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PLASTIFY OF ALLOY BASED ON THE MATRIX OF INTERMETALLIC FeAl PHASE**UPLASTYCZNIENIE STOPU NA OSNOWIE FAZY MIĘDZYMETALICZNEJ FeAl**

An alloy based on the FeAl intermetallic compound which can be hot worked was obtained thanks to proper choice of its chemical composition and production technology parameters. The main factor leading to the improved plasticity of the alloy is the deviation from the stoichiometry of the equilibrium intermetallic FeAl phase. The tendency to grain increase and strengthening of the grain boundaries was obtained by introducing of modifiers and interstitial atoms. The obtained alloy shows tendency to micropores creation, which was eliminated by the use of the oriented crystallization. The alloy was rolled in industrial conditions.

Skład chemiczny odpowiednio kształtowany i opracowanie warunków wytwarzania umożliwiły otrzymanie stopu na osnowie fazy międzymetalicznej FeAl, który można obrabiać plastycznie na gorąco. Do głównych czynników prowadzących do podwyższenia plastyczności należy odchylenie od stechiometrii równowagowego związku międzymetalicznego FeAl. Obniżenie skłonności do rozrostu ziarn oraz wzmocnienie granic ziarn uzyskano przez wprowadzenie do stopu modyfikatorów i atomów międzywęzłowych. Opracowany stop wykazuje skłonność do tworzenia mikroporów, którą usuwano przez stosowanie krystalizacji kierunkowej. Odlany stop walcowano w warunkach przemysłowych. Odształcenie plastyczne na gorąco różnicuje jednak strukturę stopu. Obok obszarów całkowicie zrekrystalizowanych występują obszary, które nie uległy zdrowieniu.

1. Introduction

Alloys on the matrix of iron aluminides, primarily FeAl phases, raise a high interest as construction materials for work at high temperatures in chemically

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aggressive atmospheres. This interest results mainly from their high resistance to wear, to gaseous corrosion and to a small density (compared with steels and nickel superalloys) while raw materials for their production are cheap and easily available [1–3]. These alloys achieve their properties basically thanks to properties structure revealing a long range order. It originates due to differences in the configuration and in physico-chemical properties of the main components, i.e. iron and aluminium.

Direct application of intermetallic compounds including FeAl is limited by their high susceptibility to brittle fracture and by a very low plasticity at room temperatures. Studies on reasons of such behaviour of materials with a long range order structure have shown three sources of low plasticity of alloys on the matrix of FeAl phase [4–9]:

1. an increase of alloys' brittleness caused by sensitivity to hydrogen brittleness,
2. a low strength of grain boundaries in long range order structures,
3. a susceptibility to point defect strengthening.

An environmental brittleness of the alloys is caused by the moisture contained in the air that is decomposed due to a high chemical affinity of aluminium to oxygen [10–15]. The other two reasons have a lesser contribution to a decrease of alloy plasticity at room temperature and may be easily removed by an appropriate choice of the chemical composition and by a proper heat treatment [11, 12, 17]. Where intercrystalline fracture dominates in alloys on the matrix of FeAl phase then a low strength of grain boundaries has a substantial share in the low plasticity. To remove brittleness connected with point defect strengthening a properly slow cooling from high annealing temperatures shall be used or performing an additional heat treatment [18].

The work undertaken aimed at designing and manufacturing an alloy on the matrix of intercrystalline FeAl phase suitable for hot plastic deformations.

2. Scope of investigations

The investigations carried out were directed towards a choice of alloy chemical composition in the view of increasing its plasticity and working out such conditions of the metallurgical process as to achieve an alloy suitable for hot plastic working. Hence the research tasks were reduced to the following objectives:

1. designing in an experimental way the chemical composition of an alloy on the matrix of intermetallic FeAl compound,
2. working out the recipe for melting the designed alloy,
3. characterising the structure and mechanical properties of the alloy in the as-cast state,
4. working out conditions of plastic working,
5. determining the alloy structure and properties after plastic deformation.

3. Preparation of material for studies

The choice of alloy's chemical composition on the matrix of intermetallic FeAl compound was started from melting in a laboratory induction vacuum furnace a stoichiometric FeAl phase. Its strength-plastic parameters obtained in a compression test at room temperature as well as hardness and grain size are specified in table 1. These results show a high brittleness of FeAl phase, the yield stress R_{ec} for the equiatomic phase is close to the strength R_{mc} . With reducing the aluminium content in a binary alloy within the range of FeAl phase stability, the yield stress falls compared with the value for the equiatomic phase. Unit elongation also increases, while no changes are observed in the hardness and the grain size. On the basis of data from table 1 one may assume that hardness depends stronger on the grain size than on the aluminium content in the alloy. A relatively large grain size results probably from application of directional solidification that was used to reduce the amount of casting defects.

