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Influence of Alloying Titanium Carbonitride by Transition Metals of Groups IV–VI on the Interaction with the Nickel Melt

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Abstract—The influence of alloying titanium carbonitride $TiC_{0.5}N_{0.5}$ by transition metals of Groups IV–VI on the mechanism of contact interaction with the nickel melt is studied. It is established that alloying metals exert a strongly destabilizing influence on titanium carbonitride $TiC_{0.5}N_{0.5}$, simultaneously increasing both its dissolution rate in nickel and the degree of process incongruence (the preferential transition of alloying metal and carbon into the melt). The influence of alloying on the phase stability of titanium carbonitride $TiC_{0.5}N_{0.5}$ in contact with a nickel melt manifests itself in its dehomogenization or phase separation. The destabilizing effect of alloying additives enhances in a series Me^{IV}–Me^{V}–Me^{VI} parallel with a decrease in their nitrogen affinity.

Keywords: alloyed titanium carbonitride, nickel, contact interaction, reactions, microstructure

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INTRODUCTION

It is known that the optimal combination of operational properties of cermets based on titanium carbide and carbonitride, in contrast with classical hard alloys of the WC–Co type, is attained only in a certain intermediate chemical, phase, and structural state [1–3]. Therefore, the knowledge of specific features and regularities of dissolution, phase formation, and structure formation running during the liquid-phase sintering of cermets is the necessary condition for their successful development [4–7].

This article is aimed at investigating the influence of alloying titanium carbonitride $TiC_{0.5}N_{0.5}$ with transition metals of Groups IV–VI on its chemical and phase stability in contact with the nickel melt. No data on this question can be found in publications.

Initial powderlike preparations $Ti_{1-n}Me_nC_{0.5}N_{0.5}$ of various compositions were fabricated by the carbothermic reduction of briquetted mixtures of TiO_2 and $Me^{IV-VI}O_y$ in the nitrogen stream at 1800°C for 30–50 h. The synthesized preparations were subjected to hot pressing in the nitrogen atmosphere at a temperature of 2800°C and pressure of 30 MPa. The residual porosity of compact samples was no larger than 3%.

The formula composition and lattice parameters of $Ti_{1-n}Me_nC_{0.5}N_{0.5}$ samples used in our study are presented in the table.

The methodical features of experiments were reduced to the following.

Hot-pressed samples of carbonitrides $Ti_{1-n}Me_nC_{0.5}N_{0.5}$ (∅10 mm, $h = 5$ mm) were placed into an alundum crucible on a nickel pellet with the same shape and sizes. Isothermal annealing was performed under rarefaction of 10^{-1} Pa at 1450°C for 1 h. After finishing the experiment, the samples were cut perpendicularly to the contact boundary, grinded, and polished with diamond pastes. The chemical composition of the phases and structural features of the contact interaction region were investigated by scanning

Formula composition and lattice parameters of carbonitride phases of the Ti–Me–C–N system

Phase composition	Lattice parameter ($\Delta a = \pm0.001$ Å)
$TiC_{0.5}N_{0.5}$	4.282
$Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5}$	4.288
$Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.4}O_{0.1}$	4.286
$Ti_{0.80}Zr_{0.20}C_{0.5}N_{0.5}$	4.410
$Ti_{0.80}Hf_{0.20}C_{0.6}N_{0.4}$	4.404
$Ti_{0.95}V_{0.05}C_{0.5}N_{0.5}$	4.277
$Ti_{0.80}V_{0.20}C_{0.6}N_{0.4}$	4.258
$Ti_{0.95}Nb_{0.05}C_{0.5}N_{0.5}$	4.286
$Ti_{0.60}Nb_{0.40}C_{0.6}N_{0.4}$	4.345
$Ti_{0.60}Nb_{0.40}C_{0.5}N_{0.4}O_{0.1}$	4.343
$Ti_{0.95}Ta_{0.05}C_{0.5}N_{0.5}$	4.285
$Ti_{0.95}Mo_{0.05}C_{0.5}N_{0.5}$	4.283
$Ti_{0.95}W_{0.05}C_{0.5}N_{0.5}$	4.280

