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Influence of Mechanical Activation of a Cordierite –Based Filler on Sedimentation Stability of Lost Foam Refractory Coatings

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Abstract:

For the development of the Lost Foam refractory coatings with controlled rheologic properties, the influence of the mechanical activation process on the cordierite-based filler's properties change was examined. First of all, the test referred to the change of filler particles' size and shape, as well as to dispersion ability and stability of the coating suspension. Cordierite was obtained by synthesis in a solid state, out of the mass consisting of kaoline, alumina, quartz, sepiolite. For characterization purposes, the following methods were used: X-ray diffraction, differential thermal analysis, SEM and optical microscopy. Mechanical activation of filler was performed in a vibrating mill. The upper boundary of the grain size was 100% -30 x 10-6 m, the times were (min): 15; 30; 60. The new composition of Lost foam refractory coatings has been developed with a change of the coating production process, as well. These newly synthesized coatings proved to be effective in terms of a positive influence on a surface quality, structural and mechanical properties of aluminium castings. Test results may be useful to have the Lost Foam refractory coatings specified together with other process parameters used for the production of castings according to this casting method.

Keywords: *Cordierite; Mechanical activation; Lost Foam refractory coatings.*

1. Introduction

Application of the Lost Foam process for the production of pre-defined quality castings has not been examined well enough yet. It requires a systematic research referring to the triangle: structure-properties-technology. Unlike sand mold casting where liquid metal flows into the "mold cavity", with the Lost Foam process, patterns and inlet systems made of polymers are retained in the mold until the liquid metal has flown in ("full mold casting"). In contact with liquid metal, polymer patterns degrade and evaporate; at the same time, castings solidification takes place. As a consequence of degradation and evaporation of the polymer pattern, a large amount of liquid and gas products are released. These products are a frequent cause for castings' defects [1-5]. To obtain quality castings, it is necessary to apply highly permeable Lost Foam refractory coatings [1, 6-8].

Over the last decades, leading world foundries have intensively tested and developed the Lost Foam process; it is now used for the production of complex castings for aircraft and automotive industry. The structure and properties of these castings are much influenced by

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critical process parameters. Therefore, these parameters require control and optimization in order for the desired useful properties of the castings to be reached [9-13]. Special attention is paid to process management referring to the polymer pattern degradation, as well as to optimization of degradation products with the application of polymers with a lower density which is specially designed for this casting method [1, 2]. Casting temperature is an important parameter; its value depends on the type and density of a polymer pattern. Degradation and evaporation processes are much influenced by the type and thickness of the Lost Foam refractory coating, grain type and grain size of the molding sand, i.e. mold permeability, design of castings and inlet systems, and, primarily, the castings wall thickness [14-17]. Polystyrene evaporation rate is directly dependent on both the pattern density and the Lost Foam coatings and sand mold permeability. This paper addresses this issue with particular attention. To prevent castings defects to be caused by the pattern degradation and evaporation products to be retained in the mold, a lower density polymer (polystyrene with density up to 19 kg m^{-3}) was chosen; then, dry quartz molding sand with larger grain size (0.36 x 10^{-3} m) was applied, as well as the Lost Foam refractory coatings with a cordierite-based, mechanically activated filler with controlled rheologic properties and with thinner, highly-permeable layers of coat (up to 0.5 x 10^{-3} m).

Systematic research of the Lost Foam refractory coatings properties have shown that there are some general conditions which must be met by these coatings: suiTab. refractoriness; low heat expansion coefficient; suiTab. gas permeability; resistance to liquid metal current with no penetration into the mold wall; inertia towards liquid metal; easy application and attachment to mold and pattern surfaces; easy adjustment of coating thickness; short drying time with no occurrence of coat cracks or erasure; formation of visible thin film of coat firmly bound to the surface of the polymer pattern [3, 18-22].

