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## EFFECT OF PHENOMENA ACCOMPANYING WEAR IN DRY CORUNDUM ABRASIVE ON THE PROPERTIES AND MICROSTRUCTURE OF AUSTEMPERED DUCTILE IRON WITH DIFFERENT CHEMICAL COMPOSITION

### WPLYW ZJAWISK TOWARZYSZĄCYCH ZUŻYCIU W SUCHYM ŚCIERNIWIIE KORUNDOWYM NA WŁAŚCIWOŚCI I MIKROSTRUKTURĘ ŻELIWA SFEROIDALNEGO AUSFERRYTYCZNEGO O RÓŻNYM SKŁADZIE CHEMICZNYM

The research described in this article is a fragment in the series of published works trying to determine the applicability of new materials for parts of the mining machinery. Tests were performed on two groups of austempered ductile iron – one of which contained 1.5% Ni and 0.5% Mo, while the other contained 1.9% Ni and 0.9% Cu. Each group has been heat treated according to the three different heat treatment variants and then the material was subjected to detailed testing of mechanical properties and abrasion wear resistance, measuring also hardness and magnetic properties, and conducting microstructural examinations. The results indicated that each of the tested materials was sensitive to the surface hardening effect, which resulted in high wear resistance. It has been found that high temperature of austempering, i.e. 370°C, favours high wear resistance of ductile iron containing nickel and molybdenum. Low temperature of austempering, i.e. 270°C, develops high wear resistance in ductile iron containing nickel and copper. Both these materials offer completely different mechanical properties and as such can be used for different and specific applications.

*Keywords:* austempered ductile iron, wear resistance, alloying elements

Badania przedstawione w artykule wpisują się w cykl publikacji określających przydatność nowych materiałów na elementy maszyn górniczych. Testy przeprowadzono dla dwóch grup żeliwa sferoidalnego ausferrytycznego – jedna z nich zawierała 1,5%Ni i 0,5%Mo, druga natomiast 1,9%Ni i 0,9%Cu. Każdą grupę obrobiono cieplnie w trzech różnych wariantach i materiał taki poddano badaniom: własności mechanicznych, odporności na zużycie ściernie, pomiarom twardości i magnetycznym oraz ocenie mikrostruktury. Wyniki wskazują, że każdy z analizowanych materiałów umacnia się powierzchniowo, co powoduje wysoką odporność na zużycie. Stwierdzono, że wysoka temperatura hartowania izotermicznego tj. 370°C, sprzyja wysokiej odporności na zużycie żeliwa zawierającego nikiel i molibden. Niska temperatura hartowania izotermicznego tj. 270°C, sprzyja natomiast wysokiej odporności na zużycie żeliwa zawierającego nikiel i miedź. Oba te materiały charakteryzują się diametralnie różnymi własnościami mechanicznymi stąd też możliwość ich wykorzystania do odmiennych, specyficznych zastosowań.

## 1. Introduction

It is known that, in addition to heat treatment parameters, chemical composition is a major factor in determining the properties of austempered ductile iron. The use of different elements in different content, especially the additions of elements like Ni, Mo, Cu often results in significant advantages, thus allowing the new properties of ductile iron to be effectively created. However, the new chemical composition can interfere with the possibility of obtaining other properties at a level that is required by European standards (PN-EN 1564: 2012). This statement is illustrated in the diagram shown in Figure 1, which displays the results of static tensile test carried out on austempered ductile iron of different chemical composition. The scatter of the results is quite significant, but it also proves a very wide range of the potential properties of this material depend mostly on heat treatment parameters but also on chemical composition.

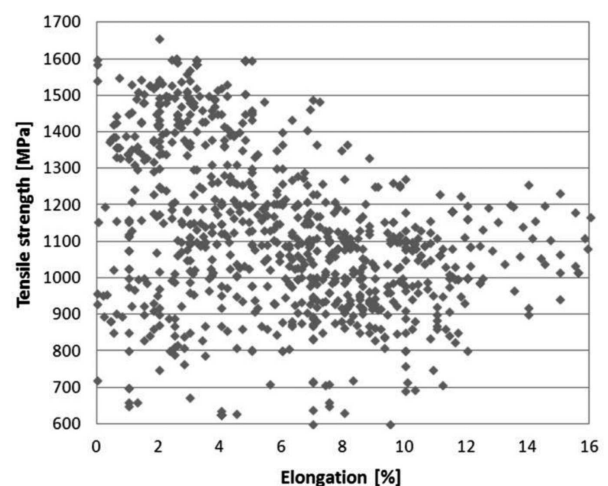


Fig. 1. Austempered ductile iron tensile strength vs elongation

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Elements that control the microstructure of austempered ductile iron matrix (a mixture of ferrite and austenite called ausferrite) are carbon, silicon, manganese, nickel, molybdenum and copper. Particularly, nickel, molybdenum and copper as additional elements are considered in research done by few investigators [1-3] as the most predictable in obtaining the required effect of changing some specific properties, like e.g. strength, ductility, fracture toughness, wear resistance, etc.

This is due to their strong influence on the formation of ductile iron matrix microstructure during heat treatment. This effect is most pronounced in the case of austenite, increasing its stability during pearlitic transformation. Introduced together or separately, they cause an increase of hardenability, thereby increasing also the critical diameter.

