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Solid-Phase Interactions In Sintered Compositions Based On Quartzite, Quartz Sand And Kaolin

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Abstract: The results of a study of solid-phase interaction in sintered compositions based on quartzite, quartz sand and enriched kaolin are presented. It has been established that the nature of the location of the melting isotherms on the triple diagram indicates the spread of new formation processes into the area of internal compositions of the triangle and the formation of crystalline phases of the minerals α-quartz, tridymite, cristobalite, mullite, which gives the necessary technological properties of finished refractory materials. It is shown that on the basis of quartzite and partially introduced quartz sand in combination with kaolin, it is possible to develop popular compositions of dinas refractory masses for lining non-standard places in high-temperature furnaces.

Keywords: Phase relationships, quartzite, quartz sand, kaolin, new formations, crystalline phase, chemical, tridymite, cristobalite, mullite.

Introduction

In recent years, there has been a rapid development of research and innovative development of new refractory materials to create industrial production of a wide range of refractory composites that are competitive in the world market. Progress in the production of refractory materials is determined by the achievements of fundamental and applied research work, which is especially relevant today, since modern materials science meets the trends in the design of popular products for various purposes.

In addition, general and special issues of permissible conditions for the use of aluminosilicate semi-acid and acid molded refractory products have not been sufficiently studied in the literature.

It should be noted that in the production of glass products, melting of glass melt is carried out in glass furnaces, the lining of which uses silica refractory bricks and unmolded refractory masses [1 - 3]. In the Republic of Uzbekistan, silica products, which are in demand in glass and metallurgical industries, are not produced; they are mainly imported abroad. At some industrial enterprises, they are mass-produced for their own needs, based on mixtures of imported and local high-silica rocks [4 - 5]. In this regard, the development of popular compositions of dinas and high-silica refractory materials based on domestic mineral raw materials and secondary resources of the Republic is of current importance.

To develop charge compositions of dinas refractory materials, high-silica mineral raw materials are used - quartzites, quartz sands, in rare cases quartz sandstones, with mineralizing and binding additives. In the production of silica refractories, high demands are placed on the permissible contents of certain impurities in the quartzites and other charge components used [6 - 8].

Unshaped refractories made from available natural and technogenic raw materials - quartzites, quartz sands, refractory clays, silica waste - have found wide application in metallurgical production. The requirements for materials used for the production of unshaped high-silica masses are determined by their ability to sinter with silica and the activity of silica, interactions with other components of the charge when exposed to high temperatures. For such unshaped refractories, the prevention of loosening, porosity, high refractoriness and the desirability of sintering the masses at sufficiently low temperatures are important.

It has been determined from literature sources that a composition of quartzite or quartz sand and refractory clay, on a phosphate bond, is used in quartzite-kaolin or quartz-clay masses for stuffed, shotcrete and monolithic linings of steel ladles of metallurgical industries. In the chemical composition of such refractory masses, the SiO2 content is 91-92 wt., % and Al2O3 is 4 - 8 wt., % [9, 10]. To develop the composition of refractory mertels, a mass composition containing 90 - 94 wt., % is used silicon dioxide and 2 - 6 wt., % alumina. In this regard, it is of particular importance to study the processes of solid-phase interaction in the high temperature range between silica and clay components in developing the optimal composition of unformed refractory masses for lining non-standard places of refractory aggregates. In this regard, based on the conducted critical analysis of the available information, the purpose and objectives of this study are formulated.

The Experimental Part

To study the sintering process in the ternary composition “quartzite - quartz sand - kaolin”, samples of raw materials were used - Jerdanak quartzite, Yakkabag quartz sand and enriched kaolin from the Alliance deposit, as well as prototypes of concentration compositions based on them.

The phase composition of the studied raw materials and fired experimental masses was determined by the X-ray method. Diffraction patterns were obtained using the powder method using a DRON-4.0 installation, CuK radiation, and a Ni filter. The radiograph was taken at a rate of generally 2 deg/min. Monocrystalline quartz was used as an internal standard. In the calculations and identification of phases, we used tables and reference books compiled by the authors of the works as well as the international card index on X-ray powder patterns [11 - 14].

