VACUUM DIFFUSION WELDING OF STAINLESS STEEL THROUGH POROUS NICKEL INTERLAYERS

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Features of formation of permanent joints by diffusion welding through an intermediate interlayer based on porous nickel, produced by vapour phase vacuum deposition (EB PVD) were studied in the case of stainless steel Kh18N10T. It is shown that application of vacuum porous nickel condensates promotes lowering of temperature-force parameters of diffusion welding (welding temperature of 800 °C + cyclic load with 20 MPa amplitude). Such an influence of the interlayer on the conditions of producing the welded joints is associated with superplastic behaviour of porous nickel and its non-equilibrium structure (small grain size and presence of vacancy type defects). It is established that pore healing in the interlayer is observed and diffusion processes between the interlayer and stainless steel are activated during diffusion welding. This results in formation of a defectfree zone of the joint based on nickel, alloyed with iron and chromium with strength properties only slightly different from those of base metal. 18 Ref., 6 Figures.

Keywords: *diffusion welding, stainless steel, intermediate layer, porous nickel interlayer, temperature-force parameter, foil deformation behaviour*

Application of ductile («soft») interlayers, having a low yield point, compared to the material being welded, promotes lowering of temperature and pressure, required to produce permanent joints by the method of vacuum diffusion welding [1]. Interlayer materials used usually are such metals as gold, silver, nickel, copper and aluminium in the form of foils, powders, coatings applied on the surfaces being joined by electroplating or vacuum deposition.

Application of «soft» interlayers in hard material welding is attributable to the fact that the conditions for realization of diffusion welding at relatively low temperature-force parameters of the process are favourable for establishing physical contact of the surfaces being welded and activation of diffusion processes in them. Such an influence of interlayers is related to the fact that their higher ductility, compared to the material being welded, leads to plastic deformation at lower loads, that results in filling of microroughnesses of the surfaces being joined, promoting their plastic deformation due to contact interaction with the interlayer.

It is shown in [2] that stress distribution in the interlayer is non-uniform. In welded joint central part stresses are determined chiefly by the normal component, and at greater distance from the joint center the tangential component becomes larger. Plastic flow of the interlayer under the impact of tangential component of stresses creates the necessary conditions to produce permanent joints at minimum temperature-force parameters of the process, predominantly over the contact surface periphery. Equalizing of the conditions for producing joints over the entire surface being welded is achieved at lowering of interlayer material yield point. It is believed that formation of full physical contact over the entire surface being welded, requires plastic deformation of the interlayer by not less than 5–10 % of its initial thickness.

As shown in [3, 4] plastic flow of interlayer material can be intensified at certain temperature-force parameters, for instance, by application of «perforated» foil, metal net, etc. as the interlayer.

Producing welded joints through an interlayer during diffusion welding can be promoted also by grain refinement, as with increase of grain boundary density the diffusion processes are greatly accelerated, due to diffusion coefficient being higher by several orders of magnitude along grain boundaries than through the grain bulk [5].

Effectiveness of application of porous highly-dispersed materials as interlayers is shown in [6] in the case of ultradispersed powder interlayers, produced by reduction of nickel, copper and cobalt formates.

Note that application of interlayers is usually accompanied by a change of composition in the joint zone, that can have a negative effect on corrosion and physical properties of the item. To reduce such an influence, researchers try to minimize the interlayer thickness. On the other hand, it turned out that such possibilities are limited. This is due to the fact that interlayer yield point rises dramatically with reduction of its thickness.

To lower the yield point of thin foils (10–20 μm), they should be heated up to the temperature, at which interlayer material becomes superductile, and this leads to welding temperature increase. Considering that structural superplasticity is associated with the action of plastic deformation mechanism in the material through grain slipping, factors, promoting the realization of this deformation mechanism, will lead to lowering of the temperature of material transition into superplastic state. Such factors include, primarily, grain refinement and presence of pores.

It is known that thin foils (10–20 μm) based on ductile metals can be produced by vacuum deposition from the vapour phase [7], and changing their deposition conditions allows varying grain size and porous structure characteristics [8]. It can be assumed that such a foil structure will promote lowering of the temperature of its transition into superplastic state and decrease of plastic flow stress. Application of such foils as interlayers can ensure lowering temperature-force parameters of diffusion welding. To study such a possibility, conditions for producing permanent joints by diffusion welding through interlayers based on porous nickel foil, made by EB PVD process, were investigated in this work for the case of stainless steel of Kh18N10T grade.

