SYNTHESIS AND CHARACTERISATION OF THE MULLITE-BASED PROTECTIVE COATINGS

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Abstract

Composition and production procedures of the mullite-based coatings for the protection of metal surfaces in the conditions of cavitation, wear and corrosion were investigated. An ultrasonic vibration method according to the ASTM G32 standard was used to characterize the coating. The change in the mass of samples as a function of the cavitation time was monitored and the cavitation rate was determined. The occurrence and development of the coating surface damage were monitored using a scanning electron microscope. Based on the value of cavitation rate and analysis of the surface damage morphology, the cavitation resistance of coating was determined. Coatings have shown the satisfactory resistance in the conditions of cavitation and possibility of application for the protection of parts of equipment in metallurgy and mining.

Keywords*: protective coatings, mullite, cavitation, cavitation resistance*

1 INTRODUCTION

Mullite is a ceramic material containing about 72% Al_2O_3 and 28% SiO_2 (chemical formula $Al_6Si_2O_{13}$). It rarely appears as the natural mineral. Production processes are designed to maximize the formation of a complex type of mullite ($3Al_20_3{\cdot}2SiO_2$). The synthesis of mullite is influenced by a number of parameters: purity, crystal shape and particle size of reactants Al_2O_3 and SiO_2 in the initial mixture for obtaining mullite, temperature and reaction time, type and amount of additives affecting the synthesis process, application of mineralizers (LiF, NaF, Cr_2O_3, Na_2WO_3, TiO_2) [1,2]. Modern mullite synthesis focuses on the sol-gel methods that allow obtaining mullites with a homogeneous structure. The sol-gel methods open up the possibility of developing the production of composites based on mullite, thin films and mullite fibers with any purity of phases, phase distribution and grain morphology [2,3]. Mullite is industrially produced in the transparent, porous and non-porous forms. These materials can have high optical and electronic properties [4]. Mullite ceramics can be an excellent construction material thanks to its excellent properties, such as: good stability of volume and strength at high temperatures, high resistance to creep and thermal expansion and conductivity, high resistance to abrasion and erosion, corrosion resistance to slag, alkali, liquid metals, corrosive gases, resistance to cavitation. All this makes it an attractive material for the production of both traditional ceramics and advanced ceramics [2,5,6]. This work investigates the use of mullite as a filler in the refractory coatings for the protection of metal structures.

2 EXPERIMENTAL

2.1 Materials

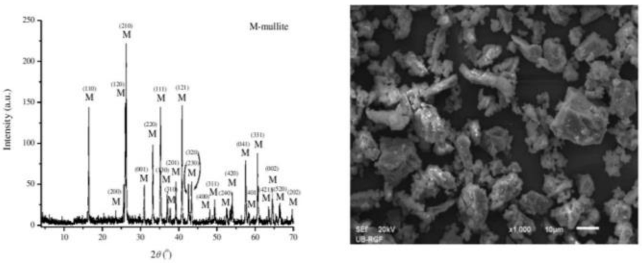
In the research of composition the protective coatings, a mullite obtained in previous researches of the synthesis of refractory coatings for use in the foundry was used as a refractory filler [1]. For the needs of these researches, the filler was additionally ground to a grain size of 15 μm. Important properties of mullite, based on which it was chosen as a filler in the composition of protective coatings are: density 3.20 g/cm^3, high hardness 7.5 by the Mosh scale, relatively low coefficient of thermal expansion ($6 \cdot 10^{-6}$ °C^{-1}), coefficient of thermal conductivity (1.3 W/m·K); it is corrosion resistant and resistant to high temperatures [1,4]. In addition to the mullite-based fillers, a protective coating based on epoxy resin, an organic solvent and organic additives, were used.

