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Research article
Научная статья

Influence of coatings for urea-based patterns on the quality of shell molds produced using colloidal silica binders

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Abstract: In the investment casting process, in addition to wax patterns, water-soluble salt patterns made of urea are also used. It is known that urea-based patterns provide high strength and allow the patterns to maintain their shape even if the temperature in the foundry increases. However, due to environmental and production-related reasons, there is currently a growing demand for transitioning to a technological process involving colloidal silica binder. This transition presents challenges related to the manufacturing of ceramic shell molds due to the interaction between the pattern compound and the colloidal silica binder slurry. This study examines the effectiveness of protective coatings based on repair wax, varnish (AK 593), and varnish with rosin, applied to water-soluble urea-based patterns containing additives such as magnesium sulfate, potassium nitrate, polyvinyl alcohol, and dimethylglyoxime. The degree of interaction was assessed by measuring the wetting angle and the spreading area of the colloidal silica binder over the surface of pattern samples with various coatings. It was found that all coatings contributed to an increase in the wetting angle and a reduction in the spreading area. Additionally, ceramic molds and castings made of nickel superalloy were produced using a series of pattern compounds with protective coatings. The surface roughness and dimensional accuracy of the castings were evaluated. It was demonstrated that the protective properties of the repair wax-based coating were insufficient, leading to the formation of cracks and sagging in the mold. This resulted in penetration defects in the castings and a significant decrease in dimensional accuracy. In contrast, when using coatings based on varnish and varnish with rosin, no defects were observed in the mold or castings, making these coatings recommended as protective solutions for urea-based pattern compounds in contact with colloidal silica binder slurries.

Keywords: urea-based pattern compounds, water-soluble salt patterns, colloidal silica binders, protective coatings, wetting angle.

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Влияние покрытий для карбамидных моделей на качество оболочковых форм, полученных с применением водных связующих

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Аннотация: При изготовлении отливок методом литья по выплавляемым моделям наряду с восковыми выплавляемыми используют и водорастворимые солевые модели на основе карбамида. Известно, что карбамидные модельные массы обеспечивают высокую прочность и позволяют сохранять форму моделей даже в случае повышения температуры в цехе. Тем не менее в силу экологических и производственных причин в настоящее время актуальным является переход на технологический процесс с применением готовых водных связующих на основе силиказоля. При этом возникают проблемы, связанные с изготовлением керамической оболочковой формы, из-за взаимодействия модельной массы и суспензии на водном связующем. В работе рассмотрена эффективность защитных покрытий на основе ремонтного воска, лака (АК 593) и лака с канифолью, нанесенных на водорастворимые модели на основе карбамида с добавками сульфата магния, нитрата калия, поливинилового спирта и диметилглиоксима. Степень взаимодействия оценивали по краевому углу смачивания и площади растекания водного связующего по поверхности образцов модельных масс с различными покрытиями. Было установлено, что все покрытия обеспечивают увеличение краевого угла смачивания и уменьшение площади растекания. Также с использованием ряда модельных составов с защитными покрытиями были получены керамические формы и отливки из никелевого жаропрочного сплава, для которых оценивали шероховатость и размерную точность. Было показано, что в случае нанесения покрытия на основе ремонтного воска защитные свойства недостаточны, что приводит к появлению трещин и наплывов в форме. В отливке это выражается в образовании механического пригара и значительном снижении размерной точности. В случае применения покрытий на основе лака и лака с канифолью каких-либо дефектов в форме или отливке не наблюдается, и именно эти покрытия можно рекомендовать в качестве защитных при использовании модельных составов на основе карбамида и суспензий на основе водных связующих.

Ключевые слова: литье по выплавляемым моделям, карбамидные модельные массы, растворимые солевые модели, водные связующие, защитные покрытия, краевой угол смачивания.

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Introduction

Among the currently applied foundry technologies for the production of large thin-walled castings from nickel superalloys, the investment casting method is predominantly used [1—2]. This method is based on obtaining a pattern of the future casting in a metallic die, most often from pattern compounds based on waxes [3]. However, wax-based pattern compounds have several drawbacks, prompting aerospace enterprises to continue using water-soluble salt patterns made of urea (CH_4N_2O) for the production of large shell castings [4—9]. The advantages of water-soluble salt patterns include high strength, hardness, low linear shrinkage, and low ash content [10; 11]. In addition to minimal shrinkage, the absence of softening and creep of the water-soluble salt pattern at elevated temperatures, which is typical for wax patterns, contributes to the

high dimensional accuracy of the resulting castings [12]. Urea is also used in the production of foamed materials by the investment casting method [13—15]. Alongside the conventional method of producing water-soluble salt patterns in dies, additive manufacturing technologies can also be employed [16].

Since salt-based patterns are water-soluble, it is necessary to prevent their dissolution during the formation of the ceramic shell [17]. One of the solutions to this problem is the use of slurries that do not contain chemically free water, such as those based on hydrolyzed ethyl silicate solution (ETS) [18]. However, several issues arise with the use of ETS in production. It is known that the curing of ETS-based slurry occurs in an acidic environment, while urea has a nearly neutral to alkaline pH [17]. As a result, contact between the pattern and the slurry leads to a deterioration of the inner mold layer quality and its weakening, which affects the quality of the castings [10]. At the same time, the use of ETS-based slurries in modern production is environmentally unfriendly due to the requirement for ammonia vapor to dry the investment shell layers, and the ETS-based mold manufacturing process cannot be automated [18].

Currently, slurries with colloidal silica binders are gradually replacing ETS-based slurries. Unlike the latter, they are non-flammable and cure by air drying, making them suitable for automated or robotic foundry production. However, the issue of dissolution and subsequent interaction with salt patterns is even more acute for colloidal silica binder-based slurries compared to hydrolyzed ethyl silicate-based slurries [17].

One way to reduce the intensity of interaction between salt patterns and shell molds is to modify the composition of the pattern compound, for example, by adding components that reduce the dissolution rate of the patterns. The main additives to urea in salt pattern compounds include magnesium sulfate, potassium nitrate, polyvinyl alcohol, ethylene vinyl acetate, and wax [19; 20]. In [12], the addition of dimethylglyoxime to the pattern compound was proposed to reduce hygroscopicity. The analysis of the wetting angle when applying an colloidal silica binder to pattern compounds containing magnesium sulfate, polyvinyl alcohol, potassium nitrate, and dimethylglyoxime showed that polyvinyl alcohol and dimethylglyoxime contribute to an increase in the wetting angle and a decrease in the interaction of the binder with the pattern compound [21].

A more preferable approach to preventing the interaction of the pattern compound with the colloidal silica binder slurry is to protect the salt-based patterns by applying water-resistant protective coatings to their surface. For example, urea-based patterns can be briefly immersed in liquid paraffin or another wax-based pattern compound to form a hydrophobic film on their surface [10]. According to [22], a protective coating formed by dissolving 3 wt. % of mixture of stearine and paraffin (1/1) in 100 mL of rubber solvent petrol provides the maximum wetting angle and the smallest spreading area of the slurry on the pattern surface. The use of such a protective coating allowed for the production of ceramic shells with low surface roughness. In [23], the use of bituminous and perchlorovinyl varnishes with mixed solvents is proposed as protective coatings. Depending on the degree of dilution, these varnishes allow for protective film thicknesses ranging from 4 to 10 μm, which does not significantly affect the dimensional accuracy of the resulting castings.