According to Pachowski [4] the strongest influence on the transformation of undercooled austenite has molybdenum or its combination with nickel and copper. The content of molybdenum in ductile iron as shown by Pietrowski [5] is usually limited to  $\leq 0.5\%$  due to its strong tendency to segregation and the formation of  $(\text{FeMo})_3\text{C}$  carbides on the eutectic cell boundaries. Starting with the molybdenum content of about 1% Mo, carbides of the  $(\text{FeMo})_3\text{C}_6$  and  $\text{Mo}_2\text{C}$  type can appear in the ductile iron.

In most cases, the ductile iron is enriched with the additions of up to 1.5% Cu, up to 4% Ni and up to 0.3% Mo. According to [6] it is understood that 1.5% of copper is equivalent to 0.3% Mo. However, maximum addition of copper has to be avoided because of its graphite despheroidising effect. Following further behind authors [5,7], copper in ductile iron fundamentally changes the course of the pearlitic and bainitic transformation. As regards the bainitic transformation, copper improves austenite stability by slowing down the process of its decomposition.

Nickel added in an amount of up to 1.5% does not significantly increase the stability of austenite within the range of pearlitic transformation, and up to 1.0% within the range of bainitic transformation. The effect similar to nickel has also copper, and therefore cases are reported by Santos H., Pinto A., Torres V. [8] when copper is used as a substitute for nickel or both these elements are introduced together in order to, for example, further suppress the formation of molybdenum or manganese carbides. Nickel, like copper, also reduces the rate of austenite transformation and suppresses its decomposition process within the range of bainitic transformation. Restrictions on the use of Cu and Mo cause that in practical applications it is nickel that plays the role of the element used in control of the ductile iron microstructure.

The materials used in the agricultural, mining, special, etc. industries have to meet more and more demands to which also belongs the resistance to wear, static and dynamic loads, etc. [9-17]. Due to the complexity of the phenomenon during this type of influences materials are subject to specific, different than static, types of tests. They define the properties in a way that allows to re-characterize the already known material and allow for finding new uses for it. That analysis is incredibly useful due to the possibility to make a wide comparison with other materials and the possibility to translate the results to the application possibilities of the tested material.

Therefore, studies were carried out on two groups of ductile iron containing original combinations of Ni-Cu and

Ni-Mo. The investigated ductile iron was heat treated to produce the required ausferritic matrix, and then was evaluated for its abrasion wear resistance using for this purpose an original test stand simulating the abrasive wear conditions in a loose corundum environment generally encountered in mines. The resulting samples were subjected to detailed material research. Differences were found in materials treated under the same thermal regime. Some material-related characteristics making the austempered ductile iron suitable for certain mining applications were also stated.

## 2. Methodology of studies

For tests, ductile iron in two groups of the chemical composition was selected, one of which contained 1.5% Ni and 0.5% Mo, and the other contained 1.9% Ni and 0.9% Cu. The test material was cast in the form of rods with dimensions of  $\text{Ø}60 \times 450 \text{ mm}$  – 3 rods for each of the two chemical compositions shown in Table 1. To obtain austempered ductile iron, the following heat treatment processes were performed: austenitising in a Nabertherm furnace and austempering in a salt bath at a temperature of 370, 320 and 270°C (Table 2). From castings, samples were cut out for the abrasion wear test (Figs. 2 and 3), for the static tensile test, for hardness measurements and for the metallographic studies.

TABLE 1  
Chemical composition of the tested ductile iron [wt%]

Ductile iron designation	C	Si	Mn	P	S	Mg	Ni	Cu	Mo
ADI CuNi	3,6	2,45	0,32	0,035	0,04	0,065	1,9	0,93	0,01
ADI NiMo	3,85	2,90	70,61	0,050	0,010	0,08	1,50	0,02	0,47

TABLE 2  
Heat treatment parameters applied to the tested ductile iron

Sample designation	Heat treatment			
	Austenitising		Austempering	
	[°C]	[min.]	[°C]	[min.]
ADI CuNi 370_150	900	120	370	150
ADI CuNi 320_150			320	
ADI CuNi 270_150			270	
ADI NiMo 370_150	900	120	370	150
ADI NiMo 320_150			320	
ADI NiMo 270_150			270	

Abrasion wear tests were performed by Dolipski A., Wieczorek A., (2010) on a stand designed and built at the Institute of Mining Mechanisation (IMG) (Fig. 2). Samples had the shape of rings (their design is shown in Figure 3), which made their front faces exposed to wear. Tests were carried out using corundum abrasive with grain size of 0.05-0.2 mm. Measurements were taken for two different variants of compressive stress maintained in samples, i.e. 0,063 MPa and 0,125 MPa.