In order that a non-metallic raw material may be used as a filler for casting coatings, grain and metric composition must be such that the upper boundary grain size of the raw material be below 40 x 10^{-6} m. This is required to attain a suiTab. rheology and the adhesive properties of the coating [2, 23-25]. This paper shows that the presence of the 35 x 10^{-6} m filler particles is suiTab. to form an even and uniform, a thinner film of the coat. It is of particular importance when the coat is applied for the Lost Foam casting process, because particles with different (grain and metric) composition are a better match; the filler particles get “stacked” among themselves to form thin, highly permeable layers of coat on the pattern concerned [3, 8, 16].

According to:

- the results of research referring to complex notions and processes carried out within the pattern, metal and mold at the liquid metal inflow stage and referring to polymer pattern degradation and castings solidification [1-4];
- examination of the influence of various process parameters on the structure and properties of castings [6],

the idea was to check the possibility to apply mechanical activation of a cordierite-based filler in order to improve rheologic properties of the Lost Foam refractory coatings. To accomplish high sedimentation stability, the choice was made among various types and amounts of additives in order to enable adsorption of the additive on the mechanically-activated filler’s particles and to keep the filler dispersed in suspension, preventing its precipitation. Special attention is paid to the type and amount of the binding agent, both for a better connection among activated filler’s particles and for good adhesion of the film of the coat to the surface of the polymer pattern. As thinner filler particles were applied, the amount of binding agent was increased in reference to previous examinations [26, 27]. Water was used as a solvent. The density of refractory coatings was ranging from 1800-2000 kg m^{-3}. Design of cordierite-based refractory coatings and choice of production method were carried out in accordance with the analysis of the influence of mechanical activation of non-metallic mineral raw materials on the filler’s quality, i.e. on the grain size and shape of the filler, according to the works [28-42].

2. Materials and Experimental Procedures

2.1. Synthesis of Cordierite-Based Filler

Cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) was chosen as the filler for the Lost Foam refractory coatings due to the following properties: high refractoriness (16 SK/1470 ºC); hardness acc to Mosh chart is 7; density 1900-2200 kgcm^{-3}; low heat conductivity coefficient, λ = (2.3 – 2.9) W/mK; low linear thermal expansion coefficient, α = 1.7x 10^{6}/ºC (20-1000); high thermal shock resistance; relatively high melting temperature, applicable up to 1380 ºC; high inertia in reference to liquid metal, absence of gases in contact with liquid metal, non-wetTab. by liquid metal [18, 22, 43, 44].

The cordierite used in experiment (marked ''C'') was the product of synthesis of the following raw materials: kaoline, alumina, quartz and sepiolite which chemical compositions are shown is Tab. 1. Initial materials, except kaoline, were made thin, with upper grain size boundary of 100% -0.1 x 10^{-3} m. Then, they were blended in the ratio $2MgO{:}2Al_2O_3{:}5SiO_2$. After homogenization, powder mix was pressed under 1MPa and then sintered at the temperature of 1350 ºC over the time of 8 hours in a laboratory oven with oxidation atmosphere.

Tab. I Initial compositions of components used to produce the filler C (%).

	Compound	SiO_2	MgO	Al_2O_3	Fe_2O_3	CaO	Na_2O+K_2O	LoI
		%						
Pre-cursor	Alumina	0.14	0.01	95.96	0.09	0.16	0.05	3.20
	Sepiolite	52.33	29.25	1.19	0.22	0.54	0.18	18.30
	Kaolin	53.55	/	28.93	1.35	0.65	0.07	8.14
	Quartz	97.50	0.11	0.15	0.28	0.17	0.4	2.20

The obtained samples of C were subject to the milling procedure in a vibrating mill. They were milled down to the boundary grain size of 100% -30 x 10^{-6} m. It was the initial boundary size of the filler C. It was then exposed to the process of mechanical activation in a vibrating mill over different times (min): 15 (mark: C_1); 30 (mark: C_2); 60 (mark: C_3).

2.2. Characterization of Filler C

XRD analysis was performed by the X-ray diffraction meter, model PW-1710 (Philips Analytical, Almelo, Netherlands); It involved a curved graphite mono-chromate meter and a scintillation counter. Intensities of the diffracted CuKα of the X-ray radiation (λ=1,54178 Å) were measured at room temperature with intervals of 0,02° 2θ , over the time of 1 s, within the range from 4° to 70° 2*θ*. The X-ray tube was under the voltage of 40 kV and current of 30 mA, while primary and diffracted rays' slots were 1° and 0,1 mm.

