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NITROGEN IN Fe–Mn–Al–C–BASED STEELS

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ABSTRACT

Nitrogen and AlN solubility in Fe–Mn–Al–N, Fe–Mn–Al–C–N and Fe–Mn–Al–Ni–C–N alloys is calculated using the methods of interaction parameters and up-to-date thermodynamic data. The obtained values of balanced nitrogen solubility in these alloys are rather close to the convergence values obtained for liquid metal according to Thermo-Calc data base. Polythermic sections of the phase diagrams of these alloys are calculated and balanced residual values of dissolved nitrogen are determined for the liquidus and solidus temperatures; these temperatures are lower than nitrogen purity limited values, achieved by the modern technologies (0.0030%) for the most investigated alloys. It is possible to obtain up to 0.010% of dissolved nitrogen in the melts Fe–20%Mn–5%Al and Fe–25%Mn–5%Al at the temperatures of steel melting and alloying by nitrogen (1550–1600 °C), while in presence of carbon and with aluminium content more than 5% this value becomes lower than nitrogen purity limited value. Nitrogen content in a solid solution depends strongly on Al and C content. If Al content is 5%, the maximal nitrogen content in a solid solution Fe–20%Mn–Al can be equal to 9.3·10⁻⁴% at the solidus temperature. This value does not correspond to possibilities of the up-to-date industrial technologies. Thereby, AlN nitrides will form in liquid metal at micro-alloying of the steels of Fe–Mn–Al–Ni–C system by nitrogen, before and during crystallization. At the temperature lower 1100 °C, practically complete whole nitrogen is presented in the form of aluminium nitrides. It is displayed experimentally that complete nitrogen content in these alloys has effect on their strength (σ_{\max}) at hot deformation towards its elevation. In the case of warm and cold deformation, the tendency of lowering of alloys strength with increase of nitrogen content in these alloys is observed.

1. Introduction

The new materials should have more wide complex of properties compared with the existing ones, i.e. they should have high values of strength, ductility, corrosion resistance, structural stability and workability during production process, and that will add them the universal features in applications. High-strength alloys on the base of Fe–Mn–Al–C system, presenting a new group of so-called TRIPLEX alloys with high Mn and Al content, can be considered as prospective ones [1, 2]. The alloys on the base of Fe–Mn–Al–C system, having high content of Al, C and Si, are characterized by specific strength comparable with specific strength of light metal alloys [3]. In order to increase substantially specific strength, aluminium content should not be less than 5%¹ [4]. Rise of Al content higher than 5% ex-

pands the area of δ (α)-ferrite and supports the process of $\gamma \rightarrow \alpha$ transformation during cooling. Mn, Ni, C and N facilitate expanding of austenite area and austenite stabilization.

Nitrogen is an efficient element for austenite stabilization. The steels containing nitrogen are used more and more widely [5]. Alloying by nitrogen increases stability, strength and corrosion resistance, in particular the resistance to local corrosion kinds and intercrystalline corrosion of austenite steels [6].

This paper examines possibilities of nitrogen usage in steels on the base of Fe–Mn–Al–Ni–C system for hard solution and dispersion hardening.

2. AlN solubility in the alloys of Fe–Mn–Al–Ni–C–N system

AlN solubility in several liquid alloys of Fe–Mn–Al–Ni–C–N has been calculated using well-known thermodynamic parameters and Thermo-Calc data base.

¹ Here and below in the paper — mass. % (if another does not mentioned).

Equilibrium of dissolution reaction of solid AlN in the alloys of this system can be presented by the following equations:



$$K_{\text{AlN}} = a_{\text{Al}} a_{\text{N}} / a_{\text{AlN}} = f_{\text{Al}} f_{\text{N}} [\text{Al}] [\text{N}]. \quad (2)$$

The activity coefficients of [Al] and [N] during usage of the method of interaction parameters are determined by the equations of the following type (Taylor's expansion, 3)

$$\lg f_i = \sum_{j=2}^n e_i^j [j] + \sum_{j=2}^n r_i^j [j]^2 + \sum_{j=2}^n \sum_{k=2}^n r_i^{jk} [j][k] + \dots, \quad (3)$$

where $[i]$, $[j]$, $[k]$ — elements dissolved in liquid or solid metal; $i = \text{Al}, \text{N}$; $j, k = \text{C}, \text{Mn}, \text{Ni}$; a_i, f_i — activity and activity coefficient of i element in the solution; e_i, r_i — interaction parameters of 1st and 2nd order; K_{AlN} — equilibrium constant of (1) reaction.

