Surface Decarburization Behaviour of A Novel Die Steel

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Abstract. The surface decarburization behaviour of a new kind of hot-working die steel under oxidative environment was systematically investigated. The thickness of the decarburization layer under different temperatures and different holding time were measured using metallographic analysis. The results show that the thickness of decarburization layer has linear relationship with the square root of holding time under a certain temperature. The decarburization sensitive temperature of this die steel is 1100℃.

1. Introduction
When steel is heated to a high temperature under oxidative environment, in the air for example, it will be coated with oxidation and decarburization layer [1-2]. This is a type of metal erosion, a very important metallographic issue about diffusion as well as one of the common scientific problems in the quality control of medium or high carbon steel [3-5], especially for hot working die steel. The study of the oxidation and decarburization of die steel would help to improve its quality and extend the service-life.

The main factors related to oxidation and decarburization are environment, heating temperature and holding time [6-9]. When the steel is heated in the air, normally it starts to be oxidized at the temperature of 500℃, and to be decarbonized between 800℃ and 850℃, while slight below 900℃ and severe above 1000℃. Both oxidation and decarburization reactions are accelerated with different extent when the temperature increases. Due to the impact of iron-oxide cyclical thickening and spalling during heating process, so large discrepancy exists between theoretical calculation value and the actual value of the oxide layer thickness that only qualitative analysis can be done on oxidation layer thickness. Therefore, the effect of oxidation on decarburization cannot be considered in order to simplify the analysis of decarburization, which can guide production fast and practically.

As for die steel, the oxidation and decarburization behaviour is heavily influenced by alloy elements, resulting in the different sensitive temperature of decarburization for different kind of die steel. It is important to determine the temperature for each kind of steel before heating during forging.

However, previous studies on decarburization were primarily concerned with the total thickness of the decarburization layer [11-13]. However, the control of the decarburization layer and temperature is of great significance and would be helpful to the application of a novel die steel. In this article, the new kind of die steel was heated to different temperatures under air condition followed by air cooling.
The relationship between the thickness of decarburization layer and holding time under each temperature was studied.

2. Materials and Methods
The specimen was made with a new die steel RX, with the size of 15mm×15mm×15mm. Its composition was shown in Table 1. They were heated in Muffle under air atmosphere condition (the precision of the thermostat was ±3℃) followed by air cooling. The heating temperature was chosen as 1030℃, 1050℃, 1080℃, 1100℃ and 1130℃ respectively, and the holding time was chosen as 30 min, 40 min, 50 min and 60 min respectively.

| Table 1. Chemical Composition of the Specimen (wt. %) |
|-----------------|--------|-------|-------|--------|--------|-------|
|                | Fe     | C     | Cr    | Si     | Mo     | Mn    | V     | S、P               | X: 0.02 |
| Balance        | Balance| 0.40  | 3.12  | 0.54   | 2.36   | 0.70  | 0.62  | <0.03              | X: 0.02 |

Specimens were cut, ground and mechanically polished according to the standard procedures for metallographic observations, and then etched with 4% nitric acid and alcohol solution for 40s. Optical microscopy and scanning electron microscopy (SEM, ZEISS EVO18) were used to examine the microstructure and the thickness of decarburization layer. Vickers hardness testing of the decarburization layer was performed at a loading of 500gf for 15s. For each test condition, three indentations were measured to calculate the average values.

3. Results
The optical micrographs and the corresponding hardness curves of the decarburization layer of typical decarburization samples are shown in figure 1. It can be seen that there is a clear dividing line between the complete decarburization layer and the partial decarburization layer, and so is the clear jump in microhardness between the two layers.

The thickness of the complete decarburization layer is shown in table 2. The SEM microstructure of the samples is shown as figure 2. As shown in SEM observation, complete decarburization layer, partial decarburization layer and none decarburization layer distribute from surface to the core. The complete decarburization is characterized by netted ferrite microstructure, partial decarburization layer is characterized by ferrite and tempered martensite mixture, and none decarburization layer is characterized by tempered martensite microstructure. The microstructure observation is consistent with the results of hardness test. Previous literature [14, 15] reported that with the extension of holding time (always more than 90 min), the growth rate of decarburization layer thickness gradually reduced. As for die steel, high temperature exposure occurs mainly in the forging process, while forging is generally a relatively shorter-time process. Therefore, decarburization behavior in no more than 60 min is discussed in this work.

| Table 2. The thickness of the complete decarburization layer under certain temperature and holding time (um). |
|-----------------|--------|--------|--------|--------|--------|
|                 | 1030℃ | 1050℃ | 1080℃ | 1100℃ | 1130℃ |
| 30min           | 0.103  | 0.14   | 0.245  | 0.26   | 0.273  |
| 40min           | 0.167  | 0.196  | 0.286  | 0.31   | 0.335  |
| 50min           | 0.214  | 0.245  | 0.34   | 0.35   | 0.383  |
| 60min           | 0.269  | 0.31   | 0.385  | 0.399  | 0.425  |
Figure 1. Optical micrographs and corresponding hardness gradient curves of decarburization samples: (a) 1030℃/60min; (b) 1050℃/60min; (c) 1100℃/60min.

