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### RESEARCH ARTICLE

#### EFFECT OF CRYOGENIC TREATMENT ON TOOL STEELS.

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#### Abstract

The objective of this review is to understand the effect of cryogenic treatment on tool life and its performance. As cutting tool life is an important factor in reducing the cost of production. In this process of cryogenic treatment, tools are generally subjected to the process such as heat treatment for achieving the hardness which will improve the performance of the tool to some extent. Cryogenic treatment also known as cold treatment, in which the phase transformation of the material takes place, is mainly responsible for changes in the wear resistance, tensile strength, toughness etc. which increases the tool life. From the review it was understood that has great effect on cutting tool properties. Especially soaking period varied from 6 hours to 36 hours are giving different effect on the phase transformation of the material, consider as one of the important aspect for tool life improvement. Various soaking range namely shallow cryogenic treatment (SCT), Cryogenic treatment (CT) and deep cryogenic treatment (DCT) produce different properties in tool material. This treatment or process can be very useful in case of machinability of work-piece, quality of machining in terms of surface finish and economics of the tooling.

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#### Introduction:-

In metal working industries, the tool materials used for cutting tools are all belong to a separate class of steels called as Tool Steels. Tool steels are called so because of a few very important machinability properties they possess such as 'abrasion resistance' 'resistance to deformation' 'high hardness' 'hot hardness' and so on. Because of these, tool steels are used for applications involving shaping other materials. High quality of the tool steels are achieved through controlled chemical compositions which give them the desired properties for shaping materials. The type of tool steel depends on the percentage of carbon (usually 0.1% to 1.6%) and the alloying elements used. Molybdenum, vanadium, chromium are very commonly used and their benefits are very well documented and widely used in the metal industry. Tool steels are widely used for applications such as die casting, plastic molding, making cutting tools, various sheet metal tooling's such as blanking, shearing, forming and so on. Tool steels offer better choice than common construction and engineering steel materials because of their corrosion resistance, durability, stability at high temperatures.

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In steel making industries there has been special attention set on producing tool steels particularly the high alloyed steels intended for the purpose of plastic molding, forming, blanking, die casting, forging extrusion, recycling and wood-working. Steels are also produced by following the Powder Metallurgy. Tool steels can be procured in soft annealed condition because of which they are easily made into cutting tools. The soft annealed condition tool steel are also well suited for hardening. The microstructure provides pockets to embed the carbide particles.

Generally Iron Carbides  $Fe_3C$  are the particles which give high strength to carbon steels. Whereas in alloy steels Tungsten (W), Vanadium (V), Molybdenum (Mo), Chromium (Cr) carbides depending on the composition of the steel. The carbides are characterized by high hardness. The wear resistance improves with the increase in percentage of various carbides and their distribution in the steel crystal structure. Non carbide forming elements when alloyed, get absorbed into the metal matrix. Cobalt and Nickel are one such alloying elements. Cobalt improves the red hardness in high speed steels, nickel improves the hardening properties as well as the toughness in hardened condition. Other inclusions in steel like manganese are kept low to avoid cracking due to thermal load during water quenching

Metal cutting is the backbone of manufacturing industry. Each and every product ever made in history can trace its origin to the basic metal cutting process. The cutting tool therefore is the important element for any metal cutting operation to realize its true potential. Since the last few decades, in a bid to make the processes economic, a lot of effort has been put into the research of metal cutting. The results have thus given birth to newer tool materials with marked improvement in performance thereby improving productivity. Lighter yet stronger tool materials have been the catch words among the cutting tool manufacturers. Some of the other properties which are highly desired are

- Resistance to diffusion.
- Resistance to brittle fracture.
- Ease and economy of fabrication of the tool.
- High resistance to thermal and mechanical shocks.
- Ability to retain strength at high speeds and withstand the heat generated during cutting operation.

The enormous developments taking place in the cutting tool technology is driven by the knowledge about the capabilities of the tool materials to work under different conditions of stress, high temperatures at the interface between tool and work piece. The tool wear occurs by different mechanisms which include thermal cracking, chipping at the cutting edges of the tool and abrasive wear. The tool materials should therefore have good mechanical, physical, chemical properties at such elevated temperatures<sup>1-4</sup>

#### **Tool steel grades:-**

Various tool steel grades have been developed over the years and are well established in AISI and SAE tool steel grades. The different alloy tool steels in each of the grades are given a number to be identified with. In total, there are 6 different grades of tool steel material. The choice of the tool steel grade is influenced by the cost, surface hardness required, working temperature, toughness, shock resistance, durability, and strength. If the working conditions are expected to be severe, the respective alloying element is chosen depending on the properties they enhance on the steels.

