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Phase Composition of Sulfate-Resistant Barium-Containing Portland Cement Clinker

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Abstract. Approximately half of the buildings and structures in various industrial sectors (e.g., hydroelectric power plants, bridges, tunnels, oil and gas wells, marine piers, nuclear power facilities) are exposed to diverse aggressive environments, such as saline soils, mineralized waters, seawater, etc. Sulfate attack is the most prevalent form. Sulfate content in groundwater and industrial effluents varies widely and can exceed 5000 mg/L (calculated as SO_4^{2-}). In such cases, even high-density concrete structures based on sulfate-resistant Portland cement require specialized waterproofing, significantly increasing construction costs.

1 Introduction

The chemical degradation of concrete due to sulfate attack is a complex process [1, 2] that typically follows two primary mechanisms:

Ettringite Formation: Sulfates react with calcium hydroaluminates within the cement paste [3, 4]. This leads to the crystallization of secondary ettringite, which occupies a significantly larger volume than the initial reactants [5, 6]. The resulting internal expansive pressure causes micro-cracking and eventual disintegration of the concrete matrix [7].

Gypsum Formation: At high sulfate concentrations, calcium ions from calcium hydroxide (portlandite) react with sulfate ions to form gypsum. This process leads to the softening of the cement paste and a substantial reduction in the overall compressive strength of the material [8- 10].

Beyond technical challenges, the premature degradation of concrete due to sulfate attack poses a significant environmental burden. The need for frequent repairs or complete structural replacements leads to excessive resource consumption and increased CO_2 emissions associated with cement production. Consequently, developing innovative methods to enhance the chemical resistance of concrete is critical not only for economic viability but also for achieving sustainable construction goals [11, 12, 13].

Partial substitution of calcium oxide (CaO) with barium oxide (BaO) in Portland cements is considered a promising approach for enhancing physicochemical and construction properties. Barium-containing cements possess several valuable characteristics: enhanced refractoriness, radiation shielding capabilities, increased specific gravity, and chemical resistance to aggressive media. Consequently, it becomes possible to expand the application limits of concrete in sulfate environments without additional protection by using barium-containing Portland cement, which demonstrates superior corrosion resistance to sulfate attack compared to conventional sulfate-resistant Portland cement [14].

Barium oxide, a chemical analog of calcium oxide, undergoes the same reactions as CaO during the firing of Portland cement raw mixes, forming independent or mixed barium-calcium compounds. There is conflicting literature data regarding the distribution of barium oxide between the silicate and aluminoferrite phases of the clinker. According to [15], barium oxide primarily

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incorporates into calcium aluminates and aluminoferrites during clinker firing, whereas according to [16], it integrates exclusively into silicate minerals. Conversely, the authors of [17] report that barium oxide not only incorporates into the Ca_2SiO_4 lattice, forming mixed crystals, but also reacts with Al_2O_3 to form a certain amount of monobarium aluminate by reducing the $Ca_3Al_2O_6$ content in the clinker.

Since the final properties of the hardened cement paste are primarily determined by the chemical and mineralogical composition of the clinker, as well as its microstructure, the phase composition of two barium-containing Portland cement clinkers containing 55 wt.% (BPC-1) and 50 wt.% (BPC-2) BaO, respectively, was investigated using X-ray diffraction (XRD) and petrographic methods of physicochemical analysis.

2 Main Part

The main constituents of Portland cement clinker are alite (based on tricalcium silicate), belite (based on dicalcium silicate), tricalcium aluminate, and tetracalcium aluminoferrite.

Materials and Methods. When formulating raw mixes for the synthesis of barium-containing Portland cement clinkers, the following characteristics were considered:

1) in the presence of BaO, tricalcium silicate decomposes to form free calcium oxide and solid solutions based on dicalcium silicate [16];

2) calcium orthosilicate (Ca_2SiO_4) does not coexist with barium orthosilicate (Ba_2SiO_4); instead, a ternary compound with the composition $Ba_5Ca_3Si_4O_{16}$ exists between them (in the concentration range of 49.4–75.7 mol.% Ba_2SiO_4) [18, 19, 20]. Therefore, all barium-containing Portland cement compositions must be designed taking this compound into account [21, 22, 23, 24].

