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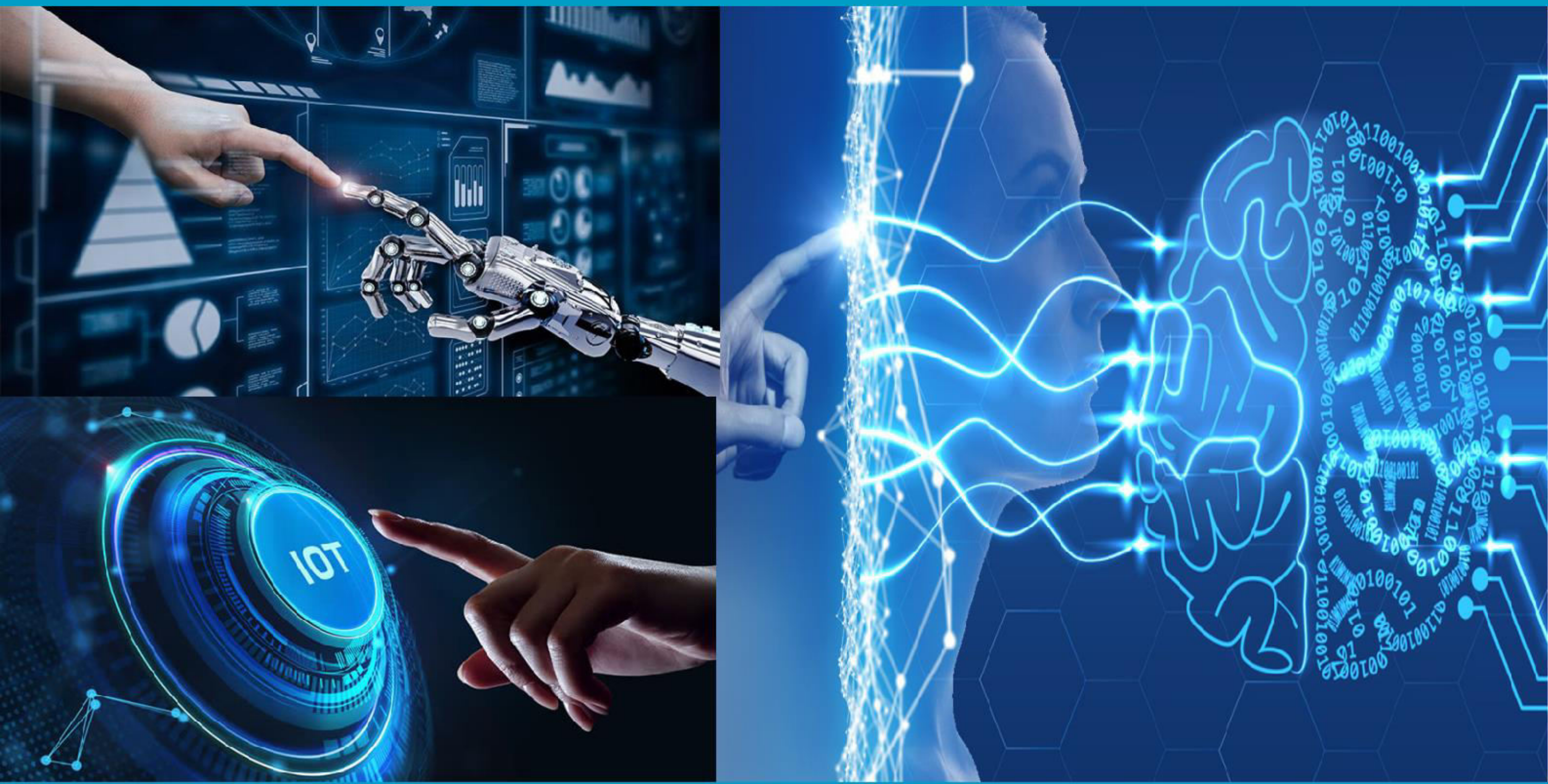

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Compositions of Refractory Adhesive Compositions Based on Industrial Waste with Clay Additive

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ABSTRACT: The results of petrographic studies of structure formation and the formation of the phase composition of the developed aluminophosphate refractory adhesive compositions Al_2O_3*-Cr_2O_3-H_3PO_4 at different temperatures with the addition of clay components are presented. It has been established that polarizing cristobalite tridymite and $AlPO_4$ crystals of rhombic modification are observed in the phase composition of the composition of fired samples, which are thermally stable compounds that provide high performance properties when gluing refractory materials.