Thermal behavior of cordierite was examined by the device Shimadzu DTA -50 (Shimadzu, Tokyo, Japan). Heating rate was 10 º/min with the interval of 20-1200 ºC in nitrogen atmosphere. Referential sample was α-Al_2O_3 (corundum) powder.

The microstucture of the samples was characterized with the scanning electronic microscope "JEOL" model JSM 6610 LV (JEOL, Tokyo, Japan). In order to improve conductivity, the sample was vapoured with gold powder.

2.3. Synthesis of Lost Foam refractory coatings with activated fillers: C_1; C_2; C_3

After mechanical activation, the cordierite samples obtained (marked: C_1; C_2; C_3) were used to produce water-based refractory coatings. The grain size of the cordierite samples were: C_1 (100% -24 x10^{-6} m); C_2 (100% -20 x10^{-6} m); C_3 (100% -12 x10^{-6} m). Refractory coatings and their production processes were designed according to the works [3, 8, 22], our works [24, 26] and domestic norms [43, 44]. Based on the analysis of the mechanical activation influence on the filler grain size and shape [27, 28-33] as well as of the filler grain size influence on coating suspension sedimentation stability [3, 7], cordierite-based, mechanically activated fillers were chosen: first filler C_2 (100% -20 x10^{-6} m) and the second combination of fillers C_2 (100% -20x 10^{-6} m) and C_3 (100% -12 x10^{-6} m). In reference to the filler grain size chosen, two series of the Lost Foam refractory coatings were designed as follows: coating with filler C_2 (marked: A) and coating with combination of fillers C_2 + C_3 (marked: B), which were corrected according to test results for the coating suspensions obtained. These coatings were tested: at the temperature of 22 ºC; in the suspension density of 1800-2000 kgm^{-3}; with thickness of wet film layers (x 10^{-3} m): 0.2; 0.5; 0.9. Refractory coatings were applied in the following way: they were immersed into the tank containing the coating suspension or were poured. During application – in order to improve homogenization of coating suspension – stirring was applied at the speed of 1 ^{o}/min. Water-based Lost Foam refractory coatings were air-dried over the time of 24 hours.

2.4. Characterization of Lost Foam Refractory Coatings

In order to establish filler and binding agent's distributions, Lost Foam refractory coating suspension was analyzed on the polarized microscope for transmitted light JENAPOL, manufactured by Carl Zeiss-Jena, Germany, with Microphoto System STUDIO PCTV; Pinnacle System, Mountain View, CA. Measurements of the filler grain size and shape were carried out with 4000 grains, while the analysis was conducted by means of the software application package OZARIA 2.5 (interval 0-1), (VAGA Lab, B. Kljajevic, Belgrade, Serbia). Shape factor is: for 0- corresponding to the position of the needle, for 1- corresponding to the circle. There is the following division according to the grain shape factor: from 0,0-0,2 – *angular*; from 0,2-0,4 – *subangular*; from 0,4-0,6 – *sub-rounded*; from 0,6-0,8 *rounded* and from 0,8-1,0 – *well rounded* grain shape. Properties of the Lost Foam refractory coatings obtained were examined pursuant to the norms [43, 44]. That is, the coatings were applied on the surface of the test pieces made of polystyrene, with density of 19 kg/m^3. Sedimentation stability was tested when the samples of refractory coatings were left idle in a cylindrical vessel with volume of 1 x 10^{-4} m^3 and height of 2.8 x 10^{-1} m for 24 hours. The results of the test are expressed in percentages by the fact that the number of readings milliliters of the transparent layer is equal to the precipitation in percentages.

To assess the quality of the Lost Foam coatings obtained, simple, plate-shaped castings were casted. They had the following dimensions (0.2 x 0.05 x 0.02) m and were made of the alloy AlSi12CuMg, while the casting temperature was 778 ºC. Liquid cast was prepared through the salt-refinement and degassing. These salts were sodium and potassium chloride-based and sodium was used as a modifier. Dry quartz sand was used as a material for the mold involved in the Lost Foam casting process. Mean grain size of the sand was 0.36 x 10^{-3} m. Polymer patterns used in the experiment were made of polystyrene with density of 19 kgm^{-3} and with mean grain size of 1x 10^{-3} m.

