XI Naučno/stručni simpozij sa međunarodnim učešćem
„METALNI I NEMETALNI MATERIJALI“ Zenica, BiH, 21-22. april 2016.

INFLUENCE OF MICRONIZED GRINDING FILLER ON THE RHEOLOGICAL PROPERTIES OF COATINGS BASED ON CORDIERITE

UTICAJ MIKRONIZIRAJUĆEG MLEVENJA PUNIOCA NA REOLOŠKA SVOJSTVA PREMAZA NA BAZI KORDIJERITA

Marko Pavlović, dipl. inž., prof. dr. Marina Dojčinović, vanredni profesor, prof. dr. Tatjana Volkov-Husović, redovni profesor
University of Belgrade, Faculty of Technology and Metallurgy,
Karnegijeva 4, 11 000 Belgrade, Serbia

Key words: rheological properties, coating, cordierite, micronized grinding, activated filler

ABSTRACT
For the development of refractory coatings with controlled rheological properties the influence of micronized milling process was investigated. Change the properties of fillers based on cordierite, primarily on changing the size and shape of grain fillers, dispersion and stability of the suspension of the coating was investigated, also. Cordierite was obtained by synthesis in the solid state from the weight of the assembled components: kaolin, alumina, talc. For the characterization of cordierite these methods were applied: X-ray diffraction, differential thermal analysis, SEM and optical microscopy. Micronized grinding of filler was done in a vibro mill in time (min): 15, 30, 60. Optimization of composition and improvement of utility properties of the coating was achieved by a choice of sizes and form factors grain activated filler, using different components of coatings, as well as changing the process of developing coatings. The resulting coatings showed sizable sedimentation stability (precipitated matter content of less than 4 % at 24 h), satisfactory fire resistance and permeability to gases, easy application and adhesion to the surface of the sand mold, easy to adjust the thickness of the coating layers, no bubbles, no cracking and abrasion the dried coating layers.

Ključne reči: reološka svojstva premaza, kordijerit, mikro mlevenje, aktivirani punioc

REZIME
Za razvoj vatrostalnih premaza sa kontrolisanim reološkim svojstvima istraživan je uticaj procesa mikronizirajućeg mlevenja na promenu svojstava punioca na bazi kordijerita, pre svega na promenu veličine i oblika zrna punioca, disperznost i stabilnost suspenzije premaza. Kordijerit je dobijen sintezom u čvrstom stanju mase sastavljene iz komponenata: kaolin, glinica, talk. Za karakterizaciju kordijerita korišćene su metode: X-ray difrakcija, diferencijalno termijska analiza, SEM i optička mikroskopija. Mikronizirajuće mlevenje punioca vršeno je u vibracionom mlinu u vremenima (min): 15; 30; 60. Optimizacija sastava i poboljšanje upotrebnih svojstava premaza postignuto je izborom veličine i faktora oblika zrna aktiviranog punioca, primenom različitih komponenti premaza, kao i izmenom postupka izrade premaza. Dobijeni premazi pokazali su poveću sedimentacionu stabilnost (sadržaj istaloženih materija manji od 4% za 24 h), zadovoljavajuću vatrostalnost i propustljivost za

gasove, lako nanaošenje i prianjanje na površine peščanih kalupa, lako podešavanje debljine slojeva premaza, bez pojave mehurića, bez pucanja i otiranja osušenih slojeva premaza.

1. INTRODUCTION

Coatings are an integral part of casting. They prevent reactions on the contact surface of the mould - metal phase in pouring the metal, cooling and solidification. They provide crisp, and smooth surface finish, without glued sand and errors caused by penetration of metal into a mould (bumps, dents, rough surface, porosity, etc.) [1, 2]. Systematic studies of the properties of refractory coatings have shown that there are general conditions which coatings must meet: adequate fire resistance; low coefficient of thermal expansion; adequate permeability to gases; resistance to current liquid metal without tearing and penetration into the wall of the mould; that does not produce gases in contact with liquid metal; that is easy to apply and adhere to the surface of the mould; fast drying without cracking or abrasion coatings [3, 4].

In accordance with the specified quality requirements were selected cordierite (general formula $2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) as a filler coating. Important properties of cordierite are hardness by Mosh scale 7; density 1900-2200 kg/cm^3; low coefficient of thermal conductivity λ = (2.3 - 2.9) W/ m · K; low coefficient of linear thermal expansion, $\alpha = 1.7 x 10^6$ /ºC (20-1000); high resistance to thermal shock; relatively high melting temperature, with the possibility of application to 1380°C; high inertness to the liquid metal, does not develop fumes in contact with liquid metal; extreme resistance on liquid metal absorption [5].

