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NON-DESTRUCTIVE EXAMINATION OF SINTERED ASTALOY CRL AND ASTALOY CrM-BASED STRUCTURAL STEELS

BADANIA NIENISZCZĄCE SPIEKANYCH STALI KONSTRUKCYJNYCH WYKONANYCH NA BAZIE PROSZKU ASTALOY CrL I ASTALOY CrM

This work presents the results of non-destructive examination (NDE) of Charpy impact test samples prepared from low-alloyed Astaloy CrL and Astaloy CrM iron based powders. The natural frequencies, excited in a dynamic way (by impact), of the samples were determined in three directions, perpendicular to each other. The Rayleigh dispersion corrections for the rod velocities were taken into account. The respective dynamic modulus of elasticity E were calculated. The velocities C_l of propagation of the longitudinal ultrasound waves were calculated. The mean values of the magnetic Barkhausen noise voltage were measured.

Keywords: non-destructive examination, NDE, pre-alloyed Astaloy CrL powder, pre-alloyed Astaloy CrM powder, Barkhausen magnetic effect, Young modulus, ultrasound waves

W pracy przedstawione zostały wyniki badań nieniszczących, jakie prowadzone były na spiekanych stalach konstrukcyjnych manganowo-chromowo-molibdenowych wykonanych na bazie proszków stopowych Astaloy CrL i Astaloy CrM.

W celu przygotowania próbek do badań, proszki wyjściowe Astaloy CrL i Astaloy CrM z dodatkiem niskowęglowego (1,3%C) żelazomanganu (77%Mn) oraz grafitu C-UF (0,3% mas.) poddano mieszaniu w mieszalniku dwustożkowym w czasie 60 minut. Z przygotowanych mieszanek, stosując ciśnienie prasowania 800 MPa, sprasowano kształtki prostopadłościenne o wymiarach 5x10x55 mm o dwóch różnych składach chemicznych: Fe-3%Mn-3%Cr-0.5%Mo-0.3%C i Fe-3%Mn-1.5%Cr-0.25%Mo-0.3%C.

Proces spiekania prowadzony był w dwóch temperaturach – 1120°C i 1250°C w czasie 60 minut w zasypce o składzie: $Al._2O_3$ + 15% FeMn + 10%C. Gęstość względna spieczonych kształtek mieściła się w zakresie od 87,1% do 88,6%.

W czasie badań zastosowano następujące techniki badawcze: metodę echa ultradźwięków, metodę dynamiczną przy niskich częstotliwościach oraz metodę szumów magnetycznych Barkhusena. Przeprowadzone eksperymenty pozwoliły na wyznaczenie dynamicznego modułu elastyczności wzdłużnej E spiekanych stali manganowo-chromowo-molibdenowych. Badania przeprowadzone były w trzech prostopadłych do siebie kierunkach zależnych od kierunku prasowania próbek: zgodnie z kierunkiem prasowania, prostopadle do kierunku prasowania wzdłuż szerokości próbki oraz prostopadle do kierunku prasowania wzdłuż długości próbki. Określana została również prędkość rozchodzenia się fal ultradźwiękowych C_l w spiekanym materiale.

Przeprowadzone badania wykazały, że metody ultradźwiękowe są odpowiednimi technikami badawczymi dla określania stopnia niejednorodności spiekanych stali, a efekt Barkhausena jest parametrem wystarczającym do klasyfikowania spieków ze względu na rodzaj proszków wyjściowych zastosowanych do ich produkcji.

