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Synthesis of the new lost foam refractory coatings based on talc

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Abstract: This paper is focused on the preparation procedures, using a talc-based filler, to improve the rheologic properties of the lost foam refractory coatings. Talc, with grain size of 40 µm, was mechanically activated in a vibration mill over the following times: 10, 20 and 30 min. Depending on the time required for the mechanical activation, the change in the filler grain size and shape was analysed along their effects on the talc-filled lost foam coating dispersion ability and stability. In order to characterize the filler, the following methods were used: X-ray diffraction, scanning electron microscopy and optic microscopy. The coating composition was tuned by choosing the suitable grain size and shape factor of the activated filler. In addition, different coating components (additives, solvent) were applied to alter the coating generation procedure. It was shown that the application of this type of the lost foam refractory coatings – water-based coatings – have a positive influence on quality of the aluminium alloy castings, which contributes to reducing the cost of cleaning and processing of the castings. Also, alcohol-based refractory coatings with talc-based activated fillers were tested and used to have the castings produced in the sand moulds.

Keywords: mechanical activation; talc-based fillers; rheologic properties of coating suspensions.

INTRODUCTION

Lost foam process is a new method for production of high quality low-cost castings. The technological possibilities for the lost foam casting process are examined and basic laws for the influence of numerous parameters on the process

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flow and castings quality are determined. The process includes a large number of insufficiently examined phenomena in connection with physicochemical and thermodynamic changes in the system: evaporative pattern–refractory coating–liquid metal–sand.^{1–3} The technological parameters important for the process to unfold and obtain high quality castings are: pattern density, granulometric contents of moulding sand (mould permeability), thermophysical features of coating materials, pouring temperature and pouring system.^{4,5}

Unlike sand mould casting, where liquid metal flows into the “mould cavity”, with the lost foam process, the patterns and pouring systems made of polymers are retained in the mould until a liquid metal has flown in (“full mould casting”).^{1,6–9} In contact with a liquid metal, polymer patterns degrade, evaporate and simultaneously the castings solidification takes place. As a consequence of the degradation and the evaporation of the polymer pattern, a large amount of liquid and gas products is released. These products are a frequent cause for castings’ defects. To obtain quality castings, it is necessary to apply highly permeable lost foam refractory coatings.^{10–17}

Basic role of the refractory coating is to form an efficient refractory barrier between the sand substrate and the liquid metal flow during the casting phase, and to control the solidification and the forming of the castings.¹¹ This provides a smooth and clean surface of castings, with no adhered sand or defects due to metal penetration into the mould (*e.g.*, rough surface, lumps, dents). The application of higher quality refractory coatings significantly can provide either reduction or elimination of the expensive foundry cleaning and mechanical operations, thus directly reducing production costs of a castings. The coating properties are strictly defined by standards; therefore, it is very important to make the right choice of coating, as well as its preparation and application procedures in the foundry working conditions.^{18,19}

Contemporary coatings, depending on their use, represent thermophysical mixtures of ceramic materials in a suspending medium which contains suspension bonding agents. The coating composition analyses show that the coating consists of a number of components of which the most important are: refractory filler, bonding agent, suspension stability agent, liquid carrier and the solvent.^{1,15,20–23}

Complexity of the casting solidification process is influenced by the insulation effect, governed by the lost foam refractory coatings. At the stage of filling the mould, when degradation and evaporation of polymer pattern take place, the refractory coating causes a temperature drop of the liquid metal. As soon as the mould is filled with liquid metal, *i.e.*, while polymer pattern is evaporating, the refractory coating, through the insulation effect, causes the decrease in the castings’ cooling and solidification rate. The endothermic degradation of the polymer pattern causes subcooling of the liquid metal and affects the formation of a fine structure of the

castings. If subcooling is considerably fast, a fine tiny-grained structure is formed on castings.^{1,7,13} The pattern degradation and the evaporation rate depend on polymer density, casting temperature, lost foam refractory coatings' permeability and sand mould permeability. Here described research paid particular attention to these factors.

