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Nitrogen diffusion along the layer boundaries after nitriding of multilayer materials

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Abstract. Diffusion processes play a key role in formation of the structures of new materials and technological processes of strengthening heat treatments, since diffusion is the reason for redistribution of substances in solids. An urgent task is to develop technologically advanced and effective methods for strengthening materials in order to improve their performance properties. There is an increasing need to improve chemical heat treatment methods, which directly affects the wear resistance of working surfaces, and, consequently, the product service life. Near-surface volumes experience increased loads, so the formation of high-strength layers becomes an important task. Quite a few methods of surface hardening are known, among which carburization, nitriding, nitrocarburization and others are widely used. The most interesting is nitriding, since it increases hardness, strength, fatigue limit, and heat resistance. However, despite the proper advantages, nitriding has a number of disadvantages, including the holding duration and small thickness of diffusion layers. The solution is related to intensification of the technological process by increasing the nitriding temperature, activating the nitriding media or directly the parts surface. All these solutions are aimed at accelerating diffusion processes, both in grain volume and along grain boundaries, the velocity along which is many times higher than the velocity of volumetric diffusion. It may be effective to use a new type of structural metal materials with a multilayer structure of hundreds of layers, with thicknesses in the micron and submicron ranges separated by large angular boundaries. The results of metallographic studies showed the effect of the steel layers interchange in the multilayer metal material on diffusion depth after chemical heat treatment. The authors proposed an accelerate diffusion model of diffusible element along the layer boundaries.

Keywords: multilayer metal materials, chemical heat treatment, nitriding, layer boundaries, diffusion

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Диффузия азота по границам слоев при азотировании многослойных материалов

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Аннотация. Диффузионные процессы играют ключевую роль в формировании структур новых материалов и технологических процессов упрочняющих термических обработок, так как именно благодаря диффузии происходит перераспределение вещества в твердых телах. Актуальной задачей является разработка технологичных и эффективных методов упрочнения материалов в целях повышения их эксплуатационных свойств. Возрастает потребность в усовершенствовании методов химико-термической обработки, что напрямую влияет на износостойкость рабочих поверхностей, а, следовательно, и на ресурс изделия. Приповерхностные объемы испытывают повышенные нагрузки, поэтому важной задачей является формирование высокопрочных слоев. Известно достаточно много методов поверхностного упрочнения, среди которых широкое применение получили цементация, азотирование, нитроцементация и др. Наиболее перспективным является азотирование, поскольку при этом происходит повышение твердости, прочности, предела усталости, жаростойкости. Однако при должных достоинствах азотирование имеет ряд недостатков, среди которых длительность выдержки и малая толщина диффузионных слоев. Поэтому разработка методик, позволяющих интенсифицировать рассматриваемый процесс, также является актуальной задачей. Традиционно, решение связано с интенсификацией технологического процесса путем повышения температуры азотирования, активации сред азотирования или непосредственно поверхности деталей. Все эти решения направлены на ускорение процессов диффузии как по объему зерна, так и по межзеренным границам, скорость по которым многократно превосходит скорость объемной диффузии. Учитывая это, эффективным может оказаться использование нового типа конструкционных металлических материалов с многослойным строением из сотен слоев с толщинами микронного и субмикронного диапазона, разделенными между собой большими угловыми границами. В работе приведены результаты металлографического исследования, показывающие влияние чередования слоев сталей в многослойных металлических материалах на глубину диффузии при проведении химико-термической обработки азотированием. Предложена модель ускоренного проникновения диффундирующего элемента по границам слоев.

Ключевые слова: многослойные материалы, химико-термическая обработка, азотирование, границы слоев, диффузия

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Introduction

Currently various mechanical wood treatments by milling are widely used. The main unit of a router is the cutter. There are numerous cutters of different designs and geometries, but, in each case, they should be characterized by high strength and wear resistance, which can be achieved by chemical heat treatment [1]. The multilayer materials used after nitriding for manufacturing cutters can enable to enhance tool durability, preserve tool geometry, increase tool life, and improve processing performance due to a significantly hardened layer. Fig. 1 shows an example of such a cutter.