The refractoriness of the tested raw materials and experimental masses was determined experimentally for each sample using the cone method. For this purpose, a finely ground sample is molded into a special collapsible metal sample form - a triangular truncated pyramid of a standard size 30 mm high with the sides of the lower base 8 mm and the upper 2 mm, one edge of which is perpendicular to the base. Pre-dried samples in an air-dry state and at a temperature of 110 – 135°C on a special stand with standard pyroscopes are introduced into ovens for further heat treatment. The temperature rise is controlled by an optical pyrometer and thermocouple, and the softening and falling temperature of the cones is recorded.

It is known that the refractory clays used require high binding capacity up to the sintering temperature of the refractory shard and high refractoriness of the clay. Replacing quartzite with quartz sand in high-silica unmolded refractories is of interest because it is a naturally comminuted and widespread material. The results of the study of chemical compositions showed that in the initial samples of Jerdanak quartzite and Yakkabag quartz sand, the silicon oxide content ranges from 94.5 to 96.5 %. To develop the compositions of popular silica products and high-silica unshaped refractories, such silicon oxide contents are insufficient [15 - 17].

In this regard, the original siliceous raw materials were previously subjected to enrichment by washing them from various impurities, in particular clay minerals, feldspars, hydromica, calcite, as a result of which an increase in the content of silicon oxide in the rocks was achieved.

Samples of enriched Jerdanak quartzite, Yakkabag quartz sand and kaolin from the Alliance deposit were subjected to qualitative X-ray phase analysis using the powder method. The obtained X-ray patterns of the prototype are shown in Fig. 1.

The X-ray diffraction patterns of enriched Jerdanak quartzite (Fig. 1a) and Yakkabag quartz sand (Fig. 1b) clearly show lines of diffraction maxima related to the β-quartz mineral with interplanar distances d = 0.165; 0.166; 0.181; 0.197; 0.212; 0.224; 0.227; 0.245; 0.279; 0.333; 0.422 nm, etc.

The X-ray diffraction pattern of Yakkabag quartz sand is characterized by high intensity and clarity of β-quartz lines, which indicates perfect crystallization of the mineral. The presence of diffraction maxima with low intensity d = 0.590; 0.324; 0.289; 0.191 nm indicates the presence of feldspar.

