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# Х А Б А Р Л А Р Ы

## ИЗВЕСТИЯ

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## NEWS

OF THE ACADEMY OF SCIENCES  
OF THE REPUBLIC OF KAZAKHSTAN  
Kazakh national research technical university  
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### ГЕОЛОГИЯ ЖӘНЕ ТЕХНИКАЛЫҚ ҒЫЛЫМДАР СЕРИЯСЫ



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**MULTILAYER STRUCTURE FORMED  
IN DIFFUSION ZONE OF Al-Co AND Al-Ni SYSTEMS**

**Abstract.** The Diffusion Zone (DZ) of Al-Co and Al-Ni systems using the diffusion couple method was studied. The microstructure and element composition of samples were studied in cross-sectional geometry by mean of Scanning Electron Microscopy and Electron Probe Microanalysis (SEM/EPMA) and X-rays diffraction. After isothermal treatment in the range between 1000°C and 1375°C the multilayer intermetallic structure has been formed. It was shown that the diffusion couples method demonstrates its efficiency in comparative experiments having cross-sectional geometry. Depending on the established concentration of components in DZ several layers of different phase composition have been formed. The well-known intermetallics having constant compositions were detected: Al<sub>9</sub>Co<sub>2</sub>, Al<sub>13</sub>Co<sub>4</sub>, Al<sub>3</sub>Co, AlCo, Al<sub>5</sub>Co<sub>2</sub>, as well as Al<sub>3</sub>Ni<sub>5</sub>, Al<sub>4</sub>Ni<sub>3</sub>, AlNi, AlNi<sub>3</sub>. New compounds having variable composition were revealed. For of Al-Co system, these four layers were detected after treatment at high temperature (1300-1375 °C): Al<sub>21</sub>Co<sub>79</sub> (78.72 at.% Co), Al<sub>44</sub>Co<sub>56</sub> (55.69 at.% Co), Al<sub>20</sub>Co<sub>80</sub> (79.55 at.% Co), and Al<sub>27</sub>Co<sub>73</sub> (73.29 at.% Co). For of Al-Ni system, the corresponding layers having clear boundaries were detected: Al<sub>51</sub>Ni<sub>49</sub> (49.07 at.% Ni), Al<sub>36</sub>Ni<sub>64</sub> (64.06 at.% Ni), Al<sub>30</sub>Ni<sub>70</sub> (69.62 at.% Ni), and Al<sub>32</sub>Ni<sub>68</sub> (67.86 at.% Ni).

Similar structural features in multilayer DZ in both systems (porosity, dendrites, and shift of intermetallics having constant composition to higher temperatures), as well as evidence of changes in local densities not only for different layers, but also within one layer, can contribute to specification of application of both systems to AM.

**Keywords:** diffusion couple, scanning electron microscopy, electron probe microanalysis, X-rays diffraction, diffusion zone, multilayer structure, intermetallics.

**Introduction.** In case of study of pattern and structure of diffusion zone (DZ) the diffusion couple method can facilitate to disclose the character of element distribution and conditions for the formation of intermetallic layers. This method is based on the close contact between two metals followed by the heat treatment at the corresponding temperature for the period of time sufficient to provide the diffusion redistribution of elements. It is expected that DZ will contain all phases in that subsequence, which is observed for them in the corresponding phase diagram at the given temperature. Therefore one can evaluate the fragments of phase diagrams through measurements of component concentration in each layer.

The concentration discontinuity is considered as the boundary line between on-phase areas in the diagrams. Adjacent phases in layers are considered ones having equilibrium with each other [1]. The DZ method can be also applied for practical purposes, e.g., to enhance the oxidation resistance of Ni-superalloys using NiAl-diffusion coatings [2]. In the most of binary systems, the mutual diffusion is accompanied by the formation of phases: solid solutions existing in the limited range of concentrations, intermetallic phases having the strictly constant composition (stoichiometric compounds, i.e., daltonides), or with variable composition (non-stoichiometric compounds, i.e., berthollides) [1]. From point of view of concentration localization of the berthollides takes the intermediate position between solid solutions and daltonides [1 Page 229, 3].