The raw mixes were calculated to obtain the ternary compound $Ba_5Ca_3Si_4O_{16}$ in the clinker, employing two distinct calculation approaches: the BPC-1 composition was calculated considering the ternary compound, whereas the BPC-2 composition accounted for calcium and barium orthosilicates. The calculated phase and chemical composition of the barium-containing Portland cement clinkers is presented in Table 1.

Table 1. Calculated phase and chemical composition of barium-containing Portland cement clinker

Cement type	Phase composition*, [wt.%]					Chemical composition, [wt.%]				
	C_3A	C_4AF	$B_5C_3S_4$	C_2S	B_2S	SiO_2	Al_2O_3	Fe_2O_3	CaO	BaO
BPC-1	7	9	84	–	–	17.18	4.53	2.96	20.53	54.8
BPC-2	4.12	9.28	–	26.81	59.79	19.15	3.5	3.05	24.3	50.0

*C – CaO, B – BaO, A – Al_2O_3, F – Fe_2O_3, S – SiO_2

High-purity (reagent grade) calcium and barium carbonates, anhydrous silicic acid, and analytical grade aluminum and iron (III) oxides were used as raw materials for the synthesis of barium-containing Portland cement clinker. Grinding and mixing of the raw charges were performed using the 'wet' method (mixture moisture content was 50 wt.%) for 10–15 hours until complete passage through a № 006 sieve.

Cylindrical specimens were formed from the dried mixtures by semi-dry pressing (material moisture content reached 4–6 wt.%, specific pressing load was 50 MPa). The samples were fired in a cryptol furnace at a peak synthesis temperature of 1350–1380°C with an isothermal holding time of 3 hours, followed by air cooling. The synthesis completeness was monitored by chemical analysis based on the absence of free calcium and barium oxides [25]. Phase composition analysis was conducted on a DRON-3M diffractometer using Cu-Kα radiation. Petrographic examinations were performed using a microscope in transmitted light (immersion mounts) and reflected light (polished sections).

X-ray diffraction (XRD) analysis of the synthesized clinkers (Figs. 1, 2) revealed the presence of the ternary compound $Ba_5Ca_3Si_4O_{16}$ ($d \cdot 10^{-10}$ = 4.099; 2.937; 2.866; 2.431; 2.349; 2.052; 1.854;

1.668; 1.656; 1.47; 1.433 m). The diffraction peaks of the ternary compound coincide with data [18, 19] for $Ba_5Ca_3Si_4O_{16}$ ($Ba_{1.25}Ca_{0.75}SiO_4$), as well as with the characteristics of synthesized silicates $Ba_6Ca_4Si_5O_{20}$ ($Ba_{1.2}Ca_{0.8}SiO_4$, very similar in composition to $Ba_5Ca_3Si_4O_{16}$) [26], $Ba_{1.4}Ca_{0.6}SiO_4$ ($4.5BaO{\cdot}1.9CaO{\cdot}3.2SiO_2$) [17, 18], and $Ba_{1.31}Ca_{0.69}SiO_4$ (T-phase) [29]. Notably, all these compounds lie within the concentration boundaries of the phase discovered in [30], which reports a homogeneous phase with hexagonal symmetry, structurally similar to glaserite, existing in the concentration range from $0.8CaO{\cdot}1.2BaO{\cdot}SiO_4$ до $0.45CaO{\cdot}1.55BaO{\cdot}SiO_4$.

