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THE APPLICATION OF NANOSTRUCTURAL FOIL FOR DIFFUSION WELDING OF COMPOSITE BASED ON ALUMINIUM ALLOY 6061 CONTAINING 55% SiC

ДИФУЗІЙНЕ ЗВАРЮВАННЯ КОМПОЗИТУ НА ОСНОВІ АЛЮМІНІЄВОГО СПЛАВУ 6061, ЩО МІСТИТЬ 55% SiC З ВИКОРИСТАННЯМ НАНОСТРУКТУРНОЇ ФОЛЬГИ

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Abstract. The process of diffusion welding in vacuum (DWV) of an aluminum-based composite using nano-multilayer aluminum–nickel and aluminum–titanium foils is investigated. Aluminum alloy 6061 hardened silicon carbide (55 at.%). It has low ductility, which makes deformation activation of the surfaces being joined difficult. Therefore, DWV perform through the intermediate layer, for example, aluminum foil. In this work, the layers obtained by the method of electron beam deposition were used. In layers with alternating nanolayers with rapid heating, reactions of self-propagating high-temperature synthesis take place, in which the amount of generated heat is sufficient for additional heating of the composite and it’s melting, which ensures activation of the surfaces to be joined. At the first stage, the creep processes of the composite under DWV conditions are investigated, the parameters and the creep equation are determined, as well as the allowable deformations that do not cause cracks. For DWV, Al/Ni and Al/Ti foils with thicknesses of 20, 40, 50, and 94 microns were used. The effect of the thickness of the interlayers on the formation of compounds has been established. It is shown that, regardless of the interlayer system, at the majority of the junction metal has a composite structure with common grains and the boundary between the connected samples is not determined either by structure or by chemical analysis. In areas with a layer of its maximum thickness does not exceed 9 microns. At a thickness of 41 μm, the structure is slightly different from the structure at a foil thickness of 20 μm. With a foil thickness of 90 microns, an interlayer 60...70 microns thick extends along the entire length of the joint. She reacted completely. In the center of the interlayer, its composition is close to the composition of the Ni_3Al phase. It has been established that the use of nano-multilayer foils of the Al/Ni and Al/Ti system ensures the formation of high-quality defect-free compounds with the realization of self-propagating high-temperature synthesis of intermetallic compounds. To calculate the parameters of the DWV regime, it is sufficient to use the creep equation of the composite at the steady state.

Keywords: composite; nano-layered foil; creep; self-propagating high-temperature synthesis; chemical composition; microhardness.

Анотація. Досліджений процес дифузійного зварювання у вакуумі (ДЗВ) композиту на основі алюмінію з використанням наноструктурних фольг систем алюміній–нікель та алюміній–титан. Сплав алюмінію 6061 зміцнюється карбідом кремнію (55 % ат.). Він має низьку пластичність, що ускладнює деформаційну активацію з'єднуваних поверхонь. Тому ДЗВ виконували через проміжні прокладки, наприклад алюмінієву фольгу. Застосовані прокладки, отримані методом електронно-променевого осаджування. У прокладках з поперемінними наношарами під час швидкого нагрівання проходять реакції самопоширюваного високотемпературного синтезу, внаслідок якого кількість виділеного тепла достатня для додаткового нагрівання композиту і його підплавлення, що забезпечує активацію з'єднуваних поверхонь. На першому етапі досліджені процеси повзучості композиту в умовах ДЗВ, визначені параметри і рівняння повзучості, а також допустимі деформації, що не призводять до утворення тріщин. Для ДЗВ використали фольгу систем Al/Ni і Al/Ti товщиною 20, 40, 50 і 94 мкм. Установлено вплив товщини прокладок на формування з'єднань. Показано, що незалежно від системи прошарків на більшій частині стику метал має структуру композиту із загальними зернами і межі між з'єднаними зразками не визначаються ні за структурою, ні за хімічним аналізом. На ділянках з прошарком його максимальна товщина не перевищує 9 мкм. При товщині 41 мкм структура мало відрізняється від структури при товщині 20 мкм. При товщині фольги 90 мкм прошарок товщиною 60…70 мкм спостерігається на всій довжині стику. Вона прореагувала повністю. У центрі прошарку його склад близький до складу фази Ni_3Al. Установлено, що застосування нанобагатошарових фольг системи Al/Ni і Al/Ti забезпечує формування якісних бездефектних з'єднань з реалізацією самопоширюваного високотемпературного синтезу інтерметалідів. Для розрахунку параметрів режиму ДЗВ достатньо використати рівняння повзучості композиту на усталеній стадії.

