

## Influence of the chemical composition on the hardness of defibrator plate segments

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**Abstract:** *Influence of the chemical composition on the hardness of defibrator plate segments.* The paper presents the results of the hardness measurements of the material of defibrator plate segments, in connection with their chemical composition. The investigations of the chemical composition were performed using Energy Dispersive X-ray Spectroscopy (EDS). The hardness was measured using the Rockwell method. The increased number of the alloy components leads to an increase in hardness by approx. 3%, in comparison with the normalized L210H21S cast steel. Changes of the chemical composition and increase in the number of alloy components are ineffective from the viewpoint of the alloy hardness.

Keywords: defibrator plate segments, hardness, chemical composition,

### INTRODUCTION

The method of defibration of plant-origin materials (mainly chips) i.e. transforming them into a fibrous form, patented in 1931 by Swedish engineer Arne Asplund, is widely used in industry in the process of fiberboard manufacture for over 90 years, i.e. since 1934 (Marchlewska-Szrajerowa 1946).

The problems of defibration, equipment design and novel solutions in the fiberboard industry were widely discussed (see Roll 2010 and Nicewicz and Kowaluk 2017).

The two key components of the defibrator chamber are defibrator plates; a stationary and the rotating ones. These plates are composed of defibrator segments characterised by a specific pattern, which may depend on the type of defibrator, the processed material and the required quality of the pulp (Oniśko 2000). The milling segments operate in a highly corrosive chemical atmosphere, i.e. in the environment of saturated and overheated water vapor at temperatures exceeding 100°C (Haber et al. 2002, Starecki et al. 1986, BN-79/7122-25), in the presence of organic acids and at a continuous operation of tribological wear mechanism. Consequently, they should be made of materials resistant to corrosion and abrasive wear. The basic material used for defibrator plate segment is cast steel alloyed with carbon and other elements (alloy components), which determine the specific cast steel properties, containing up to 2% of carbon, obtained in cast process and cast into cast moulds. The casting products may be used either directly after solidification, i.e. without thermal treatment or may undergo thermal or chemo-thermal treatment (Bartocha and Jura 2001, Dobrzański 2006, Roźniatowski and Szałowski 1995).

The way of marking the alloy cast steels is identical as that of construction alloy steels, the difference being that the cast steel symbol is preceded with letter G or L (older notation). The letter G or L is followed with a number determining the mean carbon content with .01% accuracy and the letters (symbols) determining alloy elements with numbers giving the appropriate content with 1% accuracy, exactly as in case of constructional steels. If the content

of the alloyed element does not exceed 2% on an average, only the element symbols are given (Roźniatowski and Szałowski 1995).

The main alloy elements in cast steels include:

- chromium, Cr - improves hardenability, increases the abrasive wear resistance (at a content above 2%), increases resistance to high temperature and oxidation corrosion (at a content above 10.5%),
- vanadium, V - improves hardenability and enhances the effect of precipitation hardening (up to 0.2%). Increases the abrasion wear resistance at higher content,
- nickel, Ni - improves hardenability (at a content of 0.5-1%), reduces the brittle state transition temperature (3-9%), stabilizes austenite in corrosion and high temperature oxidation resistant cast iron (above 8%),
- molybdenum, Mo - increases hardenability, increases strength and creep resistance, reduces brittleness, reduces cast iron weldability at higher content,
- manganese, Mn - initiates the deoxidation process (removal of harmful iron oxide), neutralizes the undesired sulphur activity (up to 0.7%),
- silicon, Si - prevents sulphur and phosphorus segregation, reduces the oxygen content, increases cast iron castability, increases hardness and tension resistance, deteriorates weldability,
- copper, Cu - increases material resistance against atmospheric corrosion, enhances the precipitation hardening (up to 0.2%),
- sulphur, S - deteriorates plastic properties, leads to hot brittleness, improves the cast steel workability (above 0.035%),
- phosphorus, P - may lead to hot brittleness (above 0.1%), reduces strongly plasticity, leads to segregation,
- oxygen, O - induces sulphur and phosphorus segregation, leads to material brittleness,
- nitrogen, N - may shift the material brittleness threshold to higher temperature and hence induce microcracks (Maruszczyk et al. 2015).

