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INFLUENCE OF MICROSTRUCTURE OF MULTILAYER Al/Ni FOILS ON PHASE TRANSFORMATIONS INITIATED BY HEATING

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ABSTRACT

At heating of multilayer Al/Ni foil the component interdiffusion is accompanied by phase transformations, which can occur by a two-channel multistage or single-channel one-stage schemes. It is shown that the type of the scheme by which the phase transformations develop in the studied multilayer foils, is related to the process of formation of a metastable Al_9Ni_2-phase on the layer interfaces. The work is a study of the influence of multilayer foil microstructure on formation of a metastable Al_9Ni_2-phase. It is found that the thickness of Al_9Ni_2-phase layers is determined by the thickness of aluminium layers in the initial multilayer foil. At the thickness of Al layers of 70–80 nm and greater, the thickness of Al_9Ni_2-phase layers practically does not change, and is equal to approximately 30–35 nm; at reduction of Al layer thickness, the thickness of Al_9Ni_2-phase layers decreases abruptly, and at A1 thickness less than 10–12 nm, the layers of a metastable Al_9Ni_2-phase do not form. The process of formation of metastable Al_9Ni_2-phase layers is characterized by a high rate and incubation time. Proceeding from the obtained results, a structural-kinetic diagram was proposed, which allows determination of the conditions for prevention of the multistage process of achievement of the foil equilibrium state during its heating.

KEYWORDS: multilayer foils; electron beam deposition; phase transformations; intermetallics; SHS reaction

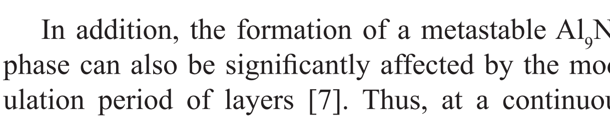
INTRODUCTION

It is known that multilayer foils (MF) consisting of Al and Ni layers can be used as intermediate layers when producing permanent joints. The process of joining materials with the use of MF is realized either by initiating the reaction of self-propagating high-temperature synthesis (SHS), which provides local heating of the joint zone, or due to the development of solid-phase reactions in MF initiated by continuous heating, which activate diffusion processes in the joint zone [1, 2]. Therefore, the possibility of predicting the kinetics of the development of phase transformations in MF, depending on their chemical composition and modulation period both in the process of continuous heating and also as a result of SHS reaction is a necessary condition for their effective practical use.

The phenomenological models, describing the propagation of the SHS reaction front in MF are based on the assumption that on the interface of the layers, a thin intermediate layer with the intermetallic structure exists, which forms in the process of MF preparation. Calculations based on these models show that the thickness of the layer of several nanometers can significantly affect the characteristics of the SHS reaction [3]. Based on this fact, it is assumed that it predetermines a high sensitivity of the SHS reaction characteristics to the method of MF preparation.

On the other hand, a number of works show that the method of MF preparation affects the initial stages of phase transformations initiated by continuous heating. For example, during heating of MF produced by the methods of magnetron [4] or electron beam deposition, instead of a stable Al_3Ni phase, first a metastable Al_9Ni_2-phase is formed [5], whereas during heating of MF produced by the methods of cold rolling of laminates [6], first, the formation of a stable Al_3Ni phase is observed intermetallic formation. A number of authors [4] associate this difference in phase formation during continuous heating of MF with a different structure of interfaces between the layers of the components, which depends on the method of foil preparation.

In [5], on the example of Al/Ni MF produced by the method of electron beam deposition with different ratios of original components and modulation periods from 50 to 500 nm, it was shown that initial stages of phase transformations with continuous heating are characterized by the formation of Al_9Ni_2-phase in the range of temperatures of 200–250 °C. At a further increase in temperature, phase transformations in MF are realized according to the two-channel scheme:



(1)

In addition, the formation of a metastable Al_9Ni_2 phase can also be significantly affected by the modulation period of layers [7]. Thus, at a continuous heating of Al/Ni foils produced by the methods of magnetron deposition, the formation of Al_9Ni_2-phase was observed only in the case of foils with a modulation period of more than 20–25 nm, and in MF with a modulation period of less than 20–25 nm intermetallic formation, that corresponds to the foil steichiometry without the formation of intermediate phases:

$$Al + Ni \rightarrow Al_nNi_m. \quad (2)$$

A similar single-channel scheme of transformations in MF, as the authors of the work [8] assume, is also realized during initiation in the SHS reaction.