Additionally, multilayer materials can be used to manufacture gear wheels that also operate under wear (Fig. 2).

This structure can be obtained if steels with different crystalline structure are included in the initial composition [2 – 4]. The developed technological route (Fig. 3) enables to produce sheet billets, 2 to 10 mm thick [5].

The material microstructure has a multilayer laminar structure with the layers, 100 to 0.8 μm thick. At the same time, the layers are characterized by crystallographic disorientation from 15 to 20°, which corresponds to the large-angle grain boundaries in the initial steels (Fig. 4) [6; 7].

The works [8; 9] show that with an appropriate choice of steels included in the initial composite billet, the multilayer structure is preserved up to the temperature of 1000 °C, which corresponds to the temperature of hot

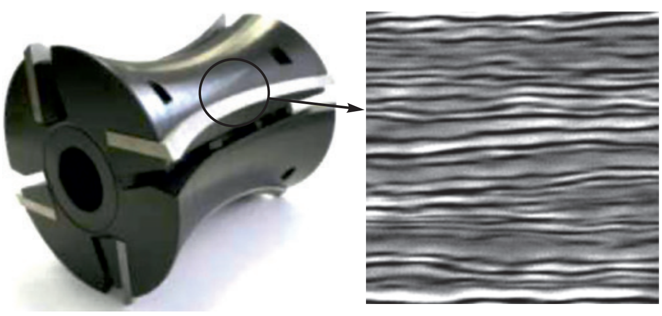


Fig. 1. Layout of a cutter from a multilayer material

Рис. 1. Схема фрезы из многослойного материала

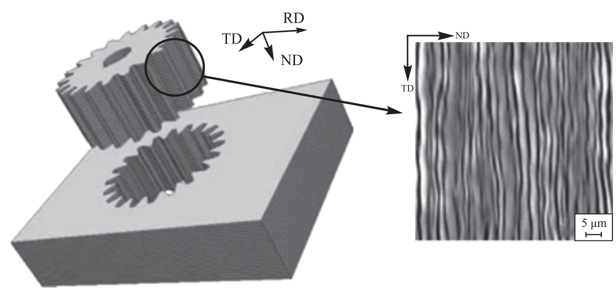


Fig. 2. Scheme of manufacturing a gear wheel from a multilayer material

Рис. 2. Схема изготовления зубчатого колеса из многослойного материала

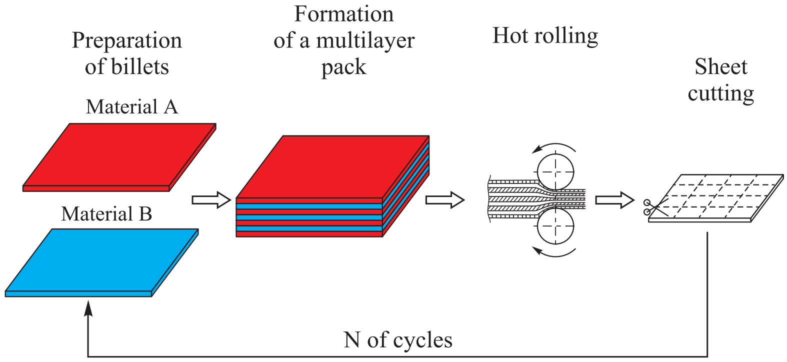


Fig. 3. Scheme of technological route

Рис. 3. Схема технологического маршрута

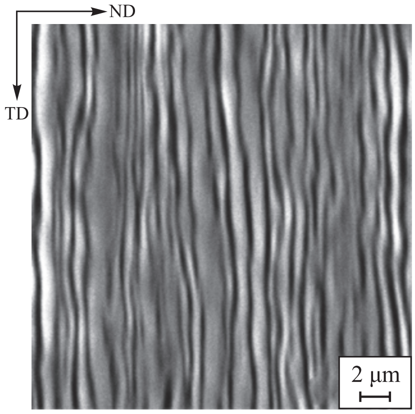


Fig. 4. Typical microstructure of cross section of a multilayer material (composition based on steels AISI420 and AISI304)