3. Results and Discussion

3.1. Properties of Synthesized Filler C

The composition of the synthesized cordierite sample C, which was mechanically activated and then used for production of refractory coatings, was: SiO_2 - 51.01 %, Al_2O_3 - 30.09 %, MgO – 13.5 %, Fe_2O_3 – 1.2 %, CaO – 3.2 % and K_2O+Na_2O – 0.02 %.

Fig. 1 shows an X-ray graph for the synthesized sample C indicating a prevailing presence of cordierite. To a lower extent, presence of corrundum, quartz, spinnele, peryclass is also noted. Based on semi-quantitative chemical analysis, it was found that the analyzed sample C contains smaller amounts of K, Ca and Fe apart from the main cationes Mg, Al and Si.

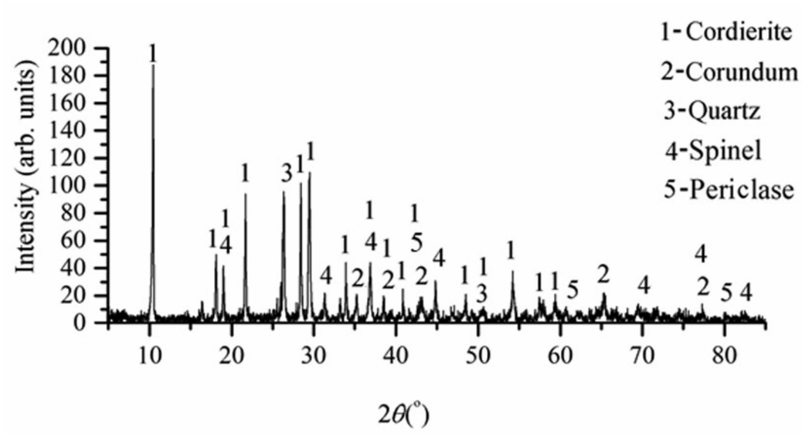

Fig. 1. X-ray diffractogram of the cordierite sample (C).

Fig. 2 shows the DTA curve for the sample C. The thermal treatment of the cordierite comprises several separate regions that are monitored by individual peaks. The first endothermic peaks occurring at lower temperatures, below 250 ^{o}C and it corresponds to the evaporation process of physically bounded water. At an interval of 600 to 900 ^{o}C, the structural water must be released. Second endothermic peaks occurring at 517 ^{o}C and it corresponds to the α-tridymite →α-quartz polymorphic transformation. The peak at 950 ^{o}C may be appropriate to the decomposition of talc to magnesium meta silicate (enstatite), amorphous silica and vapor water. Exothermal effect occurring at 1013 ^{o}C. Based on literature data [41] it can be concluded that the kaolin transformed into a phase type of spinel.

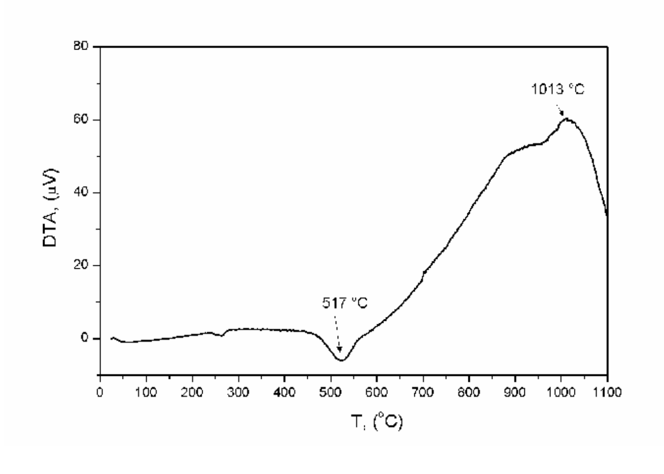

Fig. 2. DTA curve of the cordierite sample (C).

