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## MANUFACTURING OF INTERMETALLIC ALLOYS BY CONVENTIONAL POWDER METALLURGY TECHNOLOGY AND TERMOCHEMICAL TREATMENT

### OTRZYMYWANIE SPIEKÓW MIĘDZYMETALICZNYCH TECHNOLOGIĄ KONWENCJONALNEJ METALURGII PROSZKÓW I OBRÓBKII CIEPLNO-CHEMICZNEJ

Variety of P/M alloys, which are characterized by optimized mechanical properties, find increasing applications on large scale in various industries. The type of application basically depends on the material compatibility vis-a vis application, which in turn, is based on the chemical nature and the P/M manufacturing route. Often, the P/M alloys are subjected to thermochemical treatment in order to increase their surface hardness and wear-resistance. In the present experiments P/M intermetallic alloys of the type FeAl and FeAlNi have been prepared by conventional powder metallurgy via compacting and sintering. The surface hardness was improved by nitriding. The mapping of the microhardness profiles and structural examination of nitrided P/M alloys have been performed. The influence of the technology parameters on the development of nitrided layers of P/M alloys is discussed.

Materiały spiekane charakteryzujące się optymalnymi właściwościami mechanicznymi znajdują szerokie zastosowanie w różnych gałęziach przemysłu. Rodzaj zastosowania przede wszystkim zależy od składu chemicznego tych materiałów oraz od procesu ich wytwarzania. Bardzo często stopy otrzymywane technologią metalurgii proszków poddawane są obróbce cieplno-chemicznej, ażeby podwyższyć ich powierzchniową twardość i odporność na ścieranie. Spieki międzymetaliczne typu FeAl i FeAlNi otrzymywano technologią metalurgii proszków w wyniku prasowania i spiekania. Powierzchniową twardość tych spieków podwyższono poprzez zastosowanie określonego procesu

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azotowania. W toku eksperymentów sporządzono profile twardości i przeprowadzono badania strukturalne tych materiałów. Szczegółowo przeanalizowano wpływ parametrów procesu technologicznego na tworzenie warstw azotowych na powierzchni spieków międzymetalicznych FeAl oraz FeAlNi.

## 1. Introduction

The sintered intermetallic compounds have been receiving much attention in recent years because of their very attractive features as a low density, excellent oxidation and sulphidation resistance, good mechanical properties and low cost. Until now, Fe-Al and Ni-Al intermetallic compounds have been manufactured by casting, rapid solidification, hot extrusion plus hot isostatic pressing, hot pressing, injection molding and reactive sintering [1–4].

Most other methods e.g. rapid solidification, hot extrusion, isostatic pressing successfully applied, but the producing costs are relatively high, therefore P/M technology is a reasonable technique for obtaining intermetallic compounds [4, 5]. P/M technology for fabricating intermetallic compounds has become a suitable technique in view of the advantages like of achievement microstructure homogeneity, minimization of segregation, near net shapes, and significance lower producing costs.

During sintering of Fe-Al powders, swelling and shrinkage have been observed. That behavior is explained by imbalance solubility ratio and by a large diffusivity difference between iron and aluminium. The swelling results by outward aluminium diffusion during heating. Additions of aluminium are made to ferrous alloys in order to form a fine dispersoid structure. Such dispersions prevent high temperature grain growth and provide improved creep resistance, excellent high temperature oxidation and improve mechanical properties [6–8]. Dilatometric experiments performed by Lee and German [6] explained sintering process of Fe-Al system. The swelling observed in iron-aluminium compacts is determined by the liquid reaction at the eutectic temperature of 928 K temperatures due to oxide films. The various reaction products continue to homogenize during heating above 1323 K leading to densification. The fine pores are able to close, but large pores close slowly during sintering temperature.

The amount of swelling depends on alloy composition (amount of aluminium), particle sizes, green density, heating rate, sintering temperature, sintering time and prealloying [6, 9–11].

Reactive sintering as a one process which has shown great potential for forming NiAl and Ni<sub>3</sub>Al [11–14]. In general, reactive sintering involves a transient liquid phase. The initial compact is composed of mixed powders, which are heated to a temperature where they react to form a compound product. Often the reaction occurs on the formation of a first liquid, typically an eutectic liquid at the interface between contacting particles.