For the development of the lost foam refractory coatings with the controlled rheologic properties, the influence of the mechanical activation process on the talc-based filler's properties was examined. The test preferentially referred to the change in filler grain size and shape, and afterwards to the dispersion quality and the stability of the coating suspension.^{24–30}

The composition and the production procedures for the lost foam refractory coatings, with a mechanically activated talc-based filler, were planned along with the research activities, and refer to the dependence of the castings structure and properties on the casting process parameters.

Talc is a magnesium hydrosilicate with the general formula $H_2Mg_2(SiO_3)_4$ or $Mg_6(OH)_4(Si_8O_{20})$ with the additions of Al_2O_3 , FeO , NiO and CaO . Talc was chosen to be the filler for lost foam coatings, owing to its following properties: Mosh hardness 1; density 2700–2800 kg/cm^3 ; low heat conductivity coefficient, $\lambda = 3.5–4.0$ $W/(m\ K)$; low linear thermic expansion coefficient, $\alpha = 2.7 \times 10^{-6}/^\circ C$ (20–1000); high adherence and cladding ability to surfaces; high melting point, 1400–1550 $^\circ C$; high inertia, *i.e.*, resistance to acids, alkalines and heat.^{16,31,32}

The influence of following parameters on the casting process is analyzed: casting temperature, mould permeability and the design of the pattern and pouring systems. The subject of research was the correlation between these parameters and the polymer pattern density, as well as the type and thickness of refractory coating layers.

EXPERIMENTAL

Talc-based filler synthesis

Talc used as filler was obtained with the use of combined preparation procedures from mineral raw materials; talc excavated at the site Studenica, Serbia, was crushed and leached. The particular attention was paid to the talc purification procedure, as well as to reduction of the Fe_2O_3 and CaO content as follows. The initial talc sample was of a heterogeneous chemical composition, Table I.

TABLE I. The initial composition of talc used for production of the filler T

| Compound | SiO_2 | MgO | Al_2O_3 | Fe_2O_3 | CaO | Na_2O+K_2O | LoI |
|-----------------|---------|-------|-----------|-----------|-------|--------------|------|
| Content, mass % | 61.50 | 29.45 | 1.78 | 2.84 | 2.50 | 1.90 | 8.50 |

Talc purification was performed by means of leaching to reduce the Fe_2O_3 and CaO content. Talc was leached by hydrochloric acid (15 wt. %). The leaching was performed at a constant temperature ($t = 80$ $^\circ C$), in a three-neck bottle during 3.5 h of reflux. Calcite (green) precipitated as bottom solid, whereas talc concentrate (white) appeared as an upper layer. Car-

bon dioxide (CO₂) was produced during the leaching process, which lifts up the fine talc particles concentrate, lighter than calcite particles remaining at the bottom. Next, warm talc concentrate was rinsed by water, then filtered and dried. The same process was applied on calcite and intermediate product (talc–chlorite) which remain as final tailing. The talc samples produced were ground in a ceramic ball mill down to the limit grain size of 100 % – 40 µm. It was the initial grain size of the filler T; then, it was subjected to the mechanical activation process in a vibration mill over different times (min): 10, 20 and 30, with the filler sample codes: T₁, T₂ and T₃, respectively; Table II reports the grain size and shape data.

TABLE II. Talc-based refractory fillers after mechanical activation

| Sample | Mechanical activation time, min | Grain size, µm | Grain shape factor |
|----------------|---------------------------------|----------------|--------------------|
| T ₁ | 10 | 100%- 30 | 0.62 |
| T ₂ | 20 | 100% - 22 | 0.69 |
| T ₃ | 30 | 100% - 10 | 0.72 |

Characterization of filler T

To identify the composition and structure of the filler T, the method of X-ray diffraction analysis was applied. The XR diffractometer, model PW-1710 (Philips Analytical, Almelo, the Netherlands) was used; it involved a curved graphite mono-chromate meter and a scintillation counter. The intensities of the diffracted CuK α of the X-ray radiation (wavelength 1.54178 Å) were measured at room temperature with the 2θ intervals of 0.02° over the time of 1 s, within the 2θ range from 4 to 70°. The X-ray tube was under the voltage of 40 kV and the current of 30 mA, while the primary and the diffracted rays' slots were 1° and 0.1 mm.