Regardless of the fact that Al-Co and Al-Ni systems are well-studied in a number of original works nevertheless new phases are still discovered for them (including the phases having variable compositions) [3-4].

The knowledge on temperature-time conditions for the creation of new intermetallic phases can be laid into the basis for updated methods of the powder intermetallics metallurgy for broad application. It should be noticed that the powder intermetallics metallurgy obtains good prospects along with rapid development of additive manufacturing (AM) [5-7]. AM has a great potential in drastic reduction of costs in the manufacturing of parts in the aircraft industry, power plant industry, instrumentation manufacturing industry, i.e., in every branch where items having sophisticated geometry are used. In this context, the intermetallics inevitably will find their application in AM. It is very true from point of view of high prices on intermetallics; it is not appropriate to permit any substantial wastes of expensive intermetallics.

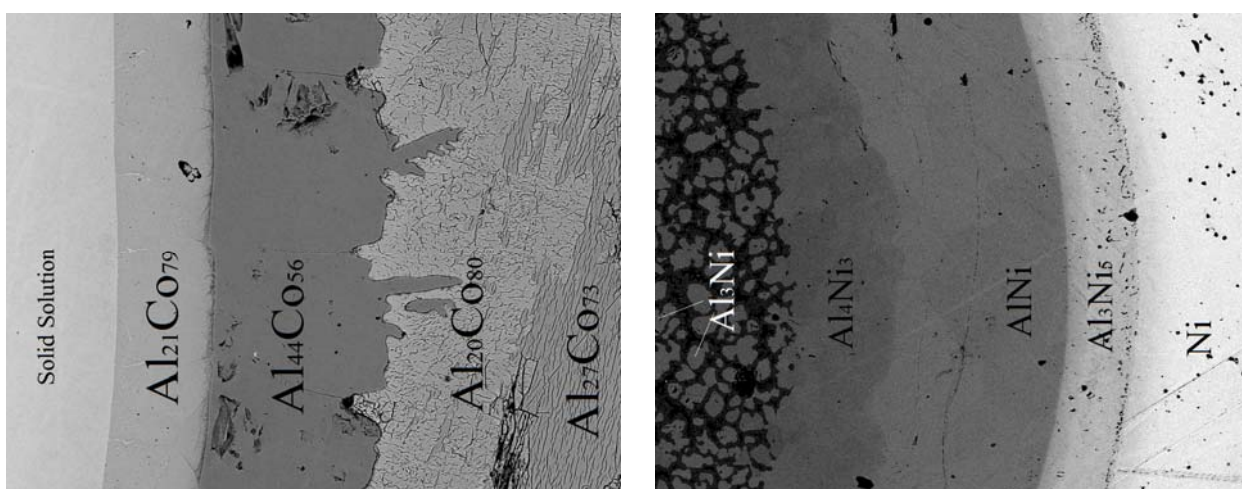
**The goal of this work** is to compare the formation of multilayer DZ structures formed between aluminium and cobalt as well as between aluminium and nickel.

**Materials and experimental methods.** The diffusion couple method has been applied to Al-Co and Al-Ni systems. Experimental details for this method are shown in [8, 9]. Samples were prepared at temperatures of isothermal treatment from 1000 °C to 1375 °C with duration from 1 to 4 hours. The initial materials were used as follows: high-grade aluminium (99,99 % Al), commercial cobalt (99,98 % Co), and nickel (99,80% Ni) in form of plates having masses up to 30 g. SEM/EPMA studies were performed on JXA-8230 (JEOL) at magnifications from  $\times 75$  to  $\times 1,000$  using the original software EPMA. Element composition of phases formed in DZ was determined by means of the spot, multipoint, and linear detection techniques using energy-dispersion spectrometry (EDS) and wave-dispersion spectrometry (WDS) demonstrated a high reproducible data obtained.