Apart from the above described alloy additions and impurities, cast steels are also supplemented with such elements like boron B, titanium Ti or rare earth elements REE, which may essentially influence the mechanical properties of the modified material (Sobula et al. 2013).

By the way of an example, as regards cast steel used for defibrator plate segment casting, we may refer to an alloy tool cast steel marked as a 21SL210H and mentioned in PN-90/H-83161 Polish Standard. This standard encompasses 18 types of cast steel of wide applications for both cold, and hot work, used after heat treatment to provide appropriate hardness (Konfederak 2007). These steels contain more carbon and alloyed elements. The chemical composition of L210H21S cast steel is presented in Table 1. The material density is at a level of 7.6 g/cm<sup>3</sup>, the specific heat is 460-500 J/kgK, and the linear expansion coefficient is 1e-5 1/K in the temperature range 20-100°C.

**Table 1.** Chemical composition of L210H21S cast steel (CTNTW)

Iron Fe	Carbon C	Chromium Cr	Nickel Ni	Molybdenum Mo	Manganese Mn	Silicon Si	Sulfur S	Phosphorus P
rest	1.95-2.45%	20-25%	0.5-1.5%	≥0.8%	0.3-0.9%	0.3-0.9%	≤0.04%	≤0.04%

In this place, it is worthwhile to notice the existing differences and discrepancies among various data describing cast steels in the references. By the way of an example, the general cast steel definition determines the maximum carbon content as about 2%, while the PN-90/H-83161 Polish Standard allows the content of this element as high as up to 25% above the former

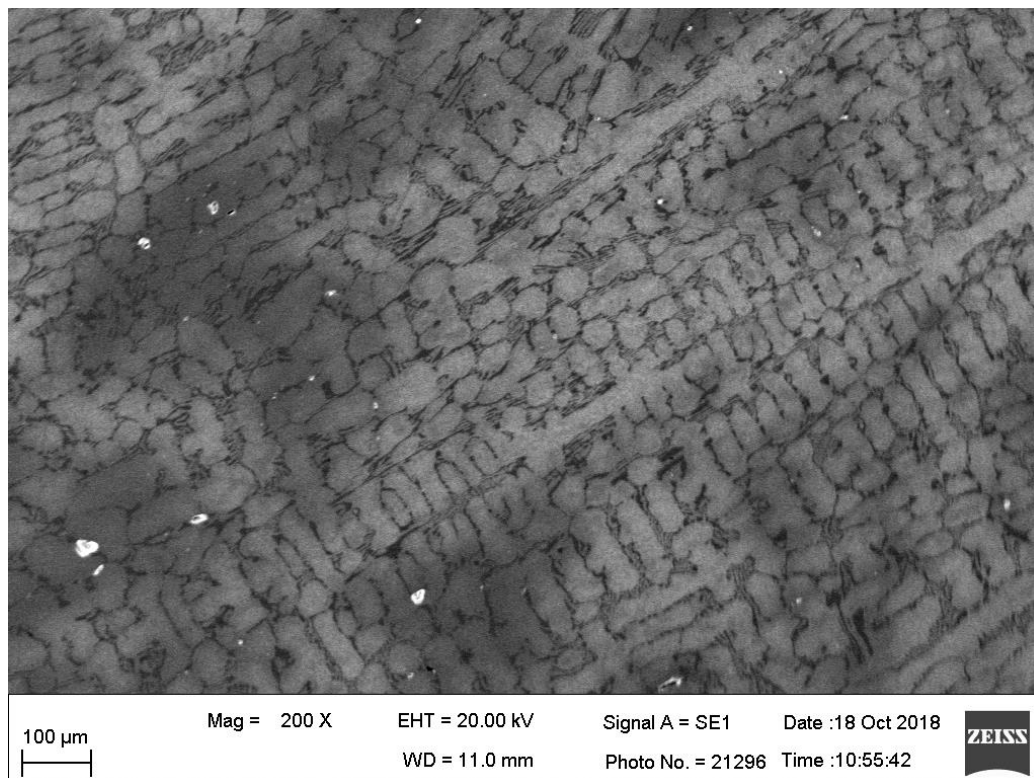
value. The Standard determines the nickel content at a level 0.5-1.5% and the lack of vanadium in the chemical composition of L210H21S cast steel, whereas it follows from other information (Dobrzański 2006) that “the L150HSM, L90HMF, L180H20F, L40H5MF, L75HMF, L120HNMF, L210H21S types containing no nickel contain, apart from chromium, molybdenum at a level 0.15-1.6%, the enhanced content of silicon 0.3-1.7% and vanadium”.

