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# EXPERIMENTAL INVESTIGATIONS ON HEAT TREATMENT OF COLD WORK TOOL STEELS: PART 1, AIR-HARDENING GRADE (D2)

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## Abstract

The present experimental investigations deal with the improvement of mechanical properties of cold work tool steels through different heat treatment processes. An attempt was made to get optimal combination of hardness and toughness through changes of microstructure by heat treatment. The toughness of D2 tool steel increases with tempering temperature. The hardness of D2 tool steel decreases with increase of tempering temperature. At lower tempering temperature range (160$^{o}$C-200$^{o}$C), the effect of tempering temperature on hardness is very small but as the tempering temperature increases hardness becomes prominent. The hardness of D2 tool steel increases with austenitizing temperature and the lowest and highest hardness were obtained at 970$^{o}$C and 1000$^{o}$C respectively. Results were compared with standard conventional test results. It is observed that different heat treatment processes significantly improves the mechanical properties like hardness, toughness and microstructure of cold work tool steel.

**Key words:** hardness, toughness, heat treatment, hardening grade.

## 1. Introduction

Most of the die materials are subjected to extremely high loads that are applied rapidly. Dies must be able to withstand these loads a number of times without breaking and without undergoing excessive wear or deformation. During hot forging or die-casting, various damage mechanisms may act simultaneously to produce cumulative damage to the die and cause deviations from the original die geometry due to wear, micro chipping, heat cracking or failure of the die. In case of stamping dies, which are made of cold work tool steels, the stamping force suddenly increases to a maximum value with sudden decrease to a minimum value. This causes a tremendous varying load on the die and punch member. High wear resistance, sharp cutting edge, high hardness and measurable toughness are the desirable qualities of a stamping die. The process parameters and the die material determine the dominant damage mechanism. Prevention of instantaneous die failure is often connected with a critical hardness level that must not be exceeded for a specific application. For many dies, ductility has greater effect on die life than toughness. Hardness is closely related to ductility and toughness.

The die manufacturers are commonly facing the problem of selecting proper system parameters while doing heat treatment operations of different die components. They are selecting the parameters either based on their experiences or using trial and error method. Thus the mechanical properties of the dies are not optimized and die failure occurs within a short period of time.

## Nomenclature

| | |
|---|---|
| EDM | Electric Discharge Machine |
| AJM | Abrasive Jet Machine |
| D2 | High carbon high chromium tool steel |
| EN31 | Emergency Number,31 grade (British specification of steels). |
| EN42 | Emergency Number,42 grade (British specification of steels). |
| H-series | Hot die series of tool steels |
| P-series | Plastic mold series of tool steels |
| O-series | Oil hardening tool steels |
| Ni | Nickel |
| Cr | Chromium |
| Mo | Molybdenum |
| V | Vanadium |
| FCC | Face Centered Cubic |
| BCC | Body Centered Cubic |
| BCT | Body Centered Tetragonal |
| LCS | Low Carbon Steel |
| $Fe_3 C$ | Iron carbide |
| MPa | Mega paskel |
| DNC | Direct Numerical Control |
| Si | Silicon |
| Mn | Manganese |
| HP | Horse Power |
| ASTM | American Society of Testing of Materials |
| NDT | Non Destructive Testing |
| A | Ferrite |
| Rc | Rock well C-scale |
| $K_{ic}$ | Fracture toughness |