2.2 Methods

To determine the resistance properties of the obtained protective coatings based on mullite, an ultrasonic vibration method with a stationary sample according to the standard ASTM G32 was applied [7]. The testing methodology was described in previous papers [6,8]. The aim of the test was to determine the quality of coatings and possibility of application for the protection of metal surfaces in the conditions of wear, corrosion and elevated temperatures. For testing, the obtained coatings were applied in two layers on a metal plate. After drying the coating in the air for 60 min, samples with the coating were fixed with holders located on the bottom of water bath. In accordance with the standard, the characteristic parameters for this method were chosen: vibration frequency 20 kHz; amplitude at the top of concentrator 50μm; distance between the test sample and concentrator 0.5 mm; water temperature in the bathroom 25°C; water flow in the bathroom 5-10 ml/s. Interval of exposure of samples to cavitation (min): 15; 30; 45; 60. The loss of coating weight as a function of cavitation time was monitored. Cavitation rate was determined as an indicator of coating resistance under the cavitation loads. The X-ray diffraction analysis was used to characterize the refractory filler. Morphology of the sample surface damage was monitored using the scanning electron microscopy. Based on the values of cavitation velocity and analysis the morphology of surface damage, the cavitation resistance of the tested protective coatings was determined.

3 RESULTS AND DISCUSSION

Figure 1a shows an XRD of a mullite - based filler showing the dominant presence of mullite. Figure 1b shows a SEM microphotograph of the filler where it is clearly shown that this material occurs in the irregular shapes of different dimensions.



a) b)

Figure 1 *Mullite-based filler: a) X-ray diffraction; b) SEM microphotograph*

During the research, the composition of protective coatings, based on mullite, was defined: 85 - 90 % refractory filler; 7.5 - 11 % epoxy resin-based binder; with the organic additives in order to improve the rheological properties of coating and organic solvent (toluene). Figure 2 shows the surfaces of the coating samples before and during the cavitation test. A small number of pits was observed after 30 min of exposure, which change slightly during further testing up to 60 min. The pits slightly change the shape and dimensions during cavitation testing, which indicates the increased resistance of the coating surface to cavitation, Figure 3. Figure 4 shows a cavitation rate diagram for mullite - based coatings.

0 min 15 min 30min 45min 60 min

Figure 2 *Surface photographs of coating samples before and during the cavitation test*

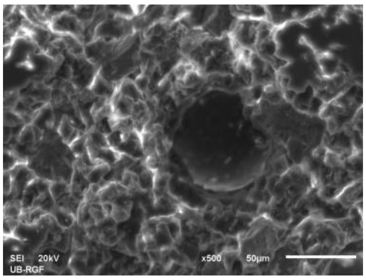


Figure 3 *SEM microphotograph of a mullite-based coating sample after 60 min of cavitation*

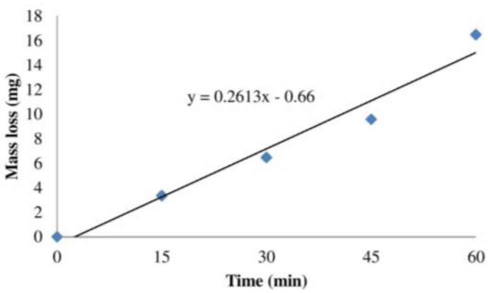


Figure 4 *Cavitation rate of mullite-based protective coating samples*

A short incubation period of about 3 min was observed, when there was no mass loss, Figure 4. After that, slight damage to the coating surface occurs, which develops at a low speed. This can be seen in the small mass loss, of 16 mg in 60 minutes of cavitation. Calculated cavitation rate was V=0.27 mg/min. The formation and development of surface damage takes place at a relatively low cavitation rate. Figure 3 shows the presence of damage to the surface of the sample, showing the pit of the relief surface and uneven edges.

Satisfactory cavitation resistance of the tested protective coatings can be explained by the high properties of the filler based on mullite, primarily high hardness and strength, resistance to high temperatures and under the action of pressure.

4 CONCLUSION

Satisfactory resistance of the tested coatings based on mullite with the possibility of application for the protection of metal surfaces under conditions of cavitation was shown. Further research to improve the cavitation resistance of coatings should focus on changing the composition of coating, application of the new organic additives, changes in coating procedures, all in order to improve the rheological properties of coating and durability of coating under the cavitation load.

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