Tool steels prepared by water quenching are known as water hardening group, these steels can have a maximum hardness of 66HRC. O - series and A - Series are also based on quenching medium used (Oil for O - series and Air for A - series). The Oil hardening series have up to 7 types numbered from O1 to O7 and D - series (High Carbon High Chromium steels) in which the chromium content is up to 13%. Next is the shock resistance alloyed tool steels which are prepared by alloying elements such as chromium, silicon, tungsten, molybdenum, manganese and approximately 0.5 % C is maintained where higher toughness is expected. Most widely used is High speed steels which are used in cutting tools such as turning tool, drill bits, saw blades. These are used predominantly in cutting operations of mild steels and other softer materials. When the materials are to be cut, formed at high temperatures, a special type of tool steels called Hot Working tool steels is used. The hot hardness and wear resistance are provided because of the presence of carbide content of the alloying element. This is the most populated group of tool steels with close to 60 types. The types numbered from H1 to H19 are high chromium content (up to 5%), H20 to H39 are tungsten alloy (9% to 18%), H40 to H59 being molybdenum based. These have low carbon content.

### Introduction to Cryogenic Process:-

Cryogenic is a term used to refer to things at very low temperatures. The idea of very low temperatures is not a modern one. It has been used as back in the time as the 16<sup>th</sup> century

when it was used to enhance some mechanical properties. Cryogenic treatment induced positive effects on the key parameters such as tool performance, tool wear. Mohan et al have achieved a 110% gain in tool life after studying the effects of cryogenic treatments. Its applications are very wide in range as it has been used for ferrous, non - ferrous metal alloys, ceramics, plastics, carbides. In conventional heat treatment methods, the changes take place on the surface or only up to certain depth. Whereas in Cryogenic treatments the changes take place at the basic crystal structure of the material,<sup>1</sup> As a bonus there is no negative influence on the corrosion resistance of the material. The effect of DCT on various other grades of steel like the stainless steel type by conducting Qualitative salt spray corrosion test confirming the same.<sup>2</sup> Thus Cryogenic treatment has been a very effective process to obtain many desirable properties in metals.

The actual process of Cryogenic treatment is quite simple. The material samples will be cooled to very low temperatures by means of a cooling system regulated by a control unit. Gases like nitrogen, helium are used to cool to extremely low temperatures like  $-80^{\circ}\text{C}$  to  $-196^{\circ}\text{C}$ . The abundant availability of nitrogen in the atmosphere and its suitability for carrying out cryogenic treatment has made it the quintessential component of Cryogenic Treatment. It has a melting point of  $-210^{\circ}\text{C}$  and boiling point of  $-196^{\circ}\text{C}$ . The samples will be kept at cryogenic temperatures for some time. This period is called 'holding time'. It is during this holding time that the change to the microstructure takes place. After this the material samples are brought gradually to room temperatures. Figure 1 shows the Schematic presentation of cryogenic treatment applied in a controlled manner<sup>3</sup>.

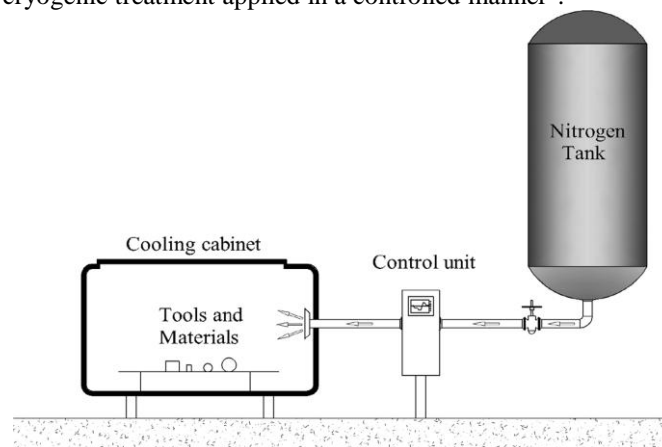


Figure 1. Schematic presentation of cryogenic treatment applied in a controlled manner<sup>4</sup>