Abbreviations

b, b_0	– width of samples
h, h_0	– height of samples
f	– recorded frequency
m, m_0	– weight of samples
kl	– transcendental factor
l	– sample length

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l_e	–	thickness of the sample
l_x	–	thickness of the model specimen
m_s	–	echoes from the sample
n_m	–	echoes from the model specimen
k	–	number of detected marks
p	–	total number of the scale marks
C_e	–	velocity of propagation of the longitudinal ultrasound waves in the model
C_b	–	rod velocity of the elastic waves
C_{l2}	–	velocity of the longitudinal ultrasound waves
C_x	–	velocity of propagation of the longitudinal ultrasound waves in the sample
E	–	Young modulus
E_1	–	modulus of elasticity along direction of pressing
E_2	–	modulus of elasticity transversely to the direction of pressing along the sample width
E_3	–	modulus of elasticity transversely to the direction of pressing, along the sample length
DV	–	digital voltmeter
K_{R1}, K_{R2}, K_{R3}	–	Rayleigh corrections for the dispersion of the rod velocity
NDE	–	non-destructive examination
M	–	microphone
MS	–	mechanical striker
P	–	porosity
PC	–	personal computer
PT	–	piezotransducer
S	–	cross section area
ST	–	sintering temperature
U_B	–	Barkhausen noise voltage
UT	–	ultrasound tester
V, V_0	–	calculated volume
ρ, ρ_0	–	calculated density

1. Introduction

Powder metallurgy materials are widely used in mechanical engineering. Their mechanical and physical properties are being continuously improved and optimised through investing and examining new materials, obtained by various processes.

Non-destructive examinations (NDE), are very popular way to investigate materials and give good description of the material condition. Problems related to preliminary pre-sintering or post-sintering control, determination of the structural inhomogeneity and the structural defects, control of the mechanical and physical properties, sorting, etc., are solved by using such methods. The correlations between the destructive and non-destructive parameters such as the dependencies of the non-destructive parameters on the porosity, the compaction rate, the sintering time or the degree of alloying are successfully used in controlling the production of end-use powder metallurgy products [1–3].

The results presented in the current work were obtained from non-destructive examinations of sintered steels made from low-alloyed Astaloy CrL and Astaloy CrM iron based powders, designed for Charpy impact toughness tests (ISO 5754).

2. Starting materials

Two groups of PM steels, based on pre-alloyed Höganäs Astaloy CrL and CrM powders were manufactured and examined. The manganese was added in the form of low-carbon (1.3% C) ferromanganese (77% Mn) having nominal particle size 12 μm as measured by sedimentation method. Elemental carbon (in amount of 0.3%) was added to powders mixtures in the form of ultra fine graphite powder. Mixtures of powders containing 3% Mn, 0.3% C, 1.5 or 3% Cr and 0.25 or 0.5% Mo were prepared by blending ferromanganese, graphite and pre-alloyed powders in a double cone mixer for 60 min.

Single uniaxial pressing in a metal die, with a bumper, under the pressure of 800 MPa and at room temperature was used for compaction of the samples.

Sintering was carried out during 60 minutes in the laboratory muffle furnace at temperature 1120°C and 1250°C in atmosphere of hydrogen/nitrogen mixture.

The accuracy of temperature control was ±1°C. The heating rate to sintering temperature was 20°C min^{-1}. Input gas rate and the sintering atmosphere dew point were controlled. The usage of ladle covers and a getter (Al_2O_3 + 15% FeMn + 10%C) ensured minimization of hydrogen losses. The experimental procedures used for sample preparation are shown in Table 1.

TABLE 1

Experimental procedures used for sample preparation

Powder, grade	Sintering atmoshpere, A_i		Sintering temperature, °C
Astaloy CrL	A_1	100% H_2	1120
	A_2	75% H_2 + 25% N_2	
	A_3	25% H_2 + 75% N_2	
	A_4	5% H_2 + 95% N_2	
	A_5	100% N_2	
Astaloy CrM	A_1	–	1250
	A_2	75% H_2 + 25% N_2	
	A_3	25% H_2 + 75% N_2	
	A_4	5% H_2 + 95% N_2	
	A_5	100% N_2	

Two types of samples of dimensions 5x10x55 mm were prepared for each experimental procedure. Average weight and average porosity (P) of the samples were in the range 20.2–20.8 g and 11.4–12.9%, respectively. Information about the conditions of the experimental procedures, used for the preparation of the samples, was given in Table 1.

