



## QUALITY MANAGEMENT OF PROTECTIVE COATINGS BASED ON ZIRCONIUM SILICATE

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**Abstract:** *The composition and production procedures of refractory coatings based on zirconium silicate for the protection of metal structures in conditions of wear, cavitation and corrosion were investigated in the paper. The composition of the coating was defined with a filler based on zirconium silicate, a binder based on epoxy resin, organic additives and an organic solvent. A method for quality control of obtained coatings was investigated using the ultrasonic vibration method with a stationary sample according to the ASTM G 32 standard.*

*The following methods were used to characterize the obtained filler samples: X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). The change in the mass of the samples as a function of the cavitation time was monitored and the cavitation rate was determined. The formation and development of damage to the coating surface was monitored using a scanning electron microscope. The obtained test results showed a satisfactory resistance of the protective coatings to the effect of cavitation, with small damage to the coating surface and a cavitation rate of 0.6 mg/min. Tests have shown that the application of the ultrasonic vibration method is suitable for a quick assessment of the quality of protective coatings during synthesis and a forecast of their resistance and stability during exploitation in the protection of equipment parts in mining and metallurgy.*

**Keywords:** *zirconium silicate, protective coatings, cavitation resistance*

### 1. INTRODUCTION

Equipment in mining and metallurgy is exposed to rigorous exploitation conditions. Various coatings and coatings with a good combination of mechanical properties (hardness and strength), resistance to wear, resistance to corrosion and cavitation are used to protect the metal parts of the equipment [1, 2]. Cavitation is the phenomenon of formation, growth and implosion of vapor or gas bubbles in a flowing liquid. This creates shock waves and micro-jets that damage the surfaces of hydrosystem elements. The formation and development of material surface damage under the effect of cavitation depends on the type of material, its structure and properties. In the cavitation zone, material damage is also affected by the presence of electrochemical processes, chemical corrosion, and high temperatures and pressures generated locally, in very short time intervals, contribute to additional material damage [3]. The destruction of materials under the effect of cavitation is a surface degradation process, so the application of different types of refractory coatings contributes to increasing the resistance of metal surfaces, their protection and safety in exploitation [4-6].

In the paper, protective coatings based on zirconium silicate with epoxy resin as a binder were investigated, which represent a novelty and have not been used in practice so far. Compositions and procedures for the production of protective coatings were investigated. A coating characterization method was developed using the ultrasonic vibration method with a stationary sample according to the ASTM G 32 standard in order to assess the cavitation resistance of the investigated coatings and the possibility of application under cavitation conditions [7].

### 2. EXPERIMENTAL

#### 2.1. Materials

Zircon ( $ZrSiO_4$ ) is an attractive refractory material, which is widely used for the synthesis of new materials due to its properties: high melting temperature or high refractoriness, low coefficient of thermal and linear expansion, high resistance to thermal shock. An important application of this refractory material is in foundries for the manufacture of sand molds and cores for casting high-quality steel castings, for the production of refractory coatings for molds and cores, as well as for the manufacture of ceramic shells in precision casting [8, 9].

Zirconium silicate used in the composition of protective coatings as a filler is of high purity (99,99 wt. %  $ZrSiO_4$ ), grain size  $20\mu m$ . Filler samples were obtained by mechanical processing, purification and grinding of zircon sand. In the composition of protective coatings, the following were used (%): refractory filler based on zirconium silicate (80-85); binder based on epoxy resin (14-15); additives (1-1,2); organic solvent to a coating density of  $\rho=2,5g/cm^3$ . During the synthesis of the coating, all components from the composition of the coating were gradually added with constant mixing. For testing, the obtained protective coatings were applied in two layers on a metal plate, drying time in air for 60 min.

