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\*e-mail: [guizira.83@mail.ru](mailto:guizira.83@mail.ru)**MICROSTRUCTURAL PECULIARITIES OF ALUMINIDES OF NICKEL, TITANIUM, AND COBALT**

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**Abstract.** Scanning electron microscopy, electron probe microanalysis, X-rays diffractometry, and microhardness measurements were used for studies of diffusion zones in systems Al-Ti, Al-Co, and Al-Ni formed by means of diffusion couples method in a temperature range of 1,300-1,350 °C. It was shown that diffusion zones have the multilayer structure with numerous peculiarities. For a number of intermetallic compounds formed in all three systems there were revealed their temperature shifts using the electron probe microanalysis methods and confirmed with X-rays diffractometry. The correlation between the individual intermetallic layers and their microhardness numbers are not always evident. For some aluminides the microhardness can reach  $7,060 \pm 1,200$  MPa (in globular area of  $Ti_5Al_{11}$  and TiAl) and 4,938 MPa (in multilayer area of titanium aluminide  $TiAl_2$ ). For very thin (about 10  $\mu m$ ) layers in multilayer area the microhardness has been measured in the first time 4,000 MPa (TiAl) and 4,450 MPa ( $Ti_3Al$ ). The obtained microhardness numbers demonstrated reasonable coincidence with previous published results. Microhardness in Al-Ni system was measured as  $5,200 \pm 500$  MPa (for  $\beta$ -NiAl) and  $5,300 \pm 860$  MPa (for  $\gamma'$ - $Ni_3Al$ ). For cobalt aluminide CoAl from Al-Co system the microhardness showed numbers  $3,900 \pm 200$  MPa, whereas for  $CoAl_3$  it was higher up to 6,600 MPa. The intermetallic compound  $\gamma'$ - $Ni_3Al$  considered as the most interesting from practical point of view had small (up to 10  $\mu m$ ) width whereas the next layer  $Ni_3Al_5$  had the wider (100  $\mu m$ ) width. There were also revealed some globular  $Ti_5Al_{11}$  having thin shells which consist of intermetallic  $Ti_9Al_2$ .

**Keywords:** intermetallic phases, aluminides, microstructure, scanning electron microscopy, electron probe microanalysis, microhardness.

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**НИКЕЛЬ, ТИТАН ЖӘНЕ КОБАЛЬТ АЛЮМИНИДТЕРІНІҢ МИКРОҚҰРЫЛЫМДЫҚ ЕРЕКШЕЛІКТЕРІ**

**Түйіндеме** Растрлық электрондық микроскопия және рентгеноспектралды микроталдау көмегімен, рентгендік дифрактометрия және микроқаттылықты өлшеу арқылы Ti-Al, Co-Al және Ni-Al жүйелеріндегі 1300-1350°C температуралар аралығында диффузиялық жұптасу әдісімен алынған диффузиялық аймақтар зерттелген. Диффузиялық аймақта көптеген ерекшеліктері бар күрделі көпқабатты құрылым түзілген. Интерметаллидті қосылыстардың бірнеше түрінің температуралық қозғалысы растрлық электрондық микроскопия және рентгеноспектралды микроталдау көмегімен расталды. Микроқаттылық көрсеткіштерін жеке қабаттармен байланыстыруға мүмкіндік әркезде бола бермеді. Жеке аймақтағы түйіршік тәрізді интерметаллидтер үшін микроқаттылық  $7060 \pm 1200$  МПа құрады, ал көпқабатты аймақтағы  $TiAl_2$  титан алюминидінің микроқаттылығы 4938 МПа көрсетті. Сонымен бірге көпқабатты аймақтағы ені өте жұқа (шамамен 10 мкм) болып келетін интерметаллидтердің микроқаттылықтары: 4000 МПа (TiAl) и 4450 МПа ( $Ti_3Al$ ) өлшеу іске асты. Микроқаттылық көрсеткіштері әдебиеттегі дереккөздермен сәйкес келеді. Al-Ni жүйесіндегі микроқаттылық ( $\beta$ -NiAl үшін)  $5200 \pm 500$  МПа және ( $\gamma'$ - $Ni_3Al$  үшін)  $5300 \pm 860$  МПа аралығында табылады. Al-Co жүйесіндегі кобальт алюминидінің CoAl микроқаттылығы  $3900 \pm 200$  МПа көрсетсе, дәл сондай  $CoAl_3$  алюминидтің көрсеткіші 6600 МПа құраған. Соның ішінде, практикалық тұрғыда маңыздылығы жоғары болып келетін интерметаллидтің  $\gamma'$ - $Ni_3Al$  ені 10 мкм құра, және ол енінің жалпақтығы 100 мкм-нан тұратын  $Ni_3Al_5$  интерметаллидтің жанында орналасқан. Жеке орналасқан  $Ti_5Al_{11}$  түйіршік тәрізді интерметаллидтің, сыртқы беті  $Ti_9Al_2$  интерметаллидпен көмкерілгені байқалған.