Figure 2. The SEM microstructure of the samples (1100℃/60min) from surface to the core: (a) complete decarburization layer; (b) partial decarburization layer; (c) none decarburization layer.
4. Discussion

Generally, decarburization are realized by atomic diffusion, and the degree of decarburization depends on the diffusion rate related to the temperature, the diffusion distance related to time, the oxygen supply state of steel surface and the state of interface microstructure. The thickness of oxidation and decarburization layer is directly related to temperature and holding time. The decarburization behavior is promoted with the increase of temperature and prolonging of holding time.

The first period of decarburization is restricted by interface chemical reactions, of which equation was:

\[ 2\text{Fe}_3\text{C} + \text{O}_2 = 6\text{Fe} + 2\text{CO} \quad (1) \]
\[ \text{Fe}_3\text{C} + \text{H}_2\text{O} = 3\text{Fe} + \text{CO} + \text{H}_2 \quad (2) \]
\[ \text{Fe}_3\text{C} + \text{CO}_2 = 3\text{Fe} + 2\text{CO} \quad (3) \]

When the decarburization layer grows to a certain thickness, the diffusion velocity of carbon becomes much smaller than reaction velocity. Then the decarburization is controlled by diffusion process. According to Fick’s Law, the flux of carbon atoms across the decarburization layer in unit time under a certain temperature is:

\[ j = -D \frac{dC}{dx} \quad (4) \]

Where \( D \) is diffusion coefficient, \( C \) is carbon atom concentration and \( x \) is the distance along the diffusion direction.

The decarburization layer is so thin that the carbon concentration in the layer can be treated as linear distribution [4].

\[ j \int_0^Y dx = -D \int_{C_0}^{C_Y} dC \]

\[ j = \frac{1}{Y} D(C_Y - C_0) \quad (6) \]

Where \( C_0 \) is the carbon concentration of \( x=0 \) point and \( C_Y \) is the carbon concentration of \( x=Y \) point. Besides, the diffusion flux is proportional to the growing velocity of decarburization layer:

\[ j = K \frac{dY}{dt} \quad (7) \]

Then,

\[ K \frac{dY}{dt} = \frac{1}{Y} D(C_Y - C_0) \]

\[ \int_0^Y Y dY = \int_0^t K \, dt \]

\[ Y^2 = K \cdot t \quad (10) \]

It was shown that the relationship between the thickness of decarburization layer and holding time is parabolic, while the relationship of thickness of decarburization layer and square root of holding time is linear. The Linear Regression Analysis was performed using Origin, with the square root of holding time as Variable and the thickness of decarburization layer as Dependent Variable:

\[ Y = A + B \sqrt{T} \quad (11) \]

Where \( Y \) is the thickness of decarburization layer and \( T \) is the holding time. The constants \( A \) and \( B \) were calculated using the data of table 2 and returned to equation (11), resulting in the equation under different heating temperature. The values of \( A \) and \( B \) are shown in table 3. The relationship between the thickness of decarburization and holding time are shown in figure 3. According to figure 3, the thickness of the complete decarburization layer has linear relationship with the square root of holding time under a certain temperature when precision permitted.

| Table 3. The fitting values of \( A \) and \( B \) in equation (11). |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 1030°C | 1050°C | 1080°C | 1100°C | 1130°C |
| \( A \) | -0.2764 | -0.2677 | -0.1021 | -0.0672 | -0.1024 |
| \( B \) | 0.0694 | 0.0737 | 0.0625 | 0.0597 | 0.0688 |
Figure 3. Relationship between the thickness of the decarburization layer and holding time.

According to the equation $Y = A + B \sqrt{T}$, if $Y=0$, then $T_0 = (-A/B)^2$, where $T_0$ is the beginning time when decarburization surface is formed. Apparently, the smaller $T_0$ is, the easier decarbonizing. According to equation (4)-(11), the values of $T_0$ for different temperatures are displayed in table 4.

### Table 4. Values of $T_0$ for different heating temperatures.

| Tem. (℃) | 980  | 1000 | 1030 | 1050 | 1080 | 1100 | 1130 |
|----------|------|------|------|------|------|------|------|
| $T_0$ (min) | 19.03 | 17.70 | 15.86 | 13.20 | 2.67 | 1.27 | 2.21 |

From table 4, it was shown that $T_0$ decreases sharply when the temperature is 1080℃, to the lowest at the temperature of 1100℃. So the decarburization sensitive temperature of this die steel is 1100℃, which should be avoided when heat processed.

In addition, the constant $B$ in the equation shows the changing speed of the thickness of decarburization layer with the prolonging of the heating time. The larger of $B$, the faster the thickness of decarburization layer grows when the holding for longer time at this temperature.

5. Conclusions

The surface decarburization behavior of a new kind of hot-working die steel under oxidative environment was systematically investigated. Main conclusions can be drawn as follows:

1. The relationship between the thickness of decarburization layer and holding time was Linear Regression analyzed using Origin, with the square root of holding time as Variable and the thickness of decarburization layer as Dependent Variable parabolic. The result shows the surface decarburization layer thickness has linear relationship with the square root of holding time.

2. The beginning time when decarburization surface is formed under different temperatures was calculated. The results indicated the decarburization sensitive temperature of this die steel was 1100℃, which should be avoided when heat processed.

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