X-rays measurements were performed on a Bruker D8 Advance diffractometer operated at 40 kV voltage and 40 mA current using Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). Software EVA has been used to process XRD patterns and to compute the interplanar distances. Interpretation of samples and phase searching were performed by means of the software Search/Match using the Powder Diffraction Database PDF-2.

The modern phase diagrams for Al-Co and Al-Ni systems are not final ones; they are under permanent detailing [10-13]. Therefore widely using data taken from scientific-technical handbooks [14-17] become quickly out of date. For this reason, in this work, the data references taken from PDF-2 updating database were used.

**Results of study and their discussion.** The cross-section pattern of DZ has the following peculiarities: DZ having two or more phases the corresponding layers (as structural fragments) have different concentration profiles. In other words, there is DZ containing several layers having different contrast in the image of back-scattered electrons (figure 1).



(a) Al-Co, 1350 °C, 1 h

(b) Al-Ni, 1300 °C, 1 h

Figure 1 – Layers in DZ

In-depth sequences of phase formation (in temperature scale) in Al-Co system were reported in details in [9]. At 1350 °C all observed compounds represent were identified from a highly diluted solid solution of Al in Co to  $Al_{67}Co_{33}$ . The following intermetallic phases having constant composition were observed in Al-Co system:  $Al_9Co_2$ ,  $Al_{13}Co_4$ ,  $CoAl_3$ ,  $Al_3Co$ , and  $AlCo$  [9]. However, there are observations when layers have variable compositions.

Figure 1 (a) shows microphotography of sample after treatment at 1350 °C where DZ contain four layers with clear border lines, contrast, and characteristic structure. From Cobalt side, the layers are seemed more homogenous with “smooth” boundaries whereas next layers (to Aluminium side) have more irregular boundaries with the appearance of certain dendrites. These layers were identified as compounds with variable composition:  $Al_{21}Co_{79}$  (78.72 at.% Co),  $Al_{44}Co_{56}$  (55.69 at.% Co),  $Al_{20}Co_{80}$  (79.55 at.% Co), and  $Al_{27}Co_{73}$  (73.29 at.% Co). The similar pattern was also observed after other treatments at high temperatures 1300-1375 °C. It should be noted that new similar compound for Al-Co system with non-standard formula was obtained in case of alloying of a mixture of Al powder with intermetallic  $Al_9Co_2$  [4]. Presumably, these compounds should be referred to berthollides [18].

Similar approach was used to identify intermetallic phases having constant and variable compositions in Al-Ni system: daltonides -  $Al_3Ni$ ,  $Al_3Ni_2$ ,  $Al_3Ni_5$ ,  $Al_4Ni_3$ ,  $AlNi$ , and  $AlNi_3$ ; berthollides –  $Al_{51}Ni_{49}$  (49.07 at.% Ni),  $Al_{36}Ni_{64}$  (64.06 at.% Ni),  $Al_{30}Ni_{70}$  (69.62 at.% Ni), and  $Al_{32}Ni_{68}$  (67.86 at.% Ni). Dendrites were also observed in Al-Ni system (see figure 1 (b)) between  $Al_3Ni_5$  and pure nickel.

It should be noticed that common feature for both systems was the well-developed porosity in certain layers of DZ.

All observed intermetallics having constant compositions in Al-Co and Al-Ni systems after all isothermal treatments are compiled in table 1.

