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The Orthogonal-Regression Analysis on Cooling Rate of PVP Quenchant

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Newly developed polyvinyl pyrrolidone (PVP)-based quenchants have more oil-like cooling characteristics and thus can replace oil for quenching high alloy steel with complicated sections. The factors influencing the cooling rate are studied in this paper using the method of orthogonal regression. The regression equations show that concentration, bath temperature, and agitation are highly significant factors in the maximum cooling rate. For the cooling rate at 300 °C, concentration is critical, temperature is related, but agitation is not related. Based on the regression equation, suggestions regarding the better control of cooling rate in practice are given.

Keywords cooling rate, orthogonal regression, polymer, PVP, quenchants

1. Introduction

Polyvinyl pyrrolidone (PVP) is a newly developed polymer quenchant with more oil-like cooling characteristics compared with polyalkylene glycols (PAGs)-based quenchants and thus can possibly replace oil for the quenching of high-alloy content steel.[1] However, its properties are not widely well known among users. Orthogonal test and regression analysis used to be separate mathematic branches, but now have been combined and a new method, orthogonal regression method, has been developed.[2]

The cooling speed of polymer quenchants is strongly influenced by concentration, bath temperature, and agitation. Regarding the cooling rate, users are more interested in the maximum cooling rate and the cooling rate at 300 °C; the former is the guarantee for hardening and the latter has a close relationship with the tendency of distortion and crack. This article, using a method of orthogonal regression, studies the effect of concentration, bath temperature, and agitation upon the maximum cooling rate as well as the cooling rate at 300 °C of PVP-based quenchants and the respective regression equations. Based on the equations, the suggestions for better control of the cooling rate are then given.

2. Test Design and Results

In Table 1, X_1 taking 1, 0, and −1 represents concentration C(wt.%) with high level (20%), zero level (15%), and low level (10%); X_2 taking 1, 0, and −1 represents temperature T(°C) in high level (60 °C), zero level (40 °C), and low level (20 °C); X_3 taking 1, 0, and −1 represents agitation speed V(m/s) in high level (1.6 m/s), zero level (0.8 m/s), and low level (0). Lines 9, 10, and 11 are the repeated tests under the zero levels.

The cooling rate test is made with the ivf quenchotest (Walfson Heat Treatment Centre, Birmingham, UK), the ISO standards, i.e., the probe made of Inconel 600 with the dimension of ϕ12.5 mm × 60 mm with a thermocouple is welded in the center. The test results—maximum cooling rate and the cooling rate at 300 °C—are listed in the last two columns.

3. Regression and Orthogonal Analysis

$$X = \begin{pmatrix} 1 & 1 & 1 & 1 \\ 1 & -1 & 1 & 1 \\ 1 & 1 & -1 & 1 \\ 1 & -1 & -1 & 1 \\ 1 & 1 & 1 & -1 \\ 1 & -1 & 1 & -1 \\ 1 & 1 & -1 & -1 \\ 1 & -1 & -1 & -1 \end{pmatrix}$$

$$B = X'Y = \begin{pmatrix} \sum_a Y_a \\ \sum_z X_1 \ aYa \\ \sum_a X_2 \ aYa \\ \sum_a X_3 \ aYa \end{pmatrix} = \begin{pmatrix} B_0 \\ B_1 \\ B_2 \\ B_3 \end{pmatrix} = \begin{pmatrix} 966 \\ -130 \\ -220 \\ 116 \end{pmatrix}$$

$$A^{-1} = (X'X)^{-1}$$

$b = A^{-1}B$ Thus, $b_0 = 120.7$; $b_1 = -16.25$; $b_2 = -27.5$; $b_3 = 14.5$

Therefore, the regression equation is as follows:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3$$

$$Y = 120.7 - 16.25X_1 - 27.5X_2 + 14.5X_3 \qquad \text{(Eq 1)}$$

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Table 1 Orthogonal Regression Table