TABLE 1

The course of chemical composition formation of the alloy designed on the matrix of intermetallic FeAl compound

Stages of designing	Alloy symbol	Mechanical properties in a compression test at room temperature			Hardness [HV]	Grain size [μm]
		$R_{0.2\epsilon}$ [MPa]	R_{mc} [MPa]	A_{ϵ} [%]		
I	Fe50Al	1311	1471	21	464	497 ± 250
	Fe45Al	996	1988	33	393	410 ± 190
	Fe40Al	503	1895	35	437	490 ± 300
II	Fe40A10.2Ti0.2B	417	2529	36	274	256 ± 50
III	Fe40Al5Cr0.2Ti0.2B	578	2702	42	256	232 ± 50
IV	Fe40Al5Cr3Ni0.2Ti0.2B	715	2616	42.5	253	235 ± 50

Modifiers were used to refine a coarse microstructure. The combination 0.2%Ti0.02%B reduces the average grain size from 490 μm to 260 μm . Microstructure refinement results in decreasing the hardness and yield stress. The subsequent stage of chemical composition formation of the alloy consisted in introducing Cr at the amount equal to the solubility limit, i.e. 5 at%. The action of this macro-alloying addition leads to an increase of relative shortening compared with the alloy without chromium. The average grain size also decreases and the compression strength increases. The addition of nickel to the designed alloy does not introduce a noticeable improvement of plastic properties; the yield stress increases compared with the alloy without nickel.

On the basis of the obtained results it was considered that the alloy Fe40Al5Cr0.2Ti0.02B (numbers given at the symbols are contributions of elements in the alloy expressed in atomic percents) has achieved the optimum chemical composition. It was assumed that at appropriate technology of melting the designed alloy may be formed plastically. The designed alloy was melted from pure components: ARMCO iron and the remaining components were used in the degree of chemical purity, as conditions of executing a heat in a laboratory vacuum furnace have limited possibilities of performing a deep refining of the melt. In the conditions of melting only an incomplete degassing could be performed under a reduced pressure. The pressure 10^{-2} — $5 \cdot 10^{-3}$ MPa used at melting of the designed alloy, due to the apparatus possibilities, appeared to be insufficient. Gaseous pores were found in the alloy and the hydrogen concentration in the as-cast state ranged from 1.00—1.37 ppm. This proves that metal was not completely degassed and despite the directional solidification, that was supposed to prevent creation of gaseous pores in the solidified metal.

Heats were cast in a vacuum atmosphere of the furnace to ceramic moulds hardened with various binders. Ingots for physical metallurgy examinations and tests of mechanical properties were in the form of ingots of diameter $\phi = 8-10$ mm and ingots for plastic working had diameters $\phi = (20-25) \cdot (350-400)$ mm.

The chemical composition of the designed alloys was specified in table 2. The contributions of individual alloying elements are given in weight percents.

After casting the alloy had a dendritic structure. To remove inhomogeneities of the chemical composition and of the structure the alloy was homogenised at 1050°C for 72h followed by cooling with the furnace to 600°C and then in the air to the room temperature.

TABLE 2

Chemical composition of the designed Fe-40Al-5Cr-0.2Ti-0.02B alloy

Alloy Fe-40Al-5Cr-0.2Ti-0.02B	Elements contents [wt. %]					
	Fe	Al	Cr	Ti	B	C
Type of element						
Assumed	69.50	24.47	5.00	0.2	0.02	—
Actual	68.21	23.66	5.77	0.15	0.015	0.046

4. Structure and mechanical properties of the alloy after homogenisation

The observation of microstructure was carried out on cross-sections perpendicular to axes of ingots and was performed by means of an optical microscope. It revealed a coarse grain microstructure after homogenisation. Grain boundaries have a zigzag shape (Fig. 1a). Moreover, the alloy preserved gaseous micropores. Some of them (internal) were revealed during examination of fracture surface on plastometric samples (Fig. 1b and c). The dendritic structure of the alloy in as-cast state was clearly revealed on the surface of fractures.

