

On the structure of cement stone with fillers from metallurgical waste

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Abstract. The results of studies of the grinding of metallurgical slags in ball mills and data on the optimization of the grinding process are presented. The results of studies of the influence of the dispersion of the filler and the degree of filling on the formation of the strength of the cement stone are presented.

1 Introduction

For the production of modern high-quality and high-strength concretes of a new generation, mineral powders of natural and technogenic origin are widely used [1]. In Europe, composite binders are produced with a content of up to 35% of mineral additives [1-2].

The distribution of composite binders with a high content of mineral additives is due, firstly, to the need for energy saving in the production of expensive Portland cement. According to statistical data, it is known that the production of 1 ton of Portland cement clinker requires, on average, 223 kg of standard fuel, while for drying and mechanical activation of mineral additives only about 20 kg/t [3].

Secondly, the introduction of mineral additives opens up the possibility of a significant increase in the hydraulic reactivity of the binder, as a result of which a positive effect on the formation of the structure of cement stone and concrete is ensured, and the physical-mechanical and construction-technical properties of the material being designed are improved [2].

Mineral additives (MA) obtained from natural (diatomites, opal-like siliceous schists, gliezhs, tuffs, volcanic ash, etc.) or technogenic raw materials (blast-furnace slags, fuel ash, microsilica, metallurgical slags, etc.) are powdered materials used as a component of concrete (mortar) mixtures and differ from fine aggregates in particle sizes from 0.16 mm to several microns or less [4]. One of the most common man-made raw materials used as active mineral additives for cement concretes and mortars is metallurgical waste. The main criterion that predetermines their use as a modifier is the presence of silicon dioxide in the composition.

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Amorphous silica, entering into a chemical reaction with cement minerals, forms new strong compounds that compact and strengthen cement structures by reducing capillary pores and binding calcium hydroxide $\text{Ca}(\text{OH})_2$. It should also be noted that all processes of directional structure formation occur at the optimum dispersion of the mineral additive.

At the metallurgical plants of Uzbekistan, large-tonnage wastes have accumulated that require disposal and are valuable modifiers in improving the physical and mechanical characteristics of cement stone [5].

The article presents the results of studies on the mechanical activation of metallurgical slags of the Foundry and Mechanical Plant JSC "O'zbekiston temir yo'llari" and experimental data from studies to establish the effect of mechanically activated filler on the properties of cement binder.

2 Research methods and characteristics of raw materials

The process of grinding waste from metallurgical production was carried out in a laboratory ball mill ШЖМ-100 in shock-attrition mode. The mass and number of balls of the mill are shown in fig. 1. In the study, waste from metallurgical production was dried to a constant weight at a temperature of $\pm 105^\circ\text{C}$ and loaded into drums in the amount necessary for grinding. The grinding process was carried out before the onset of particle aggregation. [6].