## METHODS AND MATERIALS

The investigations were performed using 4 defibrator plate segments applied in the process of fiberboard manufacture by the wet process. The plates were cast by the order of the Research & Development Centre for Wood-Based Panels Ltd. in Czarna Woda (Poland) with long tradition in the design and research of this type machine parts (Przegląd Techniczny 2009). Two types of segments “A” and “B” of a thickness of 3 to 5 cm were applied. It follows from an information of the supplier that they were cast from a high chromium and carbon content cast steel.

- A-1 - high alloy tool cast steel of L210H21S type (Polish Standard PN-90/H-83161), hardened to a Surface hardness of about 55 HRC,
- A-2 - high alloy cast steel of non-standard and undefined chemical composition,
- B-1 - high alloy cast steel of non-standard and undefined chemical composition,
- B-2 - high alloy cast steel of non-standard and undefined chemical composition,

A typical microstructure of the investigated cast steel, revealed after etching the material surface for 20 s in a commercial Oberhoffer etchant (Krawiarz) is presented in Fig. 1.



**Figure 1.** The microstructure of the investigated cast steel

The samples for X-Ray EDS and hardness measurements were cut from several spots of the steel segments, primarily from variable geometry places where material and cast defects are most probable.

Fig. 2 presents the photographs of the two types of the studied segments showing their structure and the type of patterns. The places of sample cut out are marked with numbers and arrows. The added 30 cm scale reflects the size of the parts.

The samples were prepared in several stages. In the first stage, in view size, form, and hardness of the segments, the cutting of “long” samples (through the whole width of the segment), a high pressure water cutting with abrasive was used, since water cutting does not modify the structure of the processed material. The water pressure during cutting was 360 MPa. A Garnet mesh 80 endure abrasive was used. The total cutting length was close to 5 m.

In the second stage, the “long” samples were cut transversally into smaller fragments, suitable for their processing (lapping, polishing) and study. At this stage the samples were cut with a grinder. A relatively small amount of the cut material and an intense cooling made it possible to reduce the material temperature to less than 100°C, i.e. much below a temperature where essential material microstructure modifications may occur. In view of the sample size and form, they were resin included after cutting.

In the third stage, the samples were manually lapped. Preliminary lapping was performed with diamond abrasive coated plates (220 granularity in resin) and SiC abrasive papers of 320 granularity (in water). Finishing lapping was performed using plates and water suspension of diamond abrasive of 9 μm grain size. In this case, rotation speed of the plates was 150 r.p.m. The total lapping time was between 30 and 90 min., depending on the degree of surface development and material grindability.