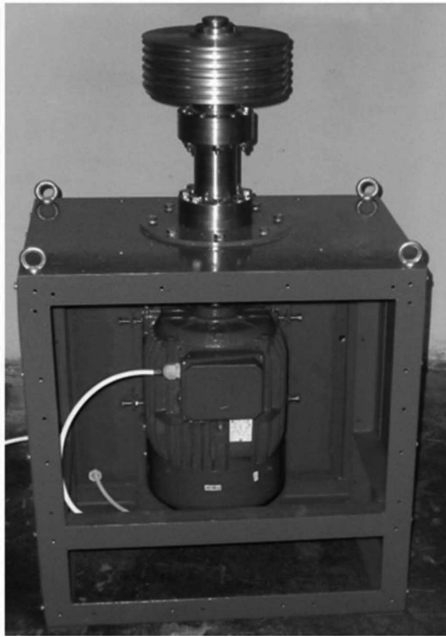


Fig. 2. Test stand to measure the abrasion wear resistance of materials used for chain drums of the scraper conveyors – general view of the stand

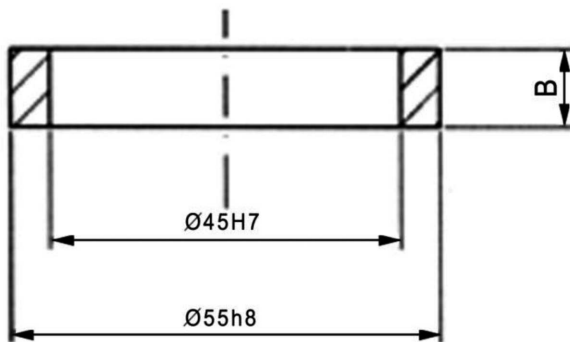


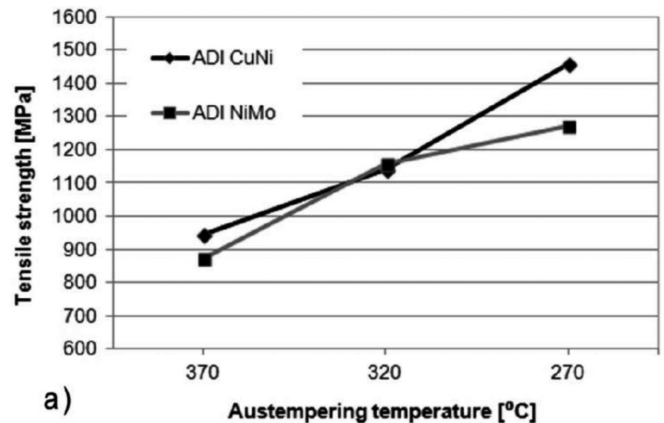
Fig. 3. The design of samples used for the loss of mass measurement on the IMG stand; B – sample width: for upper lid B = 10 mm, for lower lid B = 6 mm

All samples subjected to abrasion were next cut in two, and on the resulting cross-sections, material-related research was made and measurements were taken (hardness measurements, macro- and microscopic examinations, and measurement of magnetic properties). Vickers hardness under a load of 100 g was measured as a distribution of values from the worn out front face of the sample towards its inside; i.e. towards the core. Macroscopic evaluation was performed on a stereoscopic microscope examining the degree of abrasion wear and damage mode on the sample front face. Microscopic observations were made on metallographic sections unetched and etched with 3% Nital at a magnification of x200 and x1000 identifying the working edge under the conditions of abrasion wear. Magnetic measurements were taken with a ferrite meter using the method of eddy currents and estimating the content of ferromagnetic phases (ferrite and/or martensite) in each sample.

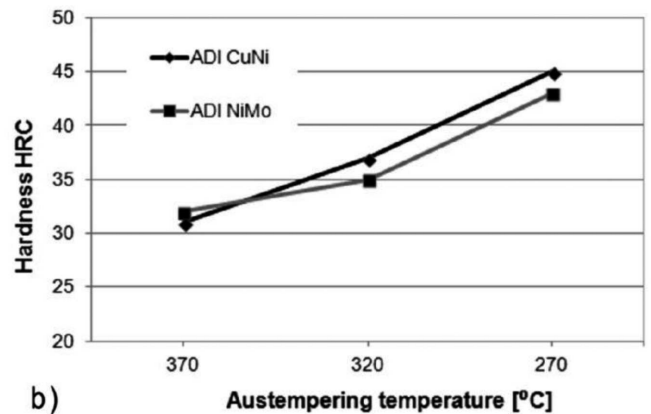
### 3. Results and discussion

The results of static tensile test and hardness HRC measurements give a general idea about the properties of the mate-

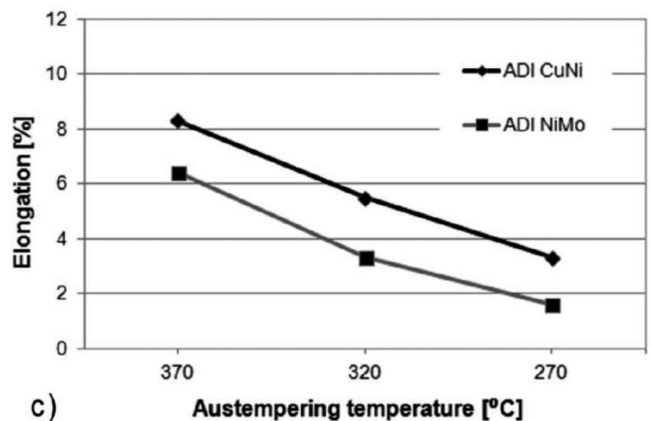
rial produced. The graph in Figure 4 shows that the ductile iron with nickel and molybdenum has nearly all parameters lower than the ductile iron containing copper and nickel. This is not consistent with the theory that states that the choice of Ni-Mo composition improves hardness and allows practical application of higher requirements for abrasion wear resistance. The mentioned ductile iron, heat treated under established conditions, has also a lower level of ductility than the ductile iron containing copper and nickel.