KEYWORDS: adsorbent, aluminophosphate glue, amorphous phase, phosphotridymite, phosphocristobalite, adhesion.

I. INTRODUCTION

The intensification of technologies in industries associated with now higher temperature processes puts forward increased requirements for refractory and binder materials. The destruction of the refractory masonry to a large extent occurs not so much from the failure of refractory products, but from the weakening of the intermediate layer created by hardened refractory adhesive solutions. The use of traditional clay-shamotte or cement mortars [1, 2] in the monolithization of enclosure constructions of thermal units often does not provide the necessary technical properties, both in terms of strength and thermal characteristics, of the resulting masonry. In recent years, intensive research has been carried out to create fundamentally new binders and composite materials used as refractory adhesives (mortars).

Currently used in the production of phosphate adhesive compositions, pure inorganic powder materials were used as fillers (in mass%): fireclay 60-75, clay 6-10, AHFS 4-6, chromite 3-5, technical alumina 10-15, magnesite 2-4 [3], and also recommended compositions in the composition (wt %): fireclay sand 69, finely ground clay 10, phosphoric acid (65%) 11, [4]. However, it should be noted that the production of components requires high energy consumption, the materials themselves are scarce and expensive. In [5, 6], a catalyst, kaolin, and AHPS were used to prepare phosphate glue, but its cost is still quite high.

Taking into account the above, we have set the goal of developing new phosphate refractory adhesive compositions using industrial waste, in particular, waste adsorbent (activated aluminum oxide Al_2O_3*) used in the production of polyethylene by LLC Shurtan Gas Chemical Complex (GCC) and local raw materials - kaolin and clays of the Angren deposit. The use of industrial waste and local raw materials allow not only to expand production, but can solve the problems and tasks of industrial waste disposal and reduce the cost of the products obtained.

II. EXPERIMENTAL METHODS

Determination of the chemical composition of raw materials was carried out in accordance with GOST 2642 (0-12) 81.

The spreadability of the phosphate refractory adhesive was determined using a TI-2 flowmeter designed by MM named after D.I. Mendeleev.

Water absorption, apparent density, open and total porosity were determined according to GOST 2409-80.

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The true density of the hardened refractory adhesive was determined by the pycnometric method, according to GOST 2211-65.

The compressive strength of the adhesive material was determined on samples - cubes with a size of 30x30x30 mm according to the method according to GOST 473.6-81, the tensile strength was determined according to GOST 14760-69 and GOST 473.7-81, the shear strength was determined according to the method according to GOST 14759 - 69.

Refractoriness was determined according to GOST 4069-69 (taking into account "Update No. 1 of 01.01.81") on cones made of the tested material. The coefficient of linear thermal expansion was determined on a vertical quartz dilatometer KDTR-16, designed by VNIIS. When determining the heat resistance of samples 70x70x20 mm in size, the requirements of GOST-20910-82 were taken into account.

The petrographic study and the study of the microstructure were carried out on transparent samples and polished sections. The refractive index was determined in immersion preparations. Separate sections of thin sections and the contact zone of glued samples were recorded by microphotography. Thin sections were made from adhesive compositions and immersion preparations were made from heat-treated material at 300 and 1100 ^oC.

III. RESULTS AND DISCUSSIONS

To solve the set tasks, the ultimate goal of which was the development of optimal compositions of refractory adhesive compositions and the study of physical, mechanical, technological properties of structure formation and the formation of the phase composition of the obtained adhesive compositions fired at different operating temperatures. In this regard, variants of refractory adhesive compositions with a clay additive were developed, the optimal compositions of which are shown in Table 1.

Table 1. Compositions of refractory adhesive compositions

№ of composition	Al_2O_3* %	Cr_2O_3 %	Refractory clay, %	Kaolin, %	H_3PO_4, %
Composition -1	31	6,6	15	-	47,4
Composition -2	31	6,6	-	15	47,4
Composition -3	34,3	8,3	13	-	47,4
Composition -4	34,3	8,3	-	13	47,4

The chemical composition of kaolin and clays of the Angren deposit, as well as the spent catalyst containing actuated aluminum oxide Al_2O_3* - waste of the Shurtan GCC, are given in Table 2.