For avoiding such failure of the dies, the selection of proper die material and its proper heat treatment is very important. Qualities of a die material directly depend on percentage of carbon, percentage of alloying elements present, microstructure of the die steel, grain size after heat treatment, and heat treatment operation carried out on it. By varying the process parameters, an attempt has been made in this experimental investigation to heat-treat the cold work tool steels to improve its mechanical properties, to increase the die life, and get optimum service out of it so that the die manufacturers are able to optimize mechanical properties of the dies. Many relevant research works has been carried out by different research scholars on improvement of mechanical properties of cold work die steels and other tool steels through different heat treatment and allied techniques. A brief review of the following research papers was given so that it helps to create a better understanding of previous researches supporting the present experimental investigation. The possibility of improving properties of D2 cold work tool steel by an unconventional vacuum furnace heat treatment. A double quenching, the first quenching from a temperature higher than that for the second quenching, followed by normal tempering enables an excellent combination of toughness and hardness as a consequence of improved micro-structural homogeneity (first quenching) and re-precipitation (second quenching) of carbides, due to a large amount of fine dispersed carbides in the matrix and due to the preset of a controlled amount of retained austenite[1]. A martensitic hot work tool steel die block for use in the manufacture of die casting die components and other hot work tooling components and developed a method for manufacturing the same. The die block has hardness within the range of 35 to 50 HRC and a minimum transverse Charpy V-notch impact toughness of 5 foot pounds when heat treated to a hardness of 44 to 46 HRC and when tested at both $72^0$C and $600^0$C[2]. Experiment taken martensitic hot work tool steel die block for use in the manufacture of molds for plastic injection molding. The die block has hardness within the range of 35 to 50 HRC, a minimum Charpy V-notch impact toughness of 3 foot pounds when heat-treated to a hardness of 44 to 46 HRC. The die block contains sulfur within the range of 0.05 to 0.30 weight percent. The hot work tool steel includes mar-aging and precipitation-hardening steels of this type [3]. The effect of austempering technology on microstructure and properties of H13 steel and compared with formal treatment, the duplex structure is obtained in H13 steel after $250^o$C austempering and tempering; the high temperature impact ductility increases 33.4% without reducing high temperature strength and toughness [4]. The experimental investigation used electron beam for surface hardening of D3 tool steel. The results showed that the microstructure of the hardened layer consists of martensite, a dispersion of fine carbides and retained austenite while the transition area mainly consisted of tempered Sorbite [5]. The modified heat treatment produces a mixed structure of martensite and lower bainite through short-term isothermal transformation at just above the martensitic transformation temperature. The $M_s$ Temperature, followed by oil quenching (after conventional austenitization), has been applied to three high-carbon low-alloy steels with different levels of nickel and chromium contents at similar molybdenum levels. The carbon allowed replacing relatively expensive additions of nickel and chromium, for their ultra-high strength application [6]. The cryogenic treatment was an inexpensive supplementary process to conventional heat treatment, which improves the properties of tool steels [7].

The earlier research works clearly indicate that most of the mechanical properties of good quality die steel can be further improved by performing different heat treatment processes and thus the life of a die can be significantly increased. Austenitizing is the most critical of all heating operations performed on die steels. Excessively high Austenitizing temperatures or long holding times may result in excessive distortion, excessive grain growth, loss of ductility and low strength. Under-heating may also result in low hardness and low wear resistance. Austenitizing is the treatment where the final alloy element separating between the austenitic matrix (which will transform to martensite on quenching) and the retained carbides. Quenching is also another important treatment to control the mechanical properties of die materials. The quenching medium must cool the work piece rapidly to get full hardness. Hot quenching minimizes distortion without affecting hardness. Tempering modifies the properties of quench-hardened tool steels to produce a more desirable combination of strength, hardness, toughness and wear-resistance. The as-quenched structure is a heterogeneous mixture of retained austenite, un-tempered martensite and carbides.

## 2. METHODOLOGY:

The following steps were carried out in the present experimental investigations:

- Samples of cold work tool steels D2 was prepared for metallographic tests and toughness measurement.
- Microstructures were examined; hardness and toughness were measured and recorded.
- By varying the system parameters necessary hardening and tempering operations were carried out in conventional furnace.
- Metallographic tests were carried out to observe the changes in microstructures after heat treatment.
- Hardness and toughness were measured for each specimen after heat treatment operations.
- From the different readings, curves were plotted to know the trends of the properties.

## 3. EQUIPMENT USED:

- Digital Specimen Mounting Press
- Laboratory Muffle Furnace
- Hand Polishing Steel Stand
- Metallurgical Specimen Polishing Machine
- Portable Electrolytic Polisher/Etcher
- Belt Polisher
- Inverted Stage Binocular Metallurgical Microscope
- CCD Camera
- Photo Micrographic Equipment
- Izod charpy Impact Testing Machine
- Rockwell cum Brinel Hardness Tester
- Quenching Bath:
- Grain Size Eye-piece
- Horizontal spindle surface grinder:
- Vertical spindle milling machine

## 4. METALLOGRAPHIC TEST

The High carbon high chromium- D2(C =1.0%, Mn =1.0%, Cr =12%) used for investigation. Different tests needed samples of different sizes. The details of the sample preparations for Metallographic test, for Hardness and Toughness tests are: Small pieces were cut from raw materials of D2. A soft grade rubber bonded abrasive wheel was used to cut the pieces. Bakelite thermo-set plastic granules were used to mould the samples by using specimen-mounting press (Fig. 1). After moulding, several steps required for grinding and polishing of specimens. Motor driven abrasive belt grinder (Fig. 2) was used with 240,320,400 and 600 grit belts. Then the samples were polished with successive emery papers of 1/0, 2/0, 3/0 and 4/0 followed by polishing with fine Alumina slurry. Final polishing was done with diamond paste on a disc-polishing machine (Fig. 3). The polished specimens were etched with 2% Naital (98% $C_2H_5OH$ with 2% $HNO_3$) for observations under metallographic microscope (Fig. 4).

