Technical procedure features of nickel dry-powder developer manufacturing by method of plasma-electrolytic dispergating

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Abstract. The following work describes means of nickel dry-powder developer manufacturing by plasma-electrolytic dispergating. For that purpose, a plasma-electrothermic device was developed that allows to manufacture dry-powders with wide range of parameters. Technical conditions for nickel dry-powder developer manufacturing by method of plasma-electrolytic dispergating were defined. By factorial design method regression equations were obtained, that make it possible to plan the process of dry-powders manufacturing with set average particle size and set manufacturing process rate. The described means allows to manufacture nicol dry-powder developer with a particle size of aprox. 100 nm.

1. Introduction

Dry-powder developers based on ferrous-ferric oxide, maghemite and other metal ferrites are widely used in mechanical engineering and aerospace industry through their unique properties. Magnetic features of the dry-powder developers make them effective in high-frequency magnetic conductors units and SHF cannons manufacturing; moreover, their relatively high conductivity and outstanding resistance to anodic dissolution make them valuable in the manufacturing of nonexpendable electrodes for electrochemical industry as well as of induced current cathodic protection and thermal protection, corrosion-resistant, radar-absorbent coating.

The modern technology extension offers a challenge of constant improving of dry-powder developers performance and technical features. Main disadvantage of dry-powder developers is heterogeneity of the chemical composition as a result of unconverted feedstock incorporation appearance. Solving this problem by manufacturing dry-powder developers through nicol plasma-electrolytic dispergating might be advantageous as the previously mentioned process shows simplicity of implementation, low cost and availability of basic materials, broad range of possibilities for automation, which promotes homogeneous structure of dry-powder developers.

There are various means and devices for electrical discharge generation under atmospheric pressure between metal and electrolytic electrodes [1, 2]. Complexity of such devices is a disadvantage. To manufacture nickel dry-powder developer through plasma-electrolytic dispergating a plasma-electrothermic device was developed; one of the features of this device is simplicity of its design.

In order to regulate the process of nickel dry-powder developers manufacturing, the device is provided with control system. This particular way or regulation is of a great interest, as detecting element is simple. A current sensor can be used as a detecting element. Current sensor might be installed on the out-port of discharge power supply unit, before the discharge chamber. To process the signals from this sensor a special control system should be developed, that would transform the current
sensor signals into drive engine control signals. By combinations of control actions on technic parameters it is possible to set various process modes to manufacture dry-powder developers and to provide their quality and necessary optimum properties. Such kind of control system implements inverse link between input parameter (current) and output parameter (electrode position).

2. Research methods
The device (Figures 1) works in a wide range of parameters of electrode gap \( l = 1 \div 100 \) mm, electrode diameter \( d = 3 \div 15 \) mm, discharge current \( I = 0,01 \div 10 \) A, discharge voltage \( U = 0,1 \div 4 \) kV, volume electrolyte velocity \( G = 15 \div 34 \) g/s, electrolyte pump velocity \( \nu = 0,02 \div 0,05 \) m/s.

![Figure 1. Flow chart of plasma device (PD) for nickel dry-powder developer manufacturing under atmospheric pressure](image)

1 - powdered metal electrode; 2 - manufactured powder; 3 - continuous electrolyte flow bath; 4 - electrode holder; 5 - step motor; 6 - screw-type shaft; 7 - tank with heat exchange unit; 8 - electrolyte transfer pump; 9 - isolation valves; 10 - air compressor; 11 - current sensor; 12 - PD control system.

With suggested means the nickel dry-powder developer manufacturing was achieved through discharge allowance between flow electrolytic anode from utility water and nickel electrode Mk N-0 (H-0 ru); setting the current within \( I = 0,6 \div 1,3 \) A, voltage \( U = 400 \div 800 \) V under atmospheric pressure, electrode gap \( l = 1 \div 30 \) mm. Dry-powder developer manufactured through method of plasma-electrolytic dispergating was collected in container and purged with ethanol. Ethanol overflow was removed, the powder was air-cured up to its constant weight. After that the powder weight was measured with microchemical balance Ohaus Adventurer pro AV2264.

Under atmospheric pressure, \( jc = 1 \) A/cm\(^2\); \( U = 600 \) V, powder production rate is 14 g/h. Decrease of electrode diameter leads to increase of dry-powder developer production rate. Microscopic examination with light microscope Mk Micromed MET, as well as electron microscope Zeiss EVO-40 showed that nickel powder particles have spherical shape with diameter within 0,1 to 1,5 \( \mu m \). Consequently, depending on the operating mode and conditions it is possible to manufacture nickel dry-powder developer of particular degree of dispersion; in comparison to another know method [3] the outcome is as much as 6 times more [4].

3. Results and discussions
By factorial design method coefficients of regression equation were calculated and checked, where linear effects and interaction effects were taken into account. Based on preliminary investigations were chosen main factors of the process that affect manufacturing rate of dry-powder developer as well as degree of dispersion the most [5, 6]: \( j \) – current intensity on anode, A/cm\(^2\); \( P \) – pressure, Pa; \( L \) – electrode gap, mm.