The results of cryogenic treatment are obvious and their improvements over untreated samples are astounding. Marked improvements in abrasion resistance, toughness, hardness, electrical conductivity can be seen in cryogenic treated samples. These can be attributed to generally the two underlying mechanism (i) conversion of austenitic structure into martensitic. (ii) Finer distribution of the carbides in the metal matrix. The presence of nanosized carbide particles through the study of microstructures. As a result Cryogenic treatment is widely used to increase the performance of chip breaker tools. When undertaken on bigger scale these methods also offer economy over other methods such as coating. Cryogenic treatments are also applied on cutting tools, race car engines, tool steels, brake discs, composites and on some plastics.<sup>4</sup> Figure 1 shows the Schematic presentation of cryogenic treatment applied in a controlled manner<sup>5</sup>

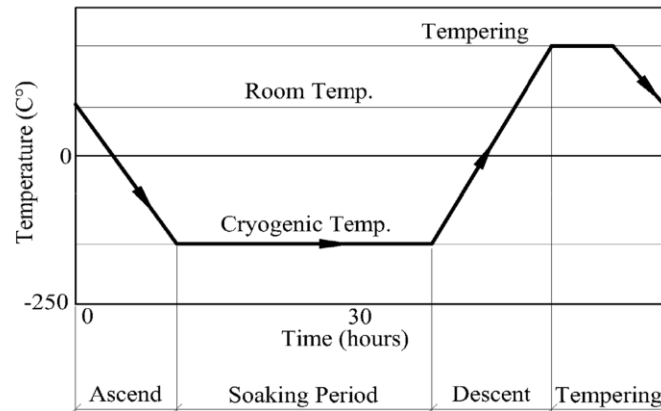
Basically the Cryogenic treatments can be broadly classified into two types. (i) Shallow Cryogenic Treatment – SCT & (ii) Deep Cryogenic Treatment – DCT. The treatment in which the cryogenic treatments are kept at  $-80$  to  $-149^{\circ}\text{C}$  is called SCT and DCT involves even lower temperatures at  $-140^{\circ}\text{C}$  to  $-196^{\circ}\text{C}$ . Figure 2 shows the Variations of temperatures during Cryogenic Treatment<sup>5</sup>

### Rate of cooling:-

The material should be brought to the cryogenic temperature and then it undergoes soaking at same temperature for period of 2 hrs to 48 hrs depending on requirement, figure 2 shows the variations of temperatures during cryogenic

treatment. In rate of cooling there are two main approaches, in the first approach the samples slowly brought to cryogenic temperature in between (-80°C to -196°C). in the second approach, the samples are slowly brought to cryogenic temperature. According to the study the second approach is recommended as some damages like micro cracks are developed on the sample due to thermal shock.

Cryogenic Treatment Schedule

Figure 2. Variations of temperatures during Cryogenic Treatment <sup>4</sup>

The used carbide cutting tools at two different rates of cooling/heating (0.5 °C/min and 1 °C/min ) and completed the cryotreatment in 8h and 4h and reported that the wear resistance was better in the sample (0.5 °C/min,8h) compared to (1 °C/min,4h). sudden decrease in temperature (thermal shock) reduces the cost of cryotreatment but increases the risk of occurrence of micro cracks in the microstructure of the specimen due to which the specimen may not give better performance. So in cryotreatment the slow cooling/heating process is recommended. <sup>6</sup>

#### Soaking Period:-

After the literature review of cryotreatment (DCT) on the tool steels the samples were subjected to soaking period in between 1 and 40 h were the conversion of material from austenite to martensite takes place and the formation of new carbides takes place for carbide distribution. The soaking of Ti-6Al-4V alloy up to 72h (DCT) at -196°C. Therefore the soaking period should be optimized to increase the wear resistance efficiency and reduce the cost of cryogenic treatment. <sup>7</sup>

#### Soaking Temperature:-

Cryogenic treatment also known as sub-zero heat treatment shows the variation according the type of tool steel material. Cryogenic treatment temperatures applied to cutting tools range between -80°C and -196 °C. Taking as -80°C- Cryogenic treatment, -120°C- Shallow Cryogenic treatment and -196 °C- Deep Cryogenic treatment. The study shows that to increase the wear resistance to the samples the soaking temperature plays an important factor. From the literature studies it is found that -196 °C (DCT) gives the best result in the improvement of mechanical properties of the specimen.

