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VACUUM DIFFUSION WELDING OF FOIL FROM POWDER NICKEL-CHROMIUM ALLOY

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The paper deals with the influence of diffusion welding parameters on formation of joints from foil of Ni–20Cr powder alloy 25 μm thick. It is shown that welding of nichrome alloy in the temperature range of 800–1200 °C without application of interlayers does not allow producing defectfree joints. Features of formation of Ni–Cr alloy joints at application of interlayers from foils, produced by the technology of electron beam deposition and condensation in vacuum were studied. Foil with multilayer structure of Ni–Al, Ti–Cu systems and foil with porous structure from Cu, Ni and Cr was used in the work. Microstructure and chemical composition of the joints were studied, using optical and electron microscopy. Strength properties of metal in the joint zone were assessed by the results of microindentation and tensile testing of flat samples. It is found that application of such interlayers in welding allows producing defectfree microstructure of the joint zone. It is shown that joints with strength properties on the level of those of base metal are formed in welding through an interlayer from copper-based porous foils. 18 Ref., 2 Tables, 9 Figures.

Keywords: *vacuum diffusion welding, Ni–Cr powder alloy, porous foils, multilayer foils, microstructure, microindenting*

Development of a reliable and cost-effective thermal protection structure of windward part of reusable space vehicles is a complex science and technology problem. Development of such promising thermal protection structures with outer metal panel has been conducted in the USA and Europe starting from the middle of the 20th century. However, thermal protection ensuring normal functioning of the space vehicle during the required number of launches is practically non-existent [1].

In Ukraine a thermal protection structure with outer honeycomb panel from high-temperature Ni–Cr-based powder alloy YuIPM-1200 is also being created [2]. At up to 1100 °C temperatures this alloy has the strength of the order of 34 MPa and 30 to 40 % relative elongation at tension that ensures its performance under the conditions of considerable deformations at elevated temperatures [3]. In fabrication of a three-layer honeycomb panel of thermal protection structure the final and most critical operation is joining its elements, namely upper and lower covers with honeycomb core [4].

Various technologies of joining three-layer panel elements are available, namely fusion welding, brazing and pressure welding.

It should be taken into account that to preserve the powder alloy characteristics, welding should be performed in the solid phase that eliminates application of fusion welding: laser and electron beam processes. Moreover, fusion welding of this group of materials is difficult, because of their hot cracking susceptibility. Producing sound joints is possible only at their preheating up to the temperature of 1100–1200 °C [5]. This kind of structures can be produced by brazing. However, high-temperature operation of brazed joints leads to oxide formation in the zone of contact of the metal being joined with braze alloy. This leads to essential intensification of oxidation and intercrystalline corrosion of base material [6].

The most promising method of joining three-layer structure elements into a panel is pressure welding, namely diffusion welding.

Complexity of producing joints of high-temperature nickel-based alloys by diffusion welding consists, primarily, in presence of a heat-resistant oxide layer on their surface and low ductility of this material group. Standard preparation of the surface of samples or items before welding consists in machining, namely grinding and chemical etching that provides removal of surface layer of metal together with oxide films. Such a surface, however, is unstable under atmospheric conditions and it is very quickly covered again by a layer of oxides [7]. Thus, an oxide layer is always present on surfaces being welded, which should be removed during heating prior to conducting the welding process. As a rule, more stringent welding conditions should be applied in welding without interlayers, in view of the presence of a heat-resistant oxide film on the surface of high-temperature nickel alloys.

Table 1. Parameters of porous and multilayer foils

Interlayer	Chemical composition, wt.%					Layer alternation period, μm	Thickness, μm	Porosity, vol.%
	Ni	Al	Ti	Co	Cu			
Ni	100	–	–	–	–	–	25	23
Co	–	–	–	100	–	–	50	25
Cu	–	–	–	–	100	–	30	30
Ni/Al	86.68	13.32	–	–	–	0.40	32	–
Cu/Ti	–	–	48.32	–	51.68	0.86	40	–

Mechanical removal of oxides from the contact zone can also have a positive impact on cleaning of the surfaces to be welded. Owing to differences in plastic characteristics of the alloy and its oxides, surface layer deformation, particularly share deformation, leads to violation of integrity of oxide layer, its cracking and breaking up into fragments. Low ductility of high-temperature nickel-based alloys has a negative impact on formation of physical contact, and, consequently, on the process of adhesion, as well as bulk interaction of the surfaces being joined.

In view of the above-said, in diffusion welding of high-temperature nickel-based alloys, it is recommended to apply enhanced welding modes: temperature $T = 1140$–1240 °C, pressure $P = 20$–60 MPa, process time $t = 30$–120 min [8, 9].