Thus, the results of the conducted studies show that the sequence of phase transformations in MF depends on a number of factors that can be conditionally divided into three groups: method of MF manufacturing, characteristics of MF microstructure (chemical composition, period of layers alternation, etc.), mode of the synthesis reaction development (heating rate).

However, there are still no works in the literature aimed at establishing parameters that determine the change in the modes of the synthesis reaction depending on the characteristics of the MF microstructure and the nature of this phenomenon.

In connection with the above-mentioned, the study of the effects of Al/Ni MF microstructure on the regularities of phase transformations initiated in them by heating was conducted in order to establish the factors that determine the change in the scheme of phase transformations from the two-channel multi-stage into a single-channel one-stage.

EXPERIMENTAL STUDIES

Multilayer foils were produced by the method of electron beam evaporation of elements in vacuum with their layer-by-layer deposition on the substrate, which is described in detail in [9]. The source components were ingots of Al (99.95 %) and Ni (99.98 %). The substrate temperature in the process of deposition was 150–170 °C, the deposition rate was about 2–5 nm/s. The choice of a certain ratio between the density of the vapor flow and the rotation speed of the substrate allowed producing foils with different ratios of elements and different thicknesses of Al and Ni layers. The total thickness of foils ranged from 30 to 150 μm, and the period of alternation (λ) of layers of Al and Ni was from 25 to 600 nm. The chemical composition of foils varied from $Al_{25}Ni_{75}$ to $Al_{75}Ni_{25}$ (at.%).

The temperature intervals of phase transformations during heating of MF were studied by the DTA method in the VDTA-8 installation at a heating rate of foils of 0.8 degrees/s. To quantify thermal effects, differential scanning calorimeter (DSC) DuPont 1090 Thermal Analyser was used in the work. X-ray measurements were carried out in 0–2θ geometry in the Dron-4-07 diffractometer in the Cu–K_α radiation. The microstructure of the samples was investigated using the Camscan-4 scanning electron microscope equipped with the energy dispersive spectrometer Energy200 to determine the chemical composition.

RESEARCH RESULTS AND THEIR DISCUSSION

The study of the process of formation of a metastable Al_9Ni_2-phase was carried out by X-ray structural analysis after isothermal annealing of MF ($Al_{50}Ni_{50}$, λ = 110 nm) depending on time. It was established that the formation of Al_9Ni_2-phase occurs during heating of MF to the temperature of 250 °C (Figure 1). Exposure at a set temperature in the first moments of time leads to a slight increase in the volume fraction

