Phase Composition Using a Variable Magnetic Field

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(Received on July 19, 2013; accepted on October 8, 2013)

In the present work the application of the variable magnetic field to investigate the composition of phases present in the alloys is presented. In order to make a study in the variable magnetic field a prototype apparatus was performed. The magnetic leakage of the alloy \( k_s \) was measured in the reversible range of magnetic field.

Experimental tests were conducted on samples of Fe–Cu alloys have contained the different volume fraction of phases components. Comparison of volume fraction of the solid solution \( \alpha_{Fe} \) determined by weight and magnetic methods was performed. The linear dependence between the magnetic coupling and the volume fraction of phases components was observed. This observation can be used to estimate the quantitative phase composition in a biphase alloys. It has been shown that in the reversible range of the magnetic field, the magnetic leakage of the alloy is equal to the sum of products of volumetric proportions of individual phase components and their magnetic leakage.

In the present work a new method for quantitatively assessing the phase composition using a variable magnetic field is presented which is universal for two-phase alloys if the two phases have different magnetic leakage. This can be particularly useful for assessing the proportion of austenite and retained austenite in heat-treated alloys of iron and carbon.

KEY WORDS: metallic alloys; phase composition; variable magnetic field; ferromagnetic properties.

1. Introduction

The qualitative and quantitative assessment of phase composition is of major importance in specifying and optimising mechanical, physical and usage characteristics of metals and their alloys. This assessment is performed both during research work and in industrial production conditions.

Although a series of methods for determining phase composition exists, none can be considered to be fully universal. In many cases, the results of an examination carried out by one method must be confirmed using other methods. The methods most frequently used to determine phase composition are as follows: the metallographic one, X-ray phase analysis and the chemical isolate (weight) method. However, other methods are also used, namely: the magnetic, dilatometric, ultrasound, calorimetric, neutron diffraction, Mössbauer spectroscopy and electrical resistance methods.

The traditional magnetic method of quantitatively determining phase composition makes use of a constant magnetic field. This method is based on the linear dependence of the magnetisation or the magnetic induction of examined alloys (determined in magnetic saturation conditions) on the volumetric proportions of particular phase components.

In metallographic examinations, a variable magnetic field is used mainly to assess the kinetics of phase transformations, identifying the temperature at which they occur, and also for flaw detection. Publications on structural examinations performed using a variable magnetic field describe a qualitative and quantitative influence of the phase composition on the results obtained, but a relationship between these results and the volumetric proportion of phase components is not unambiguously identified. The authors believe that for technical alloys, because of the multitude of variable, interdependent factors, no detailed data on the type of this dependence can be presented.

The purpose of this study is to identify the relationship between the magnetic leakage of the alloy \( k_s \) and the volumetric proportion of the phase component with ferromagnetic properties in this alloy. This research was carried out on model samples produced of an alloy of iron and copper. The results of examinations of two-phase alloys containing phase components of different magnetic characteristics carried out in a variable magnetic field can be used for the quantitative assessment of phase composition.

2. Research Methods

Experimental examinations were conducted on model samples made of iron, copper, and alloys of these metals. Iron and copper alloys are two-phase alloys. The components of these alloys are solid solutions \( \alpha_{Fe} \) and \( \alpha_{Cu} \), in which the solubility of copper and iron, respectively, is close to zero at the ambient temperature. These solutions differ in their magnetic properties: \( \alpha_{Fe} \) is ferromagnetic while \( \alpha_{Cu} \) is diamagnetic. The structural components of iron and cop-
per alloys may, depending on the composition, consist of solid solutions of $\alpha_{Cu}$ and $\alpha_{Fe}$ as well as the eutectoid mixture $\alpha_{Cu} + \alpha_{Fe}$.