In order to achieve high sedimentation stability of the slurry coating was performed micronized grinding filler based on cordierite. Bearing in mind that the finer particles of filler slowly accumulate in the suspension of the coating has been chosen filler grain size 100 % - $20 \cdot 10^{-6}$ m. In order to enable maintenance of the filler in a dispersed state in the coating have been added to various types and amounts of additives. Special attention was paid to the selection of the type and quantity of binding agent, and it was done in relation to the size and shape of the grain filler. The role of the binder is to facilitate networking particulate fillers and ensure good adhesion of the filler to the inflicted areas of sand moulds. As the solvent used is water and alcohol. The density refractory coating was 2000 kg/cm^3.

2. EXPERIMENT: MATERIALS AND METHODS

2.1. Synthesis of fillers based on cordierite

For the synthesis of cordierite raw material used is a mixture of 29% kaolin, 25% alumina, 46% talc. Chemical composition precursor is shown in Table 1. The starting materials, except kaolin, are fragmented to the upper limit of size 0.1 mm, and then mixed in relation:

$$2MgO : 2\,Al_2O_3 : 5SiO_2 \text{ composition.}$$

After homogenization, the mixture powder was pressed under the pressure of $1 \cdot 10^6$ Pa, and then sintered at a temperature of 1350°C in a time of 8h in the laboratory furnace in an oxidation atmosphere.

Table 1. The initial compositions of components for obtaining cordierite (%).

Pre-cursor	*SiO_2*	*MgO*	*Al_2O_3*	*Fe_2O_3*	*CaO*	*Na_2O+K_2O*	*LoI.*
Kaoline	*53.55*	*0.01*	*28.93*	*1.35*	*0.65*	*0.07*	*8.14*
Alumina	*0.14*	*0.01*	*95.96*	*0.09*	*0.16*	*0.05*	*3.20*
Talc	*60.15*	*31.40*	*1.18*	*0.28*	*1.5*	*0.10*	*6.5*

The synthesized cordierite (symbol: C) is subjected to a grinding in ball mill to a grain size of 100 % $-30 \cdot 10^{-6}$ m. Then the filler C is subjected to a process of milling in a vibratory mill at

a time (min): 15; 30; 60 wherein the fillers are obtained in different sizes and shapes of the grains:
• C_1 (100% -24 · 10^{-6} m, the mean particle shape factor 0.61);
• C_2 (100 % -20 · 10^{-6} m, the mean particle shape factor 0.67);
• C_3 (100% -12 · 10^{-6} m, the mean particle shape factor 0.71), respectively.

2.2. Characterization of filler based on cordierite
To identify the phase composition and structure of fillers based on cordierite was applied X-ray diffraction analysis. We used X-ray diffractometer model PW-1710, Philips. The characteristic temperature at which it comes to unwinding various reactions in the solid state of cordierite was performed in differential thermal analysis on the device Shimadzu DTA-50. Morphological and quantitative chemical analysis was performed by Scanning Electron Microscopy brand "JEOL" model JSM 6610 LV.

2.3. Synthesis of refractory coatings with activated fillers
For synthesis of refractory coating with activated fillers were selected fillers with smaller grain size. It was expected that the smaller grain fillers will be slowly sedimentation in the coating. The survey defined compositions of refractory coatings based on water and alcohol base.
Composition of refractory coatings based on water:
- Filler: mixture of activated filler ($C_2 + C_3$) in an amount of 92-93 %;
- Binder: dextrin in an amount of 3.5-5.5 %;
- Additives: cellulose (CMC) in an amount of 1-1.5 %.
Compositions of refractory coatings on alcoholic basis:
- Filler: mixture of activated filler ($C_2 + C_3$) in an amount of 93-95 %;
- Binder: resin ($C_{20}H_{30}O_2$) in an amount of 4.5-5 %;
- Additives: Bent 25 in an amount of 1-1.8 %.

The resulting refractory coatings were deposited on sand moulds pouring and by brush. During the application, homogenization of the coating suspension was carried out gently at mixing rate of 1 revolution/min. Drying aqueous coatings was carried out in air for 10 h.

2.4. Characterization of refractory coatings
In order to determine the distribution of fillers and binders with the analysis slurry coating composition prepared by observing the suspension of the coating on the polarization microscope for transmitted light JENAPOL, company Carl Zeiss Jena, the system for photomicrography STUDIO PCTV. Measuring the size and form factor of grain fillers was performed on 4000 grain, and the analysis was performed using the software program package OZARIA 2.5 (range 0-1). The division according to the shape factor is from 0.0-0.2 -*angular*; from 0.2-0.4 –*sub-angular*; from 0.4-0.6 -*sub-rounded*; of 0.6-0.8 *rounded* and 0.8-1.0 - *well rounded* grain shape.

