Though the properly treated tools do not require subsequent cryogenic treatment, many users attest to the superior performance of cryogenically treated tools.

TEMPERING AND ITS EFFECT ON CRYOGENIC TREATMENT

Tempering the steel after quenching from austenitizing temperature to the temperature lower than the A_{c_1} temperature and soaking, relieves the internal stress developed during quenching and improves the toughness by the precipitation of carbides uniformly throughout the structure. In turn, it reduces the carbon from both martensite and retained austenite. This process enables the steel to lower its retained austenite partially, but not completely even after the steel is subjected for suggested double tempering. The only way to reduce the retained austenite percentage is by subjecting the steel to cryogenic treatment (extended quench) immediately after quenching from austenitizing temperature. Also, it enhances the precipitation of η-carbides during subsequent tempering. The η-carbides that form is uniformly distributed throughout improved hardness, toughness, wear resistance and resistance to fatigue cracking.

New time-temperature "rules" must be applied to the post cryogenic treatment temper to obtain these benefits consistently. If traditional tempering practice is followed the potential advantage of deep cryogenic treatment may not be realizedcases, the net effect on properties could be negative.

ROLE OF CARBIDES

The present study is also important to the development of this technology, where the results of practice focused on deep cryogenic treatment at –196 ℃ of AISI O-1, D-2 and H-12 tool steel. The study identified martensite decomposition and precipitation of fine η-carbides as the main mechanisms responsible for the beneficial effects of deep cryogenics.

Mechanical properties of the alloy tool steels subjected to cryogenic treatment are optimized if -196 ℃ "extended quench" is followed with single conventional temper. The implication is that the multiple tempers commonly incorporated in conventional heat treatments can be eliminated. The precipitation of η-carbides in tool steel occurs only during the temper that follows deep cryogenic treatment, and lengthens the tool life as the amount of η-carbides increases. The amount of η-carbides that forms is directly proportional to the tempering time and temperature. Typical heat treatment cycle using cryogenic treatment can be seen in Fig. 4

CRYOGENIC TREATMENT AND ITS EFFECT ON MECHANICAL PROPERTIES

Cryogenic treatment improves the mechanical properties like hardness, wear resistance, toughness, and resistance to fatigue cracking. The possible reasons for this improvement are as follows.

- According to one theory of this treatment, transformation of retained austenite is complete – a conclusion verified by X-ray diffraction measurements.
- Another theory is based on the strengthening of steels by the precipitation of submicroscopic carbides. An added benefit is said to be a

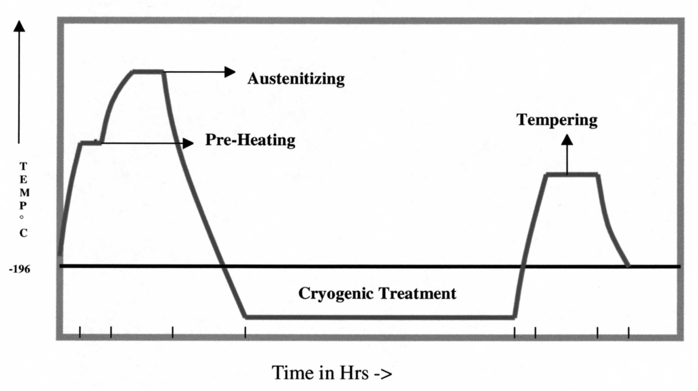

Figure 4. Typical heat treatment cycle using cryogenic treatment.

reduction in internal stresses in the martensite developed during carbide precipitation, which in turn reduces tendencies to micro-crack. Table 1 shows, the wear resistance of different tool steels with the cryogenic treatment.

Table 1. $R_w = FV/WH_v$, Where F is the normal force in Newtons for pressing the surfaces together, V is the sliding velocity in mm/s, W is the wear resistance in mm/s, and H_v is the Vickers hardness in MPa. R_w is a numeric value

	Wear Resistance, $R_w(n)$	
Alloy	Untreated	Soaked –196 ℃(–310 °F)
52100	25.2	115
D-2	224	878
A-2	85.6	565
M-2	1961	3993
O-1	237	996

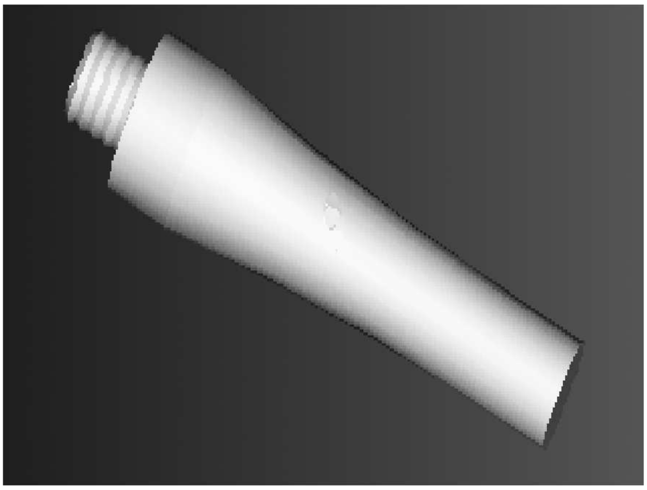

Figure 5. Pilger Mill Mandrel.