#### Tempering Process:-

Tempering is usually done after the cryogenic treatment. The process is mainly performed to remove the internal stresses of the specimen that occur due to excessive cooling in cryogenic treatment. Generally, the process is applied to cutting tools by holding the specimen at 150–200 °C for 1.5–2 h. In some previous studies has under gone double tempering after cryogenic treatment and a fine distribution of Carbide particles have been obtained by double Tempering after CT <sup>8</sup> as represented in Figure 3. In some studied the results show that Tempering indeed maximizes the gain in tool life after DCT (86% without Tempering, 126% with Tempering). <sup>9</sup> through investigations have found that tempering before actually decreases wear resistance as against increasing it. On a hind note it has been observed that multiple cycles of Tempering after DCT hasn't improved on the wear resistance, but has a negative impact. Also there has been decrease in wear resistance after multiple cycles of Tempering, compared to CT and HCT samples. <sup>10</sup>

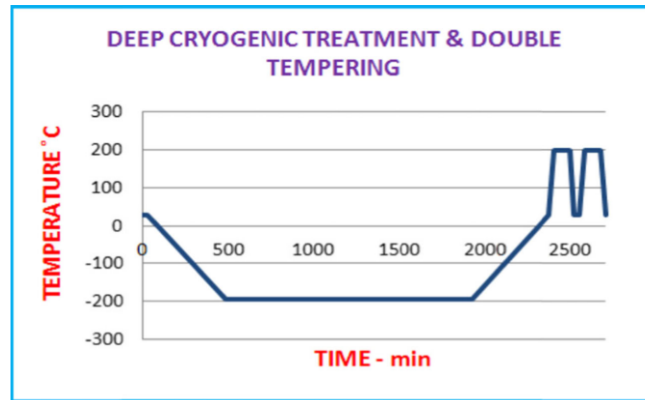


Figure 3. Variations of temperatures during the Tempering process, after Cryogenic Treatment <sup>6</sup>

## Results and Discussion:-

### Tool life:-

The actual length of the cutting time that the tool can be used is known as tool life. Generally the tool failure happens due to sudden breakage of tool tip or due to the gradual wear of the tool is known as flank wear or crater wear, the tool wear is mainly caused due to increasing cutting forces and cutting temperature, which leads to the poor surface finish and accuracy of the components. The tool life was found increasing generally by the research conducted there was the improvement of about 86% for M2 tool steel and 48% for D3 tool steel compared to heat treated tool steel.<sup>12</sup>

There are mainly three stages of flank wear rapid growth region, steady state region (Temperature insensitive region) and Catastrophe failure (Temperature sensitive region). Flank wear formation mainly depends on cutting condition i.e. Parameters like speed, feed and depth of cut as well as the properties of the work and tool material.

### Microstructure:-

A detailed study of the mechanism which improves the mechanical properties in Deep cryotreated materials has been provided were presence of different alloyed carbide has been absorbed in the sample even before, have also reported about various carbides being present.<sup>13,14,15</sup> The carbides have been classified into primary carbides and secondary carbides. Primary carbides are large, spherical in shape and their sizes are in excess of 5 $\mu$ m whereas secondary carbides are in irregular shapes. Secondary carbides are further classified into small (i) Secondary carbides (SSC) of size from 0.1  $\mu$ m to 1  $\mu$ m and (ii) Large secondary carbides (LSC) of size from 1  $\mu$ m to 5  $\mu$ m. All studies point to benefits of DCT in multiple ways. A great reduction in the amount of retained austenite is seen – in most cases retained austenite content is reduced to as low as 4%. The microstructural changes increase the wear resistance of the material.

The martensitic phases in the steels are further conditioned during DCT leading to precipitations of high amount of ultrafine secondary carbides. Secondary carbides are generally hard and thus contribute to improved wear resistance. The micro structural changes that occurred during DCT as shown in figure 4, 5 and can be compared to the figure 6 which is of cryoheat treated (CHT) for D2 tool steel. From these illustrations it can be seen that DCT enables precipitation of fine and ultrafine carbides in the material. The reduction in the amount of Retained Austenite is confirmed from XRD profile obtained for such DCT material specimen. A very scant amount of retained austenite has been detected by TEM studies from the DCT specimen. The secondary Carbides are made up of M<sub>23</sub>C<sub>6</sub> where as eutectic M<sub>7</sub>O constitutes primary carbides along with small amount of C<sub>7</sub>Cr<sub>3</sub> carbides.<sup>16</sup>

