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SYNTHESIS AND CHARACTERIZATION OF NEW REFRACTORY COATINGS BASED ON TALC, CORDIERITE, ZIRCON AND MULLITE FILLERS FOR LOST FOAM CASTING PROCESS**SYNTEZA I CHARAKTERYSTYKA NOWYCH POWŁOK OGNIOTRWAŁYCH NA BAZIE TALKU, KORDIERYTU, CYRKONII I MULLITU DO ODLEWANIA METODĄ PIANY TRACONEJ**

Refractory coatings based on different refractory fillers (talc, cordierite, zircon and mullite) for application in Lost Foam casting process were investigated. Design and optimization of the coatings composition with controlled, rheological properties included, and consequently synthesis were achieved by application of different coating components, namely different suspension agents and fillers and by alteration of the coating production procedure. Morphologic and microstructural analysis of fillers was carried out by means of scanning electronic microscope. X-ray diffraction analysis by means of X-ray diffractometer was applied in determination and monitoring the phase composition changes of the refractory fillers. An analysis of the particle size and shape was carried out by means of the PC software application package OZARIA 2.5. To assess the effects of application of individual refractory coatings, a detailed investigation of structural and mechanical properties of the moldings obtained was performed. Highlight was placed on revealing and analyzing surface and volume defects present on moldings. Radiographic molding tests were carried out by means of the X-ray device SAIFORT type-S200. Attained results are essential for the synthesis of refractory coatings based on high-temperature fillers and their applications in Lost Foam casting process for manufacturing of moldings with in-advance-set properties.

Keywords: high-temperature materials, refractory coatings, talc, cordierite, zircon, mullite, Lost Foam casting

W pracy przedstawiono wyniki badań materiałów wysokotemperaturowych – powłok ogniotrwałych na bazie talku, kordierytu, cyrkonii i mullitu, które są stosowane w procesie odlewania metodą piany traconej. Projektowanie i optymalizację składu powłoki o kontrolowanych właściwościach reologicznych, a następnie syntezę osiągnięto poprzez zastosowanie różnych składników powłok, tj. różnych stabilizatorów zawiesiny i wypełniaczy oraz poprzez zmiany w procesie produkcji powłok. Analizę morfologiczną i mikrostrukturalną wypełniaczy przeprowadzono za pomocą elektronowego mikroskopu skaningowego. Analizę dyfrakcji promieni rentgenowskich zastosowano do określania i monitorowania zmian składu fazowego wypełniaczy ogniotrwałych. Analizę wielkości i kształtu cząstek przeprowadzono za pomocą pakietu oprogramowania użytkowego PC OZARIA 2.5. Żeby ocenić skutki stosowania poszczególnych powłok ogniotrwałych, przeprowadzono szczegółowe badania właściwości strukturalnych i mechanicznych otrzymanych form. Nacisk został położony na ujawnienie i analizę defektów powierzchni i objętości obecnych w formach. Radiograficzne testy formowania przeprowadzono za pomocą urządzenia SAJFORT typu S200. Uzyskane wyniki są niezbędne do syntezy powłok ogniotrwałych w oparciu o wysokotemperaturowe wypełniacze i ich zastosowań w procesie odlewania metodą piany traconej do produkcji form o z góry ustalonych właściwościach.

1. Introduction

The most important characteristic of the Lost Foam casting process is that patterns and moulds gating made of polymers remain in the cast until the liquid metal inflow. In contact with liquid metal, polymer pattern is being intensely decomposed and evaporated in relatively short time. Previously described process is accompanied by molding crystallization. In order to achieve quality-like and profitable molding production by the Lost Foam method application, it is necessary to reach the balance in following system – *evaporable polymer pattern*

– *liquid metal – refractory coat – sandy cast* during the phase of metal inflow, decomposition and evaporation of polymer pattern and formation and solidification of castings (Scheme is given in Figure 1.). That requires systematical researches on complex appearances and processes occurring in the pattern, metal and cast as well as on appearances and processes in contact zone metal – pattern and metal – refractory coating – sand [1-7].

