ҚАЗАҚСТАН РЕСПУБЛИКАСЫ ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ

Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК РЕСПУБЛИКИ КАЗАХСТАН Satbayev University

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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАНРК сообщает, что научный журнал «Известия НАНРК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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EVALUATION OF THE PROPERTIES OF THERMODIFUSION ZINC COATING OF COUPLINGS OF PUMP-COMPRESSOR PIPES PRODUCED BY «KSP STEEL»

Abstract. The protection of tubing pipes from corrosion destruction is now becoming especially important. In world practice, one of the promising directions for preventing corrosion damage is the use of protective coatings for couplings. The choice of the method of applying a protective coating is an important step in ensuring the long-term operation of tubing. The wrong choice of the type of coating leads to additional operating costs. Among the existing methods of protection against corrosion, the operation of couplings with a protective thermal diffusion zinc coating is relevant, which makes it possible to increase the operational reliability and service life of tubing couplings and provide protection for the "pipe-coupling" threaded connection.

This article presents the results of a comprehensive assessment of the properties of a protective coating applied by the method of vapor-phase thermal diffusion galvanizing on couplings for tubing in the conditions of "KSP Steel LLP". In the course of the work, the properties of the coating were determined, which ensure the corrosion resistance of metal products. Methods of assessment are stated and the results of studies of technological, operational and physical-mechanical properties of the coating are presented.

The results of the study of the technological, physical-mechanical and operational properties of the coating showed that the properties of the coating that provide corrosion resistance, such as: thickness, wear resistance, adhesion strength of the coating to the surface of a metal product, porosity, corrosion protection properties correspond to the normalized values.

Key words: Thermal diffusion galvanizing, protective coatings, pump-compressor pipes, porosity, corrosion resistance.

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«KSP STEEL» ӨНДІРІСІНІҢ НАСОС-КОМПРЕССОРЛЫҚ ҚҰБЫРЛАР МУФТАЛАРЫН ТЕРМОДИФУЗИЯЛЫҚ МЫРЫШПЕН ҚАПТАУ ҚАСИЕТТЕРІН БАҒАЛАУ

Аннотация. Түтік құбырларын коррозияның бұзылуынан қорғау қазіргі уақытта ерекше маңызды болып отыр. Әлемдік тәжірибеде коррозиялық зақымдануды болдырмаудың перспективалы бағыттарының бірі муфталар үшін қорғаныс жабындарын қолдану болып табылады. Қорғаныс жабындысын қолдану әдісін таңдау түтіктердің ұзақ мерзімді жұмысын қамтамасыз етудегі маңызды қадам. Қорғаныс жабынының түрін қате таңдау қосымша пайдалану шығындарына әкеледі. Коррозиядан қорғаудың қолданыстағы әдістерінің ішінде қорғаныс термиялық диффузиялық мырыш жабыны бар муфталардың жұмысы өзекті, бұл құбыр муфталарының пайдалану сенімділігі мен қызмет ету мерзімін арттыруға және «құбыр- муфтаның» бұрандалы қосылымын қорғауды қамтамасыз етуге мүмкіндік береді.

Мақалада «KSP Steel» ЖШС жағдайында құбырларға арналған муфталарда бу- фазалы термиялық диффузиялық мырыштау әдісімен қолданылатын қорғаныс жабынының қасиеттерін кешенді бағалау нәтижелері берілген. Жұмыс барысында металл бұйымдарының коррозияға төзімділігін қамтамасыз ететін жабынның қасиеттері анықталды. Бағалау әдістері баяндалған және жабынның технологиялық, пайдалану және физикалық-механикалық қасиеттерін зерттеу нәтижелері берілген. Қаптаманың технологиялық, физика-механикалық және эксплуатациялык касиеттерін зерттеу нәтижелері жабынның коррозияға төзімділігін қамтамасыз ететін қасиеттерін көрсетті, мысалы: қалыңдығы, тозуға төзімділігі, жабынның металл бұйымның бетіне жабысу беріктігі, кеуектілік, коррозиядан қорғау қасиеттері нормаланған мәндерге сәйкес келеді.

Түйін сөздер: термиялық диффузиялық мырыштау, қорғаныс жабындары, сорғы-компрессорлық құбырлар, кеуектілік, коррозияға төзімділік.