Table 2. Chemical composition of the components

Name of component	l.i.i.	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	$TiCl_4$	V_2O_5
AKS-30	12,2	52,46	0,52	31,4	0,61	0,5	0,74	0,83	0,33		
AKS-78	13,2	46,8	0,36	36,9	0,51	0,18	0,24	0,02	0,38		
ARC-20	9,55	49,73	1,20	35,32	0,78	0,5	0,72	0,52	1,80		
Al_2O_3*	6,05	0,2	-	86,6	-	-	-	2,0	-	2,2	2,5

The results of determining the main physical, mechanical and technological properties of refractory adhesive compositions with a clay additive are given in Table 3.

Table 3. Main physical and mechanical technological properties of adhesive compositions.

№	Properties of composition	Comp-1	Comp-2	Comp-3	Comp-4	AXFS of OK grade
1.	True density (1 hour after curing)	1,77	1,72	1,82	1,81	1,80
2.	True density kg/m3 after heat treatment 300-1100 ^oC	1,60/1,61	1,42/1,39	1,42/1,39	1,57/1,54	1,50/1,48
3.	Correlation l/t h/kg	0,9	0,9	0,8	0,8	0,88
4.	Spreadability, in mm	80	84	87	86	90
5.	Setting time start/end in g	2,5/3,0	2,3/2,9	2,3/2,9	2,3/3,4	2,6/3,2
6.	Water absorption W, in % 300-1100 ^oC	30/33	32/35	24/26	25/26	26/27
7.	Porosity, in % 300-1100	38/40	39/42	35/36	36/38	32/34

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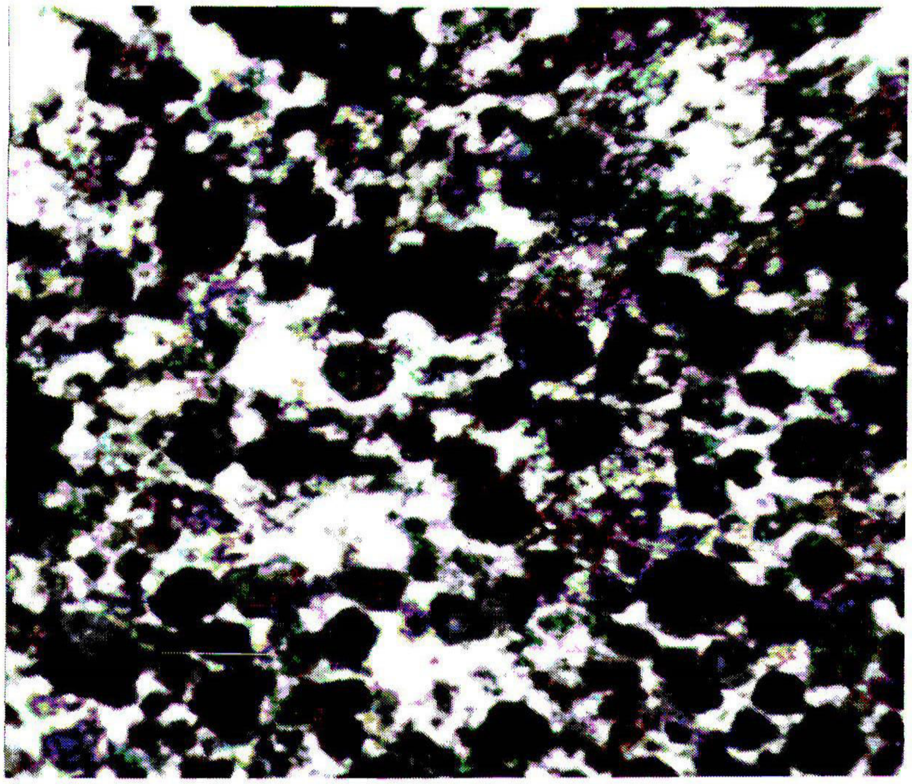
	°C					
8.	τ - shift, in MPa 300-1100 °C	11,4/10,4	9,5/9,0	6,2/5,7	4,5/4,7	7,5/7,0
9.	τ - compression, in MPa 300-1100 °C	23,2/29,9	24,5/28,9	28,0/33,1	28,0/29,5	26,2/32,4
10	τ - gap, in MPa 300-1100 °C	7,9/9,0	7,3/8,0	4,1/6,2	3,2/5,8	5,2/7,4
11	CTLR in $\alpha*10^{-6}$ 1|^{0}C	6,6	5,3	8,1	8,0	7,0
12	Deformation temperature under load, in °C	1430/1510	1430/1500	1380/1440	1390/1450	1400/1430
13	Thermal resistance, per cycle	20	20	18	19	18
14	Fire resistance, in °C	1750	1750	1710	1710	1700

From Table 3, we can make a positive conclusion that the developed refractory adhesive compositions, in terms of their physical and chemical technological properties, fully meet the modern requirements for adhesive materials.

