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Effect of SrO Addition on Recrystallization of ZrSiO$_4$ in Raw Glass-Ceramic Glazes from the SiO$_2$-Al$_2$O$_3$-CaO-MgO-K$_2$O-ZrO$_2$ System

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Abstract: The aim of the experiment was to determine the effect of strontium oxide addition on zirconium silicate recrystallization when adding strontium oxide to the glaze composition. Zirconia glazes (4 different contents) were prepared to which strontium oxide was added in amounts of 0, 1, 3, 6, and 12 mass%. SrO. The characteristic temperatures of the raw glazes were measured, based on which the maximum firing temperatures were determined. The fired glazes were subjected to study of the phase composition and observation of the microstructure. Analysis of the characteristic temperatures showed a fluxing effect, but it was not as strong for all glazes. Differences in the amount of the crystalline phase of zirconium silicate obtained in fired glazes and the partial transition of zirconium silicate to the amorphous phase were observed. Observations of the microstructure clearly indicated an increase in the homogeneity of the distribution of zirconium silicate.

Keywords: glazes; zirconium silicate; strontium oxide; recrystallization; thermal treatment

1. Introduction

Ceramic glazes are a group of materials that contain, apart from the amorphous phase, crystalline phases. Their presence is significant due to the role they play in the material. The crystalline phases have greater mechanical strength than the glaze itself, increasing the mechanical strength of the entire system, as well as resistance to abrasion or chemical corrosion. [1,2]

The crystallization process in glazes is usually desirable, except for the group of transparent glazes, where the presence of crystalline phases could reduce the transparency of the glaze layer. Depending on what crystalline phase is planned in the finished product, it is necessary to conduct the design process and glaze production in a specific way. Some crystalline phases are formed in glazes very easily; others require meeting specific conditions related to the chemical composition of the glaze, i.e., the number of substrates for crystallization, firing temperature, holding temperature or viscosity of the system. [3–6]

Zirconium silicate as a crystalline phase is desirable in glazes with high brightness, increased mechanical parameters, and chemical resistance. The crystalline phase of this mineral is obtained by adding the raw material, zirconium silicate (rarely zirconium oxide), in the amount of at least 3% by mass. Adding a smaller amount may cause no crystallization and transition of the entire amount of zircon to the amorphous phase. [1,5–9]

The zirconium silicate crystallization process is described in numerous publications. The introduction of zirconium silicate into the system causes its partial decomposition in the temperature range of 1100°C-1150°C, and its recrystallization occurs above these temperatures. An important element here is the phase composition; in the case of a low SiO$_2$/Al$_2$O$_3$ molar ratio, only partial recrystallization is observed, while the remaining amount of zirconium in the fired product is present as zirconium oxide, usually in monoclinic form. [9,16–24]

As already mentioned, the crystallization processes are influenced by the chemical composition. An important element of glazes are fluxes, the main role of which is to reduce the firing temperature

of glazes. Their actual participation in crystallization processes depends on the type of flux used. In addition to the most widely used oxides, such as potassium, sodium, calcium, or magnesium oxides, strontium oxide seems to be interesting. It is usually added in the form of carbonate. Its presence improves the gloss of the surface; however, the addition of a larger amount may cause crystallization on the surface of the glazes. [1–3,20–27]

The aim of this experiment is to determine the effect of strontium oxide addition to zirconia glazes on the amount of zirconium silicate crystalline phase obtained and changes occurring in the microstructure of the analyzed glazes with a variable amount of strontium oxide.

2. Preparation

The compositions of the glazes have been designed based on the composition of the production glaze used in the production of sanitary ceramics. The only variable in the compositions analyzed during the design process was the amount of strontium oxide added (five different amounts) and the varying amount of zirconia in the composition (four different amounts). The high molar fraction of SiO_2 / Al_2O_3 is because zirconium oxide was introduced into the glaze as zirconium silicate, it also introduced silicon oxide into the composition of the glaze. Simplified compositions of the analyzed glazes are presented in Table 1.

Glazes were prepared from natural raw materials such as quartz powder MK 40 (Strzeblowskie Kopalnie Surowców Mineralnych Sobótka), KOC kaolin (Surmin Kaolin), potassium feldspar Quantum DS (Sibelco Polska), wollastonite (Ottavi), talc (Luzenac), zirconium silicate (Kreuzonit) and strontium carbonate (Sigma Aldrich).

Table 1. Simplificited composition of the analyzed glazes.

Glaze	Seger formula [molar friction]				Amount of SrO
	SiO_2/Al_2O_3	CaO/MgO	K_2O	ZrO_2	[%mass.]
1Zr0Sr					0
1Zr1Sr					1
1Zr3Sr				0.05	3
1Zr6Sr					6
1Zr12Sr					12
3Zr0Sr					0
3Zr1Sr					1
3Zr3Sr				0,1	3
3Zr6Sr	8.52	1.01	0,37		6
3Zr12Sr					12
6Zr0Sr					0
6Zr1Sr					1
6Zr3Sr				0,21	3
6Zr6Sr					6
6Zr12Sr					12
12Zr0Sr					0
12Zr1Sr				0,43	1

12Zr3Sr	3
12Zr6Sr	6
12Zr12Sr	12

The weighted glazes were ground wet in a planetary mill for 30 minutes. After that, they were dried in a laboratory dryer at 110°C. The dried glazes were used to determine the characteristic temperatures and the maximum glaze firing temperature. Then 60 g of each glaze was placed in biscuit containers and fired according to a designated curve.