3. RESULT AND DISSCUSION

3.1. Properties of fillers based on cordierite
In Table 2. shows the composition of the synthesized cordierite which after micronized milling used as a filler of refractory coatings.

Table 2. Composition of a filler C (%)

Mark	*SiO_2*	*Al_2O_3*	*MgO*	*Fe_2O_3*	*CaO*
C	*52.12*	*31.09*	*13.6*	*1.01*	*2.18*

In Fig. 1. shows the radiograph of the sample C, which shows dominant presence of cordierite, and smaller amounts are present cristobalit, spinel and quartz.
DTA curve of samples of cordierite is shown in Fig. 2. From the DTA curve it is noted the appearance of the endothermic effect at a temperature of 517.37°C and exothermic effects in temperature 1155°C, at lower temperatures, up to 250°C cordierite loses water, which corresponds to an endothermic effect.
In Fig. 3. shows the microstructure of filler C where clearly visible grains of irregular shape and different dimensions. Based on semi-quantitative chemical analysis, it was found that the analysed sample C contains smaller amounts of K, Ca and Fe apart from the main cationes Mg, Al and Si.

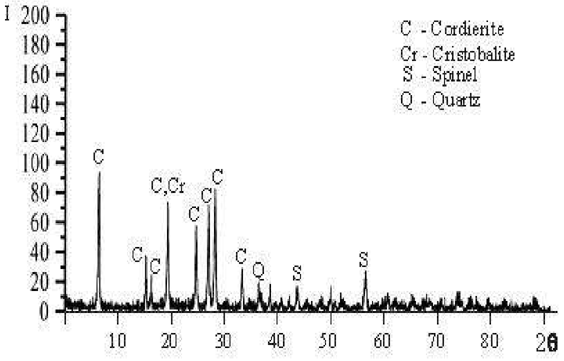

Fig. 1. XRD of starting powder sample.

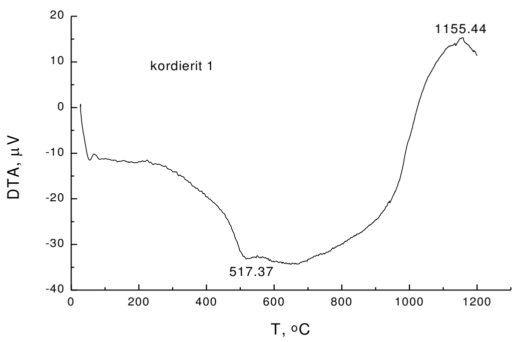

Fig. 2. DTA curve of cordierite.

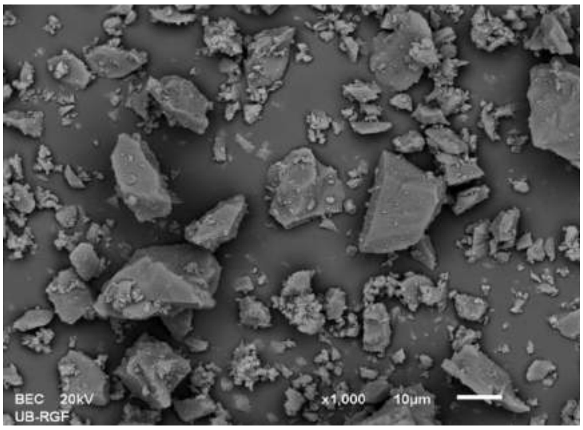

Fig. 3. Microphotography of refractory filler C.

Figures 4. - 5. show the results of measurements of the size and particle shape factor of activated fillers based on cordierite in time (min): 15; 30; 60s.

Based on data on the mean grain fillers, Figure 4., is expected that the finer grains more slowly to precipitate in suspension and the suspension of the coating easier to homogenize.

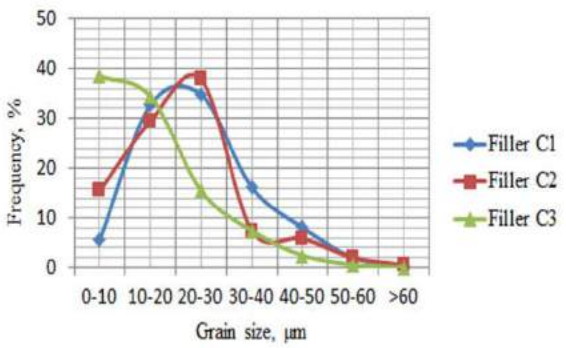

Fig. 4. Histogram of grain size distribution for the fillers C_1; C_2; C_3.