Subsequently, to determine the microstructure of the fired samples, petrographic studies of refractory adhesive compositions were carried out, the optimal compositions of which are given in Table. 1. The results of studying the macrostructure of the fired samples in Comp-1 and Comp-2 are shown in the pictures: Fig. 1 A (x200, transmitted light, without analyzer) shows the macrostructure of the glue in Comp-1. As can be seen from the picture, the pores are fairly evenly distributed between the aggregate grains and the particles of the binder mass. The pore sizes are commensurate with the filler particles (5-20 µm), in fig. 1B there are areas with larger pores - up to 100 µm. At a magnification of 400x (Fig. 2, transmitted light, without an analyzer), one can observe the location of the cementing mass on aggregate grains and in the intergranular space. On fig. 3 shows the contact zone of glued samples, the thickness of the seam is -0.75 - 0.90 mm (reflected light, x 50).

Determination of microhardness gave the following results:
glue on Comp-1 d = 38 mkm; H = 670 MPa
glue on Comp-2 d = 41 mkm; H = 420 MPa,
where: d - imprint diagonal, H - hardness number.



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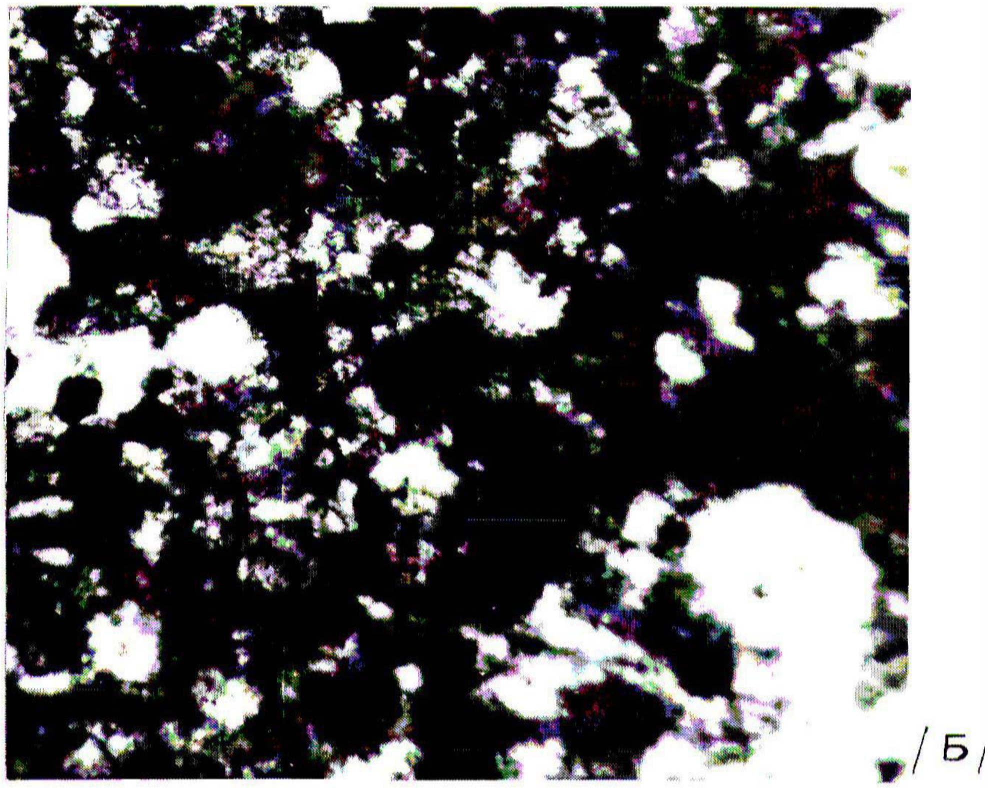

Fig. 1. Macrostructure of refractory adhesive based on Comp-1, finely porous area (A), large porous area (B). Light transmitted, x 200.