Fig. 3 shows micro-structure of the filler C. It clearly indicates the grains of irregular forms and different sizes.

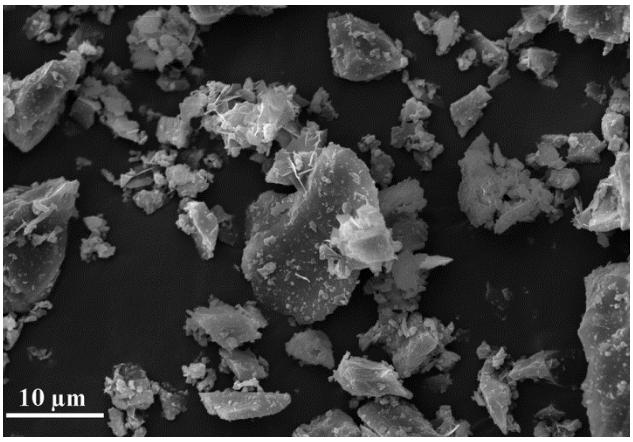

Fig. 3. Microphotography of refractory filler based on cordierite (C).

3.2. Properties of Activated Fillers: C_1 ; C_2 ; C_3

Mechanical activation of the filler C (100% -30 x 10 $^{-6}$ m) over the times of (min): 15; 30; 60, was used to obtain fillers of different sizes and shapes: C_1 (100% -24 x 10^{-6} m, mean grain shape factor 0.67); C_2 (100% -20 x10^{-6} m, mean grain shape factor 0.69); C_3 (100% -12x10^{-6} m, mean grain shape factor 0.7), respectively. Results of the analysis of the mechanically activated filler grain size and shape factor are given in the Fig. 4-5.

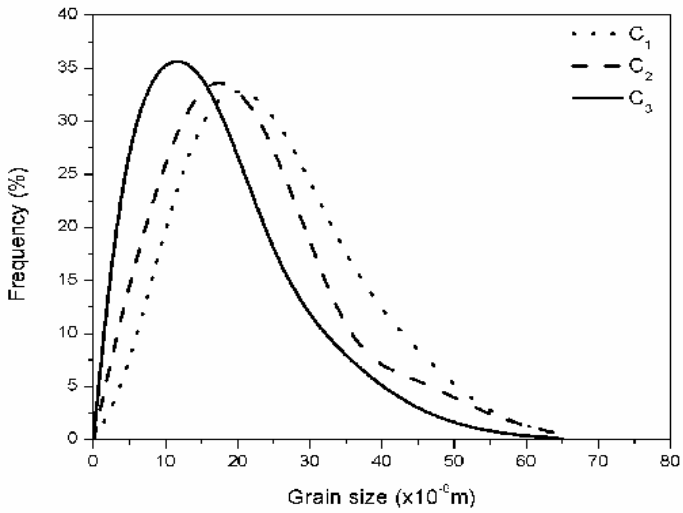

Fig. 4. Histogram of grain size distribution for the fillers C_1; C_2; C_3.

Based on the data on the shape factor, the C_1; C_2; C_3 filler grains are classified in the category of rounded grains. 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.

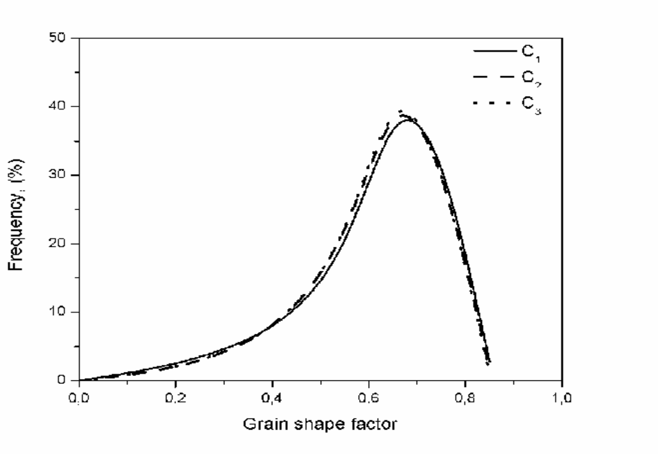

Fig. 5. Histogram of grain shape factor for the fillers C_1; C_2; C_3.