## 2.2. Methods

During the experiment to determine the parameters of the synthesis of refractory coatings based on zirconium silicate and to evaluate the quality of the obtained samples, the ultrasonic vibration method was applied with a stationary sample according to the ASTM G32 standard [7]. To evaluate the resistance of the sample to the effect of cavitation, the surface of the sample was examined before and during the test. The surfaces of the samples were recorded on a scanning electron microscope in order to monitor and analyze the morphology of surface damage. Test methodology described in earlier works [2, 6]. In accordance with the standard, the characteristic parameters for this method were chosen: vibration frequency 20 kHz; amplitude at the top of the concentrator  $50\mu m$ ; the distance between the tested sample and the concentrator is 0.5mm; water temperature in the bathroom  $25^\circ C$ ; water flow in the bathroom 5-10 ml/s. Interval of exposure of samples to the effect of cavitation (min): 15; 30; 45; 60. The loss of coating mass as a function of cavitation time was monitored. After each test interval, the change of the sample was measured with an analytical precision balance 0.1mg. The cavitation rate was determined as an indicator of the resistance of the coating under conditions of cavitation loads. The method of X-ray diffraction analysis was used to characterize the refractory filler. The morphology of the surface damage of the samples was monitored using scanning electron microscopy. Based on the value of the cavitation rate and the analysis of the morphology of the surface damage, the cavitation resistance of the examined protective coatings was determined. This made it possible to control the quality of the obtained coatings and predict their behavior under exploitation conditions.

## 3. RESULTS AND DISCUSSION

Figure 1a shows the XRD of the filler based on zirconium silicate showing the dominant presence of  $ZrSiO_4$ . Figure 1b shows a SEM microphotograph of the filler, where it can be seen that this mineral occurs in irregular forms with a characterization shell fracture and different dimensions. The morphology of the zirconium silicate grains shows that they are suitable for even packing of the grains with each other, which is a condition for obtaining continuous coating layers and good protection of the surfaces on which the coating is applied.

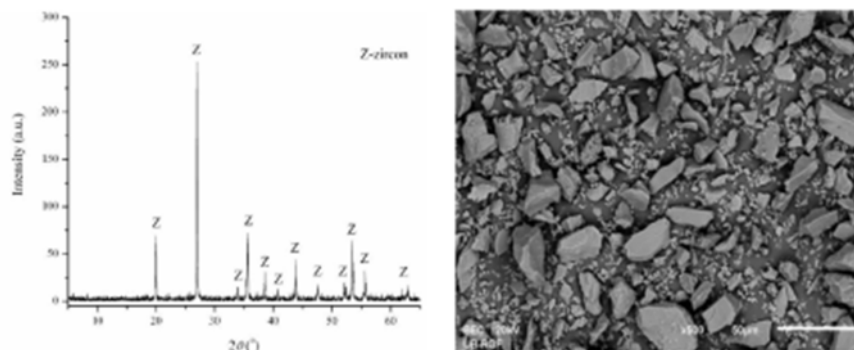


Figure 1. Zircon based filler: a) X-ray diffraction; b) SEM microphotograph.

Figure 2 shows the surfaces of zirconium silicate coating samples during the cavitation effect test. After 30 min of exposure, the appearance of a smaller number of shallow pits is observed, which change slightly during further testing up to 60 min. A small change in the shape and dimensions of the pits during testing indicates and increased resistance of the coating surface to the effect of cavitation, Figure 3.

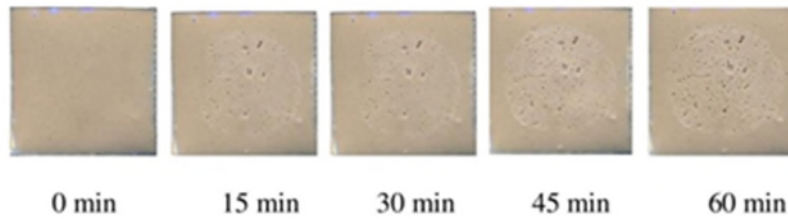


Figure 2. Surface photographs of coating samples before and during the cavitation test.

In order to assess the effect of the coating components on the resistance properties, primarily the effect of the refractory filler, the cavitation rate was determined as a measure of the resistance of protective coatings during exposure, Figure 4. A short incubation period (about 7 min) is observed in which there is no damage to the coating and no loss of mass. With further exposure, the surface of the coating changes slightly, the damage to the surface occurs at a low speed. The mass loss of the coating samples is less; in 60 min the mass loss is below 30 mg. Calculated cavitation rate is 0.6 mg/min.

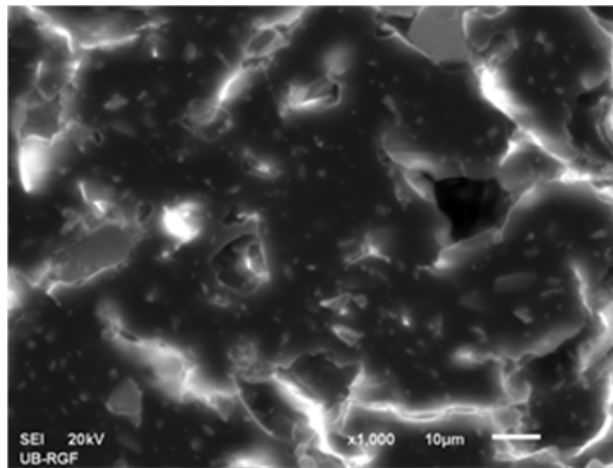


Figure 3. SEM microphotograph of a zircon-based coating sample after 60 min of cavitation.