No.	Elements				Max. Cooling Rate Y (°C/s)	Cooling Rate at 300 °C Z (°C/s)
	X_0	X_1	X_2	X_3		
1	1	1 (20)	1 (60)	1 (1.6)	90	16
2	1	−1 (10)	1	1	125	70
3	1	1	−1 (20)	1	148	51
4	1	−1	−1	1	178	75
5	1	1	1	−1 (0)	62	15
6	1	−1	1	−1	96	65
7	1	1	−1	−1	118	50
8	1	−1	−1	−1	149	70
9	1	0 (15)	0 (40)	0 (0.8)	135	50
10	1	0	0	0	131	53
11	1	0	0	0	132	54
B_j (a)	996/412 (b)	−130/−148	−220/−80	116/12		
b_j	120.7/51.5	−16.25/−18.5	−27.5/−10	14.5/1.5		
B_j^2		16 900/21 904	48 400/6400	13 456/144		
$Q_j = b_jB_j$		2112.5/2738	6050/800	1682/18		

(a) Lines following line 11 are relative calculations.
(b) Numbers before / refer to maximum cooling rate; after the / refer to cooling rate at 300 °C.

$$\frac{1}{N+m}\left(\sum Y_a + Y_{01} + Y_{02} + Y_{03}\right) = \frac{1}{8+3}(966 + 135 + 131 + 132) = 124$$

may be used to replace b_0, and let $X_1 = 2 \times (C - 20)/10 + 1$; $X_2 = 2 \times (T - 60)/40 + 1$; $X_3 = 2 \times (V - 1.6)/1.6 + 1$

Equation 1 will be as follows:

$$Y = 220 - 3.25C - 1.37T + 18.13V \quad \text{(Eq 2)}$$

Equation 2 is the regression equation of maximum cooling speed over concentration, C; Temperature, T; and the agitation, V. The regression equation of cooling rate at 300 °C can be determined as follows:

$$Z = 125.7 - 3.7C - 0.5T + 1.87V \quad \text{(Eq 3)}$$

4. Discussion

4.1 Significance Level

To Eq 2:

$$f_t = 8 - 1 = 7 \quad S_r = Q_1 + Q_2 + Q_3 = 9844.5,$$

$$St = \sum Ya^2 - \frac{B_0^{\ 2}}{N} = 9853.5$$

$$S_1 = S_t - S_r = 9; f_1 = f_t - f_r = 7 - 3 = 4$$

$$F = (S_r/f_r)/(S_1/f_1) = 1458.44 >> F_{0.01}(3,4) = 16.69$$

Thus, the regression equation of maximum cooling rate has a high level of significance.

$$F_1 = Q_1/(S_1/f_1) = 938 >> F_{0.01}(1,4) = 21.2$$

$$F_2 = Q_2/(S_1/f_1) = 2688.89 >> F_{0.01}(1,4) = 21.2$$

$$F_3 = Q_3/(S_1/f_1) = 747.5 >> F_{0.01}(1,4) = 21.2$$

Therefore, all the coefficients have a high level of significance.

To Eq 3:

$$f_t = 8 - 1 = 7 \quad S_r = Q_1 + Q_2 + Q_3 = 3556$$

$$S_1 = S_t - S_r = 458; f_1 = f_t - f_r = 7 - 3 = 4$$

$$F = (S_r/f_r)/(S_1/f_1) = 10.35 < F_{0.01}(3,4) = 16.69, \text{ but greater than } F_{0.05}(3,4) = 6.59$$

Thus, the regression Eq 3 of cooling rate at 300 °C is significant.

$F_1 = Q_1/(S_{1/}f_1) = 23.9 > F_{0.01}(1,4) = 21.2$, the coefficient of Concentration has a high level of significance.

$F_2 = Q_2/(S_{1/}f_1) = 6.99 < F_{0.05}(1,4) = 7.71$, but greater than $F_{0.10}(1,4) = 4.54$, i.e., bath temperature is related to cooling rate at 300 °C.

$F_3 = Q_3/(S_{1/}f_1) = 0.16 < F_{0.10}(1,4) = 4.54$; thus we can with a 90% confidence level say that agitation is not related to cooling rate at 300 °C. The agitation term in the regression Eq 3 may be caused by test errors and should be eliminated. Then we get the following regression equation for cooling rate at 300 °C.

$$Z = 125.7 - 3.7C - 0.5T \quad \text{(Eq 4)}$$

4.2 The Effect of Concentration and Its Control

According to Eq 2 and 4, C is related to maximum cooling rate and the cooling rate at 300 °C with a high level of significance. Therefore, the concentration of quenchants should be strictly controlled in practice.