Interlayers are applied in diffusion welding for acceleration of formation of physical contact and activation of surfaces being welded. Interlayers from foil (50–500 μm) produced by the technology of casting and subsequent rolling, are the most widely applied in welding. These foils are relatively inexpensive and adaptable to fabrication, but to ensure interlayer deformation, the welding process should be conducted at increased values of welding pressure. Here, a significant chemical inhomogeneity develops in the joint zone. Application of powder [10] or perforated foils [11] as interlayers in diffusion welding, allows lowering welding temperature and pressure, as well as increasing the uniformity of element distribution in the joint zone. However, their manufacture is a quite complex and labour-consuming process.

In order to decrease chemical inhomogeneity in the butt joint, thinner foils should be applied, capable of plastic deformation during welding. Such foils include rapidly-solidified foils [12], and condensates, produced by spraying processes, in particular, electron beam evaporation and condensation in vacuum [13]. This technology allows producing foils of different chemical composition and structural state: multilayer, porous, and gradient. A feature of both multilayer, and porous foils is the fact that they promote formation of structurally nonequilibrium state, both in the foil proper, and in subsurface layers of metal being welded. Foil heating and application of tensile stresses causes an abrupt increase of their deformation rate, which acquires an exponential dependence that is characteristic for materials at their transition into superplastic state. Application of such condensates as an interlayer in diffusion welding, promotes improvement of the conditions of physical contact formation in the butt and increase of diffusion mobility of atoms [14].

The objective of the work is studying the features of formation of welded joints from Ni–Cr powder alloy, using interlayers of different chemical composition, produced by the method of electron beam evaporation and condensation in vacuum.

Materials and methods of investigation. Studies were performed using experimental alloy YuIPM-1200 (Ni–20Cr–3–4Fe–0.40–0.6Al–0.25–0.35Ti–0.5Y, wt.%), produced by powder technology. Diffusion welding of samples from Ni–Cr alloy foil was performed in a free state in vacuum, using U-394M unit.

Ni–Cr foils of 18×11×0.025 mm size were welded. After welding the samples, sections were prepared for conducting metallographic studies and for more precise determination of the influence of thermodeformational cycle of welding on structure of the produced joints. Parameters of the welding process were

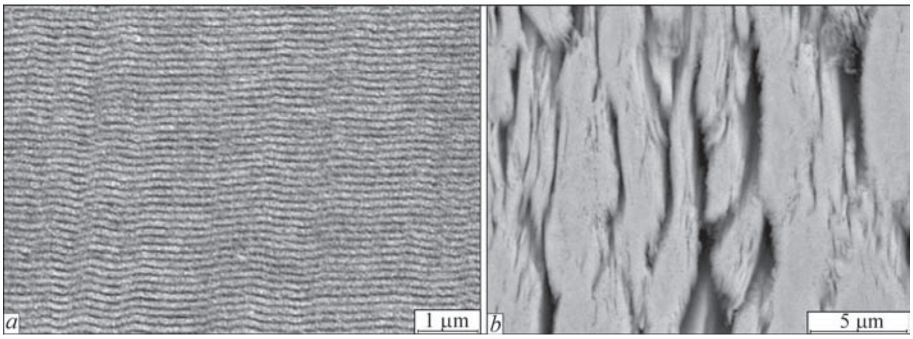


Figure 1. Microstructure of foil cross-section: *a* — multilayer Al/Ni (light layers correspond to nickel, dark layers — to aluminium); *b* — porous foil from Ni

as follows: welding temperature T = 800–1200 °C, welding duration t = 5–30 min, welding pressure P = 5–40 MPa, vacuum in the working chamber was maintained on the level of $1.33\cdot10^{-3}$ Pa.

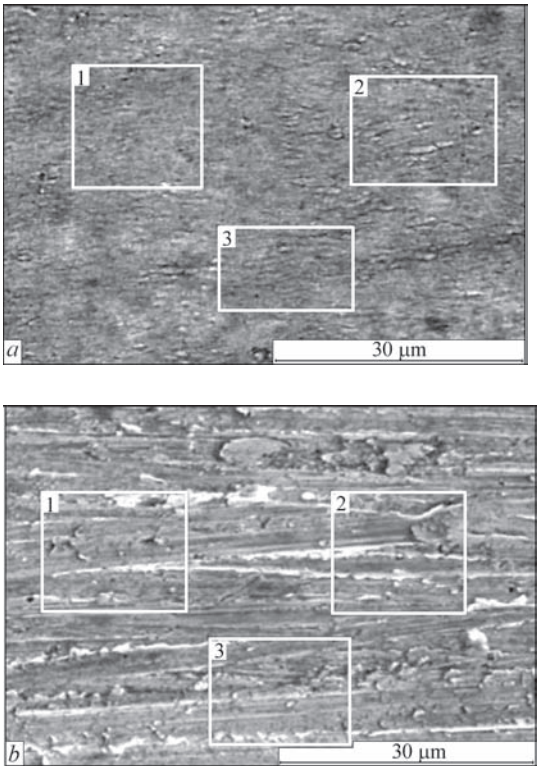
For activation of adhesion at the stage of formation of physical contact, the possibility of application of vacuum condensates (Table 1) with multilayer (Ni/Al, Ti/Cu) (Figure 1, *a*) and porous structures (Ni, Co, Cu) (Figure 1, *b*) as interlayers was studied.

