A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 53 2008 Issue 1

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IDENTIFICATION OF VOLUMETRIC PHASE SHARE ON THE BASIS OF THE RELATIVE MAGNETIC LOSS OF AN ALLOY

OCENA UDZIAŁU OBJĘTOŚCIOWEGO FAZ NA PODSTAWIE WZGLĘDNEJ STRATNOŚCI MAGNETYCZNEJ STOPU

The text presents the results of research aimed at determining the influence of a volumetric share of the phases constituting the microstructure of metal alloys on their relative magnetic loss. The research aimed at identifying the above-mentioned relation was carried out on model samples of iron and copper alloys with the univocally determined volumetric share of phases with ferromagnetic and non-ferromagnetic properties. The relative magnetic loss of the examined alloys was determined by means of a prototype device for magnetic research, designed by the author. The presented results, confirmed by examinations of engineering iron alloys, allow the following relationship to be formulated: in multi-phase alloys, the minimum relative magnetic loss of an alloy is equal to the sum of products of relative magnetic losses of particular phases and their volumetric shares. The above relationship allows one in particular to carry out comparative assessment of the volumetric share of the ferromagnetic and non-ferromagnetic phases, that constitute the microstructure of metal alloys. It can also be used to control on the line the correctness of heat treatment of iron alloys, especially with respect to the volumetric share taken by the retained austenite.
Keywords: phase analysis, ferromagnetic, non-ferromagnetic

W referacie przedstawiono wyniki badań zmierzających do określenia wpływu udziału objętościowego faz wchodzących w skład mikrostruktury stopów metali na ich względną stratność magnetyczną. Badania zmierzające do określenia powyższej zależności wykonano na modelowych próbkach o jednoznacznie określonych udziałach objętościowych faz o właściwościach ferromagnetycznych i nieferromagnetycznych przygotowanych ze stopów żelaza i miedzi. Względną stratność magnetyczną badanych stopów określono za pomocą prototypowego urządzenia do badań magnetycznych autorskiego projektu. Przedstawione wyniki potwierdzone badaniami dla technicznych stopów żelaza pozwoliły na sformułowanie zależności, że w stopach wielofazowych minimalna względna stratność magnetyczna stopu jest równa sumie iloczynów względnych minimalnych stratności magnetycznych poszczególnych faz i ich udziałów objętościowych. Powyższa zależność pozwala w szczególności na porównawczą ocenę udziału objętościowego faz o właściwościach ferromagnetycznych i nieferromagnetycznych wchodzących w skład mikrostruktury stopów metali. Może być również zastosowana do bieżącej kontroli prawidłowości obróbki cieplnej stopów żelaza, zwłaszcza pod względem oceny udziału objętościowego austenitu szczątkowego.

1. Research objectives and methodology

The volumetric share of phase components in alloy microstructure is one of their basic parameters, decisive for mechanical, physical and performance properties of metal alloys, including casting alloys. The parameter often indicates, whether heat treatment applied to iron alloys has been carried out correctly. It is used in particular to assess the volumetric share of austenite and retained austenite in iron alloys. The volumetric phase share is most commonly assessed by the metallographic method and the x-ray phase analysis [1–3]. The dilatometric and magnetic methods are also used for the purpose [1–4].

In the case of multi-phase alloys, the classical magnetic method is based on the linear dependence of magnetization intensity – determined in the conditions of magnetic saturation – on the volumetric share of particular phase components [5]. The framework of the published research comprised the examination of the relative magnetic loss in alloys, where the volumetric shares of the phases with ferromagnetic and non-ferromagnetic properties are determined unequivocally. The objective of the assumed research methodology is to develop a magnetic method, which allows the volumetric phase share to be assessed on the basis of the relative magnetic loss in alloys. The concept of phases with non-ferromagnetic

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phases refers to the phases with paramagnetic, magnetic neutral and dimagnetic properties.