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ОЦЕНКА СВОЙСТВ ТЕРМОДИФФУЗИОННОГО ЦИНКОВОГО ПОКРЫТИЯ МУФТ НАСОСНО-КОМПРЕССОРНЫХ ТРУБ ПРОИЗВОДСТВА "KSP STEEL"

Аннотация. В настоящее время особое значение приобретает защита насосно-компрессорных труб от коррозионного разрушения. В мировой практике одним из перспективных направлений предотвращения коррозионных повреждений является применение защитных покрытий муфт. Выбор способа нанесения защитного покрытия является важным этапом в обеспечении длительной эксплуатации НКТ. Неправильный выбор типа защитного покрытия приводит к дополнительным эксплуатационным затратам. Среди существующих способов защиты от коррозии актуальным является эксплуатация муфт с защитным термодиффузионным цинковым покрытием, позволяющим повысить эксплуатационную надежность и срок службы муфт НКТ и обеспечивающим защиту резьбового соединения «труба-муфта».

В данной статье представлены результаты комплексной оценки свойств защитного покрытия, нанесенного методом парофазного термодиффузионного цинкования на муфты для НКТ в условиях ТОО «KSP Steel». В ходе работы были определены свойства покрытия, обеспечивающие коррозионную стойкость металлических изделий. Изложены методы оценки и представлены результаты исследований технологических, эксплуатационных и физико-механических свойств покрытия.

Результаты исследования технологических, физико-механических и эксплуатационных свойств покрытия показало, что свойства покрытия, обеспечивающие коррозионную стойкость, такие как: толщина, износостойкость, прочность сцепления покрытия с поверхностью металлического изделия, пористость, защитные свойства от коррозии соответствуют нормируемым значениям.

Ключевые слова: термодиффузионное цинкование, защитные покрытия, насосно-компрессорные трубы, пористость, коррозионная стойкость.

Introduction. Violation of threaded joints "pipe-coupling", due to metal corrosion, is the cause of up to 55% of all accidents and failures of tubing in the fields (Penner, 2013).

One of the promising methods of corrosion protection is the operation of pipes and couplings with protective coatings, which make it possible to increase the operational

reliability and service life of tubing, significantly reduce metal losses from corrosion, and improve the environmental situation in production (Proskurkin, 2003).

The effectiveness of protecting metal products from corrosion depends on the following properties of the coatings:

the strength of adhesion to the surface of a metal product, on which the resistance to corrosion, abrasive wear, oxidation, cavitation and erosion depends (Proskurkin, 1988);

microhardness, which determines the resistance to corrosive aggressive media during operation;

porosity, which determines the protective function of the coating against aggressive media (Jain, 2021);

- the main phase in the structure of diffusion coatings is the δ _1-phase (iron 7-12% (wt) and 93-88% zinc), which provides the coating with corrosion resistance (Proskurkin, 2015 a).
- the thickness of the coating, which determines the service life (the duration of its destruction from exposure to the environment to exposure of the base) of the coating;
- uniformity of coating distribution along the thread profile is an important indicator that provides complete surface sealing and high protective properties (Proskurkin, 2015b).

Research materials and methods. The object of the study was couplings for seamless tubing made of carbon steel, standard size 108×15.5 mm, strength group N80, type Q according to GOST 31446, with a coating applied by the method of vaporphase thermal diffusion galvanizing (TDG) at the base of enterprise KSP Steel LLP (GOST 31446-2017).

The technological process was carried out with the following parameters:

heating at a temperature of 435°C for 140 minutes;

cooling for 90 minutes, with a vacuum in the furnace retort of 0.7-0.8 bar.

Heat treatment of the sleeve consisted of quenching and subsequent tempering. The physical and mechanical properties of the couplings are shown in Table 1.

Indicator name	Mechanical properties, for steel grade		
	Normalized values	Actual values	
Ultimate tensile strength $\sigma_{_{B}}$, not less, MPa	689	822,8	
Yield strength στ			
not less, MPa	650-758	702,9	
no more, MPa			
Relative elongation Af,%, not less	16	24,6	

Table 1 - Physical and mechanical properties of couplings with thermal diffusion coating

The appearance of the coating was evaluated in accordance with GOST R 9.316.