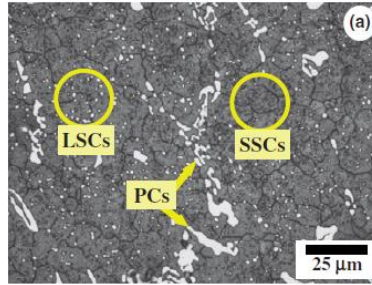


Figure 4. Optical Micrograph showing various carbides <sup>13</sup>

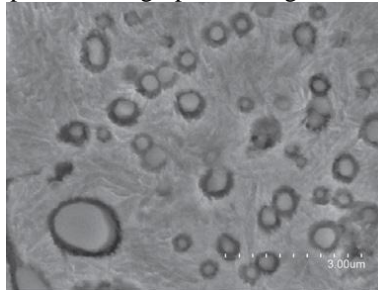


Figure 5. SEM Micrograph of DCT on AISI D2 Steel <sup>13</sup>

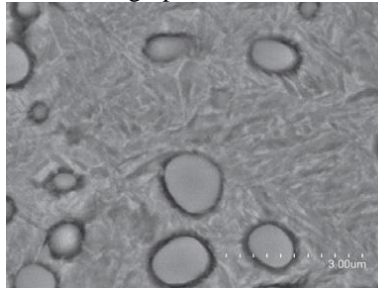


Figure 6. SEM Micrograph of CHT on AISI D2 Steel <sup>13</sup>

The reciprocator friction and wear monitor test on SCT and DCT samples of En 52 and 21-4N valve steels. The results have shown that the wear resistance has increased in DCT sample compared to SCT samples. The microstructure of the En-52 and 21-4N (CHT, SCT and DCT) variants is shown in figure 7 and 8. A very large white patches can be seen in CHT sample in which martensite and retained austenite both exist together whereas in cryotreated specimens (DCT) the austenite is fully transformed into martensite form, in SCT the austenite is retained in small fraction. Since the martensitic phase is the last phase that enhances the hardness uniformly throughout the material which increases the wear resistance of the material. The clusters of primary and secondary carbides are also seen in SCT and DCT samples. <sup>17</sup>

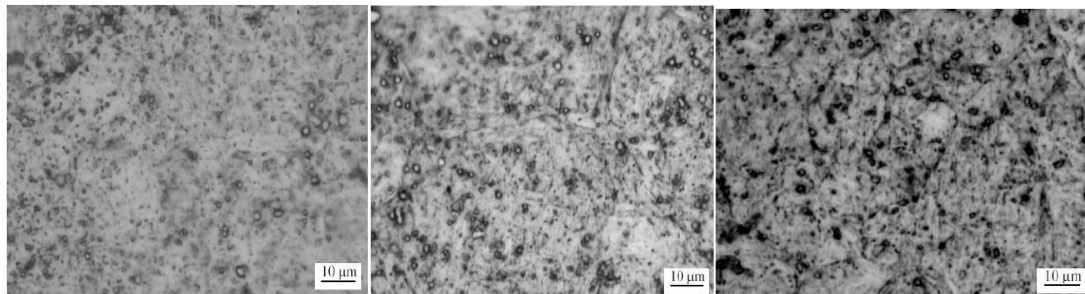


Figure 7. The microstructure of En 52 CHT, SCT and DCT <sup>14</sup>



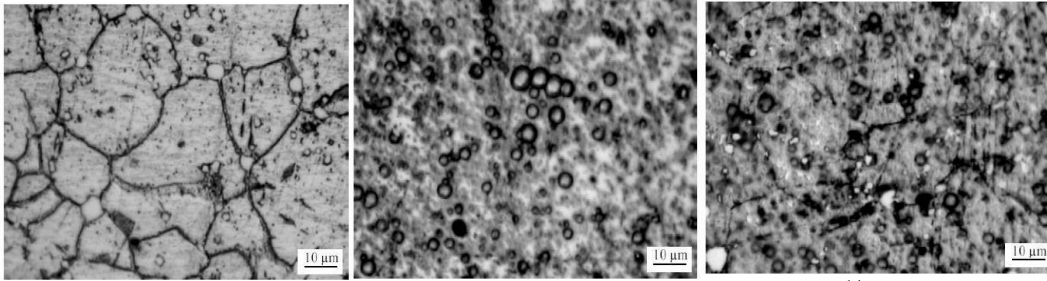


Figure 8. The microstructure of 21-4N CHT, SCT and DCT<sup>14</sup>

The optical microstructure studies<sup>18</sup> on CHT and DCT samples for D2 tool steel also confirmed the same as in other studies. The DCT sample again exhibit higher carbide precipitation and a finer distribution of secondary carbide than none at all in CHT samples which shows irregular shaped primary carbides. Comparative microstructural images are given figure 9.