3. Non-destructive methods of examinations

3.1. High frequency ultrasound echo-pulse method

Ultrasound methods are widely used for investigations of physical and mechanical characteristics of PM materials. In method presented schematically in Figure 1, the sample is locally subjected to high-frequency acoustic impulses, created by the Krautkramer USIP 11 ultrasound tester (UT). Piezo-transducer (PT) type K5K with operating frequency 5 MHz was used. Numbers 1, 2, and 3 in Figure 1 are the measure points. Measurements were performed through a thin adhesive tape to avoid impregnation of the sample by the grease used as an acoustic contact environment.

The times for propagation of the longitudinal ultrasound waves into the model specimen and the investigated samples were measured. The velocities of the longitudinal ultrasound waves into the samples were determined by the multiple reflected signals method, using the following equation:

$$C_x = C_e \cdot \frac{l_x}{l_e} \cdot \frac{m_s}{\left(n_m + \frac{k}{p}\right)}, \qquad (1)$$

where: C_x is the velocity of propagation of the longitudinal ultrasound wave in the sample and in the model specimen with known velocity C_e; l_x, l_e – thickness of the sample and the model specimen; m_s, n_m – echoes from the sample and from the model specimen; k, p – number of the detected and total number of the scale marks, on the device [4].

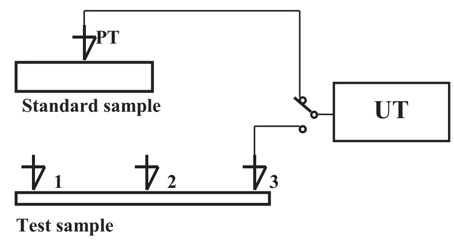


Fig. 1. Block scheme of the experimental equipment for high frequency ultrasound echo-pulse method

3.2. Low frequency dynamic method

An integral assessment of the elastic features within the whole volume of the samples can be obtained, which makes it possible to use this method.

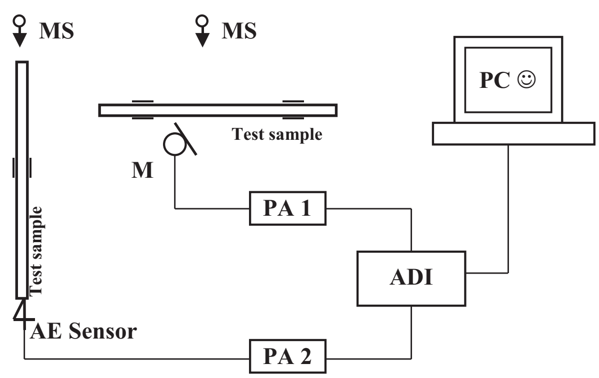


Fig. 2. Block scheme of the experimental equipment for low frequency dynamic method

A block scheme of the instrumentation used for low frequency dynamic method is shown in Figure 2. The samples were placed in a metal stand, with taut metal fibres, which help in reduction of the acoustic losses through the supports. The free oscillations of the samples were excited by a mechanical striker (MS) and recorded by the B&K 8313 type AE Sensor (acoustic emission sensor) or the RFT microphone (M) with a passband up to 30 kHz. Signals were amplified by the respective PA 1 Robotron 00 023 preamplifiers (microphone bandpass preamplifier) and B&K 2638 type PA 2 (acoustic emission widerband preamplifier). Then signals were transmitted to the input of the ProLab CMC S2A type ADI analogue/digital interface with a passband up to 160 kHz. By this interface, the signals were discretised for digital processing and recording in the personal computer (PC).