Figure 1 : Specimen mounting press

Figure 2 : Abrasive belt grinder

Figure 3 Single disc polishing machine

Figure 4 : Metallographic microscope

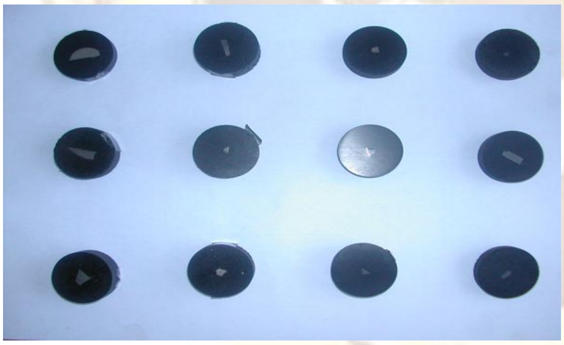

Figure 5 : Final test specimens

For metallographic tests of hardened specimens and tempered specimens, small pieces were cut from the specimens and moulded in the press. The cut specimens were prepared in the same manner as the preparation of pieces cut from the raw material. The final for metallographic tests are shown in Fig. 5.

### 4.1 Impact test

The rectangular test specimens of D2 tool steel material of size 10.5 mm x 10.5 mm x 76 mm were prepared from the raw materials purchased from local market (Dehradun). The specimens undergone for milling and light grinding to obtain the desired finished size of 10 x 10 x 75 mm. Then a U-notch of depth 2 mm and width of 2 mm has cut on a surface grinder by using a rubber bonded grinding wheel. The prepared samples have shown in Fig.6. The impact test was carried out on the on an impact test machine. The broken samples after impact test are shown in Fig. 7.

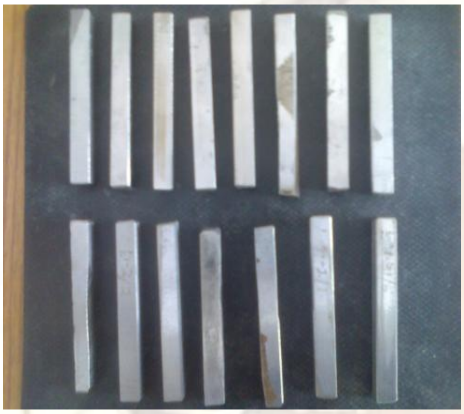

**Fig. 6, Prepared samples**

**Fig. 7, Broken specimens after impact test specimens**

### 4.2 Hardness test

The same rectangular samples of D2 prepared for impact testing were used for hardness testing. The hardness values after heat treatment were measured on hardness tester on Rockwell C-scale. For measurement of toughness after heat treatment, U-notch of 2 mm depth x 2mm width was cut on the specimens with the help of a rubber bonded parting off wheel on a surface grinder.

## 5. HEAT TREATMENT OF SPECIMENS

Six additional specimens were also made in same way for measuring the toughness and hardness before and after annealing. To remove the effect of machining and residual stresses, cyclic annealing was done by using the muffle furnace for all D2 specimens by heating to900$^{o}$C and holding for two hours, then cooling to 775$^{o}$C and holding for six hours, finally cooling in open air. The eighteen annealed specimens of D2 divided into four groups consisting of six, three, three and six specimens for investigating their hardness and toughness after hardening and after tempering. The temperature range and hardening/tempering soaking times for the experimental investigations were selected based on the material composition of the specimens. The microstructures of the specimens of D2 before and after annealing, hardening and tempering have also been studied in the present investigation.

