

# Tempering of die-steels based on indigenous materials

R. K. DUBEY, S. P. CHAKRAVARTY and P. K. GUPTA

*This paper presents studies on the effects of quenching temperature, tempering temperature and time on the hardness of three die steels of similar carbon contents but having increasing chromium percentages. While tempering in the range of 150°C to 400°C it was observed that hardness decreased as quenching temperature was raised. All the steels showed secondary hardening phenomenon in relation to the variables mentioned above. It was also observed that on tempering the steels above 450°C, after prior quenching from increasing temperatures, hardness gradually increased up to the quenching temperature of 1100°C, beyond which followed a decrease in hardness. This phenomenon was the same at all tempering temperatures upto 601°C, but the increase in hardness from low quenching temperatures to the maximum quenching temperature was less at 450°C than at the higher temperatures and attained a maximum at 600°C. At 650°C secondary hardening decreased with the increase of quenching temperature.*

THE development of die steels based on indigenous materials is closely associated with the phenomenon of decomposition of martensite and retained austenite and formation and distribution of carbides in a tempered steel. The end use of die-steel is primarily a question of compromise between hardness and toughness characterised and developed by proper heat-treatment. It is therefore necessary to examine the changes that occur during heating, quenching and tempering. Extensive studies have been made on the tempering behaviour of tool steels by several investigators<sup>1-3</sup> who established that steels are normally characterized in the quenched state by substantial amounts of retained austenite in addition to untempered martensite and excess carbide and who also observed the phenomenon of decomposition of retained austenite during cooling from the tempering temperature. It is known that the rate of decomposition of retained austenite is not the same throughout the tempering range; the isothermal reactivity of retained austenite is very prompt in the lower range of tempering temperature and rather slow as tempering temperature is increased above 425°C. As a result more austenite is available for transformation during cooling and it has probably a structure composed of bainite or tetragonal martensite or both depending on the conditioning of retained austenite.

The effects of quenching temperature, tempering temperature and time on hardness have been studied on

Messrs R. K. Dubey & P. K. Gupte, Scientists & Mr. S. P. Chakravarty, Senior Laboratory Assistant. National Metallurgical Laboratory, Jamshedpur.

three die steels of similar carbon content with increasing chromium percentages. Secondary hardening phenomena were observed in steels containing chromium in relation to these three variables.

## Experimental procedure

The investigation was carried out on three commercially important die-steels based on indigenous materials such as chromium and vanadium. These steels were produced in the laboratory in a 25-lb basic lined electric induction furnace. The cast ingots were forged to  $\frac{1}{2}$ " sq. bars after proper annealing.

The steel bars were subjected to a long homogenizing treatment for uniform dissemination of segregated atoms.

Table I gives the chemical analysis of the steels investigated.

TABLE I Chemical compositions of steels investigated (weight %)

Steel	C	Si	Mn	Cr	V	Ti	Mo	W
Ds <sub>2</sub>	1.13	0.37	1.03	1.58	0.14	Trace	0.10	—
Ds <sub>3</sub>	1.09	0.51	0.88	2.46	0.11	Trace	0.09	—
Ds <sub>4</sub>	1.01	0.32	0.75	4.82	0.27	Trace	0.15	—

The steel samples of  $5/8" \times 3/8" \times 1/4"$  size were surface ground on both the faces and bright nickel plated in order to prevent oxidation during heating at high temperatures. In each test, the 1" dia. tube furnace used for the austenitizing treatment provided a mild carbonaceous atmosphere in order to minimise the loss in surface carbon due to oxidation during heating of the specimen.

Mineral oil was used for quenching and hardening throughout the experiments. After suitably heating the specimens to various austenitizing temperatures, they were quenched in oil and immediately after cooling to hand warm temperature they were re-heated to a series of temperatures between 150°C and 650°C. Salt-bath was used for lower temperature tempering upto 350°C, and lead-bath for higher temperature tempering.

After tempering the specimens were generally cooled in air.