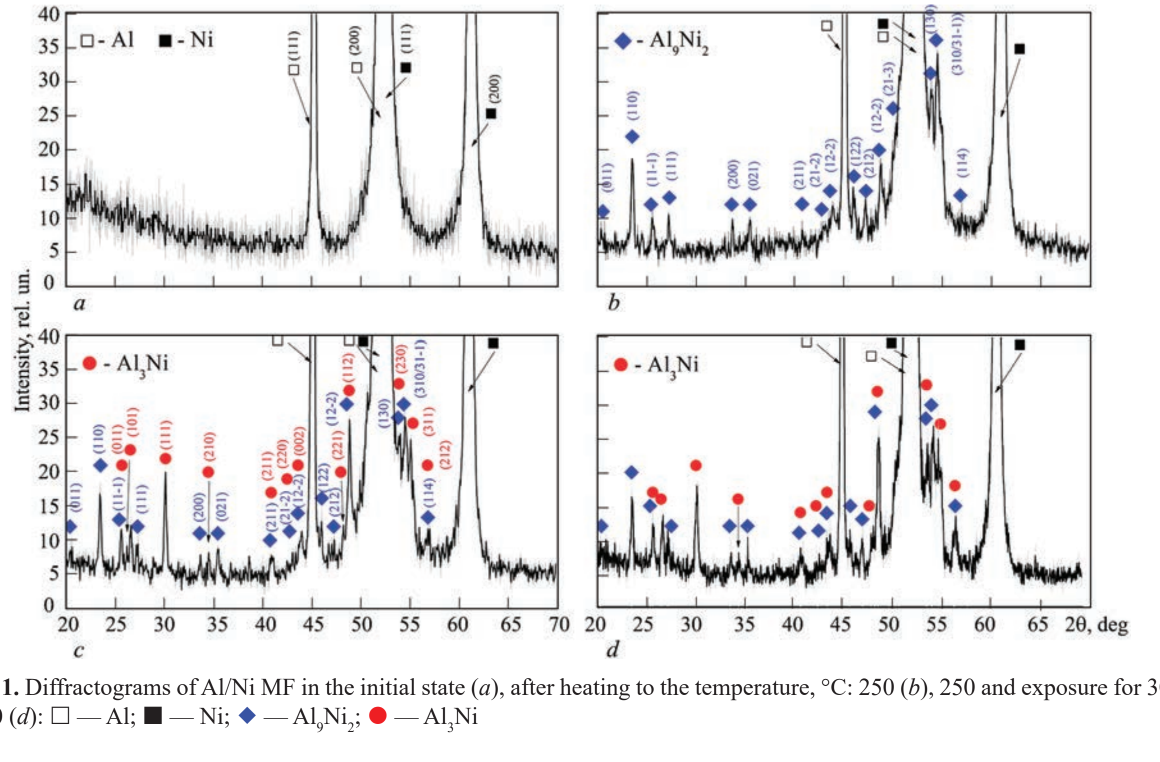


Figure 1. Diffractograms of Al/Ni MF in the initial state (*a*), after heating to the temperature, °C: 250 (*b*), 250 and exposure for 30 min (*c*), 300 (*d*): □ — Al; ■ — Ni; ◆ — Al_9Ni_2; ● — Al_3Ni

of Al_9Ni_2-phase. With the further increase in annealing time, the formation of Al_3Ni phase is observed, accompanied by a decrease in the intensity of diffraction peaks of Al and Ni. In this case, the formation of Al_3Ni-phase does not reduce the intensity of diffraction peaks (volume fraction) of Al_9Ni_2-phase. An increase in the annealing temperature of MF to 300 °C also does not lead to a further increase in the volume fraction of Al_9Ni_2-phase.

The time interval of the process of Al_9Ni_2-phase formation was evaluated by the method of in-situ X-ray diffractometry at a continuous heating of MF at 20 degrees/min. From the data of diffractograms in Figure 2, one can see that the characteristic diffraction peaks of Al_9Ni_2-phase appear for about 1 min after reaching the temperature of 250 °C, and at a further increase in temperature, their intensity remains almost unchanged. This indicates a high rate of Al_9Ni_2-phase formation and a discrete nature of transformation.

The similar results were obtained earlier in [10] while studying the kinetics of forming Al_9Ni_2-phase in the process of isothermal annealing of MF at a temperature of 200 °C, which was manufactured by magnetron deposition (Figure 3). It was found, that formation of Al_9Ni_2-phase is preceded by some delay period (incubation time). At the end of the incubation time, the formation of Al_9Ni_2-phase begins, but this process occurs only in some limited interval of time, the duration of which depends on the annealing temperature (with an increase in annealing temperature, the time of Al_9Ni_2-phase formation is reduced). According to the diagram presented in Figure 3, at annealing temperatures of about 250 °C, the transformation time should be about 1 min, which is satisfactory consistent with the obtained results. The dashed lines indicate the temperature and time interval of Al_9Ni_2-phase formation, found in this work in Al/Ni MF produced by the method of electron beam deposition (dashed lines, applied above on the diagram).

The authors of [10] believe that the presence of an incubation period in the formation of Al_9Ni_2-phase in the process of isothermal annealing of MF, the low energy of activation of the transformation process and a high rate of its developing are predetermined by the fact that Al_9Ni_2-phase formation is preceded by the process of forming of Al(Ni) solid solution on the interphase boundaries. To confirm this, the authors provide DSC data, according to which, before a low-temperature peak of the heat removal appears, characteristic of Al_9Ni_2-phase formation, a blurred peak is observed, which may be associated with the heat removal caused by the formation of a solid solution of nickel in aluminum.

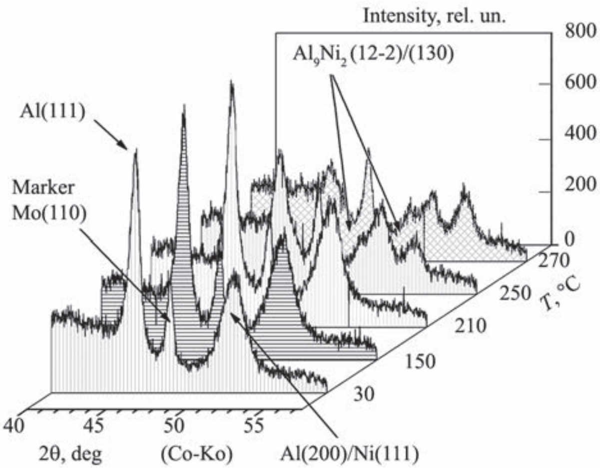


Figure 2. Fragments of diffractograms obtained directly in the process of heating Al/Ni MF (3Al:1 Ni, λ = 120 nm) at a rate of 20 °C/min (shooting frequency is 1 min^{-1})