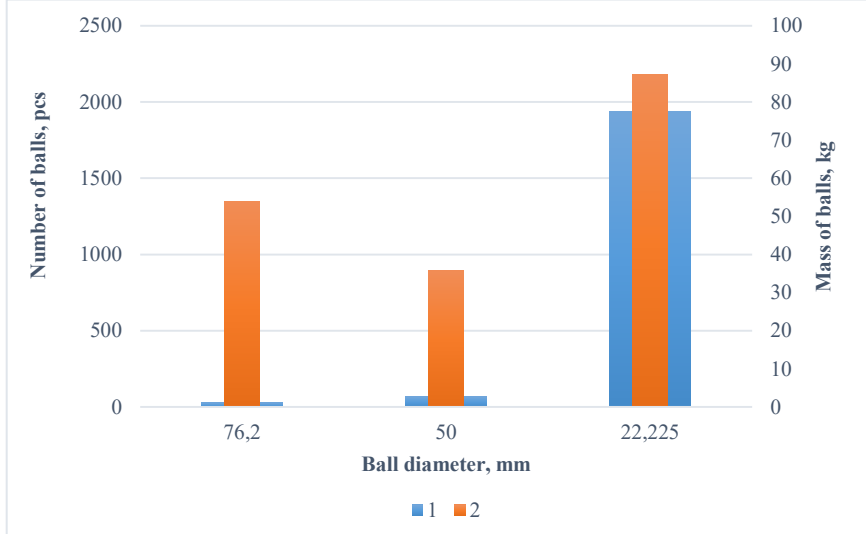


Fig.1. Number and diameter of balls when loading into a ball mill:
1-number of balls; 2-mass of balls.

The fineness of grinding was evaluated by the specific surface on a ПСХ-11А surface meter. The phenomenon of aggregation was determined by sifting through a No. 008 sieve. In experimental studies, Portland cement grade M400 D0 of the Akhangaran cement plant was used. The chemical composition of fillers and cement is given in table. 1.

Table 1. Chemical composition of waste from metallurgical production and Portland cement

Compounds, %	SiO ₂	Al ₂ O ₃	MnO	FeO ₂	MgO	CaO	TiO ₂	ZnO
Portland cement PC400 D0	22,1	4,5	-	4,2	1,7	65,8	-	-
Steelmaking slag (SS)	48,21	19,27	17,62	7,63	5,99	0,27	0,651	0,133
Casting and molding waste (CMW)	91,56	4,58	0,232	1,12	0,1	0,2	0,061	0,022

To establish the optimal value of the specific surface area and the degree of filling, samples of 4x4x16 cm in size were made from fine-grained concrete containing sand with a particle size modulus $M_{kp} = 2.5$, and after normal hardening at the age of 28 days, the compressive and bending strength was determined. When preparing a cement-sand mortar, water was introduced in two steps. First, half of the mixing water was sprayed and the cement-sand mortar was moistened, then the remaining part of the water was mixed with the moistened mortar. After the set hardening mode, the samples were tested on a CT-P2000 hydraulic press [7-8].

3 Results and discussions

The results of studies of the grindability of metallurgical production waste are presented in fig. 2-4.

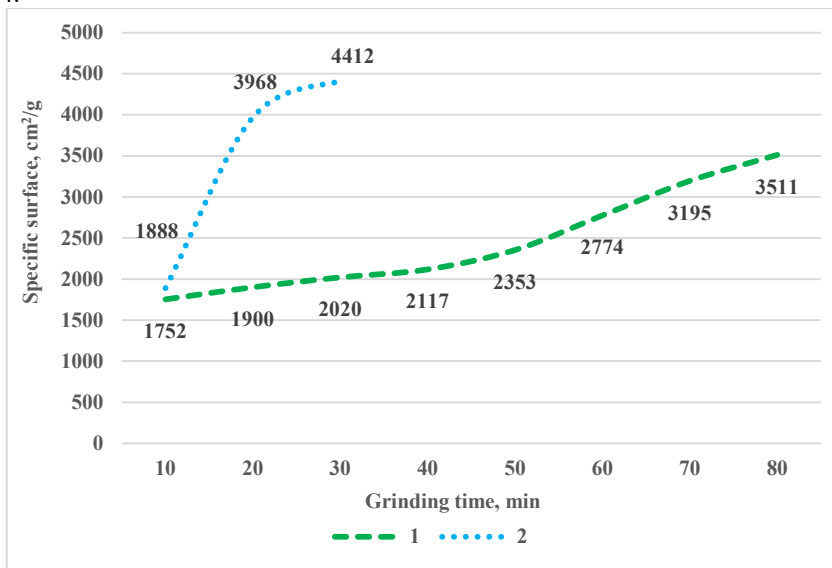


Fig. 2. Influence of the duration of grinding on the change in the value of the specific surface area (1) SS and (2) CMW

The obtained results of changes in the specific surface area show that after 80 min of SS grinding, the specific surface area reaches a value of 3511 cm²/g. In CMW in 30 minutes you can reach $S_{ss} = 4412$ cm²/g.