The fired glazes were ground to a grain size of less than 63 μm. This powder was used to determine the phase composition of the fired glaze. The phase composition (qualitative and quantitative) of the examined glazes was determined by X-ray diffraction. The study was performed using the Philips X'Pert Pro X-ray diffractometer. The qualitative identification of the phases was carried out by comparing the positions of the reflections and their intensity, obtained during the measurement, with the data collected in the JCPDS ICCD database (Join Committee for Powder Diffraction Standards, International Center for Diffraction Data). Quantitative determination of the phase composition, including the amount of amorphous phase, was performed using the internal standard method. The analysis of the diffractograms (quantitative and qualitative) was carried out using the HighScore Plus program. The quantitative share of the crystalline phases was determined using the Rietveld method. As an internal standard for quantitative analysis, zinc oxide (Huta Oława) was used, which was added to the samples in the amount of 5% by weight.

On the remaining pieces of glaze, microsections were made, which were used to observe the microstructure of the fired glazes. These observations were carried out using the NOVA NANO SEM 200 scanning electron microscope, manufactured by FEI Europe, enabling the observation of the surface of materials in the backscattered electron detection (BSE) system and resolution up to 2 nm. Microstructure observations were carried out each time at magnifications of 1000, 3000 and 5000 times, which greatly facilitates the comparison of sample images. For selected interesting crystalline phases, visual inspection was carried out at 10,000 times magnification. During the microscopic observations, the individual crystalline phases present in the image were also identified using the EDS X-ray microanalyzer by EDAX.

3. Results and Discussion

3.1. Hot-Stage Microscopy (HSM)

Table 2 presents the characteristic temperatures determined for the fired glazes. Their analysis was carried out as a function of the amount of strontium oxide added and the amount of zirconium oxide in the glazes.

**Table 2.** Characteristic temperature of analyzed glazes.

Glaze	Characteristic temperatures [°C]				Firing temperature [°C]
	Sintering	Sphere	Halfsphere	Melting	
1Zr0Sr	1136	1278	1330	1365	1330
1Zr1Sr	1120	1269	1316	1356	1315
1Zr3Sr	1116	1242	1292	1328	1290
1Zr6Sr	1122	1262	1304	1342	1305
1Zr12Sr	1106	1182	1222	1246	1220
3Zr0Sr	1139	1275	1320	1355	1320
3Zr1Sr	1135	1261	1301	1331	1300

3Zr3Sr	1126	1242	1290	1329	1290
3Zr6Sr	1110	1219	1278	1323	1280
3Zr12Sr	1130	1197	1244	1285	1245
6Zr0Sr	1132	1282	1326	1359	1325
6Zr1Sr	1122	1276	1319	1355	1320
6Zr3Sr	1150	1270	1313	1352	1315
6Zr6Sr	1125	1233	1290	1329	1290
6Zr12Sr	1142	1218	1260	1306	1260
12Zr0Sr	1180	1300	1346	1376	1345
12Zr1Sr	1146	1285	1339	1376	1340
12Zr3Sr	1135	1260	1311	1357	1310
12Zr6Sr	1131	1221	1279	1320	1280
12Zr12Sr	1124	1182	1224	1258	1225

On the basis of the analysis of the obtained values of sintering temperatures (Table 2, Figure 1), it can be observed that the obtained glazes belong to the group of high-temperature glazes. The values obtained unequivocally indicate that all processes significant from the point of view of crystallization take place above the temperature of 1100 °C.

The addition of strontium oxide to the glazes reduces the value of this temperature, which is consistent with data from the literature and the fluxing effect of this oxide. However, in not all cases, a decrease in the value of the sintering temperature was observed with the addition of strontium oxide. This may be due to the chemical composition of the glazes, which changes the rate of subsequent reactions in the glazes.