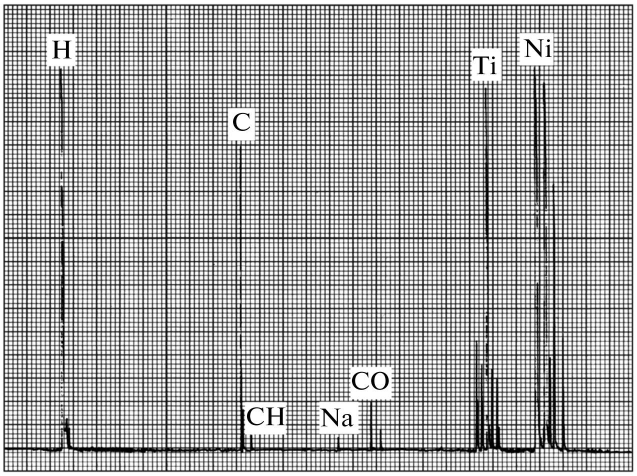

Fig. 1. Mass spectrum of eutectic isolations in the metallic zone of the $TiC_{0.5}N_{0.5}$/Ni sample.

electron microscopy and electron probe microanalysis (EPMA) using a JCXA-733 installation.

The results of the investigation are presented below in the form of schemes of running the corresponding chemical processes. Similar results on the $TiC_{0.5}N_{0.5}$–Ni base system that we published in [8] are presented here for the comparison.

We used the following conditional notation for clearness and description completeness of analyzed processes:

$\dashrightarrow$ is the dissolution of the carbonitride phase in the melt;

$\longrightarrow$ is the crystallization of the nickel-based melt;

$\longleftrightarrow$ is the phase separation of the initial carbonitride phase;

$\|$ are the phases jointly crystallizing from the melt;

$Ni(\ell)$ is the nickel-based melt;

Ni(ss) is the nickel-based solid solution;

₪ is the eutectic.

1. $TiC_{0.5}N_{0.5}$–Ni SYSTEM

According to results [8], the interaction mechanism of hot-compacted titanium carbonitride $TiC_{0.49}N_{0.48}$ with the nickel melt (1450°C, 1 h) can be described by the following reaction scheme:

$$\begin{gathered} TiC_{0.49}N_{0.48} + Ni \dashrightarrow TiC_{0.49-x}N_{0.48+x} (x = 0.05\text{–}0.07) + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 4.0\%) + \text{₪}\, Ni/TiC. \end{gathered} \tag{1}$$

It is seen from this scheme that dissolution of titanium carbonitride in nickel is incongruent; i.e., it is accompanied by the change of the composition of the dissolving phase (titanium and carbon preferentially transfer into the melt). The metallic phase has a hypoeutectic structure after cooling the system and includes primary isolations of the nickel-based solid solution in the surrounding of eutectic isolations of the carbide phase. Nitrogen is almost absent in these eutectic isolations according to the data of laser microprobe mass spectrometric analysis (Fig. 1).

2. SYSTEMS $Ti_{1-n}Me_n^{IV}C_{0.5}N_{0.5}$–Ni

Alloying the initial titanium carbonitride with small zirconium additives ($Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5}$) increases its dissolution rate and degree of process incongruence, and preferentially carbon and zirconium transfer into the melt. This results in the fact that the peripheral segments of dissolving grains are strongly enriched with nitrogen and titanium.

The metallic zone of the sample has a hypoeutectic structure after cooling, and primary grains of the nickel-based solid solution are surrounded by isolations of two sequentially crystallizing carbide eutectics, notably, binary Ni/TiC (N < 0.1%, Zr is absent) and ternary Ni/(Ti,Zr)C/(Zr,Ti)C (Fig. 2).