The morphological and the quantitative chemical analysis was carried out by means of the scanning electronic microscope “Jeol”– model JSM 6610 LV (Jeol, Tokyo, Japan). In order to improve conductivity, the sample was coated with gold powder.

Refractory coating synthesis with activated fillers T₁, T₂ and T₃

The talc samples produced after mechanical activation were used to produce both water and alcohol-based refractory coatings, Table III.

TABLE III. Talc-based refractory coatings composition

| Component | Refractory coating | | |
|-----------------------------|---|---|--|
| | Type I | Type II | Type III |
| Refractory filler | T ₂ (82–85 %) | T ₂ (72–75 %) + T ₃ (9–11 %) | T ₂ (83–86 %) |
| Bonding agent | Bentonite 4–4.5 %; Bindal H 4–4.5 % | Bentonite 4.5–5 %; Bindal H 4.5–5 % | (C ₂₀ H ₃₀ O ₂) 3.5–4.5 % Dextrin 0.5–1 % |
| Additive | Suspension maintenance agent: carboxymethyl cellulose (CMC) 1–1.5 % | Suspension maintenance agent: carboxymethyl cellulose (CMC) 1.5–2 % | Bentone 25 1.5–2.5 %; phenolformaldehyde resins, 0.2–0.5 % |
| Solvent | Water | Water | Isopropylalcohol (C ₃ H ₈ O) |
| Density, kg m ⁻³ | 2000 | 2000 | 2000 |

The paper investigates refractory coatings based on activated talc filler for casting aluminium alloy castings in sand moulds. The sand moulds and cores were coated with a Type III

refractory coating. Also, Type I and Type II coatings for casting aluminium alloy castings by the lost foam process were investigated. These refractory coatings were used to coat polymer models. The coatings were tested in accordance with the standards for this type of refractory materials,^{18,19} and with respect to our previous works in this field,^{11,16,22,25,32} which also report the results of testing of the influence of mechanical activation on the structure and properties of talc.²⁶⁻³⁰ The sedimentation stability of the produced refractory coatings was tested at a temperature of 22 °C; thickness of the wet coating film layers was: 0.3, 0.6 and 0.9 µm. Refractory coatings were applied to polymer patterns through immersing and pouring procedures, while sand moulds were coated by means of brushes.

In order to obtain a homogeneous suspension of the coating, during application, light mixing was performed at a speed of 1 rpm. The water-based lost foam refractory coatings were air dried during 24 h. The alcohol-based coatings were burned to get dry.

Characterization of lost foam refractory coatings

In order to elucidate the filler and the binding agent's distributions, the lost foam refractory coating suspension was analysed on the polarized microscope for the transmitted light JENAPOL, manufactured by Carl Zeiss-Jena, Germany, with Microphoto System Studio PCTV; Pinnacle System, Mountain View, CA, USA. Measurements of the filler grain size and shape were carried out on 4000 grains, while the analysis was conducted by means of the software application package Ozaria 2.5 (interval 0–1), Vaga Lab, Belgrade, Serbia. The shape factor was: for 0 – corresponding to the position of the needle, for 1 – corresponding 158 to the circle. There is the following division according to the grain shape factor: from 0.0–0.2 159 – angular; from 0.2–0.4 – sub-angular; from 0.4–0.6 – sub-rounded; from 0.6–0.8 rounded and 160 from 0.8–1.0 – well rounded grain shape.

Properties of the refractory coatings obtained were examined in accordance with the standards.^{18,19} The test procedure is described in a previous paper.²³ The sedimentation stability of the suspension of the obtained coatings was tested by keeping the prepared coating samples for 24 h in a cylindrical vessel with a plug, with a volume of 10^{-4} m³ and a height of 0.28 m.