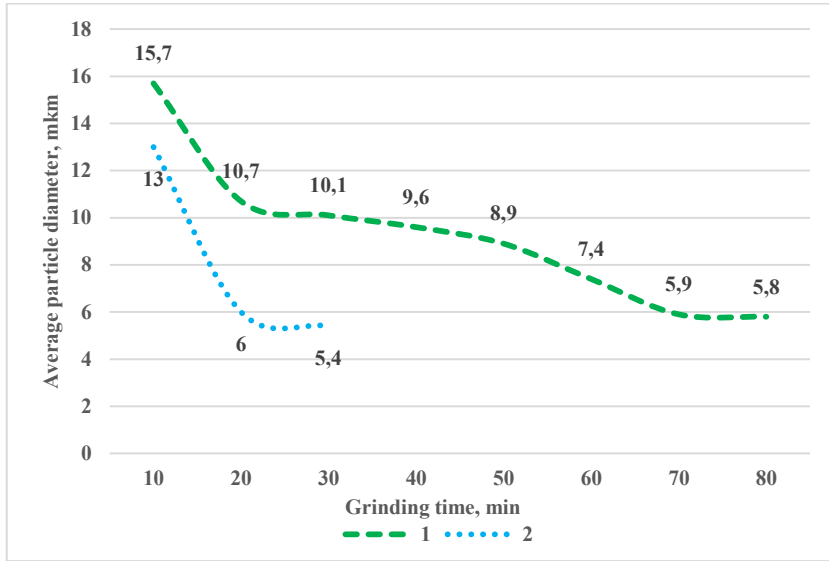


Fig. 3. Influence of the duration of grinding on the change in the average particle diameter of (1) SS and (2) CMW

At the same time, the average diameter of the SS particles decreases from 15.7 mm to 5.8 mm, and the average diameter of the CMW particles decreases to 5.4 mm.

With a decrease in the size of each particle, the total surface of the crushed substance increases rapidly, while the volume of particles remains constant when the fragments are added. The surface, which rapidly increases with grinding, has a reserve of surface energy, which is subsequently consumed when forming products from a mixture with reactions occurring along the interfaces. Upon reaching a certain dispersion limit, the potential energy of the surface may increase, which often leads to spontaneous aggregation of particles with a decrease in the specific surface area and an increase in the inhomogeneity of the initial product [9-10].

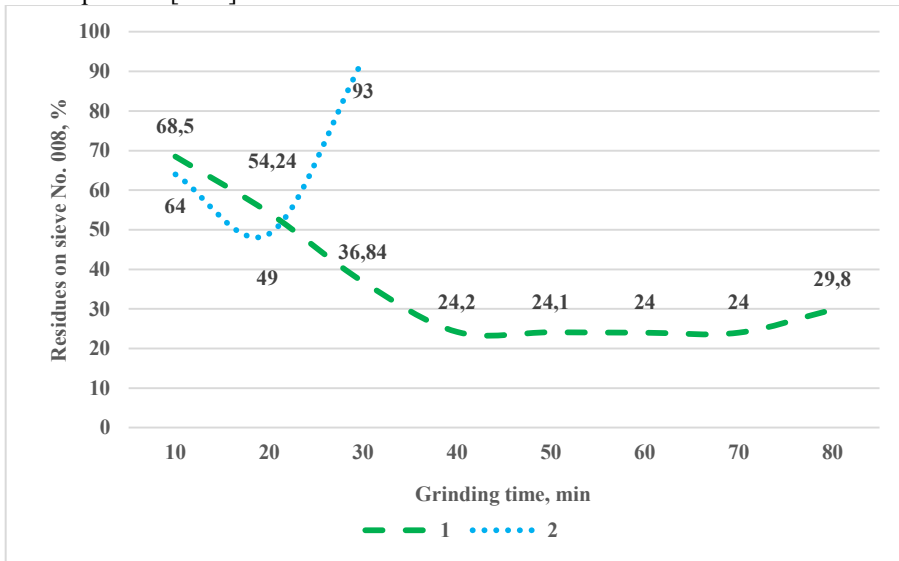


Fig. 4. Influence of grinding duration on particle aggregation (1) SS and (2) CMW