Ключові слова: композит; нанобагатошарова фольга; повзучість; самопоширюваний високотемпературний синтез; хімічний склад; мікротвердість.

Аннотация. Исследован процесс диффузионной сварки в вакууме (ДСВ) композита на основе алюминия с применением наномногослойных фольг систем алюминий–никель и алюминий–титан. Сплав алюминия 6061 упрочняется карбидом кремния (55 % ат.). Он имеет низкую пластичность, что затрудняет деформационную активацию соединяемых поверхностей. Поэтому ДСВ выполняют через промежуточные прослойки, например алюминиевую фольгу. Применены прослойки, полученные методом электронно-лучевого осаждения. В прослойках с чередующимися нанослоями при быстром нагреве протекают реакции самораспространяющегося высокотемпературного синтеза, при котором количество выделяющегося тепла достаточно для дополнительного нагрева композита и его подплавления, что обеспечивает активацию соединяемых поверхностей. На первом этапе исследованы процессы ползучести композита в условиях ДСВ, определены параметры и уравнение ползучести, а также допустимые деформации, не вызывающие трещин. Для ДСВ использовали фольгу систем Al/Ni и Al/Ti толщиной 20, 40, 50 и 94 мкм. Установлено влияние толщины прослоек на формирование соединений. Показано, что независимо от системы прослоек на большей части стыка метал имеет структуру композита с общими зернами и граница между соединенными образцами не определяется ни по структуре, ни по химическому анализу. На участках с прослойкой ее максимальная толщина не превышает 9 мкм. При толщине 41 мкм структура мало отличается от структуры при толщине фольги 20 мкм. При толщине фольги 90 мкм прослойка толщиной 60…70 мкм идет по всей длине стыка. Она прореагировала полностью. В центре прослойки ее состав близок к составу фазы Ni_3Al. Установлено, что применение наномногослойных фольг системы Al/Ni и Al/Ti обеспечивает формирование качественных бездефектных соединений с реализацией самораспространяющегося высокотемпературного синтеза интерметаллидов. Для расчета параметров режима ДСВ достаточно использовать уравнение ползучести композита на установившейся стадии.

Ключевые слова: композит; наномногослойная фольга; ползучесть; самораспространяющийся высокотемпературный синтез; химический состав; микротвёрдость.

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Problem statement. Composite materials with dispersion hardening are known for a long time. Such materials with matrices of aluminum alloys and their properties are described in [1]. Dispersion-strengthened materials based on aluminum and copper are most often produced by powder metallurgy methods.

In dispersively strengthened materials, the carrier element is a matrix in which, using ultrafine particles of oxides, carbides or other particles that are practically insoluble in the matrix, effective deceleration of the dislocations is ensured and high strength characteristics and functional properties are maintained up to the melting point. The problem of their widespread use is the lack of reliable methods of joining in both homogeneous and heterogeneous combinations: fusion welding leads to the destruction of their structure and loss of mechanical properties. The most widely used diffusion welding with intermediate layers [2–4], but soldering and welding in the solid state are difficult due to poor wettability and activation of the joined surfaces. Therefore, this work is relevant.