Figure 6. Ceramic Compaction Die Set.

CRYOGENIC TREATMENT PRACTICED BY TOOL ROOM, NFC

NFC Tool Room incorporated the cryogenic treatment along with heat treatment cycles of various tools used for the powder (UO_2) compaction Komage press dies, pilger mill tools, hot extrusion dies and chisels, see Fig. 5 and 6. Other application experiments are discussed as follows.

A Powder Compaction Die Sets (Press Tools) **-** Tools of this type demands high wear-resistance and strength during its service. The improvement observed in the performance of powder compaction dies was about three times to that of tools manufactured without cryogenic treatment. This improvement in life of the Komage press dies is corroborating the theoretical studies on wear resistance as explained above. The heat treatment cycle (with cryogenic treatment) used for powder compaction dies is shown in the following Fig. 7.

B Pilger Mill tools **-** These tools are made out of AISI H11, H12 and H13. 25VMR Dies, HPTR rollers, HPTR support plates and mandrels are the different pilger mill tools treated cryogenically. The increase in life of the pilger mill tools is double. The heat treatment cycle followed is shown in Table 2.

C Hot Extrusion Tools: **-** These tools are made out of AISI H12. Extrusion dies are treated cryogenically. It was observed that there was no increase or decrease in life of extrusion tools, after cryogenic treatment. It was also observed that, liquid nitriding as a finishing operation enhanced the life of extrusion tools to one and half time to that of hardened and tempered tools. The heat treatment cycle followed for these tools was as given in Table 3.

CRYOGENIC TEMPERATURES AND THE WAYS TO GENERATE SUCH TEMPERATURE

The temperatures well below room temperature, i.e. 0 to –269 $^\circ C$, are called cryogenic temperatures. Normally these temperatures can be generated using solid carbon dioxide or mechanical refrigeration or liquefied gas system. The solid carbon dioxide method is the oldest method and is capable of cooling components down to –80 $^\circ C$. The mechanical refrigeration

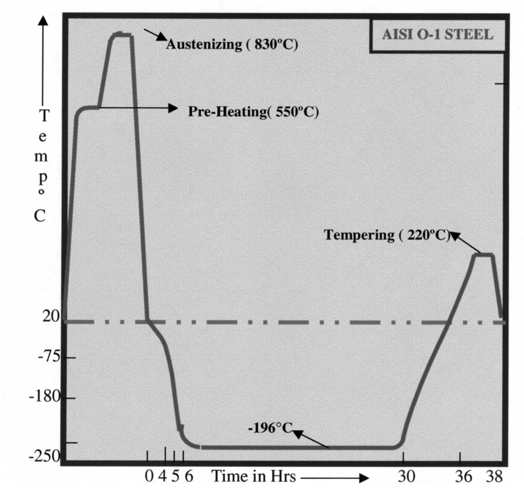

Figure 7. Cryogenic Treatment Cycle Practiced By NFC, Tool Room.

Table 2. Treatment for pilger mill tools

Step	Conventional	Cryogenically
1	Stress relieving at 560 ℃	Stress relieving at 560 ℃
2	Pre-heating at 560 ℃	Pre- heating at 560 ℃
3	IInd–heating at 830 ℃	IInd –heating at 830 ℃
4	Austenitizing at 1050 ℃	Austenitizingat 1050 ℃
5	Air Blast Cool to room temperature	Air Blast Cool to room temperature
6	Ist Tempering at 540 ℃	Cryogenic Treatment
7	IInd Tempering at 540 ℃	Tempering at 540 ℃
8	IIIrd Tempering at 540 ℃	

method may be capable of cooling to about –100 ℃using freon as a convection fluid. The last and very important method in cryogenic technology is the liquefied gas system which is capable of cooling to around –250 ℃. The gases that are used for generating the cryogenic temperatures are oxygen, nitrogen, neon, hydrogen and helium. Table 4 shows boiling temperatures of the different elements. Next, let us discuss about the liquefied gas system using liquid nitrogen as the cooling medium.

LIQUID NITROGEN SYSTEM

Components can be cooled to around –196 ℃. The liquid nitrogen system compared to other systems is more advantageous where it can be used for applications with a wider temperature range. The system is capable of cooling the components to desired temperatures at controlled rate. British Oxygen Company originally developed this method. There are two different techniques for utilizing liquid nitrogen in a controlled manner:

1 The Ellenite gas-cooled system, Fig. 8, which cools the components by forced convection of cold nitrogen gas through the work-piece (–196 ℃).