It can be assumed that the formation of a solid solution of nickel in aluminum is possible if thermodynamic factors exist that prevent the nucleation of Al_9Ni_2-phase in the initial stages of annealing, and diffusion mobility of nickel atoms is high enough at so low temperatures.

Delay in the nucleation of Al_9Ni_2-phase may be predetermined by the presence of a significant concentration gradient on the interfaces between the layers of nickel and aluminum in MF in its original state. According to [11] in such conditions, the formation of phase nuclei becomes thermodynamically disadvantageous. On the example of a layered Al/Co system, it was shown that the nucleation of $A1_9Co_2$-phase occurs only at the moment, when the concentration profile on the elements interface becomes more gentle (after annealing at 300 °C for 5 min).

Thus, the presence of a significant concentration gradient on the elements interface can interfere with

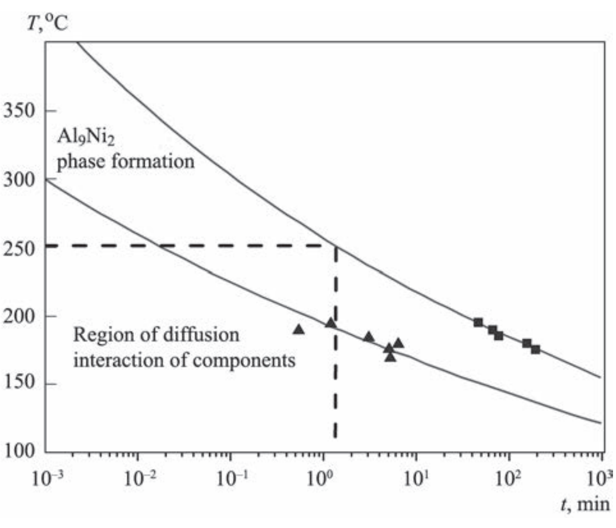


Figure 3. Temperature and time diagram of Al_9Ni_2 phase formation (solid lines and experimental points) at isothermal exposure of Al/Ni MF (the period of layers alternation λ = 80 nm) produced by magnetron deposition [10]

the formation of nuclei of the new phase at the initial stages of annealing and will promote a diffusion redistribution of elements, provided that diffusion mobility of atoms will be quite high at such low temperatures.

Taking into account that such phenomena are observed only in MF produced by the methods of physical deposition of elements at relatively low temperatures, it was suggested that their abnormally high diffusion mobility may be predetermined by the presence of a large number of non-equilibrium (excessive) vacancies. According to numerical modeling carried out in [12], the concentration of excessive vacancies in metal condensates depends on the temperature and rate of their deposition and may exceed their equilibrium value by 5–10 orders of magnitude. Considering that the concentration of vacancies significantly affects the coefficient of atoms diffusion [13], it can be assumed that the presence of a significant number of excessive vacancies in MF produced by the mehods of physical deposition, provides the process of forming of a layer of a solid solution of nickel in aluminium at the interfaces of these elements in a short time and at relatively low temperatures.

The work [5] shows the possibility of realizing the process of Al_9Ni_2-phase formation from the solid solution of Al(Ni) by the shear mechanism. Such a transformation mechanism provides the orientation ratios between the crystallographic lattices of aluminum and Al_9Ni_2-phase, whereas the transformation proper does not require a high energy of activation and can be carried out at relatively low temperatures.

From the comparison of crystalline lattices of aluminum and Al_9Ni_2-phase, it follows that during realization of this type of phase transformation, in addition to the presence of a shear component of deformation in the transformed volume, there will also be a component of deformation associated with a change in the volume, which with an increase in the thickness of Al_9Ni_2-phase layer will be accumulated. Considering that the diffusion of Al–Ni pair is characterized by the presence of significant asymmetry of diffusion flows with the predominance of nickel atoms movement into aluminum layers [14], the growth of the thickness of Al_9Ni_2-phase layer will cause significant elastic distortions in the areas of aluminum that do not participate in the transformation. As a result, when a certain value of Al_9Ni_2-phase layer is achieved, its further growth can be energetically disadvantageous. This can explain the found discrete nature of the process of Al_9Ni_2-phase formation and growth.