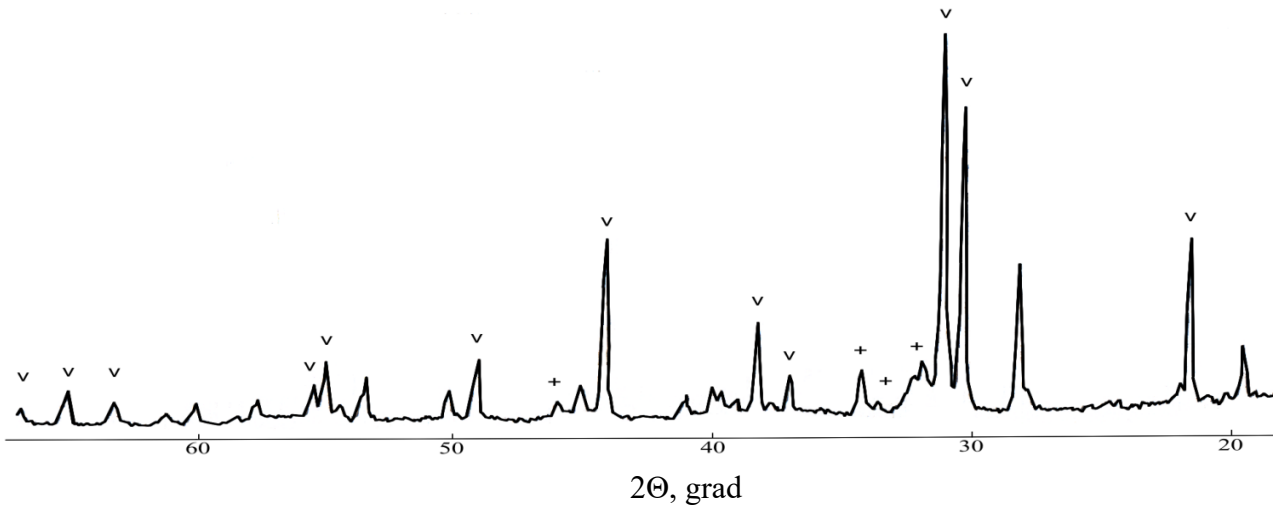


Fig. 1. XRD pattern of the BPC-1 clinker: V – $Ba_5Ca_3Si_4O_{16}$; + – $Ca_4Al_2Fe_2O_{10}$

Since the optical constants of these compounds are also very similar, it is likely that they represent the same compound (or solid solution) – a barium-calcium silicate with the general composition $xBaO{\cdot}yCaO{\cdot}nSiO_2$, where $(x + y) : n = 2$. The XRD patterns also exhibit reflections of brownmillerite $Ca_4Al_2Fe_2O_{10}$ ($d \cdot 10^{-10} = 2.774$; 2.666; 2.618; 1.976 m).

The high sulfate resistance of barium-containing Portland cement (the sulfate resistance coefficient KS reaches 1.2) is attributed to the presence of these specific ternary phases, which are characterized by stable service properties under aggressive sulfate attack [14].

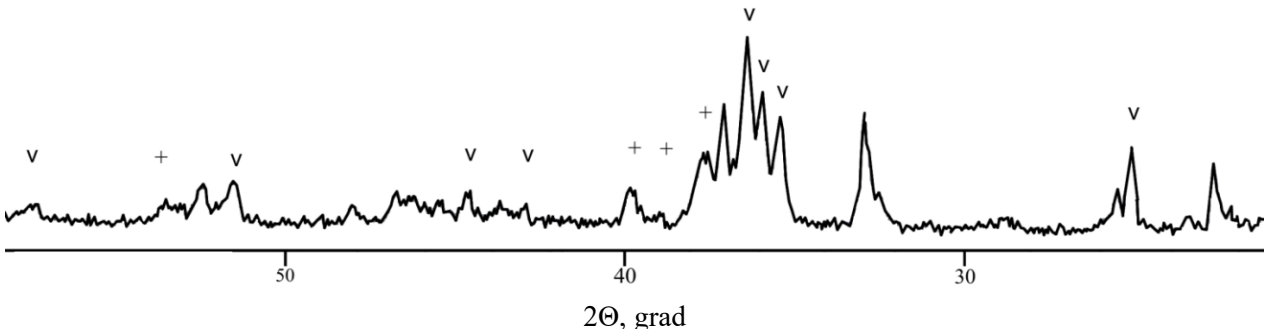


Fig. 2. XRD pattern of the BPC-2 clinker: V – $Ba_5Ca_3Si_4O_{16}$; + – $Ca_4Al_2Fe_2O_{10}$