According to the EPMA data, the interaction mechanism of carbonitride $Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5}$ with the nickel melt can be described by the following reaction scheme:

$$\begin{gathered} Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow \text{“TiN”}(Zr \sim 0.8\%, C \sim 1.5\%) + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 4.5\%, Zr \sim 0.2\%) + \text{₪}\, Ni/TiC + \text{₪}\, Ni/(Ti, Zr)C/(Zr, Ti)C. \end{gathered} \tag{2}$$

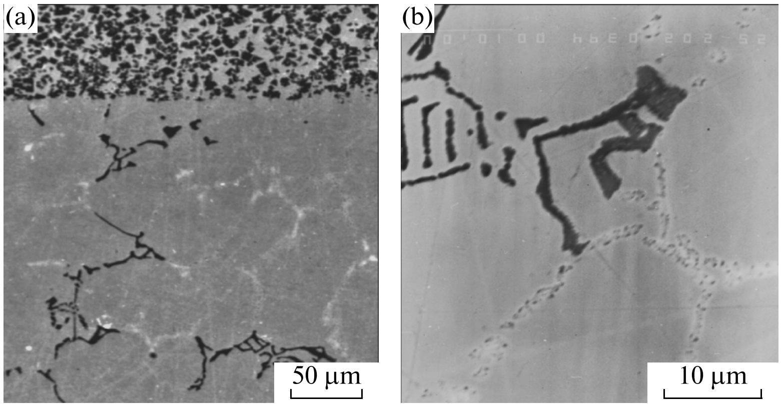

Fig. 2. Microstructure of the contact interaction region in the $Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5}$/Ni sample (1450°C, 1 h). (a) General appearance of the contact boundary and (b) fragment of the carbide eutectic.

We note that the preferential transition of zirconium into the melt, despite its low solubility in nickel compared with titanium, means that the energy of elastic lattice deformation introduces a decisive contribution to destabilization of the carbonitride phase.

The partial substitution of nitrogen by oxygen in carbonitride $Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.5}$ ($Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.4}O_{0.1}$) lowers its stability in contact with nickel even more. This results both in an increase in the dissolution rate of the refractory phase in nickel and in a decrease in the degree of process incongruence. This is accompanied by a corresponding decrease in the content of zirconium (from 7.2 to 0.6%), carbon (from 9.5 to 0.5%), and oxygen (from 2.6 to 0.4%) in peripheral regions of the grains of the dissolving phase. The appearance of separate isolations of oxide ZrO_2 in the metallic zone of the sample, along with the disappearance of the zirconium-containing carbide eutectic, evidences the active interaction of oxygen with carbon and zirconium in the melt with the formation of CO and ZrO_2, respectively.

Thus, the interaction mechanism of titanium–zirconium oxycarbonitride $Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.4}O_{0.1}$ with the nickel melt can be described by the following reaction scheme:

$$\begin{gathered}Ti_{0.95}Zr_{0.05}C_{0.5}N_{0.4}O_{0.1} + Ni \dashrightarrow \text{“TiN”}(Zr \sim 0.6\%,\ C \sim 0.5\%,\ O \sim 0.4\%) + Ni(\ell) + CO\uparrow,\\ Ni(\ell) \longrightarrow Ni(Ti \sim 5.5\%,\ Zr \sim 0.3\%) + \text{₪}\, Ni/TiC + ZrO_2.\end{gathered} \tag{3}$$

If the zirconium content in titanium carbonitride is sufficiently high ($Ti_{0.80}Zr_{0.20}C_{0.5}N_{0.5}$), its interaction mechanism with nickel becomes similar to that implemented in the $Ti_{0.75}Zr_{0.25}C$–Ni system [9]:

$$\begin{gathered}Ti_{0.75}Zr_{0.25}C_{0.96} + Ni \dashrightarrow Ti_{0.94}Zr_{0.06}C_{0.96-x} + Ni(\ell),\\ Ni(\ell) \longrightarrow Ti_{0.9}Zr_{0.1}C_x \| Zr_{0.8}Ti_{0.2}C_x + \text{₪}\, Ni/(Ti, Zr)C/(Zr, Ti)C.\end{gathered} \tag{4}$$