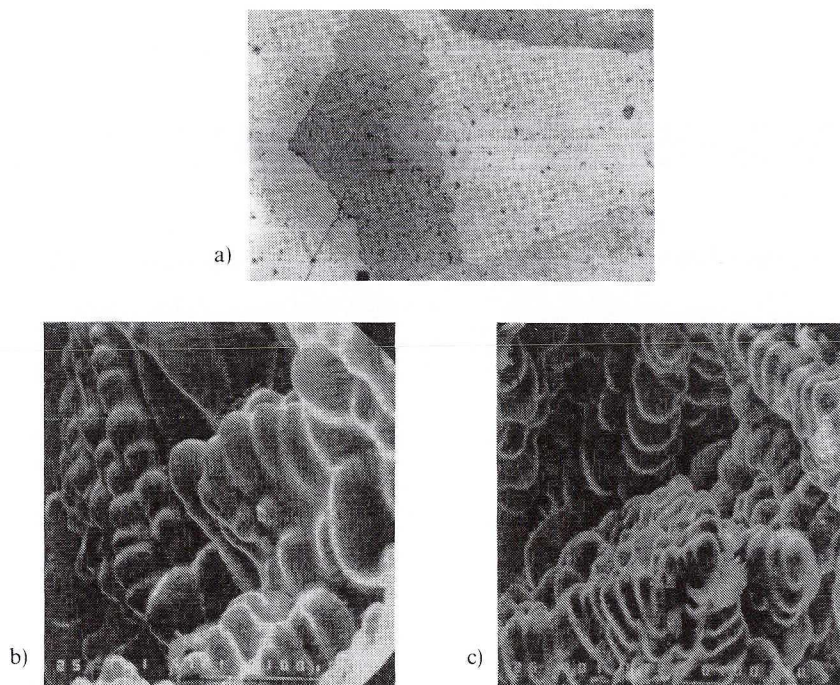


Fig. 1. Microstructure of Fe₄₀Al₁₅Cr_{0.2}Ti_{0.02}B alloy in the as-cast state after homogenisation: a — image of metallographic microsection magnification 80 ×, b — and c — fracture surfaces after plastometric tests at temperatures 1050°C

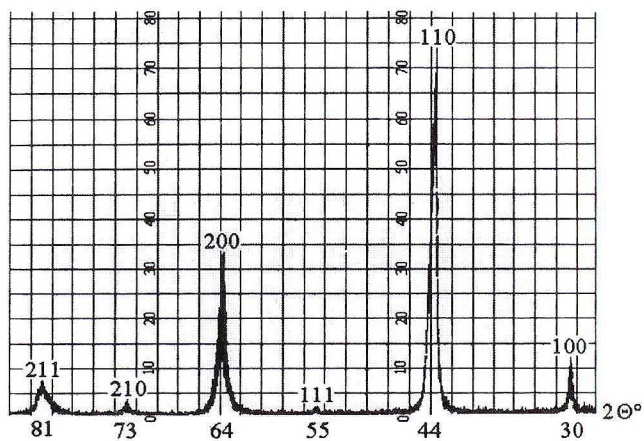


Fig. 2. X-ray diffraction pattern of Fe₄₀Al₁₅Cr_{0.2}Ti_{0.02}B alloy in the as-cast state after homogenisation

The existence of diffraction lines originating only from FeAl phase of CsCl type B2 structure was found in the diffraction pattern of the alloy (Fig. 2). Apart from basic lines of indices the sum of which is an even number, there are lines of sums of odd indices. Such a diffraction pattern in the as-cast state confirms its monophasic structure (the content of decomposition phases does not exceed an X-ray detection limit) and a high degree of long range order.

The monophasic state of the manufactured alloys was also confirmed by the analysis of a Mössbauer spectrum. The spectrum obtained for the alloy after primary crystallisation, as well as after homogenisation, contains only one spectrum line originating from a paramagnetic phase with a long-range order (Fig. 3). Parameters of this spectrum are listed in table 3. Average highest obtained values of isometric shift (IS) obtained for the studied alloy are smaller than the value reported in the literature for the binary FeAl alloy; for the compound IS = 0.23 mm/s. The difference

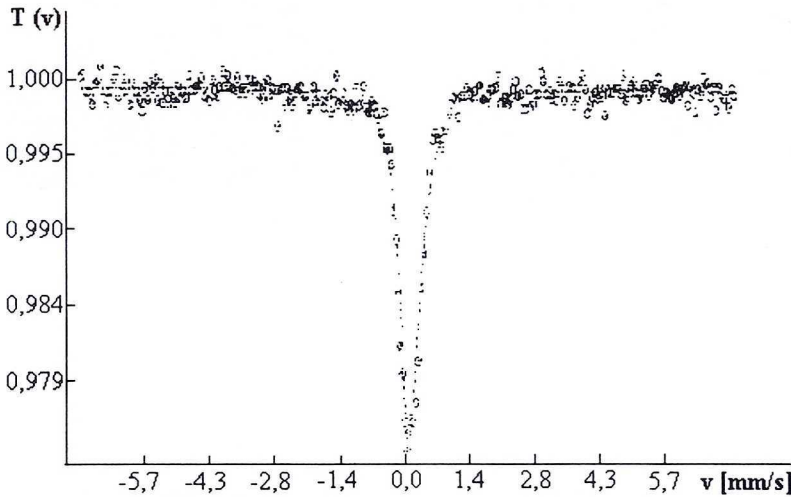


Fig. 3. Mössbauer spectrum of Fe₄₀Al₅Cr_{0.2}Ti_{0.02}B alloy in the as-cast state after homogenisation