Interlayers for welding were produced by electron beam vacuum deposition by a procedure, described in [15].

Analysis of structural characteristics of interlayers and welded joints was performed, using scanning electron microscope CAMSCAN 4, fitted with energy-dispersion analysis system EDX INCA 200 for determination of local chemical composition on flat samples. For this purpose, transverse sections of foils and welded joints were prepared by a standard procedure, using grinding-polishing equipment of Struers Company.

Mechanical properties of welded joints were assessed by the method of automatic indenting in the plane of welded joint cross-section with recording of the diagrams of indenter loading and unloading in Mikron-gamma unit [216], and tensile testing of flat samples in MTS-810 machine.

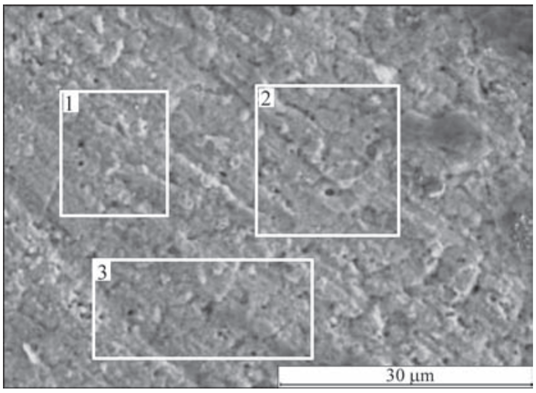
Experimental results and discussion. As shown earlier, oxide film is always present on the surface of Ni-Cr alloys [7]. Our results demonstrate that oxygen content on the alloy surface can be higher than 16 % (Figure 2, *a*). Foil surface cleaning with R1000 sand paper to metal lustre and degreasing in alcohol allow reducing the oxide film thickness, and oxygen content on foil surface to 3%, respectively (Figure 2,



Batch number	Chemical composition of as-delivered foil surface (wt.%)							
	C	O	Al	Ti	Cr	Fe	Ni	Y
1	4.86	16.75	0.66	0.55	18.22	2.27	56.69	–
2	4.60	16.38	0.45	0.18	17.51	2.51	57.66	0.71
3	4.41	16.98	0.53	0.57	18.70	2.57	56.24	–

Batch number	Chemical composition of foil surface (wt., %) after mechanical treatment and degreasing							
	C	O	Al	Ti	Cr	Fe	Ni	Y
1	9,29	3,39	–	–	15,87	3,30	68,15	–
2	7,65	3,05	–	–	16,36	3,13	69,81	–
3	6,87	3,17	0,47	0,68	15,98	3,95	68,87	–

Figure 2. Appearance of the surface of foil from Ni–Cr alloy and its chemical composition: *a* — as-delivered; *b* — after mechanical treatment by R1000 sand paper and washing in alcohol



Batch number	Chemical composition of foil surface, wt.%							
	C	O	Al	Ti	Cr	Fe	Ni	Y
1	6.64	12.35	6.70	3.42	15.17	2.46	53.26	–
2	8.62	13.25	6.87	2.18	13.81	1.73	53.54	–
3	10.60	15.13	6.76	2.63	13.71	2.38	48.80	–

Figure 3. Appearance of the surface of Ni–Cr alloy foil and its chemical composition after mechanical cleaning and heating in vacuum

b). Therefore, the sample surfaces were scraped and degreased in alcohol directly before welding.

As shown by our experiments, foil heating in vacuum of $1.33\cdot10^{-3}$ Pa at temperature $T = 1000$ °C for 30 min also leads to oxidation of precleaned surface. Figure 3 gives the foil appearance and its chemical composition. As we see, after heating in vacuum, oxygen content on foil surfaces rises from 3.05–3.39 to 12.35–15.13 wt.%. Note that our data are in agreement with the results of works [8, 17]. The authors of these works recommend applying nickel coatings in welding Ni–Cr alloys that provides protection of the alloy surface during heating.

Investigations of annealing temperature influence on metal structure were conducted to select the parameters of diffusion welding of Ni–Cr alloy.