Conventional powder metallurgy technology basing on mixing elementary powders, compacting and sintering — is very simply method and demands rather low manufacturing costs. Until now, experiments concerning fabricating of Fe-Al alloys via conventional powder metallurgy route have not been applied, therefore this paper as aimed at obtaining intermetallic compounds, type Fe-Al and Fe-Al-Ni through conventional powder metallurgy method by using elementary powders: iron, aluminium and nickel.

Because of sintered intermetallic compounds type Fe-Al-Ni ought to be applied as different kind of high performance structural parts, especially in aerospace, they have been subjected to thermo-chemical treatment (nitriding) so as to improve their surface properties like hardness and wear resistance.

## 2. Experimental and procedure

Elementary 99.5% iron, aluminium and nickel powders were used to investigations. Chemical composition of intermetallic alloys have been presented in table 1. The mixtures made from Fe, Al, Ni powders, having a pre-set chemical composition, were compacted at 300 MPa; afterwards, the compacts were sintered at about 923 K, during 15 minutes, in argon atmosphere and at 1503 K for 2 hours in vacuum. The used sintering parameters were chosen basing upon the performed dilatometric investigations of Fe-Al sinters and NiAl [6, 13, 14]. In turn, sinters were subjected to thermo-chemical treatment consisting of nitriding at the temperature

TABLE 1

Chemical composition of intermetallic alloys

Sample No	Chemical composition
Fe15Al	Fe-15 wt.%Al
Fe15.8Al	Fe-15.8 wt.%Al (stoichiometric composition of intermetallic compound type $Fe_3Al$ )
Fe20Al	Fe-20 wt.%Al
Fe25Al	Fe-25 wt.%Al
Fe15Al10Ni	Fe-15 wt.%Al-10 wt.%Ni
Fe15.8Al10Ni	Fe-15.8 wt.%Al-10 wt.%Ni (stoichiometric composition of intermetallic compound type $(FeAl)_3Ni$ )
Fe20Al10Ni	Fe-20 wt.%Al-10 wt.%Ni
Fe25Al10Ni	Fe-25 wt.%Al-10 wt.%Ni

range 793–843 K for 3 hours and at 903 K for 6 hours, using  $N_2$  or  $N_2+H_2$  (50%:50%) atmosphere.

Carried out the technological operations, the density and porosity of Fe-Al and Fe-Al-Ni sinters were determined. After the nitriding process, hardness test were performed ( $HV_{0.02}$ ) of the sinters and the specimens' hardness profiles were worked out. In order to determine changes in the morphology and to identify the phases of Fe-Al and Fe-Al-Ni sinters, structural examinations were performed, following the sintering and nitriding processes by using an light microscope and SEM, type Philips XL 30 with an attachment for X-ray microanalysis (EDX). Similarly, the was carried out an X-ray analysis of the phase composition from the specimens surfaces, following the nitriding process (the phase composition of the diffusion layer under nitriding), by means of a cobalt and copper lamp, in an X-ray diffractometer, type Philips PW 3020. There were applied two kinds of X-ray lamps such as to get information on the diffusion layer at its surface, as well as at a deeper level.

### 3. Results and discussion

An increase in consolidation and decrease in the porosity degree in the sinters Fe-Al depend on pressure, sintering temperature and the chemical composition of the specimens (table 2). Fe-Al specimens, compacted under 300 MPa and sintered at 923 K, for 15 minutes in argon atmosphere, exhibited a porosity level of 20–30%, that is approximately 80% of the theoretical value. Instead, Fe-Al specimens with a 10 wt.% nickel addition, compacted and sintered at the same conditions, reached a lower porosity level, that is 17–19%, what is 81–83% of the theoretical value. A similar effect was observed in Fe-Al and Fe-Al-Ni specimens, compacted under 300 MPa and sintered at 1503 K, for 2 hours in vacuum. For Fe-Al sinters, obtained in above mentioned technological conditions, the porosity level reached approximately 17% and in Fe-Al-Ni sinters — amount 15%. While comparing the investigation results of density and porosity of Fe-Al and Fe-Al-Ni specimens having different chemical compositions and prepared in different technological conditions (table 2), there may be found a distinct effect of nickel addition upon decreasing the porosity level of Fe-Al-Ni sinters. This observation corroborates the previous tests made [4]. The applied technology of powder metallurgy, consisting in mixing elementary Fe, Al, Ni powders having a determined chemical composition, compacting and sintering, has confirmed a possibility of obtaining Fe-Al and Fe-Al-Ni intermetallic compounds.