TABLE 3

Parameters of the Mössbauer spectrum of the Fe₄₀Al₅Cr_{0.2}Ti_{0.02}B alloy around iron atoms

State of alloy	IS ¹ [mm/s]	G ² [mm/s]
As cast	0.186	0.56
After homogenisation	0.181	0.51
After hot rolling	0.178	0.51

¹IS — isometric shift,
²G — half-width of the line

of the isometric shift for the FeAl compound and for the studied alloy originates as a result of a stoichiometric deviation from the equiatomic composition of the FeAl compound towards the excess of iron in respect to aluminium and of the presence of alloying elements that change the environment (table 3).

The width of spectral line (G) suggests that homogenisation slightly increases the degree of long-range order compared with the state after primary crystallisation.

Investigations carried out on thin foils prepared from the alloy in the as-cast state after homogenisation have revealed trace amounts of secondary phases of two shapes:

1. irregular fine particles (Fig. 4),
2. large plates (Fig. 5).

Using the chemical composition of particles determined by means of TEM Quant spectrometer (table 4) the electronographic identification of precipitated phases was performed.

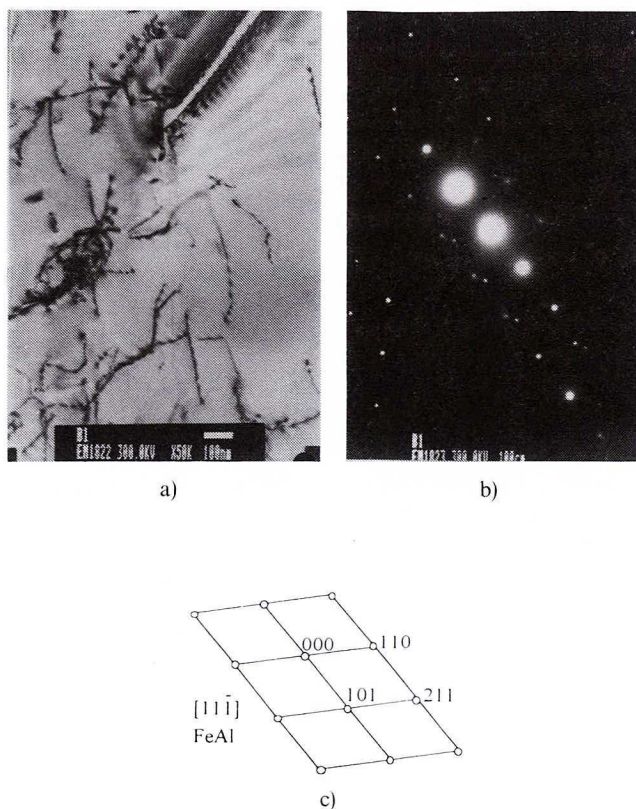


Fig. 4. Structure of Fe₄₀Al₅Cr_{0.2}Ti_{0.02}B alloy in the as-cast state after homogenisation: a — TEM image of thin foils, b — electron diffraction pattern, c — diagram with an electronographic identification