Table 1 – Intermetallics in Al-Co and Al-Ni systems

Intermetallics	Landolt-Börnstein [11-12]	Temperatures, °C	Ref.
$Al_9Co_2$	17.97 at.% Co	700	[17]
$Al_{13}Co_4$	23.19 at.% Co	800	[18]
$Al_3Co$	24.93 at.% Co	800, 900	[19]
$Al_5Co_2$	28.26-29.13 at.% Co *	1150,1200**, 1350**, 1375**	[20]
$AlCo$	50.00 at.% Co *	1150,1200, 1300, 1350, 1375	[19, 21]
$Al_3Ni$	25.78 at.% Ni *	1300**	[22]
$Al_3Ni_2$	40.00 at.% Ni *	1300	[23]
$Al_4Ni_3$	41.39-45.04 at.% Ni	1300**, 1350**	[24]
$AlNi$	50.00 at.% Ni *	1250,1350	[23]
$Al_3Ni_5$	63.51-68.07 at.% Ni *	1250**, 1300**, 1350**	[25]
$AlNi_3$	74.00 at.% Ni	1250,1350	[23, 26, 27]
<i>Note.</i> *Phase has been confirmed by X-rays measurements.			
**The corresponding phase diagram does not have this phase at given temperature.			

Table 1 demonstrates that experiments have resulted in all intermetallic compounds existing in the corresponding phase diagrams for Al-Co and Al-Ni systems. Some of these compounds were detected in those areas of phase diagram where they have not been determined before. It means that the diffusion couple method applied to Al-Co and Al-Ni systems has resulted to shift to higher temperatures of formation (or/and existence) for certain intermetallics in both systems. It is necessary to note that from a methodical point of view the discrepancy between experimentally EDS measurements and concentrations shown in Landolt-Börnstein database for each intermetallic compound [11, 12] does not exceed 0.5 at.%.

X-rays measurements confirmed the accuracy of identification of intermetallics having constant composition in DZ for both systems (figure 2).

However, some of the intermetallic layers were too small to be identified by means of X-rays pattern presumably because of its insufficient sensitivity.