In addition, from the abovementioned assumptions, it follows that the growth of the thickness of Al_9Ni_2-phase layer can be influenced by both the elastic fields created by it in the matrix (aluminum), as well as by the elastic fields of adjacent Al_9Ni_2-phase layers, as when the thickness of the aluminum layers is decreased, the distance between the adjacent Al_9Ni_2-phase layers will also decrease.

While studying the microstructure of the hardened Al–Ni melt, it was found that as a result of decomposition of the oversaturated solid solution of nickel in aluminum, Al_9Ni_2-phases may precipitate in the form of plates, which are less than 30 nm thick and about 100–150 nm long [15]. Electron microscopic examinations of the morphology of Al_9Ni_2-phase, which is formed in MF in the process of its annealing, showed that in this case Al_9Ni_2-phase has a shape close to laminar one and is located mainly along the interface of the aluminum and nickel layers [7].

If we assume that Al_9Ni_2-phase is formed in MF on the boundaries between aluminum and nickel layers (Figure 4) and has a shape of plates, we can calculate the total amount of heat (Q_{teor}), which is formed by this phase transformation, proceeding from the volume of Al_9Ni_2-phase and the specific heat of its formation ΔH as:

$$Q_{theor} = Sd\rho_{Al_9Ni_2} \Delta H \frac{2d}{\lambda}, \quad (3)$$

where S and d are the surface area and the thickness of MF, respectively; λ is the period of alternation of MF layers; $\rho_{Al_9Ni_2}$ is the density of Al_9Ni_2-phase.

Figure 5 shows a characteristic diagram of heat release, obtained by the DSC method at a continious

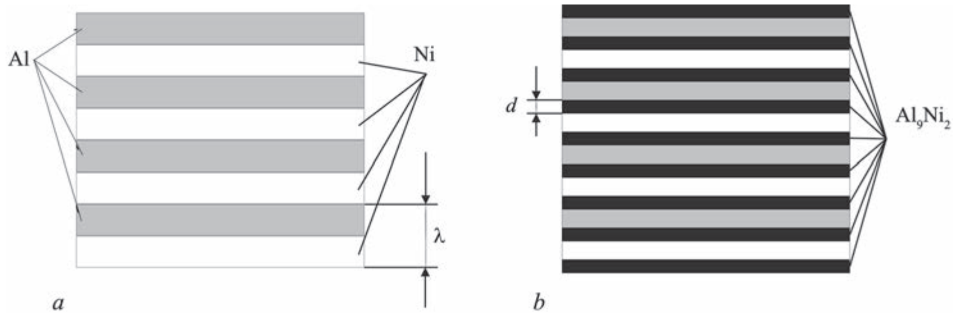


Figure 4. Scheme of microstructure of MF cross-section with the period of layers alternation λ in the initial state (*a*) and after the formation of layers of Al_9Ni_2-phase of *d* thickness (*b*)

heating of Al/Ni MF with the period of layers alternation of 110 nm and an equitatomical ratio of components. According to previously conducted studies, the first peak of heat release in the DSC diagram corresponds to the formation of Al_9Ni_2-phase. The area under the first peak (described, for example, using Gauss function) corresponds to the heat of the formation of Al_9Ni_2-phase layers.

Then the total amount of heat, released in MF at the first stage of phase transformation (Q_{exp}) during its heating, is possible to be evaluated according to DSC as:

$$Q_{exp} = ESd\rho_{MF}, \quad (4)$$

where E is the specific heat, that falls on the 1st peak of the DSC diagram (Al_9Ni_2-phase formation) during heating of MF; ρ_{MF} is the densityof MF.

Neglecting the temperature dependence of the value ΔH, which is the heat of Al_9Ni_2-phase formation in [7], let us suppose that $Q_{theor} = Q_{exp}$ and evaluate the thickness of the layer of Al_9Ni_2-phase, which is formed in the process of continuous heating of MF as:

$$d = \frac{E\rho_{MF}\lambda}{2\rho_{Al_9Ni_2}\Delta H}. \quad (5)$$

The results of the calculation of the specific value of the heat release, which falls for the 1st peak of the DSC curve, and the values of the thickness of the layer of this phase, calculated for the foils with a close chemical composition and different modulation periods, are presented in Table 1.