Additionally, the presence of the following diffraction peaks was established on the XRD patterns: ($d \cdot 10^{-10} = 4.539$; 3.157; 2.249; 2.014; 1.598; 1.514 m). In studies [27, 28, 31, 32], where the authors previously investigated the composition of barium-containing Portland cement clinkers, these diffraction peaks were also observed. However, while the authors of [27, 28] failed to identify

the indicated phase, the authors of [31, 32] attributed it to barium monoaluminate. Since the characteristics and intensity of the identified diffraction peaks were fully consistent with those of barium monoaluminate, petrographic examinations of the samples were conducted to refine the X-ray phase analysis results.

Petrographic examinations of the barium-containing Portland cement clinkers revealed the following characteristics.

Sample BPC-1 (Figs. 3, 4): The structure is fine-grained. The sample primarily consists of colorless Ba_2SiO_4-Ca_2SiO_4 crystals with isometric, polygonal, and tabular (plate-like) shapes, ranging in size from 7-25 μm (maximum 45 μm). These are cemented by films and small grains (prisms) of brownmillerite $Ca_4Al_2Fe_2O_{10}$. The brownmillerite varies from brownish to (more frequently) greenish in color, suggesting that it likely contains a small amount of calcium aluminates in solid solution. The following phases are primarily distinguished among the crystalline phases:

I – anisotropic and brightly anisotropic crystals with $Ng' \geq 1.780$; $Np' \leq 1.767$; some crystals exhibit straight extinction (or near-straight), negative elongation, while certain sections show oblique extinction. As mentioned above, researchers attribute this phase to mixed barium-calcium silicates with the following compositions:

$Ba_6Ca_4Si_5O_{20}$ ($Ba_{1.2}Ca_{0.8}SiO_4$);

$Ba_5Ca_3Si_4O_{16}$ ($Ba_{1.25}Ca_{0.75}SiO_4$);

$4.5BaO{\cdot}1.9CaO{\cdot}3.2SiO_2$ ($Ba_{1.4}Ca_{0.6}SiO_4$).