## 5.1 RESULT AND DISCUSSION

### 5.2 Investigations on D2 tool steel

Before studying the effect of heat treatment, the specimen microstructure was made stress free and uniform in structure. Due to the machining operations performed on D2 material for sample preparation, there were changes in hardness and toughness, which were measured as 47Rc and 32 Joules respectively. This is due to the formation of martensite in the microstructure. By annealing, the hardness was reduced to 38Rc and toughness was increased to 45 Joules. The microstructure was also got refined with transformation of martensite to cementite. The said material properties of D2 tool steel are given in Table- 1

**TABLE 1 : MATERIAL PROPERTIES OF D2 TOOL STEEL**

| **Material properties** | **Before Annealing** | **After Annealing** |
|---|---|---|
| Hardness (Rc) | 47 | 38 |
| Toughness (Joules) | 32 | 45 |
| Microstructure | Cementite - carbide- martensite | Cementite – alloy carbides |

The six D2 specimens of first group, three D2 specimens of second and third groups each and six D2 specimens of fourth group were hardened at the austenitizing temperature of 970°C, 980°C, 990°C and 1000°C respectively. After hardening, hardened D2 specimens of each group were tempered at 160°C, 200°C, 250°C, 350°C and 550°C respectively. The specimens used for tempering in second and third group have increased from three to five because of one pre-tempered specimen being used for toughness measurement thereby breaking that specimen into two pieces. In the first and fourth group, the pre-tempered specimen used for toughness measurement was discarded, thereby reducing the group specimen to five. Hardening soaking time of 30 minutes and tempering soaking time of 180 minutes were used for D2 tool steel material. After hardening and tempering, their hardness and toughness were measured and the respective values are shown in Table 2. The microstructure of D2 specimens after hardening is martensite with retained austenite and carbides whereas after tempering the microstructure obtained under metallographic microscope was only tempered martensite and carbides. Taking the data from the Table 2, the hardness Vs austenitizing temperature curve, the ardness Vs tempering temperature curve, the tempering temperature Vs toughness curve and toughness Vs austenitizing temperature curve have been drawn.

**TABLE 2 EXPERIMENTAL DATA OF D2 SPECIMENS AFTER HEAT TREATMENT.**

| **Test-specimens** | **After hardening** | | | **After tempering** | | |
|---|---|---|---|---|---|---|
| | Austenitizing temperature, °C | Hardness, Rc | Toughness, Joules | Tempering temperature, °C | Hardness, Rc | Toughness, Joules |
| 1st Group | $970_{1,2,3,4,5,6.*}$ | $65_1$ | $01_1$ | $160_2$ | $65_2$ | $04_2$ |
| | | | | $200_3$ | $64_3$ | $06_3$ |
| | | | | $250_4$ | $63_4$ | $07_4$ |
| | | | | $300_5$ | $61_5$ | $08_5$ |
| | | | | $550_6$ | $57_6$ | $09_6$ |
| 2nd Group | $980_{7,8,9}$ | $66_7$ | $01_7$ | $160_7$ | $66_7$ | -<br>$5_8$<br>-<br>- |
| | | | | $200_8$ | $65_8$ | |
| | | | | $250_7$ | $64_7$ | |
| | | | | $300_7$ | $62_7$ | |
| | | | | $550_9$ | $55_9$ | $10_9$ |
| 3rd Group | $990_{10,11,12}$ | $70_{10}$ | $01_{10}$ | $160_{10}$ | $69_{10}$ | -<br>$5_{11}$<br>-<br>- |
| | | | | $200_{11}$ | $67_{11}$ | |
| | | | | $250_{10}$ | $65_{10}$ | |
| | | | | $300_{10}$ | $64_{10}$ | |
| | | | | $550_{12}$ | $54_{12}$ | $12_{12}$ |
| 4th Group | $1000_{13,14,15,16,17,18}$ | $72_{13}$ | $01_{13}$ | $160_{14}$ | $71_{14}$ | $04_{14}$ |
| | | | | $200_{15}$ | $69_{15}$ | $05_{15}$ |
| | | | | $250_{16}$ | $67_{16}$ | $06_{16}$ |
| | | | | $300_{17}$ | $65_{17}$ | $08_{17}$ |
| | | | | $550_{18}$ | $53_{18}$ | $13_{18}$ |

*Subscripts denote specimen numbers

It is clear from the fig. 8 that if the austenitizing temperature of the sample is increased, the hardness also increases. It was observed that the hardness was lowest (65Rc) for the sample using 970°C of austenitizing temperature and highest hardness (72Rc) was observed for the sample having 1000°C of austenitizing temperature and the curve has a rising trend.