**Түйін сөздер:** интерметаллидті фазалар, алюминидтер, микроқұрылым, расторлық электронды микроскоп, рентгеноспектралды микроталдау, микроқаттылық.

## ОСОБЕННОСТИ МИКРОСТРУКТУРЫ АЛЮМИНИДОВ НИКЕЛЯ, ТИТАНА И КОБАЛЬТА

**Резюме.** Методами растровой электронной микроскопии, рентгеноспектрального микроанализа, рентгеновской дифрактометрии и измерения микротвердости были исследованы диффузионные зоны систем Ti–Al, Co–Al и Ni–Al, полученные методом диффузионных пар в интервале температур 1300–1350 °С. Установлено, что в диффузионной зоне образуется сложная многослойная структура со многими особенностями. Обнаруженные с помощью растровой электронной микроскопии, и рентгеноспектрального микроанализа температурные сдвиги для ряда интерметаллических соединений подтверждены рентгеновской дифрактометрией. Полная привязка показаний микротвердости к интерметаллическим слоям не всегда представилась возможной. Для отдельных алюминидов микротвердость составляет  $7060 \pm 1200$  МПа (глобулярный участок  $Ti_5Al_{11}$  и TiAl) и 4938 МПа (многослойный участок алюминидов титана  $TiAl_2$ ). При этом в многослойном участке удалось определить показания микротвердости в тонких (порядка 10 мкм) слоях: 4000 МПа (TiAl) и 4450 МПа ( $Ti_3Al$ ). Полученные показания микротвердости практически совпадают с литературными источниками. Микротвердость в системе Al–Ni находится в интервале от  $5200 \pm 500$  МПа (для  $\beta$ -NiAl) или от  $5300 \pm 860$  МПа (для  $\gamma'$ -Ni<sub>3</sub>Al). Для системы Al–Co алюминид кобальта CoAl показал значения микротвердости  $3900 \pm 200$  МПа, в то же время для CoAl<sub>3</sub> она составляет 6600 МПа. В частности, такой важный с практической точки зрения интерметаллид, как  $\gamma'$ -Ni<sub>3</sub>Al, имеет ширину до 10 мкм и образуется рядом со слоем Ni<sub>3</sub>Al<sub>5</sub>, который составляет 100 мкм. Также обнаружено, что отдельные глобулы  $Ti_5Al_{11}$  имеют тонкие оболочки, состоящие из интерметаллида  $Ti_6Al_2$ .

**Ключевые слова:** интерметаллические фазы, алюминиды, микроструктура, растровая электронная микроскопия, рентгеноспектральный микроанализ, микротвердость.

**Introduction.** The developments of new construction materials, which have better performance and cost-effective properties for numerous units in different areas of application, should result in relatively light compounds. They are able to work in conditions of long-term thermal cycling. Therefore these materials should have high refractory properties, low density, and high mechanical properties. Irregular-shaped and heavy-duty items with unique and serial properties, e.g. based on the titanium powders, are manufactured using additive technologies. For these purposes ductility and hardness of modern metallic and intermetallic compounds can be modified using the different heat treatments [1]. It seems that it is impossible to raise resistance of metallic materials to thermal shocks and thermo-cycling using the traditional approaches for alloys [2].

Intermetallic compounds are considered as having a great potential to improve operation specifications for such heavy-duty conditions [2]. Today there are more than 1,500 intermetallic compounds forming about 200 different crystal structures [3]. The most prospective intermetallics are as follows: phases  $A_3B$  having cubic crystal lattice, phases  $A_3B$  having hexagonal crystal lattice, aluminides of type  $MA_3$  and phases of type  $AB$  [3]. The best results in area of development of structural refractory intermetallics were reached for titanium aluminides [4]. Among the nickel aluminides one can emphasize so-named  $\gamma'$ -phase ( $Ni_3Al$ ). It appeared in refractory superalloys and became attractive subject in other systems [5]. Studies conducted on the cobalt aluminides are just getting started [6]. Today the diffusion couples technique is the effective to study strengthening phases in intermetallics. It is convenient for comparative studies not only in

diffusion zone in its classical understanding [7], but also in reaction zone (RZ) resulted from transformation of diffusion zones (DZs) to formation and development of intermetallic compounds [8]. Merging of these terms into single conception “Diffusion zone and Reaction zone” (DZ&RZ) fully corresponds to processes proceeding in the same area of substance.