3.3. Properties of Lost Foam Coatings with Activated Fillers

Compositions of the A&B series Lost Foam refractory coatings are shown in Tab. II.

Tab. II Compositions of the A & B series Lost Foam refractory coatings (%).

	Type A:	**Type B:**
Refractory filler	-Cordierite C_2, grain size 20 x10^{-6} m, 92-93%	-Cordierite C_2, grain size 20 x10^{-6} m, 80-82 % -Cordierite C_3, grain size 12x10^{-6} m, 11-12 %
Bonding agent	-Bentonite 1.5-2.5% -Bindal H 1-1.5%	-Bentonite 2.5-3.5 % -Bindal H 1-1.5%
Suspension maintenance agent	-$Na_3P_3O_3$ 1-3.5 % -Carboxymethil cellulose (CMC) 0.5%	-$Na_3P_3O_3$ 1-3.5 % -Carboxymethil cellulose (CMC) 0.1%
Solvent Density- (kg/m^3)	Water 2000	Water 2000

Microphotography of suspensions of the A& B series Lost Foam refractory coatings are shown on Fig. 6-7.

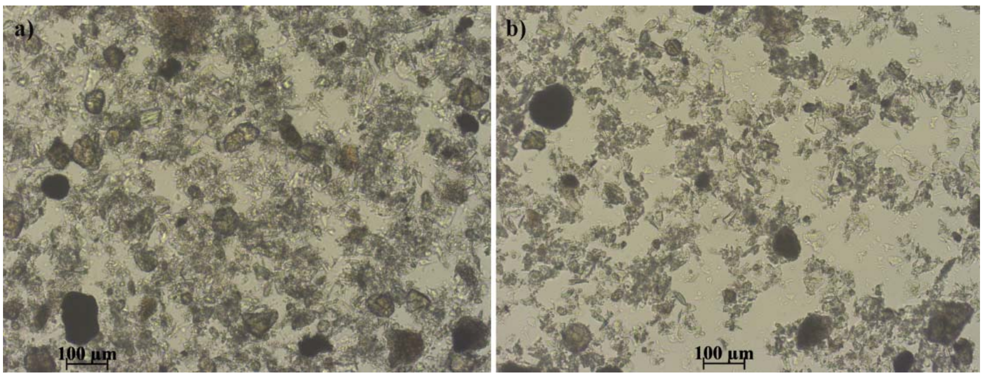

Fig. 6. Microphotography of suspensions of the A series Lost Foam refractory coating: a. homogenous suspension; b. dilluted suspension.

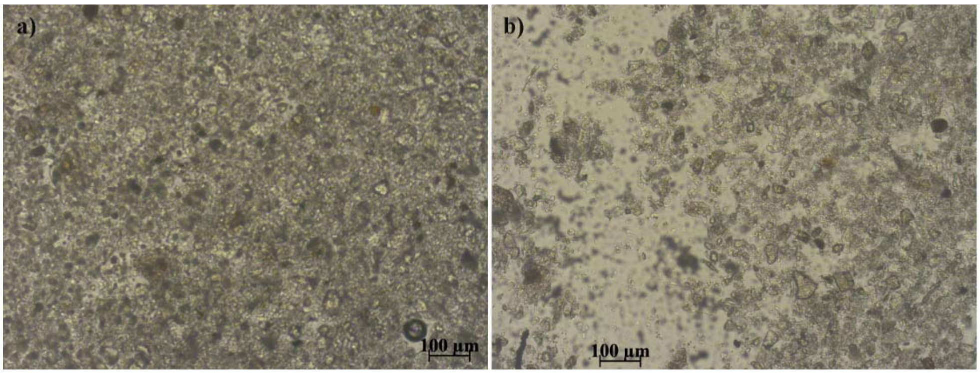

Fig. 7. Microphotography of suspensions of the B series Lost Foam refractory coating:a. homogenous suspension; b. dilluted suspension.