On the values of sintering temperatures for different contents of zirconium oxide in the glazes, it is difficult to notice any dependence. Both an increase and a decrease in the value of this temperature are observed with the same addition of strontium oxide.
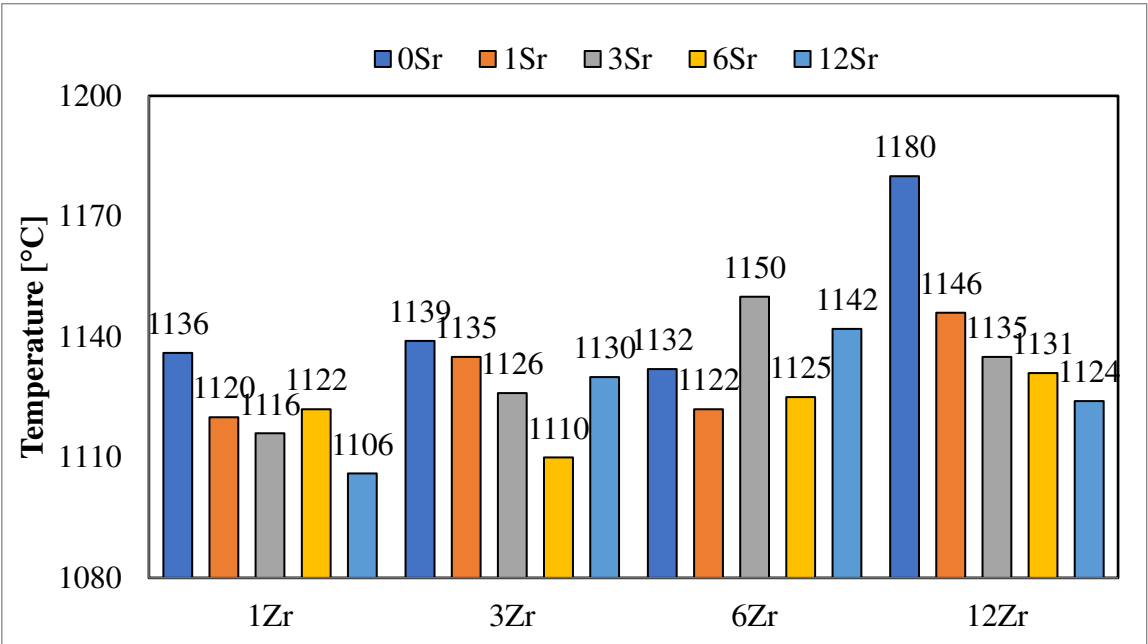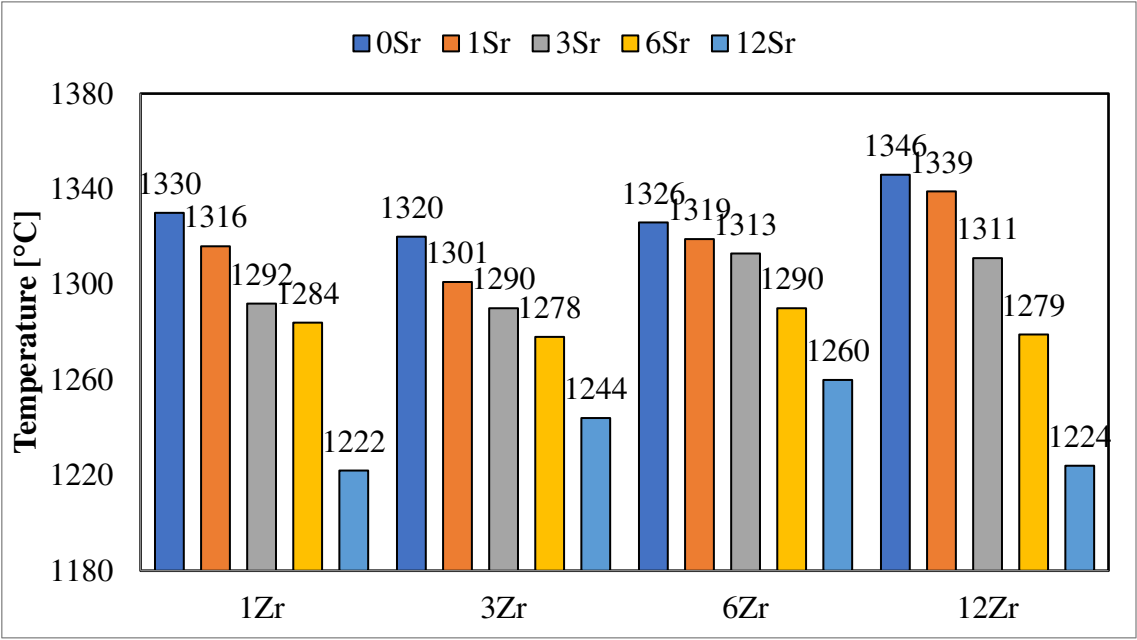

Figure 1. Determined sintering temperatures of tested samples with variable content of strontium oxide.

The determined sphere temperatures (Figure 2) for the analyzed glazes clearly indicate a decrease in the value of this temperature during the addition of strontium oxide. The higher the content of strontium oxide in the glazes, the lower the designated temperature of the hemisphere. The decrease in temperature shows a relationship with the amount of strontium oxide added to the glazes. For glazes with the same strontium oxide content, a slight increase in hemispheric temperatures is observed, along with a higher zirconium content in the glaze. This is especially noticeable with low-stontium oxide glazes. For a larger amount of strontium oxide in the glazes, such a relationship is not observable.