The method using an oscillating elastic rectangular-section rod with free ends allows calculation of the respective modulus of elasticity following equations (2) and (5), which apply to transverse and longitudinal oscillations, respectively [5, 6]:

$$E_1 = \rho \cdot C_b^2 \cdot K_{R1}, \quad (2)$$

$$E_2 = \rho \cdot C_b^2 \cdot K_{R2}, \quad (3)$$

$$C_b = \frac{2 \cdot \pi \cdot f \cdot l^2}{(kl)^2} \cdot \sqrt{\frac{S}{J}}, \quad (4)$$

$$E_3 = 4 \cdot l^2 \cdot f^2 \cdot \rho \cdot K_{R3}, \quad (5)$$

where: E_1 – modulus of elasticity, along the direction of pressing, in the case of impact excitement, E_2 – modulus of elasticity, transversely to the direction of pressing, along the sample width, in the case of impact excitement, E_3 – modulus of elasticity, transversely to the direction of pressing, along the sample length, in the case of impact excitement, ρ – density, C_b – rod velocity of the waves, f – recorded frequency, l – sample length, kl – transcendental factor (kl = 4.73), S – cross section area, J – moment of inertia of the cross section, K_{R1} = 1.07, K_{R2} = 1.27 and K_{R3} = 1.015 – Rayleigh corrections for the dispersion of the rod velocity [6].

3.3. Barkhausen magnetic noise method

The Barkhausen magnetic effect is related to sudden irreversible magnetisation of separate domains, or parts of them, under the effect of a magnetic field, which changes at a constant rate. Using a measuring coil, impulse signals of duration in the range from 10^{-4} to 10^{-8} were recorded. The amplitude and the energy of the signals were proportional to the material volume, which had been magnetised. The jumps in the magnetisation of the material are related to random processes in ferromagnetic materials: fixing and motion of the domain walls, formation and disappearance of nuclei of magnetisation, change of the magnetisation in some domains, formation and motion of interdomain 90° and 180° walls, overcoming of potential barriers, related to crystallographic anisotropy, alloying, internal stresses, etc. [1].

Figure 3 shows the experimental equipment used for above mentioned method, which includes an UBK-MM type meter of the mean value of the magnetic noise voltage, magnetising coil C 1, measuring coil C 2 and a digital voltmeter (DV). The sample was placed into the coil and after adjustment of the magnetising current, the voltage value is read from the digital voltmeter.

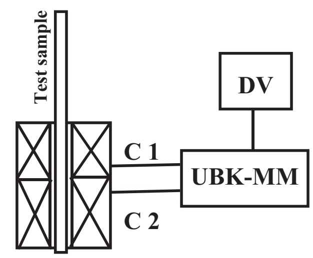


Fig. 3. Block scheme of the experimental equipment for the magnetic noise method

4. Results

Before the non-destructive examinations were performed, the dimensions, the weights, the volumes, also green and as-sintered densities of the samples were measured. The mean values for both investigated steels and the two sintering temperatures applied for the most frequently used protective atmosphere A_4, are shown in Table 2, where m, m_0 stands for weight, h, h_0 – for height, b, b_0 – for width, l, l_0 – for length, V, V_0 – calculated volume and ρ, ρ_0 – calculated density of the samples. The accuracy of the measurements was: 0.01 mm for h, h_0 and b, b_0, 0.05 mm for l, $l_0$and 0.01 g for m, m_0. The data applies to the best samples in the sets.

TABLE 2

Dimensions, green and as-sintered densities of Astaloy CrL and Astaloy CrM-based steels – mean values

Alloy	ST, °C	green compact						sintered and tempered steel					
		m_0	h_0	b_0	l_0	V_0	ρ_0	m	h	b	l	V	ρ
		g	mm	mm	mm	cm^{-3}	gcm^{-3}	g	mm	mm	mm	cm^{-3}	gcm^{-3}
Astaloy CrL	1120	21.25	5.03	10.79	55.57	3.017	7.04	21.25	5.09	10.85	55.68	3.073	6.92
	1250	21.28	5.00	10.86	55.60	3.020	7.05	21.21	5.05	10.85	55.56	3.041	6.97
Astaloy CrM	1120	20.88	4.78	11.36	56.12	3.046	6.86	20.87	4.82	11.35	56.20	3.077	6.78
	1250	20.75	4.71	11.51	56.25	3.047	6.81	20.70	4.78	11.51	56.13	3.088	6.70