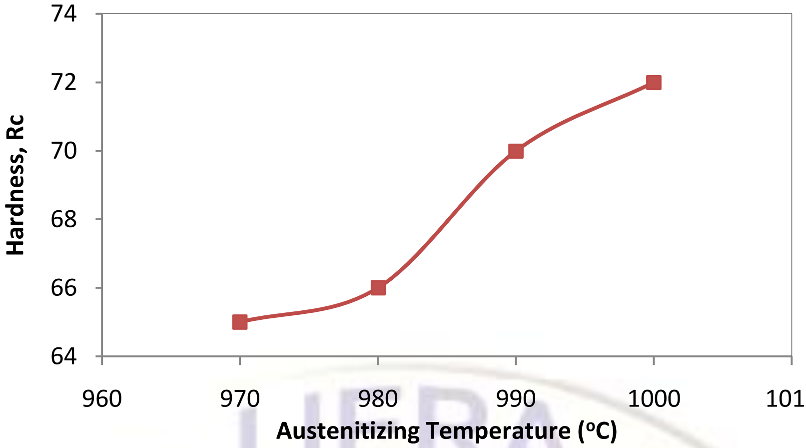

**Figure 8 : Effect of austenitizing temperature on hardness of D2 tool steel.**

The variation of hardness of D2 at various tempering temperatures in the range of 150°C-300°C. It was observed that the hardness of the sample decreases with the increase of tempering temperature. At lower tempering temperatures, there were nominal effects on hardness, but as the tempering temperature goes up, the effect of tempering temperature on its hardness becomes prominent. By tempering operation, the toughness of the material was increased at the cost of marginal loss of hardness. For the specimens hardened at 1000°C, at the tempering temperature range of 160°C- 200°C, for rise of 25% (40°C) of tempering temperature, the hardness decreases from 71Rc to 69Rc (2.81%).

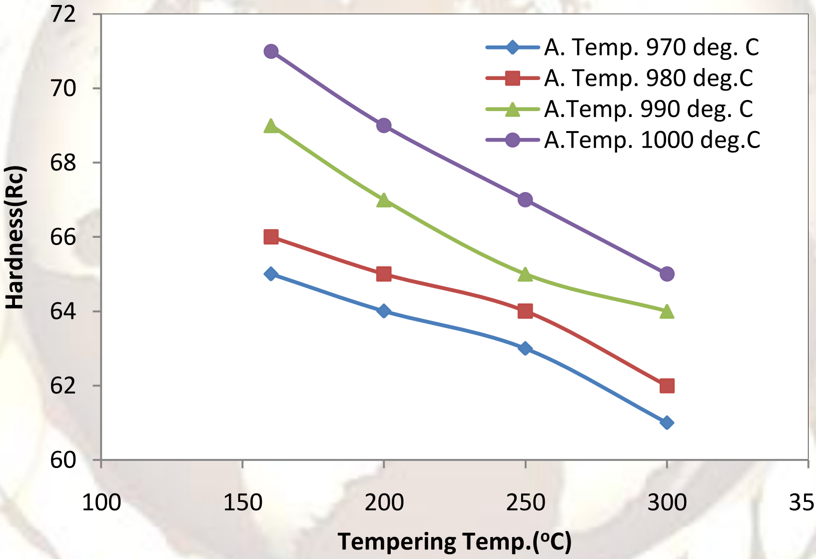

**Figure 9 : Effect of tempering temperature on hardness of D2 tool steel at 970, 980,990, and 1000°C austenitizing temperatures.**

At tempering temperature range of 200°C- 250°C, for the rise of 25% (50°C) of tempering temperature, the hardness decreases from 69Rc to 67Rc (2.89%). At tempering temperature range of 250°C- 300°C, for rise of 20% (50°C) of tempering temperature, the hardness decreases from 67Rc to 65Rc (2.98%). Thus the rate of decrease in hardness varies between nominal value of 2.81% to 2.98%. For the other samples hardened at 970°C, 980°C and 990°C, the rate of decrease in hardness due to rise of tempering temperature is having same trends. The reason of decrease in hardness due to increase in tempering temperature is that the martensite (hard constituent) is being transformed to comparatively soft troos tite also called tempered martensite.