The only difference is that the metallic zone of the sample is now characterized by the hypoeutectic structure because of the lower dissolution rate of titanium–zirconium carbonitride compared with the corresponding carbide. According to EPMA results, the interaction can be now described by the following reaction scheme:

$$\begin{gathered}Ti_{0.80}Zr_{0.20}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.95}Zr_{0.05}C_{0.5-x}N_{0.5+z} + Ni(\ell),\\ Ni(\ell) \longrightarrow Ni(ss) + \text{₪}\, Ni/(Ti_{0.9}Zr_{0.1})C/(Zr_{0.8}Ti_{0.2})C.\end{gathered} \tag{5}$$

The influence of hafnium on dissolution processes in the $Ti_{1-n}Hf_nC_xN_z$–Ni system is in general similar to the influence of zirconium (Fig. 3). The characteristic feature illustrating this fact is presented below:

$$\begin{gathered}Ti_{0.80}Hf_{0.20}C_{0.6}N_{0.4} + Ni \dashrightarrow Ti_{0.90}Hf_{0.10}C_{0.6-x}N_{0.4+z} + Ni(\ell),\\ Ni(\ell) \longrightarrow Ni(ss) + \text{₪}\, Ni/Ti_{0.9}Hf_{0.1}C/Hf_{0.7}Ti_{0.3}C.\end{gathered} \tag{6}$$

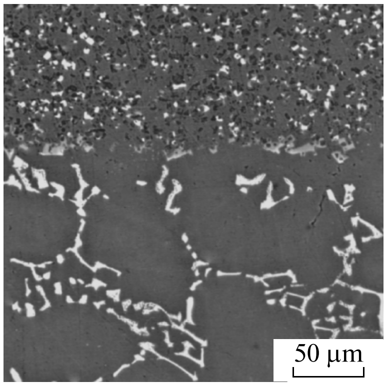

Fig. 3. Microstructure of the contact interaction region in the $Ti_{0.80}Hf_{0.20}C_{0.6}N_{0.4}$/Ni sample (1450°C, 1 h).

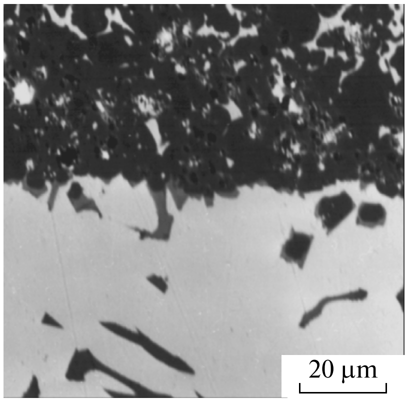

Fig. 4. Electron image of the contact zone of carbonitride $Ti_{0.8}V_{0.2}C_{0.6}N_4$ with the nickel melt (1450°C, 1 h).

3. SYSTEMS $Ti_{1-n}Me_n^{V}C_{0.5}N_{0.5}$

Small additives of vanadium into initial titanium carbonitride ($Ti_{0.95}V_{0.05}C_{0.5}N_{0.5}$) also destabilize the forming solid solution, thus increasing its dissolution rate in nickel and degree of process incongruence. The consequence of the preferential (compared with titanium) transfer of vanadium into the melt is its increased content in metallic matrix and carbide eutectic:

$$\begin{gathered} Ti_{0.95}V_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.99}V_{0.01}C_{0.5-x}N_{0.5+z} + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 6.5\%, V \sim 1.5\%) + \text{₪}\, Ni/(Ti_{0.9}V_{0.1})C. \end{gathered} \tag{7}$$

The interaction mechanism in the system under consideration undergoes a qualitative change upon going to the more vanadium-enriched composition $Ti_{0.8}V_{0.2}C_{0.6}N_{0.4}$. Indeed, the total carbonitride dissolution rate now does not increase like we could expect taking into account the destabilizing role of small vanadium additives and a relatively lower nitrogen content, instead noticeably dropping. We assume that the cause of this circumstance is the unusual fact that the diffusion redistribution rate of elements between the periphery and grain bulk becomes higher than their transfer rate into the melt. Such an inversion of mass transfer rates in turn results in the exfoliation of the grains of initial carbonitride into two phases, notably, based on titanium nitride (grain core) and based on vanadium carbide (reaction (8), Figs. 4, 5):

$$\begin{gathered} Ti_{0.8}V_{0.2}C_{0.6}N_{0.4} + Ni \dashrightarrow \text{“TiN”} \longleftrightarrow \text{“VC”} + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 2.5\%, V \sim 3.0\%) + \text{₪}\, Ni/(V_{0.6}Ti_{0.4})C. \end{gathered} \tag{8}$$