Another interesting method for reducing the interaction between urea-based patterns and colloidal silica binders is the application of a mixed mold-making technology. In studies [24; 25], it is proposed to apply a layer of slurry based on hydrolyzed ethyl silicate solution first, followed by a layer of slurry with a colloidal silica binder. This approach partially protects the water-soluble pattern from interacting with the colloidal silica binder; however, it introduces additional technological operations and does not fully resolve the issue associated with the use of hazardous components in production.

Thus, there are two main ways to limit the interaction between the pattern compound and the slurry: modifying the composition of the pattern compound and applying a hydrophobic coating to the pattern surface. Their combined study presents both scientific and practical interest. In this regard, the aim of this study was to investigate the interaction of various pattern compounds after applying protective coatings with colloidal silica binders.

Materials and methods

Five urea-based pattern compounds with additives of magnesium sulfate, polyvinyl alcohol, potassium nitrate, and dimethylglyoxime were selected as materials for the study (Table 1). Their primary properties having been previously investigated in [21]. It was shown that the additives of polyvinyl alcohol and dimethylglyoxime contribute to an increase in the wetting angle between the colloidal silica binder and the pattern compound [21].

Water-soluble patterns, featuring a sprue with attached parallelepiped elements measuring $10 \times 25 \times 25$ mm (Fig. 1), were produced using an aluminum alloy die.

Protective coatings based on repair wax and varnish AK 593 were examined (see Table 2). The varnish is based on a copolymer of methacrylic acid and methacrylic acid butyl ester. It is uncertain whether the slurry will remain on the pattern surface after applying varnish AK 593, as it may run off due to an excessively high wetting angle. Therefore, an additional coating option was considered, where a layer of rosin was applied over the varnish to improve surface wettability [26].

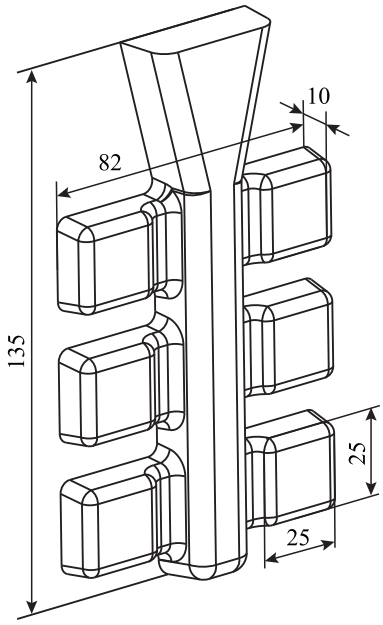

Fig. 1. Schematic representation of the water-soluble pattern

Рис. 1. Схематичное изображение растворимой модели

Three protective coatings were applied to the water-soluble patterns produced using each of the five pattern compound variants. The composition of the coatings and the sequence of their application are presented in Table 2. The coatings used include wax-based (#W), varnish AK 593-based (#V), and varnish with rosin (#V + R).

To assess the degree of interaction between the pattern compounds and the colloidal silica binder, as well as the protective effectiveness of the coatings, the wetting angle and the spreading area of the colloidal silica binder on the surface of the coated pattern compounds samples were determined. The lower the wetting angle and the larger the spreading area, the more intensive the interaction between the liquid and the substrate.

To determine the wetting angle using the sessile drop method and to measure the spreading area, three drops (0.08—0.09 mL) of the binder UltraCast One+ were

Table 1. **Composition of pattern compounds**

Таблица 1. Состав модельных масс

Compound	Components of the pattern compound and their content, wt. %				
	Urea CH_4N_2O	Magnesium sulfate $MgSO_4$	Polyvinyl alcohol $(C_2H_4O)_x$	Potassium nitrate KNO_3	Dimethylglyoxime $C_4H_8N_2O_2$
#1	98	2	–	–	–
#2	96	2	2	–	–
#3	90	–	–	10	–
#4	88	–	2	10	–
#5	88	–	–	10	2

Table 2. **Investigated protective coatings**

Таблица 2. Исследуемые защитные покрытия

Coating type	Composition and application sequence
#W	3 g of repair wax (a mixture of ceresin and petrolatum) in 100 mL of rubber solvent, applied in 2 layers using a sponge
#V	Varnish AK 593, applied in 1 layer by dipping
#V + R	Varnish AK 593, applied in 1 layer by dipping, followed by 5 g of pine rosin in 100 mL of ethanol, applied in 2 layers using a sponge

applied to the surface of pattern samples using a plastic pipette. The droplet behavior was recorded from the side using a SONY NEX EA50H video camera in macro mode with a Sony E PZ 18-200 mm F3.5—6.3 macro lens and Meike MK-S-AF3A macro rings. Frames were extracted from the video at the moment of droplet application and subsequently every 10 s during the first minute. From the second minute onwards, the frame extraction interval was increased to 1 min. The final frame corresponded to a 6-min hold time. After the experiment, a top-view image of the droplet was captured to determine the spreading area. For each pattern composition and type of coating, the experiment to determine the wetting angle and spreading area was conducted on three samples. The wetting angle and spreading area were measured from the extracted frames using the image analysis software ImageJ 1.52a (National Institutes of Health, USA).

To assess the quality of the castings obtained using the experimental pattern compounds and coatings, ceramic molds were produced and used to cast samples from the nickel superalloy. This alloy is widely used in domestic aerospace applications for the production of large shell castings employed in structural elements of combustion chambers. For this reason, the alloy is often cast into ceramic molds made using fused quartz as a filler.

Patterns produced from compounds #2 and #5 with all coating variants were used to fabricate ceramic molds. Various properties of colloidal silica binders, slurries, and ceramic samples obtained with their use were previously investigated in [27]. The ceramic mold fabrication technology and materials described below were selected based on the results of that study.

The slurry was prepared by mixing 5 L of binder with 10 kg of fine fused quartz with fraction 0.045 mm (supplied by LLC "Kefron", Yekaterinburg). The first and second ceramic layers were made using the binder UltraCast One+, while the subsequent layers were made using UltraCast Prime (both produced by LLC "Technopark", Moscow). After mixing, the slurry was left to stand for 24 h to ensure proper wetting of the micropowder with the binder and to remove air bubbles. Before use, the slurry was thoroughly mixed again. Its viscosity was determined using a DIN flow cup viscometer. If the viscosity did not meet the target values (60 s for the slurry used in the first and second layers, and 40 s for the third and subsequent layers), additional binder was added, the slurry was remixed, and viscosity measurements were repeated until the required values were achieved. The slurry was applied to the pattern by dipping, followed by stuccoing with fused quartz of varying particle sizes: 0.25—0.4 mm (for layers 1—2), 0.4—0.6 mm (for layers 3—4), and 0.5—1.0 mm (for layers 5—7), all supplied by LLC "Kefron". The final 8^{th} layer of slurry was applied without subsequent stuccoing (as a finishing layer). The first layer was dried in air at a temperature of 21—22 °C and relative humidity of 55—72 % for 2 h, while each subsequent layer was dried with air circulation at 21—22 °C and relative humidity of 64—85 % for 2 h.

The pattern was removed 24 h after applying the final layer by dissolving it in hot water at a temperature of 95 ± 5 °C.