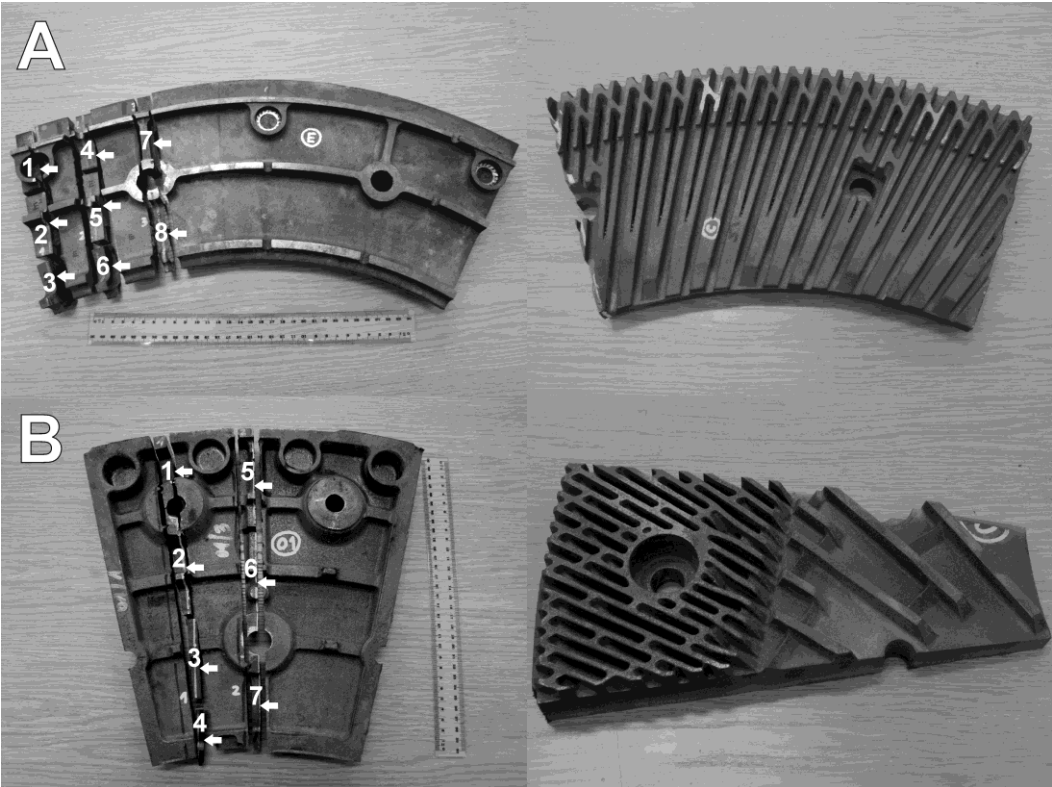


Figure 2. The photo of the types of investigated segments

In the fourth stage, the prepared samples were polished using polishing discs covered with acetate fiber woven fabric and a water suspension of diamond abrasive of a grain size up to 3 μm, and next, using polishing discs covered with synthetic fiber and a water suspension of diamond abrasive of a grain size up to 1 μm. In both cases, the rotation speed of the plates was 150 r.p.m. The total time of polishing individual samples was between 10 and 30 min. In view of complicated sample form, relatively high development of their surface and a high hardness

of the used materials, the processes of lapping and polishing were terminated at the moment when a major part of the samples under preparation has achieved a smoothness sufficient for investigations. The regions less essential from the viewpoint of investigations were left without preparation. At this stage, the samples were not etched.

In the next step, chemical composition of the prepared samples was determined using Scanning Electron Microscopy (SEM) equipped with EDS detector. A fast (300 s/measurement) less precise standardless ZAF method was used. It accounts for correction coefficients for atomic number Z, absorption A and fluorescence F (Mikroanaliza rentgenowska). This analysis was carried for typical spots on the sample surface at  $\times 1000$  magnification, i.e. for regions of a surface of  $269 \times 192 \mu\text{m}^2$ . The accelerating voltage was 20 kV. The range of 20 kV electrons in the sample material, as determined by an EDS dedicated Quantax 200, Esprit 1.9 (Quantax) program was about  $1.1 \mu\text{m}$  in all cases. In addition, the material density was determined on the basis of the material chemical composition.