Fig. 10 shows toughness of D2 at various tempering temperatures ranging from 160°C to 300°C. It was observed that in the lower tempering temperature range of 160°C- 200°C, for the rise of 25% (40°C) in tempering temperature, the rate of increase in toughness is 25% (4 to 5 Joules). At the tempering temperature range of 200°C-250°C, for the increase of 25% (50°C) in tempering temperature, the toughness increases by 20% (5 to 6 Joules). As the tempering temperature range increases to 250°C-300°C, for 20% increase of tempering temperature, the increase of toughness reaches from 6 Joules to 8 Joules and rate of increase in toughness is 33%. It is due to the fact that the lower tempering temperatures only relieve the hardening stresses without any changes of microstructures but higher tempering temperature causes the changes in microstructures.

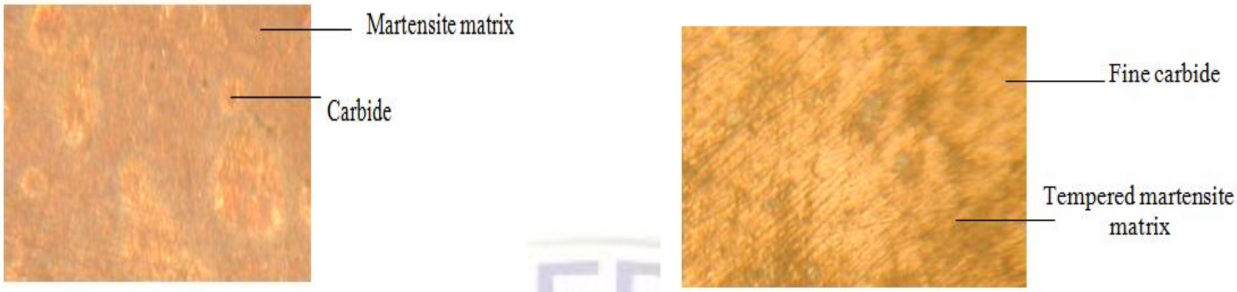

**Figure 10 : Effect of tempering temperature on toughness of D2 tool steel.**

Fig. 11shows the influence of austenitizing temperature on toughness of the investigated cold work tool steel (D2) at selected tempering temperatures. It was observed that the influence of austenitizing temperature on toughness at lower tempering temperature (200°C) is negligible.

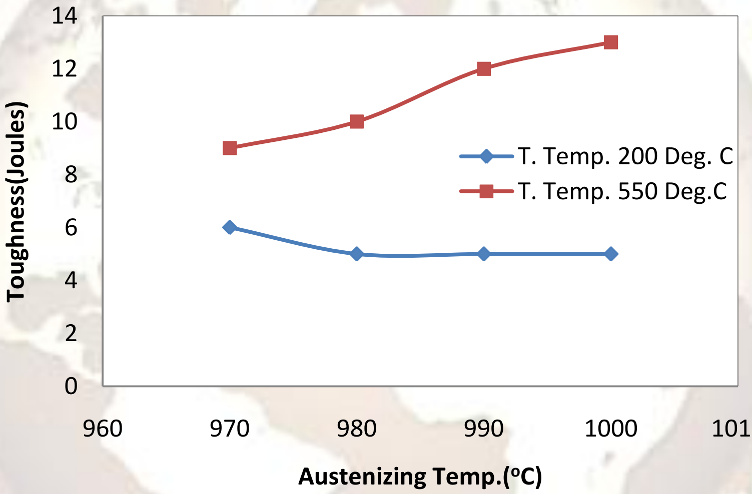

**Figure 11 : Effect of austenitizing temperature on toughness on D2**

When specimens were tempered at 200°C the toughness of samples changes from 5 to 6 Joules at Austenitizing temperature from 970 to 1000°C. Moreover, toughness varies between 9 to 13 Joules for same Austenitizing temperature when tempered at 550°C. It is due to the fact that at low temperature tempering only hardening stresses are removed but at high temperature tempering there is change of microstructures from martensite to tempered martensite that have better combination of mechanical properties.The microstructures of D2 specimens before annealing (Fig. 12), was expected to be same as that of the microstructure of specimens after annealing (Fig. 13). But it was not so because of the effects across the cross-section of the specimens due to heat developed during machining. It causes the formation of martensite along with the cementite-carbide/pearlite. The annealed specimens of D2 show the uniform distribution of microstructure of carbides and cementite combination.