As output parameters the following were picked: \( P \) – dry-powder production rate, g/h; \( D \) – dry-powder particles average diameter, \( \mu m \).
According to the factorial design method equations of regression were calculated for average size of particles and for production rate using nickel N-O as metal cathode.

Table 1. Results of the factorial design method equations

| Set of tests | Average particles size $D_{av}$, µm | Dispersion assessment $S_D^2$ | Production rate $P_{av}$, g/h | Dispersion assessment $S_P^2$ |
|--------------|------------------------------------|-------------------------------|-------------------------------|-------------------------------|
| 1            | 0.1000                             | 0.0011                        | 8.6333                        | 0.2022                        |
| 2            | 0.3000                             | 0.0066                        | 7.5000                        | 0.1666                        |
| 3            | 0.2000                             | 0.0066                        | 14.0000                       | 2.0000                        |
| 4            | 0.4000                             | 0.0066                        | 8.3333                        | 1.5555                        |
| 5            | 0.5666                             | 0.0022                        | 13.0000                       | 0.6666                        |
| 6            | 0.6333                             | 0.0022                        | 10.6666                       | 0.2222                        |
| 7            | 0.5333                             | 0.0022                        | 8.0000                        | 0.6666                        |
| 8            | 0.4333                             | 0.0155                        | 8.9000                        | 0.4866                        |

Coefficients calculations for equations of regression:

$$b_0 = \frac{1}{N} \sum_{j=1}^{N} y_j$$ (1)

$$b_i = \frac{1}{N} \sum_{j=1}^{N} x_{ij} \cdot y_j$$ (2)

$$b_{i0} = \frac{1}{N} \sum_{j=1}^{N} x_{ij} \cdot y_j$$ (3)

$$b_{iun} = \frac{1}{N} \sum_{j=1}^{N} x_{ij} \cdot x_{jm} \cdot y_j$$ (4)

Conforming to it (1) – (4), obtained,

for average size of particles: $b_{00} = 0.3958$; $b_{01} = 0.1458$; $b_{02} = -0.0041$; $b_{03} = 0.0458$; $b_{012} = -0.0541$; $b_{013} = -0.0541$; $b_{023} = -0.0208$; $b_{0123} = -0.0208$.

for dry-powder manufacturing production rate: $b_{00} = 9.8792$; $b_{10} = 0.2625$; $b_{20} = -0.0708$; $b_{30} = 1.0291$; $b_{11} = -1.6208$; $b_{12} = 0.6708$; $b_{13} = -0.1625$; $b_{23} = 0.9708$.

Considering orthogonality of multifactor experiment, evaluation of dispersion degree is the same for each coefficient calculation.

Subsequently:

$$S_{(b_i)} = \frac{S_{(p)}}{\sqrt{8}} = \frac{0.0254}{\sqrt{8}} = 0.0090$$ (5)

$$S_{(b_{i0})} = \frac{S_{(p)}}{\sqrt{8}} = \frac{0.2887}{\sqrt{8}} = 0.1020$$ (6)

Test of significance of equation of regression coefficients was conducted through Student’s coefficient, eq. 2.12.

Equation coefficient is to be considered significant if the following condition works:

$$|b_i| \geq T \cdot S_{(b_i)}$$
Conforming to it and (5) – (6):

\[ |b_D| \geq 2,12 \cdot 0,0090 = 0,0190 \]
\[ |b_P| \geq 2,12 \cdot 0,1020 = 0,2163 \]

Therefore, coefficients \( b_D \) are considered as insignificant for equation of dependence regression of average particles size. Coefficients \( b_P \) and \( b_{P23} \) are considered insignificant for equation of dependence regression of dry-powder manufacturing production rate.

In such case equations of regression are written as:

For average particles size:

\[ D_{Ni} = 0,395 + 0,145 \cdot x_1 + 0,045 \cdot x_3 - 0,054 \cdot x_1 \cdot x_2 - 0,054 \cdot x_1 \cdot x_3 - 0,02 \cdot x_2 \cdot x_3 - 0,02 \cdot x_1 \cdot x_2 \cdot x_3 \]

For dry-powder manufacturing production rate:

\[ P_{Ni} = 9,879 + 0,262 \cdot x_1 - 1,029 \cdot x_3 - 1,62 \cdot x_1 \cdot x_2 + 0,67 \cdot x_1 \cdot x_3 + 0,97 \cdot x_1 \cdot x_2 \cdot x_3 \]

4. Conclusion

With help of multifactor experiment method equations of regression were deduced. The equations allow to pick the necessary parameters of processing for nickel dry-powder developer manufacturing with required particles size and appropriate production rate. The deduced regression equations can be used to calculate the operation modes for plasma devices with both flow-type and non-flow-type electrodes in a wide range of current and discharge voltage, pressure and electrode gap.

References

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