a)



b)



c)

Fig. 4. Graphical display of the results of basic studies of the mechanical properties

The results of abrasion wear tests, graphically presented in Figures 5 and 6, show an interesting picture for both grades of the ductile iron heat treated under different conditions. It is worth to analyse first a high level of wear resistance of the ductile iron containing nickel and molybdenum, austem-

pered at 370°C – designated as ADI NiMo 370\_150 (Figs. 5a and 6a). This material, although relatively ductile, gives many times better results in abrasion wear test than its counterparts with higher hardness levels. Attention certainly deserves the fact that this trend is maintained even under heavy loads of 0.125 MPa.

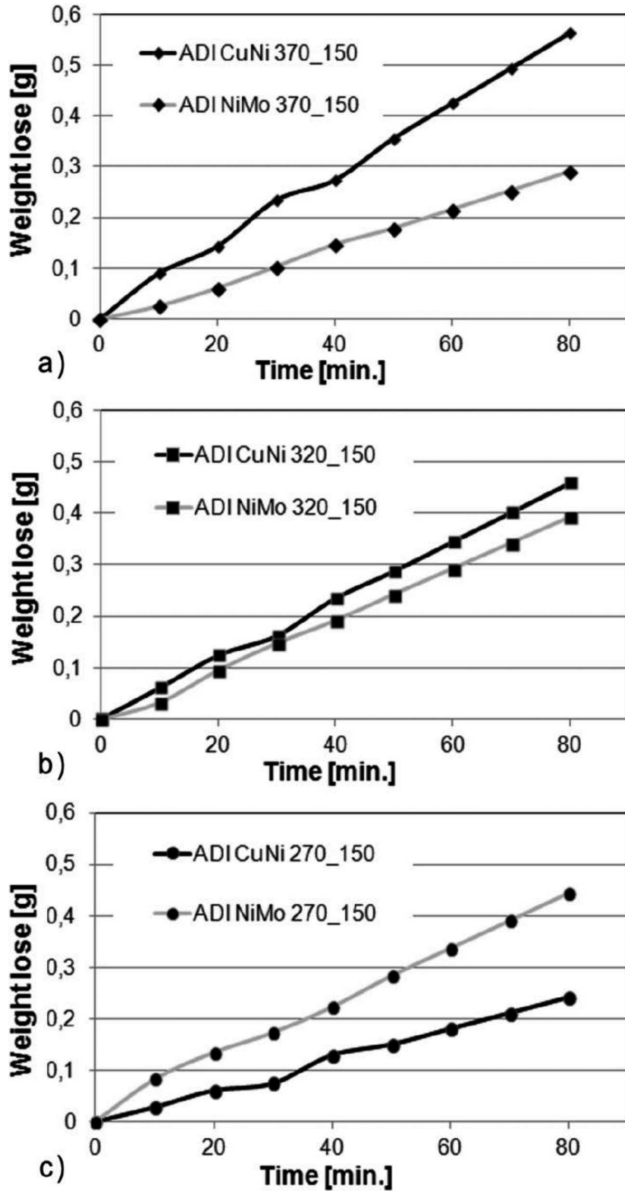


Fig. 5. The results of tests in dry corundum abrasive. Compressive stress: 0.063MPa

It is also easy to notice that in both groups of austempered ductile iron, differences in the run of the wear resistance curves are smaller for the isothermal quenching temperature of 320°C (Figs. 5b and 6b). Close analysis of these diagrams allows concluding that ADI CuNi and ADI NiMo heat treated at this temperature are comparatively resistant to abrasion wear, although some preference is with the ADI NiMo 320\_150. It is possible that the ADI containing Cu in the case of high transition temperature ausferritic (370°C), a relatively long transition time (150 min) allows to start processes, the precipitation of carbide from the austenite, which helps strengthen the surface and increases abrasion resistance [18].