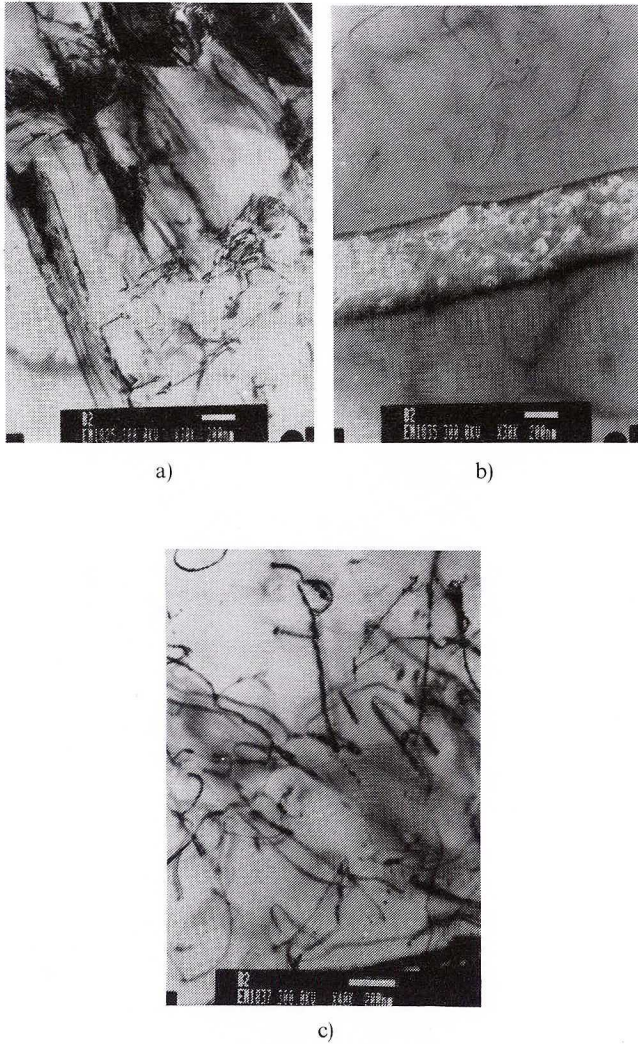


Fig. 5. Precipitations of secondary phases in Fe40Al15Cr0.2T; 0.02B alloy after primary crystallization: a. Plate precipitations; b. disperse precipitations in the grown twins; c. precipitations on the dislocations

Fine particles in which chromium was the dominating component were identified as carbide $M_{23}C_6$, while plate precipitates of a high titanium content as carbide TiC.

The distribution of dislocations far from precipitated particles is moderately homogeneous. Numerous dislocations lines are split into two components (Fig. 6). Some dislocations lines reveal themselves in the form of straight-line sections, some have got curved lines.

TABLE 4

Chemical composition of precipitates determined spectrometrically

Shape of particle	Al	Ti	Cr	Fe	Si	Al	Ti	Cr	Fe	Si
	% -weight					% -atomic				
Fine irregular particles	0.42	1.69	80.95	8.60	8.26	0.76	1.92	75.69	7.48	14.30
	1.95	1.40	85.56	10.94	—	3.71	1.50	84.58	10.07	—
	2.77	1.75	84.06	11.13	—	5.23	1.86	82.48	10.16	—
Average value	1.71	1.61	89.57	10.22	8.26	3.23	1.79	80.91	9.23	14.90
Plates	2.01	87.84	2.84	7.27	—	3.55	87.59	2.61	6.22	—

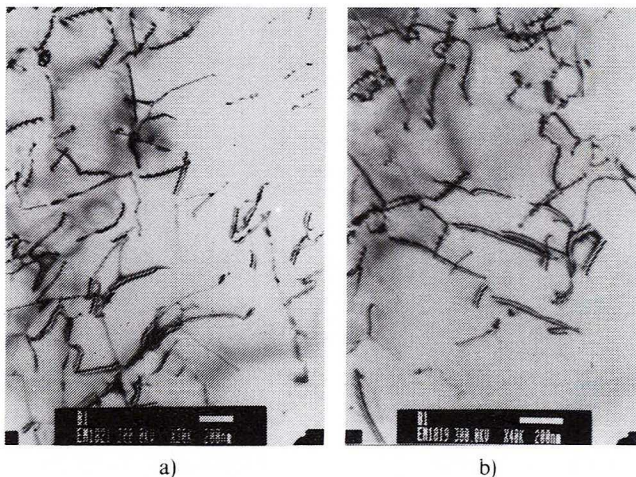


Fig. 6. Dislocation structure of Fe40Al5Cr0.2Ti0.02B alloy in the as-cast state after homogenisation

TABLE 5

Results of static tensile test for Fe-40Al-5Cr-0.2Ti-0.02B alloy after homogenisation

Temperature [°C]	R_m [MPa]	$R_{0.2}$ [MPa]	A_5 [%]	Z[%]
room	312.5	—	—	—
500	282.1	—	—	—
600	271.9	—	—	—
700	267.5	172.4	—	—
800	115.3	77.4	2.8	21.1
900	52.6	42.7	6.9	35.2

Results of a static tensile test of the alloy after homogenisation in the as-cast state are specified in table 5.

The studied alloy shows a very low ductility in the as-cast state. Its unit elongation at 900°C does not exceed 7%. Fracture surfaces of samples broken in a tensile test at 500°C and 700°C show a transcrystalline fissile appearance (Fig. 7a and b). Fracture surfaces of samples broken at 800°C and 900°C have slightly different appearance (Fig. 7c and d). Areas with some degree of plastic deformation occur in the surface of the latter fractures. The size of these ductile areas increases with increasing temperature up to 900°C. However, the areas of fissile fracture dominate despite the presence of ductile areas. The observation of fractures shows that a large mechanical energy is necessary to break samples.