**Goal** of this work is a detection of microstructural peculiarities in the DZ&RZ and microhardness of aluminides of nickel, titanium, and cobalt.

**Experimental.** DZ&RZs of Al–Ti, Al–Ni, and Al–Co systems were prepared by means of the diffusion couple method. This method used the mode “Solid metal (Co, Ni, Ti) – liquid aluminum” at temperatures between 1,000 °C and 1,350 °C from 1 to 4 hours and cooling on air [9]. For these purposes the metals, which have a technical purity, were used: aluminum A99 grade, cobalt K0 grade, nickel H2 grade, and titanium BT1-00 grade (all grades were written in Russian).

For sample preparations in the longitudinal and cross sections STRUERS equipment (Secotom-50 and Tegramin-25) was used. Studies of scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) were implemented using the microanalyzer JXA-8230 (JEOL). Element composition of phases formed in DZ&RZs was identified with spot detection using the Energy Dispersive Spectrometer (EDS). Detailing of phase compositions in DZ&RZs was performed using the X-rays diffractometer (XRD) Bruker D8 Advance. Microhardness of samples was measured with PMT-3M device using a load 1HV; some samples were measured with DuraScan G5 (Emco Test, Austria) at loads from 0.025 to 1HV.

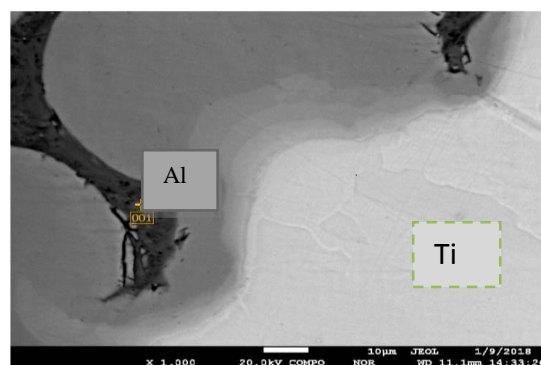
**Results and their discussion.** Our result in DZ&RZ between Al and Ti consists of three fragments: area with smooth growth of Al concentration in titanium, area in form of multilayer structures of intermetallic phases, and area in the form of large globular particles of intermetallics and gaps between them filled with non-reacted aluminum (Figure 1 a, c). Large globes can reach sizes up to 100  $\mu\text{m}$  (Figure 1c, d); they consist of phases TiAl (51.69 at. % Ti) and  $\text{Ti}_5\text{Al}_{11}$  (31.2 at. % Ti). Thin shells can be formed around these globes; they consist of phases  $\text{TiAl}_2$  (33.07 at.% Ti) and  $\text{Ti}_9\text{Al}_{23}$  (37.15 at. % Ti). The share of non-reacted aluminum was estimated by means of the metallographic methods for evaluation of phase volumes [10]. It is decreasing with growth of temperature from 23 % at 1,100  $^{\circ}\text{C}$  to 6 % at 1,300  $^{\circ}\text{C}$ .

These globes shown on Figure 1d have Ti concentration 31.2 at. % indicating on intermetallic phase  $\text{Ti}_5\text{Al}_{11}$ . Their microhardness is about  $7,065 \pm 255$  MPa. Its order of magnitude is close to values of titanium aluminides formed by method of powder shock-wave compression: 4,086 MPa for TiAl and 4,492 MPa for  $\text{Ti}_3\text{Al}$  [11]. The microhardness numbers were also determined for thin (about 10  $\mu\text{m}$ ) layers: 4,000 MPa (TiAl) and 4,450 MPa ( $\text{Ti}_3\text{Al}$ ).