It is necessary to draw attention to the fact, that the thickness of Al_9Ni_2-phase layers decreases with a decrease in the thickness of aluminum layers (Figure 6). Moreover, this dependence has a significantly nonlinear nature. Thus, reducing the thickness of Al layers to 70–80 nm has almost no effect on the thickness of Al_9Ni_2-phase layers, which is about 30–35 nm, whereas at the thicknesses of the aluminum layers smaller than 70 nm, a sharp decrease in the

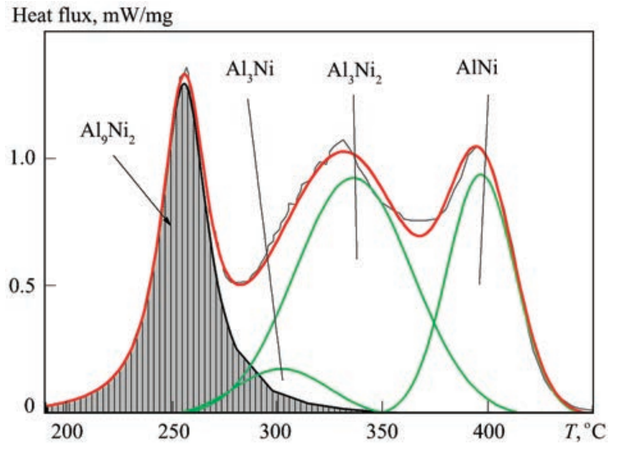


Figure 5. DSC diagram obtained during continuous heating of Al/Ni MF of equatomic composition with a period of 110 nm

thickness of Al_9Ni_2-phase layers is observed, which at the values of the thickness of the aluminum layers of about 12 nm reaches the value of the order of 2 nm. Obviously, when the thickness of aluminum layers is reduced, the distance between the adjacent Al_9Ni_2-phase layers will also decrease. In view of this, it can be assumed that when some critical value of the thicknesses of aluminum layers is reached (of the order of 10–12 nm), elastic distortions that will occur in the aluminum matrix as a result of Al_9Ni_2-phase layers formation will suppress their further growth. In the case of equatomic composition of MF, these conditions are reached during a modulation period of about 20–25 nm.

Previously, a number of studies showed that in MF with a modulation period of less than 25 nm, Al_9Ni_2-phase is not formed [7], and transformations occur under a single-channel one-stage scheme (2).

Therefore, it can be assumed that the thickness of aluminum layers can affect the sequence of phase transformations in MF during their continuous heating. If the thickness of the aluminum layer of MF is larger than some critical value, then the phase transformation will develop according to a two-channel multistage scheme (1), and if the thickness of the alu-

Table 1. Characteristics of MF microstructure and thickness of layers of Al_9Ni_2 phase formed in it during heating

Number	Chemical composition of MF, at.%	Specific heat release which falls on the 1st peak (E), J/g	Period of layers alteration, nm	Thickness, nm	
				of Al layers	of Al_9Ni_2 phase layers
1	$Al_{86.5}$–$Ni_{13.5}$	+261	266	230	37.5
2	Al_{51}–Ni_{49}	+208	200	137	34.5
3	$Al_{61.2}$–$Ni_{38.8}$	+192	135	95	27.5
4	Al_{67}–Ni_{32}	+294	140	93	33.0
5	Al_{63}–Ni_{37}	+332	110	69	29.0
6	$Al_{86.5}$–$Ni_{13.5}$	+445	63	55	14.8
7	Al_{42}–Ni_{58}	+107	106	44	10.4
8	Al_{49}–Ni_{51}	+103	26	13	2.5

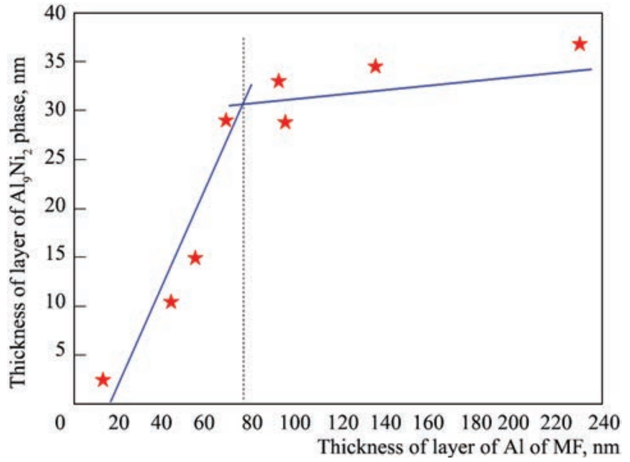


Figure 6. Dependence of thickness of the plate of Al_9Ni_2-phase formed in MF during its heating on the thickness of aluminum layers

minum layer is less than this value, it will develop according to a one-stage scheme (2).