Polymer patterns decomposition is endothermic staged process which starts with liquid metal inflow. The patterns decomposition kinetics is the function of liquid metal temper-

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ature with which the pattern gets in contact. During the inflow stage, while metal passes through polymer pattern, 70-90% of products of metal decomposition are liquid. During the process, the decomposed liquid products are being pushed toward the casts cavity upper surface, afore the liquid metal front. In case of minor permeability of refractory coating and sand for modeling, those liquid products of pattern decomposition remain in upper parts of moldings causing appearance of surface, subsurface and volumetric defects. Further decomposition of liquid phase is effected by evaporation (boiling phase layers creation) with formation of a solid residue of polymer chain, monomer and benzene, small quantities of toluene and ethyl benzene [3, 8-11].

Important factors influencing patterns decomposition and evaporation process, besides temperature and patterns density, are: type and refractory coat layer thickness which the evaporable pattern is getting covered with, type and size of sand grain for modeling, respective permeability of sand for modeling, moldings and gating of moulds construction. The pattern density and permeability of refractory coating and sandy cast determine polymer evaporation velocity. The velocity of liquid metal entering the cast and its contact with the pattern is controlled by a proper definition of the gating of moulds [12-20].

In order to obtain moldings of a priori desired quality, critical process parameters should be determined for each particular polymer pattern, as well as the type of alloy for casting. That requires long-lasting researches with a goal to achieve optimization of Lost Foam casting process and to obtain the moldings of a priori specified properties. In order to correctly understand Lost Foam casting process optimization, it is necessary to know that various types of molding structure determine their various properties [21-28]. Beside this dependency to obtain the moldings of a priori specified properties, fundamental structure dependence on technology should also

be determined, implying critical process parameters control and control of useful molding properties. Thus, a special consideration in this work was dedicated to this correlation.

2. Materials and methods

Four series of experiments were carried out in order to analyze the possibilities of application of refractory coatings based on talc (series mark: A), cordierite (series mark: B), zircon (series mark: C) and mullite base (series mark: D) in Lost Foam casting process. The coating compositions were defined (Table 1) and the coating components preparation methods were determined. Grinding of refractory fillers talc, cordierite, zircon and mullite was conducted in a mill with balls of Cr-Ni steel ("Retsch-PM4" type), capacity 20 kg/h, with mill load of 70% and grinding time 45-60 minutes.

Refractory talc, cordierite, zircon and mullite powders used both for coating production and application in Lost Foam casting process crucially depend on rheological coating quality and on sedimentary coating stability. During research, both the coating compositions and their preparation procedures were altered in order to achieve specified coating properties. While applying refractory coating on polymer pattern by techniques of immersion into the tank containing the coating, overflowing and coating with brush, a special attention was given to the coating quality control. The basic criteria for quality evaluation of this type of refractory products were: pertinence for applying, drying behavior, resistance to attrition, sedimentation and penetration.

The experimental parameters of Lost Foam casting process (selection of the composition and preparation of refractory coats were carried out according to these parameters) are showed in Table 2.

TABLE 1

Compositions of the refractory coatings series A, B, C and D

Coat type	Refractory filler		Bonding agent	Coating maintenance material	Solvent for coating density: 2 g/cm ³
	Granulation, μm	Quantity, %			
A	talc		Bentonite 4%; Bindal H 8%	dextrin 0.4%, lucel 0.4%	water
	30-35	87			
B	cordierite		Bentonite 1-3%; Bindal H 6%	dextrin 0.5%, lucel 0.5%	water
	35-40	89			
C	zircon		Sodium silicate (water glass) 3%	dextrin 0.5%, lucel 0.5%	water
	35-40	97			
D	mullite		Bentonite 4%; Bindal H 3%	dextrin 0.5%, lucel 0.5%	water
	40-45	89			

TABLE 2

Experimental parameters of Lost foam casting process

Parameter	Parameter description
Tested alloy	AlSi10Mg
Preparation methods of liquid die	-refinement by compounds based NaCl and KCl in quantity of 0.1% on die mass; -degasification by briquette C ₂ Cl ₆ in quantity of 0.3% on die mass; -modification – by sodium in quantity of 0.05%.
Casting temperature (°C)	735; 760; 795
Evaporable polystyrene pattern	-density (kg/m ³): 20; 25. -pattern construction: plate (200×50×20) mm and staged probe with different wall thickness (mm): 10; 20; 30; 40; 50. -polystyrene grain size 1-1.5 mm.
Mounting pattern for casting	"cluster" with four patterns-plates set on central runner gate and "cluster" with two staged probes set on central runner gate
Gating of moulds	-central runner gate (40×40×400)mm, -ingates (20×20×10) mm, 2 pieces.
Dry quartz sand for cast production	Grain size (mm): 0.17; 0.26; 0.35.