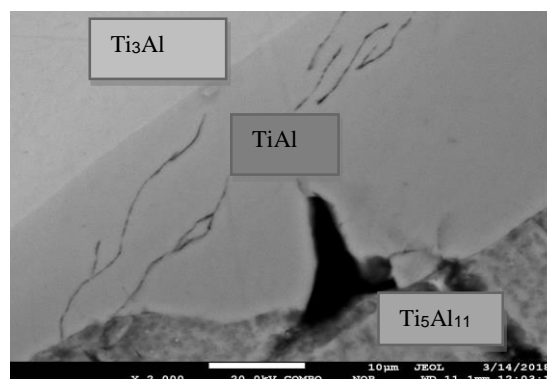
Figure 2 shows microstructure of DZ&RZs of Al-Ni and Al-Co systems. Figure 2a shows indents in  $\gamma'$ - $\text{Ni}_3\text{Al}$  layer with a distorted shape. Since compression of indents is observed in direction perpendicular to layer boundaries one can suppose the generation of compressing stresses due to difference of volumes in elementary unit cells belonging to different phases.

The compressive stresses can radically influence not only on shape of indents, but also on the microhardness in the affected layer [12]. The intermetallic compound  $\text{Ni}_3\text{Al}_5$  has with agglomeration of pores (Figure 2b). There are also shown the profiles of Al (red) and Ni (green) giving evidence that their concentrations in layers and on layer boundaries have non-monotonic character.

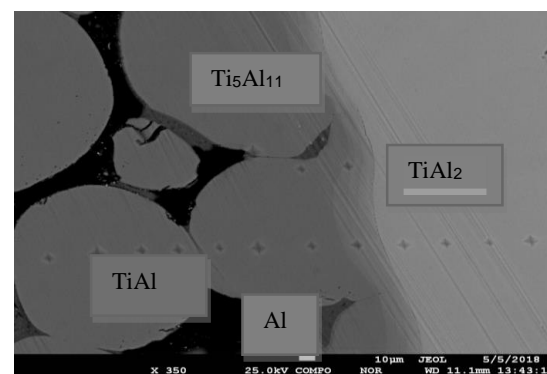
When the microhardness were not been affected by microstructural peculiarities (narrow layers, pores, etc.) its numbers have been determined for separate intermetallic phases. For  $\beta$ -NiAl it was  $5,200 \pm 500$  MPa and for  $\gamma'$ - $\text{Ni}_3\text{Al}$  –  $5,300 \pm 860$  MPa. For  $\beta$ -NiAl it is  $5,900 \pm 300$  MPa and for  $\gamma'$ - $\text{Ni}_3\text{Al}$  it is about  $5,100 \pm 500$  MPa [13]. For Al-Co system the pattern was pretty the same with indents in CoAl intermetallic phase (Figure 2c).



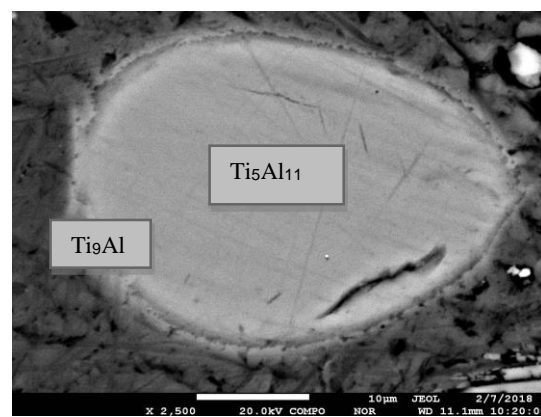
a)



b)



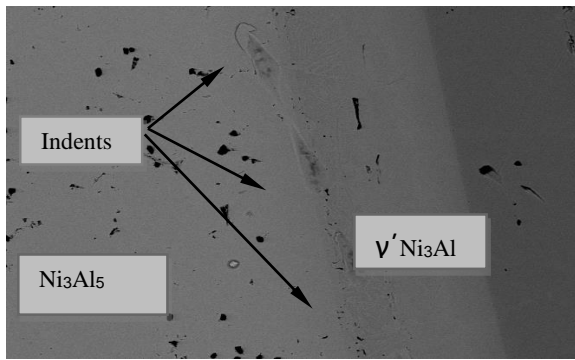
c)