The prepared molds were placed in a container and externally filled with a supporting refractory—coarse quartz sand. The molds were then subjected to burnout processing, during which they were heated to a temperature of 900 °C over 2 h, followed by a 4-hour holding period. Before pouring, the container with the molds was transferred to the pouring area. A ready-made nickel-based superalloy (wt. %: Ni — base; C — up to 0.08; Cr — up to 20.0; Mo — up to 5.0; Al — up to 1.5; Ti — up to 2.9; Nb — up to 2.8; Fe — up to 10), produced by VIAM (Moscow), was used as the charge. The melting was carried out in an induction furnace (SPE "RELEK", Ekaterinburg) using a periclase crucible (STC "Bakor", Shcherbinka). The mass of the melted alloy was 8 kg. The melt was protected by a covering flux of crushed silicate glass. The metal was poured into the ceramic molds at a temperature of approximately 1500 °C.

A distinctive feature of the investment casting method is the high dimensional accuracy and low surface roughness of the produced castings. Therefore, for the castings obtained using different pattern compounds and coatings, surface roughness and dimensional deviations were assessed. Additionally, shrinkage was calculated based on the measured dimensions, as it is an important parameter to consider when developing casting technologies.

The surface roughness of the cast samples was measured using an M300C profilometer (MarSurf, Germany). The average value was obtained from seven measurements.

To determine the dimensions of the cast samples and their linear shrinkage, laser scanning of the castings was performed using a handheld 3D scanner KScan Magic (ScanTech, China). The device's measurement accuracy was ± 20 μm. The cloud of points was processed using Geomagic Design X software (3D Systems, USA). The width of the casting (l_c) was measured at three points. Knowing the width of the mold cavity used to produce the pattern (l_p = 82 mm), the total linear shrinkage of the

pattern, ceramic mold, and alloy was calculated using the following equation: $\varepsilon = [(l_p - l_c)/l_p] \cdot 100$ %.

Since high-temperature alloys are prone to interaction with mold materials, the structure of the surface layers of the mold and castings was examined. The microstructure and phase composition of the surface layer of the castings and molds were analyzed using a scanning electron microscope (SEM) Vega SBH3 (Tescan, Czech Republic) equipped with an energy-dispersive microanalysis attachment (Oxford, UK), as well as an optical microscope (OM) Axio Observer. D1m (Carl Zeiss, Germany).

Results and discussion

Fig. 2 illustrates the dependence of the wetting angle (θ) on the holding time (τ) of the binder droplet on the surface of the tested pattern samples, both with and without protective coatings. In the absence of a coating (Fig. 2, *a*), the initial wetting angle upon application of the binder ranged from 20° to 40°, while after a 5-minute holding period, it decreased to θ = 2÷13° [21]. The presence of polyvinyl alcohol and dimethylglyoxime additives in the pattern composition resulted in higher values of θ.

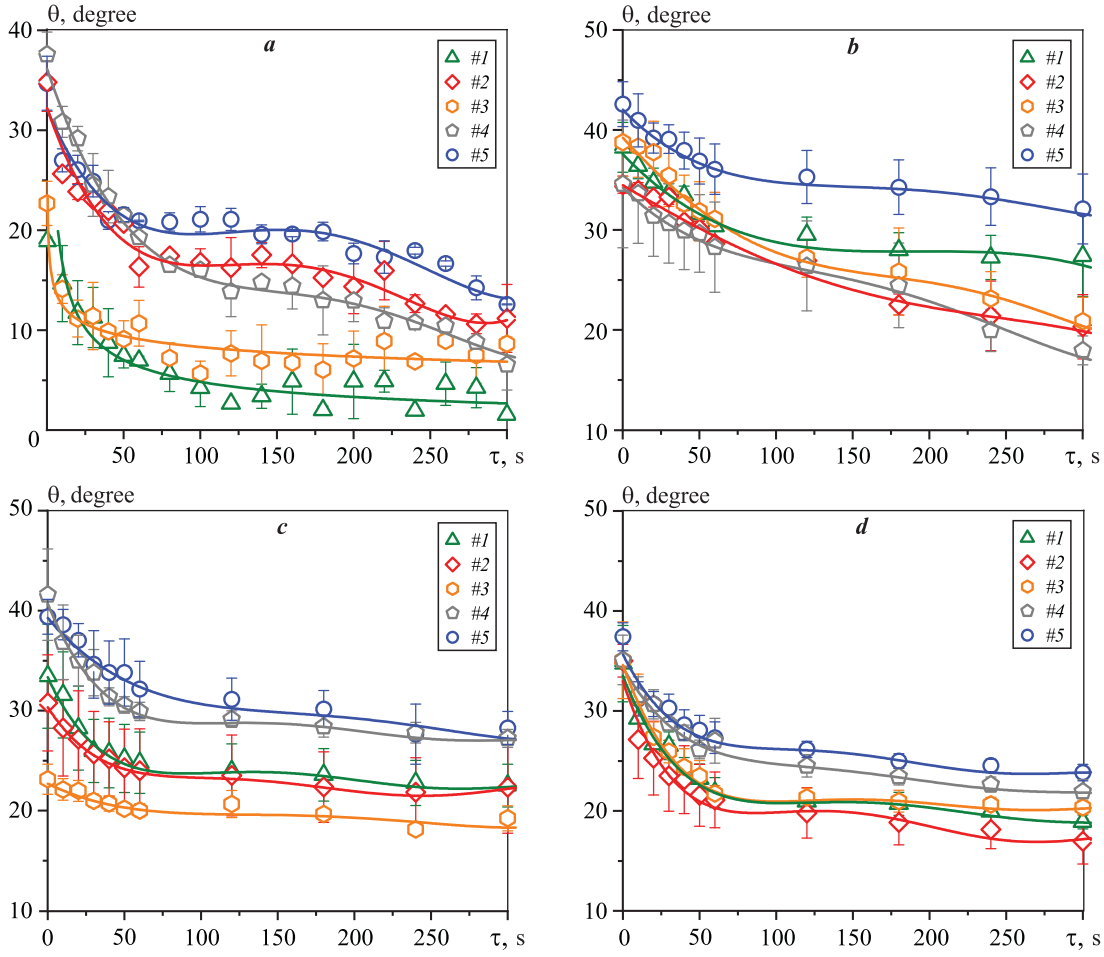

Fig. 2. Wetting angle with colloidal silica binder of the surface of pattern samples without coating (***a***) [21] and with coatings #Wax (***b***), #Varnish (***c***) and #Varnish + Rosin (***d***) (Table 2) as a function of time for pattern compounds *#1–#5*

#1 – 98 % urea + 2 % magnesium sulfate; *#2* – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#3 – 90 % urea + 10 % potassium nitrate; *#4* – 88 % urea + 10 % potassium nitrate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 2. Краевой угол смачивания (θ) водным связующим поверхности образцов модельных масс без покрытия (***a***) [21] и с покрытиями #В (***b***), #Л (***c***), #Л + К (***d***) (см. табл. 2) в зависимости от времени (τ) для модельных составов *#1–#5*

#1 – 98 % карбамида + 2 % сульфата магния; *#2* – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#3 – 90 % карбамида + 10 % нитрата калия; *#4* – 88 % карбамида + 10 % нитрата калия + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

Fig. 2, *b* presents the wetting angle values for the same pattern compositions but after the application of the wax-based protective coating. The initial values of θ immediately after applying the binder drop to samples coated with #W ranged from 35° to 43°, which is quite close to the values obtained for uncoated pattern compounds. However, it can be observed that during further holding of the droplet on the sample surface, the reduction in the wetting angle is not as significant compared to the case without a coating. Similar results were previously reported in [22], indicating that the intensity of interaction between the pattern compound and the binder decreases after the application of wax-based coatings. It is likely that when coating #W is applied and the solvent subsequently evaporates, the coating does not fully cover the sample surface, leading to localized dissolution of the pattern material. It should be noted that the composition of the pattern compound also affects the wetting angle, with the highest values of θ observed for the composition containing dimethylglyoxime. This additive reduces the hygroscopicity of the pattern compound and thus increases the wetting angle. Regarding other compositions, the values of θ after the application of coating #W range from 17° to 27°, which is fairly consistent across different formulations. In this case, no significant effect of polyvinyl alcohol on increasing the wetting angle was detected.