With a grinding time of 70 minutes, the phenomenon of aggregation and sticking of particles is observed, as evidenced by the data shown in Fig. 4. For 40 minutes of grinding SS, the residues on sieve No. 008 are reduced from 68.5% to 24.2%. After 70 minutes of grinding, the amount of residue begins to grow and the phenomenon of particle aggregation occurs. The residue on sieve No. 008 CMW is reduced from 64% to 49%.

From numerous studies of the authors [6], it follows that the reactivity of mechanically activated substances must be evaluated not only by the size of the specific surface area and the degree of amorphization of particles, but also by changes in their shape and relief, and most importantly, by the concentration of active surface Brønsted acid sites, since they have the most significant effect on the interaction with binders.

It has been established that with an increase in the fineness of grinding of mineral materials, there is an increase in the number of active adsorption centers on the surface of the resulting powders, which are responsible for their reactivity. However, this trend is observed up to a certain specific surface area (S_{sp}), after which the process slows down considerably. Therefore, when using a freshly ground filler, it is important to determine precisely the rational value of fineness, above which the activity of the surface of its particles increases insignificantly and, therefore, further milling is impractical [6].

When storing freshly ground mineral fillers, the concentration of active centers decreases. Surface decontamination is carried out due to the adsorption of water molecules and largely depends on the humidity of the environment.

The mechanism of this process is as follows. On the surface of the filler, water dissociates into protons and hydroxyls, since its proton-donor surface (and other impurity centers) participate in the formation of hydrogen bonds with H_2O molecules. This process can be not only a consequence of the adsorption of water molecules, but also occur as a result of the capture of H^+ and OH^- ions or radical products of H and OH by the surface [7]. The activity of the filler can also be affected by the adsorption of oxygen or nitrogen molecules in the air. In this case, the active centers are “clogged”, and the molecules O_2 and N_2 , being an electron donor, they change the concentration of the latter on the surface [8].

Taking into account the above theoretical prerequisites, the influence of the shelf life of fillers on the strength of the cement stone was studied (Fig. 5).

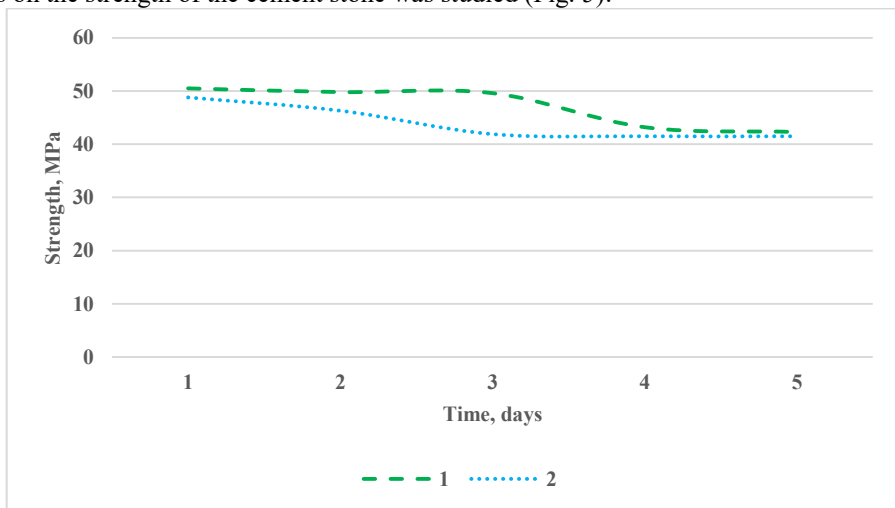


Fig. 5. Decrease in filler activity over time as a result of dampening; 1-SS: 2- CMW

On fig. 4 shows that the number of active centers on the surface of the filler for SS decreases most intensively after three days of exposure to air, and a monotonous drop in strength is observed. As for the finely ground mineral filler based on CMW, the

concentration of active centers drops after a day of storage under the same conditions, after which the strength index stabilizes.