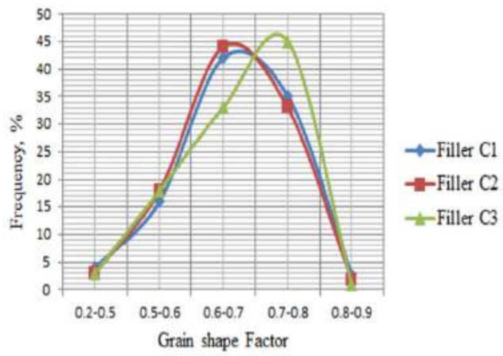

Fig. 5. Histogram of grain shape factor for the fillers C_1; C_2; C_3.

Therefore, it is chosen for the preparation of the coating mixture of filler:

• C_2 (grain size of $20 \cdot 10^{-6}$ m, the mean particle shape factor of 0.67) in an amount of 80-82% and
• C_3 (grain size of $12 \cdot 10^{-6}$ m, the mean particle shape factor of 0.71) in the amount of 11-12%.
Based on the data on the shape factor, Fig. 5., grain of fillers C_1; C_2; C_3 belong to the category of rounded grains.

3.2. Properties of refractory coatings whit activated fillers
Microphotographs of the obtained suspension of refractory coatings based on water-based activated filler (Fig. 6.) and of the alcohol base (Fig. 7.), showed a homogeneous distribution of the refractory filler in the coating suspension.

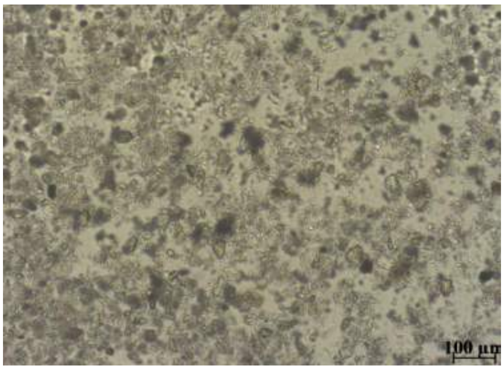

Fig. 6. Microphotography of suspension water based coatings

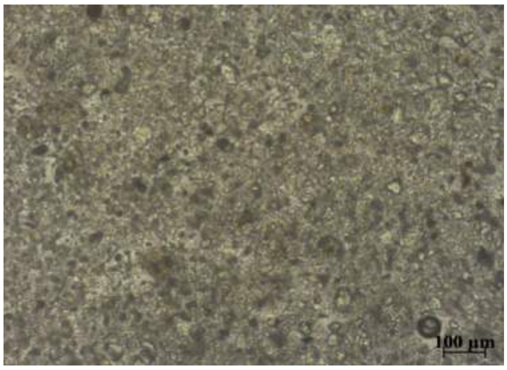

Fig. 7. Microphotography of suspension of alcohol based coatings.

Sedimentation stability tests of the coating suspension showed different results for the amount of the precipitated substance. When the coating on alcohol base amount deposited matter was below 4%, and aqueous coatings 4 - 4.5%, which, according to national standards for this type of refractory materials [3,4] considered satisfactory.

4. CONCLUSION

As a result of these studies were determined optimal composition of refractory coatings with mechanically activated filler based on cordierite. All here investigated refractory coatings gave satisfying properties, with low sedimentation of refractory filler (precipitated matter content of less than 4% at 24 h).

5. REFERENCES

[1] Monroe, R.: Expandable Pattern Casting, AFS Inc., Schaumburg, II, 1994.,

[2] Akbarzadeha Y., Rezaaeia M., Akbar Babaluoa A., Charchia A., Azimia H. R., Bahlulib Y.: Microstrukture, permeability and rheological behavior of lost foam refractory coatings, Surface&Coating Technology, 202, 4636-4643, 2008.,

[3] Serbian Standard SRPS B.H9.102:1980, Foundry Means – Materials for mould and core dressing – Classification – Technical requirements – Testing Methods.,

[4] Serbian Standard: SRPS EN 12890:2000/CEN/TC 190, Founding – Patterns, Pattern Equipment and Core Boxes for the Production of Sand Moulds and Sand Cores.,

[5] Moore, R. E.: Refractories, Structure and Properties, Encyclopedia of Materials: Science and Technology, 8079-8099, University of Missouri-Rolla, Rolla, Missouri, USA, 2001.