Fig. 2, *c* presents the wetting angle values after applying the varnish coating on the pattern samples. It can be observed that the initial values immediately after applying the binder drop are approximately 41° for compositions *#4* and *#5*, around 32° for compositions *#1* and *#2*, and about 23° for composition *#3*. Thus, the lowest wetting angle is observed for the pattern compound that contains only potassium nitrate. Compounds containing magnesium sulfate exhibit slightly higher θ values, and addition of polyvinyl alcohol having no significant impact on the wetting angle. The highest wetting angle is achieved for compounds containing potassium nitrate, polyvinyl alcohol, and dimethylglyoxime. The influence of polyvinyl alcohol on the wetting angle is attributed to its ability to reduce the hygroscopicity of the pattern compound. Previous studies have shown that samples made from pattern compound *#3* exhibit the highest surface roughness [21]. This is likely due to differences in the structure of the pattern compounds after solidification, which can lead to variations in the formation of the coating layer on their surface. In the case of varnish coating application, the wetting angle changes slightly over the holding period, decreasing by 4—15° after a 5-minute exposure. The slight reduction in wetting angle over time, as well as the influence of the pattern composition on its value, may indicate both coating integrity issues and interactions between the coating and the binder. The application of an additional rosin layer on top of the varnish coating (Fig. 2, *d*) results in an initial wetting angle that is independent of the pattern compound composition, with an average value of approximately 35°. Over time, the wetting angle values exhibit a trend similar to that observed for varnish coatings alone. This behavior can be attributed to the fact that, at the initial stage after the binder droplet is applied, the wetting angle is primarily determined by the interaction between the binder and the rosin layer. Over time, degradation of the rosin layer occurs, leading to interaction between the binder and the underlying varnish coating. On average, for all pattern compounds, the wetting angle values range from 17° to 23°.

In addition to the wetting angle, the spreading area (*S*) of the binder on the surface of the pattern samples can be used to evaluate the protective properties of the coatings. Fig. 3 presents the spreading area of the binder on the surfaces of the tested pattern compounds with different coating options after 2 h (once the binder had fully dried). The maximum spreading area of 170—180 mm^2 was observed for urea-based pattern compounds with polyvinyl alcohol additives (*#2* and *#4*) coated with the repair wax-based protective coating. A relatively high spreading area of $S = 140$ mm^2 was also obtained for the pattern compound containing magnesium sulfate (*#1*) with the wax-based coating. Considering the high values of error bars, it can be stated that for all pattern compounds coated with varnish or varnish with rosin, the spreading area of the binder ranged from 90 to 130 mm^2. Overall, the obtained *S* values confirm the conclusions drawn from the analysis of wetting angles, indicating that varnish-based coatings, unlike wax-based coatings, provide better protective properties. The wetting angle values and spreading areas are primarily determined by the protective properties of the coatings themselves rather than the pattern compositions.

It should also be noted that when examining the influence of various coatings on the spreading area of the binder across different pattern compounds, the minimum *S* values (along with the maximum wetting angle values θ) were observed for samples containing dimethylglyoxime (compound *#5*). As previously mentioned, this additive significantly reduces the hygroscopicity of the pattern compound.

Based on the results of wetting angle and spreading area measurements of the binders, two pattern

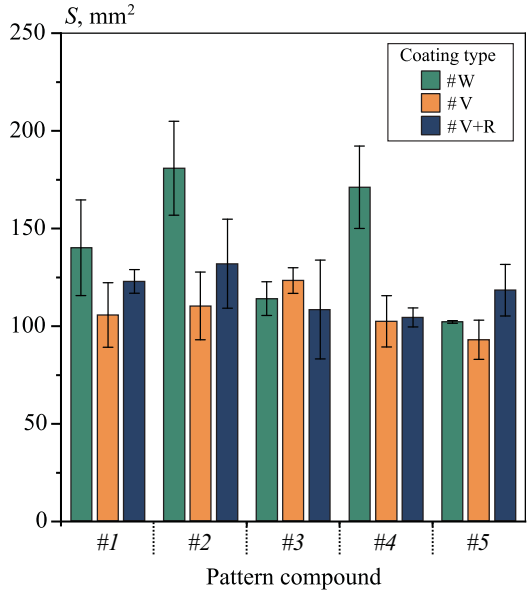

Fig. 3. Spreading area of the colloidal silica binder on the surface of pattern compound samples with coatings #W, #V and #V + R (see Table 2) after a 2-hour holding period for pattern compounds *#1–#5*

#1 – 98 % urea + 2 % magnesium sulfate;
#2 – 96% urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#3 – 90 % urea + 10 % potassium nitrate;
#4 – 88 % urea + 10 % potassium nitrate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 3. Площадь растекания водного связующего на поверхности образцов модельных масс с покрытиями #В, #Л и #Л + К (см. табл. 2) после выдержки 2 ч для модельных составов *#1–#5*

#1 – 98 % карбамида + 2 % сульфата магния;
#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#3 – 90 % карбамида + 10 % нитрата калия;
#4 – 88 % карбамида + 10 % нитрата калия + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

compounds were selected for further investigation: *#2* (96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol) and *#5* (88 % urea + 10 % potassium nitrate + + 2 % dimethylglyoxime). Fig. 4 presents photographs of ceramic molds produced using pattern compounds *#2* and *#5* with repair wax-based and varnish-based coatings at different stages of production.