Of undoubted interest is the question of the influence of the specific surface area of SS and CMW fillers on the formation of the strength characteristics of cement stone. To study this issue, samples were prepared from fine-grained concrete with $W / C = 0.45$. In order to establish rational values of the specific surface of the filler and the degree of filling of the cement binder, studies are given with different values of the specific surface of the SS and CMW of the filler: 1000, 2000, 3000, 4000 cm^2/g and the degree of filling 5% 10%, 15%, 20%, 25 %, 30%, 35%, 40%.

The strength characteristics of cement binder with different degrees of filling and specific surfaces were studied on standard beams $4 \times 4 \times 16$ in size. After statistical processing of the results of experimental data, the following graphical dependencies were obtained (Fig. 6-9) [12].

From fig. 6-8 it can be seen that with an increase in the degree of filling and dispersion, the compressive and flexural strength of the cement stone increases monotonously, and an extreme indicator was registered for both SS and CMW at $S_{sp} = 3000 \text{ cm}^2/\text{g}$ and a degree of filling of 30% by weight of the binder. Strength when filled above 30% is declining. A further increase in the degree of filling leads to a natural decrease in the strength of the cement stone, due to a decrease in the proportion of the original cement. [11].

In addition, an increase in the specific surface of fillers above 3000 cm^2/g also negatively affects the mechanical properties. The reason for this is the sticking and aggregation of particles, which occurs due to the polarization of the surface during mechanical activation. These results are in good agreement with the data shown in Fig. 4.

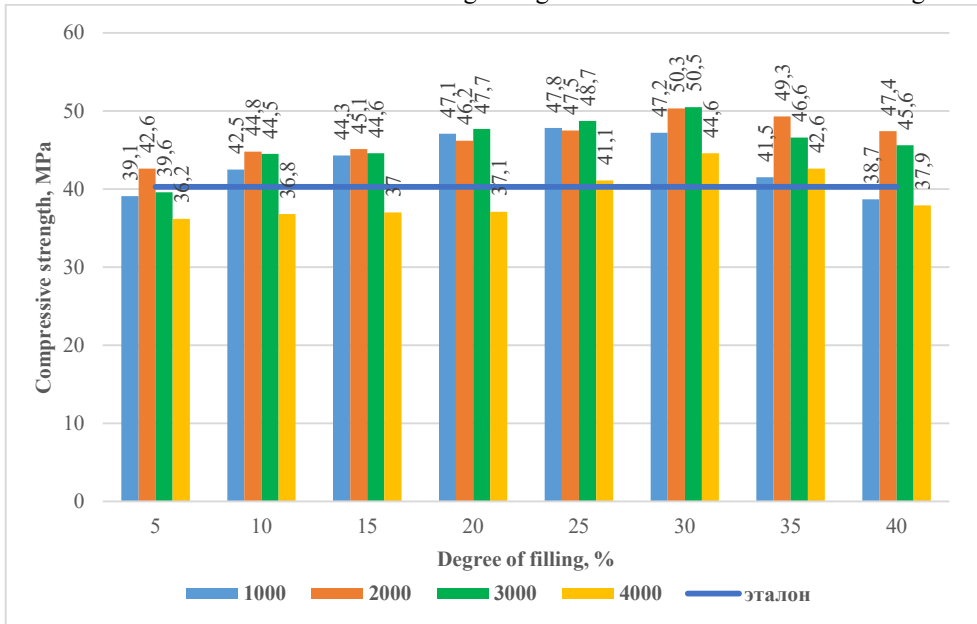


Fig.6. Influence of the specific surface area and degree of filling of the SS on the compressive strength of the cement stone