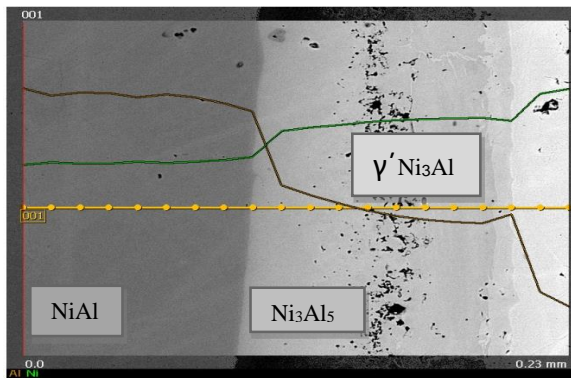
d)

a) 1,150  $^{\circ}\text{C}$ , b) 1,300  $^{\circ}\text{C}$ , c) 1,300  $^{\circ}\text{C}$ , and d) 1,150  $^{\circ}\text{C}$

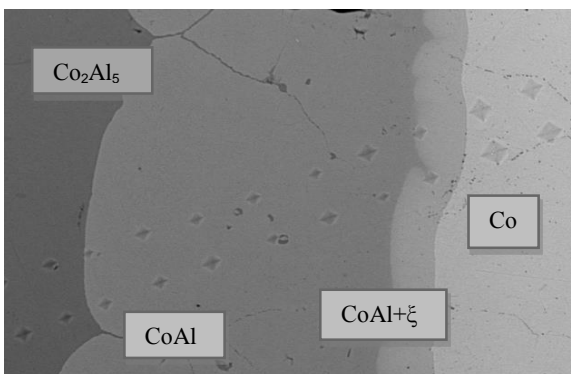
Figure 1 – DZ&RZ microstructure of Ti-Al system



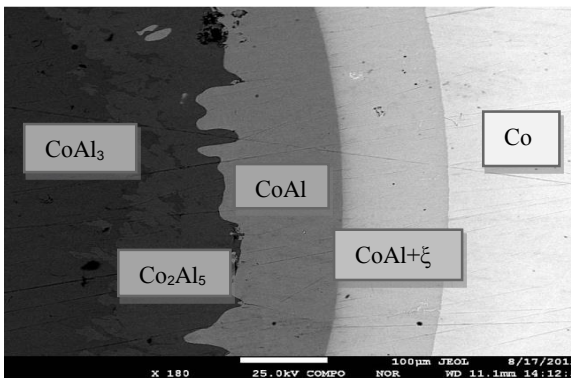
a)



b)



c)



d)

a) 1,300°C, b) 1,250°C, c) 1,150°C, and d) 1,300°C

Figure 2 – DZ&RZ microstructure of Al-Ni and Al-Co systems

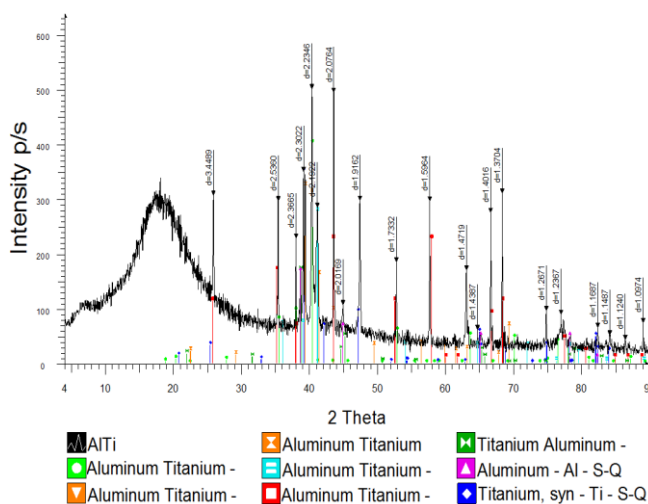
From this Figure it is notable that microhardness number of pure Co is lower compared to cobalt aluminides. CoAl layer has microhardness number about  $3,900 \pm 200$  MPa; for CoAl<sub>3</sub> layer the microhardness number is about 6,600 MPa.

Present measurements do not coincide with data demonstrating that cobalt aluminide CoAl has microhardness higher than for nickel aluminide NiAl [3]. Presumably it is related to techniques of manufacturing of intermetallic compounds and different consequences of phase transformations in multilayer structures. The accurate comparison of microhardness sometimes is very difficult even for simple experimental conditions [14], it is needless to say about differences caused by microstructural peculiarities.