When applying the first two ceramic layers to the pattern made from compound *#2* with the repair wax-based coating, minor sagging was observed (Fig. 4, *a*). The study of the wetting angle and spreading area of the binder on the pattern composition revealed that in cases of significant interaction between the binder and the pattern compound, simultaneous dissolution of the pattern occurs, forming indentations in the sample, while binder crystals grow at the pattern—binder in-

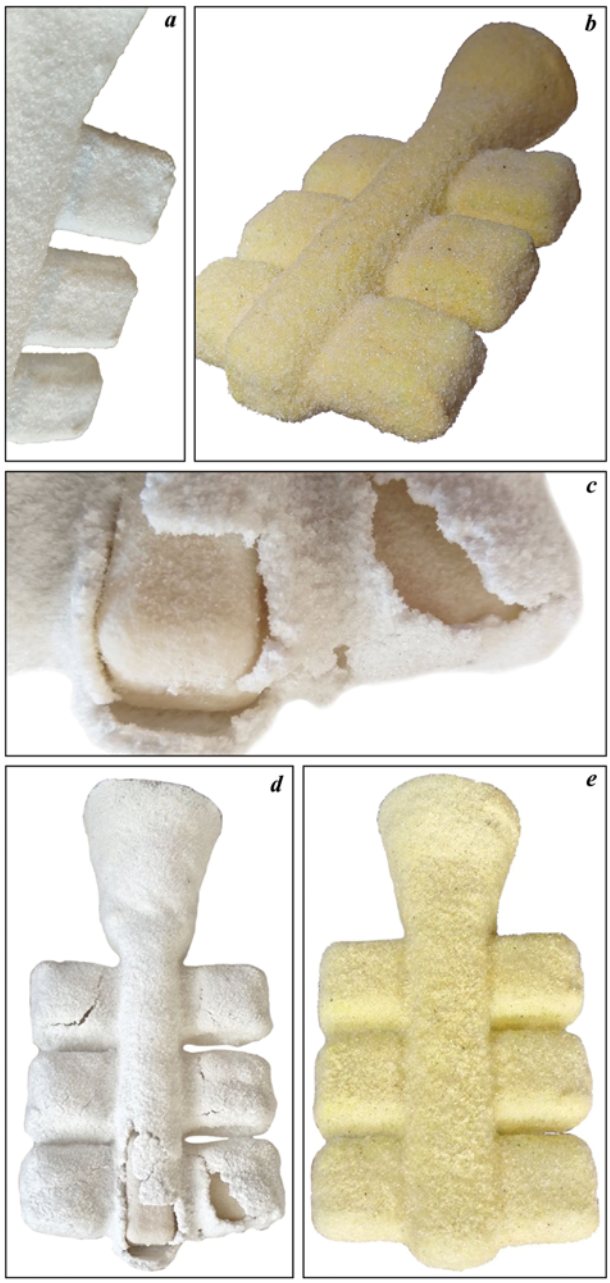

Fig. 4. Photographs of ceramic molds produced using various pattern compounds and protective coatings

a, ***b*** – pattern compound #2 (96 % urea + 2 % magnesium sulfate + + 2 % polyvinyl alcohol) repair wax-based coating #W, 2 layers (***a***) and 7 layers (***b***);
c–e – pattern compound #5 (88 % urea + 10 % potassium nitrate + + 2 % dimethylglyoxime) with a repair wax-based coating #W, 2 layers (***c***, ***d***), and varnish-based coating #V, 7 layers (***e***)

Рис. 4. Фотографии керамических форм, полученных с использованием различных модельных масс и защитных покрытий

a, ***b*** – модельный состав *#2* (96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта) с покрытием на основе ремонтного воска (#В) в 2 слоя (***a***) и 7 слоев (***b***);
c–e – модельный состав *#5* (88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима) с покрытием на основе ремонтного воска (#В) в 2 слоя (***c***, ***d***) и лака (#Л) в 7 слоев (***e***)

terface. Large crystals of the pattern compound extend beyond the original sample boundaries, likely contributing to sagging formation due to the insufficient protective properties of the coating. This partial dissolution of the pattern compound, accompanied by crystal growth, pushes the ceramic layer outward. Despite this issue, all seven ceramic layers were successfully applied to the pattern. The final appearance of the mold is shown in Fig. 4, *b*.

In the case of pattern compound *#5* containing dimethylglyoxime and coated with a repair wax-based protective layer (#W), the application of the first cera-

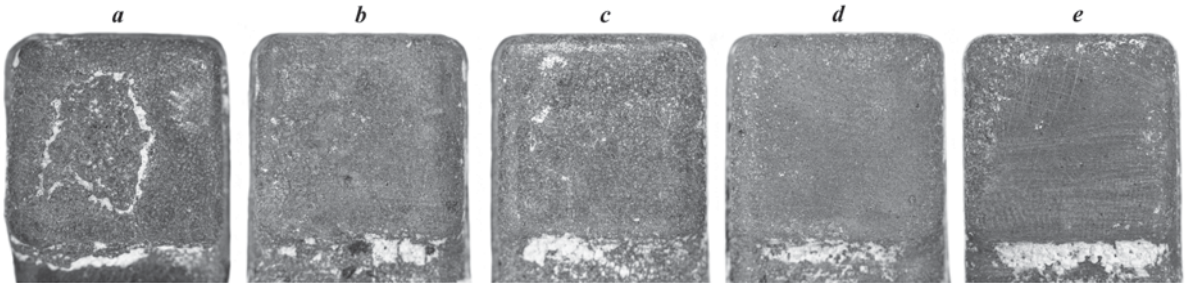

Fig. 5. Surface photographs of cast samples produced in ceramic molds using pattern compounds *#2* (***a***–***c***) and *#5* (***d***, ***e***) with protective coatings based on repair wax #W (***a***), varnish #V (***b***, ***d***), and varnish with rosin #V + R (***c***, ***e***)

#2 – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 5. Фотографии поверхности отлитых образцов, полученных в керамических формах с использованием модельных масс *#2* (***a***–***c***) и *#5* (***d***, **e**) с защитными покрытиями на основе ремонтного воска #В (***a***), лака #Л (***b***, ***d***) и лака с канифолью #Л + К (***c***, ***e***)

#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

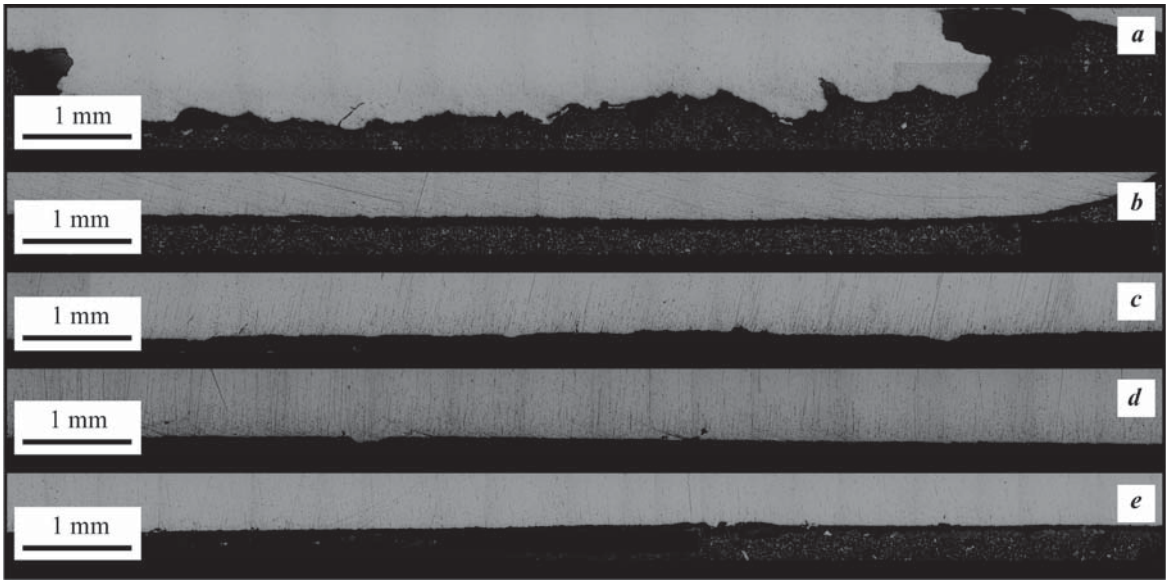

Fig. 6. Microstructure (OM) of cross-sectioned as-cast samples produced in ceramic molds using pattern compounds *#2* (***a***–***c***) and *#5* (***d***, ***e***) with protective coatings based on repair wax #W (***a***), Varnish #V (***b***, ***d***), and Varnish with Rosin #V + R (***c***, ***e***)