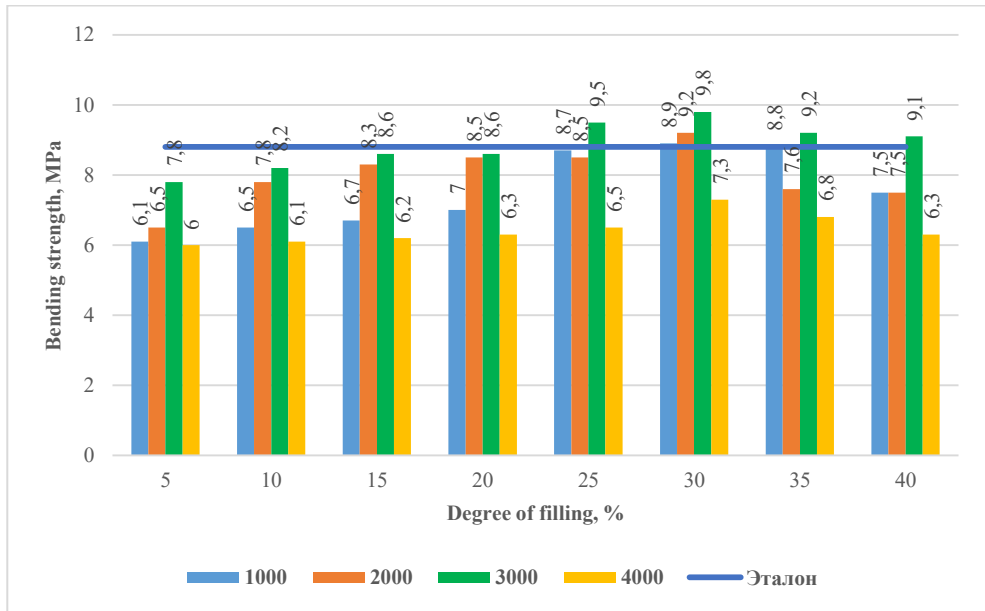


Fig. 7. Influence of the specific surface area and degree of filling of the SS on the flexural strength of the cement stone