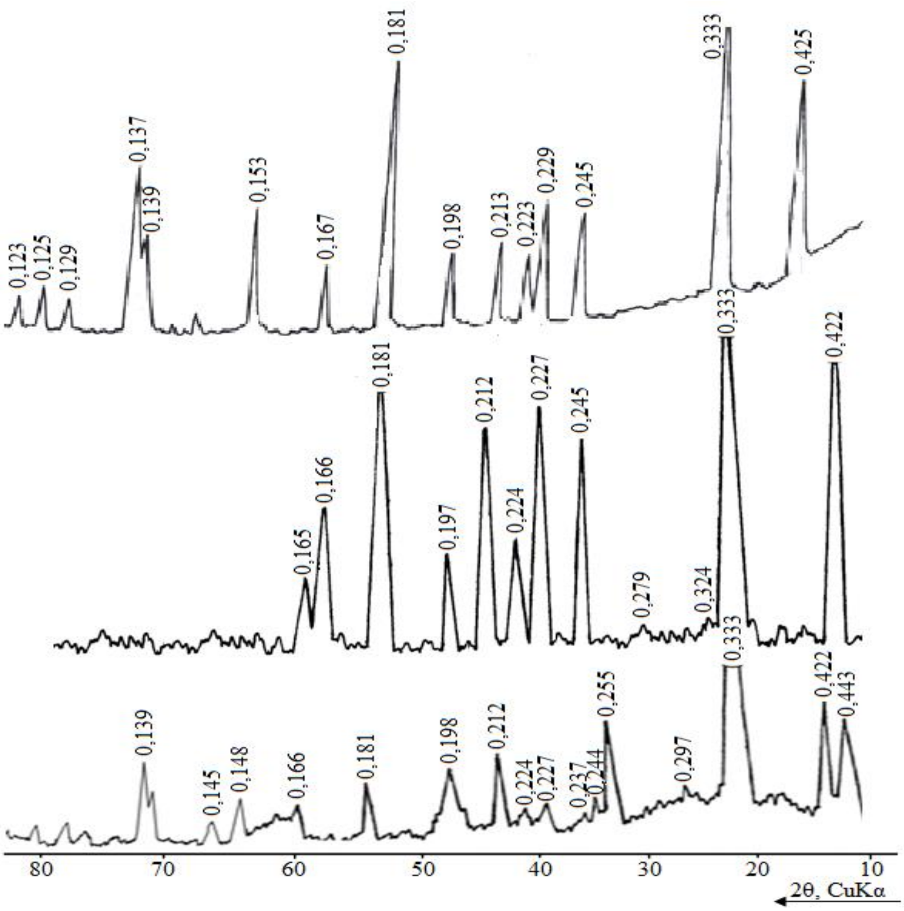

Fig. 1. X-ray diffraction patterns of the initial components: where: a-Jerdanak quartzite; b-Yakkabag quartz sand; c-Alliance kaolin.

X-ray diffraction patterns (Fig. 1c) of enriched kaolin from the Alliance deposit reveal intense diffraction lines of kaolinite (0.255; 0.443; 0.496, 0.358; 0.714 nm, etc.). The nature of the lines of interplanar distances in the X-ray diffraction pattern of samples of enriched kaolin indicates a significant increase in the content of the kaolinite mineral, a decrease in the amount of β-quartz and the absence of hydromica, when compared with the X-ray diffraction pattern of a sample of unenriched kaolin from the Alliance deposit.

The results of determining the chemical compositions of calcined samples of Jerdanak quartzite and Yakkabag quartz sand, after their preliminary enrichment by washing, as well as a calcined sample of enriched kaolin from the Alliance deposit, are given in Table 1. From the data in Table 1, it can be seen that the content of two silicon oxides in the composition of the Jerdanak quartzite and Yakkabag quartz sand is above 98 wt., % which makes it possible to obtain high-silica refractory materials based on them in combination with kaolin – plasticizing components.

Table 1.

Chemical composition of the initial components of the ternary system, in terms of calcined substance.

Name of raw materials	Oxides content, wt., %								∑
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	
Jerdanak quartzite	98,71	0,82	0,19	0,11	0,03	0,08	0,05	0,01	100
Yakkabag quartz sand	98,09	0,24	0,45	0,34	0,15	0,64	0,08	0,01	100
Alliance kaolin	57,25	38,89	0,55	0,52	0,43	0,25	1,58	0,53	100

The study of the processes of formation of compounds during sintering of mixtures with different ratios of quartzite, quartz sand and kaolin makes it possible to evaluate the high-temperature phase relationships and physicochemical properties of refractory materials. It should be noted that the identification of concentration regions of existence of polymorphic forms of quartz minerals also allows us to generalize the results obtained from studying the properties of the compositions of the triple reciprocal system “Jerdanak quartzite - Yakkabag quartz sand - Alliance kaolin”.

To study crystalline phase compositions and melting points, test samples were crushed and subjected to wet grinding in an agate mortar to obtain a fine powder. 45 experimental mixtures were prepared, the composition of the experimental points of the triple diagram “quartzite-quartz sand-kaolin”, which are given in Table 2 and their calculated chemical compositions are given in Table 3.

Table 2.

Melting temperatures and primary crystallization phases of melts of the studied compositions of the ternary diagram.

No. composition	Contents of components			T°C melt.	Primary phase of melt crystallization
	Jerdanak quartzite	Yakkabag quartz sand	Alliance kaolin		
1	70	30	-	1720	α-cristobalite
2	70	25	5	1680	α-cristobalite

3	70	20	10	1620	α-cristobalite
4	70	15	15	1560	mullite
5	70	10	20	1600	mullite
6	70	5	25	1620	mullite
7	70	-	30	1630	mullite
8	60	40	-	1710	α-cristobalite
9	60	35	5	1680	α-cristobalite
10	60	30	10	1620	α-cristobalite
11	60	25	15	1550	mullite
12	60	20	20	1590	mullite
13	60	15	25	1610	mullite
14	60	10	30	1630	mullite
15	60	-	40	1650	mullite
16	50	50	-	1710	α-cristobalite
17	50	45	5	1660	α-cristobalite
18	50	40	10	1600	α-cristobalite
19 eutectic	50	35	15	1530	α-cristobalite-mullite
20	50	30	20	1580	mullite
21	50	25	25	1610	mullite
22	50	20	30	1630	mullite
23	50	10	40	1650	mullite