Sample heating was conducted at temperature $T = 1050$ °C, pressure $P = 5$ MPa during $t = 5$ min in vacuum. At analysis of foil microstructure it was found that reduction of pore content proceeds in it under the impact of temperature and pressure (Figure 4).

It is established that porosity in the foil in the initial condition is equal to 7.7 %, and after annealing it decreases to 5.2 %. Microindentation method revealed that in initial material samples average microhardness value is equal to 3.754 GPa, Young's modulus value is 139.8 GPa, deviation of modulus of elasticity is equal to 4.6 % that is indicative of material homogeneity, as well as uniformity of pore distribution through overall cross-section of foil sample. Foil annealing under vacuum leads to lowering of microhardness values by 1.6 times, compared to material in the initial condition (up to 2.293 GPa) and increase of Young's modulus to 148.7 GPa.

Increase of the modulus of elasticity can be an indication of lowering of material total porosity [18]. However, increase of the range of deviation of its values to 7.1 % can be an indication of a less uniform nature of pore distribution through overall cross-section of the foil, compared to the sample in the initial condition.

A series of experiments to produce joints at temperatures of 800, 900, 1000, 1100, 1200 °C were performed, in order to determine the optimum parameters of welding Ni–Cr alloy. Welding was conducted without application of interlayers. Welding pressure in all the cases was equal to 40 MPa, process time was 20 min.

Microstructures of joints from nichrome alloy, produced at the temperature of 800, 1000 and 1200 °C, are given in Figure 5.

As is seen from Figure 5, welded joint zone defectiveness decreases with increase of welding temperature. However, even at welding temperature of 1200 °C, a string of oxides located along the butt, is preserved in the joint zone.

In welding of Ni–Cr alloy without application of interlayers ($T = 1200$ °C, $P = 40$ MPa, $t = 20$ min), microhardness values in the joint zone, are close to those characteristic for annealed material $H = 2.823$ GPa. Results of metallographic studies, as well as a broad range of variation of Young's modulus $E = 110.7$–154.3 GPa lead to the conclusion that influence of high values of welding temperature and pressure results, on the one hand, in pore coagulation in the foil, and on the other hand — in nonuniform redistribution of porosity in the joint zone (Figure 6).

Influence of multilayers on formation of welded joint structure was studied. Analysis of microstructure of the joints produced with application of Al/Ni system interlayers, shows that there are no defects in the butt joint (Figure 7, *a*). Width of the joint zone (JZ) is equal to 20 μm. Depth of aluminium diffusion from interlayer into Ni–Cr alloy is equal to 5–7 μm, proceeding from element distribution (Figure 7, *b*). Chemical element content in the butt joint is equal to: 85.53 Ni; 3.75 Cr; 10.16 Al; 0.56 Fe, wt.%.

Joint zone of samples produced using Al/Ni multilayer, is characterized by higher average values of both microhardness $H = 4.340$ GPa, and Young's modulus $E = 161.3$ GPa.

In the case of application of foil of Cu–Ti system, formation of several diffusion zones with different

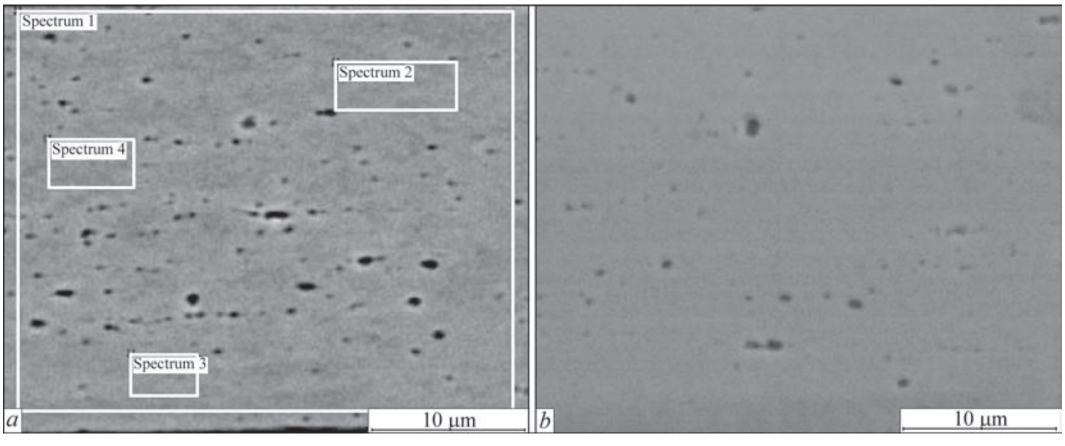


Figure 4. Microstructure of foil from Ni–Cr alloy in the initial condition (*a*) and after heating at temperature $T = 1050$ °C at the pressure of $P = 5$ MPa in vacuum (*b*)