The test result is expressed in percentages: the volume in mL of transparent layer read is equal to the precipitation of solid particles in percent. Determination of the penetration of coatings into sand moulds was investigated using tubes made of moulding mixture. After applying the coating and drying, the tubes were broken and the depth of penetration of the coating in mm was measured at the fracture. The refractoriness of the filler was determined by a comparative test with bodies whose softening points at high temperatures are known. The symbol SK (Seeger's pyramid) and the number that represents the code for the temperature.¹¹ Testing of the coating properties was performed by applying the prepared coatings on the surfaces of the test bodies made of moulded sand mixture and polystyrene. Coatings created in the described way should be easy to apply, adhere well to the surface, are not susceptible to leakage and creation of drops, bubbles, dry easily, without cracking and rubbing the dried layers of the coating.

To assess the quality of the obtained coatings, test castings of plate shape of dimensions 0.2 m×0.05 m×0.02 m were cast by casting in sand moulds and by the lost foam process. The casting alloy was AlSi12CuMg. Prior to casting, liquid casting was prepared by refining, degassing and modification processes. The refining and degassing process refers to the treatment of liquid metal with salts based on sodium and potassium chloride in order to remove impurities, inclusions and slag, as well as dissolved gases. To obtain a finer structure and better mechanical properties of castings, liquid metal is treated with structure grinding agents such as sodium, strontium, antimony, and phosphorus before pouring into moulds. Sodium was used in the experiment as a cheap modifier. The casting temperature was 778 °C. Sand moulds

are made of a moulding mixture based on quartz sand with a grain size of 0.17 mm, with 3 % bentonite and 0.15 % dextrin. The moulds for the lost foam process were made of dry quartz sand with a grain size of 0.26 mm. The polymer models in the experiment were made of polystyrene with a density of 19 kg/m³.

RESULTS AND DISCUSSION

Talc-based filler properties

Composition of the sample talc filler T, which was, after mechanical activation, used for production of refractory coatings, is shown in the Table I.

Fig. 1 shows XRD pattern of the talc samples with the dominant presence of talc in the initial sample. Fig. 2 shows SEM microphotograph of the initial talc sample before the mechanical activation. It is seen that the mineral was exclusively present in the proper foliar aggregates.

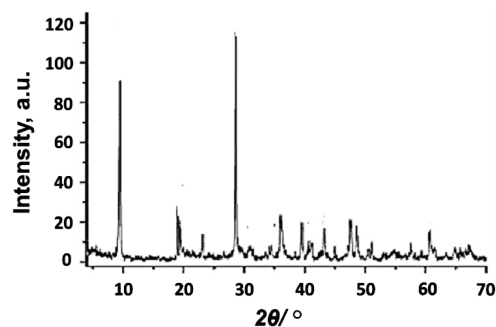


Fig. 1. XRD of talc samples before activation.

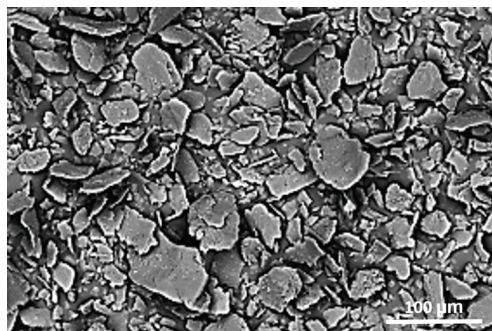


Fig. 2. SEM microphotograph of talc samples before activation.

Figs. 3 and 4 show XRD pattern and microstructure of the talc samples after activation (30 min).

Fig. 3 shows that the diffraction peaks got less intense, thus indicating the alteration of microstructure, amorphousness of the material treated, change of the filler grain size and shape (Table II), as well as the crystal defects.²⁵ During the mechanical activation, the structure was changed, the talc grain was crushed and rounded.

Figure 5 shows the histogram of the filler grain size distribution.

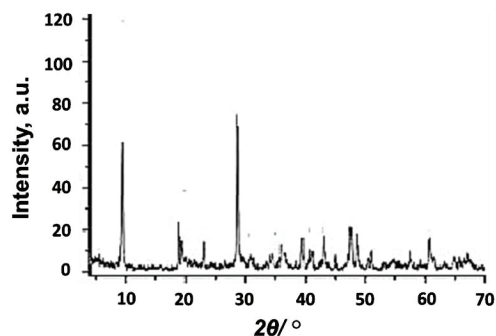


Fig. 3. XRD of talc samples after activation.