24	50	-	50	1660	mullite
25	40	60	-	1690	α-cristobalite
26	40	55	5	1650	α-cristobalite
27	40	50	10	1590	α-cristobalite
28	40	45	15	1550	mullite
29	40	40	20	1590	mullite
30	40	35	25	1610	mullite
31	40	30	30	1630	mullite
32	40	20	40	1645	mullite
33	40	10	50	1660	mullite
34	40	-	60	1675	mullite
35	30	70	-	1690	α-cristobalite
36	30	65	5	1660	α-cristobalite
37	30	60	10	1615	α-cristobalite
38 eutectic	30	55	15	1530	α-cristobalite-mullite
39	30	50	20	1570	mullite
40	30	45	25	1600	mullite
41	30	40	30	1620	mullite
42	30	30	40	1645	mullite
43	30	20	50	1660	mullite
44	30	10	60	1675	mullite

45	30	-	70	1690	mullite

As a result of the determination of the crystalline phases of sintered samples (Table 2) it has been established that the primary crystallizing phases based on the ternary composition "quartzite-quartz sand-kaolin" are the minerals alpha cristobalite and mullite, which impart the necessary technological properties of finished refractory materials. At the eutectic points of composition No. 19 and No. 38 there are mixtures of minerals alpha cristobalite and mullite.

Table 3. Calculated chemical compositions of experimental points of the triple diagram "quartzite-quartz sand-kaolin" eutectic.

No. composition	Oxide content, wt., %								T °C, melt.
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	
1	98,52	0,64	0,27	0,18	0,07	0,25	0,06	0,01	1720
2	96,48	2,57	0,27	0,20	0,08	0,23	0,14	0,04	1680
3	94,45	4,51	0,28	0,20	0,09	0,22	0,22	0,06	1620
4	92,40	6,44	0,28	0,21	0,10	0,20	0,29	0,09	1560
5	90,36	8,37	0,29	0,21	0,13	0,17	0,37	0,12	1600
6	88,31	10,30	0,29	0,23	0,14	0,15	0,45	0,14	1620
7	86,28	12,24	0,30	0,24	0,15	0,14	0,51	0,17	1630
8	98,47	0,59	0,29	0,21	0,08	0,31	0,06	0,01	1710
9	96,42	2,51	0,30	0,22	0,09	0,28	0,14	0,04	1680
10	94,38	4,45	0,31	0,22	0,11	0,27	0,21	0,06	1620
11	92,34	6,38	0,30	0,24	0,12	0,25	0,29	0,09	1550
12	90,30	8,32	0,31	0,24	0,14	0,23	0,37	0,12	1590
13	88,25	10,25	0,32	0,25	0,15	0,21	0,44	0,14	1610
14	86,22	12,18	0,33	0,26	0,17	0,19	0,51	0,17	1630

15	82,13	16,05	0,33	0,28	0,19	0,15	0,66	0,22	1650
16	98,41	0,53	0,33	0,23	0,10	0,36	0,07	0,06	1710
17	96,36	2,46	0,33	0,24	0,11	0,34	0,15	0,04	1660
18	94,32	4,40	0,34	0,25	0,12	0,33	0,22	0,06	1600
19	92,28	6,32	0,34	0,26	0,13	0,30	0,30	-	1530
20	90,24	8,26	0,35	0,26	0,16	0,28	0,37	0,12	1580
21	88,19	10,19	0,35	0,28	0,17	0,26	0,45	0,14	1610
22	86,16	12,13	0,36	0,29	0,18	0,25	0,52	0,17	1630
23	82,07	15,99	0,37	0,30	0,20	0,20	0,67	0,22	1650
24	77,99	19,86	0,38	0,32	0,24	0,17	0,82	0,28	1660
25	98,33	0,47	0,35	0,24	0,10	0,41	0,07	-	1690
26	96,29	2,40	0,36	0,26	0,11	0,39	0,14	0,03	1650
27	94,26	4,34	0,37	0,26	0,13	0,38	0,22	0,05	1590
28	92,21	6,27	0,36	0,27	0,15	0,36	0,30	-	1550
29	90,16	8,21	0,37	0,28	0,16	0,34	0,37	0,11	1590
30	88,12	10,13	0,38	0,29	0,17	0,31	0,45	0,13	1610
31	86,09	12,07	0,39	0,30	0,21	0,26	0,67	0,21	1630
32	82,00	15,94	0,39	0,32	0,21	0,26	0,67	0,21	1645
33	77,91	19,80	0,41	0,33	0,25	0,19	0,82	0,27	1660
34	73,83	23,66	0,41	0,35	0,27	0,18	0,97	0,32	1675
35	98,27	0,42	0,38	0,27	0,12	0,47	0,08	-	1690
36	96,23	2,35	0,38	0,28	0,13	0,45	0,15	0,03	1660