Structural investigations performed with an optical and scanning microscopes (Figs 1–4) have revealed that in Fe15Al, Fe20Al, Fe25Al samples sintered at 923 K, against the iron matrix there appear two intermetallic phases — a grey type: FeAl and a dark type:  $FeAl_2$ . Fe15.8Al specimens sintered at 1503 K consist of a light

TABLE 2

Influence of nickel addition on the decreasing degree of porosity and increasing densification of Fe-Al-Ni alloys

Chemical composition wt. %	Sintering conditions (temp./time/ atmosphere)	Density g/cm <sup>3</sup>	Porosity %	Chemical composition wt. %	Sintering conditions (temp./time/ atmosphere)	Density g/cm <sup>3</sup>	Porosity %
Fe-15 Al	923 K/15'/argon	4.72	22.7	Fe-15 Al-10 Ni	923 K/15'/argon	4.99	19.0
Fe-15 Al	923 K/15'/argon	4.69	23.2	Fe-15 Al-10 Ni	923 K/15'/argon	4.96	19.0
Fe-15 Al	923 K/15'/argon	4.62	24.4	Fe-15 Al-10 Ni	923 K/15'/argon	4.92	20.0
<b>Fe-15 Al (average)</b>	<b>923 K/15'/argon</b>	<b>4.68</b>	<b>23.4</b>	<b>Fe-15 Al-10 Ni (average)</b>	<b>923 K/15'/argon</b>	<b>4.96</b>	<b>19.3</b>
Fe-20 Al	923 K/15'/argon	4.45	21.7	Fe-20 Al-10 Ni	923 K/15'/argon	4.75	17.2
Fe-20 Al	923 K/15'/argon	4.58	19.6	Fe-20 Al-10 Ni	923 K/15'/argon	4.77	16.9
Fe-20 Al	923 K/15'/argon	4.54	20.3	Fe-20 Al-10 Ni	923 K/15'/argon	4.67	18.6
<b>Fe-20 Al (average)</b>	<b>923 K/15'/argon</b>	<b>4.52</b>	<b>20.5</b>	<b>Fe-20 Al-10 Ni (average)</b>	<b>923 K/15'/argon</b>	<b>4.73</b>	<b>17.6</b>
Fe-25 Al	923 K/15'/argon	4.21	20.9	Fe-25 Al-10 Ni	923 K/15'/argon	4.39	18.2
Fe-25 Al	923 K/15'/argon	4.16	21.9	Fe-25 Al-10 Ni	923 K/15'/argon	4.30	19.9
Fe-25 Al	923 K/15'/argon	4.10	22.9	Fe-25 Al-10 Ni	923 K/15'/argon	4.32	19.6
<b>Fe-25 Al (average)</b>	<b>923 K/15'/argon</b>	<b>4.16</b>	<b>21.9</b>	<b>Fe-25 Al-10 Ni (average)</b>	<b>923 K/15'/argon</b>	<b>4.34</b>	<b>19.2</b>
Fe-15.8 Al	1503 K /2h/vacuum	5.00	17.2	Fe-15.8 Al-10 Ni	1503 K /2h/vacuum	5.20	14.8
Fe-15.8 Al	1503 K /2h/vacuum	4.98	17.5	Fe-15.8 Al-10 Ni	1503 K /2h/vacuum	5.17	15.2
Fe-15.8 Al	1503 K /2h/vacuum	4.98	17.5	Fe-15.8 Al-10 Ni	1503 K /2h/vacuum	5.17	15.2
<b>Fe-15.8 Al (average)</b>	<b>1503 K /2h/vacuum</b>	<b>4.99</b>	<b>17.4</b>	<b>Fe-15.8 Al-10 Ni (average)</b>	<b>1503 K /2h/vacuum</b>	<b>5.18</b>	<b>15.1</b>

Type of intermetallic phases at the analyzing areas of selected FeAl and FeAlNi alloys, SEM with EDX

