EFFECT OF THE PARTICLE SIZE OF STARTING POWDERS ON THE RESULTS OF SPECTRAL ANALYSIS OF SINTERED MATERIALS

COMMUNICATION II

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Dependence of the Spectrally Determined Concentration of an Addition on the Particle Size of Its Powder

To determine how the intensity of spectral lines of an addition is affected by the latter's powder particle size, tests were carried out on two sets of specimens of the following compositions:

Set I - No. 1) 60.9% Ni + 39.1% Fe (iron powder particle size 150-100 μ); No. 2) 61.2% Ni + 38.8% Fe (iron powder particle size < 40 μ);

Set II - No. 3) 95.147% Ni + 4.867% Fe (150-100 μ); No. 4) 95.1% Ni + 4.9% Fe (< 40 μ).

The particle size of the matrix powder and the specimen porosity were maintained constant. The iron contents quoted above were determined by chemical analysis. Disk-shaped specimens, 50 mm in diameter and 5 mm thick, were pressed to a porosity of 20% and sintered for 2 h in a hydrogen atmosphere at a temperature of 1000°C. During the excitation of a spectrum with a high-frequency spark generator, 8.28 mg of material was transferred into the discharge cloud in 200 min from the surface of specimen No. 1 and 20.96 mg from the surface of specimen No. 2; thus, reducing the particles size of the addition from 150-100 to < 40 μ increased the amount of material transferred almost 2.5 times. Together with the determination of yields of material, spectra of these specimens were photographed, after presparking for 15 sec, on a Type 1 plate with 45 seconds' exposure, using a permanent magnesium electrode of 1.6 mm diameter. The excitation conditions were the same as in [1]. This made it possible to study changes in the absolute and relative intensities of the spectral lines of nickel (the matrix of the material) and the spectral lines of iron (the element added to the material). The mean absolute and relative intensities of spectral lines, obtained for each specimen from 50 spectra are listed in Table 1. Analysis was performed against the Fe 2599.4 Å–Ni 2583.99 Å and Ni 2416.14 Å–Fe 2417.866 Å pairs of lines. In Table 1, S is the blackening of a spectral lines and ΔS is the difference in the degree of blackening of an analytical pair of spectral lines.

Photographic plates revealed the existence of two systems of spectra. Spectra obtained by photographing specimen No. 1 proved to be much weaker than those yielded by specimen No. 2. Photometric

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Composition</th>
<th>Yield of material in 200 min mg</th>
<th>$S_{Fe}$</th>
<th>$S_{Ni}$</th>
<th>$ΔS$ ($S_{Fe}$–$S_{Ni}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>60.9% Ni + 39.1% Fe (150–100 μ)</td>
<td>8.28</td>
<td>140.0</td>
<td>65.0</td>
<td>+75.0</td>
</tr>
<tr>
<td>2</td>
<td>61.2% Ni + 38.8% Fe (&lt;40 μ)</td>
<td>20.96</td>
<td>177.0</td>
<td>83.0</td>
<td>+94.0</td>
</tr>
</tbody>
</table>

Fig. 1. Calibration curves for iron with high-frequency spark excitation of spectrum. Iron powder particle size: 1) < 40; 2) 100-150; 3) 250-450 μ.

Fig. 2. Calibration curves for iron with condensed high-frequency spark excitation of spectrum. Designations as for Fig. 1.

data (see Table 1) show that the absolute intensity of the spectral lines of Fe and Ni is less in spectra of specimen No. 1 than in those of specimen No. 2. The ratio of the yields of iron and nickel in the overall amount of material transported into the discharge plasma depends on the particle sizes of the powders. Hence, compared with the surface of specimen No. 1, the amount of iron transported from the surface of specimen No. 2 exceeded that of nickel. As a result, the relative intensity of the spectral lines of iron was greater in spectra of specimen No. 2 than in those of specimen No. 1.

Thus, when the particle size of the addition powder is increased (the particle size of the matrix powder and the porosity of the material being maintained constant), both the absolute and the relative intensity of the spectral lines of the addition decrease; conversely, with decreasing particle size of the addition powder, the absolute and the relative intensity of these lines increase. This regularity is observed in the spectral analysis of powder metallurgical materials in which the amount of an addition element being analyzed ranges from negligible to some 40-50%.

Using sets of reference standards prepared by powder metallurgy techniques, calibration curves were constructed for iron. The compositions of the reference standards are listed in Table 2. The standards were produced from electrolytic nickel powder with various amounts of iron. The particle sizes of the iron powder are shown in Table 2. The reference standards, in the form of cylinders 15 mm in diameter and 10 mm high, were pressed to 20% porosity and sintered for 2 h in a dried hydrogen atmosphere at 1000°C. The iron contents presented in Table 2 were obtained by chemical analysis. Mean relative error in operation with a high-frequency spark generator is ±4% for Fe.

Calibration curves for iron were constructed using mean results of ten measurements. Such measurements consisting of ten spectrum recordings were frequently repeated and found to give identical results. Calibration curves plotted with the aid of standard sets are illustrated in Fig. 1. The effect of the particle size of iron powder manifests itself in a shift of calibration curves. Our calculations show that the deviation of comparable results is much greater than 3σ (where σ is the root-mean-square error in iron content determination with the light source employed); it therefore follows from metrological considerations that the influence of the particle size of the addition powder may be taken as quite conclusively established.

The influence of the particle size of iron powder on analytical results may be evaluated from the positions of calibration curves 1, 2 and 3 (Fig. 1). It follows from this figure that there is a systematic deviation of about 20-25% (relative) between calibration curves 1 and 2, about 65-70% between calibration curves 1 and 3, and about 40% between calibration curves 2 and 3. Clearly, the position of a calibration curve will vary depending on the element being determined and its concentration limit. For this reason, a systematic deviation of a calibration curve will also represent a different magnitude of relative error in analysis.