Рис. 4. Типичная микроструктура поперечного сечения многослойного материала (композиция из сталей 08Х18Н10 и 40Х13)

pack rolling. In this case, the cross-section of the sheet billets has a structural orientation ready for chemical heat treatment (Fig. 5).

Preliminary evaluation of nitriding the multilayer composition that includes AISI304 and AISI430 steels showed that the nitriding depth in the multilayer material is greater than its depth in AISI304 steel which is hard to nitride. The reason behind the increased thickness of the nitrided layer in multilayer materials may be the accelerated diffusion of nitrogen along the layer boundaries with subsequent saturation of layer volumes from their boundaries as from the diffusing element sources [10].

To thoroughly investigate the resulting effect, we used model compositions of multilayer materials containing steels of different compositions (grades). The following study objects were selected for nitriding: composites consisting of 1008 and AISI304 steels, as well as W108 and AISI304 steels. After hot pack rolling, we obtained 10-mm thick samples with 100 layers, the single layer being 100 µm thick.

The samples of these compositions were nitrided, the nitriding surfaces being in all cases perpendicular to the rolling directions used to produce the multilayer samples. Nitriding was performed in the gas atmosphere containing 20 – 40 % ammonia dissociation products. We used two nitriding conditions with the following temperature and time parameters: 540 °C for 45 h and 580 °C for 25 h. To study the structure of the resulting nitrided layers and to determine their geometric characteristics, we prepared sections with the surfaces perpendicular to the rolling direction and parallel to the direction of nitrogen diffusion penetration that occurred in the course of nitriding (Fig. 6).

Fig. 7 shows the general view of diffusion profiles formed during nitriding for AISI304 and 1008 (*a*), AISI304 and W108 (*b*) compositions obtained after nitriding at 540 °C for 45 h.

The micrographs show the concentration profile of diffusing nitrogen in AISI304 steel with a considerable nitrogen penetration depth (and a long nitrogen diffusion path, respectively) along the layer boundaries of the multilayer material. Inside the AISI304 component layer, for both cases, the nitrogen penetration distance shrinks with increasing distance from the interlayer boundary. The smallest depth of nitrogen penetration is about 100 µm from the outer surface of the samples and midway between the layers of neighboring components (midway between the layers of 1008 steel (*a*) and W108 steel (*b*)) inside the layer of AISI304 steel. This indicates that the source of nitrogen penetration into the volume of AISI304 steel during nitriding is the interface between the layers of the material. The diffusion along these boundaries occurs faster than that through the layer of AISI304 steel from the outer surface.

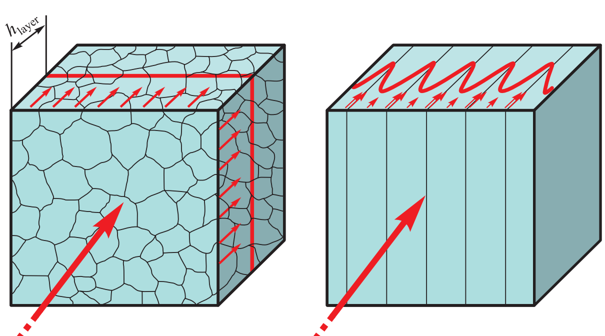


Fig. 5. Scheme of polyhedral and laminar structure of structural metallic materials indicating diffusion profiles. Arrows indicate the direction of diffuser flow

Рис. 5. Схема полиэдрического и ламинарного строения конструкционных металлических материалов с указанием диффузионных профилей. Стрелками указано направление потока диффузанта