Sample No	Analyzing area	Elements, at. %	Intermetallic Phases	Matrix
			according Figs 3–5	
Fe15Al sintered at 923 K	(1) light area	Fe–98.21	----	Fe
		Al–1.79		
	(3) dark area	Fe–33.95	FeAl <sub>2</sub>	----
		Al–66.05		
Fe20Al sintered at 923 K	(1) light area	Fe–96.44	----	Fe
		Al–3.56		
	(2) grey area	Fe–54.71	FeAl	----
		Al–45.29		
	(3) dark area	Fe–31.57	FeAl <sub>2</sub>	----
		Al–68.43		
Fe25Al sintered at 923 K	(1) light area	Fe–95.76	----	Fe
		Al–4.24		
	(2) grey area	Fe–49.98	FeAl	----
		Al–50.02		
Fe15.8Al sintered at 1503 K	(1) light area	Fe–72.75	Fe <sub>3</sub> Al	----
		Al–27.25		
Fe15Al10Ni sintered at 923 K	(1) light area	Fe–23.96	Ni <sub>3</sub> Fe	----
		Al–3.97		
		Ni–69.14		
	(2) grey area	Fe–97.83	----	Fe
		Al–1.66		
		Ni–0.51		
	(3) dark area	Fe–32.97	FeAl <sub>2</sub>	----
		Al–64.30		
		Ni–0.73		

Sample No	Analyzing area	Elements, at. %	Intermetallic Phases	Matrix
			according Figs 3–5	
Fe20Al10Ni sintered at 923 K	(1) light area	Fe–16.83	Ni <sub>3</sub> (FeAl)	----
		Al–18.70		
		Ni–64.47		
	(2) grey area	Fe–79.67	----	Fe
		Al–17.06		
		Ni–3.27		
	(3) dark area	Fe–50.83	FeAl	----
		Al–45.41		
		Ni–3.76		
Fe25Al10Ni sintered at 923 K	(1) light area	Fe–7.01	Ni <sub>2</sub> Al <sub>3</sub>	----
		Al–13.14		
		Ni–79.85		
	(2) grey area	Fe–91.36	----	Fe
		Al–6.44		
		Ni–2.20		
	(3) dark area	Fe–52.59	FeAl	----
		Al–45.63		
		Ni–1.78		
Fe15.8Al10Ni sintered at 923 K	(1) light area	Fe–41.99	FeNi	----
		Al–1.13		
		Ni–56.89		
	(2) grey area	Fe–98.34	----	Fe
		Al–0.87		
		Ni–0.79		
	(3) dark area	Fe–27.51	FeAl <sub>2</sub>	----
		Al–64.35		
		Ni–8.14		

$\text{Fe}_3\text{Al}$  phase (Figs 3 a, c, d, table 3). In  $\text{Fe}_{15}\text{Al}_{10}\text{Ni}$ ,  $\text{Fe}_{20}\text{Al}_{10}\text{Ni}$  and  $\text{Fe}_{25}\text{Al}_{10}\text{Ni}$  sintered at 923 K there appear also two intermetallic phases — a light ones nickel rich:  $\text{Ni}_3\text{Fe}$ ,  $\text{Ni}_3(\text{FeAl})$ ,  $\text{Ni}_2\text{Al}_3$  and a dark ones type:  $\text{FeAl}$  and  $\text{FeAl}_2$  (Figs 3 b, 4, table 3). The presence of intermetallic phases was confirmed by X-ray diffractometry studies and additionally the following phases were found:  $\text{Fe}_2\text{Al}_5$ ,  $\text{FeAl}_2\text{O}_4$ ,  $\text{AlNi}$  (Fig. 6). The compositions of respective intermetallic phases base of on SEM observations with point analysis (EDX) are shown table 3 and Figs 4, 5.

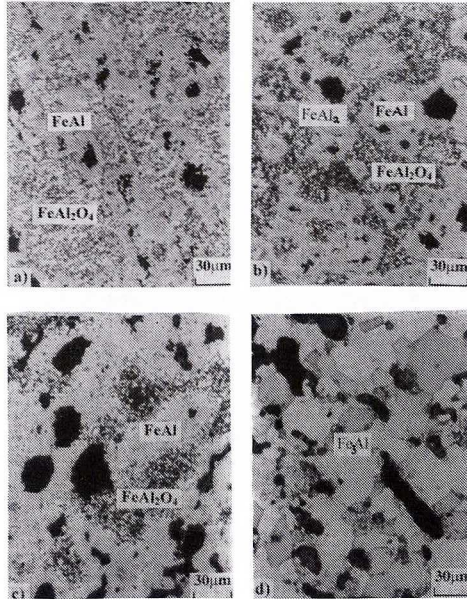


Fig. 1. Optical micrographs showing the microstructure taken from a)  $\text{Fe}_{15}\text{Al}$ , b)  $\text{Fe}_{20}\text{Al}$ , c)  $\text{Fe}_{25}\text{Al}$  sintered at 923 K, 15 minutes, argon atmosphere and d)  $\text{Fe}_{15.8}\text{Al}$  sintered at 1503 K, 120 minutes, vacuum

Comparing microhardness of some intermetallic phases in investigated samples it has been stated that a light intermetallic nickel rich phase is harder than a dark one — aluminium rich [11]. Hardness values of  $\text{FeAl}$  and  $\text{FeAlNi}$  alloys sintered at 923 K decrease with increasing of aluminium contents in the specimens (table 4).