2 The Ellenite Liquid-cooled system, Fig. 9, which cools the components indirectly by immersion in a bath of alcohol or trichloroethylene, which is cooled by a submerged liquid nitrogen spray (–150 ℃), temperature and cooling rate controls are possible. The equipment is relatively inexpensive compared to other systems.

CONCLUSIONS

Following inferences were recorded from our experiments in NFC tool room:

- AISI O1 & O2 (OHNS) press tools and powder compacting tools (even with minute cross sectional areas) worked very well. The life of tools increased.

- Performance of D2 & D3 and M2 & M6 grades cutting tools and metal forming tools were improved to 3 times to that of hardened and tempered.

Table 3. Treatment for extrusion tools

Step	Conventional	Cryogenically
1	Stress relieving at 560 ℃	Stress relieving at 560 ℃
2	Pre-heating at 560 ℃	Pre-heating at 560 ℃
3	IInd–heating at 830 ℃	IInd–heating at 830 ℃
4	Austenitizing at 1050 ℃	Austenitizing at 1050 ℃
5	Air Blast Cool to room temperature	Air Blast Cool to room temperature
6	Ist Tempering at 610 ℃	Cryogenic Treatment
7	IInd Tempering at 610 ℃	Tempering at 610 ℃
8	IIIrd Tempering at 610 ℃	

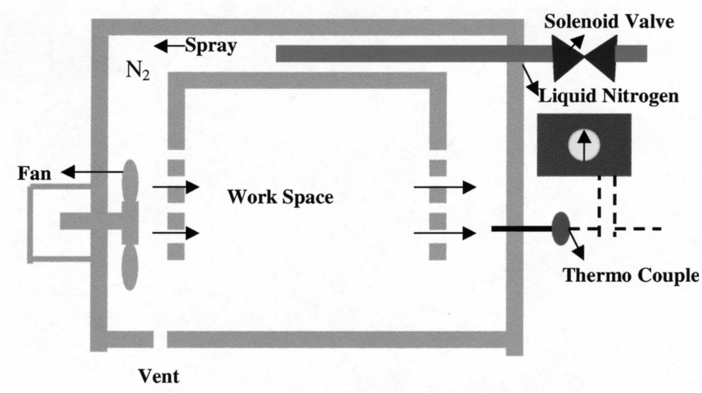

Figure 8. Liquid Nitrogen System (Gas Cooled).

Table 4. Boiling temperature of cooling media

S No	Element	Boiling Temperature
1	Oxygen	–183 ℃
2	Nitrogen	–196 ℃
3	Neon	–247 ℃
4	Hydrogen	–253 ℃
5	Helium	–269 ℃
6	Carbon dioxide	–80 ℃

- Performance of AISI H11, H12 and H13 grades metal forming and pilgering tools were found to the tune of 200% to that of hardened and tempered tools.

- Chisels made out of O1 and S1 grade steel improved their performance to 150 to 200%

- Hot extrusion tooling made out of AISI H12 steel did not show any improvement by cryogenic treatment. The reason attributed for this phenomenon is that the working temperature of these tooling are to the tune of 600 to 800 ℃where the microstructure transformation takes place and hence it behaves as hardened and tempered tools. Experiments conducted on hot extrusion tooling were

  1 Plasma ion nitriding

  2 Surface coating by detonation coating

  3 Surface coating by electro spark deposition and

  4 Surface hardening by liquid nitriding techniques and mirror polish.

Out of the above we found that hardening the layer by liquid nitriding followed by mirror polishing improved the life to 150%.

It was also observed that post-cryogenic treatment fine machining (grinding) is easier. Hence the cost of finishing operation comes down due to cryogenic treatment.

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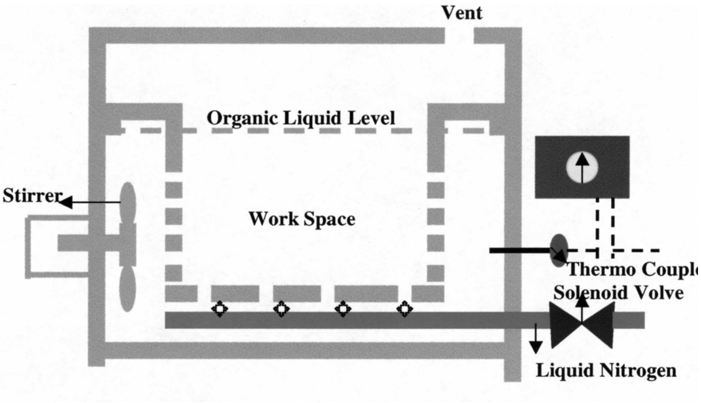

Figure 9. Liquid Nitrogen System (Liquid Cooled).