#2 – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 6. Микроструктура (ОМ) шлифов (поперечное сечение) отлитых образцов, полученных в керамических формах с использованием модельных масс *#2* (***a***–***c***) и *#5* (***d***, ***e***) с защитными покрытиями на основе ремонтного воска #В (***a***), лака #Л (***b***, ***d***) и лака с канифолью #Л + К (***c***, ***e***)

#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

mic layers resulted in cracking and destruction. Figs. 4, *c* and 4, *d* clearly show that under the cracked ceramic layer, the pattern surface exhibits significant roughness, which was not present before the ceramic application—indicating interaction between the pattern and the binder. The causes of destruction in this case are similar to those previously described for the pattern compound *#2* with the repair wax-based coating. Regarding varnish-based coatings (#V) and varnish with rosin (#V + R), no sagging was observed in any case, and high-quality ceramic molds were successfully obtained for both pattern compounds *#2* and *#5*. An example of a high-quality mold produced using the pattern compound with dimethylglyoxime (*#5*) and coated with varnish (#V) is shown in Fig. 4, *e*.

Fig. 5 presents photographs of cast samples made of nickel superalloy poured into ceramic molds produced using pattern compounds *#2* and *#5* with protective coatings based on repair wax, varnish, and varnish with rosin. As previously mentioned, it was not possible to produce a mold using pattern compound *#5* containing dimethylglyoxime and a repair wax-based coating (#W). Although the mold produced with pattern compound *#2*, containing magnesium sulfate and polyvinyl alcohol, and coated with repair wax did not fail structurally, the surface of the cast samples exhibited penetration defects (Fig. 5, *a*). These defects formed due to the destruction of the ceramic surface layer and infiltration of the molten alloy into the damaged layer. Previous observations (see Fig. 4, *a*) showed the presence of localized bulging of the shell during mold layer formation. Regarding the samples coated with varnish (#V) and varnish with rosin (#V + R) (Fig. 5, *b*—*e*), it can be observed that all sample surfaces are free of defects, confirming the high protective properties of the varnish coatings.

Fig. 6 shows cross-sectional micrographs of the samples presented in Fig. 5. It can be observed that the thickness of the burn-on defect in the sample produced using pattern compound *#2*, coated with a repair wax-based protective layer (Fig. 6, *a*), is approximately 1 mm. The other samples, in which varnish-based coatings (#V) and varnish with rosin (#V + R) were used on the pattern compounds (Fig. 6, *b*—*e*), exhibit a minimal number of surface defects.

Fig. 7 presents the surface roughness values of the cast samples produced in ceramic molds using pattern compounds with magnesium sulfate and polyvinyl alcohol additives (*#2*), as well as potassium nitrate and dimethylglyoxime additives (*#5*), coated with protective coatings based on repair wax, varnish, and varnish with rosin. The minimum surface roughness value

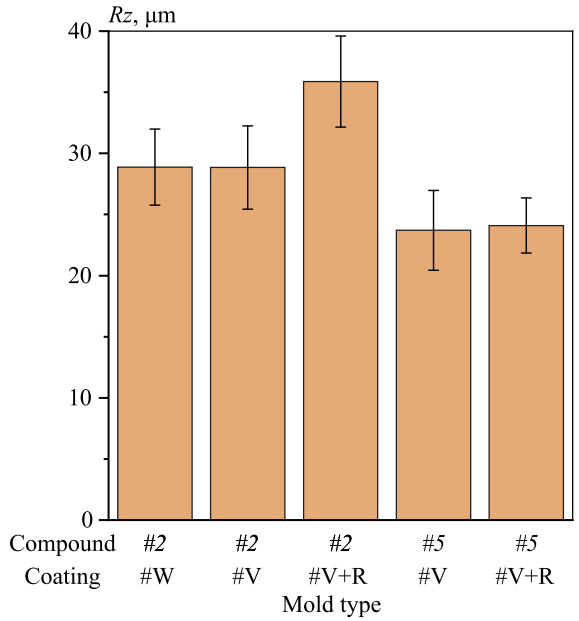

Fig. 7. Surface roughness of as-cast samples produced in ceramic molds using pattern compounds *#2* and *#5* with protective coatings based on repair wax (#W), varnish (#V) and varnish with rosin (#V + R)

#2 – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 7. Шероховатость поверхности отлитых образцов, полученных в керамических формах с использованием модельных масс *#2* и *#5* с защитными покрытиями на основе ремонтного воска (#В), лака (#Л) и лака с канифолью (#Л + К)

#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

(Rz = 24 μm) was obtained for the cast samples produced using pattern compound *#5* (with potassium nitrate and dimethylglyoxime) and the varnish-based coating. The use of the same coatings on pattern compound *#2*, containing magnesium sulfate and polyvinyl alcohol, resulted in slightly higher roughness values (Rz = 29÷36 μm). The cast sample produced using pattern compound *#2* with a repair wax-based coating demonstrated similar roughness, with Rz = 29 μm. In this case, the surface roughness was measured in areas free of burn-on defects. Thus, all protective coatings provide comparable surface roughness values in the range of Rz = 24÷36 μm. This is likely due to the fact that after pattern removal and mold burnout, no traces of the coating remain, and the surface roughness is entirely determined by the characteristics of the slurry and stucco used in ceramic mold production.

Fig. 8, *a* presents the SEM microstructure of a sample cast in a ceramic mold produced using pattern

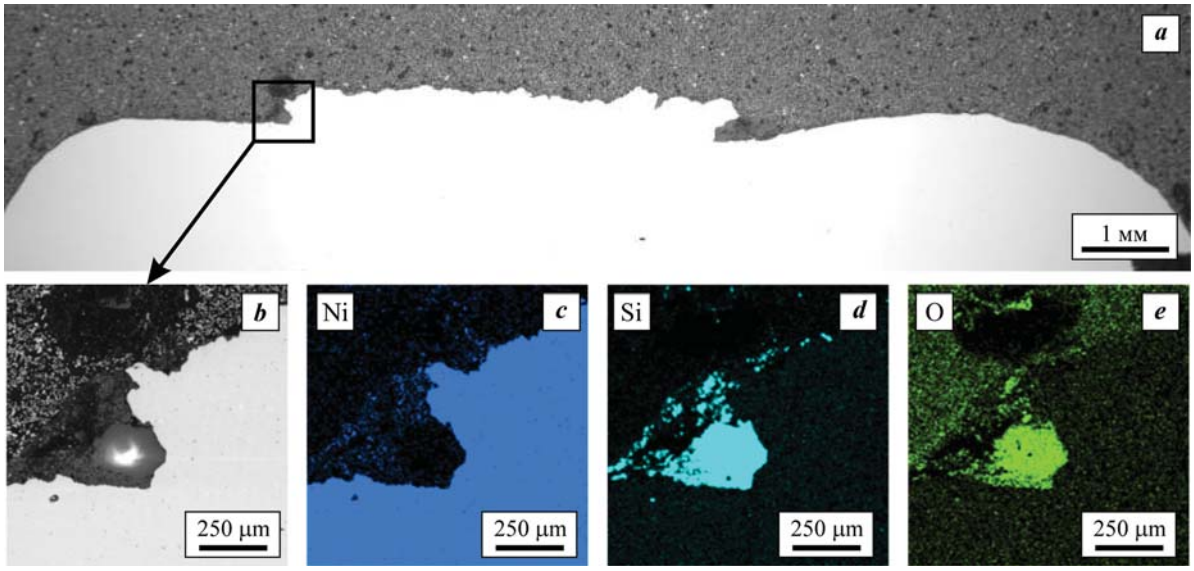

Fig. 8. Microstructure of the sample cast in a ceramic mold produced using pattern compound *#2* with a repair wax-based coating (#W) (***a***), magnified microstructure area (***b***), and elemental distribution maps of Ni (***c***), Si (***d***), and O (***e***)