4.1. High frequency ultrasound echo-pulse method

The velocities of the longitudinal ultrasound waves along pressing direction were measured in three points (in both ends and in the middle) for each sample. Figures 4 and 5 present the dependence of the C_{l2} values, measured in the middle points only, on the protective atmosphere used, for two samples made from Astaloy CrL and Astaloy CrM powders, respectively.

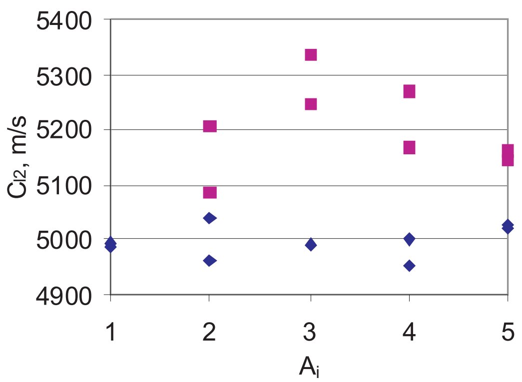


Fig. 4. Dependence of C_{l2} on the protective atmosphere for Astaloy CrL

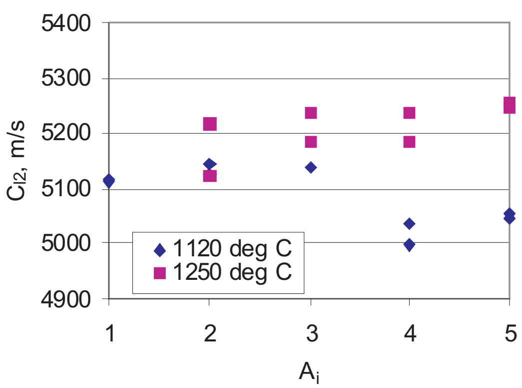


Fig. 5. Dependence of C_{l2} on the protective atmosphere for Astaloy CrM

4.2. Low frequency dynamic method

The values of modulus of elasticity for PM structural steels based on Astaloy CrL and Astaloy CrM powders, obtained using the low frequency dynamic method, are shown in Figures 6, 8 and 10, and Figures 7, 9 and 11, respectively. The Rayleigh corrections were taken into account for each sample.

Figures 12–14 present the dependencies between the modulus of elasticity of investigated PM steels, obtained from the two pre-alloyed Astaloy powders in different experimental conditions. The results were statistically processed and approximated using linear relations. The obtained regression equations of high correlation (R= 0.874 – 0.991) are given in Table 3.