### Effect of quenching temperature on hardness

For obtaining maximum hardness in steel, the primary condition is that mainly carbon and less significantly the other alloying elements should completely go into austenitic solution so that on quenching, the breakdown of austenite bears a maximum hardness relationship to the carbon content of the steel. The solubility of carbides, which depends on their nature and compositions, varies in different compositions of steels and progressively increases as the austenitizing temperature is raised.

As many as five quenching temperatures were used to study the effect of solution of carbides on hardness. The austenitizing temperatures were selected at 800°C, 850°C, 900°C, 950°C and 1000°C. Table II shows the effect of quenching temperature on hardness.

TABLE II Effect of quenching temperature on hardness

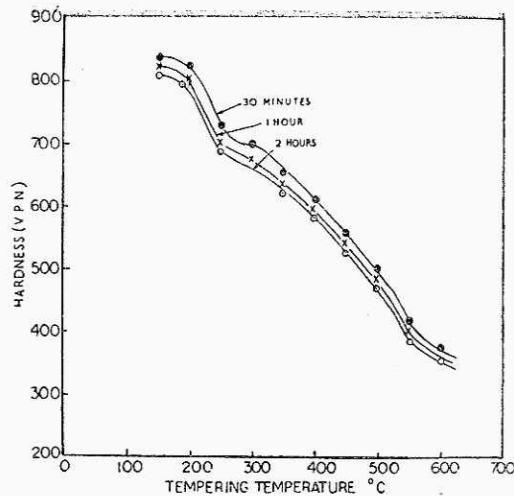
Quenching temperature °C	Hardness (VPN)		
	Steel DS <sub>2</sub>	Steel DS <sub>3</sub>	Steel DS <sub>4</sub>
800°C	880	832	—
850°C	877	880	870
900°C	823	817	905
950°C	817	771	840
1000°C	778	760	790

It is clear that as the quenching temperatures increase, steels DS<sub>2</sub> and DS<sub>3</sub> drop in hardness above 850°C. In steel DS<sub>4</sub>, the hardness reaches its maximum at the quenching temperature of 900°C. Above the quenching temperature of 900°C the amount of retained austenite was a little larger. At lower quenching temperatures hardness decreased as usual. All the steels showed a marked similarity in this behaviour.

### Effect of quenching media on the as-quenched hardness

Three quenching media e. g. water, oil and air were chosen to study the effect of cooling rate on the degree of martensitic hardness as well as the amount of retained austenite. Table III shows the changes in hardness values due to different cooling rates. Steels DS<sub>2</sub> and DS<sub>3</sub> were quenched at 850°C and steel DS<sub>4</sub> at 900°C.

It is clear from this table that after quenching from the same austenitizing temperature, rapid cooling imparts greater hardness than slow cooling. Such differences in the hardness values were attributed to the fact that more austenite is retained in slower cooling rates.



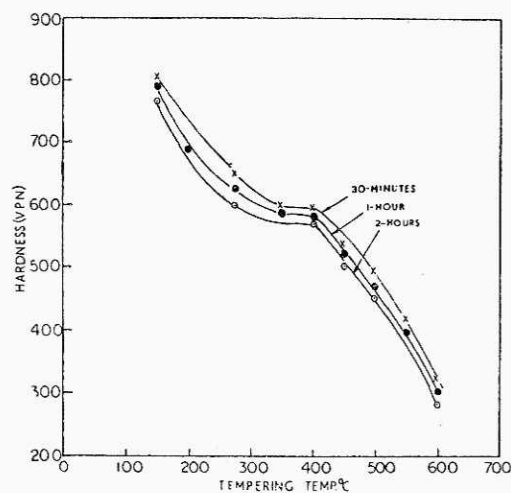
1 Effect of tempering time on hardness of steel DS<sub>2</sub>