Lost foam process phases are showed on Fig. 2.

Table 3. shows the process parameters for all series refractory coats production, methods of coating application on patterns and drying. A radiographic analysis was performed by means of the X-ray device SAIFORT type-S200 on the moldings from all series.

TABLE 3

Optimal process parameters of production and preparation of coat, lining and coat drying on polystyrene patterns

Parameter	Parameter description
Coat densities	2 g/cm ³
Coat temperature	25°C
The way of coats excess remove from the pattern after pulling out from the tank for lining	Patterns are been seeped, in vertical position, 5-10 s, and then set 5 s under 45° angle in order to coat layers on patterns surface get equally even
Slowly coat mixing in tank during the coat applying on pattern	- velocity 1 revolutions/min
Methods of coat applying on pattern	- "cluster" immersion into tank with coat - overflowing; - coating with brush
Drying	- first layer 1.5 h; - final layer 24 h
Coat layers thickness on the pattern after drying (mm)	0.5; 1; 1.5

3. Results and discussion

In the Fig. 3-4. the results of XRD phase analysis of the samples A to D are presented. The results of XRD showed a dominant talc presence in sample A Fig. 1 [22] dominant cordierite presence, and also spinel and quartz in sample B Fig. 2 [22], zircon presence in sample C Fig. 3 [23] and dominant presence of mullite in sample D Fig. 4 [23].