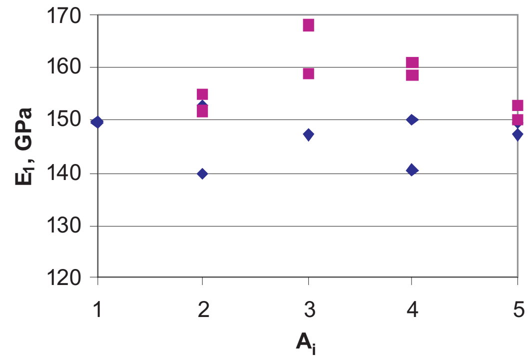


Fig. 6. Dependence of E_1 on the protective atmosphere for Astaloy CrL

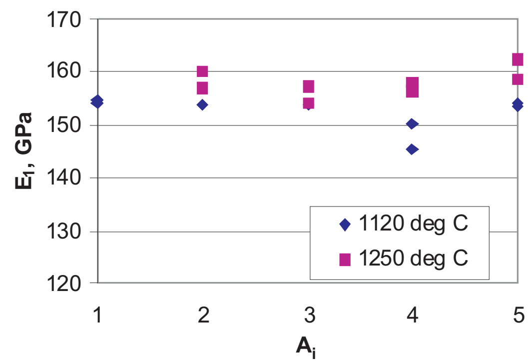


Fig. 7. Dependence of E_1 on the protective atmosphere for Astaloy CrM

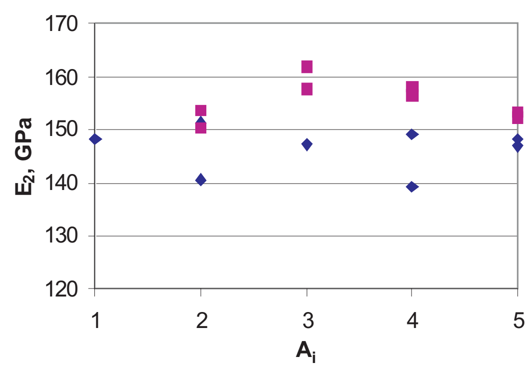


Fig. 8. Dependence of E_2 on the protective atmosphere for Astaloy CrL

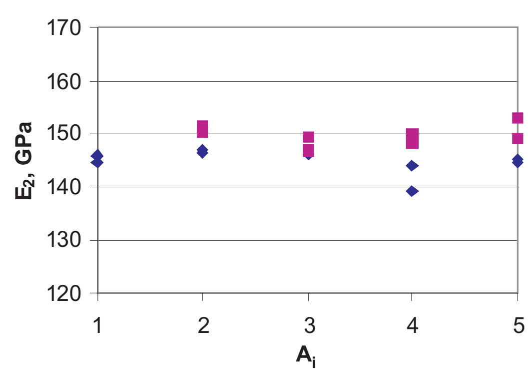


Fig. 9. Dependence of E_2 on the protective atmosphere for Astaloy CrM

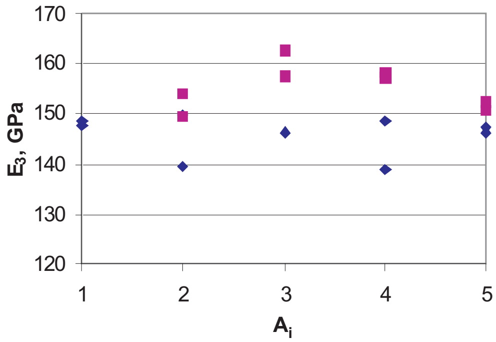


Fig. 10. Dependence of E_3 on the protective atmosphere for Astaloy CrL

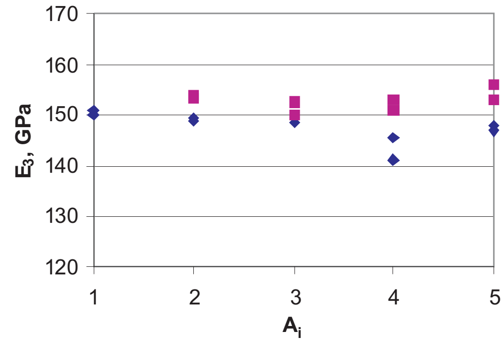


Fig. 11. Dependence of E_3 on the protective atmosphere for Astaloy CrM

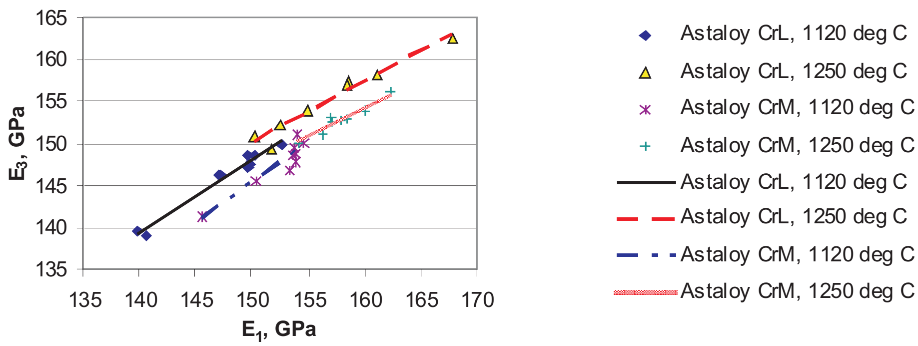


Fig. 12. Linear correlation between E_3 and E_1 for all experimental procedures applied