Similar results were observed for hemispheric temperatures (Figure 3), and with an increase in the amount of strontium oxide added, a decrease in the determined hemispheric temperatures was observed. However, the hemispheric temperature values of glazes with a higher zirconium content have higher values only for glazes without the addition of strontium oxide. The addition of this oxide causes the determined hemispheric temperatures to show a clear upward or downward trend with the higher zirconium content in the glazes. This may indicate that the presence of strontium oxide in the glaze affects the processes that take place, whereas its fluxing effect is not directly proportional to the amount of SrO added and strongly depends on the chemical composition of the glaze.
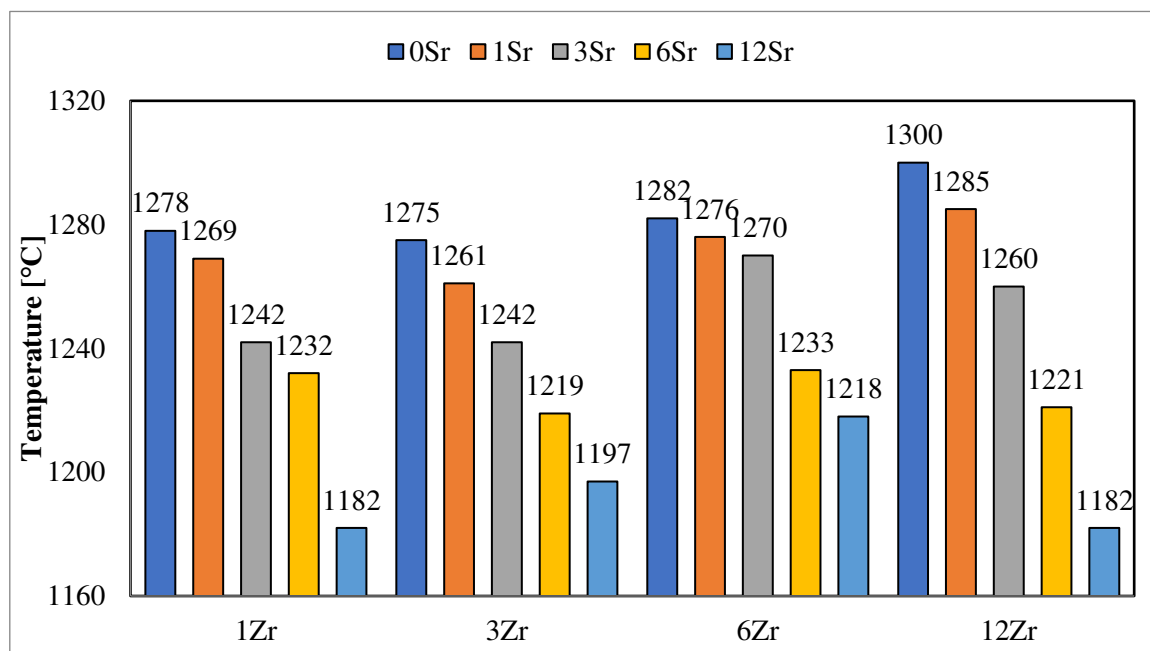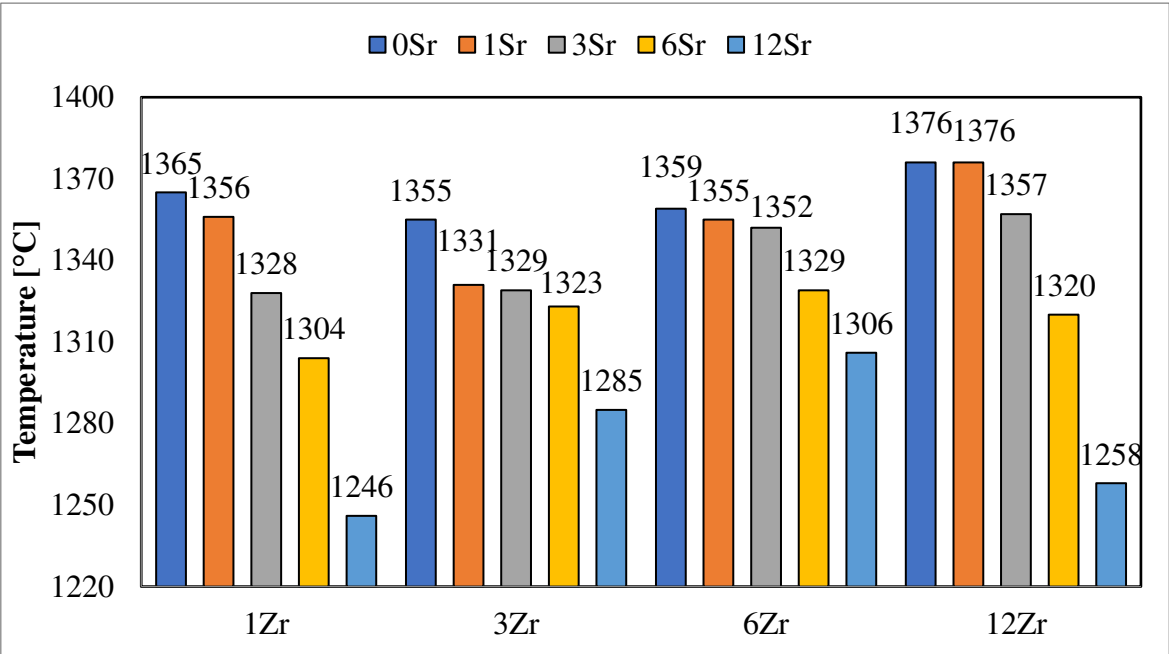

Figure 2. Determined sphere temperatures of tested samples with variable content of strontium oxide.

Figure 3. Determined half-sphere temperatures of tested samples with variable content of strontium oxide.

The addition of strontium oxide to the glazes has a slightly different effect on the determined melting temperatures (Figure 4). For the group of glazes with the lowest zirconium content, a decrease in the value of this temperature is observed with an increase in the amount of strontium oxide in the glaze. For glazes with higher zirconium oxide, a decrease in the value of the reflow temperature is observed, but it is less proportional than in the case of other characteristic temperatures. The decrease in the reflow temperature value is most visible for the highest addition of strontium oxide, compared to the glaze without the addition of SrO, with the same content of zirconium oxide.

Figure 4. Determined melting temperatures of tested samples with variable content of strontium oxide.

Because the crystallization processes are also strongly influenced by the chemical composition of the glaze and its viscosity, the maximum firing temperatures were determined by the hemisphere temperatures, so that the conditions for the crystallization process were as close to each other. According to the literature, the characteristic temperature of the hemisphere is the temperature at which the glaze sample assumes the shape of a hemisphere, and the geometric dimensions of the glaze sample at the temperature of the hemisphere, observed using a high-temperature microscope, are the following: the length of the base is equal to half the height of the sample, and the viscosity of the glazes for such a shape is 10^4 dPas. [28,29]

3.2. Phase Composition

The fired glazes were characterized by high transparency. This may indicate their high degree of amorphousness. The exception here were glazes from the 12Zr group, whose transparency was significantly low and can be classified as cloudy glazes. To determine the amount and type of amorphous phases, we subjected the glazes to X-ray radiation, and the results are presented in the Table 3.

The results obtained indicate the presence of only two crystalline phases in varying amounts: zirconium silicate (ICSD 98-000-9582) and quartz (ICSD 98-000-0174).