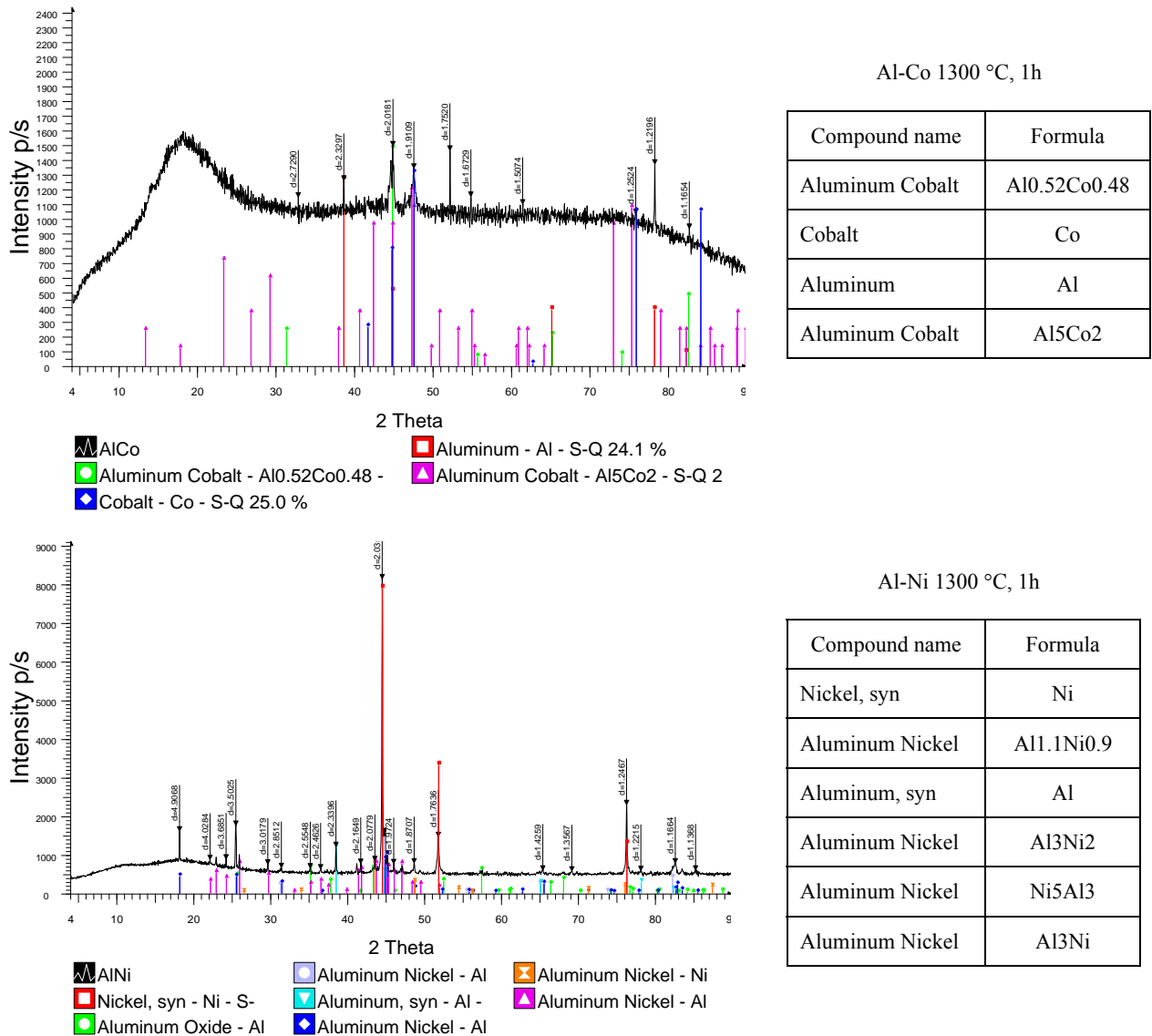
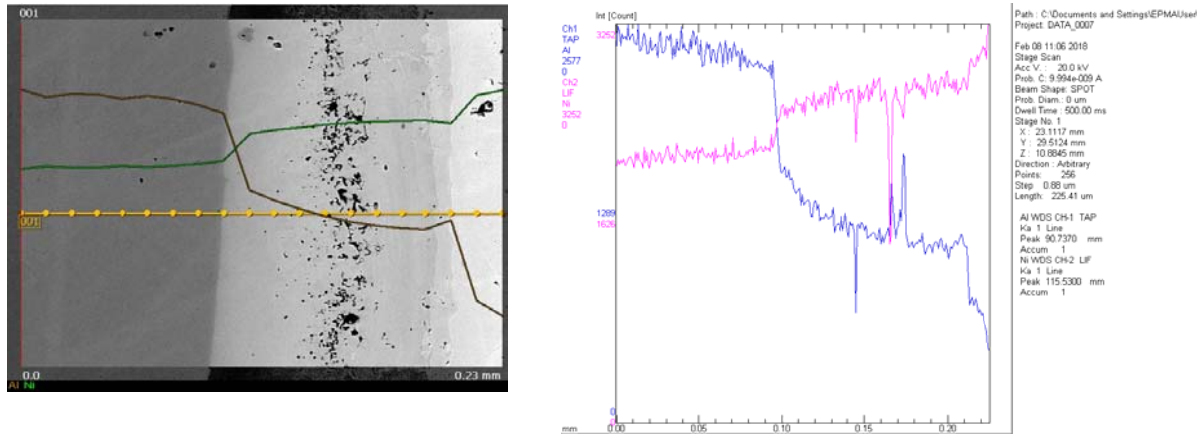