Conditions to produce permanent joints by diffusion welding without application of an interlayer were the subject of several studies [9–11]. In [9] it was established that optimal characteristics of welded joints from E10 alloy and 12Kh18N10T steel are achieved at welding temperature of 1100 °C, specific pressure of 15 MPa and soaking time of 10–15 min. In [10] 1Kh18N9T steel welding was performed at T = = 1100 °C, P = 25 MPa, and 10 min duration.

By the data of [11], to preserve austenitic structure it is recommended to perform welding of steels of 18-8 type (18 % Cr and 8 % Ni) at temperatures below austenite transformation point and to minimize welding time. Proceeding from that, the following welding mode was proposed: T = 1000 °C, P = 20 MPa, t = 10 min. During welding, metal structure changes due to intragranular and intergranular creep, as well as processes of diffusion nature. Pressure increase essentially increases the creep rate, enhances intergranular creep and grain growth in the joint, that intensifies recrystallization processes. At diffusion welding of austenitic steels, producing joints with optimum properties requires ensuring favourable conditions for recrystallization development, but it should be borne in mind here that excessive grain growth and impurity redistribution lead to lowering of joint impact toughness.

Materials and methods of investigation. Diffusion welding of samples from 12Kh18N10T steel was performed in the free state in vacuum with application of U-394M system, the block-diagram of which is described in [12]. Welding process parameters were as follows: welding temperature was varied in the range of T = 800–1100 °C, welding duration was t = 5–20 min, welding pressure P = 10–20 MPa, vacuum in the working chamber was maintained on the level of $1.33 \cdot 10^{-3}$ Pa. To intensify establishing of physical contact of the surfaces being joined, in keeping with [13], cyclic application of compressive pressure was applied in a number of cases. Load was applied to the parts by the following scheme: P = 20 MPa, t = = 3 min, unloading to P = 5 MPa with duration t = 1 min. Total cycle number was 5.

Thin foils of nickel were produced by EB PVD by the procedure described in detail in [14].

Analysis of structural characteristics of foil and welded joints was conducted with application of scanning electron microscope CAMSCAN 4, fitted with a system for energy-dispersive analysis EDX INCA 200 for local chemical composition of flat samples. For this purpose, transverse sections of foil and welded joints were prepared by a standard procedure with application of grinding-polishing equipment of «Struers» Company. Foil porosity was assessed with application of a computer method of microstructure image analysis.

Deformational behaviour of thin foils during heating under the conditions of continuous tensile loads, the magnitude of which was commensurate with tangential stresses developing in the interlayer during diffusion welding (of the order of 1/10 of applied pressure), was assessed with application of a dylatometric unit fitted with a special device [15].

Mechanical properties of welded joints were assessed by the method of automatic indenting of welded joint cross-sectional plane with recording of the diagram of indenter loading and unloading in Micron-gamma unit [15].

Results and their discussion. Figure 1 shows the characteristic cross-sectional microstructure of porous nickel foil, produced by EB PVD. It is seen that foil microstructure after deposition (Figure 1, *a*) is characterized by a columnar grain