Pattern composition *#2* – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol

Рис. 8. Микроструктура образца, отлитого в керамическую форму, полученную с использованием модельной массы *#2* с покрытием на основе ремонтного воска (#B) (***a***), увеличенный участок микроструктуры (***b***) и карты распределения Ni (***c***), Si (***d***), O (***e***)

Модельный состав *#2* – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта

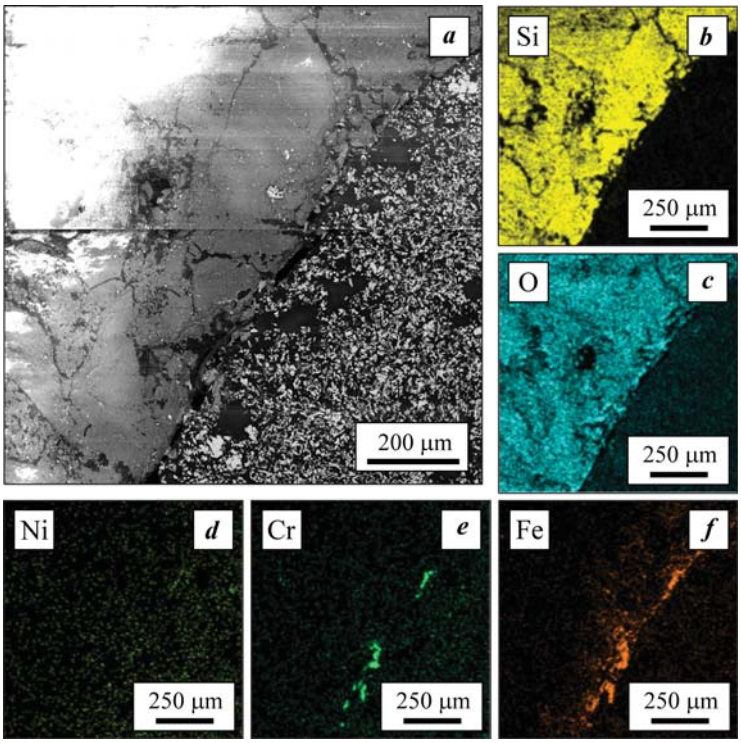

Fig. 9. Microstructure of the contact layer of the ceramic mold produced using pattern compound *#5* coated with varnish (#V) after alloy pouring (***a***) and EDS maps of Si (***b***), O (***c***), Ni (***d***), Cr (***e***), Fe (***f***)

Pattern composition *#5* – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 9. Микроструктура контактного слоя керамической формы, полученной с использованием модельной массы *#5* с покрытием на основе лака (#Л), после заливки сплава (***a***) и карты распределения Si (***b***), O (***c***), Ni (***d***), Cr (***e***), Fe (***f***)

Модельный состав *#5* – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

compound *#2* (96 % urea + 2 % magnesium sulfate + + 2 % polyvinyl alcohol) with a repair wax-based coating. It can be observed that at the transition zone between the high-quality surface and the burn-on defect, undercuts are present. The microstructure of such an undercut at a higher magnification, along with the EDS maps for this area, is shown in Fig. 8, *b*—*e*. The results of EDS analysis reveal the presence of particles containing Si and O within the undercuts. Thus, it can be assumed that these are stucco particles from the ceramic mold that detached from the mold during the casting removal process. This confirms the assumption that the previously observed sagging during mold formation occurs due to the delamination of ceramic layers.

For chemically active melts, it is essential to evaluate their interaction with mold materials. Fig. 9, *a* presents the microstructure of the contact layer in a cross-section of the ceramic mold surface produced using pattern compound *#5* (88 % urea + + 10 % potassium nitrate + 2 % dimethylglyoxime) with a varnish-based coating (#V). According to the elemental distribution maps of Si and O (Fig. 9, *b* and *c*), the ceramic mold has a well-defined boundary. A layer enriched with Cr and Fe (Fig. 9, *e* and *f*), with a thickness of no more than 50 μm, is present on its surface. It should be noted that this layer is not continuous, which may be attributed to its insufficient strength and partial destruction during the preparation of the metallographic specimen. It is known that chromium has high vapor elasticity and, upon pouring, deposits on the mold surface while interacting with atmospheric oxygen. Thus, no signs of interaction between the melt and the mold have been detected. As previously mentioned, the applied coatings are completely removed during the burnout of the ceramic mold.

The results of laser scanning of the cast samples produced in ceramic molds using pattern compounds with magnesium sulfate and polyvinyl alcohol additives (*#2*), as well as potassium nitrate and dimethylglyoxime additives (*#5*), coated with protective coatings based on repair wax, varnish, and varnish with rosin, are presented in Fig. 10. It is evident that when using the repair wax-based coating (#W), the dimensional deviations in the positive direction reach up to 2.5 mm (Fig. 10, *a*). These deviations are attributed to the formation of sagging, which resulted from changes in the mold geometry during the application of the initial ceramic layers.

Unfortunately, it is not possible to determine the effect of the coating on dimensional accuracy, as producing a mold using a urea-based pattern compound and a colloidal silica binder slurry without a protective coating is not feasible. However, considering the thin protective layer (less than 100 μm), it is unlikely that the coating significantly affects the dimensional accuracy of the castings.

