Development of a flaw detection material for the magnetic particle method

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Abstract. The issues of increasing the effectiveness of the magnetic particle method of nondestructive testing by using a new flaw detection material is considered in the paper. The requirements for flaw detection materials are determined, which ensure the effectiveness of the inspection method. A new flaw detection material – magnetic fluids from iron-containing waste products – has been developed.

1. Introduction

The magnetic particle method of nondestructive testing is based on the analysis of magnetic scattering fields using a ferromagnetic material as an indicator. This method is used to control the surface of products made of ferromagnetic materials and allows revealing surface and subsurface defects.

Currently, ferromagnetic powders, magnetic suspensions and magnetohumed pastes are used as an indicator (a flaw detection material). The national, interstate standards, industry regulations, rules, recommendations and guidelines, as well as the industry standards (STO) and technical specifications (TU) establish the requirements for the flaw detection materials used.

The national standards (GOST R) provide general requirements for flaw detection materials. In order to define the specific values of the material characteristics, an analysis of the industry requirements is necessary.

2. Main part

The analysis of regulatory documents [1-6], regulating the magnetic particle inspection method, allowed formulating universal characteristics of the flaw detection materials used. Table 1 provides the characteristics of the flaw detection material that meets the requirements for monitoring at hazardous production facilities, main oil pipelines, gas pipelines, as well as in the railway and automotive sectors.

At present, magnetic powders on the basis of which suspensions are prepared are widely used in industrial enterprises.
However, the characteristics given in Table 1 do not provide the method efficiency required by the enterprises, especially in terms of the stability of the material, since the maximum stability of flaw detection materials (suspension) is usually 120 hours. Therefore, it must be manufactured and tested for suitability for each new inspection procedure. The enterprises also have high requirements to the sensitivity level of the method, which depends on the average particle size of the ferromagnetic material, namely, the smaller the particle size, the higher the detecting ability of the flaw detection material.

**Table 1.** Characteristics of flaw detection materials for the magnetic particle inspection method.

| Item No. | Indicator name                     | Indicator characteristics (value)                                      |
|----------|------------------------------------|----------------------------------------------------------------------|
| 1        | Appearance                         | Black, colored, luminescent magnetic powders in dry form or as part of suspensions |
| 2        | Color                              | Maximum contrast in relation to the color of the surface monitored   |
| 3        | Detecting ability, above, %        | Applied magnetic field method – 98, Remanent magnetization method – 95 |
| 4        | Mass fraction of the main substance, % | For dry powders – 80-85; For use in suspensions – 20-30            |
| 5        | Concentration of hydrogen ions (pH), above | 8                                                               |
| 6        | Average particle size, μm, below   | Application by dry method – 150 μm; Use in suspensions – 50 μm       |
| 7        | Viscosity, m²/s, below             | 36·10⁻⁶ (36 cS)                                                      |
| 8        | Dynamic viscosity at (20 ± 2) °C, below, MPa·s | 5                                                               |
| 9        | Concentration of powder in suspension, g/l | (25 ± 5) – for black or colored (non-luminescent) powder; (4 ± 1) – for luminescent powder |
| 10       | Suspension stability, above, h     | 120                                                                 |
| 11       | Heat resistance                    | Materials should not degrade after 5 minutes of heating to the maximum operating temperature |
| 12       | Corrosivity                        | Does not cause corrosion                                            |
| 13       | Foaming                            | Resistance to foaming                                               |
| 14       | Sulfur and halogen content         | The sulfur content should be less than (200 ± 10) ppm; The halogen content should be less than (200 ± 10) ppm (halogens mean chlorine and fluorine). |

In this connection, it is necessary to develop a material that meets the requirements of various industries and is a multifunctional one, and also increases the effectiveness of the method by improving the stability and average particle size.

Magnetic fluid is a material that makes it possible to solve the problem of improving the method efficiency. Due to an unusual combination of the properties of magnetic materials, liquids and colloidal solutions magnetic fluids (MF) are promising materials and can find use in magnetic particle inspection [7].

Magnetic fluid is a stable ultrafine colloidal system of highly dispersed particles of magnetic material (ferro- or ferrimagnetic substances) with an average size of 5 to 50 nm dispersed in various liquids and performing intensive Brownian motion stabilized by surfactants that is capable of
interacting with a magnetic field and behaves like a homogeneous liquid in many respects [8]. However, the use of magnetic liquids made from pure components (reactive raw materials) can be limited due to their high cost; therefore, a technology for producing magnetic liquids from iron-containing waste products has been developed [9-10].

Three components are necessary to obtain magnetic fluid, namely: magnetic particles of colloidal dimensions (magnetite), a liquid base and a stabilizer, which prevents the colloid particles from sticking together. Each component must meet certain requirements, only this condition makes it possible to obtain a magnetic fluid suitable for use in a particular direction.