The situation is in many aspects similar to that one observed in the $Ti_{0.75}V_{0.25}C_{0.94}$–Ni carbide system [9]. The only distinction is that the effect of the phase separation of the solid solution is more strongly implemented in the presence of nitrogen. Subsequently, only the “VC” grain shell is dissolved in practice, which leads to the strong enrichment of the melt with vanadium. From here, its content in the metallic matrix and carbide eutectic is much higher than in titanium.

Small additives of niobium, similarly to vanadium, also destabilize titanium carbonitride, increasing its dissolution rate in nickel and degree of process incongruence:

$$\begin{gathered} Ti_{0.95}Nb_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.99}Nb_{0.01}C_{0.5-x}N_{0.5+z} + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 5.0\%, Nb \sim 2.0\%) + \text{₪}\, Ni/(Ti_{0.8}Nb_{0.2})C. \end{gathered} \tag{9}$$

On the other hand, in contrast with the vanadium-containing system, no phase separation of the grains is observed at a high niobium content in carbonitride ($Ti_{0.6}Nb_{0.4}C_{0.6}N_{0.4}$). This is probably caused by the lower diffusion mobility of niobium atoms in the solid phase and its lower interphase activity compared with vanadium. Consequently, the transfer rate of elements into the melt remains higher than their intragrain redistribution rate:

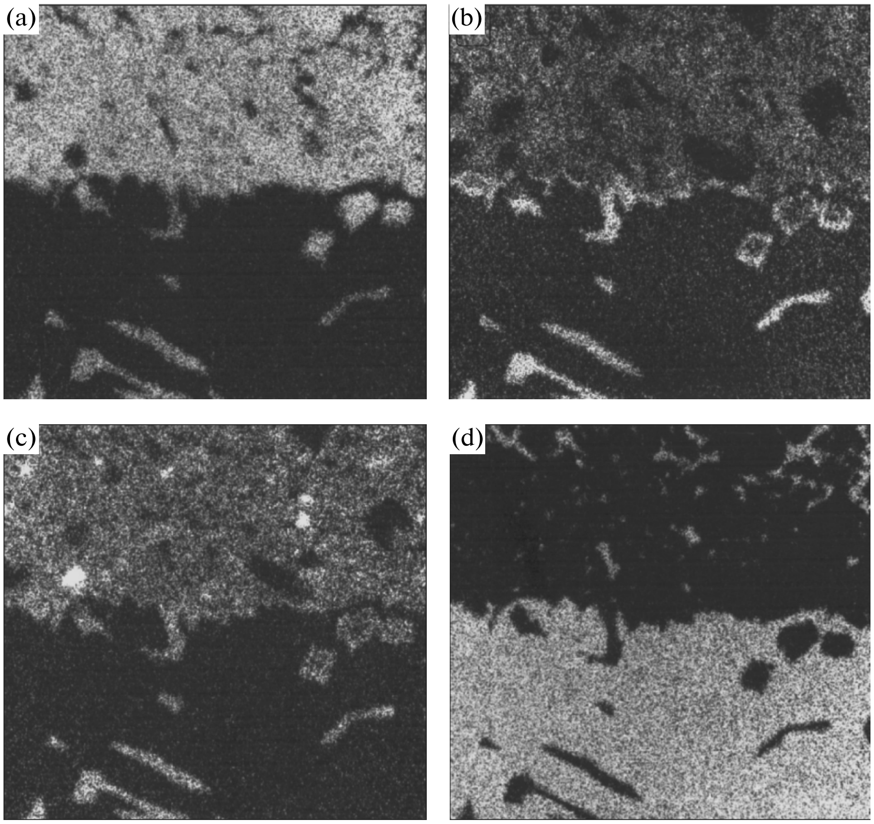

Fig. 5. Images of the contact zone presented in Fig. 4 in characteristic X-ray radiations (a) TiK_α, (b) VK_α, (c) CK_α, and (d) NiK_α.