The thickness of the coating was estimated by the eddy current method according to GOST 9.302 using the "Constant K5" device. The relative error of this method is \pm 5%. The thickness control was carried out on the lower and upper sides of the sleeve at 6 points on each inner groove (ASTM B602 – 88).

The control of the thickness of the coating on the thread by the metallographic method was carried out in accordance with GOST 9.302. Microsections polished on a grinding

polishing machine and etched with 4% alcohol solution HNO₃ were used as a material for determining the thickness of the coating on the thread of the coupling. Sections were studied using an Olympus BX51M metallographic microscope. According to the requirements of GOST 633-80, the interference fit of the coupling with a coating along the thread gauge of the plug (the distance from the end of the coupling to the end of the thread runoff on the pipe) should be from 2.5 to 7.5 mm. To comply with this requirement, the thickness of the zinc coating is regulated within 40 ± 20 microns («Pervouralsk Novotrubny Plant» JSC).

To assess the uniformity of the coating, the thickness was measured on the outer side of the coating, on the upper and lower sides of the box: along the ends and along the outer band, at six points on each surface. Uniformity was estimated as the deviation from the arithmetic mean of all thickness measurements, in percent. In foreign and domestic NTD and GOST there are no unambiguous requirements for the uniformity of the coating, according to the results of the literature review, the deviation of the thickness from the arithmetic mean no more than 5% is accepted as the standard value (Chizhov, 2015).

The assessment of the wear resistance of the coating was carried out in accordance with the requirements of API 5 B (API 5B-2017) according to the factory method, the material was pipes with a nominal diameter of 73 mm, strength group D (ASTM F3226) and couplings with a thermal diffusion coating of heat No. 5190973. The protective properties of the coating against corrosion were determined through the index of resistance to pitting formation. The test was carried out in accordance with the requirements of GOST 9.302 and the technological regulations of the enterprise (Technological regulations "Thermal diffusion galvanizing of couplings"). Microhardness was measured in accordance with GOST 9450 (Bao, 2011) according to the factory method on a Shimadzu HMV-2 series device (PA «KSP Steel» LLP). The microstructure of the coatings was determined according to GOST 5640 on polished, polished and etched with 4% alcohol solution of HNO₃ microsections, which were studied at a magnification of × 60 to × 1000 on an Olympus BX51M microscope (Elkoca, 2015). According to GOST 5640, the microstructure is assessed on microsections with a size of 30x40 mm. Samples for microsections were cold cut mechanically so that their plane coincided with the direction of the fibers (samples should be longitudinal). The adhesion strength of the coating to the metal of the coupling (adhesion) was determined according to GOST 9.302-88, by the heating method. The porosity of the coating is determined by various methods according to GOST 9.302. In this work, the corrosion method was used.

Result and discussion. A general view of samples with a coating on the outside and a threaded surface is shown in Figure 1.





Figure 1. Coupling with a thermal diffusion coating: a. appearance; b. threaded surface

The appearance assessment showed that the thermal diffusion zinc coating meets the requirements of the standards for this coating. The results of measuring the thickness of the coating are shown in tables 2, 3.

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N_{2}	Thickness, micron					
meas.	On the upper and lower inner girdle of the coupling	On the uncoated coupling (conditional zero)	Arithmetic mean - conditional zero	Taking into account the method error ± 5%		
1		ZCIO)				
1	68	9				
2	68					
3	69	9				
4	68					
5	74	8		ı		
6	73	o				
7	62	9	66,1-8,83=57,27	60,13-54,41 micron		
8	62	9				
9	60	o				
10	62	8				
11	59	10				
12	68	10				
Average	66.1	8.83				

Table 2 - The results of measurements of the coating thickness, using the device "Constant K5"

The data of metallographic control of the coating thickness are given in Table 3, the type of coating is shown in Figure 2.

Thickness of TDG coating, microns On the side Normalized values At the top of the In the socket № measuring Average tooth surface meaning 47 1 23 30 2 48 28 34 3 47 29 35 35,7 20-60 4 46 26 35 5 42 30 32 44 32 35

Table 3 - Thickness of coatings on the coupling thread





Figure 2. View of TDG coating on the coupling thread when measuring the thickness by the metallographic method: a. socket; b. apex of the tooth

According to the results of evaluating the coating thickness by the eddy current method using the Constant K5 device and the metallographic method using an Olympus BX51M microscope, the coating thickness is on average 57.27 microns (the minimum value with an error of 5% is 54.41 microns) and 35.7 microns respectively, which corresponds to the normalized values (from 20 to 60 microns).