TABLE III Changes in hardness values with different cooling rates

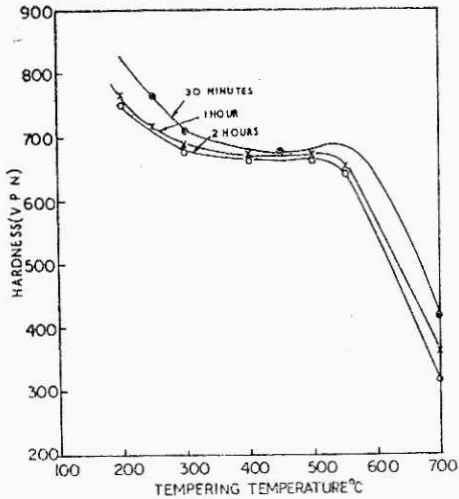
Quenching media	Hardness (VPN)		
	Steel DS <sub>2</sub>	Steel DS <sub>3</sub>	Steel DS <sub>4</sub>
Water	897	900	920
Oil	877	880	905
Air	815	820	835

### Effect of tempering time

Experiments were conducted to study the effect of holding time on breakdown of retained austenite during tempering of steels DS<sub>2</sub>, DS<sub>3</sub> and DS<sub>4</sub>. After quenching from the austenitizing temperature the specimens of steel DS<sub>2</sub>, DS<sub>3</sub> and DS<sub>4</sub> were tempered at temperatures between 150°C and 650°C for ½ hour, 1 hour and 2 hours. The results are represented in Figs. 1, 2 and 3.



2 Effect of tempering time on hardness of steel DS<sub>4</sub>

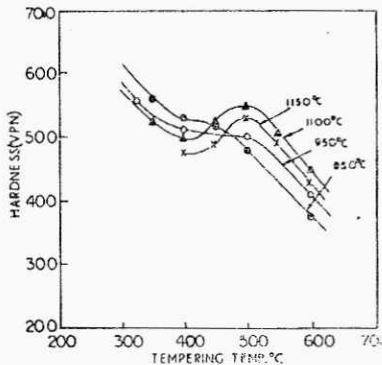


3 Effect of increasing tempering time on hardness of steel  $DS_4$

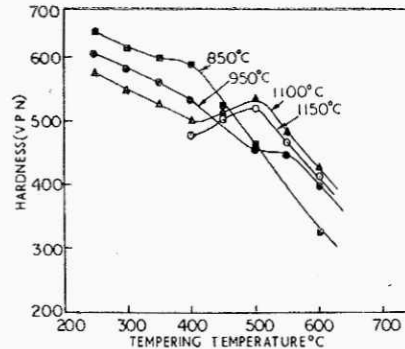
The curves show clearly that as tempering time is increased subsequent hardness decreases more or less uniformly. Steel  $DS_3$  showed a flattening in the tempering range of 350 to 400°C; in steel  $DS_4$ , this flattening tendency was found to prolong upto 500°C, while no appreciable flattening tendency was observed with steel  $DS_2$ .

#### Effect of quenching temperatures on tempering characteristics

The hardness characteristics of steels tempered at various temperatures were studied and the results are summarized in Figs. 4, 5 and 6. Vickers Pyramid hardness readings were taken on each quenched and tempered specimen with due precautions to ensure accurate results. Figs. 4, 5 and 6 show that there was no secondary hardening until the tempering temperature reached 450°C and at this point a marked increase in



4 Hardness developed in four specimens of steel  $DS_2$  quenched from 850°C, 950°C, 1100°C and 1150°C after various reheats

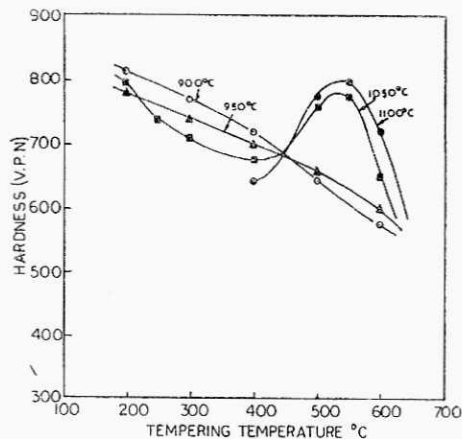


5 Hardness developed in four specimens of steel  $DS_3$  quenched from 850°C, 950°C, 1100°C and 1150°C, after various reheats

hardness with increasing quenching temperature was observed, which predominated at all tempering temperatures upto 650°C. This observation held good upto the quenching temperature of 1100°C, after which there was a drop in hardness. Hardness increased steadily due to secondary hardening in relation to quenching temperature and tempering temperature and attained a maximum at the tempering temperature of 600°C, when the increase in steel  $DS_3$  and  $DS_4$  was as much as 95 and 150 VPN respectively. At 650°C, the increase in hardness was much less and might be regarded as virtually the upper limit of tempering temperature beyond which hardness decreased rapidly without secondary hardening effect. Similar effect of secondary hardening was observed in steel  $DS_2$  also, but to a smaller degree, owing to the lower chromium content.