$$\begin{gathered}Ti_{0.6}Nb_{0.4}C_{0.6}N_{0.4} + Ni \dashrightarrow Ti_{0.9}Nb_{0.1}C_{0.6-x}N_{0.4+z} + Ni(\ell),\\ Ni(\ell) \longrightarrow Ni(ss) + \text{₪}Ni/(Nb_{0.6}Ti_{0.4})C.\end{gathered} \tag{10}$$

The partial substitution of carbon in carbonitride $Ti_{0.6}Nb_{0.4}C_{0.6}N_{0.4}$ by oxygen ($Ti_{0.6}Nb_{0.4}C_{0.5}N_{0.4}O_{0.1}$) almost does not affect its dissolution rate. However, eutectic isolations of the Ni_3Nb intermetallic compound are additionally formed in the metallic zone of the sample after its cooling, which evidences the substantial decarburization of the system resulted from the reaction [C] + [O] = CO↑ in the melt:

$$\begin{gathered}Ti_{0.6}Nb_{0.4}C_{0.5}N_{0.4}O_{0.1} + Ni \dashrightarrow Ti_{0.9}Nb_{0.1}C_{0.5-x}N_{0.4+z} + Ni(\ell) + CO\uparrow,\\ Ni(\ell) \longrightarrow Ni(ss) + Ni/(Nb_{0.6}Ti_{0.4})C + \text{₪}Ni/Ni_3Nb.\end{gathered} \tag{11}$$

Alloying the $TiC_{0.5}N_{0.5}$ carbonitride by small additives of tantalum ($Ti_{0.95}Ta_{0.05}C_{0.5}N_{0.5}$) also destabilizes it, increasing both the dissolution rate and the degree of process incongruence. In this respect, the influence of tantalum is similar to the influence of small additives of vanadium and niobium:

$$\begin{gathered}Ti_{0.95}Ta_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.99}Ta_{0.01}C_{0.5-x}N_{0.5+x} + Ni(\ell),\\ Ni(\ell) \longrightarrow Ni(Ti \sim 4.5\%, Ta \sim 2.5\%) + \text{₪}Ni/(Ti_{0.65}Ta_{0.35})C.\end{gathered} \tag{12}$$

A comparison of reactions (7), (9), and (12) allows us to reveal an interesting regularity, notably, the dissolution rate of carbonitride, which is evaluated by the amount of titanium transferred into the melt, decrease in a series of single-type additives V–Nb–Ta; the degree of its depletion with the alloying metal is almost

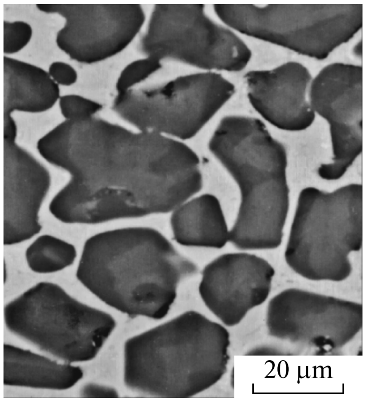

Fig. 6. Microstructure of the grains of the refractory phase in the $Ti_{0.95}Mo_{0.05}C_{0.5}N_{0.5}$/Ni sample (1450°C, 1 h).

invariable, but the amount of the alloying element in the composition of the carbide eutectic increases. A correlation is observed that the higher the crystallization temperature of the Ni/$Me^{V}C$ eutectic (1300, 1330, and 1350 ± 10°C, for systems VC–Ni, NbC–Ni, and TaC–Ni, respectively [10]) is compared with the Ni/TiC eutectic (1280°C), the larger the amount of the alloying metal enters the composition of the carbide eutectic components and the smaller its residue in the metallic matrix is.