As stated before, the cooling speed at 300 °C is very important for the tendency of quench distortion and cracking, and is mainly determined by concentration of PVP quenchants, as suggested by Eq 4. Theoretically, the parts made from steels with differing hardenability ranges should have different quenching; thus, a different concentration of PVP quenchants. In reality, this may be done to a limited extent with a small quenching tank; whereas, in many cases with a large quench tank only one concentration is normally used for quenching. The consideration of concentration is mainly used for parts with higher hardenability. For parts with less hardenability, regulating agitation and temperature, according to Eq 2, is necessary to match the need for faster quenching speed.

4.3 The Effect of Bath Temperature and Its Control

According to Eq 2, if the other elements remain unchanged,

$$\Delta C = 1.37/3.25 \times \Delta T \approx 0.42\Delta T$$

Normally, concentration must be maintained at ±2%. This is the same as keeping the temperature within 10 °C. Obviously, without the effective heat exchanger, this cannot be achieved.

The calculation of the heat exchanger requirement can be made as follows.

Given the temperature rise of quenchant is Δt,

$$\Delta t = \frac{GC_1(t_1 - t_1)}{\tau \cdot V \cdot C_2} \quad \text{(a)}$$

Δt, temperature rise (°C); G, the maximum quenched weight (kg); C_1, specific heat of parts (cal/g); τ, the interval between quenching (hour); t_1, parts' temperature of starting quenching (taking °C); t_1', parts' temperature of finishing quenching (taking 60 °C); V, volume of the tank (liters); C_2, specific heat of polymer close to water (cal/g, °C).

The amount of heat released during quenching:

$$Q = V \cdot \Delta t \cdot C\,(4.18 \times 10^3 \text{ J/h}) \quad \text{(b)}$$

According to Eq b, the cooling water volume, V_1, is as follows:

$$V_L = \frac{Q}{C_L\,(t'_L - t_L)} \times 10^{-3}\ (\text{m}^3/\text{h}) \quad \text{(c)}$$

Here, C_L is the specific heat of water (cal/g, °C), and t_L and t'_L are the inlet and outlet temperature (°C), respectively, taking the highest temperature in summer.

Based on the above calculation, choosing a suitable heat exchanger can be easy.

Table 2 Tank Volumes and Agitation Power

Volume of Tank, m^3	The Lowest Agitation Power, W/L: Quenching Oil	Polymer Quenchants	Water
<3	1.0	0.9	0.8
3-7.5	1.15	0.95	0.8
7.5-12	1.2	1.10	1.0
>12	1.40	1.20	1.0

4.4 The Effect of Agitation and Its Control

According to regression Eq 2 and 4, agitation has a highly significant influence over maximum cooling rate but little effect on the 300 °C cooling rate. Compared with concentration, the benefits of agitation adjustment is that faster agitation only increases the maximum cooling speed, but gives little increase to the cooling speed at 300 °C. Furthermore, better agitation allows for more uniform cooling and less distortion.[3] Thus, the effective and adjustable agitation is necessary for a PVP quenchant system. The power of agitators can be determined with Table 2.

5. Conclusions

The following conclusions can be made. The regression equations of maximum cooling speed and cooling rate at 300 °C for PVP based quenchants was made with the method of regression and orthogonal analysis:

Maximum Cooling Rate: $Y = 220 - 3.25C - 1.37T + 18.13V$

Cooling Rate (300 °C): $Z = 125.7 - 3.7C - 0.5T$

The first equation has a high level of significance and the second equation is less significant.

Concentration, temperature, and agitation all have a high level of significance to the maximum cooling rate. Concentration has a high level of significance, and temperature is related to the cooling rate of 300 °C. Finally, it is extremely important to strictly control concentration, bath temperature, and agitation in the application of PVP based polymer quenchants.

References

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