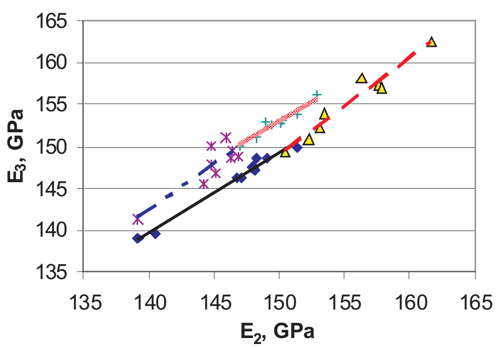


Fig. 13. Linear correlation between E_3 and E_2 for all experimental procedures applied

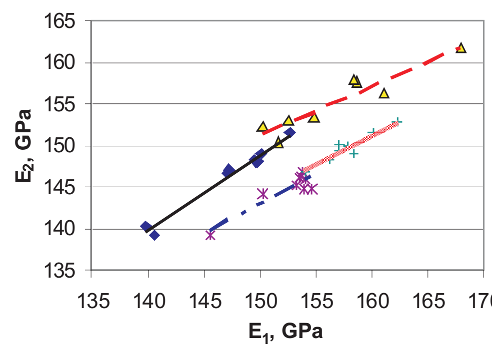


Fig. 14. Linear correlation between E_2 and E_1 for all experimental procedures applied

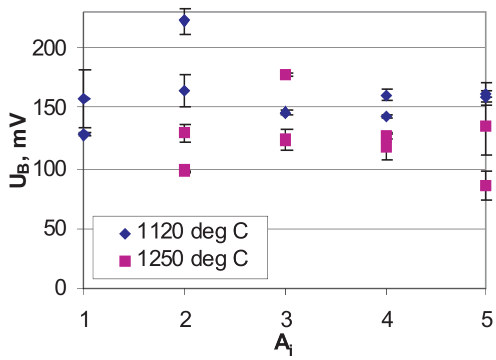
TABLE 3

Regression equations for all experimental procedures applied

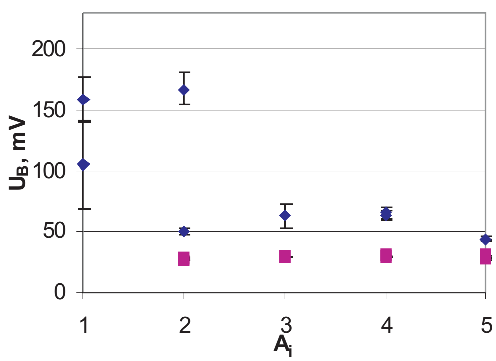
Powder, grade	T, °C	Regression equations	R
Astaloy CrL	1120	$E_3 = 0.8723\ E_1 + 17.318$	0.987
		$E_3 = 0.9598\ E_2 + 5.2895$	0.991
		$E_2 = 0.9004\ E_1 + 13.782$	0.987
	1250	$E_3 = 0.7276\ E_1 + 40.983$	0.975
		$E_3 = 1.1644\ E_2 - 25.715$	0.978
		$E_2 = 0.5878\ E_1 + 63.099$	0.938
Astaloy CrM	1120	$E_3 = 0.9359\ E_1 + 4.9509$	0.938
		$E_3 = 1.1073\ E_2 - 12.636$	0.874
		$E_2 = 0.7190\ E_1 + 35.144$	0.913
	1250	$E_3 = 0.6969\ E_1 + 42.771$	0.959
		$E_3 = 0.9477\ E_2 + 10.898$	0.971
		$E_2 = 0.6982\ E_1 + 39.504$	0.938

4.3. Barkhausen magnetic noise method

The mean magnetic noise value, U_B, was measured in both ends of the samples for alternative current magnetisation at 50 Hz (Figures 15 and 16). The mean values and the scattering of the values obtained are shown for each studied sample.