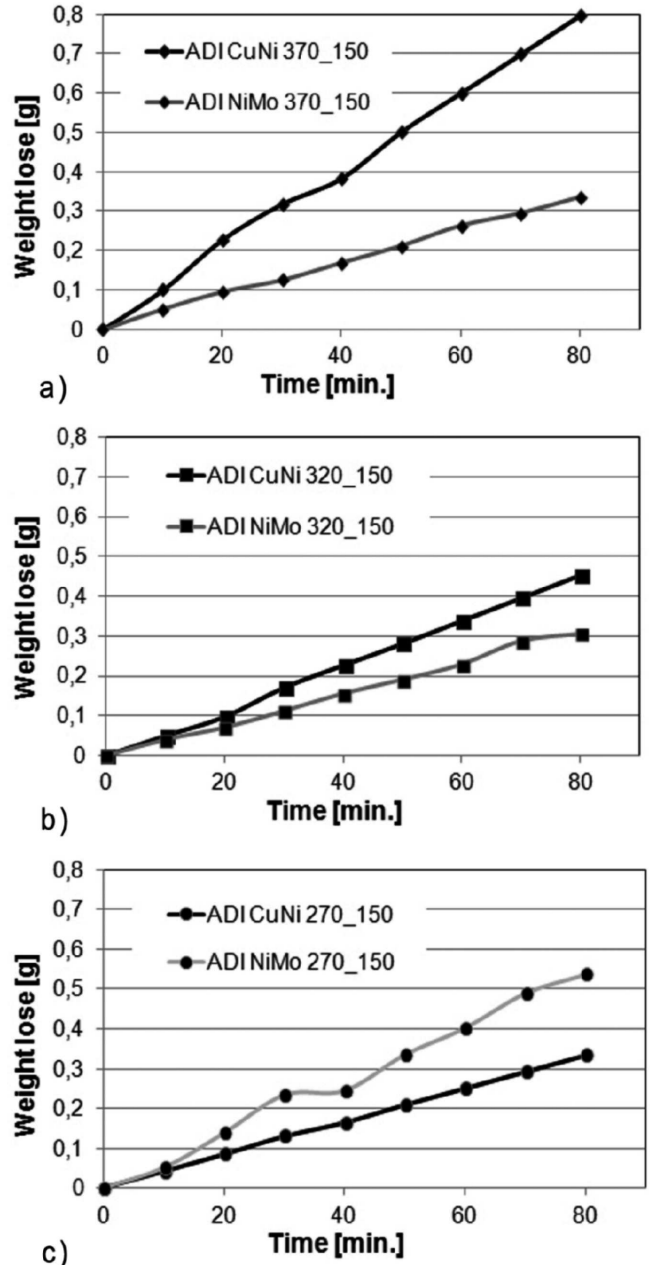


Fig. 6. The results of tests in dry corundum abrasive. Compressive stress: 0.125MPa

The lowest temperature of isothermal quenching (270°C) results in the manufacture of ductile iron characterised by the highest hardness and good wear resistance. Better characteristics to offer has the ductile iron containing copper and nickel (Figs. 5c, 6c). Charts show that the grades with low hardness are in terms of abrasion wear resistance by no means inferior to those with high hardness. A comparison of charts presented in Figures 6a, b, c, suggests indeed that the abrasion resistance is comparable for samples differing in hardness by even more than 10 HRC units.

The surfaces of samples after the abrasion wear test were subjected to careful stereoscopic observations, owing to which differences in the size of the worn out surfaces were traced. Figures 7 and 8 show the surface of one of the samples before and after the abrasive corundum impact. The milled surface clearly changes its character to a surface cut through with the crevices of uneven plastic deformation and microcutting with

the shifting hard corundum grains. The abraded surface also shows the heterogeneity of the abrasion process – some parts of the sample surface are more exposed to wear than the other parts (Fig. 8). This is the specific case of abrasion wear caused by loose abrasive, which roughly simulates the conditions of real abrasion in an industrial environment.

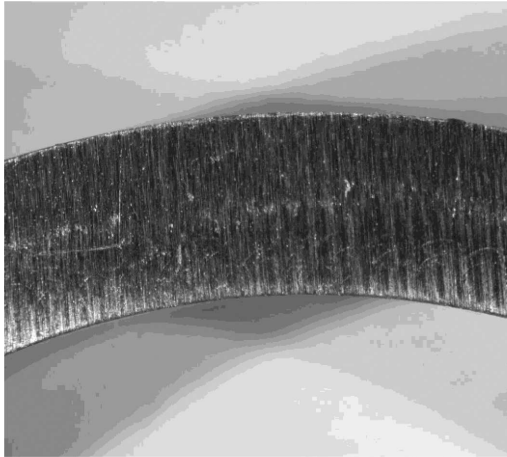


Fig. 7. Surface appearance of the ductile iron samples after milling before the wear test



Fig. 8. Surface appearance of the ductile iron samples after the wear test

The impact of abrasive corundum grains causes micro-cutting and plastic deformation on the ductile iron sample surface. The measurements of hardness taken on a cross-section of the abraded surface, displayed as a distribution of values (Fig. 9), and microscopic observations (Figs. 11 and 12) give a general idea about the way the material "responds" to a drastic impact during the abrasion wear test. It shows, first of all, in the distribution of microhardness values, starting with the subsurface micro-areas. The results of the measurements indicate that each sample has undergone the process of hardening, although it penetrated the sample to a different depth and thus ensured different levels of hardness (Fig. 10).