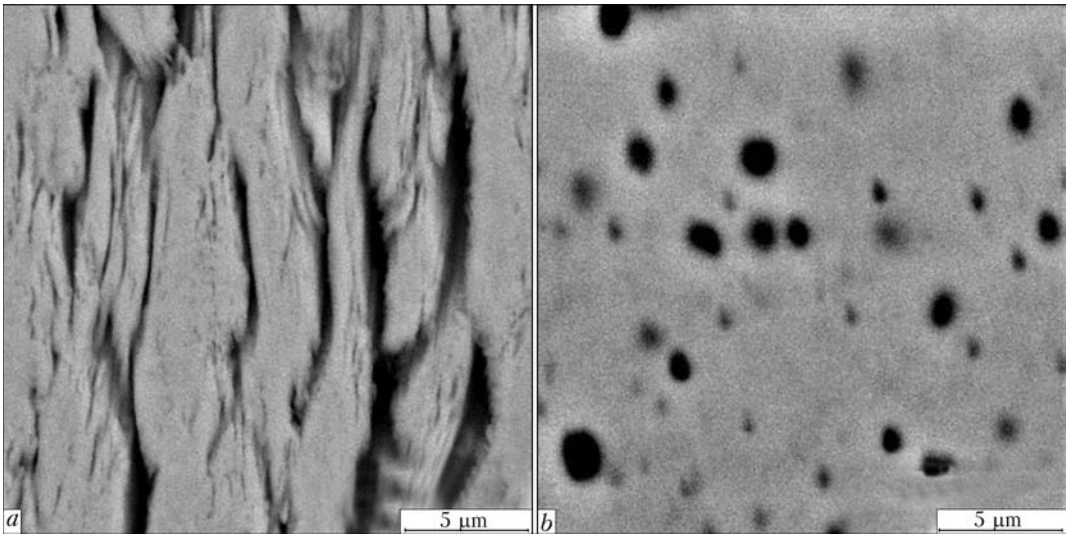


Figure 1. Cross-sectional microstructure of porous nickel foil with 25 vol.% of pores: *a* — after deposition; *b* — after annealing at 600 °C for 20 min

structure, on the boundary of which elongated open porosity is observed, which runs through the entire thickness of the foil. Low deposition temperature ($<0.3T_{\text{melt}}$) leads to formation of foil in structurally-nonequilibrium state, characterized by elongated grains with cross-section of the order of 2–3 μm, and increased concentration of vacancies.

Yield point of porous foils was evaluated by the data on their microhardness as $1/3H_\mu$ [17]. It turned out that with porosity increase foil yield point at room temperature decreases from 600 MPa for defectfree nickel foil, produced by EB PVD, to 300 MPa for nickel foil with porosity of 25 vol.%.

Investigations of deformational behaviour of porous nickel foil at heating under the conditions of continuous tensile stresses (of the order of 2–4 MPa) showed that foil plastic flow velocity non-monotonically depends on temperature. As is seen from Figure 2, *a*, foil deformation rate remains on the level of nickel thermal expansion factor (tensile stresses below foil yield point) up to 400–450 °C. At increase of foil temperature, its deformation rate starts growing continuously that is associated with its plastic deformation. In the region of 550–600 °C, dependence of deformation rate on foil temperature becomes exponential that is indicative of its transition into the superplastic state.

Note that during superplastic flow foil structure undergoes qualitative changes — open type porous structure changes to closed type porous structure (Figure 2, *b*). This is accompanied by reduction of foil porosity from 25 to 18 vol.%. Here, pores size is also significantly reduced.

Microstructures of porous foil after its annealing at 600 °C in the free state (without load application) were studied to identify the factors, which have an essential influence on pore evolution during its superplastic flow. It is seen (Figure 1, *b*) that during annealing structural trans-