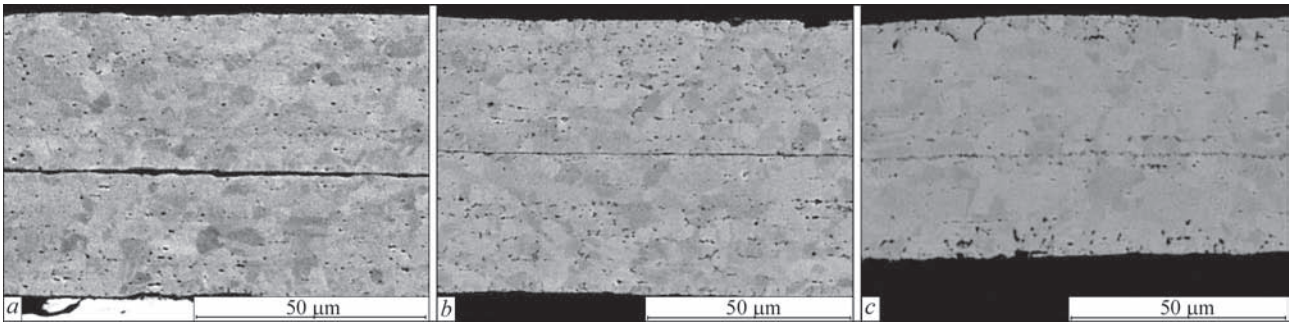
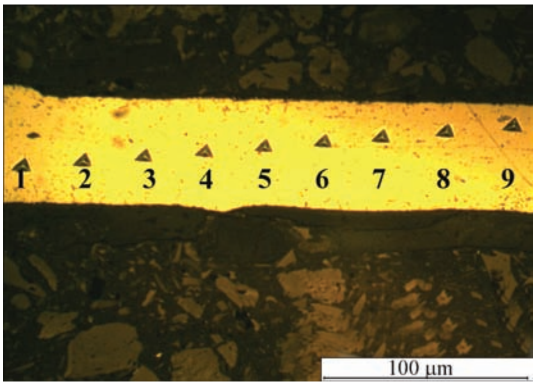


Figure 5. Microstructure of Ni–Cr alloy joints, produced at welding temperature of: *a* — 800; *b* — 1000; *c* — 1200 °C



Batch number	H, GPa	E, GPa
1	2.547	110.7
2	3.029	127.1
3	2.750	112.6
4	2.689	121.4
5	2.804	125.8
6	2.403	131.1
7	2.519	140.4
8	2.707	143.5
9	2.487	154.3
Average value	2.659	129.7

Figure 6. Results of automatic indentation of the joint zone of Ni–Cr foil sample ($T = 1200$ °C, $P = 40$ MPa, $t = 20$ min); imprints obtained by indentation; Table of calculations (indentation was conducted at the same values $P = 20$ g and $V = 2$ g/s for all the points)

chemical composition of elements is observed in the butt joint (Figure 7, *c*, *d*). Total width of JZ is equal to 25–30 µm. As follows from graphs of element distribution, during welding nickel diffusion proceeds through the entire interlayer thickness. Depth of titanium diffusion from interlayer into Ni–Cr alloy, is equal to 12–15 µm, proceeding from element distribution (Figure 7, *d*). Nickel concentration in the interlayer is equal to about 40 %. Chromium diffusion from the foil into the interlayer is insignificant, its content in the interlayer being 1.81–2.41 %. Average value of microhardness for samples produced with Cu/Ti multilayer, is equal to $H = 4.340$ GPa, and Young's modulus is $E = 161.3$ GPa.

Applicability of porous interlayers from nickel, cobalt and copper in nichrome welding was also studied (Figure 8). Welding was performed at temperature $T = 1200$ °C, pressure $P = 40$ MPa, soaking time $t = 20$ min.

It is found that application of cobalt-based interlayers leads to development of considerable porosity in the joint zone (Figure 8, *a*), and nonuniformity of element distribution (Figure 8, *b*). Content of chemical elements in the butt joint is equal to: 15.80 Ni; 6.47 Cr; 1.09 Fe; 76.64 Co, wt.%. Total width of JZ is equal to 35–38 µm. Depth of cobalt diffusion from interlayer into Ni–Cr alloy is equal to 7–10 µm, proceeding from element distribution (Figure 8, *b*). Average value of microhardness for samples, produced with porous interlayer from cobalt, is equal to $H = 3.244$ GPa, and Young's modulus is $E = 157.3$ GPa.

At application of copper-based porous interlayers the line of contact of interlayer–Ni–Cr alloy disappears as a structural element during welding (Figure 8, *c*).