Figure 2 – Examples of X-rays patterns for Al-Co and Al-Ni systems

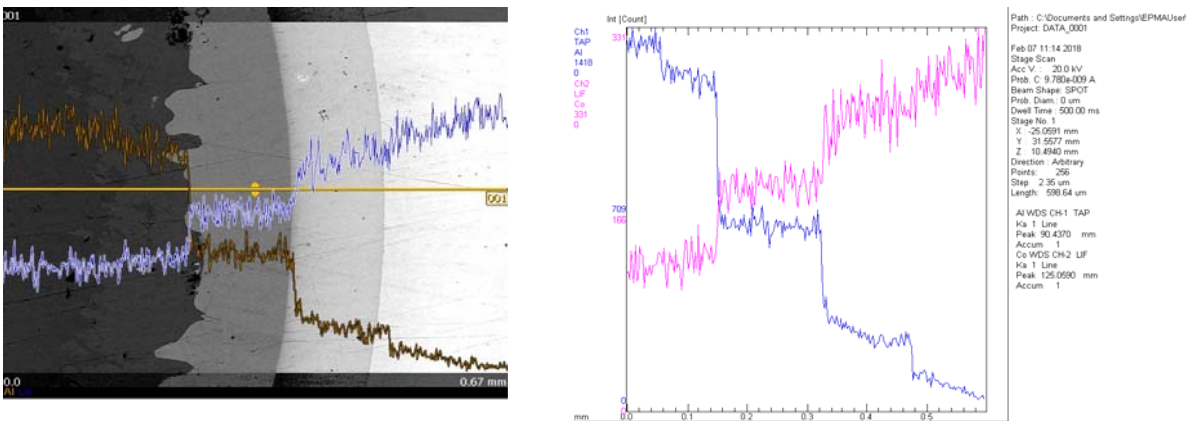
The most of samples reveal the normal element distribution in form of concentration steps on cross-sectional profiles of DZ (Figure 3 (a) and (b)). However, there are some examples of abnormal element distribution (figure 3 (c)) where one berthollide layer enriched with aluminium has two adjacent berthollide layers having lower Al concentration.

There is another important peculiarity in DZ element profiles: changes in component concentrations occur in a non-proportional manner. It is apparent that minor changes in concentration profile of Ni (or Co) correspond to more drastic changes in Al concentration profile. It is more evident when more sensitive method – WDS linear analysis – has been applied. Non-proportional changes take place not only in the area of concentration jumps (at the boundaries between layers), but also within one layer. To exclude the effect of absorption of characteristic X-rays on concentration profiles the re-measurements were performed using  $K_{\beta}(1)$  lines of cobalt and nickel instead their regular  $K_{\alpha}(1)$  lines. Behaviour of concentration profiles was pretty same. Smooth and non-proportional changes in concentration profiles should be associated with the ability of studied intermetallics to vary their component concentrations and local density.

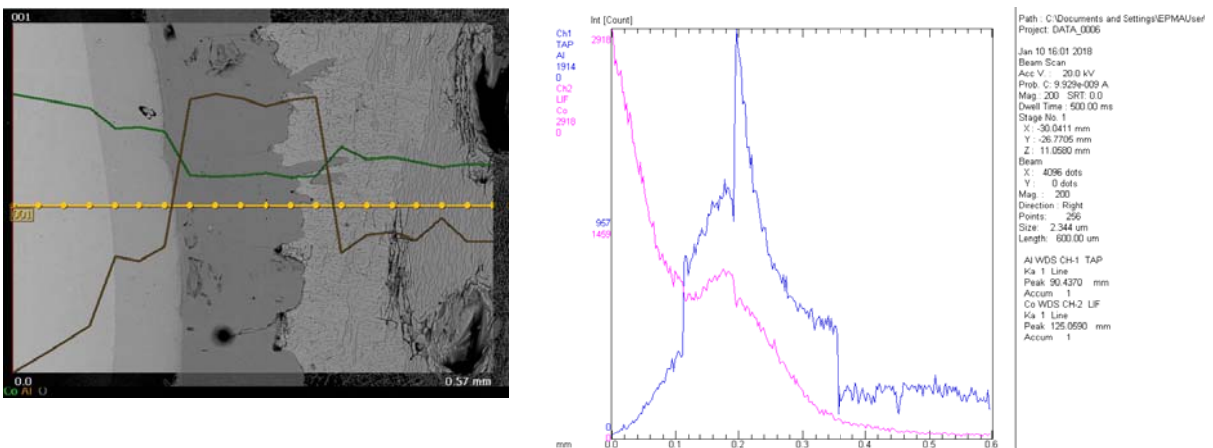
Similar features in structure of multilayer DZ in Al-Co and Al-Ni systems as well as general behaviour of their component concentration profiles could be associated with certain property (or properties) inherent to both systems. These systems are recognized as ones having omission solid solutions; therefore



(a) Al-Ni system 1250 °C, 1 h



(b) Al-Co system 1375 °C, 1 h



(B) Al-Co system 1350 °C, 1 h

Figure 3 – Element profiles in depth of DZ obtained by means of multipoint and linear EDS and WDS microanalysis

some special phenomena such as maxima in dependences of lattice parameters and density on component concentrations as well as structural defects [3 Page 232-233] can be also expected in multilayer DZ in both systems.