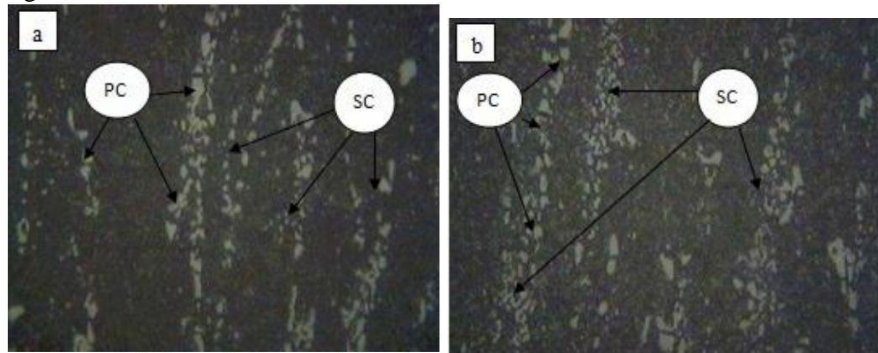


Figure 9. The microstructure of D2 CHT, DCT<sup>15</sup>

Various sequences of hardening and tempering process along with DCT were carried out on D3 tool steel by SimranPreet<sup>19</sup> the results of experimentation were studied through microstructural studies. The various sequences of heat treatment process are tabulated below. The figure 10(a) shows the microstructure of the as the received state of D3 large accumulation of carbides of all irregular shapes can be seen carbides get dissolved in the medium after hardening process and the materials takes different changes based on the subsequent process carried. It can be seen through comparison between the figure 10(b) and 10(c) for B-I and B-II sequence that the size of carbides have been reduced. When cryotreatment was followed by tempering and double tempering the carbides can be seen to getting more cores through a phenomenon called “Ostwald ripening” (smaller particles getting deposited onto larger carbides).

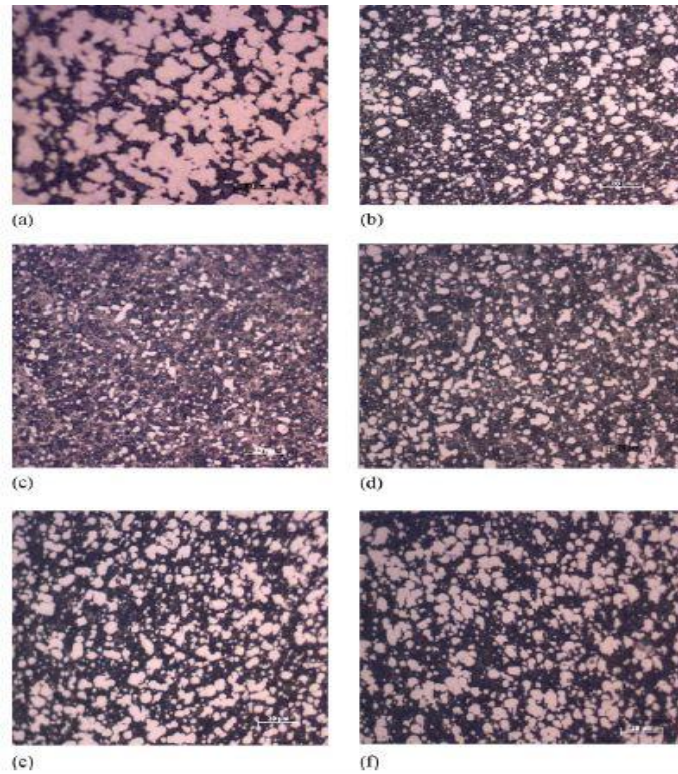


Figure 10. Microstructure of D-3 steel for treatments B-I, B-II, B-III, B-IV and B-V: (a) raw (500×); (b) HT (500×); (c) HC (500×); (d) HCT (500×); (e) HCTT (500×); (f) HCTTT (500×).<sup>16</sup>

The changes in D2 and D3 tool steel after cryogenic treatment, Globular shaped carbides of 9 micron and nodular shaped carbides of 11X4 micron can be seen in the microstructure of CHT of D2 in figure 11(a) and untempered martensite and retained austenite was seen of about 10% and 50% respectively there has been an decrease in carbides size both in globular and nodular shape as compared to just austenizing, quenching and tempering process. The distribution of carbides is more uniform figure 11(b). the carbide sizes in both forms was found to increase in AQCT process after tempering however multiple tempering process after cryogenic treatment increases the retained austenite there by neglecting the overall benefits of cryogenic treatment therefore multiple tempering treatment is not advisable after DCT.<sup>20</sup>