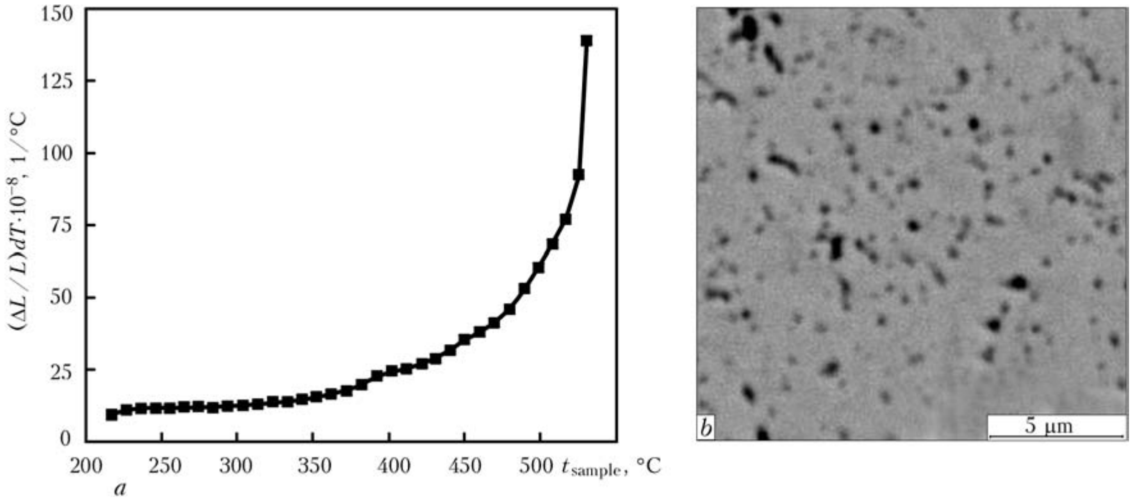


Figure 2. Temperature dependence of nickel foil deformation rate (porosity of 23 vol.%) at heating under the conditions of continuous tensile stresses of 4 MPa (*a*), and foil microstructure after its deformation (*b*)

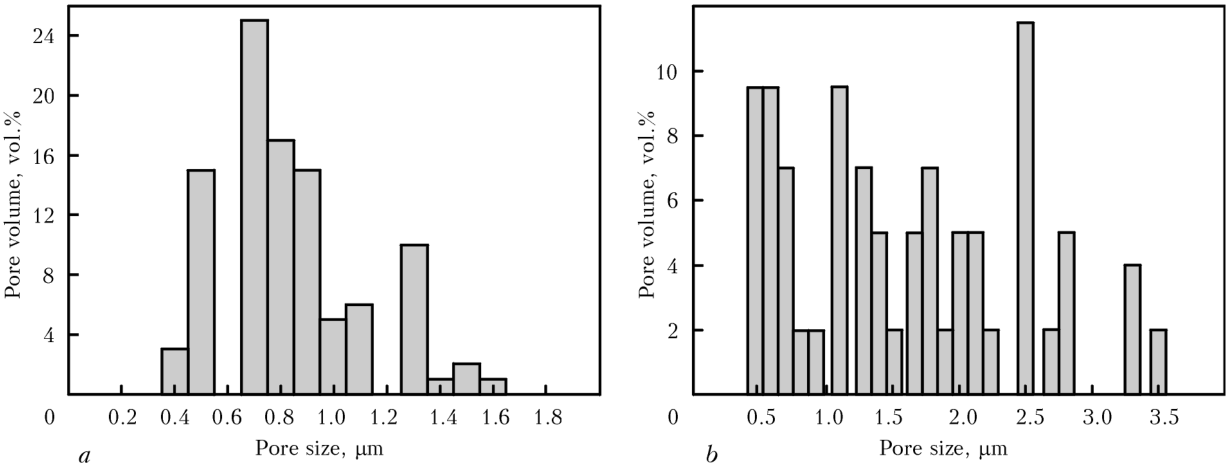


Figure 3. Size distribution of pores in nickel foil after heating up to 600 °C under the conditions of continuously applied tensile stresses (*a*) and after annealing at 600 °C for 20 min (*b*)

formations run in the foil, which are accompanied by changing of its porous structure: pores become closed and acquire a spherical shape, porosity decreases to 18 vol.%.

However, unlike foil porous structure after its plastic deformation by superplastic flow mechanism, which is characterized by narrow distribution of pore sizes (Figure 3, *a*), porous structure of annealed foil has a broader pore distribution by size (from fractions of a micron to several microns) (Figure 3, *b*). It can be assumed that formation of foil porous structure during its annealing is the consequence of running of various processes in it. Some of them are associated with reduction of free surface of open porosity. This causes its evolution, leading to open pore healing that results in formation of relatively coarse closed porosity. Other processes can be associated with relaxation of excess vacancies, inherent to vacuum condensates produced under temperature conditions, when diffusion mobility of atoms is limited (at $T_d < 1/3T_m$, where T_d is the deposition temperature, and T_m is the nickel melting temperature), which are accompanied by their combining into complexes with subsequent transformation into pores. These processes can result in formation of closed pores of relatively small (submicron) size.

Absence of coarse pores in the foil after superplastic flow leads to the assumption that plastic deformation promotes healing of coarse pores, whereas finer pores are preserved. According to [18], pore presence in the material is a prerequisite for realization of its superplastic flow by the mechanism of grain slipping.