Hardness of the defibrator plate segments was determined by Rockwell method in C scale (HRC), i.e. at a load of 1471 N (150 kG) and using a diamond  $120^\circ$  cone indenter. The measurements were carried using a semiautomatic hardness meter with electronic read-out of the measured hardness. The apply time of the preliminary load (98.07 N) was 8 s, the load time of the main force (1373 N) - 6 s and the time of indenter relief - 2 s. The hardness was determined in several tens of spots on the surface of the prepared samples at a distance of not less than  $2.5d$  from the sample edge and not less than  $4d$  between the indentations, where  $d$  is the maximum indentation diameter (Konowalski 2005). No measurements were taken for "thin" or unstable parts e.g. in the vicinity of the segment pattern. The measurements were taken at room temperature.

## RESULTS AND DISCUSSION

The results of the chemical composition of the defibrator milling plate segments are presented in Table 2. In each case, the values are the averages of three measurements, rounded to the second decimal place. Gaseous elements of the material are not included in the analysis.

**Table 2.** Chemical composition and density of the investigated cast steels

	A-1	A-2	B-1	B-2
Iron, Fe	rest	rest	rest	rest
Chromium, Cr	16.95%	21.12%	19.97%	18.21%
Carbon, C	5.57%	5.63%	5.51%	5.15%
Manganese, Mn	0.62%	0.39%	0.45%	0.74%
Silicon, Si	0.38%	0.26%	0.52%	0.42%
Molybdenum, Mo	0.27%	-	0.03%	0.59%
Nickel, Ni	0.07%	0.04%	0.04%	0.14%
Titanium, Ti	-	0.14%	0.2%	0.02%
Selenium, Se	-	-	0.05%	0.04%
Vanadium, V	-	-	-	0.19%
Aluminum, Al	-	-	-	0.03%
Sulfur, S	0.08%	0.02%	-	0.02%
Phosphorus, P	0.02%	0.01%	0.02%	-
Density ( $\text{g}/\text{cm}^3$ )	7.42	7.38	7.38	7.43

It is easy to note that the composition of Material A-1, declared by the manufacturer as L210H21S cast steel deviated from the composition described in PN-90/H-83161 Standard. Moreover, the content of carbon is surprisingly high in all cases. Several factors may be responsible for this state:

- the chemical composition at the sample surface (in our case the composition of a layer of a thickness of about 1.1  $\mu\text{m}$ ) may not be identical with the composition in the bulk, in view of sorption effects, surface oxidation, presence of adventitious carbon in the form of e.g. organic compounds etc.,
- conversion of EDS peak intensity into the element content should be treated as a rough estimation unless accurate calibration using appropriate standards is performed,
- in the case of research performed in standardless quantitative analysis, one applies the matrix ZAF correction and 100% standardization to the identified elements. Hence, if some elements are excluded from analysis, concentration of the remaining ones is artificially enhanced,
- additional peaks may appear in the EDS spectrum or a change in the height of the peaks may occur (and the content of the analyzed element may change) as a result of complicated interaction of electrons with the generated X-ray radiation,
- the peak width may increase as a result peak superposition associated with the detector resolution (Słowik 2012),
- the atoms of light elements from boron to neon (thus including carbon) have only two orbits occupied by electrons and thus only one K emission series for which the excitation energy is very low (1 keV at max.), i.e. all their peaks are contained within a low resolution part of the spectrum, where the individual peaks are superimposed on one another. Additionally, determination of the percent content of light elements by EDS is difficult as their characteristic radiation is absorbed more effectively by the sample material and their correction factor ZAF is much larger than one (Wassilkowska et al. 2014). The carbon content is estimated rather than calculated, in agreement with the program information: „element determined via net-count ratios”),
- the mass sensitivity of EDS analysis depends essentially on the signal to background ratio and hence it is difficult to separate the peaks of trace elements from the background. For concentrations of order of tenth of percent and below, the peak intensity exceeds the background by only about 30-50%. Errors in background subtraction may result from its different intensity on both sides of the peak. Therefore the background calculations include dependence of the continuous spectrum on the atomic number of the element (Wassilkowska et al. 2014).