The results of measurements of the thickness of the sleeve, to assess the uniformity of the coating, taking into account the "conditional zero" indicator are given in Table 4, the results of evaluating the uniformity are shown in Table 5.

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№ meas.	Coating thickness, microns						
	On the upper end	On the upper inner	At the	On the lower inner	Average		
	of the coupling	belt of the coupling	bottom of the	shoulder of the	value		
			coupling	coupling			
1st meas.	51,17	59,17	38,17	53,17	50,42		
2 nd meas.	44,17	59,17	37,17	53,17	48,42		
3 rd meas.	53,17	60,17	35,17	51,17	49,92		
4th meas.	48,17	59,17	37,17	53,17	49,42		
5th meas.	60,17	65,17	38,17	50,17	53,42		
6 th meas.	54,17	64,17	35,17	59,17	53,17		
Average	51,84	61,17	36,84	53,34	50,79		

Table 4 - Results of coating thickness control with the "Constant K5" device, to assess the uniformity

Table 5 - The results of evaluating the uniformity of the coating on the couplings

Type of coating			Coa	ating thickness,	microns	
		On the	On the upper	At the	On the lower	Average value
		upper end of	inner belt of	bottom of the	inner shoulder of	
		the coupling	the coupling	coupling	the coupling	
TDG, micron	max	60,17	65,17	38,17	59,17	51,42
	min	44,17	59,17	35,17	50,17	31,42
Deviation max, min	max	17,02	26,74	25,76	15,07	18,5
from the mean value,%	min	14,09	15,07	31,60	2,4	18,5

After analyzing the data in Table 5, we can conclude that the coating is not uniform enough with a deviation from the arithmetic mean of the coating thickness by 18.5% (with the accepted uniformity indicator, the deviation is no more than 5%). However, taking into account the absence of regulatory requirements for uniformity in NTD and the compliance of the minimum thickness indicators with the standard values (from 20 to 60 microns), we can say that the coating with this uniformity indicator will protect the product from the effects of aggressive media.

Wear resistance was determined after 6-times make-up-unscrewing of a sleeve with a coating with tubing pipe on a sleeve-screwing machine.

The actual make-up moments for each stage of make-up are given in Table 6, the appearance of the threaded surface of the coupling after the assessment of wear resistance is shown in Figure 3.

Make-up stage, №	Recommended make-up torques, N·m			Actual make-up
	min	optimal	max	torque, N·m
1	978,75	1305	1631	1464
2				1521
3				1428
4				1433
5				1428
6				1442

Table 6 - Make-up moments for each make-up stage

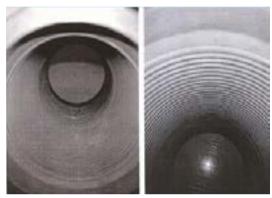


Figure 3. Threaded surface of a coupling with TDG coating after wear resistance tests

Thus, it can be concluded that the coating has good frictional properties, allowing at least six acts of make-up and unscrewing of a pipe with a coupling without damaging the threaded surface and meets the requirements of API 5B (16th edition).

To assess the protective properties against corrosion, a freshly prepared solution of copper sulfate with a concentration of 100~g / dm^3 was used, which was applied to a sample previously defatted with alcohol and held for 60 seconds. The place where the drop was applied was limited to the glass with a pencil. Samples coated and uncoated after testing are shown in Figure 4.



a



Figure 4. Samples after the study of protective properties: a. coated sample; b. uncoated sample

As you can see from the figure, after 60 seconds, a pink spot was formed on the uncoated samples. On the coated sample, there are no pink pits, which is an indicator of the provision of high-quality corrosion protection of the product.

When determining the microhardness of the coating, measurements were carried out on cross-sectional sections of the samples. The measurement results are shown in Table 7.