Parameters e_{N} и r_{N} for N, C, Mn, Ni have been taken from [7], parameters $e_{\text{Al}}, r_{\text{Al}}, e_{\text{N}}^{\text{Al}}, r_{\text{N}}^{\text{Al}}$ and the value K_{AlN} have been taken from [8,9], parameters $e_{\text{Al}}^{\text{C}}, r_{\text{Al}}^{\text{C}}$ have been taken from [10]. Taking into account presence of interaction parameters e_i, r_i in the above-mentioned references and values of these parameters, we can obtain the equations for activity coefficients of aluminium and nitrogen in the alloys of Fe–Mn–Al–Ni–C system.

$$\begin{aligned} \lg f_{\text{N}} = & e_{\text{N}}^{\text{N}} [\text{N}] + e_{\text{N}}^{\text{C}} [\text{C}] + e_{\text{N}}^{\text{Mn}} [\text{Mn}] + e_{\text{N}}^{\text{Ni}} [\text{Ni}] + \\ & + e_{\text{N}}^{\text{Al}} [\text{Al}] + r_{\text{N}}^{\text{C}} [\text{C}]^2 + r_{\text{N}}^{\text{Mn}} [\text{Mn}]^2 + r_{\text{N}}^{\text{Ni}} [\text{Ni}]^2 + \\ & + r_{\text{N}}^{\text{C,Mn}} [\text{C}][\text{Mn}] + r_{\text{N}}^{\text{N,Mn}} [\text{N}][\text{Mn}]; \end{aligned} \quad (4)$$

$$\lg f_{\text{Al}} = e_{\text{Al}}^{\text{Al}} [\text{Al}] + e_{\text{Al}}^{\text{N}} [\text{N}] + e_{\text{Al}}^{\text{C}} [\text{C}] + r_{\text{Al}}^{\text{C}} [\text{C}]^2 \quad (5)$$

The results of calculations according to the equations (1–5) are presented on the **fig. 1**. Increase of aluminium content in a metal leads to strong decrease of nitrogen solubility at any content of other elements. If aluminium content is constant, carbon content and temperature have the most strong effect, while influence of manganese and nickel are essentially weaker. Nitrogen content in the solution decreases at carbon content rise and temperature lowering. At the melting temperature 1500–1550 °C and Al content $\geq 5\%$ in the liquid alloys of Fe–20%Mn–5%Ni–1%C system, only $\leq 0,005\%$ of nitrogen can be presented in dissolved state, that is close to nitrogen content introduced with charge material as impurities. It is displayed that reaching of nitrogen content $\leq 0,010\%$ in the low-carbon alloy Fe–20% Mn at the same temperatures and with aluminium content $\geq 5\%$ is possible.

Contents of Al and N balanced with AlN have been also calculated using Thermo-Calc data base (version 3.1). Thermodynamic base TCFE7 has been used. The results of both calculation methods are presented on the **fig. 2**.

Calculation according to the equations (1–5) provides higher values of nitrogen content in the solution. However, taking into account accuracy of interaction parameters and calculations based on the Thermo-Calc

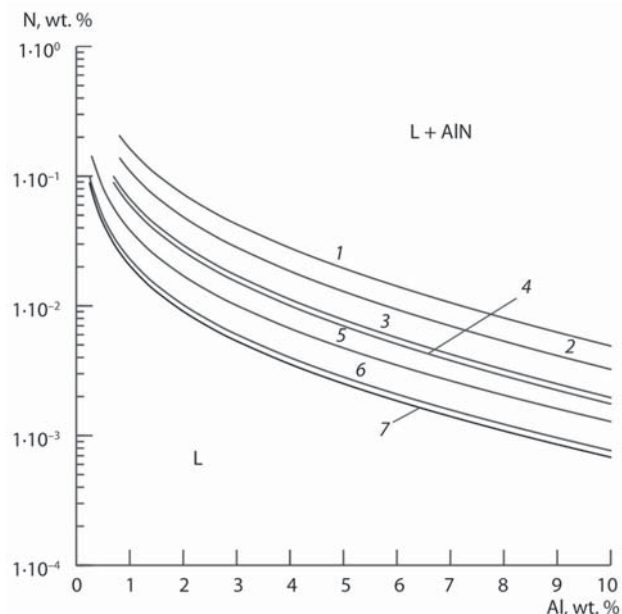


Fig. 1. Aluminium and nitrogen content in liquid metal balanced with AlN:

Alloys: 1 — Fe–25%Mn at 1600 °C; 2 — Fe–20%Mn at 1600 °C; 3 — Fe–20%Mn–1%C at 1600 °C; 4 — Fe–20%Mn–5%Ni–1%C at 1600 °C; 5 — Fe–20%Mn at 1500 °C; 6 — Fe–20%Mn–1%C at 1500 °C; 7 — Fe–20%Mn–5%Ni–1%C at 1500 °C

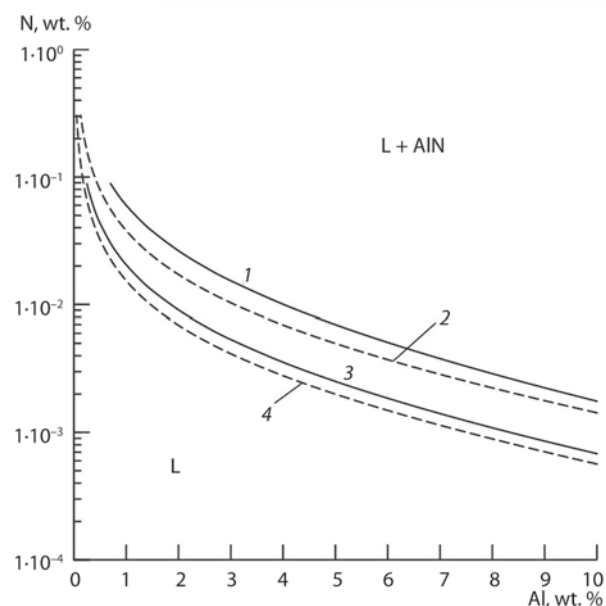


Fig. 2. Comparison of aluminium and nitrogen content in liquid metal, balanced with AlN, according to the two calculation methods:

1, 2 — Fe–20%Mn–5%Ni–1%C at 1600 °C; 3, 4 — Fe–20%Mn–5%Ni–1%C at 1500 °C. Calculation results according to the equations (1–5) — 1, 3; the results based on the Thermo-Calc data base — 2, 4

data base, the deviation can be accepted inside a methodical error. Thereby the data obtained via the Thermo-Calc data base can be used for evaluation of nitrogen content in liquid and solid alloys, when AlN is balanced with the melt at different temperatures.

Polythermic sections of several alloys of the Fe–Mn–Al–N, Fe–Mn–Al–C–N and Fe–Mn–Al–Ni–C–N systems have been built using Thermo-Calc data base. The polythermic section of the Fe–20%Mn–5%Al–N system is presented as an example on the **fig. 3**. Fig. 1 and 2 testify that temperature lowering leads to decrease of nitrogen content in the solution for all alloys. Rise of Mn content increases nitrogen solubility in the alloy, but at the same time liquidus and solidus temperatures decrease. Content of dissolved nitrogen in the alloys at liquidus and solidus temperatures are presented in the **table 1**. Nitrogen solubility in the alloy of the Fe–25%Mn–5%Al–N system at T_{liq} and T_{sol} is lower than in the alloy of the Fe–20%Mn–5%Al–N system (see table 1). This table displays that the alloy of the Fe–20%Mn–5%Al–N system is characterized by the most high nitrogen content $3.1 \cdot 10^{-3}\%$ at T_{liq} and $9.3 \cdot 10^{-4}\%$ at T_{sol} among all examined alloys. The **fig. 3** shows possibility of obtaining nitrogen content $\geq 0,007\%$ during melting of this alloy in liquid metal at the temperature $\geq 1550\text{ }^\circ\text{C}$. This is the adjustable level of content at micro-alloying. Nitrogen solubility of this alloy at the liquidus temperature makes $3.1 \cdot 10^{-3}\%$. This is an ultimate nitrogen purity level that can be achieved via up-to-date industrial technologies at present time. At solidus temperature, nitrogen content in the solution decreases for this alloy to $9.3 \cdot 10^{-4}\%$, while at the temperatures below $1100\text{ }^\circ\text{C}$ practically whole nitrogen is presented as AlN nitrides (**fig. 3**). For other alloys mentioned in the table 1, nitrogen content in the solution during crystallization is lower. Analysis of the phase diagrams displays that all examined (ferrite, binary and austenite) alloys include whole nitrogen as AlN nitrides at the temperatures lower than 1100 or $1200\text{ }^\circ\text{C}$. Thereby, nitrogen regulating effect on the properties of these alloys can be realized only via nitride phase, amount and size of nitrides. The higher is the formation temperature, the larger is nitrides size. Minimal amount and size of nitrides are observed for those nitrides that are forming in the solid solution, i.e. at the temperatures $\leq T_{sol}$; for this