Latest research and publications analysis. Diffusion welding and brazing of composite materials began to be applied at the end of the last century [2, 5, 6]. Diffusion welding was usually performed using non-melting or melting interlayers, which used adhesive-active solders [5]. In work [2] a composite material based on aluminum 2124, containing 25...30 at. %. silicon carbide, welded both directly between themselves and through an aluminum interlayer and obtained joint strength at 110 MPa (below 50 %

of the base metal strength) at a welding temperature of 500 °C, pressure of 10 MPa, welding time of 240 minutes.

Increasing the welding temperature to 570 °C of composite 2124 using aluminum foil 15 mkm thick at a pressure of 16 MPa and a welding time of 60 minutes ensures a 127 MPa cut resistance [3].

In [7, 8], the DWV of composite 6061 was investigated at temperatures of 400...650 °C, hardened at 18 % at. SiC and 30% Al_2O_3, respectively. The effects of temperature, compression pressure and welding time were investigated. For a composite with silicon carbide, the welding mode is recommended: T = 620 °C; P = 5 MPa, t = 30 min, at which the bond strength of 210 MPa (72 % of the strength of the base material) is achieved. For the composite containing Al_2O_3, the welding mode is recommended: T = 600 °C; P = 5 MPa, t = 30 min, at which the ultimate strength σ_B = 130 MPa (60 % of the strength of the base material).

The characteristics of DWV of dispersion-strengthened composite materials are described in the review [4], and the welding of a composite based on an aluminum alloy containing 20 % silicon carbide SiC (4.0 % C) was studied in [9, 10]. The authors conducted DWV at a temperature of 500...600 °C, pressure up to 3.0 MPa with a welding time of 30 minutes. There is heterogeneity of the compound and the presence of defects. When using aluminum foil, its thickness is recommended 150 mkm. The same foil thickness was recommended in [11] with an DWV of aluminum with titanium.

In published works, the recommended parameters of diffusion welding with non-melting highly plastic microslips vary over wide intervals. As such layers, use technical aluminum or alloyed with magnesium, silicon, copper or lithium, which have superplasticity. Eutectic melts are used as melting layers [12].

It is known [13] that the main parameters of the DWV mode are temperature, pressure and welding time. The change in each of them determines the amount of plastic deformations that provide the formation of physical contact, deformation activation and volumetric interaction (recrystallization and diffusion), but in the published works they determined the total deformation after welding, without its control during welding and the relationship with temperature, pressure and time . Therefore, the deformation varied from 6 to 36.3 %, which does not allow establishing the patterns of formation of joints and ensuring stable welding quality.

A new way to connect composites is the use of nanofolg. At the Paton Electric Welding Institute of NAS of Ukraine has developed a technology for producing multilayer foils by electron beam deposition method [14], which were used in DWV [15, 16]. In layers with alternating nanolayers with rapid heating, reactions of self-propagating high-temperature synthesis (HTS) occur, in which the amount of emitted heat is sufficient to heat the metal in the joint up to 1200 °C and melt it, which ensures activation of the surfaces to be joined [14]. In the Al/Ni interlayer (Al + 57.7 % wt. Ni), the intermetallic formation reaction occurs between solid nickel and liquid aluminum, and in the Al/Ti interlayers, when heated in the temperature range 250...650 °C, phase transformations occur in this sequence [17, 18]: $Al + Ti \rightarrow Al_3Ti \rightarrow$ $\rightarrow Al_5Ti \rightarrow Al_2Ti \rightarrow AlTi$.

Separation of previously unsolved parts of the general problem. In the studies discussed above, relatively plastic composites were combined, including those based on aluminum with a silicon carbide content of up to 20 %. It is known [19] that with an increase in the amount of the hardening SiC or Al_2O_3 phase, the plasticity of the composite sharply decrease. Therefore, on the instructions of the Customer, it was necessary to investigate the possibility of a DWV composite containing 55 % SiC, the information on which DWV using multilayer nanostructured foils is not available in the literature. The main material and foil were supplied by the Customer.