It is known that the interaction of aqueous solutions of salts of bivalent and ferric iron and their combined precipitation produces the formation of magnetite in the form of highly dispersed particles

\[ \text{Fe}^{2+} + 2\text{Fe}^{3+} + 8\text{OH}^- = \text{Fe}_3\text{O}_4 \downarrow + 4\text{H}_2\text{O}. \]  

(1)

In the paper presented, we used iron-containing wastes as sources of bivalent and ferric iron, the properties of which are provided in Table 2.

**Table 2. Characteristics of waste.**

| Indicator name, % | 1st group | 2nd group | 3rd group |
|------------------|-----------|-----------|-----------|
| Waste of OJSC "Severstal" | Waste of OJSC "Severstal" | Waste of Olenegorsky MPP | Waste of OJSC "Severstal" |
| FeO | 76.96±0.77 | 76.96±0.77 | 76.96±0.77 |
| CaO | 2.14±1.34 | 2.14±1.34 | 2.14±1.34 |
| Na2O | 0.10±0.07 | 0.10±0.07 | 0.10±0.07 |
| ZnO | 3.07±0.76 | 3.07±0.76 | 3.07±0.76 |
| C | 0.41±0.05 | 0.41±0.05 | 0.41±0.05 |
| CuO | 0.22±0.01 | 0.22±0.01 | 0.22±0.01 |
| P2O5 | 0.15±0.008 | 0.15±0.008 | 0.15±0.008 |
| SiO2 | 1.54±0.35 | 1.54±0.35 | 1.54±0.35 |
| Cr total | – | 1.84±0.87 | – |
| NiO | traces | 0.15±0.1 | 0.15±0.1 |
| H2SO4 | – | – | 2.9±0.02 |
| TiO2 | 0.11±0.01 | – | – |
| Al2O3 | 1.23±0.03 | – | – |
| MnO | 0.10±0.01 | – | – |
| MgO | 0.98±0.01 | – | – |
| K2O | 0.36±0.02 | – | – |
| Ba | 2.12±0.2 | – | – |
| SO3 | 3.41±1.1 | – | – |
| Mass fraction of substances insoluble in HCl | 10.01±4.6 | 10.01±4.6 | 10.01±4.6 |

These wastes are classified into 3 groups:

- Wastes containing ions of predominantly ferric iron;
- Wastes containing ions of bivalent and ferric iron;
- Wastes containing ions of predominantly bivalent iron.

The technology of obtaining magnetic fluids includes two main stages:

1. Obtaining highly dispersed magnetite particles (dispersed MF phase);
2. Stabilization of magnetite in the carrier fluid using surfactants, which prevent the aggregation of magnetite particles in the carrier fluid and ensure the MF stability.

Oleic acid was used to stabilize magnetite in the carrier fluid. Water, kerosene or various oils can be used as a liquid base. A block diagram of obtaining magnetic fluids from iron-containing waste products is presented in Figure 1.

![Diagram showing the process of obtaining magnetic fluids from iron-containing waste products.](image)

**Figure 1.** Scheme of obtaining a magnetic fluid.

The size of the obtained magnetite particles (Figure 2) is determined using the Nanotrac laser particle analyzer. As can be seen from Figure 2, the average particle size is in the range of 10 to 20 nm. The characteristics of the magnetic liquids obtained are given in Table 3.

![Chart showing the dependence of increasing total and percentage of fractions on the size of magnetite particles obtained from galvanic slime.](image)

**Figure 2.** Chart of the dependence of increasing total and percentage of fractions on the size of magnetite particles obtained from galvanic slime.

Table 3 shows that magnetic fluids obtained from iron-containing waste meet the requirements specified in Table 1.
The magnetic liquids obtained were tested as a flaw detection material for non-destructive testing of welded joints by a magnetic particle method. The investigations were carried out on single-pass welded joints made of steel 20. The control was carried out by the applied magnetic field method.

Table 3. Characteristics of magnetic liquids.

| Indicator name                            | Indicator characteristics (value) |
|-------------------------------------------|-----------------------------------|
| Appearance                                | Dark brown liquid                 |
| Concentration of hydrogen ions (pH),     | 8-9                               |
| Average particle size, nm                 | 10-20                             |
| Viscosity, m²/s, below                    | $36 \cdot 10^{-6}$ (36 cS)       |
| Dynamic viscosity at (20 ± 2) °C, below   | 5 mPa·s                           |
| Stability                                 | More than 210 days                |
| Corrosivity                               | Does not cause corrosion          |
| Foaming                                   | Resistance to foaming             |
| Saturation magnetization, kA/m            | 10-20                             |
| Volume fraction of magnetite, %           | 2-8                               |

It is shown that the use of magnetic liquids increases the efficiency of magnetic particle inspection:

1. the efficiency of detecting defects increases due to the reduction of the size of magnetic particles from 5-70 μm to 10-20 nm;
2. the stability of the flaw detection material increases (more than 210 days);
3. The cost of magnetic particle inspection is reduced.

3. Conclusions

Thus, the results of the research allowed for the creation of a new flaw detection material based on industrial iron-containing waste. The technology developed contributes to the improvement of the quality of magnetic particle inspection, and is also a method of disposal of toxic industrial wastes, negatively impacting the environment and human health.

References

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