4. SYSTEMS $Ti_{1-n}Me_n^{VI}C_{0.5}N_{0.5}$

Alloying the $TiC_{0.5}N_{0.5}$ carbonitride with small additives of molybdenum and tungsten lowers its phase stability in contact with nickel even more strongly than by Group V metals. For example, the relative dissolution rate of carbonitride in the melt increases approximately by a factor of 1.5, while the degree of the process incongruence (the degree of depletion of grain periphery by the alloying metal) increases by an order of magnitude (reactions (13), (14), Fig. 6):

$$\begin{gathered} Ti_{0.95}Mo_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.995}Mo_{0.005}C_{0.5-x}N_{0.5+z} + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 6.5\%,\ Mo \sim 1.5\%) + \text{₪}Ni/Ti_{0.6}Mo_{0.4}C; \end{gathered} \tag{13}$$

$$\begin{gathered} Ti_{0.95}W_{0.05}C_{0.5}N_{0.5} + Ni \dashrightarrow Ti_{0.995}Wo_{0.005}C_{0.5-x}N_{0.5+z} + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 6.0\%,\ W \sim 2.5\%) + \text{₪}Ni/Ti_{0.7}W_{0.3}C. \end{gathered} \tag{14}$$

It is reasonable to assume that the main cause of such behavior is the very low nitrogen affinity of molybdenum and tungsten, which prevents them from enriching the grain boundaries despite their high interphase activity. We recall that molybdenum and tungsten in similar carbide systems, in contrast with carbonitride ones, practically do not leave the limits of carbide grains concentrating at their periphery [9]:

$$\begin{gathered} Ti_{0.95}Mo_{0.05}C_{0.96} + Ni \dashrightarrow TiC_{0.96-x} \longleftrightarrow Ti_{0.85}Mo_{0.15}C_x + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 3.5\%;\ Mo \sim 0.1\%); \end{gathered} \tag{15}$$

$$\begin{gathered} Ti_{0.99}W_{0.01}C_{0.95} + Ni \dashrightarrow TiC_{0.95-x} \longleftrightarrow Ti_{0.93}W_{0.07}C_x + Ni(\ell), \\ Ni(\ell) \longrightarrow Ni(Ti \sim 4.0\%;\ W - no) + \text{₪}\,Ni/TiC. \end{gathered} \tag{16}$$

CONCLUSIONS

The influence of alloying the $TiC_{0.5}N_{0.5}$ carbonitride with the transition metals of Groups IV–VI on the contact interaction mechanism with the nickel melt is investigated systematically for the first time. An analysis of the results allows us to conclude the following:

(i) Alloying the $TiC_{0.5}N_{0.5}$ titanium carbonitride with Group IV transition metals (Zr, Hf) destabilizes it, simultaneously increasing both the dissolution rate in nickel and the degree of process incongruence (the preferential transfer of alloying metal and carbon into the melt). The main cause of the observed effects is an increase in the elastic deformation energy of the lattice of the forming solid solution because of a large difference in sizes of atoms of the master and alloying metals.

(ii) Alloying the $TiC_{0.5}N_{0.5}$ titanium carbonitride with Group V transition metals (V, Nb, Ta) even more strongly lowers its stability in contact with nickel, increasing its dissolution rate and degree of process incongruence. The higher diffusion mobility of Group V alloying metals in the carbonitride lattice compared with Group IV metals leads to the dehomogenization or even phase separation of the grains enveloping their total volume rather than only peripheral layers, as in Zr-containing and Hf-containing layers.

(iii) Alloying the $TiC_{0.5}N_{0.5}$ titanium carbonitride with small additives of Group VI transition metals

(Mo, W) lowers its phase stability in contact with nickel to the maximal degree. This phenomenon manifests itself in the almost complete transfer of alloying metals from the grain bulk into the melt during the dissolution of the carbonitride phase.

(iv) The influence of alloying metals on the destabilization of titanium carbonitride $TiC_{0.5}N_{0.5}$ in contact with the nickel melt is enhanced in a series Me^{IV}–Me^{V}–Me^{VI} in parallel with a decrease in their nitrogen affinity.

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