THE ARTICLE AIM — is to investigate diffusion welding in vacuum (DWV) of a composite based on an aluminum alloy 6061 containing 55 % silicon carbide using a multilayer nanostructured foil and to recommend its chemical composition.

Methods, object and subject of research

The object of the research is the processes of creep and formation of compounds with a DWV composite based on aluminum alloy 6061 using nanostructured foils.

The subject of the research is the creep parameters of alloy 6061 under DWV conditions, the structure and chemical composition of compounds.

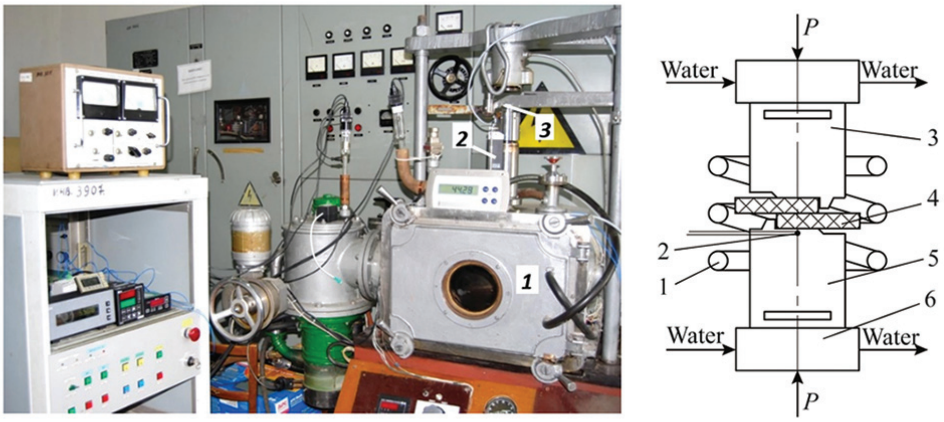
Research methodology

For the DWV study, a composite material (CM) was used, the matrix of which is aluminum alloy 6061, and the hardening phase is silicon carbide SiC in the amount of 55 %. Alloy 6061 contains (% wt.) 0.15 Cu; 0.15 Mn; (0.8–1.2) Mg; 0.25 Zn; (0.04–0.35) Cr; 0.15 Ti; (0.4–0.8) Si; 0.7 Fe; the rest of Al. We used 65×25×3 mm plates with a polished surface for welding. Welding of the samples was carried out with an overlap through an intermediate strip of Al/Ni (AN) and Al/Ti (AT) nanomultilayer foil with a thickness of 20 to 94 μm, obtained in 23… 335 deposition cycles. The maximum foil thickness of 93 microns has 335 cycles, the minimum thickness of 20 microns has 23 cycles.

DWV was performed on a UDSV-DT diffusion-vacuum unit, the general view of which is shown in Fig. 1, *a* and Fig. 1, *b* shows the assembly of samples in a vacuum chamber.

In DWV, it is necessary to provide an interatomic connection between the surfaces being welded due to local plastic deformation at elevated temperatures. The magnitude of the compressive force was chosen from the condition for ensuring the plastic deformation of the samples due to creep processes at stresses below the yield strength of the composite material.

The UDSV-DT installation provides a working vacuum of 10^{-2} Pa. The parts are heated by high frequency currents. Weldable plates *4* were installed between the rods *3*, *5* (Fig. 1, *b*). For heating, a cylindrical inductor *1*



a) b)

Fig. 1. General view of the UDSV-DT installation for DWV (*a*) and the scheme of sample assembly in the working chamber (*b*): *a*) *1* — vacuum chamber; *2* — displacement sensor; *3* — load sensor; *b*) *1* — inductor; *2* — thermocuple; *3* — top rod; *4* — weldable composite; *5* — bottom stock; *6* — cooler

was used, in which the upper and lower rods were heated, transmitting heat to the overlap plates of the composite due to heat conduction.