When analyzing the content of the crystalline phase of zirconium silicate in fired glazes, greater amounts can be observed, especially in the glazes of the 6Zr and 12Zr series. In the glazes of the 1Zr and 3Zr series, the determined amount of zirconium silicate is small. This clearly suggests that the added zirconium silicate almost completely passed into the amorphous phase. Literature data indicate that in the case of glazes to which zirconium silicate or zirconium oxide will be added, there is no recrystallization of crystalline zirconium phases, and zirconium cations are present in the amorphous matrix. For the group of 6Zr and 12Zr glazes, a decrease in the amount of the crystalline phase of zirconium silicate is observed with an increase in the amount of strontium oxide added. This suggests the depolymerizing effect of strontium oxide, which breaks the bonds of the alumina and silica sublattices and loosens the structure, due to which large zirconium cations are able to fit into the amorphous phase of the glazes.

The second crystalline phase obtained is quartz. Its amount varies depending on the amount of zirconium silicate used and the addition of strontium oxide. For the glazes of the 1Zr and 3Zr series, a decrease in the amount of quartz in the fired glaze is observed with the addition of strontium oxide in the amount of 1 and 3% by weight, and then the further addition of strontium oxide causes an increase in the amount of the crystalline phase of quartz in the glaze. For glazes of the 6Zr and 12Zr series, the determined amount of quartz in the glaze decreases with increasing strontium oxide, while in the case of glazes with the highest zirconium content, quartz appeared in a small amount only for the 12Zr0Sr and 12Zr1Sr glazes. The fluxing and depolymerizing effect of strontium oxide will cause faster dissolution of quartz grains and a reduction of its amount in the fired glaze. However, the decomposition of zirconium silicate and partial dissolution of zirconium cations in the amorphous matrix will result in incomplete dissolution of quartz grains because of its excess in the amorphous phase.

Table 3. Phase composition of the analyzed glazes.

Glaze	Crystalline phases [%vol.]		Amorphous phase [%vol.]
	Zirconium silicate	Quartz	
1Zr0Sr	1	18	82
1Zr1Sr	1	7	93
1Zr3Sr	1	8	91
1Zr6Sr		14	86
1Zr12Sr		12	88
3Zr0Sr	2	5	93

3Zr1Sr	2	4	94
3Zr3Sr	1	3	96
3Zr6Sr	1	9	90
3Zr12Sr		13	87
6Zr0Sr	7	5	88
6Zr1Sr	5	4	91
6Zr3Sr	4	4	92
6Zr6Sr	4	3	93
6Zr12Sr	3	1	96
12Zr0Sr	16	1	83
12Zr1Sr	13	1	86
12Zr3Sr	12		88
12Zr6Sr	12		88
12Zr12Sr	9		91

3.3. SEM Observations

Observations of the microstructure of the 1Zr series glazes (Figure 5) showed the presence of fine crystals of high brightness. EDS analysis confirmed that these are zirconium silicate crystals. Their presence was observed during the observation of the 0Sr, 1Sr and 3Sr glazes, which is consistent with the results obtained from the composition of the analysis of the XRD phase. In the glaze without the addition of strontium oxide, the distribution of zirconium silicate is even over the entire surface. This is different for the microstructure of 1Sr and 3Sr glazes, for which clusters of zirconium silicate were observed, areas containing more of this crystalline phase, and areas completely without this crystalline phase. Glazes with a higher addition of strontium oxide show full amorphism, but no zirconia-crystalline phases were observed.

The microstructure of the 3Zr series glazes (Figure 6) shows an uneven distribution of the zirconium crystalline phases. This is especially evident for glazes to which strontium oxide has been added. For glazes without the addition of this oxide, small areas devoid of zirconium silicate crystals are areas that, according to the EDS analysis, correspond to residual quartz crystals, although they do not have clearly marked intergrain boundaries. The addition of strontium oxide causes zirconium silicate to occur in clusters that constitute a larger or smaller area of the microstructure. For the 3Zr1Sr glaze, a photo magnified 500 times is shown in which smaller and larger areas with the crystalline phase of zirconium silicate are visible. Residual quartz crystals and empty areas without any crystalline phase are also visible.

The glazes of the 6Zr series (Figure 7) show much greater homogeneity than those of the series, with a lower zirconium content in the glaze. Areas with more crystals are still observed, but there are much fewer of them, and their amount decreases with increasing strontium oxide content in the glazes. However, there is greater diversity in terms of crystal size; There are also small $ZrSiO_4$ crystals, but slightly larger ones can be observed. The areas in which zirconium silicate crystals are not observed correspond to the composition of quartz.

The 12Zr series of glazes (Figure 8) shows considerable variation in the distribution of zirconium silicate crystals. An increase in the homogeneity of the microstructure can be observed with the increase in the amount of strontium oxide. Glaze 12Zr0Sr without the addition of strontium oxide shows significant inhomogeneity, which is visible microscopically - bright streaks on the glaze. The addition of strontium oxide causes this macroscopic inhomogeneity to decrease, and since the addition of 6Sr, the glazes appear uniform. The microstructure confirms this, since from the content of 3 mass.% SrO, the glaze is more homogeneous than the reference glaze.

The different arrangement of the crystalline zirconia phases may be due to the difference in the viscosity of the glaze during the firing process. The inhomogeneity of glazes at the microscale results

from the individual processes that take place with individual raw materials; some of them undergo reactions at a lower temperature, and others require a higher one. Thus, there are areas where the liquid phase appears much faster, and in these areas, further reactions occur earlier than in those where the liquid phase appears later.