Thus, plastic flow of porous nickel foil at relatively low heating temperatures can be related to presence of open porosity and structural changes in foil material, which are due to relaxation of excess vacancies, transformation of open pore structure into closed pore structure, and processes of pore coalescence and healing, that should promote not only lowering of temperature of foil transition into superplastic state, but also intensification of diffusion processes in it.

Figure 4, *a* shows the microstructure of welded joint of stainless steel samples, produced by the method of diffusion pressure welding at 1100 °C without application of the interlayer.

It is seen that welded joints, made without the interlayer, are characterized by presence in the joint zone of defects in the form of microdiscontinuities and pores, that results from poor physical contact of the surfaces of samples being welded.

Diffusion welding of stainless steel samples through an interlayer from porous nickel allows lowering welding temperature to 900 °C, and ensuring formation of a defect-free joint zone. Figure 4, *b* shows the microstructure of the zone of a joint produced with application of porous nickel foil 25 μm thick. It is seen that there are no defects or discontinuities in the butt joint area. The interface of foil and steel is blurred, that is indicative of diffusion processes running in this zone, and foil surface envelops the steel surface roughness contours. This is an indication of foil plastic deformation under the impact of applied load, and presence of a diffusion zone on the interface of these surfaces points to establishing of physical contact between them and diffusion interaction of the contacting surfaces. Intensive running of diffusion processes in joint zone is also indicated by nickel and iron distribution in the butt joint area (Figure 5, *a*): elements, included into stainless steel composition (2.5 % Fe, 1.1 % Cr) are present in nickel interlayer.

Note also the fact that there are no pores in the joint zone. As the processes of atom diffusion from steel into the interlayer and pore healing can be facilitated by foil non-equilibrium struc-