37	94,19	4,28	0,39	0,28	0,14	0,43	0,23	0,05	1615
38	92,15	6,21	0,39	0,31	0,15	0,41	0,30	-	1530
39	90,11	8,15	0,40	0,30	0,18	0,39	0,38	0,11	1570
40	88,06	10,08	0,40	0,31	0,19	0,37	0,46	0,13	1600
41	86,02	12,02	0,41	0,33	0,20	0,36	0,52	0,16	1620
42	81,94	15,88	0,42	0,34	0,23	0,31	0,67	0,21	1645
43	77,85	1975	0,43	0,36	0,26	0,28	0,83	0,27	1660
44	73,77	23,60	0,44	0,37	0,29	0,20	0,98	0,32	1675
45	69,69	27,47	0,45	0,39	0,31	0,20	1,13	0,37	1690

From the data given in Table 3, it has been established that when the amount of silicon oxide and aluminum oxide in these mixtures changes, respectively, a neoplasm occurs in the form of the minerals alpha cristobalite and mullite.

Next, experimental mixtures were prepared from the resulting powders, the compositions of which corresponded to the points of the ternary system composed of calcined components: Jerdanak quartzite - Yakkabag quartz sand - Alliance kaolin, in the area of compositions limited by the contents of Jerdanak quartzite from 30 to 70 wt., %, Yakkabag quartz sand from 0 to 70 wt., %, enriched kaolin "Alliance" from 0 to 70 wt., %. The concentration points of the experimental compositions on the ternary diagram are shown in Fig. 2.

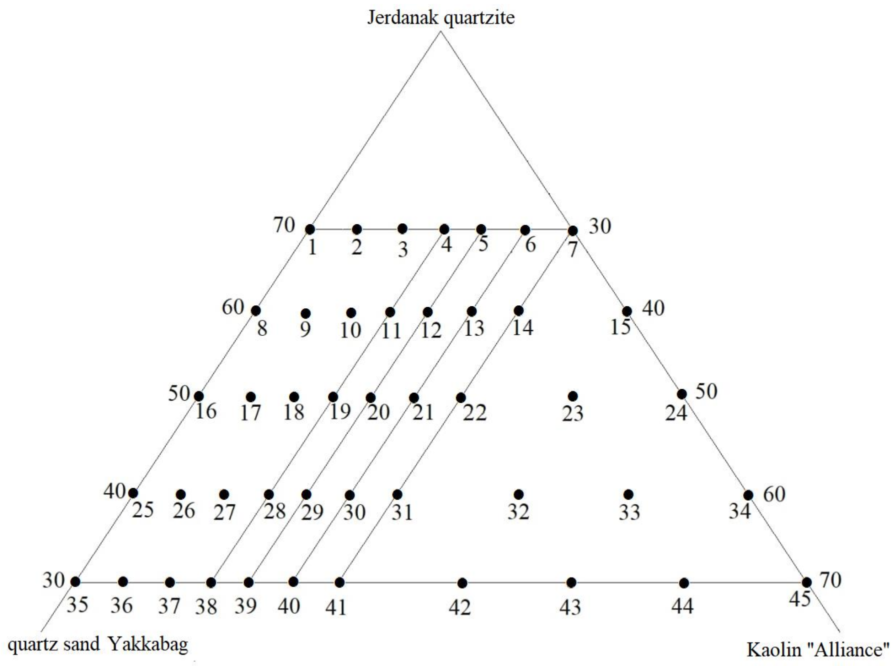

Fig. 2. Concentration points of experimental compositions on the ternary diagram “quartzite-quartz sand-kaolin”

The experimental mixtures under study were prepared by carefully finely grinding mixtures of the synthesized starting components. Test cones were then pressed into a metal mold and used to determine melting temperatures in a laboratory tube furnace and an oxy-gas flame melting unit. After the melting temperatures were established, the samples were quenched by rapid cooling in air.