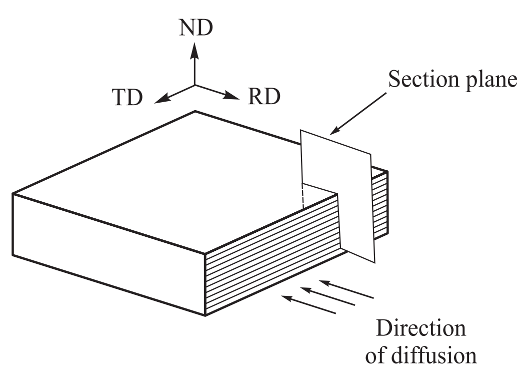


Fig. 6. Scheme of sample cutting for metallographic analysis

Рис. 6. Схема вырезки образцов для проведения металлографического анализа

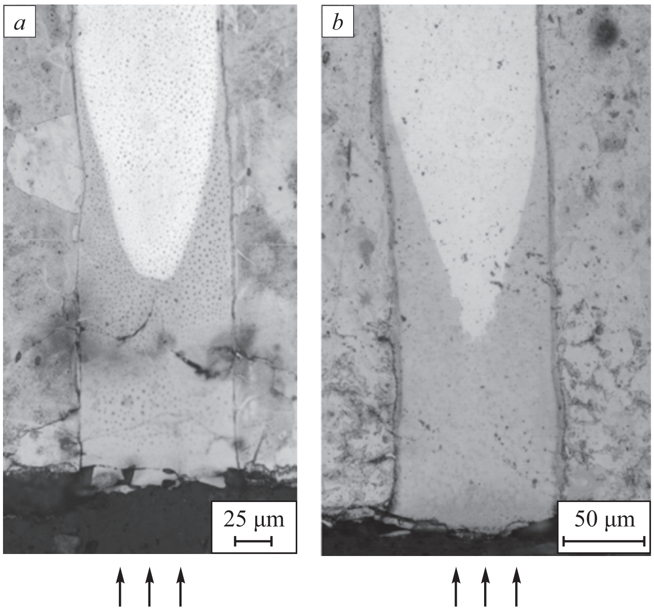
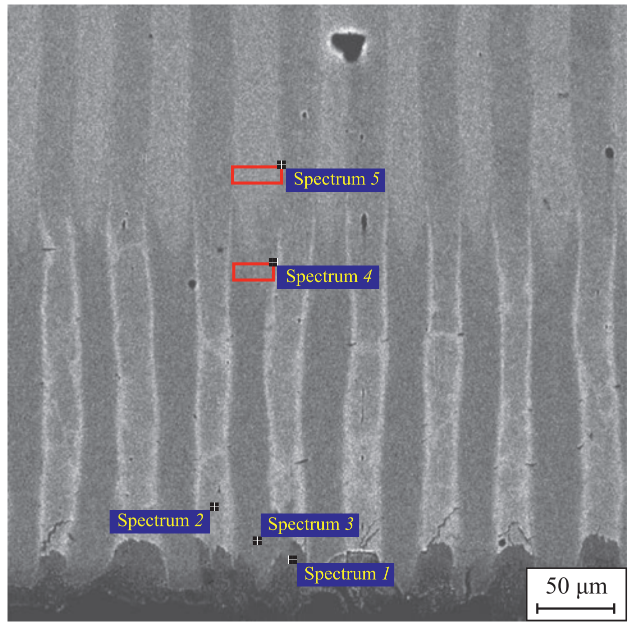


Fig. 7. Microstructure of nitrided layer of composition based on steels: AISI304 and 1008 (*a*), AISI304 and W108 (*b*). Arrows indicate the direction of nitrogen diffusion flow

Рис. 7. Микроструктура азотированного слоя композиций 08Х18Н10 + 08кп (*a*) и 08Х18Н10 + У8 (*b*). Стрелками указано направление диффузионного потока азота