Tests of sedimentation stability of the A&B series Lost Foam coatings showed different results for the amounts of precipitated matters. As for the coatings with the fillers C_2 (grain size 100% -20 x10^{-6} m, filler amount 92-93 %), the amount of precipitated matters was 5.2-5.8 %. As for the coatings with combination of the filler C_2 (grain size 100% -20 x10^{-6} m, filler amount 80-82 %) and the filler C_3 (grain size 100% -12 x10^{-6} m, filler amount 11-12 %), tests showed a lower content of precipitated matters - abt 4.3-4.5 % - indicating significant improvement of sedimentation stability when finer filler grains are used. During production of the A & B series refractory coatings, it was noticed that coating suspensions with density of 2000 kg/m^3 got homogenized faster and easier than low density suspensions, 1800 kgm^{-3}. Utilization of filler particles of different sizes and shapes (B series) led to a better mutual particle "stacking" inside layers of coats. It helped formation of a highly permeable, thin film of coat (below 0.5 x 10^{-3} m) on the pattern surfaces. This film was homogenous and constant. According to the norms [43, 44], the results of sedimentation stability of the A and B suspensions were compliant. After visual inspection of the surface casted according to the Lost Foam method, it was noted that application of both types of coatings together with the fine-grained polystyrene patterns (1x10^{-3} m) led to production of castings with fine and smooth surfaces.

When the coatings were applied on polymer patterns with rougher surface (larger polystyrene grains: 2,5 x10^{-3} m), rough surface was fully reproduced. The same happened when thinner layers of coat (below 0.5x10^{-3} m) were applied, as well as the coatings with lower density (1800 kgm^{-3}). It indicates that refractory coats of homogenous compositions must be used to attain fine and smooth castings surface; during application stage, coating suspension should be lightly stirred – at the speed of 1 ^{o}/min; coating density should be 2000 kgm^{-3}. Once these conditions are met, thinner layer of coats (up to 0.5 x 10^{-3} m) might be used. To attain fine and smooth castings surfaces according to the Lost Foam method, it is necessary to use polymer patterns with lower density and more tiny grains for any pattern defects are fully reproduced on the surface of castings.

4. Conclusion

As a result of these tests, optimum compositions of Lost Foam refractory coatings with mechanically activated, cordierite-based filler were determined (filler grain size from 100% -20 x 10^{-6} m to 100% -12 x 10^{-6} m). Coating suspension preparation procedures were defined, therefore pre-defined coating properties in terms of refractoriness, gas permeability, easy application and attachment to the pattern surfaces were accomplished, as well as an easy adjustment of coat thickness on the pattern surface, absence of bubbles, short drying time, absence of cracks and erasures of the dried coat layers. Coating suspensions with density of 2000 kgm^{-3} showed high sedimentation stability (below 4.5 % of precipitated matters). Application of thinner layers (0,5x10^{-3} m) of a water-based coating of this type, as well as application of polystirene patterns with lower density (19 kgm^{-3}) have a positive influence on surface quality and structural and mechanical properties of castings made of aluminum alloys.

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Садржај: *За развој Lost foam ватросталних премаза са контролисаним реолошким својствима истраживан је утицај механичке активације на промену својстава пуниоца на бази кордијерита, пре свега на промену величине и облика зрна пуниоца, дисперзност и стабилност суспензије премаза. Кордијерит је добијен синтезом у чврстом стању масе састављене из компонената: каолин, глиница, кварц, сепиолит. За карактеризацију добијеног пуниоца коришћене су следеће методе: XRD, DTA, SEM, optička mikoskopija. Механичка активација пуниоца урађена је у вибрационом млину са горње границе крупноће зрна 100% -30 x 10-6 м у времену (мин): 15; 30; 60. Нови Lost foam ватростални премази развијени су оптимизацијом састава премаза и изменом поступка израде премаза. Њиховом применом у пракси ливница остварени су позитивни ефекти на побољшању површине, структурних и механичких својстава добијених одливака на бази легура алуминијума. Резултати истраживања могу*

корисно да послуже за дефинисање Lost foam ватросталних премаза и других параметара процеса за производњу одливака по новој методи ливења.
Кључне речи*: кордијерит, механичка активација, Lost foam ватростални премаз.*