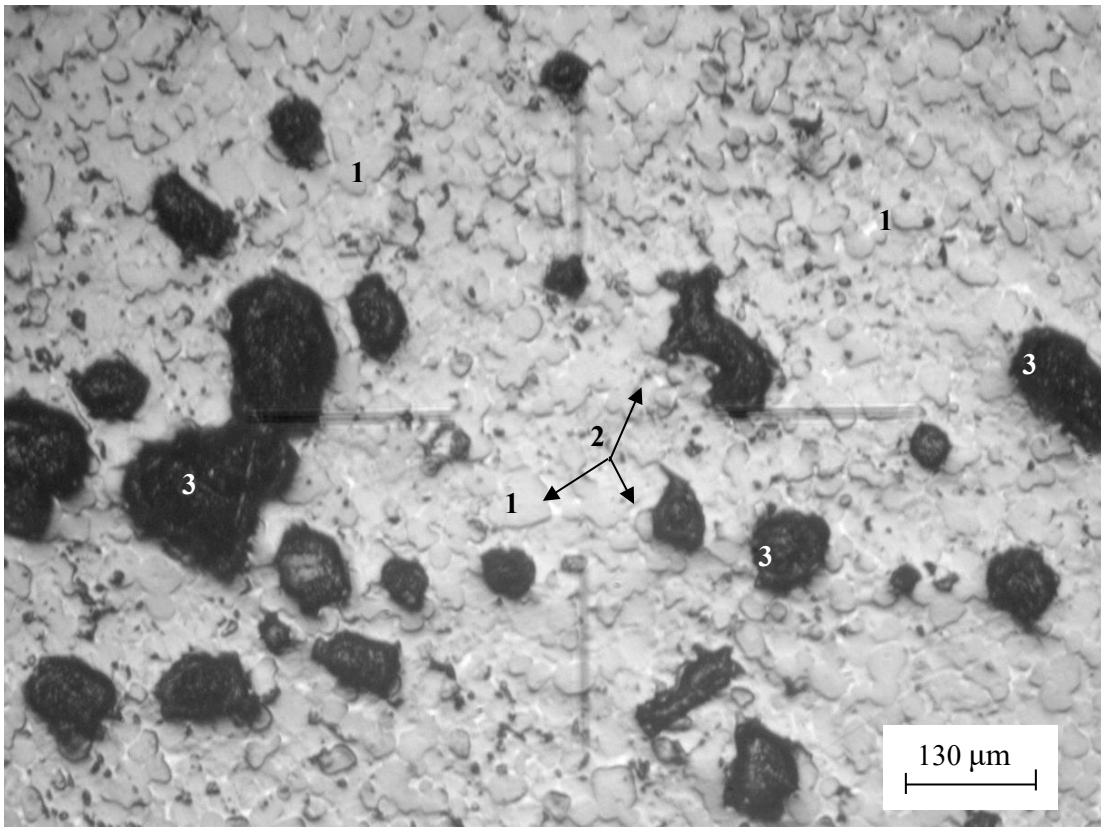


Fig. 3. Microstructure of the BPC-1 clinker: Ba_2SiO_4-Ca_2SiO_4 crystals (1) in brownmillerite films (2), pores (3)

II – weakly anisotropic or nearly isotropic crystals with $Ng' \approx 1.754$; $Np' \geq 1.737$; $\Delta \sim 0.004$–0.005. According to data [16, 33], these are:

$Ba_{1.1}Ca_{0.9}SiO_4$ or $Ba_{1.0}Ca_{1.0}SiO_4$ ($BaO{\cdot}CaO{\cdot}SiO_2$).

III – an isotropic (or nearly isotropic) phase observed in minor amounts with $N_{av} \leq 1.737$; possibly α-Ca_2SiO_4 with a small amount of Ba_2SiO_4 in solid solution:

α-Ca_2SiO_4 : $CaO{\cdot}BaO{\cdot}SiO_2$.

Occasional films of a glassy phase and films of hydrated forms of barium-calcium silicates are observed. In sample BPC-1, the pores are rounded, isometric, or irregular in shape, ranging in size from 20 μm to 600 μm, with a predominant size of 40–100 μm and a maximum of 200 μm; they are rarely interconnected. The structure is dense and well-sintered.