2. Material for research

The relative magnetic loss was examined on model samples with the dimensions Φ20x20 mm, identified by numbers from 1 through 6. The samples identified with numbers 1 and 6 were made from ingot iron (99.7% Fe, 0.1% Mn, 0.05% S, 0.05% P and others) and electrolytic copper (99.9% Cu), respectively. The samples identified with numbers from 2 through 5 were taken from test casts, made of iron and copper alloys with differentiated compositions. The melting process and casting into moulds were realized in the chamber of a vacuum furnace produced by the Baltzers company. Test casts – in the shape of cylinders with the dimensions Φ30x100 mm – were casts into a metal mould at the temperature of 500°C. All casts underwent the process of stress relief, consisting in annealing at the temperature of 650°C for 2 h and slow cooling down of the casts, together with the furnace. The samples for magnetic and metallographic examinations were taken from the comparable bottom parts of the test casts.

3. Examination results

3.1. Metallographic examination

The metallographic examinations were carried out by means of the metallographic microscope produced by the Leica company, at the magnification levels of 100, 200, 500 and 1000. The quantitative analysis by the metallographic method was carried out by means of the LeicaQWin software. The samples for metallographic examination, identified by numbers 1 through 6, were examined before and after etching with 1% solution of HNO3 in methanol. The ?Fe solid solution (ferrite) is the only microstructure component of sample No. 1. The microstructure of the samples identified by numbers 2 through 5 consists of $\alpha_{Fe} + \alpha_{Cu}$ eutectoid (the dark area in the attached microstructure images) and α_{Cu} solid solution (the light area). The α_{Cu} solid solution is the microstructure component of sample No. 6. The microstructure of samples taken from test casts is characterized by comparable microporosity U_{Vp} of about 1.5%. Selected microstructure images of the samples identified by numbers 3 and 5 are presented in figure 1. Table 1 shows the volumetric shares of the phases with ferromagnetic properties (U_{Vf}) and non-ferromagnetic properties (U_{Vn}), calculated on the basis of the prepared feeds, while allowing for microporosity. It is assumed for the calculations, that copper in solid state does not dissolve in iron and iron does not dissolve in copper [8].

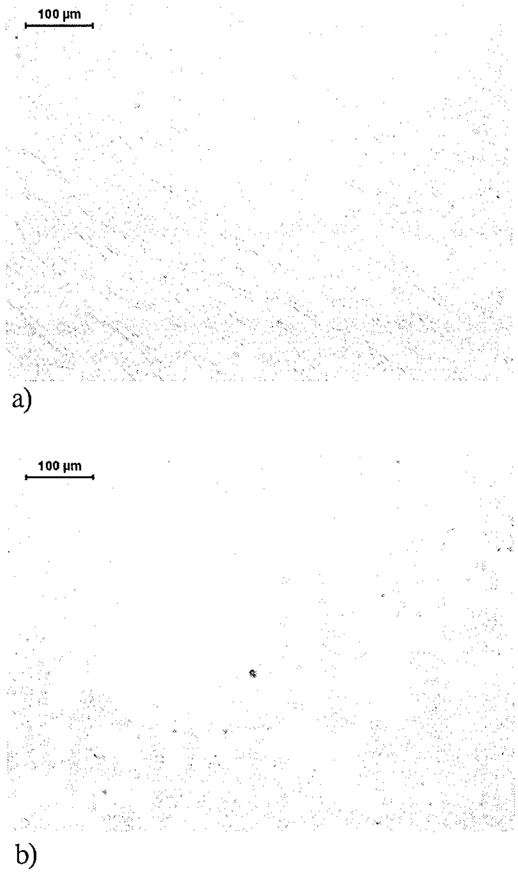

Fig. 1. Selected microstructure images: a) the sample identified by number 3, b) – the sample identified by number 5

TABLE 1

Volumetric shares of the phases with ferromagnetic and non-ferromagnetic properties in the examined samples, identified by numbers 1 through 6

Sample number	Volumetric share of the phase with:	
	ferromagnetic properties U_{Vf}, %	non-ferromagnetic properties U_{Vn}, %
1	100.0	0.0
2	88.8	11.2
3	73.6	26.4
4	49.2	50.8
5	24.7	75.3
6	0.0	100.0

3.2. Magnetic examination

The values of relative magnetic loss P_w for particular samples were determined on the basis of voltage and current characteristics $U_i(I)$, determined by means of a device for magnetic examinations, designed by the authors of the text [7].