In order to obtain samples with various proportions of phase components, melts were produced in a Balzers VSG-02 vacuum furnace. The charge comprised technically pure iron (9.97% Fe, 0.1% Mn, 0.05% S, 0.05% P and others) and electrolytic copper (99.9% Cu). After the charge materials were melted and superheated to 1600°C, the alloys were cast into a metal mould previously heated to 500°C. Trial castings in the shape of $\phi 30 \times 100$ mm cylinders were subjected to stress-relief annealing at the temperature of 650°C for two hours and slowly cooled down with the furnace. After the heat treatment, samples having the shape of $\phi 20 \times 20$ mm cylinders were taken from the castings for metallographic and magnetic examinations. The symbols of the samples and the weight proportions of the charge prepared for melting are presented in Table 1.

When ferromagnetic alloys are exposed to an external magnetic field, they are magnetized. The change of the magnetization of the alloy $J_s$ relative to the intensity of the magnetic field $H$ is called the initial magnetization curve. This relationship is a complex function along which five characteristic ranges are distinguished. Examinations by the magnetic method generally applied to quantitatively assess phase composition are carried out with a constant magnetic field, they are magnetized. The change of the magnetization of the alloy $J_s$ achieves the maximum value if $H$ equals $H_k$. This study makes use of the first range on the initial magnetization curve, which is also referred to as the range of weak magnetic fields in the literature. Ferromagnetics are magnetized in this range by the reversible movement of domain walls, and the process of changing the direction of magnetization of an alloy placed in a variable magnetic field is hysteresis-free in character.\(^5\)

The magnetic leakage of the alloy $k_s$ is determined using a prototype measuring apparatus relating to the relationship (1). When determining magnetic leakage $k_s$, the following boundary conditions were assumed: $k_s = 0$ – no leakage, $k_s = 1$ – total magnetic leakage.

$$k_s = 1 - a \cdot \frac{V_n}{V_o} \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \ quad
micropores) as well as the magnetic leakage values \( k_s \) of the studied samples. Figure 3 shows the change of the magnetic leakage \( k_s \) as a function of the volumetric proportion of the phase component with ferromagnetic properties \( V_{\alpha \text{Fe}} \) for samples numbered I–VI (Table 2).

The linear dependency of the magnetic leakage \( k_s \) on the volumetric proportion of the phase component \( \alpha \text{Fe} \) (Fig. 3, correlation coefficient close to 1) supports the conclusion that this parameter can be used to quantitatively assess the phase composition of the examined alloy.

Based on the measured values of magnetic leakage \( k_s \), the volumetric proportion of the phase with ferromagnetic properties \( V_{\alpha \text{Fe}} \) can be calculated from Eq. (3):

\[
V_{\alpha \text{Fe}} = \frac{k_{\text{Fe}} - k_s}{k_{\text{Fe}} - k_{\text{Cu}}} \times 100\% \tag{3}
\]

where:

- \( k_{\text{Fe}} \) – magnetic leakage for a sample made of an iron and copper alloy;
- \( k_{\text{Cu}} \) – magnetic leakage for a sample containing 100% of the \( \alpha \text{Cu} \) phase;
- \( V_{\alpha \text{Fe}} \) – volumetric proportion of the \( \alpha \text{Fe} \) phase [%].

Table 3 and Fig. 4 present the volumetric proportion of the solid solution \( \alpha \text{Fe} \) for samples II–V determined by the weight method (based on the weight proportions of charge materials) and by the magnetic method with a variable field. The \( k_{\text{Fe}} \) and \( k_{\text{Cu}} \) symbols found in Eq. (3) represent the values of magnetic leakage determined for samples number I and VI.

Both the correlation coefficient \( R^2 = 0.9998 \) (Fig. 3), and the high level of consistency in assessing the volumetric

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**Table 2.** Phase volume fractions of components \( V_{\alpha \text{Fe}} \) and \( V_{n} \) and values of magnetic leakage \( k_s \), for samples I ÷ VI.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>( V_{\alpha \text{Fe}} ) [%]</th>
<th>( V_{n} ) [%]</th>
<th>( k_s )</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>100.0</td>
<td>0.0</td>
<td>0.197</td>
</tr>
<tr>
<td>II</td>
<td>88.8</td>
<td>11.2</td>
<td>0.267</td>
</tr>
<tr>
<td>III</td>
<td>73.6</td>
<td>26.4</td>
<td>0.348</td>
</tr>
<tr>
<td>IV</td>
<td>49.2</td>
<td>50.8</td>
<td>0.494</td>
</tr>
<tr>
<td>V</td>
<td>24.7</td>
<td>75.3</td>
<td>0.638</td>
</tr>
<tr>
<td>VI</td>
<td>0.0</td>
<td>100.0</td>
<td>0.777</td>
</tr>
</tbody>
</table>