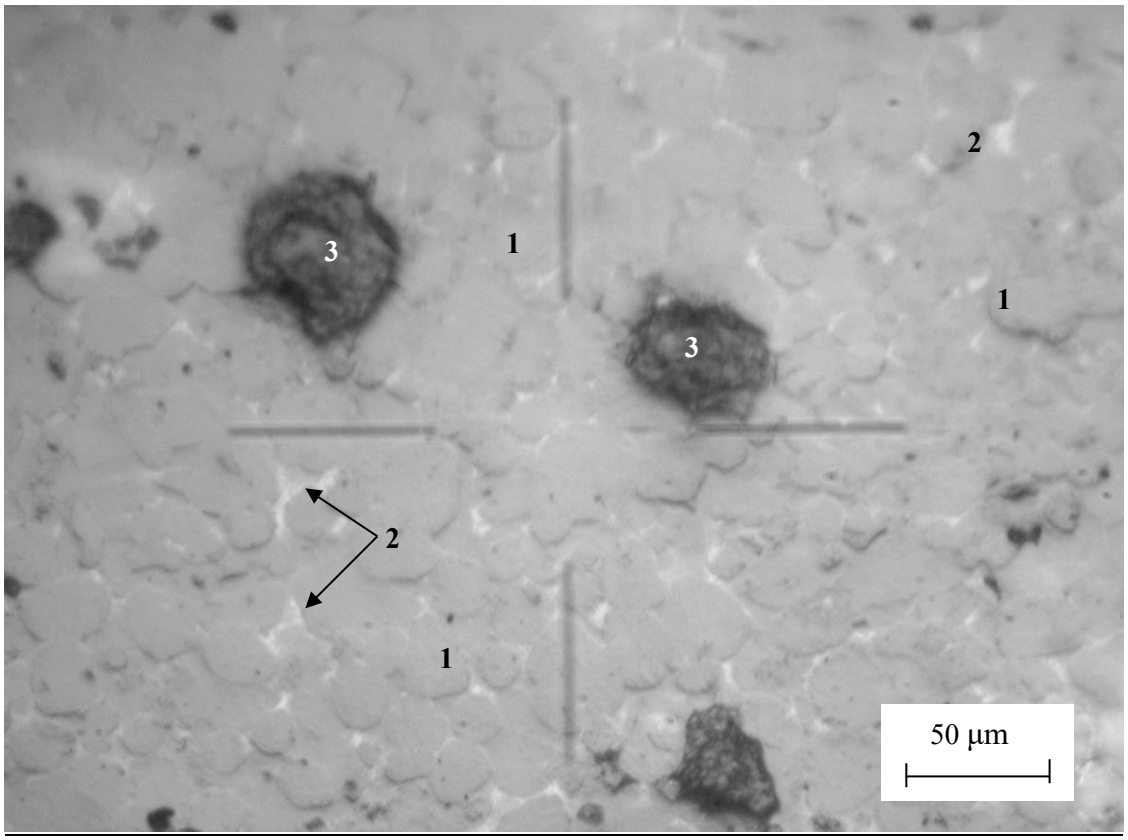


Fig. 4. Microstructure of the BPC-1 clinker (1, 2, 3 are the same as in Fig. 3)

Sample BPC-2 (Figs. 5, 6): The mineral composition and structure are similar to sample BPC-1, with the following differences:

a) the Ba_2SiO_4-Ca_2SiO_4 crystallization is somewhat larger in some areas – 7–30 μm (maximum 50 μm);

b) very fine ("point-like") precipitates (≤ 4 μm) are observed along the edges of some Ba_2SiO_4-Ca_2SiO_4 grains, possibly resulting from partial decomposition of the solid solution;

c) a slightly higher amount of brownmillerite is present as films and prisms (up to 8 μm in length), ranging from brownish to pale greenish in color, indicating a small amount of calcium aluminates in solid solution;

d) a slightly larger number of grains with $N \leq 1.737$ (close to Ng' of α-Ca_2SiO_4 containing a small amount of Ba_2SiO_4 in solid solution);

e) pores are rounded, oval, elongated, or isometric, ranging from 40-100 μm (maximum 200 μm), sometimes interconnecting to form short microcracks up to 100 μm in width.