The addition of strontium oxide causes a significant reduction of this inhomogeneity because of its fluxing effect, the appearance of the liquid phase, and the migration of system elements. Strontium oxide also has a depolymerizing effect, i.e. it loosens the structure and reduces viscosity, which promotes mixing and homogenization of the entire system.
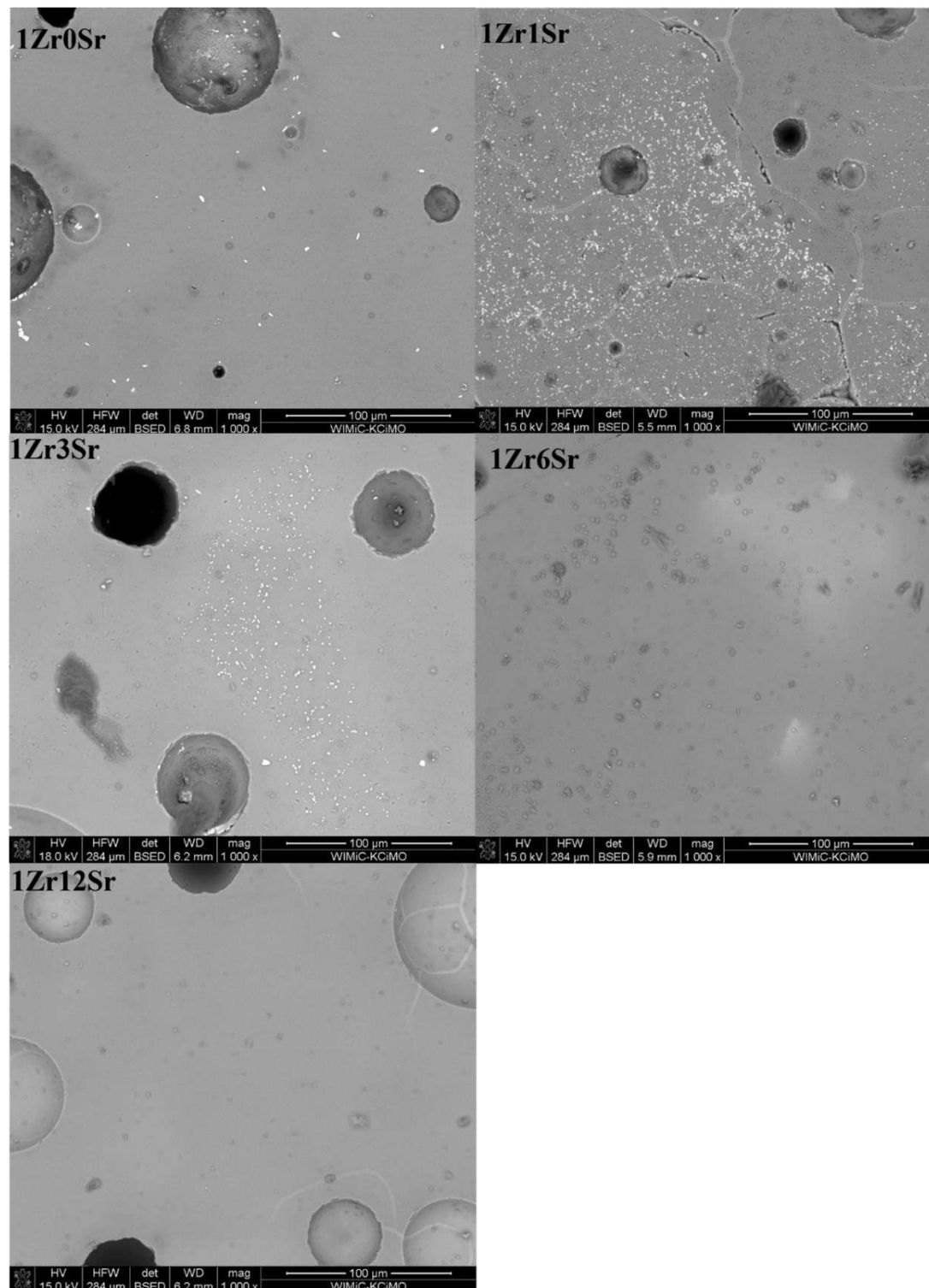
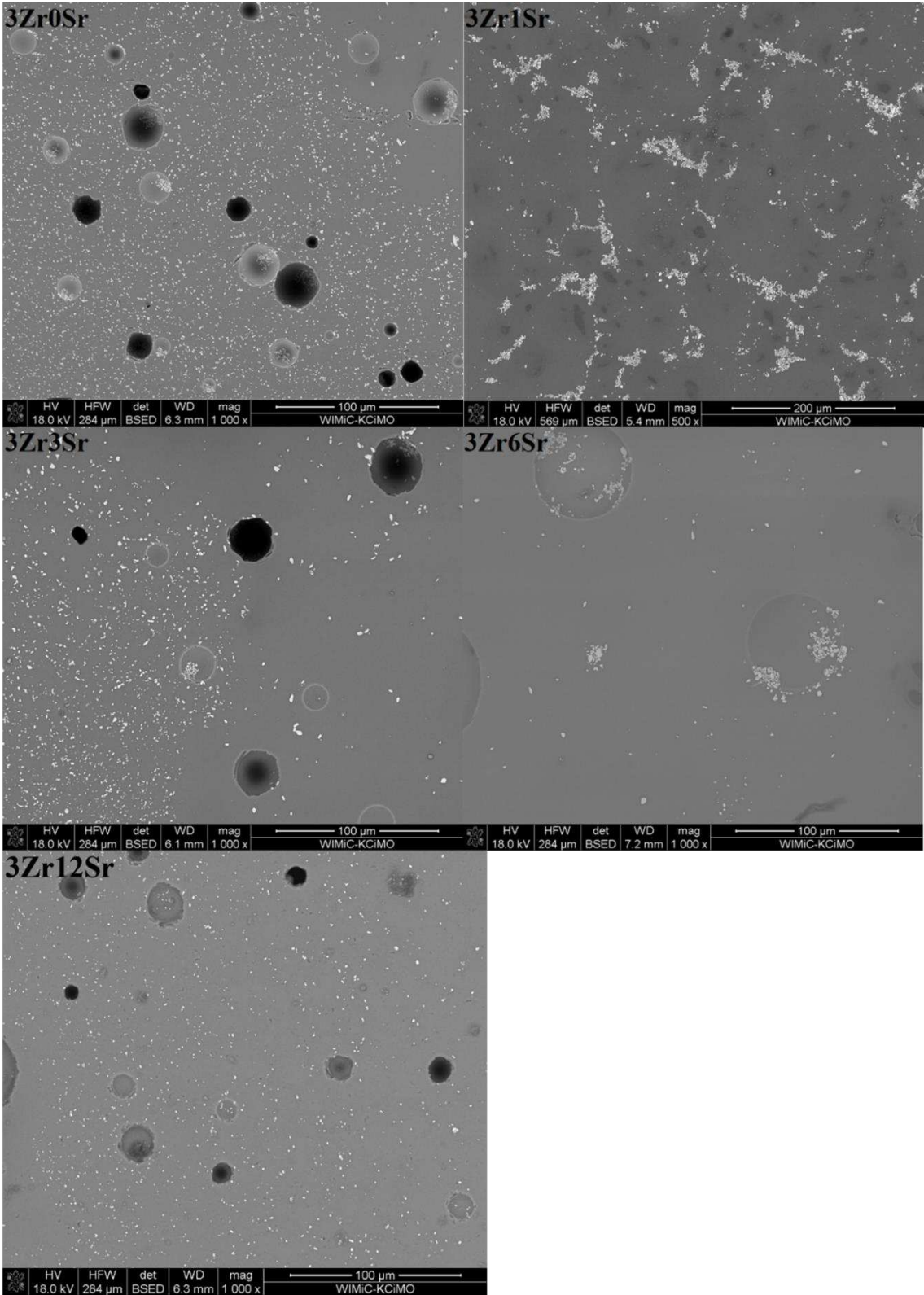**Figure 5.** Microphotographs from SEM observations of 1Zr series.