Rys. 15. Dependence of U_B on the protective atmosphere for Astaloy CrL



Rys. 16. Dependence of U_B on the protective atmosphere for Astaloy CrM

5. Discussion

The differences in the weights and dimensions of the PM structural steels results from the different apparent density and the different morphology of the starting powders (Table 2). A slight increase (up to 1–2%) in the samples' volume is observed at the same time. The steels based on Astaloy CrM powder have about 4% and 2% lower density than the ones made from Astaloy CrL powder, after sintering at 1250°C and 1120°C, respectively.

The C_l values measured in points 1, 2 and 3 (Figure 1) reveal longitudinal inhomogeneity of the sintered steels, which should be taken into consideration in the analysis of the Charpy test data.

The reduction potential increases with the increase of hydrogen content in the protective atmosphere, i.e.: oxide concentration in the material will be lower and thus higher strength of the PM materials could be expected, while nitrogen has little influence on the oxide concentration. The lowest scattering of the C_{l2} and the E values in the sample sets was observed for protective atmospheres, containing N_2 or H_2 only (A_1 or A_5), as was shown in Figures 4–11. A set contains all the samples prepared from the same powder, sintered under the same conditions (temperature, atmosphere).

The E values of PM structural steels, based on Astaloy CrL powder, sintered at T = 1250°C in mixture of 5%H_2-95%N_2, show slight scattering, while a maximum is observed for the samples sintered in atmosphere containing 25%H_2 and 75%N_2. The lowest E values were obtained for steels with higher chromium and molybdenum content after sintering at 1250°C in pure nitrogen.

The different protective atmospheres used for sintering PM 3%Mn-(Cr)-(Mo)-0.3%C steels, have insignificant influence on the mean value of the magnetic noise voltage, except those based on Astaloy CrM powder after sintering at 1120°C in hydrogen-rich atmosphere.

For Astaloy CrL and Astaloy CrM-based PM steels, the highest C_{l2} values and the lowest U_B values were recorded after high-temperature sintering.

Although the composition of material has little effect on E, it could be seen in Figures 6–11, that the of elasticity of low-chromium, low-molybdenum PM steels, sintered at 1250°C is slightly higher than for the samples made from Astaloy CrM powder, sintered at the same temperature.

The higher chromium and molybdenum content in sintered material contribute to increase of the non-magnetic component and thus results in less pronounced magnetic noise effect (Figures 15 and 16).

The different experimental procedure is well separated with regard to the protective atmosphere, as seen from the linear relations presented in Figures 12–14. Four separate groups (Table 3), from the regression equations of which one can obtain the median values of the dependencies between the elastic moduli, can be distinguished.

6. Conclusion

The following conclusions can be drawn from the results of the performed study:

1. The high frequency ultrasound echo-pulse method can be used to determine the homogeneity of PM samples subjected to mechanical tests.
2. A slightly pronounced anisotropy (up to 1–2%, related to E_1), typical for PM parts, was observed after taking into account the Rayleigh corrections and calculating E_1, E_2 and E_3 values.

3. The mean value of magnetic noise voltage is a suitable parameter for selection of PM materials from among Astaloy CrL and Astaloy CrM powders.
4. The protective atmosphere mainly decides on a stability of the modulus of elasticity.
5. A value of the modulus of elasticity increases with the increase of the sintering temperature within the range 1120–1250°C.

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