To study the sequence of structural changes in the foils of this type, MF with the ratio of components 2Al:3Ni were produced, in which the thickness of aluminum layers was about 10 nm, which, according to the evaluation, is commesurable with a critical value. From X-ray studies it is seen (Figure 7) that during heating of MF to the temperature of 240 °C, a diffraction peak (111) from aluminum disappears. However, the diffraction peaks inherent in Al_9Ni_2-phase do not appear. Moreover, even heating to a higher temperature (300 °C) also does not lead to the appearance of diffraction signs of forming of either Al_9Ni_2-phase nor other intermetallides inherent in the mentioned system. Only heating above 300 °C is accompanied by the appearance of enlarged diffraction peaks in the positions, characteristic of AlNi phase (reaction product).

Thus, within the framework of the abovementioned model of phase transformations in MF, initiated by heating, in the case of MF with the thickness of the aluminum layer which is less than a critical one, the phase transformations develop by a single-channel one-stage scheme (1).

From the comparison of the results of X-ray-structural studies obtained in the process of continuous (slow) heating of MF with a thickness of the aluminum layer, which is less than a critical value (this work), with the results obtained in-situ at a high-rate heating of MF with the thickness of the aluminum layers greater than the critical value (in the process of developing SHS reaction front) in [8], it is possible to note the similarity of changes in diffraction patterns for both cases. Thus, in MF where the thickness of the aluminum layer is larger than a critical one (λ = 100 nm) due to a high heating rate during the SHS reaction (of the order of 10^5 °/s), the formation of Al_9Ni_2-phase is not observed, but the formation of AlNi intermetallic occurs by a single-channel one-stage scheme. Moreover, in both cases, the disappearance of diffraction peaks from aluminum is accompanied by the appearance of a wide diffuse peak (halo), which may indicate the formation of an oversaturated solid solution of nickel in aluminum, the appearance of which precedes the formation of the intermetallic phase.