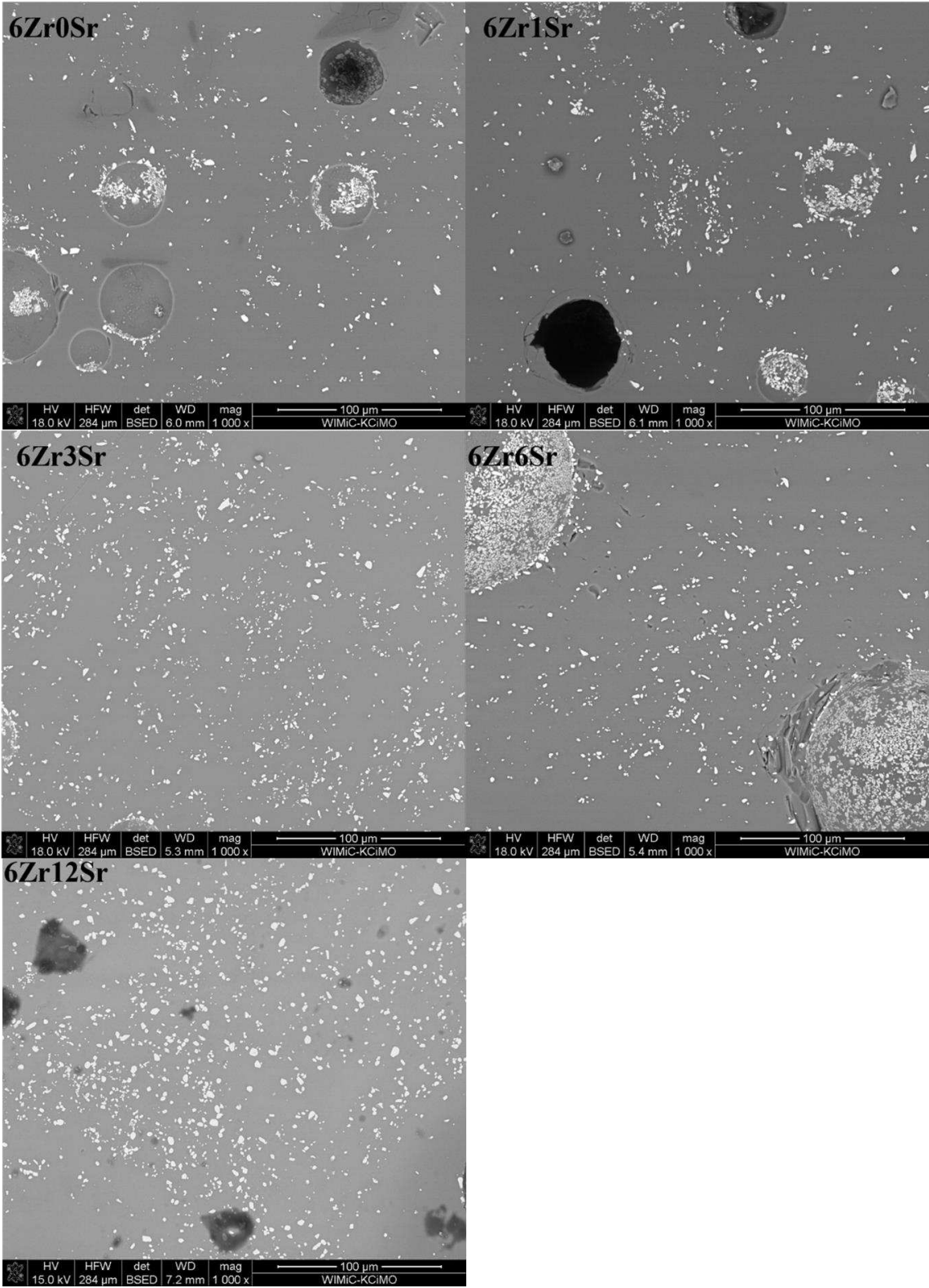**Figure 6.** Microphotographs from SEM observations of 3Zr series.