It should be noted that, as copper melting temperature is equal to 1083 °C, in this case welding was conducted in transient liquid phase diffusion bonding (TLDB) mode. Presence of liquid phase in the butt joint ensured activation of surfaces of blanks being welded and copper diffusion through the entire foil thickness.

Application of copper interlayers leads to a more uniform nature of element distribution in the butt joint (Figure 8, *d*) and minimal number of defects. Chemical element content in the butt joint is as follows: 68.46 Ni; 17.26 Cr; 0.99 Fe; 14.27 Cu, wt.%. Average value of microhardness for samples produced with porous copper interlayer, is equal to $H = 2.258$ GPa, and Young's modulus is $E = 137.1$ GPa.

In welded joints produced with application of porous nickel interlayer, formation of coarse-grained structure is observed in the joint zone. Width of the joint zone is equal to 20–25 µm (Figure 8, *e*). There are no defects in the joint zone.

However, a string of pores, located along the butt joint, is observed on the boundary of interlayer–Ni–Cr alloy. Chemical element content in the butt joint is equal to: 93.67 Ni; 4.96 Cr; 0.46 Al; 0.91 Fe, wt.%, that may be indicative of the fact that a nickel-based low alloy was formed in the butt joint.

Average value of microhardness of the joint zone of samples, produced using a porous nickel interlayer,

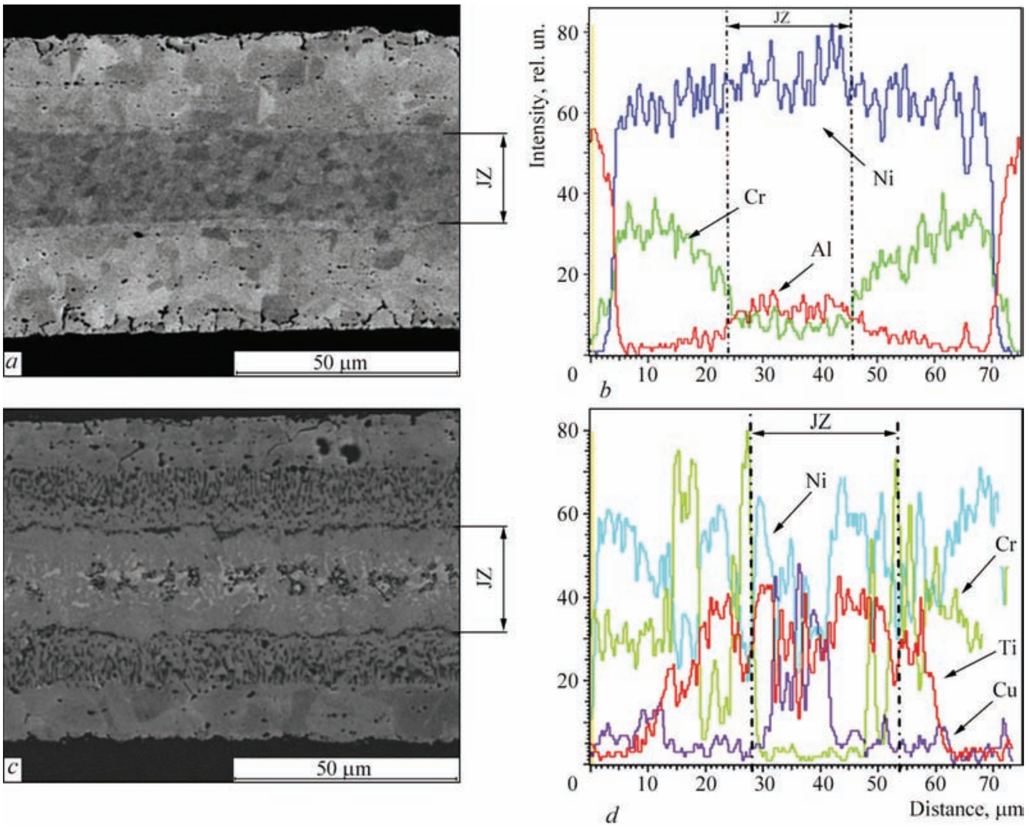


Figure 7. Microstructure and distribution of elements in joints produced with application of multilayers of Al/Ni (*a*, *b*) and Cu/Ti (*c*, *d*) systems

is equal to H = 2.119 GPa, and Young's modulus is E = 158.5 GPa.

Results of micromechanical studies of the initial material and welded joints are shown in Figure 9.