**Conclusions.** It was confirmed that diffusion couple method is efficient in comparative cross-sectional experiments on identification and characterization of phases formed in multilayer DZ in Al-Co and Al-Ni systems. Parallel with well-known intermetallic phases having constant composition new compounds having variable compositions were revealed – in fours for each system: (78.72 at.% Co),

Al<sub>44</sub>Co<sub>56</sub> (55.69 at.% Co), Al<sub>20</sub>Co<sub>80</sub> (79.55 at.% Co), and Al<sub>27</sub>Co<sub>73</sub> (73.29 at.% Co); Al<sub>51</sub>Ni<sub>49</sub> (49.07 at.% Ni), Al<sub>36</sub>Ni<sub>64</sub> (64.06 at.% Ni), Al<sub>30</sub>Ni<sub>70</sub> (69.62 at.% Ni), and Al<sub>32</sub>Ni<sub>68</sub> (67.86 at.% Ni).

Similar structural features in multilayer DZ in both systems (porosity, dendrites, and shift of intermetallics having constant composition to higher temperatures), as well as evidence of changes in local densities not only for different layers, but also within one layer can contribute to the specification of application of both systems to AM.

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### Al-Co ЖӘНЕ Al-Ni ЖҮЙЕЛЕРІНІҢ ДИФФУЗИЯЛЫҚ АЙМАҒЫНЫҢ КӨП ҚАБАТТЫ ҚҰРЫЛЫМЫ

**Аннотация.** Жұмыста түйіспелі балқыту әдісімен Al-Co және Al-Ni жүйелерінің диффузиялық аймағы зерттелген. Микроқұрылымы және элементтік құрамы үлгілердің көлденең кимасында растрлық электрондық микроскопия және рентгеноспектралды микроталдау (РЭМ-РСМА) және де рентген дифрактометриясының (РД) көмегімен зерттелді. Изотермиялық ұзақтықтан кейін 1000-1375°C аралығында интерметаллидтердің көп қабатты құрылымы қалыптасты. Түйіскен аймақта металдардың шоғырлану белгісіне қарай фазалық құрамы әртүрлі бірнеше қабаттар құрылады. Диффузиялық аймақта құрамы тұрақты белгілі интерметаллидтердің Al<sub>9</sub>Co<sub>2</sub>, Al<sub>13</sub>Co<sub>4</sub>, Al<sub>3</sub>Co, AlCo, Al<sub>5</sub>Co<sub>2</sub>, және де Al<sub>3</sub>Ni<sub>5</sub>, Al<sub>4</sub>Ni<sub>3</sub>, AlNi, AlNi<sub>3</sub>. түзілуі түйіспелі балқыту әдісімен расталды. Салыстырмалы тәжірибенің көлденең кима геометриясында түйіспелі балқыту әдісінің қолайлығы көрсетілген. Ауыспалы құрамы бар жаңа қосылыстар анықталды. Al-Co жүйесінде жоғарғы температуралар аймағында 1300-1375 °C Al<sub>21</sub>Co<sub>79</sub> (78.72 ат.% Co), Al<sub>44</sub>Co<sub>56</sub> (55.69 ат.% Co), Al<sub>20</sub>Co<sub>80</sub> (79.55 ат.% Co) және Al<sub>27</sub>Co<sub>73</sub> (73,29 ат.% Co) тұратын құрамы ауыспалы төрт қосылыс анықталды. Дәл солай Al-Ni жүйесінде де нақты шекаралары мен тиісті қабаттары бар Al<sub>51</sub>Ni<sub>49</sub> (49.07 ат. % Ni), Al<sub>36</sub>Ni<sub>64</sub> (64.06 ат.% Ni), Al<sub>30</sub>Ni<sub>70</sub> (69.62 ат.% Ni), Al<sub>32</sub>Ni<sub>68</sub> (67.86 ат.% Ni) анықталды.