After nitriding the formation of a diffusion nitrogen layer was observed. It was also established that the thickness of nitrogen diffusion layers depends on the chemical composition of  $\text{FeAl}$  and  $\text{FeAlNi}$  alloys (especially on aluminium contents and sintering temperature, (Figs 2, 3, table 5). The thickest, hardest and uniform (2488  $\text{HV}_{0.02}$ ) nitrogen diffusion layer was formed in the specimens of  $\text{Fe}_{15.8}\text{Al}_{10}\text{Ni}$ , sintered at 1503 K, for 2 hours, in vacuum. It has been stated, that  $\text{Fe}_{15}\text{Al}$  and  $\text{Fe}_{15}\text{Al}_{10}\text{Ni}$  specimens, sintered at 923 K, have no diffusion layers (Figs 2, 6, table 5).



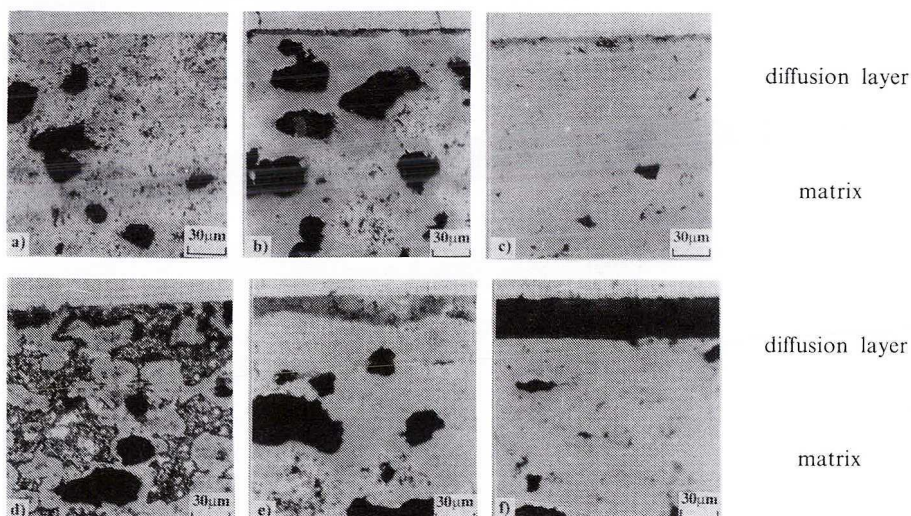


Fig. 2. Light micrographs showing the microstructure achieved in FeAl and FeAlNi samples sintered and nitrided at 793 K, 2 hours, ( $N_2 + H_2$ ) atmosphere a) Fe15Al sintered at 923 K, b) Fe25Al sintered at 923 K, c) Fe15.8Al sintered at 1503 K, d) Fe15Al10Ni sintered at 923 K, e) Fe25Al10Ni sintered at 923 K, f) Fe15.8Al10Ni sintered at 1503 K

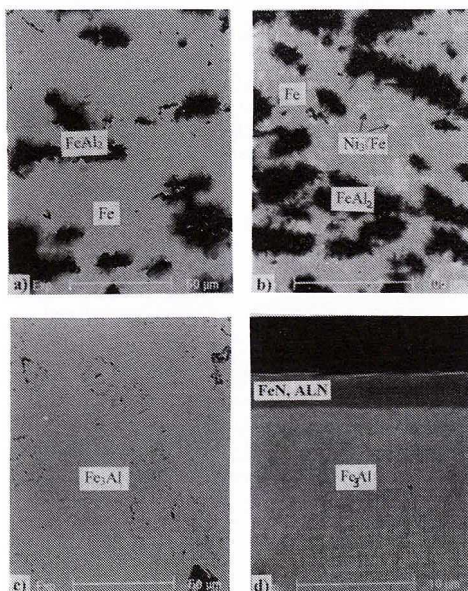


Fig. 3. SEM micrographs showing the topography in FeAl and FeAlNi samples a) Fe15Al sintered at 923 K, b) Fe15Al10Ni sintered at 923 K, c) Fe15.8Al sintered at 1503 K, d) Fe15.8Al sintered at 1503 K and nitrided at 793 K, 2 hours, ( $N_2 + H_2$ ) atmosphere