#### Effect of duple tempering

In order to obtain an idea of the effect of double tempering on these steels the samples  $DS_2$ ,  $DS_3$  and  $DS_4$  which were quenched and hardened from the austenitizing temperature of 950°C were tempered at 450°C, 550°C and 650°C for 1 hr and allowed to cool



6 Hardness developed in steel  $DS_4$  quenched from 900°C, 950°C, 1050°C and 1100°C, after various reheats

n air until their temperature was handwarm. Immediately the same specimens were retempered at the previous tempering temperatures for further one hour. It was seen that the hardness had dropped appreciably indicating the absence of retained austenite after the specimen was tempered once.

### Discussion of results

At lower ranges of tempering temperature, the quenched and hardened die steels have greater tendency to yield to softening, while on raising the tempering temperature above 425°C a sudden change in the precipitation mechanism in steel develops a secondary hardness, a vital change in the combined effect of strength and toughness during the process of tempering.

The whole process of tempering mechanism can be broadly divided into four stages. During the first stage of tempering, tetragonal martensite decomposes into cubical symmetry and is characterized by a marked softening and a decreased volume. It has been found during tempering in the range of 150° to 400°C that hardness decreased as the quenching temperature was raised. This softening, i. e. decrease in hardness is perhaps due to the agglomeration of cementite particles rejected by the tetragonal martensite, without having a chance to precipitate as alloy carbides due to the low diffusion rate of alloying elements at such low tempering temperatures.

The amount of retained austenite available for isothermal decomposition is also dependent on the holding period for which the steel is tempered. At such low tempering range retained austenite decomposes partially into bainite for want of sufficient time (1 hr) for the transformation of retained austenite. With increasing quenching temperature there is greater occurrence of retained austenite which causes a regular drop in hardness during the first stage of tempering of steels DS<sub>2</sub>, DS<sub>3</sub> and DS<sub>4</sub>.

In all the steels the second stage of tempering is characterized by the precipitation of alloy carbides from the retained austenite as the tempering temperature is raised to 450°C and above. As a result there has been an increase in hardness as well as in resistance to softening.

When tempering temperatures are varied in the range 450°C to 600°C the increase in hardness is neither the same nor is it regular. It was observed that on tempering the steels at 450°C after prior quenching

from increasing temperatures, hardness gradually increased upto the quenching temperature of 1100°C, after which it decreased. This phenomenon was constant for all the tempering temperatures. But at the tempering temperature of 450°C the increase in hardness from low quenching temperature to maximum quenching temperature is less than at the higher tempering temperatures. This increase in hardness, which is due to the secondary hardening becomes maximum as the tempering temperature reaches 600°C. As the compositions of these steels differ only by their chromium content a lower value is obtained in steel DS<sub>2</sub>. The slight increase in hardness at 450° and 500°C is due to involvement of more alloy carbides precipitated from retained austenite and possibly does not account for the decomposition of retained austenite which is likely to be very sluggish or conditioned.

The third stage of tempering attains maximum secondary hardness in a comparative study with the variation of quenching temperature in relation to a particular tempering temperature. At 550° and 600°C there was a rapid increase in hardness as quenching temperature varied. This increase is due to the decomposition of retained austenite which is greatly relieved of sluggishness by prior precipitation of carbides and the transformation of austenite take place during cooling of the steel in air after tempering.

At 650°C the secondary hardening decreased with the increase of quenching temperatures. This is followed by coagulation of cementite of tetragonal martensite with the alloying elements diffusing at a rapid rate. When quenched die steels are tempered at high temperature (above 650°C) they approach the annealed condition.

### Acknowledgements

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