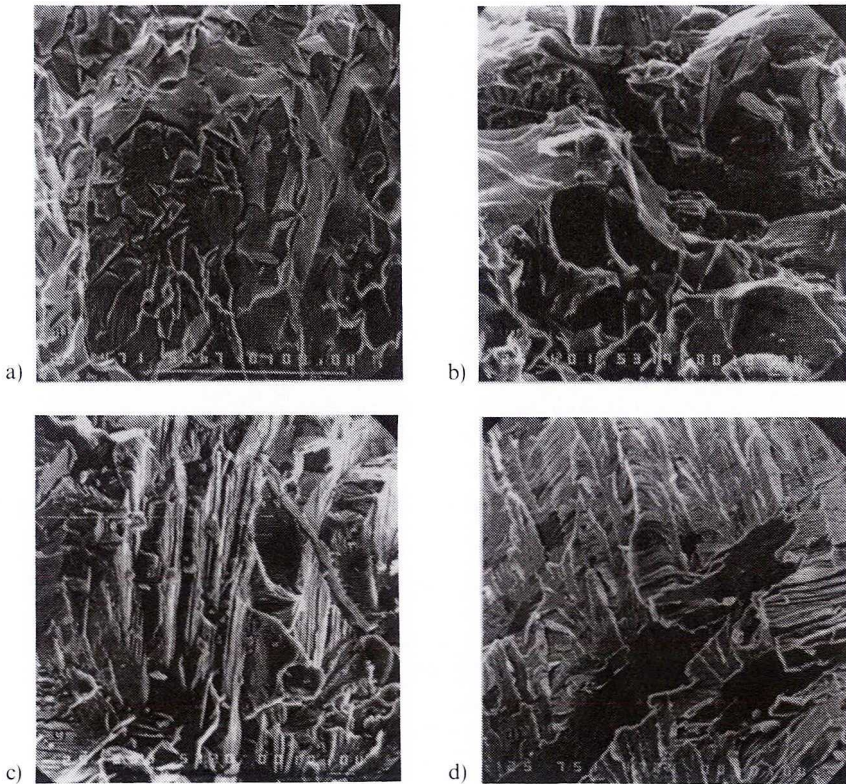


Fig. 7. Fracture surfaces of Fe40Al5Cr0.2Ti0.02B alloy in the as-cast state after homogenisation and subject to tensile tests at temperatures: a — 500°C, b — 700°C, c — 800°C, d — 900°C

5. Working out conditions of plastic working

To determine the lower limit of temperature, at which the plastic working of the designed alloy must be completed, plastometric examinations were carried out in the temperature range 800°C–1200°C. Behaviour of the alloy during the plastometric test was described with the following magnitudes: maximum intensity, strain at the maximum intensity, strain at fracture (table 6). From the data in table 6 it results that the studied alloy may be plastically deformed at temperatures higher than 1000°–1050°C. At lower temperatures the amount of plastic deformation is too small to have any importance for the process of plastic working.

TABLE 6

Results of plastometric tests

Test temperature°C	σ_{\max} MPA	$\epsilon_{\sigma_{\max}} * 10^3$	$\epsilon_{\max} * 10^2$
900	307	5.5	1.1
950	311	7.1	13.9
1000	160	9.1	17.0
1050	35	19.0	31.8

σ_{\max} — maximum stress,
 $\epsilon_{\sigma_{\max}}$ — strain at the maximum stress,
 ϵ_{\max} — maximum strain

To check the alloy's behaviour in a knock-down test samples 25 mm high and 20 mm in diameter were cut from an ingot and after heating up to the lower temperature from the range 1000°C–1050°C were compressed on cold anvils of a press. Samples were cracking on the circumference even at a minimum stress. Such a behaviour of the alloy induced to perform effort tests in "hoods" from austenitic steel heated together with the sample designed for plastic forming in a given temperature.

Deformation in hot hoods did not lead to origination of cracks. This observation provided the basis for the assumption that the designed alloy shall be worked in casings heated together with the charge material.

The designed alloy was rolled in industrial conditions of Huta Baildon (Baildon Steel Mill) on a shape mill. Ingots of 25 mm in diameter and 300 mm long after homogenisation were hammered down in tubes made from austenitic steel with 3 mm thick walls. The prepared charge was heated up in a gas-fired furnace up to about 1200°C–1250°C during 0.5–0.7 h and then rolled. Interoperational reheating was applied between passes. The rolling speed amounted to 1.5 ms⁻¹. A bar of 2–5 mm diameter was obtained from ingots after finishing the rolling and removing casings.

6. Structure and mechanical properties of the alloy after hot rolling

The alloy microstructure after hot working at temperatures higher than 1000°C assumes the form of idiomorphic grains with a large diversification of their size (Fig. 8). Fine grains occur beside large multilateral ones. The appearance of the observed microstructure suggests that the dynamic recrystallisation was not completed during the performed hot working. Average grain size amounts to about 100 μm . Fine particles of precipitates occur both on the boundaries as well as inside grains.