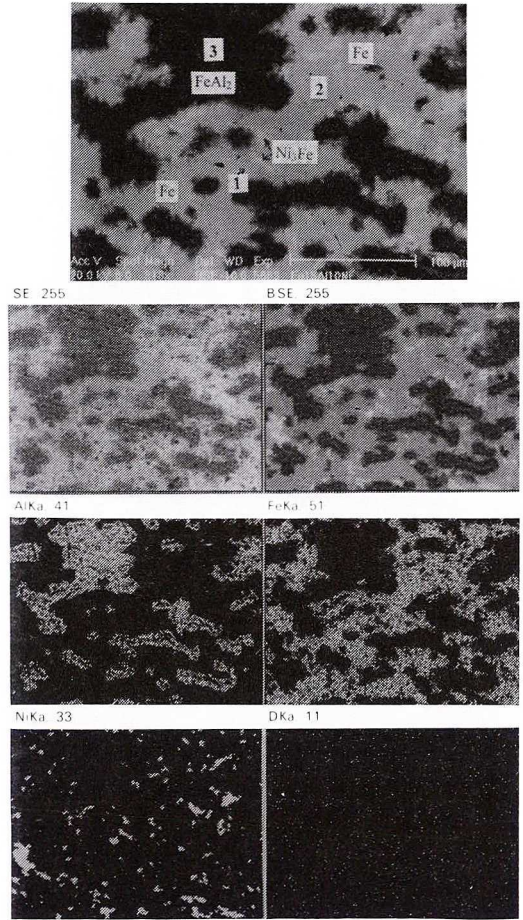


Fig. 4. SEM micrographs showing the topography and distribution of chemical elements in Fe15Al10Ni samples, sintered at 923 K, 15 minutes, argon atmosphere

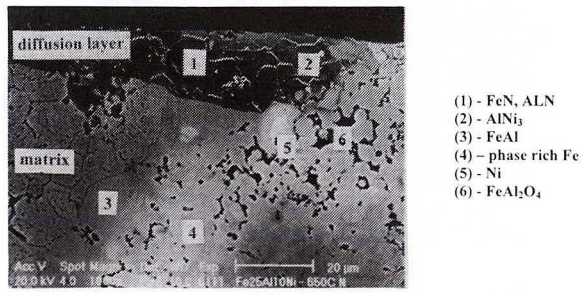


Fig. 5. SEM micrographs showing the topography and point analysis (EDX) in Fe25Al10Ni sintered at 923 K, 15 minutes, argon atmosphere and nitrided at 793 K, 2 hours, (N<sub>2</sub>+H<sub>2</sub>) atmosphere

TABLE 4  
 Hardness of FeAl and FeAlNi alloys  
 sintered at 923 K/15min./argon atmosphere

Sample No	Hardness, HV <sub>(10)</sub>
Fe15Al	68
Fe20Al	66
Fe25Al	65
Fe15Al10Ni	156
Fe20Al10Ni	119
Fe25Al10Ni	81