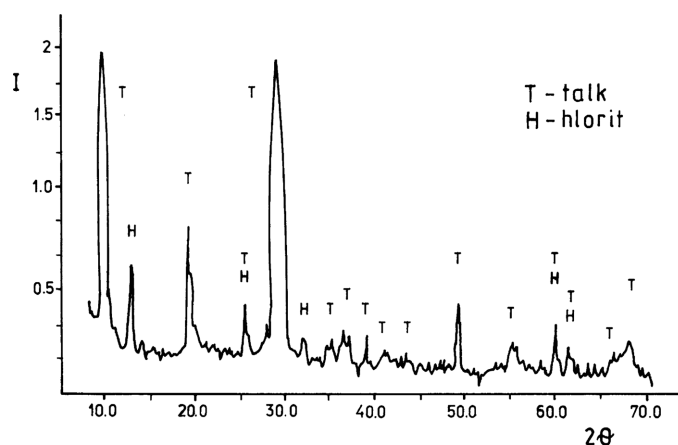


Fig. 1. Diffractograms of the filler based on talc

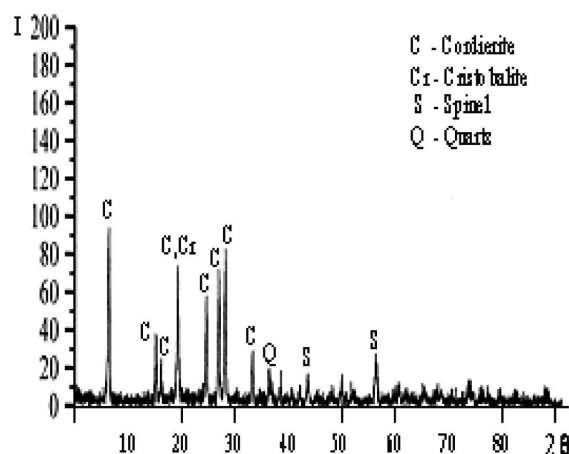


Fig. 2. Diffractograms of the filler based on cordierite

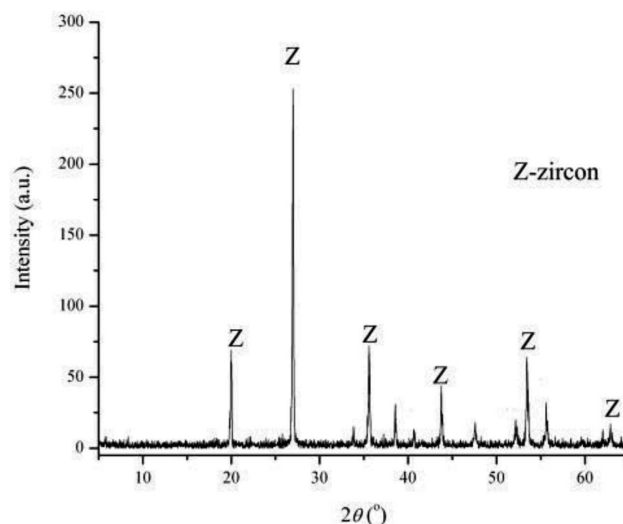


Fig. 3. Diffractograms of the filler based on zircon

Fig. 5 and 6. show diagrams of grain size and grain shape factors of the samples A to D. The mean grain size of refractory filler is between 35-40 μ m and the grain shape mean factor is 0.63-0.75, which means that the grains shape approximately are elongated round (shape factor 0 corresponds to needle shape and 1 to circle). SEM microphotographs showed that grains are in fact irregularly shaped with large number of angles which are helping the locking particles in together in the coating mixture. Such thing is preferable for obtaining homogeneous coating mixture. This conclusion is in accordance with previous investigations [8, 22, 23] emphasizing that the filler particle size and shape are crucial for the quality of casting coatings. Filler particle size, shape and surface condition depend on the type of the mill. Roughness of the surface of filler particles may cause increased absorption of the liquid metal on the coating surface, which is not favorable as it deteriorates the coating quality [29].

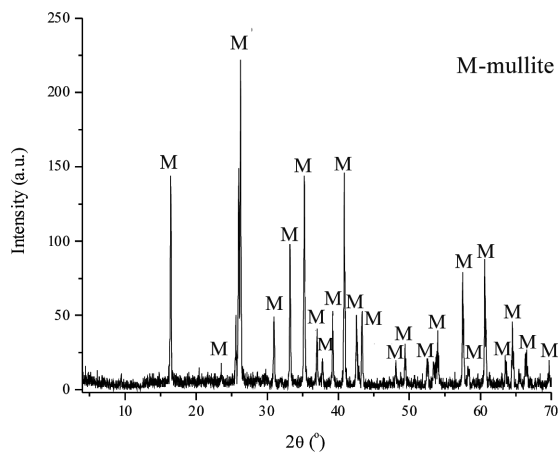


Fig. 4. Diffractograms of the filler based on mullite

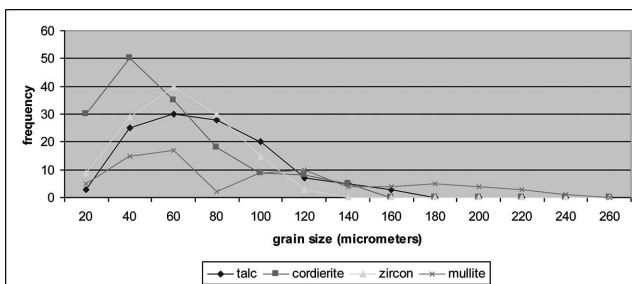


Fig. 5. Diagram of grain size of samples A to D

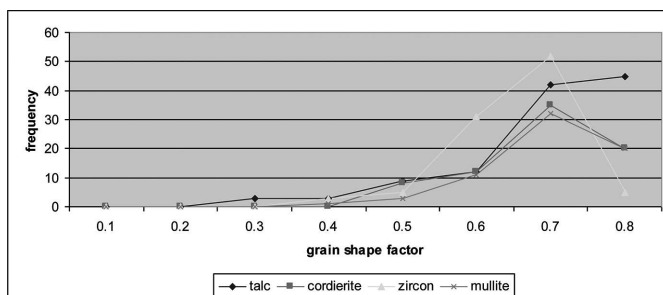


Fig. 6. Diagram of grain shape factor of samples A to D

Fig. 7-9 show results of quality-like mineralogical analysis of fillers A to D. The analysis shows that the ceramic

powders particles are principally of equal morphology, but there are differences in particle sizes. This is favorable since the particles of diverse granulations contribute to formation of equal and continuous coating layer on polymer pattern, due to better mutual harmony among the particles. It was determined that for the mullite samples preparation a longer grinding time was required (approximately 90 min) in order to achieve particles granulation below 40 μ m. Fig. 7 [23] shows that the talc-based filler has proper foliate aggregates. Morphology of the cordierite sample, Fig. 8 [32], shows that the particles have improper forms and different dimensions. Zircon powder particles have improper forms with a typical shell-like break; they are also of different dimensions (Fig. 9 [22]). Mullite sample (Fig. 10 [33]), shows that the particles have improper forms and different dimensions. Pursuant to the work [29], it was