Spectrum	Content, wt. %					
	N	Si	Cr	Mn	Fe	Ni
1	4.53	0	0.63	0.42	94.42	0
2	0	0	0.63	0.46	98.68	0.24
3	4.62	0.57	18.20	1.73	67.43	7.45
4	4.24	0.44	17.48	1.74	68.61	7.49
5	0	0.57	18.10	1.67	72.09	7.57

Fig. 8. Results of qualitative MRS analysis of the diffusion layer of composition based on steels W108 and AISI304

Рис. 8. Результаты качественного МРС анализа диффузионного слоя композиции У8 + 08Х18Н10

Nitrogen penetration into the layers of 1008 and W108 steels at this depth from the nitriding surface was not detected. This is proved by the results of electron microscopy and *X*-ray spectral analysis. There is practically no volume diffusion of nitrogen in 1008 and W108 steels due to low solubility of nitrogen in α-iron (maximum 0.1 wt. %), which is the main pathway of nitrogen diffusion in the ferrite phase [11]. The second reason inhibiting nitrogen movement in 1008 steel is the surface nitride formation consisting of nitrides (Fe_2N), which is also proved by the results of electron microscopy (Fig. 8).

Thus, it can be concluded that the nitriding process in multilayer materials of this type occurs due to the fast nitrogen diffusion along the layer boundaries [12; 13].

It can be assumed that simultaneously nitrogen atoms migrate inside the AISI304 steel perpendicular to the interlayer boundary, which can be considered as an extended source of nitrogen diffusion. This steel is an austenitic grade steel and the solubility of nitrogen in austenite (γ-iron) is about 2.8 wt. %, therefore, diffusion saturation of AISI304 layers with nitrogen is possible. According to literature data [14], the diffusion coefficient of nitrogen in γ-iron at temperatures ranging from 500 to 600 °C is determined by the following equation

$$D_\gamma = 4.6\cdot 10^{-5}\exp\left(-\frac{108{,}474}{RT}\right),\ \text{m}^2/\text{s}. \qquad (1)$$

We used the Fisher model for calculating diffusion along grain boundaries in metallic samples to determine the diffusion permeability of layer boundaries of the multilayer material [15 – 18]. According to this model, the product of the diffusion coefficient D_b along the grain boundary (layer boundaries in this case) and the boundary thickness δ, that is value of $s\delta\ D_b$, can be calculated by the formula [19]

$$s\delta D_b = (\pi t)^{1/2} D_\gamma^{3/2}\text{ctg}^2\theta, \qquad (2)$$

where θ is the angle at the top of the component concentration profile (Fig. 7), which passes into the phase (layer) volume from the grain (layer) boundary; *s* is the ratio of boundary enrichment with atoms of the diffusing component, which can be estimated based on the dependence proposed in [20]:

$$sx_0 = 6.2 \pm 4.5, \qquad (3)$$

where x_0 is the volumetric concentration of the impurity in mole fractions.

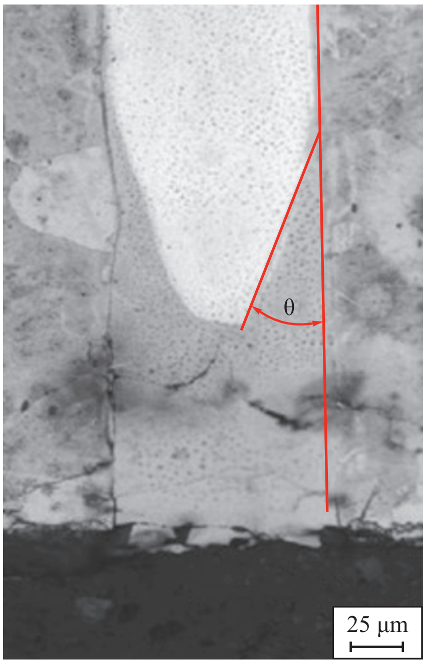


Fig. 9. Concentration angle θ for determining the diffusion coefficient in a multilayer material

Рис. 9. Концентрационный угол θ для определения диффузионной проницаемости слоевых границ в многослойном материале

If we assume that the enrichment of layer boundaries is mainly determined by the ferrite phase, since in accordance with the phase diagram it has the smallest Fe – N concentration of nitrogen, according to formula (3), the value of enrichment of layer boundaries shall equal $s = 5 \cdot 10^3$.