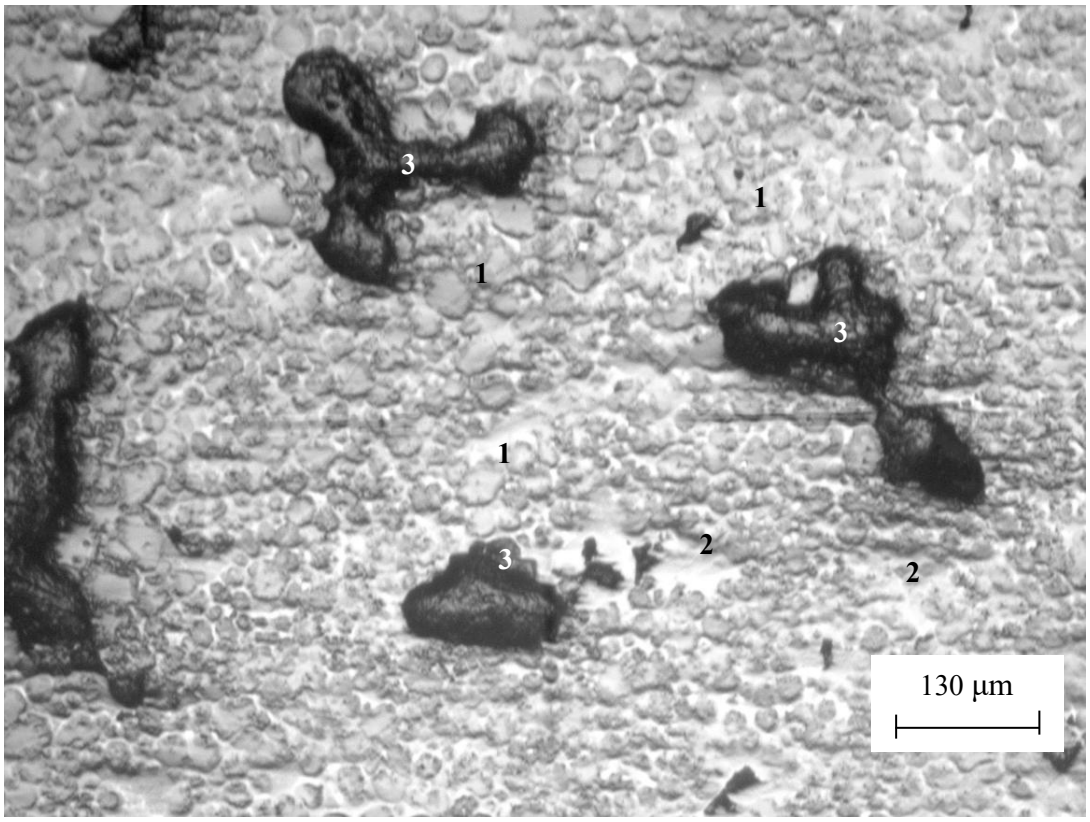


Fig. 5. Microstructure of the BPC-2 clinker (1, 2, 3 are the same as in Figs. 3, 4)