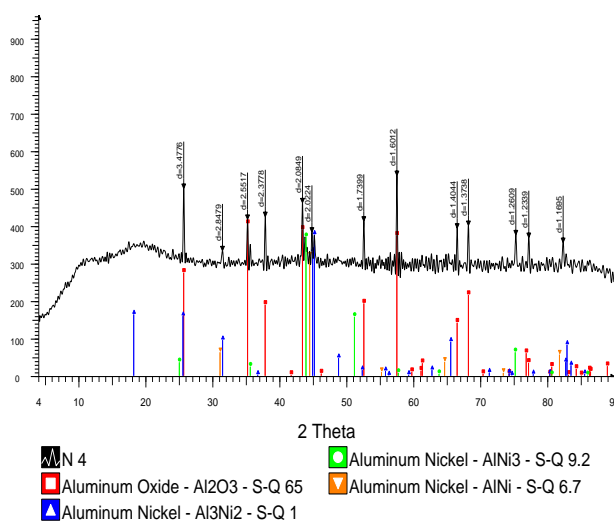
In area of higher temperatures the layer β-NiAl has been formed with width about 200 μm. For γ'-Ni<sub>3</sub>Al the width exceeds 10 μm (Figure 2a, b). Meanwhile for Al-Co system the intermetallic compound CoAl has its width about 115 μm; and this value for CoAl<sub>3</sub> was below 100 μm (Figure 2c, d). A brief summary on this issue can be formulated as follows - the direct comparison in widths will be incorrect even for the same system because of the sophisticated structural peculiarities.

DZ&RZs of intermetallic phases have complex structure, e.g. for Al-Ti system after 1,100 °C porosity has been formed whereas after 1,300 °C phase TiAl which has the brittleness at the room temperature [15] contains the specific cracks (Figure 1b). Examples of porosity were observed in other multilayer intermetallic compounds (TiAl and Ti-TiAl<sub>3</sub>) [16]. Formation of titanium aluminides was confirmed by XRD spectra (Figure 3a). These XRD measurements verified the temperature shifts of existence of a number of intermetallics in DZ&RZs in systems Al-Ni and Al-Co revealed by means of EPMA methods [17, 18]. However, some of the intermetallic layers are too narrow to be identified with XRD. It is related to fact that size of XRD spot is much larger compared to the typical sizes of DZ&RZs and its corresponding layers. Figure 3 (a, b) shows X-rays diffraction patterns for Al-Ti system with observed intermetallic phases TiAl<sub>3</sub>, Ti<sub>2.9</sub>Al<sub>2.1</sub>, TiAl, and Ti<sub>3</sub>Al and for Al-Ni system with phases NiAl<sub>3</sub>, NiAl, and Ni<sub>2</sub>Al<sub>3</sub>.

In Al-Ti system at 1,300 °C the phase Ti<sub>9</sub>Al<sub>23</sub> (27.15 at.% Ti) demonstrated the temperature shift, i.e. it should be disappeared at 780 °C according to Predel's work [19]. In Al-Ni system the temperature shift for phase NiAl<sub>3</sub> (25.78 at.% Ni) is about 446 °C taking into account that its upper limit of existence is 854 °C [20].



a)



b)

a) Al-Ti system, b) Al-Ni system.

Figure 3 – Examples of XRD analysis (after 1,300 °C, 1 hour)

The similar value for  $Ni_2Al_3$  (40 at.% Ni) is 163 °C. In Al-Co system for the phase  $CoAl_3$  (24.93 at. % Co) there is the temperature shift about 165 °C [21], whereas for phase  $Co_4Al_{13}$  (23.19 at.% Co) it is about 207 °C.

The shifts in phase diagrams are not frequently reported, however, there are communications on temperature and concentration shifts compared to hand-book data for binary and intermetallic systems [22, 23, 24, 25]

**Conclusions.** To sum up, observations and identifications of structural peculiarities of DZ&RZs in Ti-Al, Co-Al, and Ni-Al systems obtained by means of the diffusion couple technique in a

temperature range of 1,000-1,350 °C using the SEM/EPMA methods resulted in the following conclusions:

1) Complex and multilayer structure is developed with numerous peculiarities in form of narrow layers for instance in case of  $\gamma'$ - $Ni_3Al$  adjacent to layer  $Ni_3Al_5$  with the developed porosity or in form of thin intermetallic shells  $Ti_9Al_2$  surrounding the separate globes of  $Ti_5Al_{11}$ ;

2) Temperature shifts for a number of intermetallic compounds revealed by means of EPMA were confirmed by XRD;

3) Although the microhardness had been comparable to the literature data, there was no complete opportunity to link them to individual layers. Moreover comparison of the microhardness and DZ&RZ widths is a difficult task even for the same system because of complicated and sometimes incomparable structural peculiarities.

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