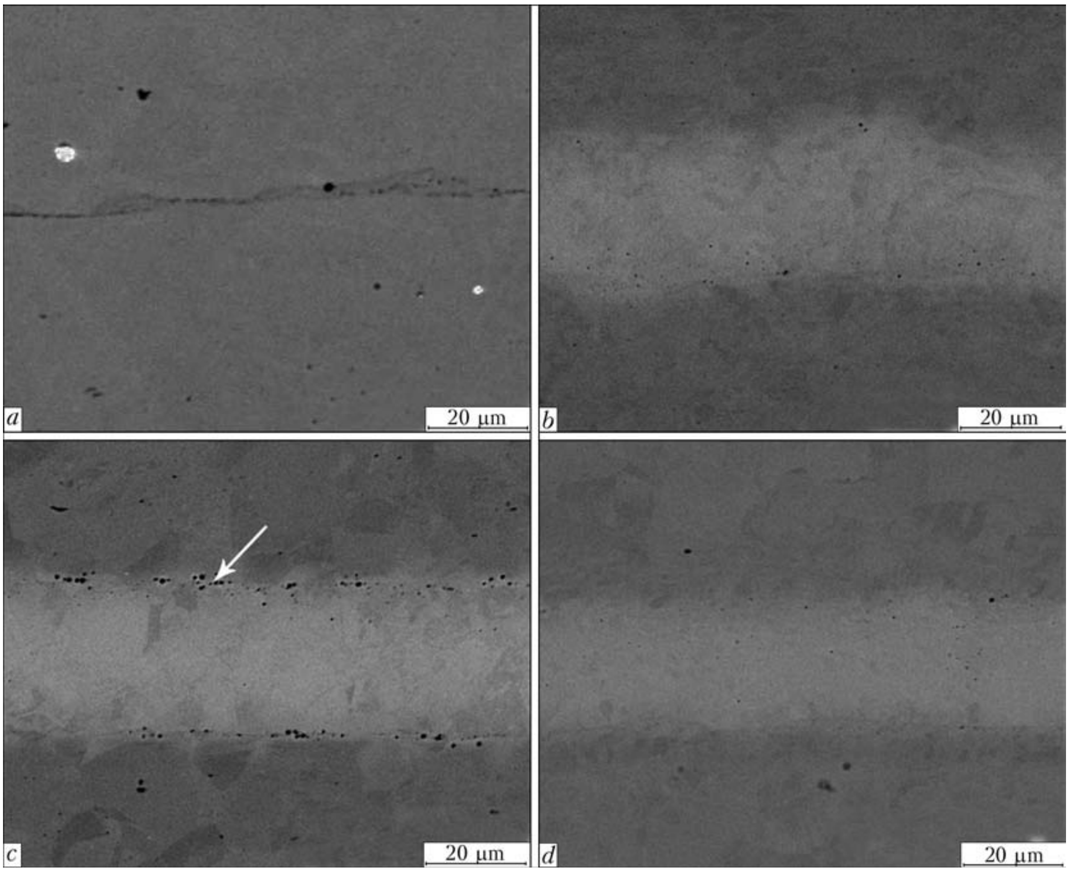


Figure 4. Microstructure of welded joint of stainless steel samples produced by diffusion welding at T = 1100 °C, P = = 20 MPa without interlayer (a), at T = 900 °C, P = 20 MPa with application of porous nickel interlayer (b), with application of porous nickel interlayer pre-annealed at 600 °C for 20 min (c), in welding at T = 800 °C, P = 20 MPa + cyclic loading with application of porous nickel interlayer (d)

ture due to excess vacancies, diffusion welding of steel samples was performed under similar conditions (T = 900 °C, P = 20 MPa) with application of porous nickel foil as interlayer, which was pre-annealed at 600 °C for 20 min. It is seen that (Figure 4, c) in this case pores and discontinuities (arrow) form on foil/steel interface.

Such a welded joint structure can be the result of both the change of foil porous structure and lower concentration of excess vacancies in it.

Both the factors can have an influence both on foil superplastic flow under the impact of applied pressure, and on diffusion processes in the joint zone, that, in its turn, influences establishing of physical contact between the surfaces being welded (foil and steel) and their diffusion interaction. Therefore, formation of porous foil with a more equilibrium pore shape and partial relaxation of excess vacancies (by their coales-

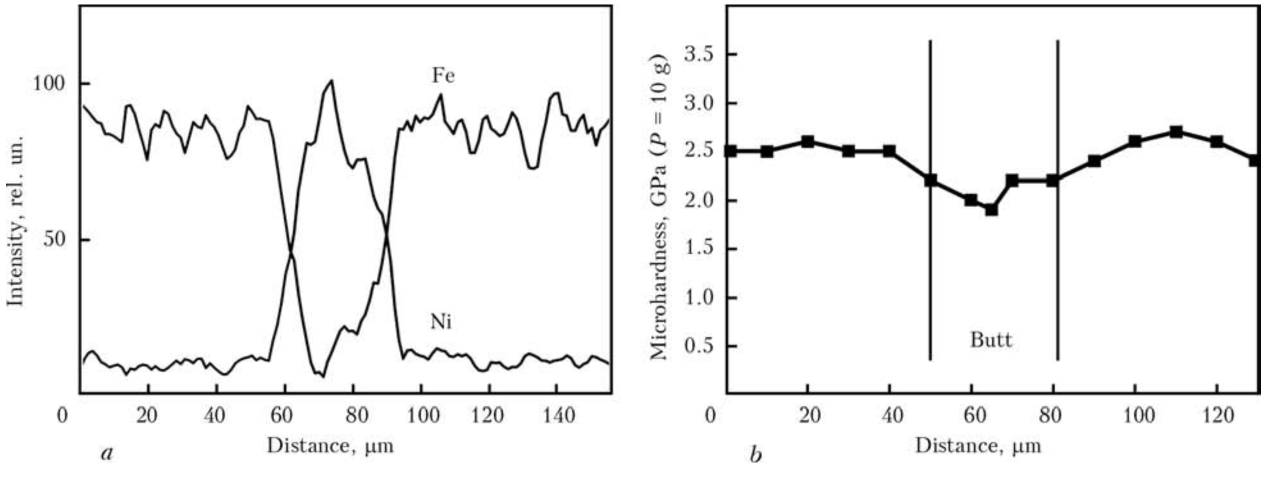
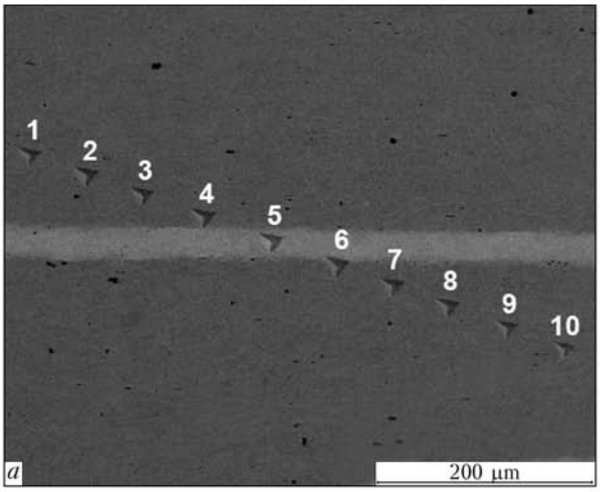