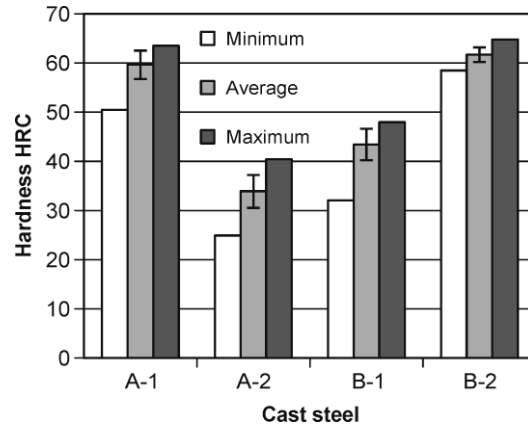
The values of material density, calculated by the program of EDS spectrum analysis were comparable for all investigated cast steels and lower by about 0.2 g/cm<sup>2</sup> in comparison with the L210H21S cast steel.

Table 3 presents the number of measurement points, the minimum, the average and the maximum values of hardness and the values of deviation and standard error for all investigated materials. Fig. 3 shows the trends in hardness changes. The vertical sections at the average hardness bar mark „ $\pm$  standard deviation”.

Among the studied materials, B-2 and A-1 defibrator segments, containing the maximum (among the determined ones) numbers of alloy components, exhibit the highest (above 60 HRC) and comparable hardness. These materials feature the lowest content of chromium (18.21 and 16.94%, respectively), which increases hardenability and the abrasive wear resistance, the highest content of manganese (0.74 and 0.62%), which neutralizes the detrimental effects of sulphur and oxygen in cast steel, the highest content of molybdenum (0.59 and 0.27%), which increases hardenability, strength and creep resistance and reduces the cast steel brittleness, and the highest value of nickel (0.14 and 0.07%), which reduces the temperature of brittle state transition.

**Table 3.** Results of hardness measurements

	A-1	A-2	B-1	B-2
Number of measurement points	102	97	75	46
Minimum HRC value	50.52	24.91	32.06	58.44
Maximum HRC value	63.46	40.43	47.94	64.78
Average HRC value	59.67	33.91	43.45	61.73
HRC standard deviation	2.86	3.33	3.22	1.59
HRC standard error	0.28	0.34	0.37	0.24

**Figure 3.** Hardness of the tested defibrator segments

The hardness of B-1 and A-2 segments was about 43 HRC and 34 HRC, respectively, i.e. it was lower by about 30 and 40%, respectively in comparison with the former materials. The hardness of A-1 cast steel with the lowest number of alloy components was only 3.3% lower in comparison to B-2 cast steel with the highest number of components. As mentioned before and according to the manufacturer declaration, A-1 is a normalized L210H21S type.

The paper (Xiang et al. 2003), presents the hardness data for 4 different plate materials containing iron, carbon, manganese, chromium, nickel, molybdenum and for two cases containing additional vanadium. The highest hardness at a level 57.5 HRC is exhibited by material of a composition (apart from iron and impurities): 2.5-2.8% C, 0.7-1.2% Si, 0.4-0.8% Mn, 22-28% Cr, 0.6-1.1% Ni, 0.6-2.0% Mo and 1.0-4.0% V. On the other hand, the hardness of material differing only in vanadium content ( $\leq 0.4\%$  instead 1.0-4.0%) was at a level of 52 HRC. The lowest hardness value, i.e. at a level of 48.5 HRC was obtained in material of a composition: 1.77% C, 1.03% Si, 0.73% Mn, 24.5% Cr, 1.02% Ni and 0.89% Mo. A reduction of chromium content from 24.5% to 17.5% and an increase of nickel content from 1.02% to 1.75% (i.e. for a composition: 1.53% C, 1.15% Si, 0.9% Mn, 17.5% Cr, 1.75% Ni and 0.89% Mo) results in the hardness increase to 53 HRC.