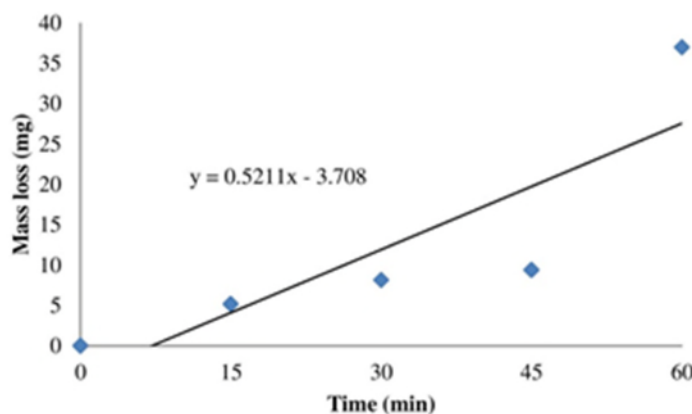


Figure 4. Cavitation rate of zircon-based protective coating samples.

The satisfactory properties of the coatings resistance to the effect of cavitation can be explained by the high properties of the filler based on zirconium silicate, above all by its high hardness and strength, high fire-resistant properties. In order to improve the resistance properties of the coating, for future research, smaller filler grain diameters (15  $\mu\text{m}$ ) should be used, which will contribute to the creation of continuous layers of protective coatings on the surface of metal structures and certainly better protection against harmful effects during exploitation.

#### 4. CONCLUSION

The research results showed satisfactory cavitation resistance of protective coatings based on zirconium silicate. In order to improve the properties of the coating and the possibility of their application in practice for the protection of equipment in mining and metallurgy, it is necessary to achieve the optimal composition of the coating, apply suitable organic additives in order to improve the rheological properties of the coating and increase the resistance of the coating to harmful influences during exploitation.

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#### REFERENCES

- [1] M. Dojčinović, V. Đorđević: The possibility of polymer materials application in reparation of hydraulic machinery parts damaged by cavitation, 3rd International Conference Research and Development in Mechanical Industry RaDMI 2003, Proceedings, p. 19-23
- [2] M. Pavlović, M. Dojčinović: Cavitation damage of refractory materials, Monograph, Akademskamisao, Belgrade, (2020), p.165. (in Serbian)
- [3] J. P. Franc, J. M. Michel (Eds): Fundamentals of cavitation, Series Fluid Mechanics and Its Applications, vol.76, 2004, XXII, 306 p., Kluwer Academic Publishers, New York, Boston, Dordrecht, London, Moscow.
- [4] T. Fukai, K. Matsumoto: Cavitation Erosion of Ceramic Coatings and its Evaluation Method, Research Center, Toyo Engineering Corporation, 11, 25, 2009.
- [5] N. Qiu, L. Wang, S. Wu, D. S. Likhachev: Research on cavitation erosion and wear resistance performance of coatings, Engineering Failure Analysis 55, 208–223, 2015.
- [6] M. Pavlović, M. Dojčinović, A. Sedmak, I. Martić, F. Vučetić, Z. Aćimović: Synthesis and characterisation of the mullite-based protective coatings, 53 IOC Bor 2022, 3-5 October 2022, Proceedings, pp. 147-150, ISBN 978-86-7827-052-9.
- [7] ASTM Standard G32-98 Standard, Test Method for Cavitation Erosion Using, Vibratory Apparatus, Annual Book of ASTM Standards, 2000, pp. 107–120.
- [8] F. L. Pirkle, D. A. Podmeyer, Zircon: origin and uses, Transactions, vol. 292, Society for Mining, Metallurgy and Exploration, Inc., USA 1988.
- [9] Aćimović Z., Terzić A., AndrićLj., Pavlović M.: Comparison of refractory coatings based on talc, cordierite, zircon and mullite for Lost foam casting, Materials and Technology 49 (2015) 1, 157-164.