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**Table 3.** Volumetric fraction of the solid solution \( \alpha \text{Fe} \) determined by the weight method and by the magnetic method with a variable magnetic field.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>( V_{\alpha \text{Fe}} ), [%] weight method</th>
<th>( V_{\alpha \text{Fe}} ), [%] magnetic method</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>88.8</td>
<td>87.9</td>
</tr>
<tr>
<td>III</td>
<td>73.6</td>
<td>74.0</td>
</tr>
<tr>
<td>IV</td>
<td>49.2</td>
<td>48.8</td>
</tr>
<tr>
<td>V</td>
<td>24.7</td>
<td>24.0</td>
</tr>
</tbody>
</table>

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**Fig. 2.** Selected images of the microstructure of samples marked with symbols; a) – IV and b) – V, Etched with 1% of HNO₃ in ethanol.

**Fig. 3.** Magnetic leakage \( k_s \) for samples of iron and cooper alloys I ÷ VI as a function of the volume fraction of the phase \( \alpha \text{Fe} \).

**Fig. 4.** Comparison of volume fraction of the solid solution \( \alpha \text{Fe} \) determined by weight and magnetic methods.
share (the absolute error does not exceed 0.9%, Table 3), allow Eq. (3) to be written in the form of Eqs. (4) and (5):

\[ k_s = \sum V_{\alpha i} \cdot k_{\alpha i} \] .............................. (4)

\[ \Sigma V_{\alpha i} = 1 \] .............................. (5)

where:

- \( k_s \) – magnetic leakage of the alloy;
- \( k_{\alpha i} \) – magnetic leakage of the \( i \)th phase component;
- \( V_{\alpha i} \) – volumetric proportion of the \( i \)th phase.

Relationships (4 and 5) indicate that in the reversible range of the magnetic field, the magnetic leakage of the alloy is equal to the sum of products of volumetric proportions of individual phase components and their magnetic leakage.

The above relationship can be considered to form the basis of a new method for quantitatively assessing the phase composition using a variable magnetic field. This method is universal for two-phase alloys if the two phases have different magnetic leakage. This can be particularly useful for assessing the proportion of austenite and retained austenite in heat-treated alloys of iron and carbon. A microstructure of steels and cast steels after quenching consists of martensite, which is a ferromagnetic material and of retained austenite of paramagnetic properties. A volume fraction of retained austenite is - in this case - one of the criteria of the properly performed heat treatment. An excessive fraction of unstable phase component, such as austenite is the reason of increasing internal stresses and a loss of dimensional stability of quenched parts of machines and devices. Due to a diphasic microstructure composition as well as different magnetic properties of both phases the proposed method can be - in such case - specially suitable. For alloys made up of more than two phases, this method used on its own does not allow the volumetric proportions of the phase composition to be unambiguously determined. Yet regardless of the discussed limitations to its use, it has important advantages over other methods, namely: it assesses the proportion with good precision, the measurements are quick and easy and the cost of examinations is low. Because the measurement is quick and easy, it can be used not only in physical metallurgy labs, but also on production lines for the non-destructive control of heat-treated alloys containing phase components of different magnetic properties.11)

4. Conclusions

(1) In the reversible range of the magnetic field, the magnetic leakage \( k_s \) for an iron and copper alloy is linearly dependent on the volumetric proportions of phase components found in this alloy.

(2) For two-phase alloys of varied magnetic properties, relationships (4) and (5) can be considered to form the basis of a new method for quantitatively assessing the phase composition using a variable magnetic field.

(3) The absolute error in the assessment of volumetric proportions by the proposed method does not exceed 0.9%.

(4) For alloys consisting of more than two phases, the \( k_s \) parameter can be used as a supporting method for a separation based on the proportion of two phases.

REFERENCES