The installation has an automatic maintenance of the welding temperature, as well as high-precision displacement sensors and loads of the type LIR-14 00PS 11 and RINC3 / 1t, respectively, with the recording of their readings and the values of the temperature values on a computer. The temperature was measured with a chromel-alumel thermocouple with an accuracy of ±2 °C, which was maintained by an electronic regulator. The installation uses 2 loading mechanisms: lever and hydraulic. The parameters of plastic deformation of the composite were determined by the method of [15, 16] at the same installation.

To research the microstructure of the compounds and the interaction of the composite material with the interlayer, a NEOFOT 21 microscope and a scanning electron microscope — microanalyzer REMMA 102-02A were used, on which a local X-ray spectral microanalysis was also performed.

Basic material. The main parameters of the DWV mode are temperature, pressure and welding time. The vacuum in the working chamber was 10^{-2} Pa. The sequence diagram of the DWV process is shown in Fig. 2.

After installing the samples in the working chamber, they were compressed by the set pressure of P_p to avoid their displacement in the device. With increasing temperature, the pressure of compression was reduced to the value of the heating pressure P_h in order to avoid plastic deformation.

When the temperature reached the welding temperature T_w, the welding pressure P_w was set. After holding at the welding temperature, the preset time was reduced to P_h and the heating was turned off.

After welding several samples, the tendency of the composite to crack formation and the need to control the level of deformation during the welding process were established. For this, the parameters of the creep process of the composite in the second stage, the effect of temperature and pressure on the creep rate were determined, and high-precision instruments were used to control the deformation.

In the second creep stage, the strain rate is steady and is determined by the equation [20, 21]:

$$\dot{\varepsilon} = A \cdot p^m \exp\left(-\frac{\Delta H_c}{RT}\right), \quad (1)$$

where $\dot{\varepsilon}$ — is the creep rate; ΔH_c — creep activation energy; p — is the compression pressure; T — is the temperature; R — is the universal gas constant; A, m — coefficients for this material.

At the first stage of research, the activation energy of creep was determined by the method of small temperature jumps [15, 16], as shown in Fig. 3.

As can be seen from Fig. 3, the strain curve of the samples was first recorded at a temperature of 605 °C, then the temperature rose to 620 °C and the strain curve at 620 °C was recorded at the same pressure. Accordingly, the strain rates were determined (lines A-A and B-B), and the activation energy was determined by the equation

$$\Delta H_c = \frac{R \cdot T_1 \cdot T_2}{T_1 - T_2} \ln \frac{\dot{\varepsilon}_2}{\dot{\varepsilon}_1},$$

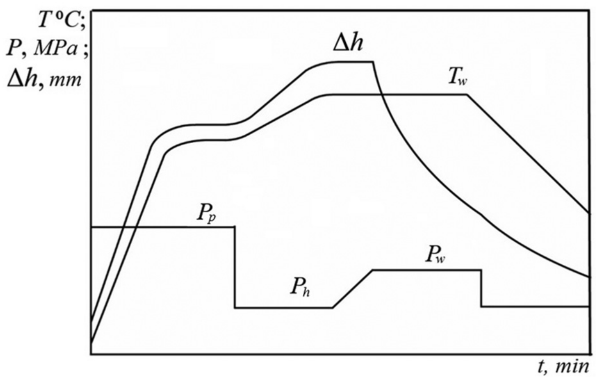


Fig. 2. The sequence diagram of the DWV composite process

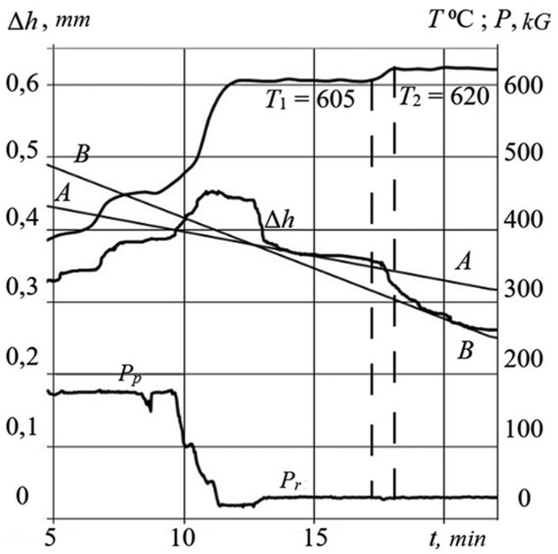


Fig. 3. Determination of the creep activation energy by the method of small temperature jumps