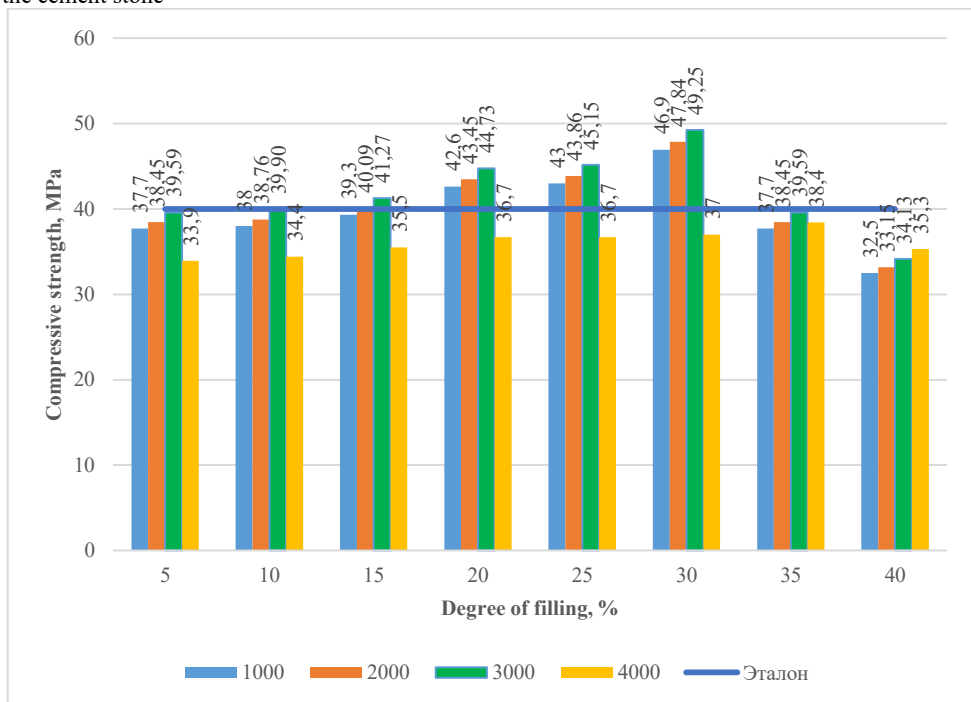


Fig.8. Influence of the specific surface area and the degree of filling of the CMW on the compressive strength of the cement stone

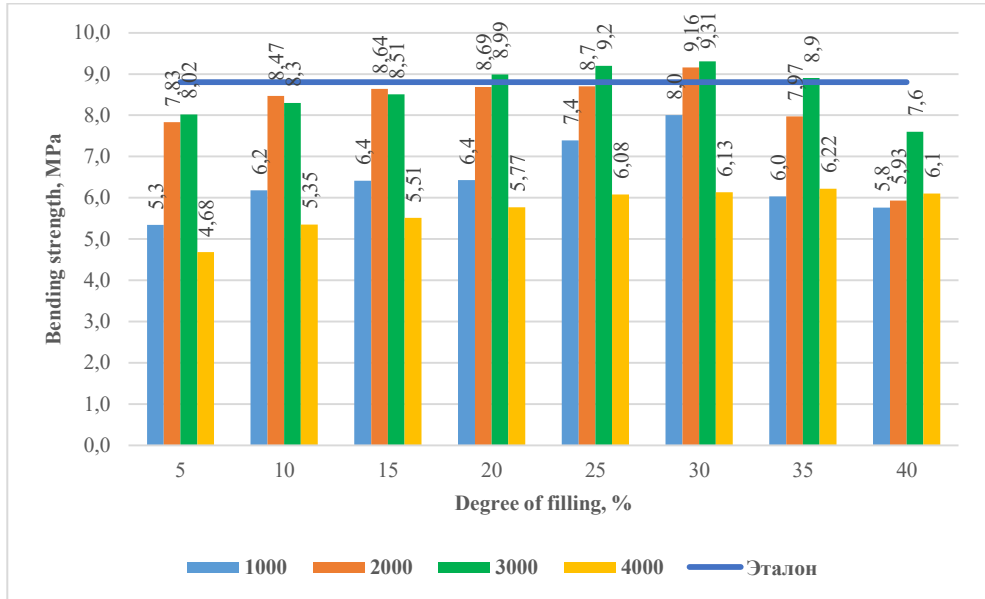


Fig.9. Influence of the specific surface area and the degree of filling of the CMW on the flexural strength of the cement stone

4 Conclusion

In the course of comparing the values of the fineness of grinding, crushed by SS and CMW, it was found that the impact-attrition mode of a ball mill is capable of grinding SS in 80 min to a specific surface of 3511 cm^2/g and an average particle diameter of up to 5.8 mm. Grinding the CMW for 30 min makes it possible to create a fineness of 4412 cm^2/g , with an average particle diameter of 5.4 mm.

Studies have shown that after reaching the specific surface area $S_{Ss}=3195 \text{ cm}^2/\text{g}$, further dispersion increases the amount of residue on sieve No. 008. The same indicator for CMW is $S_{Sp}=3968 \text{ cm}^2/\text{g}$.

The maximum strength of the cement stone ((50.5 MPa for SS) and (48.8 MPa for CMW)) is observed at dispersion $S_{Ss} = 3000 \text{ cm}^2/\text{g}$ and a degree of filling of 30% by weight of the binder.

At the same time, the same strength indicators of the original cement stone are 40 MPa.

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