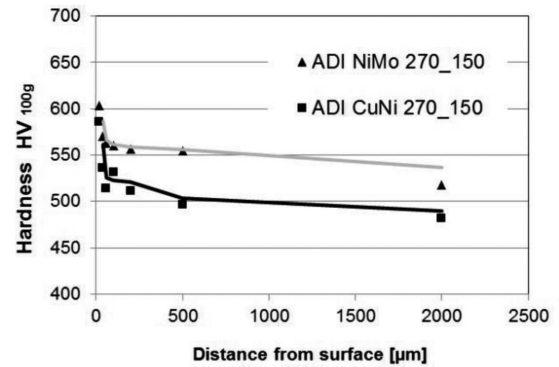
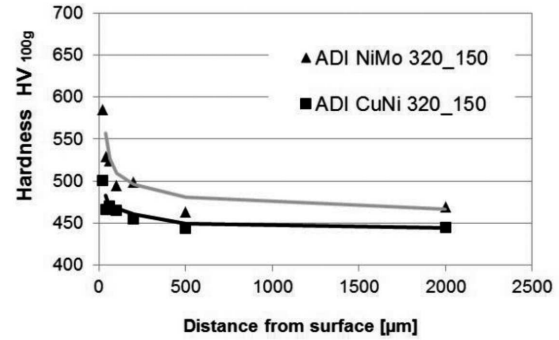
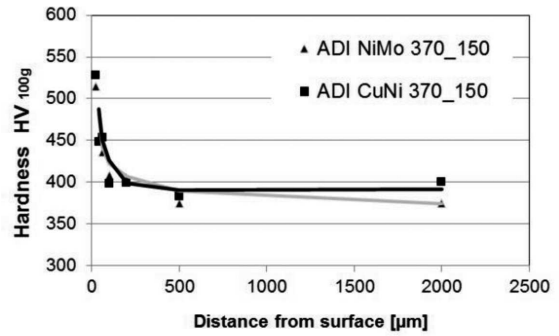


Fig. 9. The results of hardness measurements displayed as a distribution of values in direction from the sample surface: a) ADI\_370\_150, b) ADI\_320\_150, c) ADI\_270\_150

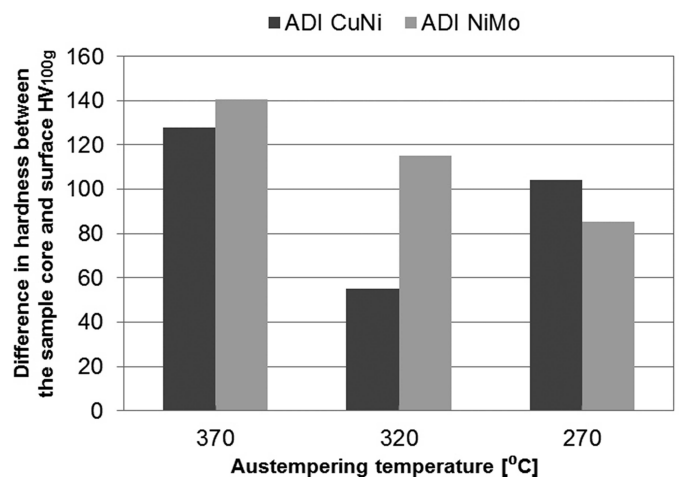


Fig. 10. Visual representation of differences in hardness between the sample core and surface after the abrasion wear test

From the analysis of hardness increase in the surface layer compared to the low-lying layers of material (sample core) it follows that the largest increase in hardness has occurred in the copper- and nickel-containing ductile iron austempered at 320 and 370°C.

On the other hand, the lowest examined temperature of austempering, i.e. 270°C, gave higher increase of hardness in the ductile iron containing nickel and molybdenum. This is consistent with the results of abrasion wear test, although it should be remembered that the differences in hardness are relatively small, and therefore drawing of unmistakable conclusions is rather difficult. Hardening, in any event, indicates the effect that the abrasive has on microstructure and formation of compressive stresses in the tested material. It can also indicate the occurrence of phase transformations associated with instability of the ductile iron structure. The highest degree of hardening recorded for samples austempered at 370°C may indicate the highest content of metastable phases, austenite – in particular, as proved by further studies.

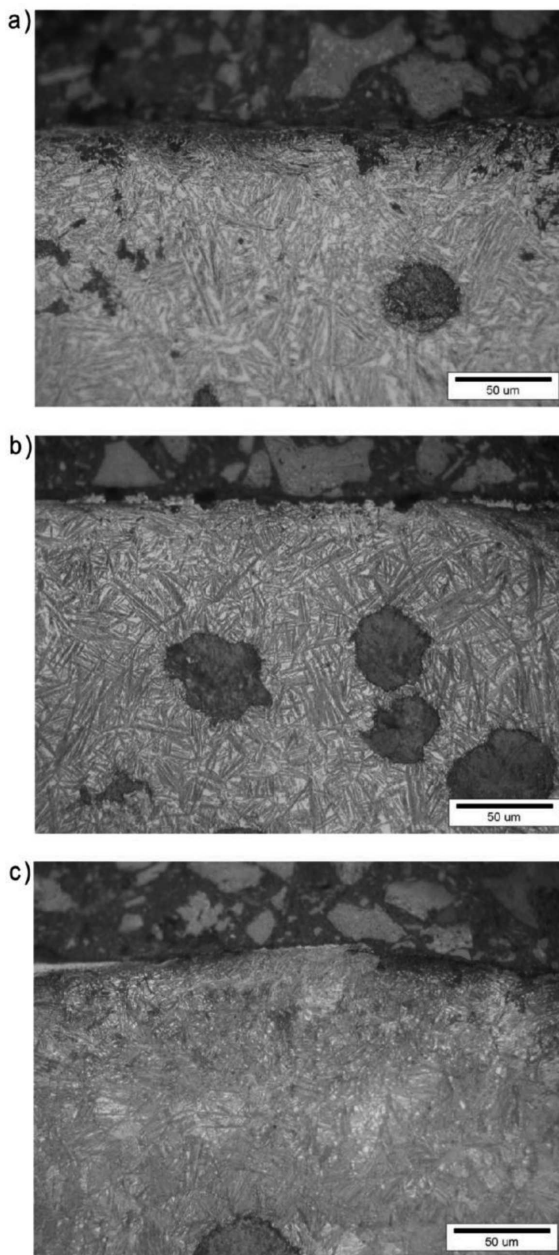


Fig. 11. Microstructure of ADI samples containing 1,5%Ni and 0,47%Mo after the abrasion wear test: a) ADI.NiMo.370.150, b) ADI.NiMo.320.150, c) ADI.NiMo.270.150