Жалпы құрылымдық ерекшеліктері көп қабатты диффузиялық аймақ екі жүйені (кеуектілік, фестоңдар және құрамы тұрақты интерметаллидті фазалардың жоғары температураға ығысуы) сондай-ақ әр жергілікті тығыздықтың куәлікті өзгеруі түрлі қабаттардың ғана емес, бір қабаттың шегінде де аддитивті технологияның қолдануына нақты үлес қосуы мүмкін

**Түйін сөздер:** түйіспелі балқыту, растрлық электрондық микроскопия және рентгеноспектралды микроталдау, рентген дифрактометриясы, диффузиялық аймақ, көп қабатты құрылым, интерметаллидтер.

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### МНОГОСЛОЙНАЯ СТРУКТУРА ДИФФУЗИОННОЙ ЗОНЫ СИСТЕМ Al-Co И Al-Ni

**Аннотация.** В работе методом диффузионных пар исследована диффузионная зона (ДЗ) систем Al-Co и Al-Ni. Микроструктура и элементный состав образцов изучены в поперечном сечении с помощью растровой электронной микроскопии, рентгеноспектрального микроанализа (РЭМ-РСМА), а также с помощью рентгеновской дифрактометрии (РД). После изотермической выдержки от 1000°C до 1375°C сформировалась многослойная структура интерметаллидов. В зависимости от установившейся концентрации компонентов в ДЗ образуются несколько слоев различного фазового состава. Установлено образование известных интерметаллидов постоянного состава в ДЗ систем, сплавленных методом диффузионных пар Al<sub>9</sub>Co<sub>2</sub>, Al<sub>13</sub>Co<sub>4</sub>, Al<sub>3</sub>Co, AlCo, Al<sub>5</sub>Co<sub>2</sub>, а также Al<sub>3</sub>Ni<sub>5</sub>, Al<sub>4</sub>Ni<sub>3</sub>, AlNi, AlNi<sub>3</sub>. Показано, что метод диффузионных пар эффективен в сравнительных экспериментах в геометрии поперечного сечения. Выявлены новые соединения переменного состава. В системе Al-Co в области высоких температур 1300-1375 °C выявлены четыре таких слоя: Al<sub>21</sub>Co<sub>79</sub> (78.72 ат.% Co), Al<sub>44</sub>Co<sub>56</sub> (55.69 ат.% Co), Al<sub>20</sub>Co<sub>80</sub> (79.55 ат.% Co) и Al<sub>27</sub>Co<sub>73</sub> (73,29 ат.% Co). В системе Al-Ni также обнаружены соответствующие слои с четкими границами: Al<sub>51</sub>Ni<sub>49</sub> (49.07 ат.% Ni), Al<sub>36</sub>Ni<sub>64</sub> (64.06 ат.% Ni), Al<sub>30</sub>Ni<sub>70</sub> (69.62 ат.% Ni), Al<sub>32</sub>Ni<sub>68</sub> (67.86 ат.% Ni).

Общие структурные особенности в многослойных ДЗ обеих систем (пористость, фестоны и сдвиг известных интерметаллидных фаз постоянного состава к более высоким температурам), а также свидетельства изменения локальной плотности не только для разных слоев, но и в пределах одного слоя могут внести вклад в детализацию применения этих систем в аддитивных технологиях.

**Ключевые слова:** диффузионная пара, растровая электронная микроскопия, рентгеноспектральный микроанализ, рентгеновская дифрактометрия, диффузионная зона, многослойная структура, интерметаллиды.

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