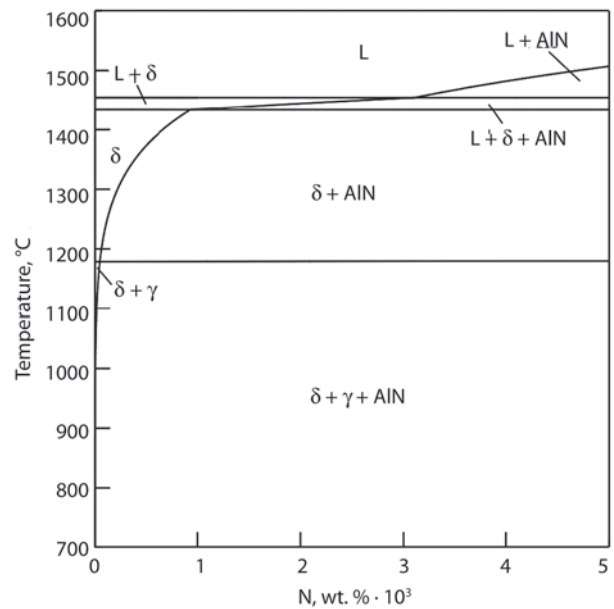


Fig. 3. Polythermic section of the state diagram for the alloy of the Fe–Mn–Al–N system an variable content of nitrogen and constant content of Mn = 20% and Al = 5%

purpose nitrogen content in the alloy should not exceed its solubility at T_{sol} at the melting temperature (see table 1). At present time such concentrations are not achievable technologically at $\text{Al} \geq 5\%$, as well as concentrations at T_{liq} (see table 1). In this connection, regulating effect on the properties of these alloys via nitride phase can be conducted by micro-alloying of these alloys, and obtaining nitrogen content in the metal not exceeding its balanced content between AlN and melt will be the most optimal variant. In this case nitrogen amount in the solid metal will correspond to its amount introduced during melting, subtracting nitrides removed out of liquid metal during cooling before crystallization and during crystallization itself. Only large inclusions are removed during this period, while small ones are forming in the solid metal. However, figure 1 displays that this variant is impossible for the alloys with high Al and C content, because the balanced nitrogen contents are too small and can't be achieved even at increased temperature above $1600\text{ }^\circ\text{C}$. Possibilities of temperature rise during melting of these alloys are restricted owing to strong manganese evaporation. Nitrogen amount added in such cases during alloying can be any possible and not depending on its balanced content, because it is determined empirically via the properties of finished metal and nitride phase. In this case controllability of this process will be rather small, because nitrides are forming and deleting out of the melt just during alloying.

3. Results of the experiment

Table 2 presents the alloys of Fe–Mn–Al–Ni–C system with different nitrogen content.

Table 1. Content of dissolved nitrogen in the alloys at liquidus and solidus temperatures

Diagram	T_{sol} , $^\circ\text{C}$	N, % (at T_{sol})	T_{liq} , $^\circ\text{C}$	N, % (at T_{liq})
Fe–20%Mn–5%Al–N	1434	$9.3 \cdot 10^{-4}$	1454	$3.1 \cdot 10^{-3}$
Fe–20%Mn–10%Al–N	1421	$3.8 \cdot 10^{-4}$	1439	$8.0 \cdot 10^{-4}$
Fe–25%Mn–5%Al–N	1412	$8.3 \cdot 10^{-4}$	1434	$2.7 \cdot 10^{-3}$
Fe–25%Mn–10%Al–N	1403	$3.5 \cdot 10^{-4}$	1420	$6.6 \cdot 10^{-4}$
Fe–20%Mn–1%C–5%Al–N	1288	$9.4 \cdot 10^{-5}$	1389	$7.5 \cdot 10^{-4}$
Fe–20%Mn–1%C–10%Al–N	1274	$1.2 \cdot 10^{-5}$	1377	$2.0 \cdot 10^{-4}$
Fe–25%Mn–1%C–5%Al–N	1277	$9.7 \cdot 10^{-5}$	1370	$6.3 \cdot 10^{-4}$
Fe–25%Mn–1%C–10%Al–N	1265	$1.8 \cdot 10^{-5}$	1360	$1.6 \cdot 10^{-4}$
Fe–20%Mn–1%C–5%Ni–5%Al–N	1270	$6.4 \cdot 10^{-5}$	1369	$5.1 \cdot 10^{-4}$
Fe–20%Mn–1%C–5%Ni–10%Al–N	1251	$1.2 \cdot 10^{-5}$	1361	$1.3 \cdot 10^{-4}$
Fe–25%Mn–5%Al–5%Ni–N	1376	$5.4 \cdot 10^{-4}$	1417	$1.9 \cdot 10^{-3}$