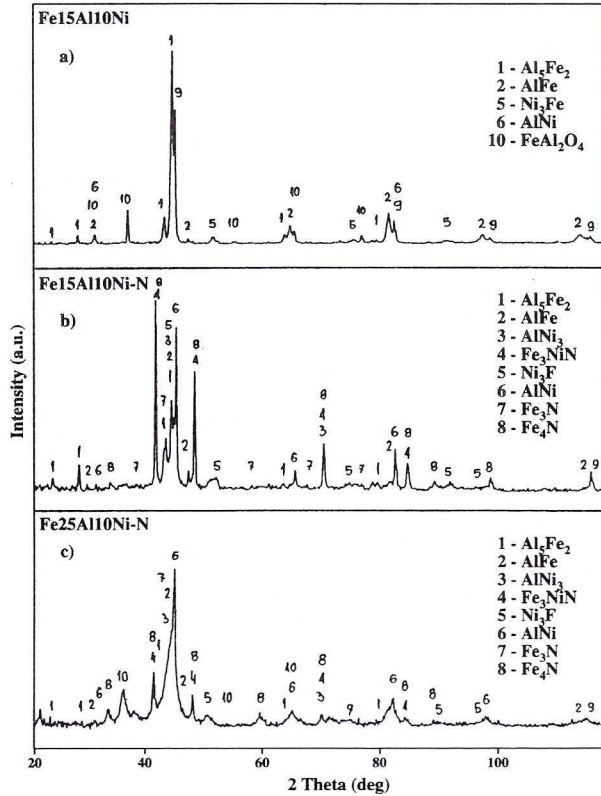


Fig. 6. X-ray diffraction patterns showing compounds in FeAlNi samples a) Fe15Al10Ni — sintered at 923 K, 15 minutes, argon, b) Fe15Al10Ni — sintered at 923 K, 15 minutes, argon and nitrided at 793 K, 2 hours, ( $\text{N}_2 + \text{H}_2$ ), c) Fe25Al10Ni — sintered at 923 K, 15 minutes, argon and nitrided at 793 K, 2 hours, ( $\text{N}_2 + \text{H}_2$ )

TABLE 5

Influence of chemical composition of samples on the thickness and hardness of diffusion layer

Sample No	Thickness of diffusion layer $\mu\text{m}$	Hardness of diffusion layer $\text{HV}_{0.02}$
Fe15Al sintered at 923 K	---	---
Fe25Al sintered at 923 K	7	2000
Fe15.8Al sintered at 1503 K	6	1300
Fe15Al10Ni sintered at 923 K	---	---
Fe25Al10Ni sintered at 923 K	30	1455
Fe15.8Al10Ni sintered at 1503 K	36	2488

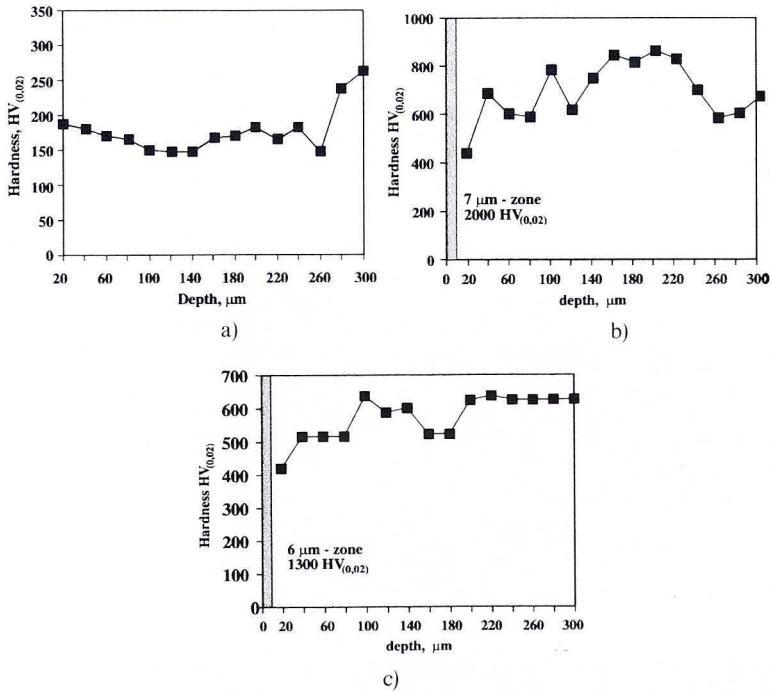


Fig. 7. Microhardness profiles of FeAl specimens a) Fe-15Al sintered at 923 K and nitrided, b) Fe-25Al sintered at 923 K and nitrided, c) Fe-15.8Al sintered at 1503 K and nitrided

The microhardness profiles  $HV_{0.02}$  of FeAl and FeAlNi specimens are illustrated in Figs 7, 8. A highest microhardness value of nitrided diffusion layers in FeAl sinters is caused by formation of  $Fe_3N$  nitride and intermetallic  $Fe_3Al$  phase, but a highest microhardness value of nitrided diffusion layers in FeAlNi samples is caused by formation of following nitrides:  $Fe_3N$ ,  $Fe_4N$ ,  $Fe_3NiN$  and intermetallic phase like  $Ni_3Fe$  (Fig. 6). The results are confirmed by a point phases analysis (SEM and EDX) — (Figs 3–5) and an X-ray phase analysis (Fig. 6). In the matrix of the nitrided FeAl specimens were found following intermetallic phases: FeAl,  $Fe_2Al_5$  and  $Fe_3Al$  but in the matrix of nitrided FeAlNi samples were formed intermetallic phases like: FeAl,  $Fe_2Al_5$  and  $AlNi_3$ , AlNi (Figs 5, 6). It is interesting that irrespectively of the distance from the diffusion interface, an equal quantity of Fe and Al nitrides were observed. It means that FeAl and FeAlNi specimens were thoroughly nitrided.