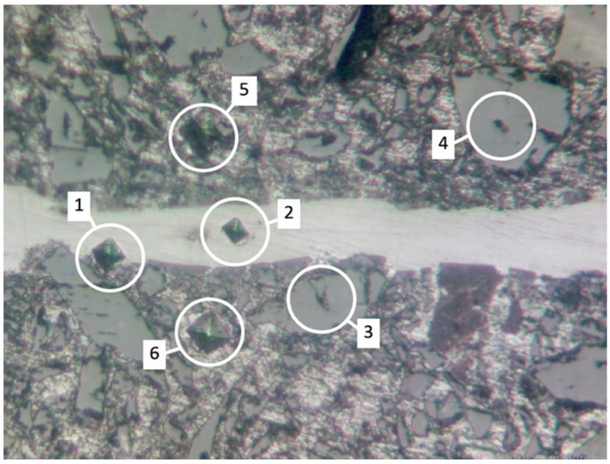


Fig. 4. The microstructure of the compound in the DWV of the composite, through an Al/Ti interlayer containing 22.9 % at. Ti, δ = 41 mkm

where $T_1 = 878$; $T_2 = 893$ on the Kelvin scale; $\frac{\dot{\varepsilon}_2}{\dot{\varepsilon}_1} = 1.75$ creep activation energy $\Delta H_c = 242$ kJ/mol.

After conducting studies at other pressures, using the equation (1), the coefficients m, A are determined: $m = 2.1$; $A = 1.3 \cdot 10^{-4}$.

Using these parameters and equation (1), it is possible to determine the combination of temperature, pressure and welding time, which will ensure the allowable deformation of the composite without the appearance of cracks. The displacement sensor allows, if necessary, to carry out adjustment of the mode. We have chosen the welding temperature of 590 ± 5 °C and determined the allowable amount of plastic deformation of 5...7 %. Depending on the thickness of the interlayer, the amount of permissible deformation varies somewhat in the case of melting of the base metal.

In our studies, Al/Ni foils with a thickness of 20, 40, 50, and 94 mkm were used. The Al/Ti system foil contained about 22.5 % at. Titanium, the Al/Ni system foil contained about 40, 45 and 55 % at. Nickel.

After welding the microstructure, microhardness and chemical composition of the metal in the joint zone were investigated. For example, Fig. 4 shows the microstructure of welded joint with Al /Ti interlayer with a thickness of 41 mkm, containing 22.9 % at. Titanium.

Hardness was measured at the center of the interlayer (p. 2), in the transition zone (p. 1), in large inclusions in the composite (p. 3, p. 4) and in the composite matrix (p. 5, p. 6). Inclusions in the composite have high microhardness (more than 19500 MPa) and represent silicon carbides. The composite matrix has a microhardness of 2600...2800 MPa. In the center of the interlayer the microhardness is 4470 MPa and in the transition zone — 2500 MPa.

The material in the center of the interlayer is the following chemical composition (% at): 67 Al; 16 Si; 15 Ti, which is different from the chemical composition of the interlayer. This indicates its interaction with the base metal. Along the junction there are areas where there is no solid layer and the joint cannot be determined, since the structure of the metal is uniform. The joint is not determined by chemical composition using microanalysis. Such a structure is shown in Fig. 5.

A similar structure is formed when using Al/Ni foil containing 54.8 % at. Ni, with a thickness of 20 mkm (Fig. 6, *a*).