The examined samples were subjected to unidirectional, variable magnetic field with the frequency f = 50 Hz. The magnetic measurements were carried out at room temperature. Figure 2 presents the voltage and current characteristics $U_i(I)$, obtained for the examined samples, identified by numbers 1 through 6. Table 2 presents the values of minimum relative magnetic loss P_{wm} determined on this basis in bending point. The following conditions were assumed during the determination of the minimum relative magnetic loss in the examined samples: the relative magnetic loss $P_w = 1$ for a magnetically-neutral environment (air); the minimum relative magnetic loss $P_{wm} = 0$ for an environment with perfect magnetic properties.

TABLE 2

The minimum relative magnetic loss P_{wst} for samples identified by numbers 1 through 6

Sample number	1	2	3	4	5	6
The minimum relative magnetic loss, P_{wm}	0.365	0.441	0.539	0.688	0.856	1.004

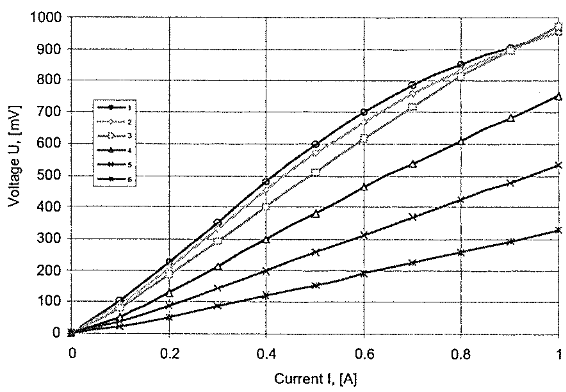

Fig. 2. Voltage and current characteristics $U_i(I)$ for model samples, identified by numbers 1 through 6, obtained by means of a prototype device for magnetic examinations

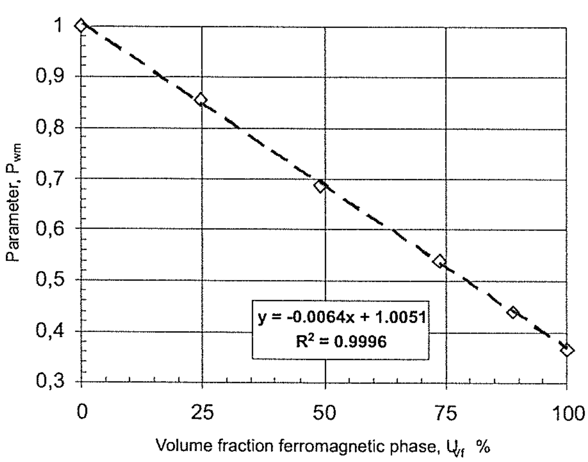

Fig. 3. The impact of the volumetric share of the phase with ferromagnetic properties U_{Vf} on the alteration of the minimum magnetic loss P_{wm} of the examined samples 1÷6

Figure 3 shows the impact of the volumetric share of the phase with ferromagnetic properties U_{Vf} (the data presented in table 1) on the alteration of the minimum magnetic loss P_{wm} of the examined samples (the data presented in table 2).

3.3. Analysis of results

The linear correlation presented in figure 3 – the correlation coefficient $R^2 = 0.9996$ – of the minimum magnetic loss on the volumetric share of the phase with ferromagnetic properties can be presented in the form of formula 1a:

$$P_{wm} = 1 - P_{wmf} \cdot UVf \quad (1a)$$

$$U_{Vn} + U_{Vf} = 100\%, \quad (1b)$$

where: P_{wm} – the minimum relative magnetic loss of an alloy, P_{wmf} – the minimum relative magnetic loss of the phase with ferromagnetic properties, U_{Vf} – the volumetric share of phases with ferromagnetic properties, P_{wn} – the relative magnetic loss of the phase with non-ferromagnetic properties, U_{Vn} – the volumetric share of phases with non-ferromagnetic properties.