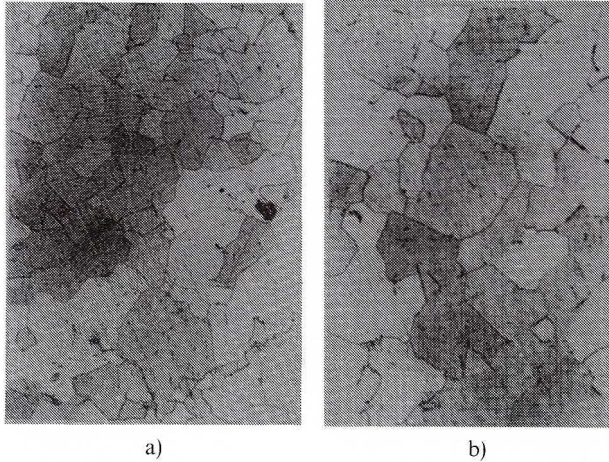


Fig. 8. Microstructure of Fe40Al5Cr0.2Ti0.02B alloy after hot rolling, a — magnification 80 \times , b — magnification 40 \times

Observations of thin foils by means of a transmission electron microscope (TEM) have confirmed the inhomogeneity of alloy's structure after hot rolling. Beside areas of total alloy's recrystallisation (Fig. 9a) there occur polygonised areas (Fig. 9b) and a substructure with the presence of low-angle boundaries (Fig. 9c) as well as areas with poorly visible effects of dynamic recovery (Fig. 9d).

TABLE 7

Results of static tensile test for Fe-40Al-5Cr-0.2Ti-0.02B alloy in the plastic worked state (8:1 degree of rolling)

Temperature [$^{\circ}\text{C}$]	R_m [MPa]	$R_{0.2}$ [MPa]	A_5 [%]	Z [%]
room	458.0	444.0	—	—
500	467.5	350.6	—	—
600	437.7	393.5	2.4	3.3
700	300.6	194.5	3.3	6.5
800	176.8	123.8	13.3	38.6
900	114.6	80.9	42.7	83.7

After plastic deformation the alloy remains still monophasic that was confirmed by *X*-ray and Mössbauer examinations. The amount of precipitates is probably smaller than the *X*-ray detection limit and the precipitated carbides are paramagnetic. The alloy's structure reveals a long-range order. The sequence of values of relative of diffraction lines corresponds with a theoretical diagram. This regularity indicates the lack of texture of the hot worked alloy. Parameters of the Mössbauer spectrum of the plastically worked alloy have the same values as in the as-cast state after homogenisation (table 3) what suggests that the plastic working changes the long-range order degree in an insignificant way.

Results of a static tensile test of the alloy Fe40Al5Cr0.2Ti0.02B after hot rolling are specified in table 7.

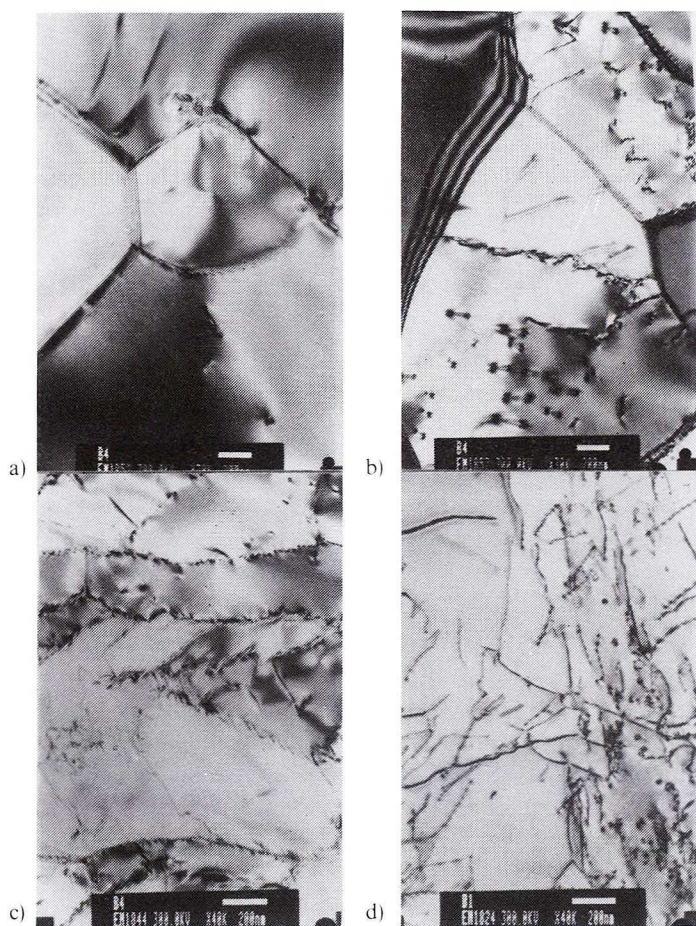


Fig. 9. Structure of Fe40Al5Cr0.2Ti0.02B alloy after hot rolling (TEM images of thin foils): a — formed and idiomorphic grains of the alloy, b — polygonised areas of the alloy, c — low-angle boundaries, d — uniform distribution of dislocations

After plastic working the alloy achieves some plasticity already during a tensile test at 600°C. So the plastic working reduces the temperature of plasticisation compared with the as-cast state, the character of fracture also changes. The fracture surface of the alloy broken in the as-cast state, in a tensile test at 500°C, has a transcrystalline fissile character without visible traces of plastic deformation, while after hot working traces of plastic deformation appear (compare Fig. 7a and Fig. 10a). Figure 10 presents fractures of samples from tensile tests of the alloy after hot working.