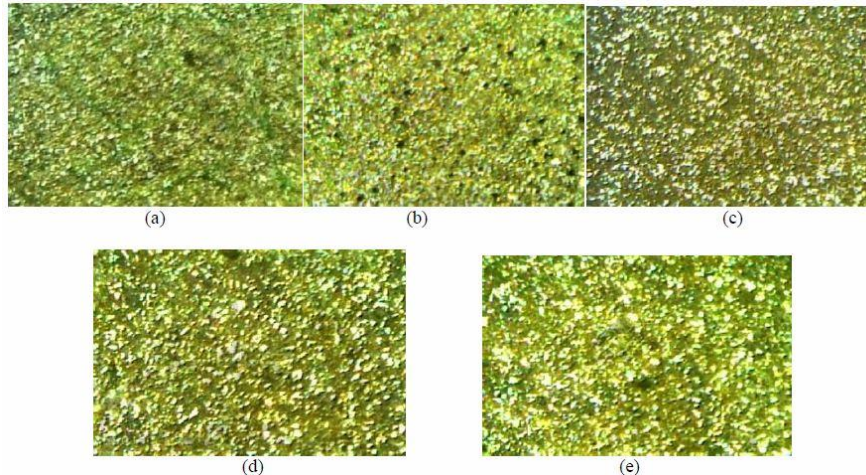


Figure 11. Microstructure of various combinations of treatments for the specimen of D2 steel a) CHT b) AQC c) AQCT d) AQCTT e) AQTCT<sup>17</sup>

Figure 12(a) which shows the microstructure of D3 tool steel of CHT, there has been up to 20% of the globular shaped carbide content of size 3x6 microns. The well distributed structure was observed with around 7% of untempered martensite the carbide content is up to 4% in AQC sequence figure 12(b) which shows the positive effect



of tempering after cryogenic temperature. The medium contains up to 95% of unstable austenite figure 12(c) the carbide distribution is more uniform. Nodular carbides of 2X4 microns are also seen amounting to 15%, on further tempering multiple times the retained austenite content goes on reducing and the untempered martensite is increasing in content from 5% to 7% in further sequences in AQCTT and AQCTCT sequences.<sup>20</sup>

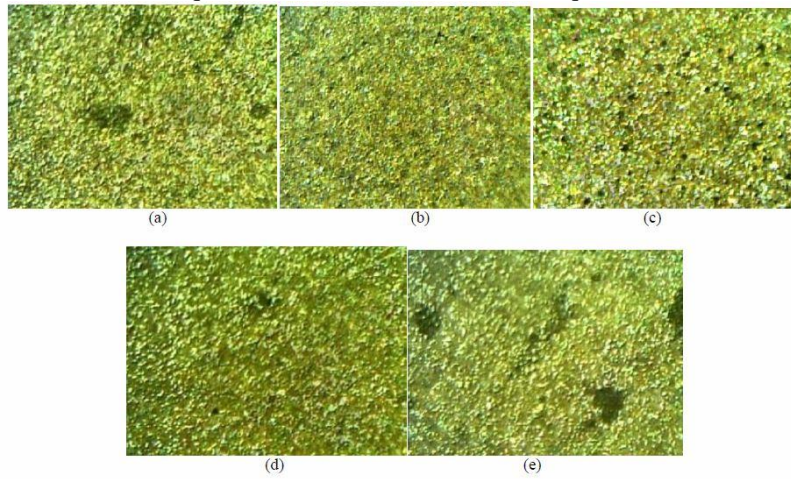


Figure 12. Microstructure of various combinations of treatments for the specimen of D3 steel a) CHT b) AQCT c) AQCTT d) AQCTT e) AQCTCT<sup>17</sup>

Huang<sup>21</sup> studied the effects of cryogenic treatment and subsequent tempering had on the precipitation of carbide in M2 tool steels using the TEM micrographs of untreated and cryotreated M-2 tool steel as shown in figure 13 and has concluded that the distribution of carbide as well as their sizes remained same in treated and untreated cryogenic samples, but the density of the carbide content was different during the treatment as the retained austenite is converted into martensite, internal stresses are developed in the samples this is caused due to the chain reaction the stress are mainly caused due to the changes in the composition and microstructure which causes dislocation and twinning in the crystal structure. High internal stresses are developed when the samples are cooled below certain temperatures, when the holding time is long enough the carbon and other alloying elements diffuses to the defected sites thus creating a cluster of carbon and alloying elements. As the martensite becomes supersaturated due to decreasing temperatures during cryogenic treatment the resulting thermal instability and lattice distortion drive the alloying atoms and carbon atoms clustering near defects. It is around these clusters where carbides are formed when the tempering process is followed after cryogenic treatments.