As is seen from the given data, foil microhardness (average value) after annealing decreases from 3.754 to 2.293 GPa. In welded joints produced using Al/Ni and Cu/Ti multilayer foils average values of microhardness in the joint zone are equal to 4.340 and 4.637 GPa, respectively, that, in our opinion, can be indicative of intermetallic phases formation in the butt joint. In joints, produced with application of porous foils from Cu and Ni, average values of microhardness in the butt joint (2.258 and 2.119 GPa, respectively), are close to microhardness values of Ni–Cr foil after annealing. In joints, produced with application of foil from cobalt, microhardness in the joint zone is equal to 3.224 GPa, that is higher than average microhardness values for as-annealed base material.

Mechanical properties of welded joints produced in vacuum diffusion welding of Ni–Cr alloy samples were studied. Sample length was 18 mm and width was 11 mm. Overlap was equal to 5 mm in sample welding. Results of tensile mechanical testing of welded joints are given in Table 2.

As is seen from Table 2, average strength of base metal of Ni–Cr alloy is equal to 405 MPa. Foil annealing leads to lowering of its strength level to 305 MPa. Application of multilayer foils of Cu/Ti and Al/Ni systems ensures average strength properties of the joints on the level of 161 and 100 MPa, respectively that, as was shown above, may be associated with considerable chemical inhomogeneity in the joint zone and increase of microhardness of individual structural elements in the butt joints.

Average strength of samples, made with application of porous cobalt interlayer, is equal to $\sigma_t =$ = 223 MPa. Samples, produced with application of cobalt interlayer, are characterized by presence of defects in the joint zone, both in the form of pores, and as considerable chemical inhomogeneity of elements, namely Cr, Ni, Al. In our opinion, development of porosity in the butt can be associated with manifestation of Kirkendale effect.

Average strength of samples, produced with application of a porous nickel interlayer, is equal to $\sigma_t =$ = 108 MPa. Proceeding from the results of metallographic studies, it can be assumed that the obtained results are associated both with formation of a zone in the butt joint which consists of low-alloyed nickel, as with the presence of pore stringers from two sides of the interlayer, that, probably, is what leads to lowering of welded joint strength.

Application of porous copper interlayer in welding Ni–Cr alloy allows producing joints with average value of strength σ_t = 317 MPa. Microstructural analysis of welded joints shows that in the case of application of porous copper interlayer and welding mode, which