It should be noted that the formula (2) is suitable for describing diffusion along the layer boundary when the diffusive removal of the component (nitrogen in this case) from the boundary into the bulk is one-sided – volume diffusion occurs only towards the layers of AISI304 steel. The experimental data shows that there is no diffusion of nitrogen toward the layers of 1008 steel (Fig. 10, *a*) or W108 steel (Fig. 10, *b*).

The values of angles θ required to calculate the diffusion coefficient D_b along the layer boundaries were determined by analyzing micrographs of cross-sections of multilayer samples of both compositions after nitriding using two processing modes.

For the 1008 + AISI304 composition, Fig. 8, *a* shows the method for measuring these angles when nitriding is performed at 540 °C for 45 h and Fig. 10, *b* – when the operating parameters are 580 °C and 25 h.

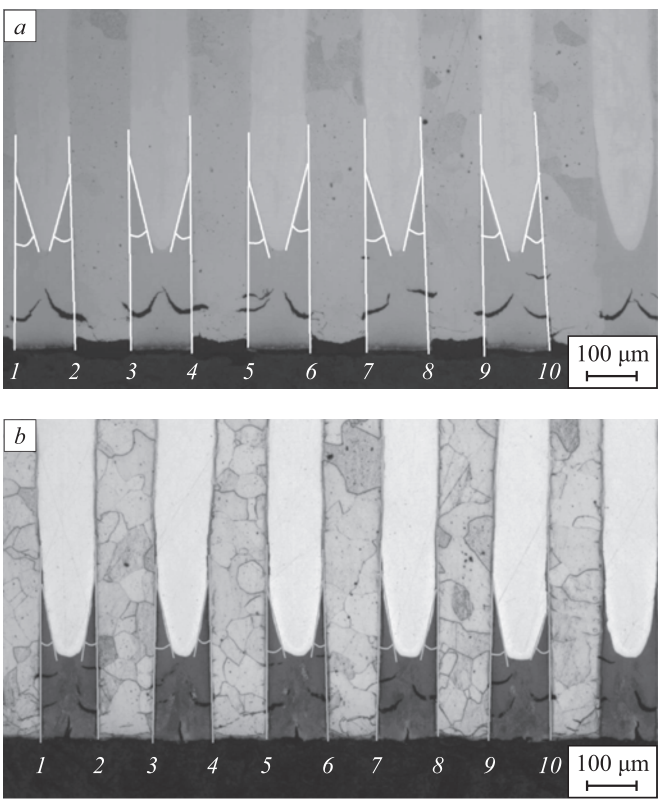


Fig. 10. Determination of the angles θ for calculating the product of δ and D_b for the composition based on steels 1008 and AISI304 after nitriding: t = 540 °C, 45 h (*a*); t = 580 °C, 25 h (*b*)

Рис. 10. Определение углов θ для расчета произведения δ D_b для композиции 08кп + 08Х18Н10 после азотирования при 540 °С, 45 ч (*a*); 580 °С, 25 ч (*b*)