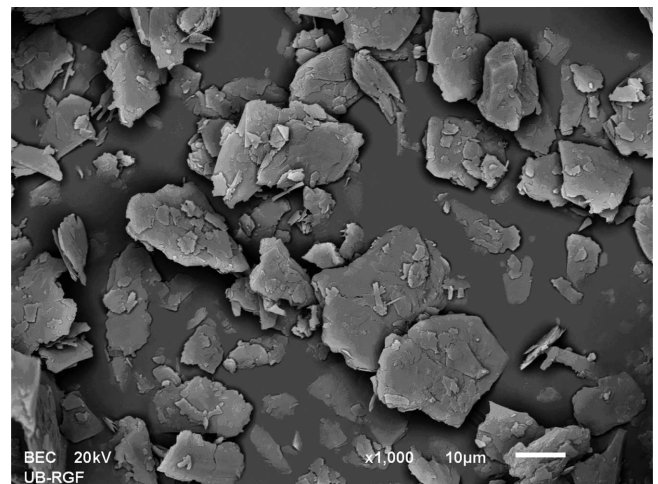


Fig. 7. Microphotograph of refractory fillers based on talc

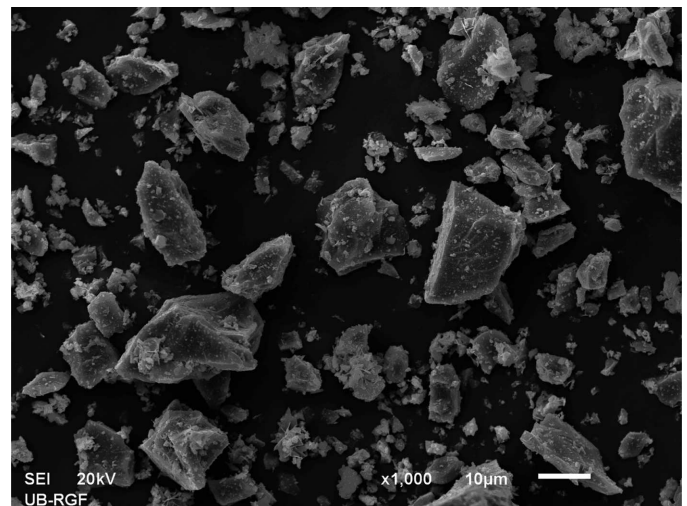


Fig. 8. Microphotograph of refractory fillers based on cordierite

showed that application of the talc-based filler with elongated and smooth particles contributed to manufacture of the coatings of higher quality, easily adhered to patterns, not forming the sintered sand fault on moldings. Applying the coating composed from zircon flour plus colloidal silica [13, 17], thin coating layers with high gas permeability were obtained, while silica content increase contributed to reduction of metal penetration into the mould. Having viewed the literature data, no cordierite application as refractory filler was recorded.

With reference to our earlier works [22, 23], this research used different filler granulations proving to be positive to get homogenous coating layers and to have the coating better adhered to the pattern surface. Further, the bentonite quantity was reduced, lucel quantity, i.e. carboxymethylcellulose (CMC) quantity increased influencing reduced appearance of cracks in dried coating layers, in accordance with the works [18,21,25].