The hardness of the coating averaged 649.33 HV0.2. The table shows that the hardness changes significantly. Thus, the hardness of the layer located closer to the base metal was 788 HV0.2, and in the layers closer to the surface, it decreases, which is comparable to the presence of phases, the hardness of which changes (mainly decreases) with approaching the outer surface of the coating. In general assessment, the hardness of the coating is high, compared to the hardness of uncoated metal, averaging 224.5 HV0.2 due to the presence on the metal surface of phases and intermetallic compounds of metal with zinc in various ratios that form the basis of the coating.

№ meas.	Hardness, HV _{0.2}			
Nº Ilicas.	Uncoated	Coated (454-471 HV)		
1	227	788		
2	225	751		
3	222	680		
4	225	610		
5	220	548		
6	228	518		
Average value	224,5	649,33		

Table 7 - Results of measurements of the hardness of the zinc coating

The results of metallographic studies at various magnifications are shown in Figure 5.

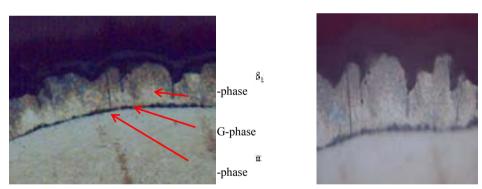


Figure 5. Microstructure of zinc coating: a. × 200 magnification; b. × 1000 magnification

In the study of thermal diffusion zinc coating, the following phase layers were found: α -phase - the transition zone from the base metal to the coating, on the microstructure of the form as a weakly etched white layer. It is a solid solution of zinc in iron;

G-phase - a dark narrow strip located on a steel base is the hardest phase in the coating;

 δ 1-phase - mainly has a columnar structure, gives corrosion resistance to the coating. When testing the strength of adhesion (adhesion) of the coating, the sample of the coated sleeve was heated to a temperature of 190°C, kept at this temperature for 1 hour and cooled in air, after which its appearance and integrity were evaluated.

The threaded surface of the coated coupling before and after the bond strength test is shown in Figure 6.





Figure 6. Threaded surface of the coated coupling before and after the bond strength test:

a. before the test: b. after the test

As can be seen from the figure, on the surface of the sleeve after heating, no swelling or delamination was found, the surface was not deformed, and the adhesion strength of the coating to the metal was high.

To assess the porosity, the samples were immersed in a solution containing potassium ferrocyanide $3 \text{ g} / \text{dm}^3$ and sodium chloride $10 \text{ g} / \text{dm}^3$ at a temperature of 22°C . The immersion time of the samples is 5 minutes. The surface of the samples after testing is shown in Figure 7.





Figure 7. Surface of samples after porosity tests: a. coated sample; b. uncoated sample

As can be seen from Figure 7, no blue pits were found on the surface of the sample with a coating, which is an indicator of the non-porosity of the coating.

Conclusion. As a result of a comprehensive assessment of the properties of a thermal diffusion zinc coating applied by the vapor-phase method, the following results were obtained:

- 1. the appearance meets the requirements of GOST R 9.316;
- 2. the average value of the coating thickness, taking into account an error of 5%, is equal to 57.27 microns, the minimum thickness is 54.41 microns and 335.7 microns on the thread, which corresponds to the standardized requirements for the coating

thickness for corrosion protection in accordance with GOST 9.302 and for interference in accordance with thread according to GOST 633-80;

- 3. the uniformity of the thickness of the coating over the surface of the product has a deviation from the arithmetic mean of the thickness by 18.5%. Taking into account the absence of regulatory requirements for uniformity in NTD and the compliance of the minimum thickness indicators (35.17 μ m) with the standard values, it can be assumed that the coating at this uniformity indicator will protect the product from the effects of aggressive media;
 - 4. the coating has high wear resistance and resistance to pitting corrosion;
- 5. the coating has a high hardness an average of 649.33 HV0.2, the outer layers of the coating have softer layers that play the role of solid lubricant and provide high wear resistance to the coating;
 - 6. the coating has high adhesion to the surface of the product;
 - 7. the coating has no pores;
- 8. there is a δ_1 -phase in the microstructure of the coating, which imparts corrosion resistance.

In general, according to the results of the complex tests carried out, it can be concluded that the coating obtained by the method of vapor-phase thermal diffusion galvanizing under the conditions of KSP Steel LLP provides reliable corrosion protection for tubing couplings.

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