The Authors of another publication (Slavković et al. 2013) present the results of hardness measurements for cast steel plates of a composition: 3.54% C, 0.71% Si, 0.42% Mn, 2.89% Cr, 0.048% Cu, 0.173% Mo, 3.85% Ni and 0.022% V, as well as 3.38% C, 0.79% Si, 0.915% Mn, 2.51% Cr, 0.178% Mo, 4.03% Ni, 0.02% V and 0.123% W. The hardness of these materials was at a level of 56 and 60 HRC, respectively.

The WO 97/40204 patent presents an alloy referred to as EX05 of a composition: 0.2-0.4% C, 0.5-1.5% Mn, 0.5-1.5% Si, max. 0.05% S, max. 0.05% P, 14-18% Cr, 2.0-5.0% Ni, 2.0-4.0% Cu, max. 1.0% Mo and 1.5-2.5% Nb. The alloy hardness in the initial state was 35 HRC and increased to 42 HRC after a heat treatment.

The WO 01/68260 patent describes an alloy of a composition: 2.96% C, 0.77% Si, 0.82% Mn, 24.2% Cr, 5.16% V, 0.04% Mo and 0.03% Ni attaining hardness 57-63 HRC after heat treatment.

The chromium content described in Ref. (Xiang et al. 2003) was close to the values obtained in the present publication, but at the same time it was several times higher than that presented in publication (Slavković et al. 2013), though the results of hardness measurement were similar to one another. It follows that differences in the material hardness result from material structure rather than its composition. By the way of an example, the material of highest hardness (57.5 HRC), presented by Xiang (Xiang et al. 2003) exhibited martensite structure with dispersion carbide and the presence of second carbide. Material with 52 HRC hardness exhibited an austenitic structure with eutectic type M7C3 carbide (Garbiak i Piekarski 2010). The material with the lowest hardness 48.5 HRC exhibited a martensite structure with flack carbide precipitations whereas material with 53 HRC hardness exhibited martensitic structure with a net carbide. As it is known, the material structure is formed not only through an appropriate chemical composition but also through the proper course of technological process as e.g. appropriate thermal treatment minimizing the distortions of the working surface of the treated objects.

## CONCLUSIONS

The performed investigations of the defibrator plate segments demonstrate that from the hardness view point, a change of chemical composition and increasing the number of alloy elements are unfavourable if their effect on material properties is not fully exploited. An increase of the number of alloy elements, in comparison to the normalized L210H21S cast steel leads to a hardness increase only in one case and only by about 3%.

The performed measurements prove also that the results obtained in national laboratories and companies are comparable or better as compared to those obtained abroad.

In general, one should notice that although it is generally believed that the material hardness contributes to its abrasive wear resistance, it has been already demonstrated (Xiang et al. 2003), that if one analyses hardness and abrasive wear resistance hardness in 4 different materials, one notices that there is no direct linear dependence between the abrasive wear resistance and hardness, and consequently, hardness is only an apparent (illusiv) determinant of the abrasion resistance.

Accordingly, the effect of alloy elements upon other defibrator plate material properties, e.g. brittleness, abrasion resistance, or corrosion resistance should be the subject of further study.

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**Streszczenie:** *Wpływ składu chemicznego na twardość segmentów tarcz mielących termorozwłóknarki.* W artykule przedstawiono wyniki pomiarów twardości materiału segmentów tarcz mielących termorozwłóknarki w powiązaniu z badaniami jego składu chemicznego. Badania składu chemicznego były prowadzone metodą Rentgenowskiej Spektroskopii Dyspersji Energii. Twardość była mierzona metodą Rockwella. Zwiększanie liczby dodatków stopowych, w porównaniu ze znormalizowanym staliwem L210H21S, powoduje wzrost twardości o ok. 3%. Zmiana składu chemicznego i zwiększanie liczby dodatków stopowych są nieefektywne z punktu widzenia twardości.

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