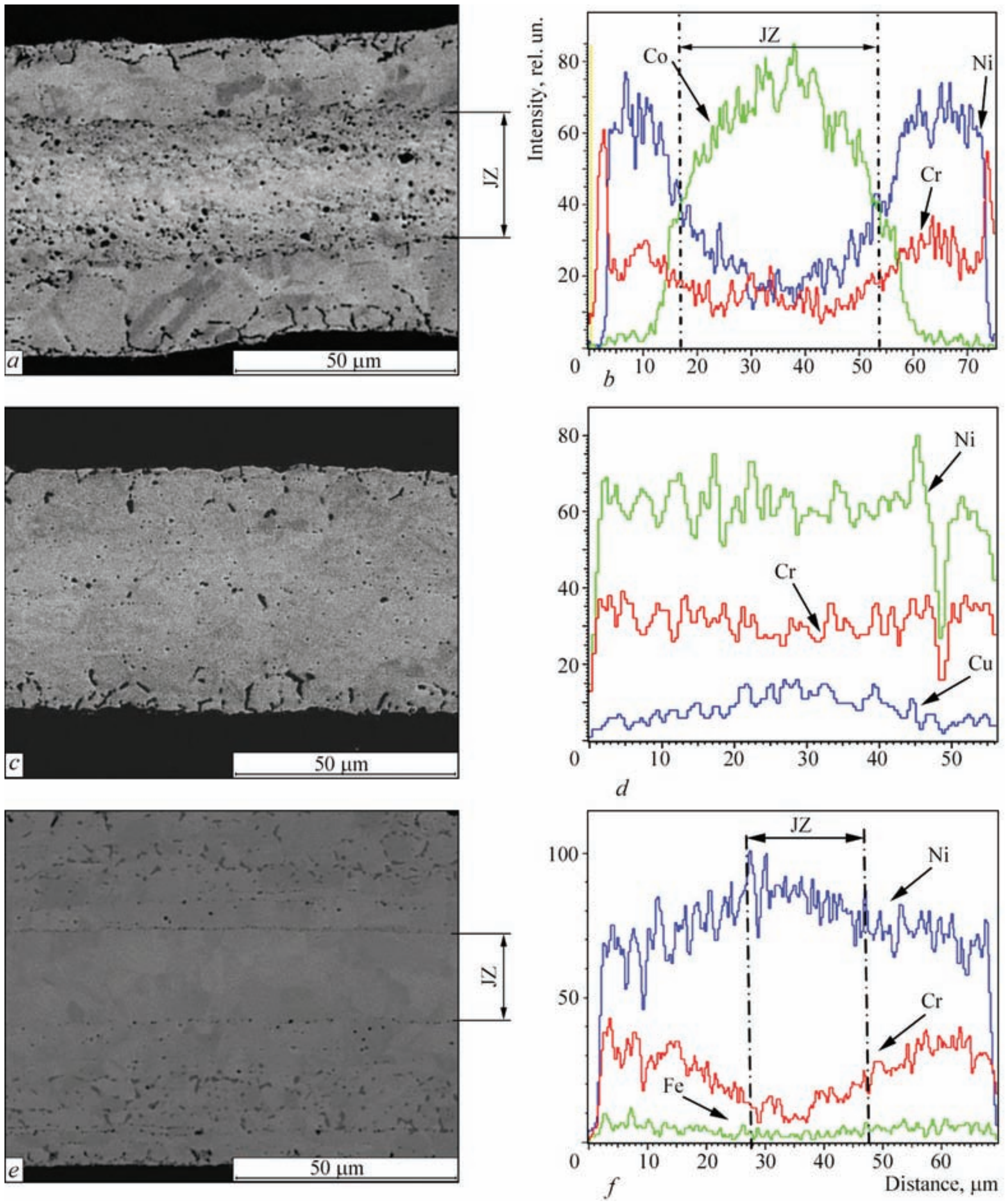


Figure 8. Microstructure and element distribution in joints produced with application of porous interlayers of Co (*a*, *b*); Cu (*c*, *d*) and Ni (*e*, *f*)

ensures running of intensive diffusion processes in the butt, the interlayer disappears as a structural element. Joint strength is on the level of that of base metal, subjected to heat treatment.

Conclusions

1. Vacuum diffusion welding of Ni–Cr powder alloy YuIPM-1200 (Ni–20Cr–3–4Fe–0.40–0.6Al–0.25–0.35Ti–0.5Y, wt.%) without application of interlayers results in formation of defects in the form of pores in the joint zone in all the studied temperature ranges of welding.

2. It is shown that annealing of Ni–Cr alloy foil in vacuum $B = 1.33 \cdot 10^{-3}$ Pa at temperature, corresponding to that of diffusion welding, is accompanied by oxidation of sample surface.

3. Application of Al/Ni and Ti/Cu multilayers in diffusion welding of Ni–Cr alloy, produced by the technology of electron beam evaporation and condensation in vacuum, promotes formation of defectfree joints. Diffusion zones with higher level of microhardness form in the joint zone.

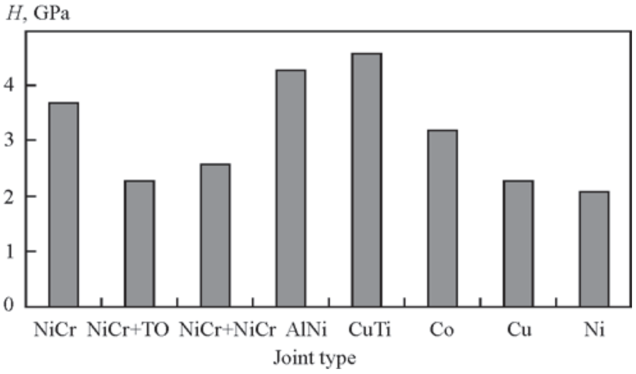


Figure 9. Microhardness values, produced for base metal and welded joints by the results of automatic indentation

Table 2. Results of mechanical tensile testing of welded joints

Sample number	Sample type	Type and thickness of interlayer, mm	Sample fracture location		σ_t, MPa	$\sigma_{t\ av}$, MPa
			Base metal	Joint zone		
1	BM	–	+	–	440	405
2			+	–	–	
3			+	–	370	
1	As-annealed BM	–	+	–	200	305
2			+	–	215	
3			+	–	500	
1	Welded joint	Cu/Ti, δ = 0.04	–	–	75	161
2			+	–	250	
3			+	–	160	
1	Same	Al/Ni	–	+	–	100
2			–	+	100	
3			–	+	–	
1	»	Ni, δ = 0.025	+	–	120	108
2			+	–	160	
3			–	+	45	
1	»	Cu, δ = 0.03	+	–	200	317
2			+	–	310	
3			+	–	360	
4			+	–	400	
1	»	Co, δ = 0.05	+	–	175	233
2			+	–	260	
3			–	+	–	
4			+	–	265	

4. Application of interlayers based on porous foil from nickel, copper and cobalt, ensures establishing of physical contact of surfaces being welded, promotes running of diffusion processes and welded joint formation. Copper-based interlayers in diffusion welding of Ni–Cr alloy, enable producing joints with the strength on the level of that of base metal after heat treatment.

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