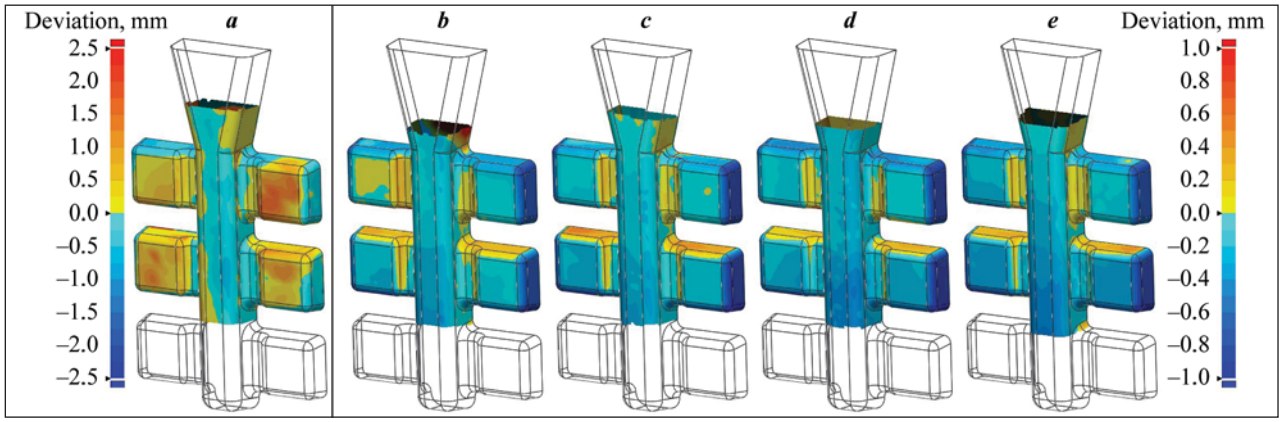

Fig. 10. Dimensional deviations of as-cast samples produced in ceramic molds using pattern compounds *#2* (***a***—***c***) and *#5* (***d***, ***e***) with coatings based on repair wax #W (***a***), varnish #V (***b***, ***d***), and varnish with rosin #V + R (***c***, ***e***)

#2 – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 10. Отклонения размеров отлитых образцов, полученных в керамических формах с использованием модельных масс *#2* (***a***—***c***) и *#5* (***d***, ***e***) с защитными покрытиями на основе ремонтного воска #В (***a***), лака #Л (***b***, ***d***) и лака с канифолью #Л + К (***c***, ***e***)

#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

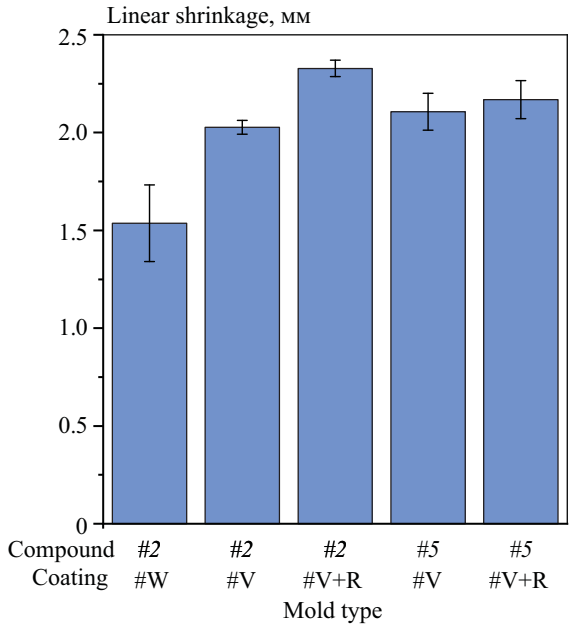

Fig. 11. Linear shrinkage of as-cast samples produced in ceramic molds using pattern compounds *#2* and *#5* with protective coatings based on repair wax (#W), varnish (#V), and varnish with rosin (#V + R)

#2 – 96 % urea + 2 % magnesium sulfate + 2 % polyvinyl alcohol;
#5 – 88 % urea + 10 % potassium nitrate + 2 % dimethylglyoxime

Рис. 11. Линейная усадка отлитых образцов, полученных в керамических формах с использованием модельных масс *#2* и *#5* с защитными покрытиями на основе ремонтного воска (#В), лака (#Л) и лака с канифолью (#Л + К)

#2 – 96 % карбамида + 2 % сульфата магния + 2 % поливинилового спирта;
#5 – 88 % карбамида + 10 % нитрата калия + 2 % диметилглиоксима

The results of laser scanning of the cast samples were used to determine their linear shrinkage. Fig. 11 presents the results of the linear shrinkage measurements for the cast samples produced in ceramic molds using pattern compounds with magnesium sulfate and polyvinyl alcohol additives (*#2*), as well as potassium nitrate and dimethylglyoxime additives (*#5*), coated with protective coatings based on repair wax, varnish, and varnish with rosin. When using the repair wax-based coating (#W), the linear shrinkage was only 1.5 %, whereas for the varnish-based coatings (#V and #V + R), the shrinkage ranged from 2.0 % to 2.3 %. The low shrinkage observed with the repair wax coating (#W) is attributed to delamination and sagging that occurred during the application of the initial mold layers. In [21], it was shown that the linear shrinkage of pattern compounds *#2* and *#5* is 0.55 % and 0.3 %, respectively. However, it is challenging to observe a significant difference in the total shrinkage of the cast samples depending on the applied pattern compound composition. Overall, it can be concluded that the total shrinkage of the ceramic mold and the alloy itself is, on average, ~1.7 %, which is almost identical to the shrinkage characteristic of ceramic molds produced using hydrolyzed ethyl silicate solution, as well as colloidal silica binders, but with wax patterns.

Large-sized castings of the “Outer Casing” and “Inner Casing” components made of the heat-resistant nickel alloy were produced under industrial conditions at PJSC “UEC-Kuznetsov” (Samara, Russia). Water-soluble patterns made of a urea-based pattern compound with a hydrophobic protective coating based on varnish with rosin were used for the production of the castings. The maximum overall dimension of the produced castings reached 1136 mm. The use of the protective coating #V + R made it possible to produce shell molds using a slurry prepared with a colloidal silica binder, achieving results comparable to those obtained using the traditional technology based on hydrolyzed ethyl silicate as the binder. The resulting castings met the technical requirements in terms of dimensional accuracy and mechanical properties.

Conclusions

1. The wetting angle of the colloidal silica binder on the surface of the tested pattern compounds increased from 3—15° to 20—30° when applying protective coatings based on repair wax, varnish, and varnish with rosin. However, no significant influence of the coating type on the wetting angle was observed. In all cases (with and without coatings), the highest θ value was obtained for the pattern compounds containing dimethylglyoxime.

2. The minimum spreading area of 90—130 mm^2, corresponding to minimal interaction between the binder and the pattern compound, was achieved using varnish-based coatings, as well as varnish with rosin. In the case of repair wax-based coatings, the spreading area was 170—180 mm^2. The modification of the pattern compound with various additives, in all likelihood, does not have a significant impact on the spreading area of the colloidal silica binder on the surface of the pattern compound samples.

3. The use of repair wax-based protective coatings in the production of ceramic molds with colloidal silica binder resulted in sagging and cracking, which prevented the successful fabrication of molds with pattern compound *#5* containing dimethylglyoxime. The mold produced using pattern compound *#2* (with magnesium sulfate and polyvinyl alcohol additives)

was successfully fabricated; however, partial destruction of the inner mold surface and sagging were observed. Molds coated with varnish and varnish with rosin exhibited no visible defects or manufacturing issues.

4. The castings produced in the ceramic molds with varnish and varnish with rosin as protective coatings on the patterns exhibited no visible defects and had a surface roughness of $Rz = 25 \div 35$ μm. The casting obtained in the mold with a repair wax-based protective coating showed mechanical penetration defects, with ceramic mold particles detected within the casting. Additionally, it exhibited significant dimensional deviations due to sagging caused by the degradation of the inner mold surface.

5. The linear shrinkage of the castings produced using molds with varnish and varnish with rosin coatings on the patterns was higher compared to those with repair wax-based coatings. This was also associated with the formation of bubbles and sagging on the pattern surface during the mold layer formation process.

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