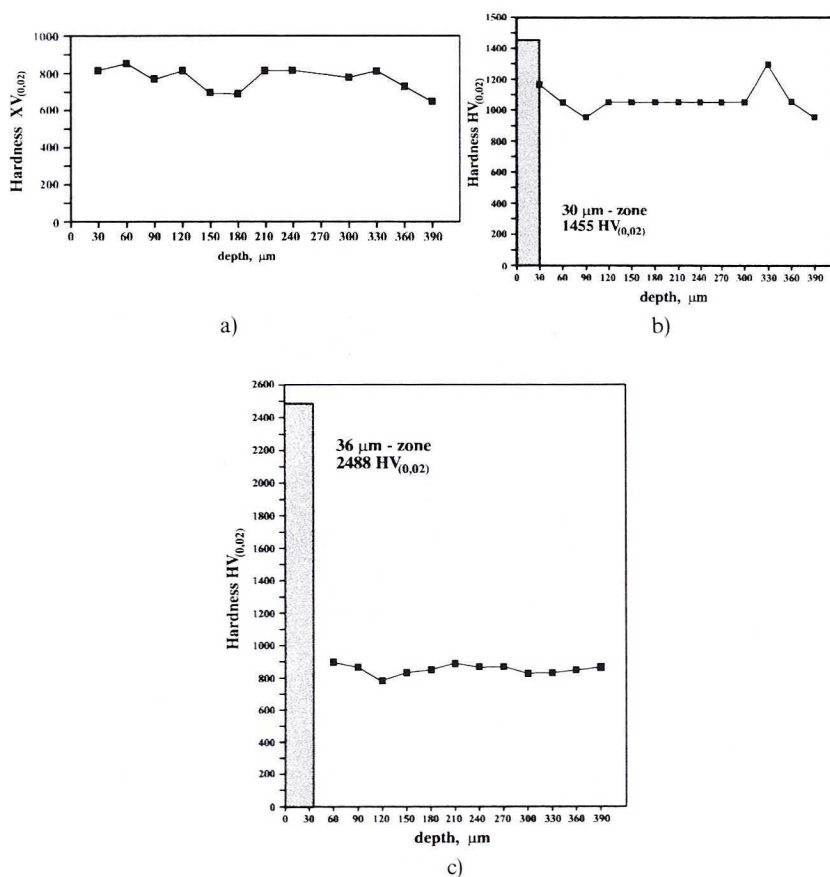


Fig. 8. Microhardness profiles of FeAlNi specimens sintered and nitrided a) Fe-15Al10Ni sintered at 923 K and nitrided, b) Fe-25AlNi sintered at 923 K and nitrided, c) Fe-15.8Al10Ni sintered at 1503 K and nitrided

#### 4. Conclusions

The present experiments carried out corroborated a possibility of obtained sintered intermetallic alloys, type Fe-Al and Fe-Al-Ni, via a conventional powder metallurgy by mixing elementary Fe, Al and Fe, Al, Ni powders, then compacting and sintering. Nitriding process of Fe-Al and Fe-Al-Ni samples allowed to form a very hard diffusion layers on the surface. The thickness of the diffusion layers depends on the aluminium contents in Fe-Al and Fe-Al-Ni samples and sintering temperature. Alloys of a stoichiometric composition — it means Fe<sub>15.8</sub>Al and Fe<sub>15.8</sub>Al<sub>10</sub>Ni sintered at 1503 K, during 2 hours, in vacuum are characterized by a lower porosity in the range of 15%–17%. The thickest and the hardest uniform diffusion layers were obtained for Fe<sub>25</sub>Al<sub>10</sub>Ni samples sintered at 923 K and for Fe<sub>15.8</sub>Al<sub>10</sub>Ni alloys sintered at 1503 K.

The experiments performed exhibited a possibility of using intermetallic alloys Fe-Al and Fe-Al-Ni for parts where high hardness and wear resistance is required.

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