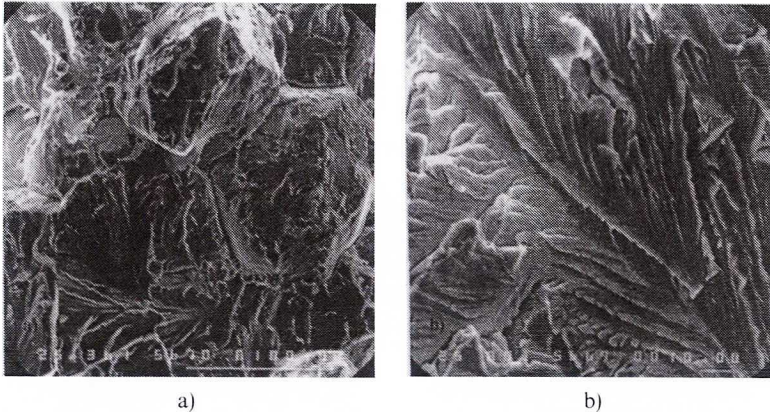


Fig. 10. Fracture surfaces of Fe40Al5Cr0.2Ti0.02B alloy samples after hot rolling and subject to a tensile test at temperatures: a — 500°C, b — 600°C

7. Discussion

The choice of chemical composition and a properly carried out metallurgical process enable manufacturing intermetallic alloys on the matrix of intermetallic FeAl compound that may be hot worked.

From among treatments modifying the chemical composition the strongest effect on the alloy's plasticity is exerted by deviation from equiatomic contribution of components in FeAl towards the excess of iron. With a decrease of aluminium contribution in FeAl from 50at% to 40at% Al the unit shortening has increased by 20% compared with the equiatomic FeAl phase. Modifiers (Ti + B) introduced to the Fe40Al alloy have caused a refinement of microstructure, which despite that remained still coarse grained. The average grain size fell virtually by a half. Besides, the character of fracture has changed from intercrystalline into transcrystalline fissile. The change of fracture character results probably from the increase of grain boundaries strength caused by a segregation of boron that is indicated by literature reports [19, 20] and by the presence of dispersed carbides. Further increase of Fe40Al0.2Ti0.02B alloy plasticity was achieved after introducing chromium. The unit shortening increased by 14% compared with the state without chromium. The

modified chemical composition of the alloy on the matrix of intermetallic FeAl compound was considered to be the optimum one in respect to plasticity. Apart from modification of the chemical composition of the designed alloy we have attempted to increase its susceptibility to plastic deformation by formation of its structure in metallurgical processes.

Performing melts in a laboratory vacuum furnace reduced using all possibilities of refining the melt, even though pure components were used for melting, but after solidification the alloy contained gaseous pores within the solid. Hence a higher than so far vacuum shall be used during melting and the charge before introducing to the furnace shall be dried. The moulds to which metal is to be cast in vacuum shall be dried. After solidification ingots revealed a high inhomogeneity that may be reduced by homogenisation.

We shall assume that removal of drawbacks of the metallurgical process will lead to further easing the susceptibility to plastic working of alloys on the matrix of intermetallic FeAl compound. Conditions for manufacturing products from these type of alloys will be created.

8. Conclusions

1. Choice of chemical composition of monophasic alloy on the matrix of intermetallic FeAl compound and formation of structure by means of metallurgical processes and heat treatment enable obtaining a material plastically workable at temperatures higher than 1050°C.

2. The structure of alloy after hot rolling reveals a high inhomogeneity in respect to the degree of advancement of dynamic recovery and recrystallisation processes. In the material there are areas after dynamic recrystallisation beside areas partially polygonised as well as areas without recovery effects that feature a high density of uniformly distributed dislocations. The diversification of structure proves that in metals with a long range order structure compared with disordered structures the plastic deformation occurs according to a separate mechanism.

3. The plastic working of Fe-40Al-5Cr-0.2Ti-0.02B alloy maybe performed by means of rolling homogenised ingots in austenitic casings in the temperature range 1250°C–1000°C. Interoperational reheating after each pass shall be used during rolling.

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