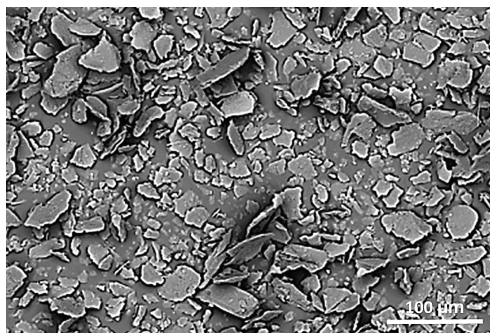


Fig. 4. SEM microphotograph of talc samples after activation.

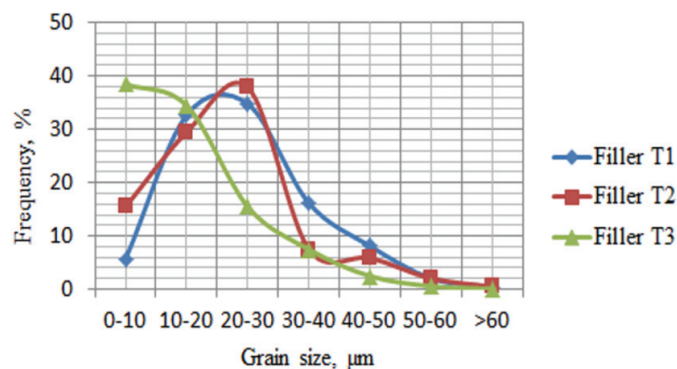


Fig. 5. Histogram of the filler grain size distribution.

Based on the data on the filler mean grain size, it may be expected that the lower-grained fillers will precipitate slower in suspension; they will keep their dispersed state longer and the coating suspension will homogenize more easily. Smaller grains have a larger active surface, they are more difficult to settle, which contributes to the improvement of sedimentation stability of the coating suspension.

Fig. 6 shows the histogram of the filler grain shape factor. Based on the data on the shape factor, the T₁, T₂ and T₃ filler grains are classified in the category of rounded grains (according to division for the grain shape factor of 0.6–0.8).

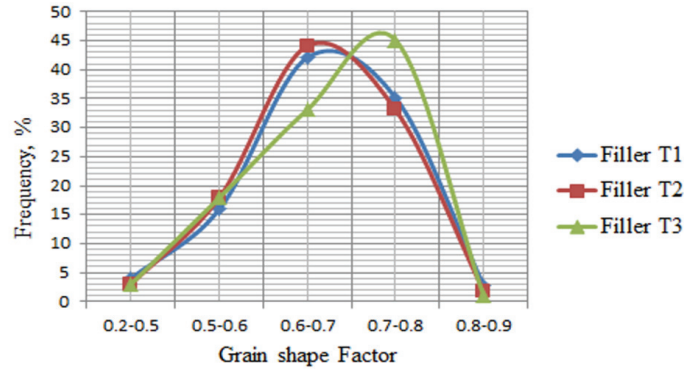


Fig. 6. Histogram of the filler grain shape factor.

Properties of refractory coatings with activated fillers

The microphotographies of suspension for all the 3 types of refractory coatings, with a talc-based activated filler (Table II), are shown in Figs. 7–9.

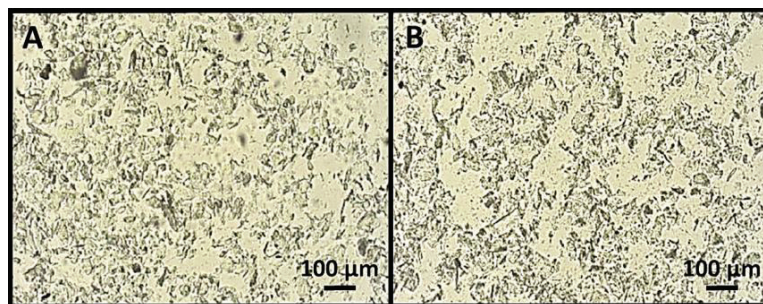


Fig. 7. Microphotographs of suspensions of the lost foam refractory coatings, Type I:
A – homogenous; B – diluted.