No.	Steel designation	Alloy group	Content of elements, %					
			Mn	Al	Ni	Si	C	N
1	39K	α -steels	23,7	7,5	<0,03	0,20	0,05	0,008
2	40K		22,6	14,4	<0,03	0,20	0,05	0,007
3	42K		23,7	8,1	<0,03	0,20	0,05	0,008
4	43K		24,3	11,5	<0,03	0,20	0,03	0,045
5	45K	γ -steels	23,8	4,6	<0,03	0,20	0,05	0,020
6	44K		24,1	5,3	<0,03	0,32	0,04	0,024
7	35K		21,2	6,2	<0,03	0,50	1,10	0,006
8	36K		19,1	7,5	<0,03	0,60	1,47	0,002
9	34K		23,9	4,0	<0,03	0,54	1,80	0,032
10	33K		19,1	9,0	<0,03	0,50	2,18	0,001
11	54K		24,1	4,93	4,71	0,22	1,47	0,0008
12	56K		20,8	8,91	4,69	0,30	1,48	0,135

Melting of 33K–45K alloys has been conducted in the laboratory vacuum resistance furnace with tungsten heater at the temperature 1600 °C via alloying of pure charge materials [11]. To mix and saturate the alloys, metal has been subjected to blowing through submerged pipe by argon and nitrogen, then samples have been taken in quartzite pipes with diameter 6–8 mm. The cylindrical specimens with diameter 5–7 mm and length 9.7–11.45 mm have been prepared from these samples. They were used afterwards for investigation in as-cast state or subjected to thermal or thermomechanical treatment with hot compression at 950–1000 °C (3 min holding, preset deformation degree ϵ at 50% testing) or with warm deformation at 550 °C with deformation rate $\dot{\epsilon} = 0.1 \text{ s}^{-1}$ along cylindrical axis, using “Gleeble System 3800” testing complex. Therefore, the deformation diagrams have been obtained, strength and deformability of cast alloys have been evaluated. After deformation the specimens have been cooled quickly

(with rate not less than 50 °C/s) using air nozzles. Mechanical properties at the room temperature have been evaluated in the “Instron 3300” testing system via bending tests until destruction or until practically complete parallelism of sides².

54K and 56K alloys are produced in the induction vacuum furnace. Pure materials have been used as charge. Metal has been cast in ingots with 9 kg mass, 68 mm diameter and 170 mm height.

Based on the results of investigations, nitrogen hardening effect manifests in hot deformation in the range of varying its concentration in α - and γ -alloys (fig. 4). Rise of nitrogen content leads to increase of maximal deformation resistance σ_{\max} both for low-carbon and high-carbon alloys. 33K alloy has anomalous high $\sigma_{\max} = 198 \text{ MPa}$ both at minimal nitrogen content 0.001%, as well as maximal carbon

content 2.18% and aluminium content 9%. The alloy structure after deformation was characterized as $\alpha + \kappa$ (kappa-carbide). Strong hardening of this alloy can be explained by kappa carbide extraction. It was shown above that all nitrogen in these alloys is presented by nitrides, and diffusion rate of elements in metal is rather high at 1000 °C and homogenization occurs during heating and deformation. All excessive phases (except nitrides) that are formed mainly in liquid state and at the temperatures above 1000 °C, are dissolving. Thereby AlN is the only hardening phase at 1000 °C, AlN amount is proportional to general nitrogen content in the alloy. Fig. 4 also displays the weak tendency of decreasing ϵ_{\max} value during hot deformation of α - and γ -alloys with increase of general nitrogen content.