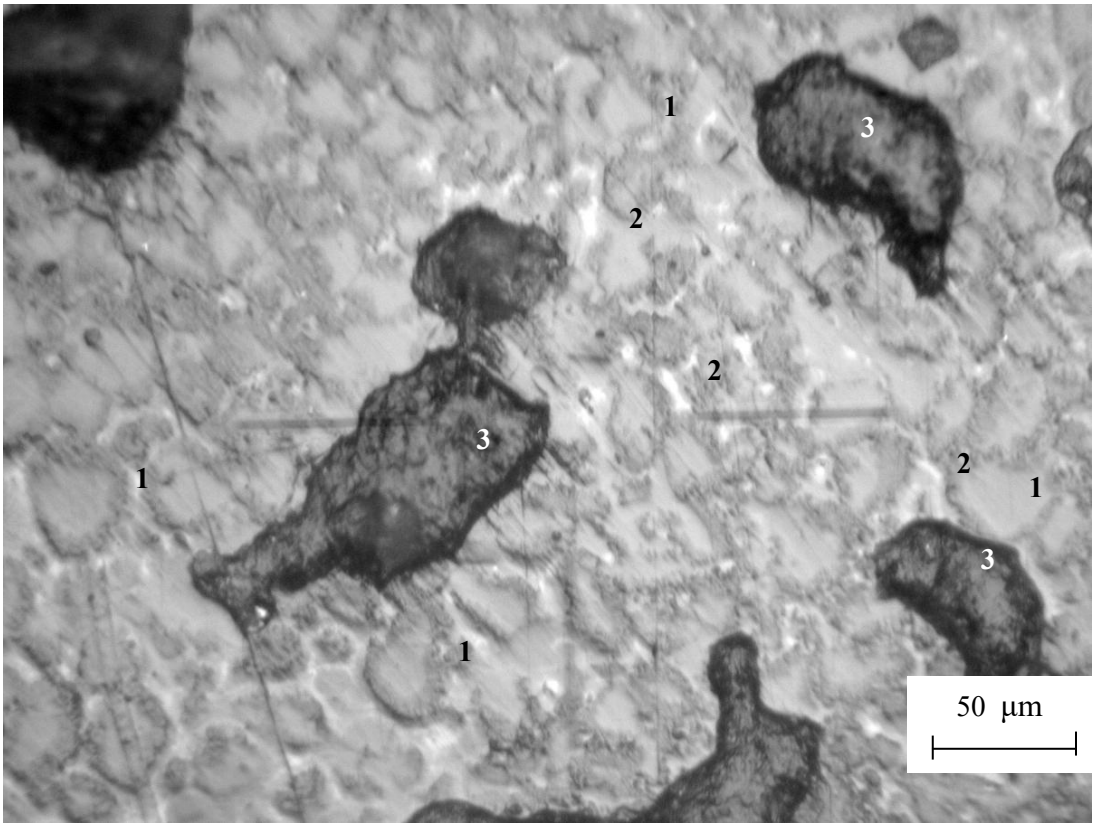


Fig. 6. Microstructure of the BPC-2 clinker (1, 2, 3 are the same as in Figs. 3-5)

Table 2 presents the quantitative phase content of the barium-containing Portland cement clinker, established as a result of petrographic studies.

Table 2. Estimated phase content of barium-containing Portland cement clinker

Sample	Estimated content, [%]				
	Phase I Ba_2SiO_4-Ca_2SiO_4	Phase II Ba_2SiO_4-Ca_2SiO_4	Phase III α-Ca_2SiO_4 : $CaO{\cdot}BaO{\cdot}SiO_2$	Brownmillerite	Glass phase + hydrates
BPC-1	40–50	35–45	2–4	10–12	1–2
BPC-2	40–45	35–45	5–7	10–15	1–2

As seen from the petrographic analysis results, two barium-calcium silicate phases predominantly form in the clinker. Since no barium monoaluminate was detected in the microscopic specimens, it can be assumed that the unidentified diffraction peaks on the XRD patterns belong to the barium-calcium silicate Phase II (presumably $Ba_{1.1}Ca_{0.9}SiO_4$ or $Ba_{1.0}Ca_{1.0}SiO_4$), which is present in the clinker in a significant amount (35-45 wt.%). However, there are no radiographic data in the literature to clearly identify this phase. Notably, our results indicate that during the firing of the Portland cement clinker, barium oxide is incorporated only into the silicate phases, which is in good agreement with the data in [16, 27, 28].

3 Conclusions

Thus, as a result of the studies on the phase composition of barium-containing Portland cement (50–55 wt.% BaO), the following was established:

1. Mixed barium-calcium silicate crystals (Ba_2SiO_4-Ca_2SiO_4) were formed in the samples.
2. Two solid solution phases predominate:

Phase I: with $Ng' \geq 1.780$; $Np' \leq 1.767$; corresponding to mixed silicates like $Ba_6Ca_4Si_5O_{20}$ ($Ba_{1.2}Ca_{0.8}SiO_4$); $Ba_5Ca_3Si_4O_{16}$ ($Ba_{1.25}Ca_{0.75}SiO_4$); $4.5BaO{\cdot}1.9CaO{\cdot}3.2SiO_2$ ($Ba_{1.4}Ca_{0.6}SiO_4$).

Phase II: with $Ng' \approx 1.754$; $Np' \geq 1.737$; $\Delta \sim 0.004$–0.005, presumably $Ba_{1.1}Ca_{0.9}SiO_4$ or $Ba_{1.0}Ca_{1.0}SiO_4$.

It is present in a subordinate quantity **Phase III** (minor amount): $Nav \leq 1.737$, likely α-Ca_2SiO_4: $CaO{\cdot}BaO{\cdot}SiO_2$ with a small amount of Ba_2SiO_4.

3. The silicate crystals Ba_2SiO_4-Ca_2SiO_4 are cemented by brownmillerite ($Ca_4Al_2Fe_2O_{10}$) films containing calcium aluminates in solid solution.
4. In the X-ray diffraction patterns of barium-containing Portland cement clinker, along with barium-calcium silicate of the composition $Ba_5Ca_3Si_4O_{16}$ and calcium aluminoferrite $Ca_4Al_2Fe_2O_{10}$, reflections of an unidentified phase (presumably $Ba_{1.1}Ca_{0.9}SiO_4$ or $Ba_{1.0}Ca_{1.0}SiO_4$) are present.

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