The resulting hardened samples of melted cones were subjected to X-ray phase analysis to identify the phases of minerals that initially crystallized from the melt and were in equilibrium with the melt. It has been established that in the hypoeutectic regions of the liquidus surface from the side of the "quartzite-quartz sand" triangle, the primary crystallizing phase is a high-temperature form of silica - the mineral α-cristobalite. In the hypoeutectic regions of the liquidus surface from the side of the quartzite-kaolin triangle, the primary crystallizing phase is the mineral mullite.

The study of phase relationships is relevant due to the need for comprehensive studies of aluminosilicate refractory materials with high silica contents, which are widely used in high-temperature thermal units - unmolded quartz-clay masses, dinas mortars, etc., in which kaolins and refractory clays are present as plasticizing and sintering component.

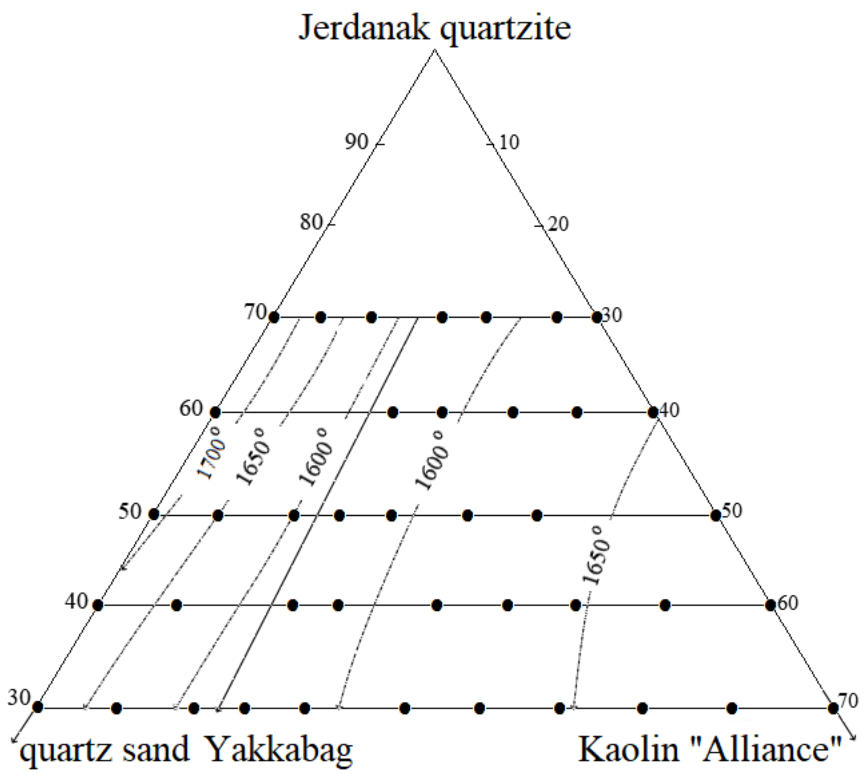

Fig. 3. Fusibility diagram of a ternary system "quartzite-quartz sand-kaolin"

The conducted study of solid-phase interaction and phase relations in sintered compositions based on quartzite, quartz sand and kaolin is relevant, due to the need for comprehensive studies of refractory materials with high silica contents, which are widely used in high-temperature thermal aggregates - unformed quartz clay masses, dinase mertels, etc., in which kaolins are present as a plasticizing and the sintering component.

Thus, it should be noted that to obtain high-silica, in particular the sought-after compositions of dinas refractory masses, the main raw materials are quartzites and partially introduced quartz sands and enriched kaolins.

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