Figure 5. Nickel and iron distribution in the zone of stainless steel joint made by diffusion welding through an interlayer of porous nickel at T = 900 °C, P = 20 MPa (a), and respective microhardness distribution (b)



Joint zone	P_{load}, g	v_{load}, g/s	*HM* GPa	*E*, GPa	K_{plast}
1	40	4.0	2.418	211.6	0.947
2	40	4.0	2.055	165.9	0.947
3	40	4.0	1.853	163.6	0.947
4	40	4.0	1.895	192.2	0.954
5	40	4.0	2.135	205.3	0.952
6	40	4.0	1.878	183.3	0.962
7	40	4.0	2.197	176.0	0.942
8	40	4.0	1.842	183.3	0.953
9	40	4.0	2.121	190.8	0.949
10	40	4.0	2.140	188.9	0.947

b

Joint zone	P_{load}, g	v_{load}, g/s	*HM* GPa	*E*, GPa	K_{plast}
1	40	4.0	2.315	185.0	0.943
2	40	4.0	2.158	177.7	0.944
3	40	4.0	2.348	192.8	0.944
4	40	4.0	2.617	203.2	0.941
5	40	4.0	2.148	189.1	0.948
6	40	4.0	2.353	190.6	0.943
7	40	4.0	2.333	188.3	0.943
8	40	4.0	1.976	172.0	0.947
9	40	4.0	2.014	162.2	0.943
10	40	4.0	2.341	187.7	0.943

c

Figure 6. Results of automatic indenting of the zone of stainless steel sample joint produced with application of intermediate foil from porous nickel: *a* — imprints from indentation; *b* — welding at $T = 900$ °C, $P = 20$ MPa; *c* — welding at $T = 800$ °C, $P = 20$ MPa + cyclic loading; P_{load} — loading; v — loading rate; *HM* — Meyer microhardness values; *E* — Young's modulus; K_{plast} — plasticity coefficient; highlighted line corresponds to butt joint area in Figure 6, *a*

cence into nanopores) during pre-annealing impairs the weld structure.

Joint mechanical properties were assessed by the method of automatic indentation of the cross-section with application of Micron-gamma instrument at 40 g load, at indenter loading rate of 4 g/s. To widen measurement range, indentation was conducted along a line oriented at an angle of 20–25° to the joint zone (Figure 6, *a*).

The Table (Figure 6, *b*) gives measurement results, where the highlighted line shows material properties in the butt joint area. Analysis of measurement results showed that mechanical properties of the joint zone practically do not differ from those of steel. So, a slight lowering of material microhardness was noted in the butt joint area (see Figure 5, *b*). Close values of the modulus of elasticity, microhardness and coefficient of plasticity of steel and material in the butt joint area are indicative of formation of a practically equivalent joint.

It turned out that transition from static load during welding to cyclic load (alternative application of loading and unloading) allows additional activation of diffusion processes in the joint zone. As a result, welding temperature can be lowered by 100 °C. Figure 4, *d* shows the microstructure of the zone of the joint produced at 800 °C under the conditions of cyclic loading. On the other hand, it should noted that lowering of welding temperature somewhat delays the growth of structural elements, namely pores and grains, in the butt joint area.

Thus, it is shown in the case of stainless steel that application of porous nickel foil produced by EB PVD, as an interlayer, allows lowering the temperature-force parameters of diffusion welding, compared to modes of diffusion welding without application of the interlayer.