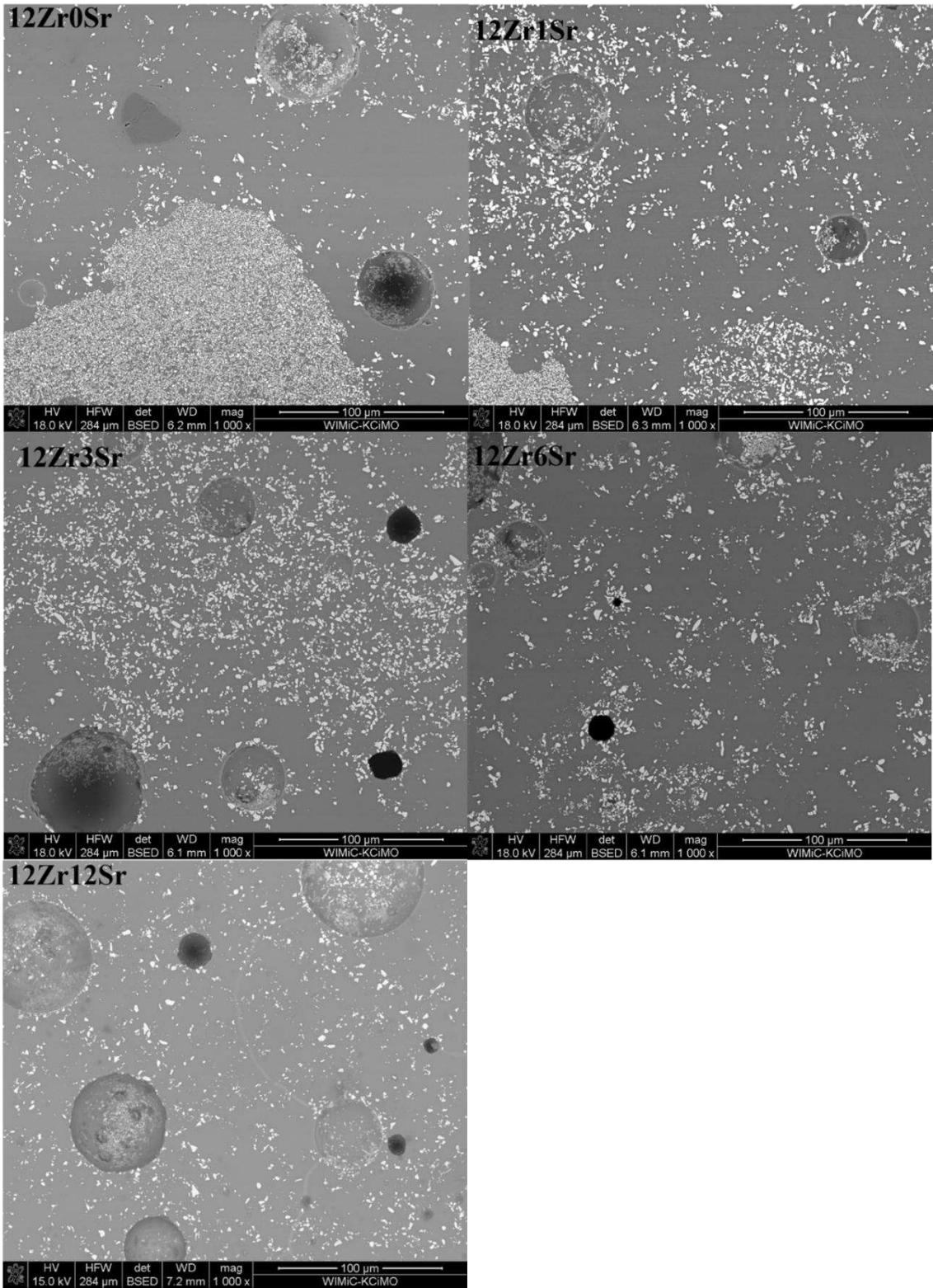**Figure 7.** Microphotographs from SEM observations of 6Zr series.

Figure 8. Microphotographs from SEM observations of 12Zr series.

4. Conclusions

This paper presents the effect of the addition of strontium oxide on the recrystallization of zirconium silicate. The characteristic temperatures of the prepared glazes were measured, on the basis of which the maximum firing temperatures were determined on the determined temperature of the hemisphere. The results obtained from the measurement of characteristic temperatures

indicated that strontium oxide exhibits a fluxing effect; however, it is not observed for each glaze with its addition and depends on the chemical composition of the entire system. In addition, an increase in the amount of zirconium silicate in the glaze does not cause a proportional increase or decrease in the characteristic temperatures, depending on the presence of other components in the glaze.

Analysis of the phase composition showed that some of the added zirconium silicate dissolves in the amorphous matrix, which is particularly evident at a lower zirconium content in the glazes. The amount of dissolved zirconium slightly increases with the addition of strontium oxide, but it does not exceed 3% by mass. This is due to the structure and geometric dimensions of the zirconium cation.

The analysis of the microstructure showed that the addition of strontium oxide increased the homogeneity of the distribution of zirconium silicate crystals. This phenomenon is the most desirable because of the lack of microareas of a purely amorphous nature. These areas will be characterized by reduced mechanical properties and the entire glazes as a material will be much weaker than the one that will be microstructurally homogeneous.

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