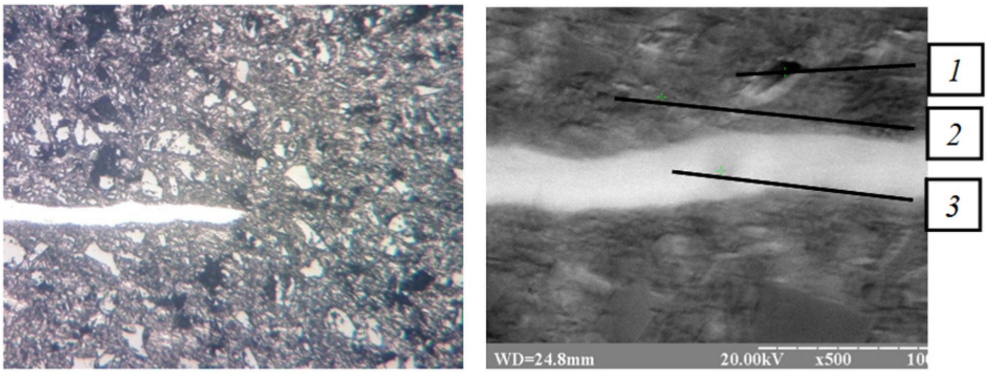
On the non-extended areas of the joint, where there is an interlayer, it does not have a clear boundary with the base metal and will smoothly disappear from the joint (Fig. 6, *b*). This indicates the interaction of the interlayer with the base metal. At the center of the interlayer is the following chemical composition (% at.): 54.1 Al; 26.0 Si; 19.9 Ni, which confirms the flow of intensive processes of interaction in the contact of the solid and liquid phases.

With thicknesses of 93 and 94 mkm interlayers, both the Al/Ti system and Al/Ni at the junction, an thickness of 60...70 mkm is formed, as shown in Fig. 7.

In the center of the layer (p. 3) has the chemical composition (% at.): 39 Ni; 53 Al; 7 Si, close to the composite (p. 4): 25 Ni; 64.5 Al; 6.2 Si with foil composition: 42.2 Ni; 57.8 Al. The concentration of nickel, which is present only in the foil, decreases with distance from the center of the layer. Silicon passes into the layer, which is possible only due to the interaction of the foil with the composite.

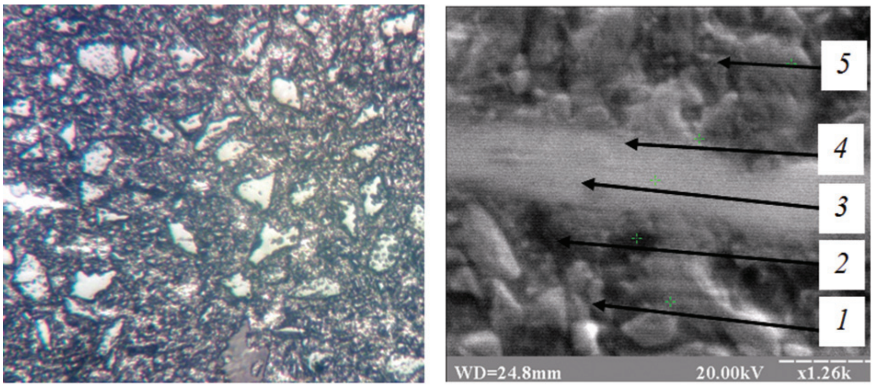
Considering the low temperatures and the short duration of the synthesis reaction, the change in the composition of the interlayer at the junction is due to the process of dissolution of the composite.