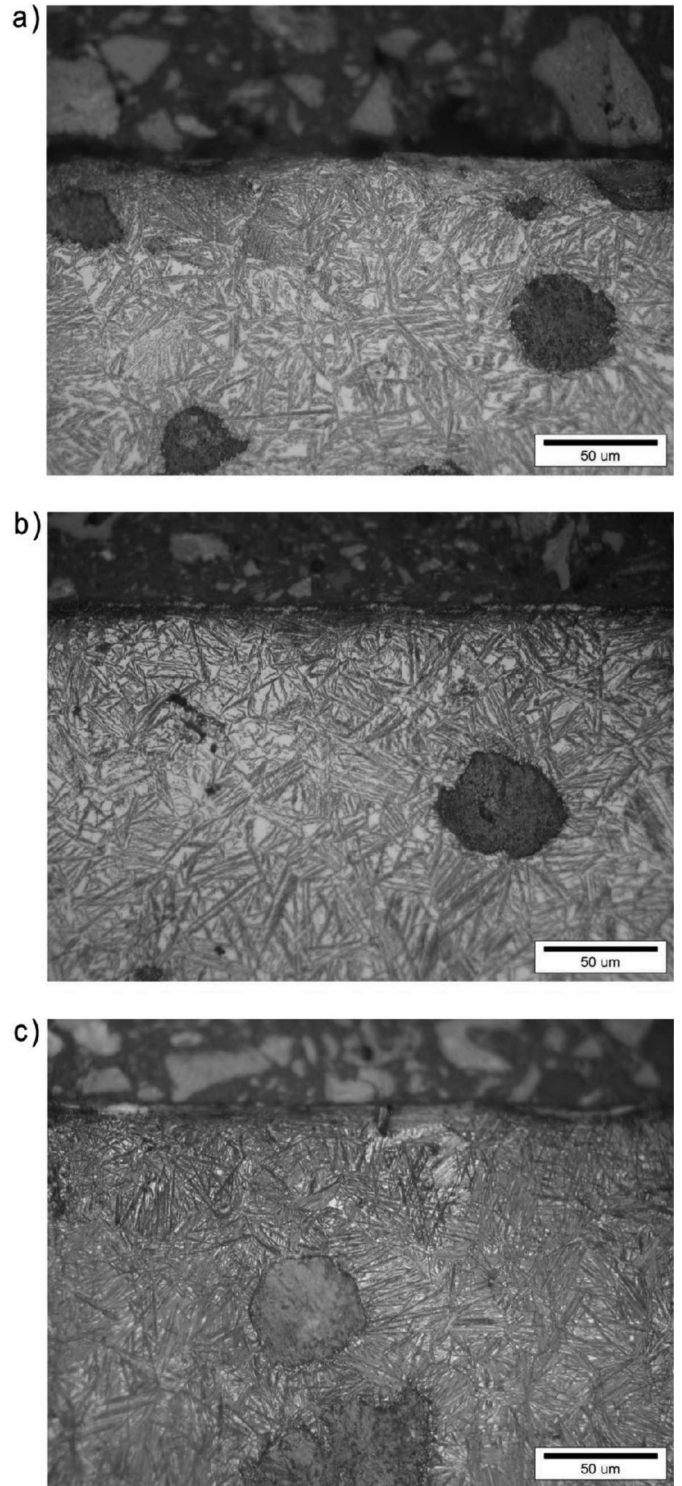


Fig. 12. Microstructure of ADI samples containing 1,9%Ni and 0,93%Cu after the abrasion wear test: a) ADI.CuNi.370.150, b) ADI.CuNi.320.150, c) ADI.CuNi.270.150

The analysis of respective images (Figs. 11 and 12) shows different microstructures in each type of material. At higher temperatures, the austempered ductile iron will be characterized by slightly higher content of austenite and ferrite lamellae morphology commonly referred to as "feathered". The lowest temperature of treatment will give the morphology of ferrite lamellae closer to martensite with decreasing content of austenite. The temperature of 320°C is characterized

by a microstructure intermediate between that obtained at the temperature of 370°C and 270°C.

From the etched micrographs it is difficult to determine the effect that the abrasive has on microstructure, but an unetched image of the ADI NiMo 370.150 sample allows us to identify a relatively strong surface deformation. The image of the microstructure (Fig. 13) shows the deformed graphite spheroids assuming an elongated form just under the surface exposed to abrasive effect.