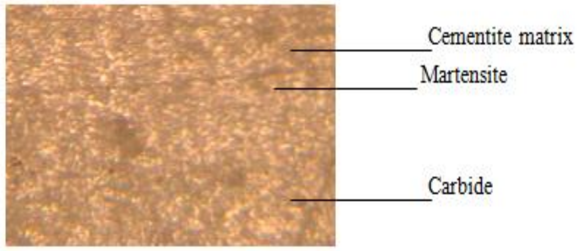

**Figure 12** D2 tool steel specimens with cementite-carbide structure after annealing.

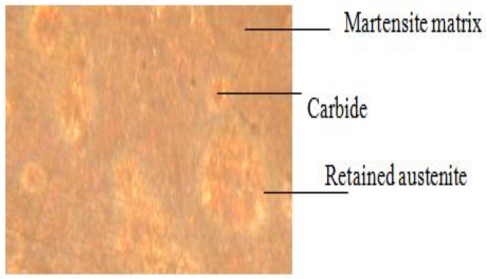

**Figure 13** Microstructures of D2 tool steel specimens before annealing.

The microstructures of D2 specimens after hardening (Fig. 14) show carbide particles throughout the martensite matrix. It was observed Very less retained austenite and precipitation of carbides were noticed along the grain boundaries. The microstructure of D2 specimen after tempering (Fig 15) is comparatively finer because (during tempering of the specimens) the martensite decomposes into emulsified form of pearlite called troostite (tempered martensite). It is very fine in nature with reasonable toughness. Thus the microstructure obtained after tempering provides a good combination of mechanical properties likes high hardness, high strength, high wear resistance, greater dimensional control and considerable toughness and temperature resistance. It serves our main objectives of increasing the die life through heat treatment.

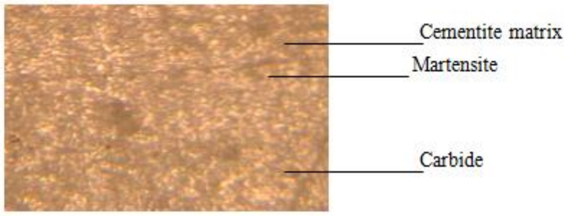

**Figure 14** D2 tool steel specimens with martensite, retained austenite and carbide particles after hardening.

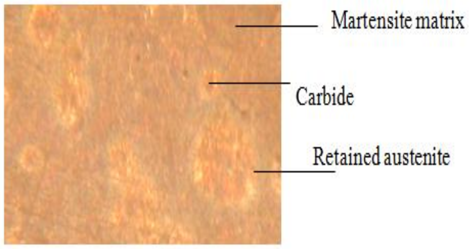

**Figure 15** D2 tool specimens with fine tempered martensite and fine carbide particles after tempering.

## 6. CONCLUSIONS

Based on the results obtained from the extensive tests and investigations performed on cold work tool steels D2, the following conclusions are drawn:

i) Annealing should be done for the tool steels before performing any heat- treatment to avoid the effects of any changes on material properties caused during the sample preparation.

ii) The hardness of D2 tool steel increases with austenitizing temperature and the lowest and highest hardness were obtained at 970°C and 1000°C respectively.

iii) The hardness of the D2 tool steel decreases with increase of tempering temperature. At lower tempering temperature range (160°C-200°C), the effect of tempering temperature on hardness is very small but as the tempering temperature increases hardness becomes prominent.

iv) At lower tempering temperature range 160°C-200°C, the rate of increase in toughness was 25% ( i.e. 4 to 5 Joules). As the tempering temperature increases from 200°C to 250°C and 250°C to 300°C, the toughness was increases by 20% and 33% respectively. It may be interpreted that at lower tempering temperatures only relieves the hardening stresses without any change in microstructures but higher tempering temperature causes the changes in microstructures.

v) When specimens were tempered at 200°C the toughness of samples changes from 5 to 6 Joules at Austenitizing temperature from 970 to 1000°C. Moreover, toughness varies between 9 to 13 Joules for same Austenitizing temperature when tempered at 550°C. It is due to the fact that at low temperature tempering only hardening stresses are removed but at high temperature tempering there is change of microstructures from martensite to tempered martensite.

vi) The microstructure of D2 specimen after tempering is comparatively finer because (during tempering of the specimens) the martensite decomposes into emulsified form of pearlite called troostite (tempered martensite). It was very fine in nature with reasonable toughness. Thus the microstructure obtained after tempering provides a good combination of mechanical properties likes high hardness, high strength, high wear resistance, greater dimensional control and considerable toughness and temperature resistance.

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