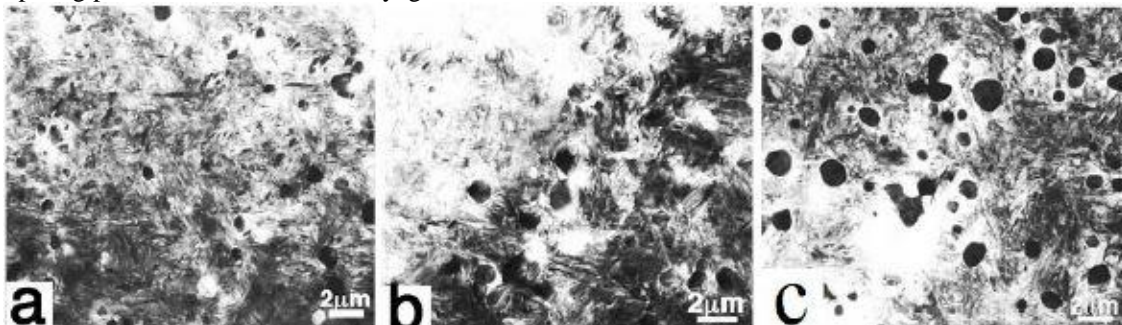


Figure 13. TEM micrographs of M-2 steel (a) and (b) Representative regions from the same untreated sample (c) Cryotreated sample<sup>18</sup>

In the series of experiment<sup>22,23,24</sup> significant precipitation of secondary carbides was observed. As in most studies of cryogenic treatments on tool steels mechanical properties have improved figure 14 shows the SEM micrographs of various cryotreatment, In cryogenic treatment holding time has a significant effect on wear resistance properties. It is found that the wear resistance increases as the holding time increases up to 36 hours and decreases beyond that. Thus a function between the density, distribution of carbides and holding time can be established which agrees in the case of M2 tool steel strongly.

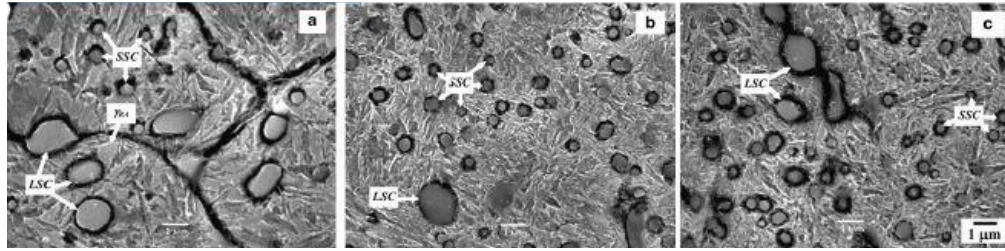


Figure 14. High-magnification SEM micrographs: (a) conventionally quenched and tempered; (b) cryogenically treated for 36 h; (c) Cryogenically treated for 84-h<sup>21</sup>

The authors also concludes that the wear resistance on the material will have a positive effect only till the holding time is 36 hours only, and beyond that the material will show the reverse effect i.e. decrease in the properties.

#### Wear behavior:-

The wear of tool during a wide range of industrial applications is a serious issue in the production prospective. Cryogenic treatment is a best solution to reduce the tool wear which is also effectively proved by the authors. There was an increase in wear resistance by 18.54% and 14.04% for a load of 50N and 25N respectively.<sup>25</sup> In some cases an improvement of wear resistance in D3 tool steel by 80%. The co-efficient of friction was also found to decrease which point to the quality surface finish was obtained in cryotreatment.<sup>26</sup>

#### Conclusion:-

- The cryogenic treatment has been used effectively to enhance the mechanical properties like wear resistance, hardness of various grades of steel.
- The core benefits of cryogenic treatment are increase in wear resistance and hardness.
- Soaking period and temperature also plays an important role in varying the properties of the tool.
- These benefits are effectively applied to various tool steels to maximize the tool life.
- The underlying mechanism had been conversion of austenite to martensite structure. The tool life also gets enhanced through increased wear resistance caused by the deeper distribution of carbide particles in the material.
- Maximum benefits from can be achieved in cryogenic treatment by controlling the parameters such as (holding temperature, holding time, identification of heat treatment to be applied before or after, etc.) which should be applied under optimum conditions.

#### Declaration of conflicting interests:-

- There are no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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