For warm deformation of γ -alloys, σ_{\max} rises definitely with increase of carbon content and lowers slightly with increase of nitrogen content. The alloys with high carbon content and minimal nitrogen content (33K, 35K) have the most high σ_{\max} value. Carbides seem to be the main hardening phase at 550 °C, while nitrides make negative effect. It is shown that σ_{\max} for α -alloys increases with rise of aluminium content, while nitrogen influence was not revealed, as well as influence of the alloy composition on ϵ_{\max} value for warm deformation.

For cold deformation, maximal deformation resistance σ_{\max} and bending angle α have been determined. It was revealed that σ_{\max} also mostly decreases for γ -alloys with rise of nitrogen content, while nitrogen influence on the bending angle was not revealed. Low-carbon alloys 44K and 45K (128 and 130 ° respectively) have the highest α value. It is shown that α -alloys are characterized by brittle destruction.

The alloy 54K had low nitrogen content (0.0008%). Single-phase γ -solution for this alloy is located in the temperature range 900–1200 °C. Thereby metal temperature made 950–1100 °C during treatment for solid solution and during hot deformation. The ingot was subjected to homogenization at 1100 °C during 3 hours, then it was forged to 35 mm thickness (with intermediate heating),

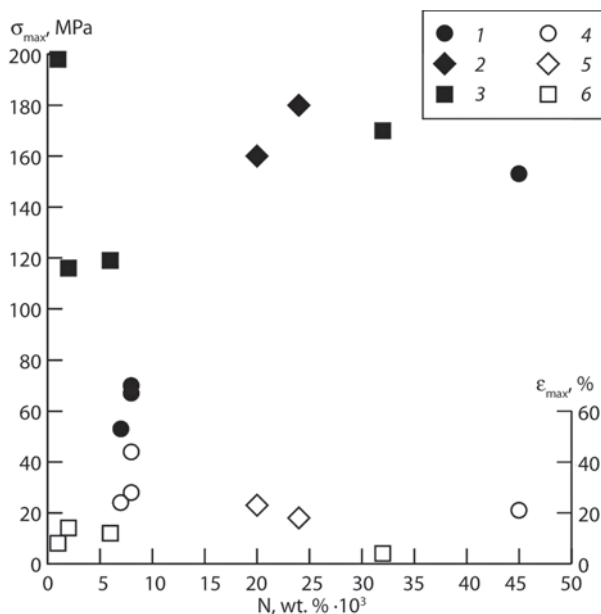


Fig. 4. Nitrogen influence on σ_{\max} (1–3) and ϵ_{\max} (4–6) at hot compression deformation: 1, 4 – α -steels; 2, 5 – γ -steels; 3, 6 – γ -steels with carbon

²A. V. Bronz participated in this work.

then it was rolled to 10, 6 and 4 mm thickness (also with intermediate heating), then it was quickly cooled in water from the finished rolling temperature. Mechanical properties of hot-deformed alloy 54K have been determined at extension testings: $\sigma_b = 995\text{MPa}$, $\sigma_{0.2} = 590\text{MPa}$ and $\delta = 39,0\%$. It is displayed that the alloy 54K is characterized by rather high ductility together with rather high strength, in spite of high carbon content.

The alloy 56K is also characterized by the wide temperature range for single-phase γ -solution (850–1220 °C). Thereby metal temperature during treatment for solid solution and during hot deformation was inside the interval 950–1200 °C. The route of thermal deformation treatment was similar to that of the alloy 54K. The ingot has been destructed in forging, due to its coarse-crystalline cast structure and appearance of nitride blisters. As soon as this alloy has too high nitrogen content (0.135%), there were too much nitrides in the structure. Supposedly, there is not substantial extraction of nitrides from liquid metal. Taking into account the results obtained for 33K–45K alloys, it can be concluded that nitrogen content at the melting and alloying temperature should not exceed seriously balanced content between AlN and melt at the liquidus temperature. In this case we can wait that coarse nitrides will be removed, while fine nitrides (remained after solidification) will support forming of fine-grained structure during heating before hot deformation.

5. Conclusions

Nitrogen in the alloys of Fe–Mn–Al–Ni–C system is practically completely in the form of AlN nitrides, for aluminium content $\geq 5\%$ and temperatures lower than 1100 °C. Controlled nitrogen effect on the properties of these alloys can be realized only via the nitride phase. For hot compression deformation at 1000 °C, increase of general nitrogen content leads to metal hardening. On the contrary, for warm and cold deformation, the tendency of lowering the metal strength with rise of nitrogen content is observed. At the same time, definite nitrogen influence on metal ductility was not revealed.

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