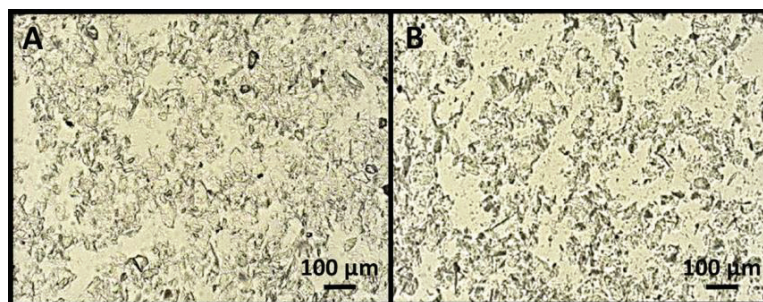


Fig. 8. Microphotographs of suspensions of the lost foam refractory coatings, Type II:
A – homogenous; B – diluted.

During the research referring to water-based coatings, Types I and II, a certain dissolution of the coating film was noticed on the surfaces of polymer

pattern, when a lower content of bentonite-based binding agent was used, up to 2.5 %, in reference to earlier works.²²

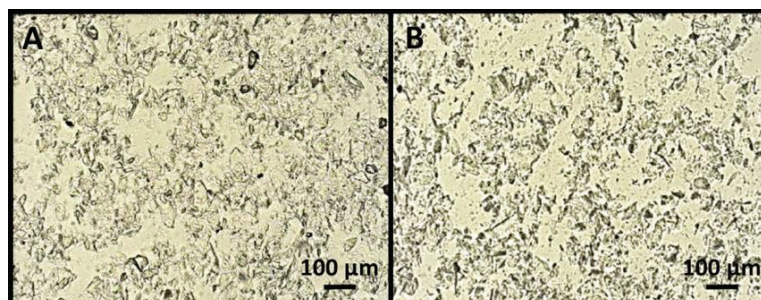


Fig. 9. Microphotographs of suspensions of the lost foam refractory coatings, Type III: A – homogenous; B – diluted.

Furthermore, due to the mechanical activation, the filler grains got crushed, and increasing the amount of bentonite-based binding agent (to 4.5–5 %), led to a better coating layers adherence to the pattern surface. A of the coating, trade name Bindal H, was also introduced to the binding agent composition (in amount of 4.5–5 %), thus increasing the binding agent ability to get firmly adhered to the polymer pattern surface (Table III). Carboxymethyl cellulose (CMC), in amounts up to 1.5–2 %, was added as an additive in order to increase the coating suspension stability (Table III).

Type III alcohol-based coatings did not show any defects. By using both colophonium-based binding agents (up to 4.5 %) and the suspension maintaining agent Bentone 25 (organic type, up to 2.5 %), Table III) uniform and homogenous coating layers were formed on the surfaces of sand moulds and cores.

Table IV shows the technological properties of the refractory coatings produced: Types I–III.

TABLE IV. Comparison of technological properties of refractory coatings

| Coating | Sedimentation (24 h) % | Penetration ^a mm | Drying | Refractoriness ^b | Layer thickness mm ² |
|----------|---------------------------|--------------------------------|------------|-----------------------------|------------------------------------|
| Type I | 5.5–5.7 | ≤1 | Air -dried | SK 14/1410 °C | 0.4–0.7 |
| Type II | 4.5 | ≤1 | Air-dried | SK 14/1410 °C | 0.4–0.5 |
| Type III | 5 | ≤2 | Heat-dried | SK 14/1410 °C | 0.3–0.8 |

^aPenetration coating suspension into the sand mould; ^bSeger's pyramid and the number that represents the code for the temperature

During the production of the Type I–III refractory coatings, it was observed that the coating suspensions with the density of 2000 kg/m³ got homogenized quickly and easy.