Conclusions

Porous structure of nickel foils, produced by vacuum deposition from the vapour phase, is characterized by a low temperature of transition into the superplastic state, that is related not only to presence of open-type porosity, but also to not-equilibrium state of foil structure, which can be due to presence of excess vacancies in vacuum condensates.

Application of porous nickel foil as interlayers in diffusion welding of stainless steel allows producing defect-free joints with a small thickness of the butt zone at lower temperature-force parameters of welding (welding temperature of 800 °C, cyclic load with 20 MPa amplitude).

Influence of porous structure of nickel foil on diffusion welding parameters is related to the features of its deformational behaviour, due to lowering of the yield point at foil transition into the superplastic state and activation of diffusion processes in the joint zone. Foil plastic flow during welding ensures establishing physical contact of the surface being welded and the interlayer, and relaxation of foil non-equilibrium state activates their diffusion interaction and promotes pore healing.

1. Musin, R.A., Antsiferov, V.N., Kvasnitsky, V.F. (1979) *Diffusion welding of heat-resistant alloys*. Moscow: Metallurgiya.
2. Bakshi, O.A. (1965) On stress state of soft interlayers in welded joints under tension (compression). *Voprosy Svarochn. Proizvodstva*: Transact. of ChPI, **33–35**, 36.
3. Musin, R.A., Lyamin, Ya.V. (1991) Application of perforated interlayers in diffusion welding. *Svarochn. Proizvodstvo*, **2**, 2–4.
4. Lyamin, Ya.V., Karakozov, E.S., Musin, R.A. *Method of pressure welding with heating*. USSR author's cert. 1463415. Int. Cl. B23 K20/16. Publ. 30.01.89.
5. Bokshtejn, S.Z. (1971) *Structure and properties of metal alloys*. Moscow: Metallurgiya.
6. Lyushinsky, A.V. (2006) *Diffusion welding of dissimilar metals*. Moscow: Akademiya.
7. Movchan, B.A. (1998) Inorganic materials deposited from vapor phase in vacuum. In: *Modern materials science of 21st century*, 318–332. Kiev: Naukova Dumka.
8. Ustinov, A.I., Lyapina, K.V., Melnichenko, T.V. et al. (2008) Influence of substrate temperature on formation of porous structure in stainless steel in case of its deposition with NaCl and KCl vapors. *Advances in Electrometallurgy*, **1**, 8–11.
9. Novikov, V.G. (1973) Development of technology of vacuum diffusion welding of electromagnet casings. In: *Proc. of 7th All-Union Sci.-Techn. Conf. on Diffusion Vacuum Joining of Metals, Alloys and Nonmetallic Materials*, 102–106.
10. Gelman, A.S., Bolshakov, M.V. (1967) Effect of oxide films on joint formation in pressure welding of metals. *Svarochn. Proizvodstvo*, **10**, 23–26.
11. Kozakov, N.F. (1976) *Diffusion welding of materials*. Moscow: Mashinostroenie.
12. Konyushkov, G.V., Kopylov, Yu.N. (1974) *Diffusion welding in electronics*. Moscow: Energiya.
13. Ustinov, A.I., Lyapina, K.V., Melnichenko, T.V. (2005) Regularities of stainless steel porous structure formation during its deposition from vapor phase in presence of sodium chloride vapors. *Advances in Electrometallurgy*, **4**, 19–24.
14. Ustinov, A.I., Matvienko, Ya.I., Polishchuk, S.S. et al. (2009) Investigation of phase transformations and plastic deformation at continuous heating of Al/Cu multilayer foil. *The Paton Welding J.*, **10**, 23–27.
15. Firstov, S.A., Gorban, V.F., Pechkovsky, E.P. et al. (2007) Equation of indentation. *Dopovidi NANU*, **12**, 100–106.
16. Cahoon, J.R., Broughton, W.H., Kutzak, A.R. (1971) The determination of yield strength from hardness measurements. *Metallurg. Transact.*, 2(**7**), 1979–1983.
17. Kuznetsova, R.I., Bryukhovetsky, V.V., Pojda, V.P. et al. (1995) Mechanism of development of grain boundary pores and local heterogeneity of deformation under superplastic flow conditions. *Metallofizika i Nov. Tekhnologii*, 17(**8**), 64–72.

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