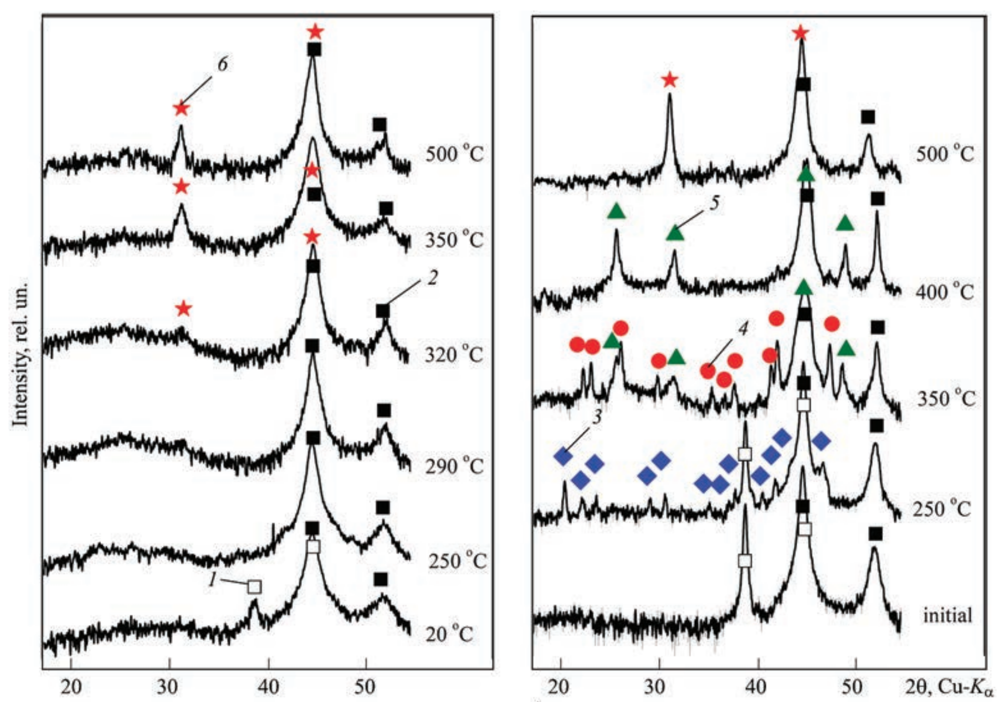


Figure 7. Diffractograms of MF with the elements ratio of 2Al:3Ni and the period of layers alternation of 25 nm (*a*) and 140 nm (*b*) in the initial state and after their heating to the temperature indicated in the pattern of temperatures: *1* — Al; *2* — Ni; *3* — Al_9Ni_2; *4* — Al_3Ni; *5* — Al_3Ni_2; *6* — AlNi

Thus, summarizing the obtained results, the patterns of developing phase transformations in Al/Ni MF can be represented in the form of a scheme shown in Figure 8. According to this scheme, the factors that affect the change in the mode of developing phase transformations from a multi-stage into a one-stage character, in the case of MF of equiatomic composition, are the period of layers alternation (thickness of the aluminum layer) and heating rate. For MF with the period of alternation of components layers smaller than 25 nm, regardless of heating rate, the phase transformation occurs by a single-channel one-stage scheme, whereas with an increase in the period of layers alternation, the realization of such a scheme is possible on the condition of high heating rates (at the level of SHS reaction or higher).

The latter is predetermined by the fact that aluminum in the initial stages is transformed into a solid solution of nickel in aluminum, which at a further increase in temperature will be transformed into an intermetallic phase inherent in this chemical composition of MF, bypassing the formation of intermediate intermetallics.

Indeed, from the kinetic diagram of Al_9Ni_2-phase formation (Figure 3), it is seen that at heating rates higher than 10^4 °C/s, the temperature of the beginning of Al_9Ni_2-phase formation becomes commeasurable with the temperature at which this phase loses its stability because, as is shown in [5], during heating of Al/Ni MF of equiatomic composition to more than 350 °C, Al_9Ni_2-phase decomposes. These model notions about the effect of heating rate on the sequence of phase transformations in MF are consistent with the results of in-situ X-ray studies of phase transformations during the SHS reaction (the rate of heating MF of the order of 10^5 °C/s), which showed that the synthesis of interametallic (reaction product) occurs without the formation of intermetallic phases [8].

CONCLUSIONS

1. For Al/Ni MF produced by the method of physical deposition (magnetron spraying or electron beam evaporation of components), the scheme of developing phase transformations (two-channel multi-stage or single-channel one-stage) is determined by the possibility of formation of the foil of a metastable Al_9Ni_2-phase in the initial stages of heating.

2. The dependence of the sequence of phase transformations on the conditions of producing MF is associated with the oversaturation of the foil with non-equilibrium vacancies that accelerate the diffusion of nickel into layers of aluminum at relatively low temperatures, which provides the formation of a solid solution of nickel in aluminum.

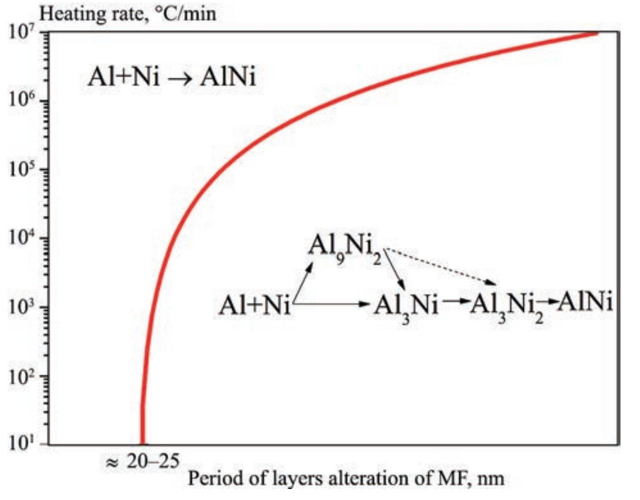


Figure 8. Scheme of effect of the modulation period of Al/Ni MF of equatomic composition on the sequence of phase transformations depending on heating rate

3. It is shown that transformation of a solid solution of nickel in aluminum into Al_9Ni_2-phase is only possible when the thickness of aluminum layers is larger than some critical value (about 10–12 nm) and the heating rate is less than 10^4 °C/s.

4. The scheme of influence of the period of layers alternation of MF and heating rate was proposed, which allows determining the scheme of realization of phase transformations initiated by heating.

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