The use of a filler with various rounded grain sizes (Type II coating) led to a better mutual grain stack within the coating layers facilitating the filler grain – water-binding agent blend which contributed to the production of a highly permeable, thin (below 0.6 mm), homogenous and continuous film of coat on the pattern surface.

The application of the activated talc-based filler significantly improved the Type I–III coating suspension stability (sedimentation 4.5 - 5.7 %, Table IV).

Earlier research referring to talc-based coatings with grain size of 100 μm , 40 μm ^{31,32} showed that the amount of precipitated matters was much higher (7–8 %).

In accordance with the standards,^{18,19} the results obtained for sedimentary suspension stability of all types of coatings were satisfactory, as well as the results obtained for the depth of their penetration into the sand mould (Table IV).

After the visual inspection of surface of the sand–produced castings (with the application of the Type III refractory coatings) and lost foam process (with application of the Type I and II refractory coatings), it was acknowledged that the surfaces were clean, smooth, and glossy with no visible surface defects like roughness or uneven spots. This will help reduce the cleaning and the mechanical operations applied for the castings, *i.e.* it will help make the production costs in cast houses lower.

CONCLUSIONS

The result of this research is the determination of the compositions of the water-based lost foam refractory coatings with the mechanically activated, talc-based filler (with grain size 10–22 μm). As for sand moulds and cores, the composition of the alcohol-based refractory coatings with talc-based activated filler (with grain size of 22 μm) was defined. As activated talc-based fillers with a smaller grain size were applied, the compositions of coatings were altered in terms of content of binding agent and additive, which helped them both improve sedimentary stability of coating suspension and utilization properties of the coatings.

The preparation procedures for the coating suspensions were defined to accomplish the pre-defined coating properties in terms of refractoriness, gas permeability, easy application and adherence to mould and pattern surfaces, easy adjustment of the coat layer thickness, no bubbles, no cracking, or erasure of the dried coat layers. The coating suspensions with density of 2000 kg/m^3 presented high sedimentary stability (precipitated matters below 4.5 % during 24 h).

The results of examination of the quality of AlSi12CuMg castings, produced by the lost foam casting process, show that the following technological parameters have the significant influence on the castings quality: pattern density, thermo-physical characteristics of the refractory coating based on talc, shape and dimension the gating system, and particularly on the process flow and balance of the evaporative pattern–refractory coating–liquid metal–sand system. The application

of thinner layers (0.5 mm) of a water-based coating of Type I and II, as well as the application of polystyrene patterns with lower density (19 kg/m^3) have a positive influence on the surface quality and the structural and mechanical properties of castings made of aluminium alloys.

Talc-based filler has lower hardness; in order to increase the mechanical properties of the dried coat layers, the further research should focus on combined talc-based fillers with the addition of cordierite, zeolite or corundum, which could reduce distortion and defects of polymer patterns during the production of moulds in the lost foam process.

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ИЗВОД

СИНТЕЗА НОВИХ “LOST FOAM” ВАТРОСТАЛНИХ ПРЕМАЗА НА БАЗИ ТАЛКА

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Истраживање је фокусирано на поступке припреме пуниоца на бази талка у циљу побољшања реолошких својстава “lost foam” ватросталних премаза. Талк величине зрна од 40 μm механички је активиран у вибрационом млину у временима од 10, 20 и 30 min. У зависности од времена механичке активације, анализирана је промена величине и облика зрна пуниоца заједно са њиховим утицајем на дисперзност и стабилност суспензије премаза. За карактеризацију пуниоца коришћене су следеће методе: рендгенска дифракциона анализа, скенинг електронска и оптичка микроскопија. Испитиван је утицај састава премаза избором подесне величине и облика зрна активiranог пуниоца на квалитет премаза. Осим тога, примењене су различите компоненте премаза и измењен је поступак израде премаза. Показано је да је примена ове врсте “lost foam” ватросталних премаза на воденој основи имала позитиван утицај на квалитет одливака од легура алуминијума. Такође, тестирани су ватростални премази на бази алкохола са активираним пуниоцем на бази талка који су коришћени за ливење одливака у калупе од песка.

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