The above-presented linear dependence of the minimum magnetic loss on the volumetric share of phases with ferromagnetic and non-ferromagnetic properties was confirmed by the authors of the text in the case of the model samples, which were prepared by the method of compaction of metal powders and industrial iron alloys subjected to heat treatment [6]. The obtained results allow one to generalize formula (1a and 1b) to the form presented in formula (2):

$$P_{wm} = \sum_{i=1}^{n} P_{wmi} \cdot U_{Vi}, \quad (2)$$

where: n – number of all phases in alloy, P_{wm} – the minimum relative magnetic loss of an alloy, P_{wmi} – the minimum relative magnetic loss of phase i, U_{Vi} – the volumetric share of phase i, $U_{Vi} = 100\%$.

In multi-phase alloys, the minimum relative magnetic loss of an alloy is equal to the sum of products of relative magnetic losses of particular phases and their volumetric shares.

The above-stated correlation allows one in particular to carry out comparative assessment of the volumetric shares of the phases with ferromagnetic and non-ferromagnetic properties, which constitute the microstructure of metal alloys. It can also be used for the purposes of non-destructive control of the correctness of heat treatment of iron alloys, especially to assess the volumetric share taken by the retained austenite.

4. Conclusions

The following conclusions can be formulated on the basis of the results obtained during the examinations, realized in the conditions indicated in point 3.2:

- the minimum relative magnetic loss of an alloy is inversely proportional to the volumetric share of the phases with ferromagnetic properties contained in the alloy (formula 1a),
- the minimum relative magnetic loss of an alloy is directly proportional to the volumetric share of the phases with non-ferromagnetic properties contained in the alloy (formula 1a and 1b),
- in multi-phase alloys, the minimum relative magnetic loss of an alloy is equal to the sum of products of relative magnetic losses of particular phases and their volumetric shares (formula 2).

Acknowledgements

Research work No 10.10.170.299.

REFERENCES

[1] K. Przybyłowicz, S. Jasieńska, Nowoczesne metody badań w metalurgii i metaloznawstwie. Skrypt uczelniany AGH, Kraków 1981.

[2] A. Kosowski, Metaloznawstwo i obróbka cieplna stopów odlewniczych. WN AKAPIT, Kraków 2003.

[3] Z. Bojarski, Mikroanalizator rentgenowski. Wydawnictwo "Śląsk" Katowice 1971.

[4] M. Grounes, Pomiary zawartości austenitu szczątkowego w stali i żeliwach za pomocą dylatometru różnicowego. Hutnik, 5-6, 265-269 (1981).

[5] E. Tyrała, Analiza ilościowa składu fazowego metodą magnetyczną. XXIX Konferencja Naukowa z okazji Święta Odlewnika 125-130, Kraków 2005.

[6] E. Tyrała, P. Tyrała, Wpływ udziału objętościowego faz na stratność magnetyczną dla wybranych stopów żelaza. XXX Konferencja Naukowa z okazji Święta Odlewnika 93-98, Kraków 2006.

[7] E. Tyrała, Ocena udziału objętościowego faz w stopach metodą magnetyczną. Sprawozdanie z pracy naukowo-badawczej NR 10.10.170.299, Kraków 2007.

[8] K. Sękowski, J. Piaskowski, Z. Wójtowicz, Atlas struktur znormalizowanych stopów odlewniczych. Wydawnictwo Naukowo-Techniczne, Warszawa 1972.

[9] S. Gąsiorek, R. Wadas, Ferryty zarys właściwości i technologii. WKŁ, Warszawa 1987.

[10] M. Nałęcz, Jaworski, Pomiary magnetyczne. PWN, Warszawa 1965.

Received: 3 December 2007.