Similarly formed at the junction of the layer when using Al/Ti foil with a thickness of 94 mkm. In some areas in the joint zone there is small loosening in the composite, directly adjacent to the interlayer.



a) b)

Fig. 5. Microstructure of CM welded joint with Al/Ti interlayer (*a*) ×100 and at points of local X-ray spectral microanalysis (*b*) with an interlayer thickness of 41 mkm



a) b)

Fig. 6. The structure of the compound at the DSB of the composite through an Al/Ni interlayer with a thickness of 20 mkm (*a*) ×200 and at points of local microanalysis points (*b*)

Discussions. One of the most important mechanisms in DWV, as in other methods of welding in the solid state, is the deformation activation of the surfaces to be joined. Composite dispersively hardened materials with a high content of hardening phase have low ductility, which often leads to the appearance of cracks during compression of parts. The idea of using nanostructured interlayers in DWV composites is that nano-multilayer foils have alternating nanolayers of metals, which form intermetallic compounds with a large thermal effect. With rapid heating, reactions of self-propagating synthesis proceed and the heat released additionally heats the metal in the joint zone, ensuring the activation of the surfaces. Analysis of the microstructures and chemical composition of welded joints shows that Al/Ni and Al/Ti systems provide active interaction of the foil with the composite, as evidenced by the presence of composite chemical elements in the resulting interlayer (see Fig. 4–6).

Studies of the effect of the thickness of the interlayers on the formation of the compounds showed that the structure of the compounds is significantly different for small thicknesses (20 mkm) and large (90 mkm). At thicknesses of 20 mkm, regardless of the foil system over a long joint, the metal has a composite structure with common grains and the boundary between the connected samples is not determined either by structure or by chemical composition (see Fig. 5 and 6). In areas with a layer of its maximum thickness does not exceed 9 mkm. When the thickness of the Al/Ti foil is 41 mkm, the structure differs little from the structure at a foil thickness of 20 mkm.

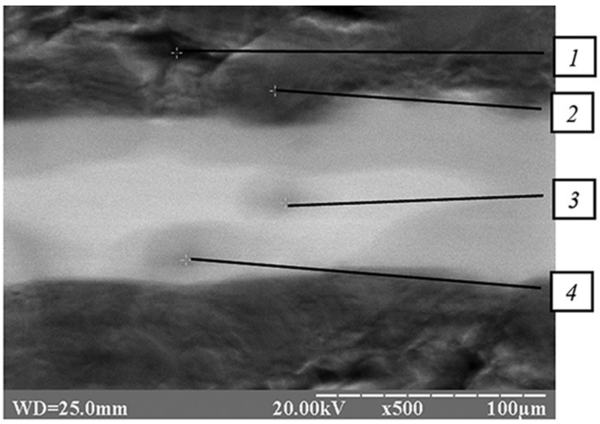


Fig. 7. The microstructure of the compound at the DSB of the composite through an Al/Ni interlayer with a thickness of 93 mkm, contains 42.2 % at. nickel

With a foil thickness of 90 mkm, an interlayer 60...70 mkm thick extends along the entire length of the joint. She reacted completely. In the center of the interlayer, its composition is close to the composition of the $NiAl_3$ phase.

According to the structure of the welded joint, the best option is a foil 20 mkm thick. The disadvantage of such a foil is its fragility and difficulty in working with it. When the foil thickness is 90 mkm and more, a thick continuous layer of brittle intermetallic is formed. Therefore, the best option is to use a foil with a thickness of about 40 mkm.

Comparing the obtained results with the literature data, it should be noted that the DWV of the brittle composite containing 55 % silicon carbide was performed for the first time using a clear method for determining the mode parameters through the allowable amount of plastic deformation determined by the established creep parameters. It is shown that to calculate the parameters of the DWV regime it is sufficient to use the creep equation at the steady state.

CONCLUSIONS. 1. It has been established that the use of nanomicrolayer foils of the Al/Ni and Al/Ti systems ensures the formation of high-quality defect-free compounds with the realization of the reaction of self-propagating high-temperature synthesis of intermetallic compounds.

2. The parameters of the DWV mode of the composite are determined taking into account the allowable plastic deformation using the established creep equation in the second stage.

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