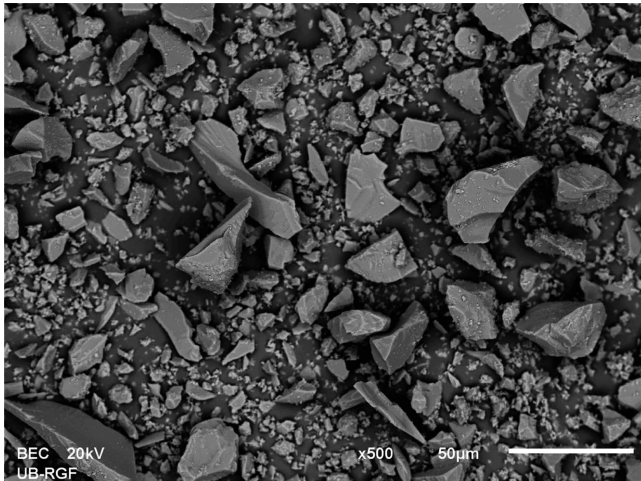


Fig. 9. Microphotograph of refractory fillers based on zircon

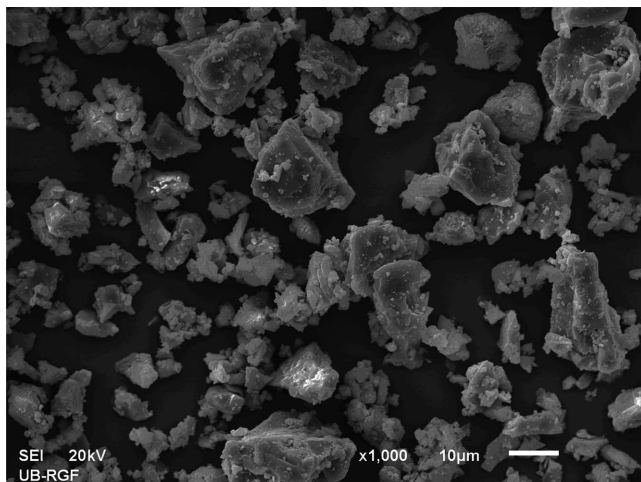


Fig. 10. Microphotograph of refractory fillers based on mullite

The control of critical process parameters for refractory coating production and the control of the coating properties determine that the coatings of all series comply with conditions for application in Lost Foam process. It was determined that coatings were easily applied on polymer patterns, being equally lined at overflowing and immersion, were easy to be coated with brush, without any mark of brush, leakage, drops and clots formation. After drying, the coating surface was smooth; coating layers were of equal thickness everywhere on pattern surface, without any bubbles, crazing, peelings or attrition. The coating quality and refractory fillers homogeneity in the coating depends on coating preparation. In order to achieve even coat layers thickness on patterns surface, it is necessary to slowly and constantly mix the coating during its application on patterns, to maintain defined density (2 g/cm^3)

and temperature (25°C) of coating. Vice-versa, the coating composition non-homogeneity occurs.

In case of applying coating layer of higher thickness and fast drying, a dried coating layers crazing and tweaking have emerged, which had negative effects on molding surface quality from those series (uneven surface, roughness, sintered sand, cracks, folds, Fig. 11 a, c.), as well as on risk of liquid metal penetration into the cast and casts sinking, Fig. 11b. Coating layer perforation and metal penetration into the cast was also noted in series during staged probes casting where coating layers of lower thickness were applied (0.5 mm) on the moldings part with wall thickness above 40 mm, at higher casting velocities. When coating layers of higher thickness were applied (above 1 mm) this fault on moldings did not emerge.

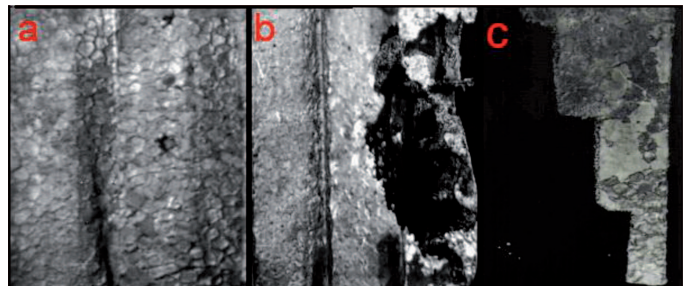


Fig. 11. Errors on castings: a) castings lumpiness of surface reproduced from the pattern, surface porosity, fold; b) metal penetration into sand; c) sintered sand on the castings surface

In order to observe the effects of casting process, evaluation of certain operation phases and analysis of applied refractory coatings influence, a visual control of the obtained moldings was affected, testing their structural and mechanical properties. After pulling out the founded "clusters" from the mould, their surface is covered with coat layer which is easy to be broken and removed from it, so the cleaning is not necessary, which significantly reduces the production costs. The refractory coatings of all series have demonstrated positive effects on the surface quality – shiny and smooth molding surfaces were obtained. The moldings are true copy of the patterns (dimensionally are precise) which indicates that the decomposition and evaporation of polystyrene pattern was in totality, and that the gating of moulds solution was satisfactory. It was noted that the lower molding parts of all series have flat and sharp edges, clean and shiny surface. At some moldings from the series with coating layers of higher thickness (above 1.5 mm) the upper molding surfaces are a bit uneven and folded, and also on certain moldings parts a surface roughness appears, and more often at the moldings from the series with patterns density above 20 kg/m^3 .