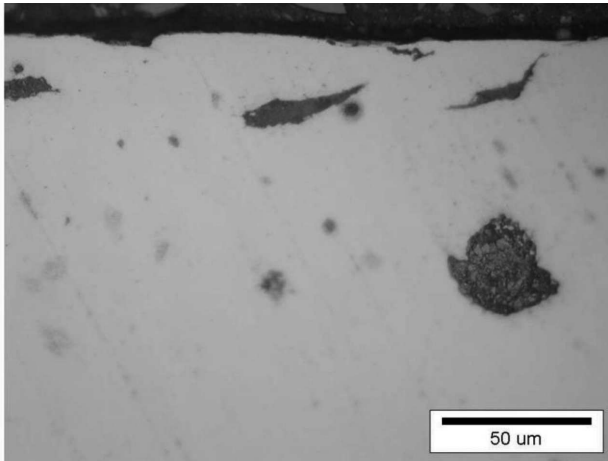


Fig. 13. The edge of ADI NiMo 370.150 sample after the abrasion wear test; unetched section

The measurement of the content of magnetic phases taken on the surface of the ADI NiMo 370.150 sample before and after the abrasion wear test allows a more accurate assessment of the deformed structure (Fig. 14). It shows that in this sample (like in the other ones) the content of paramagnetic phases has decreased in favour of the ferromagnetic ones (ferrite and martensite).

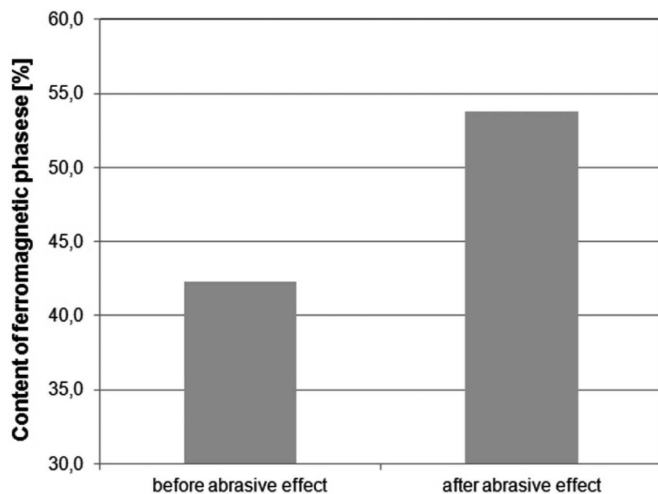


Fig. 14. Measurement of the content of ferromagnetic phases in microstructure of the NiMo ADI 370.150 sample before and after the abrasion wear test

Deformation arrested the growth of ferrite as documented by Garin J.L., Mannheim R.L. [19] so the only phase that could occur due to the impact of abrasive was martensite. Analysis of the graph in Figure 14 shows that the growth of martensite is quite significant, similar as the increase of

hardness (Fig. 10). Hardness at a level higher than 500HV suggests that indeed martensite must have appeared in the ductile iron microstructure accompanied by a strong effect of surface hardening. The presence of the resultant martensite can prove the presence of unstable austenite in the microstructure of austempered ductile iron (Fig. 15). Characteristic feature of this particular austenite is that it “rebuilds” its crystal structure under the effect of stress or deformation with the following transformation into martensite [20,21].

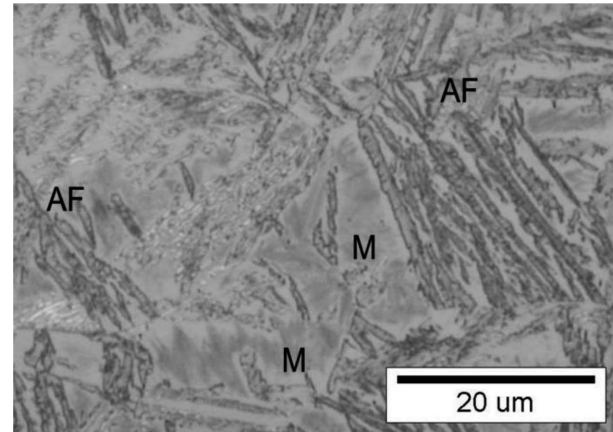


Fig. 15. Ductile iron matrix in a surface layer of ADI NiMo 370.150 sample – ausferrite AF + martensite M

#### 4. Conclusions

Studies conducted on two types of austempered ductile iron containing the combinations of Ni and Mo or Cu and Ni allowed formulating the following conclusions:

- The austempered ductile iron is a material of relatively high abrasion resistance.
- Isothermal quenching at high temperature, i.e. at 370°C and 320°C, makes this ductile iron more resistant to abrasion in the dry corundum environment when it has the addition of nickel and molybdenum.
- Low temperature of isothermal quenching, i.e. 270°C, allows obtaining the highest level of abrasion wear resistance in the ductile iron containing copper and nickel.
- Abrasion causes microcutting and plastic deformation on the surface of austempered ductile iron, resulting in strong hardening of the surface layer.
- Hardening of the ductile iron surface layer is also due to phase transformations induced by the abrasive impact, i.e. owing to the transformation of metastable austenite into martensite.

#### Acknowledgements

Research realized thanks to the development project No. N R09 0026 06/2009 financed from the Ministry of Science and Higher Education by decision No. 0481/R/T02/2009/06.

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