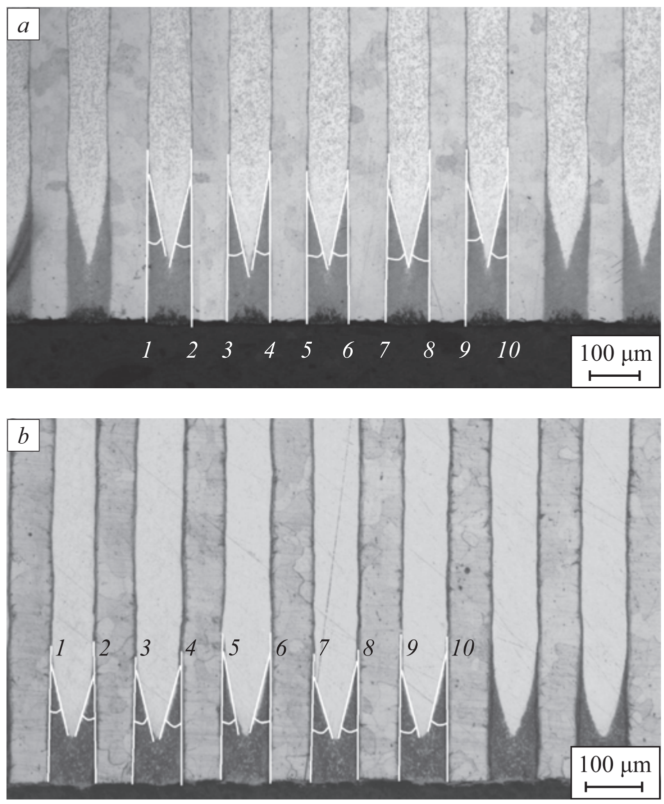


Fig. 11. Determination of the angles θ for calculating the product of δ and D_b for the composition based on steels W108 and AISI304 after nitriding: t = 540 °C, 45 h (*a*); t = 580 °C, 25 h (*b*)

Рис. 11. Определение углов θ для расчета произведения δ D_b для композиции У8 + 08Х18Н10 после азотирования при 540 °С, 45 ч (*a*); 580 °С, 25 ч (*b*)

Nitrogen diffusion coefficients along the layer boundaries D_b, m²/s

Коэффициенты диффузии азота по границам слоев D_b, м²/с

Composition	Nitriding mode	
	540 °C, 45 h	580 °C, 25 h
1008 + AISI304	$1.9 \cdot 10^{-8}$	$8.1 \cdot 10^{-8}$
W108 + AISI304	$4.3 \cdot 10^{-8}$	$15.9 \cdot 10^{-8}$

For the W108 + AISI304 composition, the similar procedure is shown in Fig. 11.

The Table presents the diffusion coefficients of nitrogen atoms along the layer boundaries for 1008 + AISI304 and W108 + AISI304 multilayer compositions obtained by analyzing experimental data as nitriding of these materials samples was investigated. The calculation assumes that the layer boundaries thickness δ is 10^{-9} m.

Conclusions

The experimental study of nitriding the samples of multilayer metallic materials with alternating layers of two different steel grades revealed that the main mechanism behind the process is mass transfer (diffusion) of nitrogen atoms along the boundaries of the material layers.

The analysis of experimental data obtained while investigating cross sections of surface layers of two compositions of multilayer materials after nitriding using two modes enabled us to obtain the estimated values of nitrogen diffusion coefficients D_b along layer boundaries. The D_b values were 10^4 times higher than the volume diffusion coefficient of nitrogen in AISI304 steel under the same conditions.

The study showed that the nitriding depth in both multilayer compositions increased due to fast diffusion penetration of nitrogen atoms along the layer boundaries of multilayer materials.

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K. B. Polikevich – literary review, selection and preparation of the research objects.
A. L. Petelin – development of a mathematical model and determination of diffusion coefficients for the research objects.

A. I. Plokhikh – statement of the problem, development of the research concept.
L. P. Fomina – carrying out the technological process of nitriding on the studied compositions.

Вклад авторов

К. Б. Поликевич – анализ литературы, подбор и подготовка объектов исследования.
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А. И. Плохих – постановка проблемы, разработка концепции исследования.
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