The study results of molding structural and mechanical characteristics were within the limits predicted by the standards for this type of alloys. That would be the moldings from series with used polystyrene patterns up to 20 kg/m^3 , refractory coatings of lower thickness layers, below 1 mm, applied quartz sand for modeling with its grain size above 0.26 mm, casting temperature within the limits of $760\text{--}780^\circ\text{C}$ and casting velocity which enabled even decomposition and evaporation of polystyrene, with complete elimination of gassy products from patterns evaporation, without any cast falling in

and liquid metal penetration into sand. This was confirmed by X-ray samples tests too, Fig. 12.

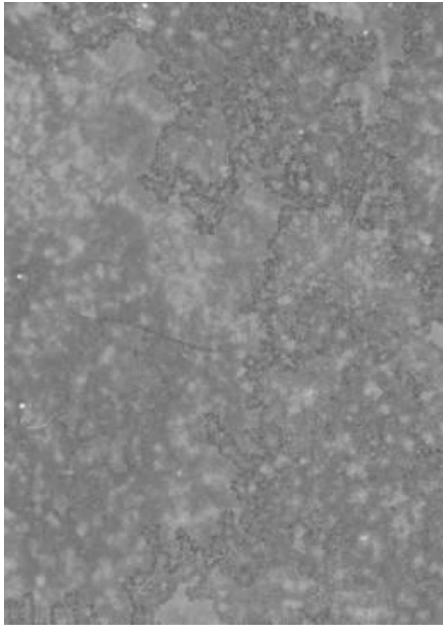


Fig. 12. Characteristic samples radiograms: casting without porosity

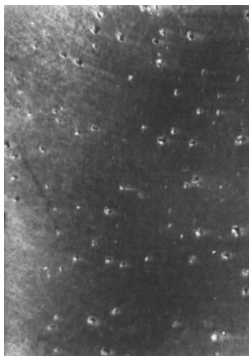


Fig. 13. Characteristic samples radiograms: casting with expressed porosity, higher patterns density and coat layers of higher thickness

On the other hand, moldings from the series with applied patterns of densities above 20 kg/m^3 and coat thickness above 1.5 mm have expressed subsurface and volumetric porosity too, Fig. 13. This indicates that the reasons for these types of defects are primarily the polystyrene pattern, and then the refractory coat and high casting velocity. It is in accordance with the works of other authors [6, 17, 24, 26, 27] expressing that the success of Lost foam casting process may be accomplished with polymer patterns with density below 20 kg/m^3 and with application of thinner layers of refractory coatings of different compositions based on silicon dioxide [30], zircon [17], talc [29], mica [26], with various components within the coating composition, with application of polysaccharide and an increased contents of CMC (1-5%). In difference to our research where we used pure components for refractory fillers, the works [4, 30, 31] showed that the combination of different refractory fillers such as zircon flour, alumina, silica, magnesite, clays, talc, chromite, mica, with application of various suspension agents for the system rheology control, may be used to attain significant effects of the increase of

both the coating refractoriness and gas permeability. All of this contributed to getting the quality of surface of moldings obtained by Lost Foam method increased. To apply this type of coating on large and complex forms, a flow coating method was successively developed [30].

4. Conclusion

Utilization of refractory talc, cordierite, zircon and mullite powders for coating production and application in Lost Foam casting process crucially depends on rheological properties of coatings, and, respectively, on sedimentary coating stability. These research results are optimum coating compositions and their preparation procedures with the goal of achieving positive effects on molding quality. Shiny and smooth molding surfaces are obtained, without distinctive defects of surface and volumetric porosity, folds, sintered sand in series of casting with polymer patterns of lower density, application of refractory coating layers of lower thickness and quartz sand for the casts of higher permeability. The achieved results of applying refractory coatings on mullite base are initial ones. Further studies should be done with the goal to determine the correlation between coatings composition and layer thickness and the molding structural and mechanical characteristics. By developing refractory coatings on talc, cordierite, zircon and mica base and optimizing technological parameters of Lost Foam casting process, the moldings with a priori defined quality i.e. desired properties could be obtained with significantly lower price cost with respect to the moldings founded in sand.

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