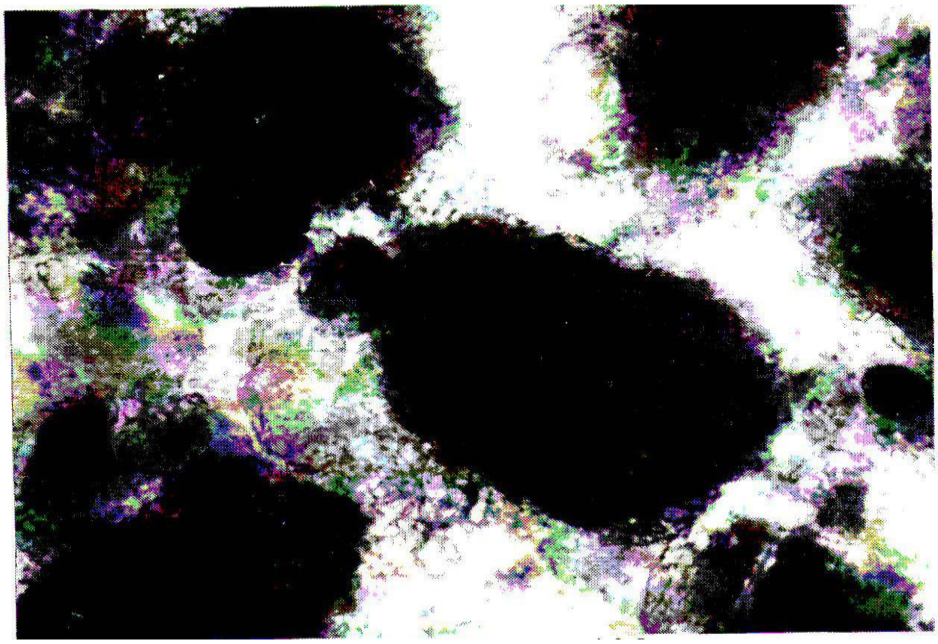

Fig. 2. Macrostructure of refractory glue in Comp-2.
Light transmitted, x 400.

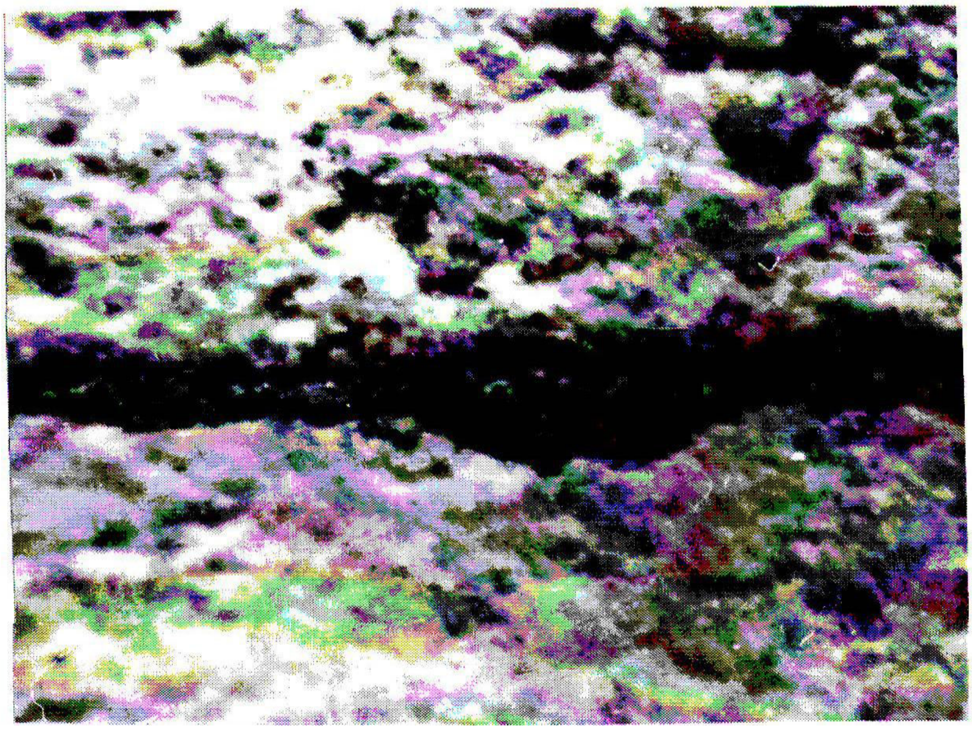

Fig. 3. Microstructure of refractory adhesive in the contact zone of glued specimens. Light transmitted, x 50.

The higher microhardness of the composition in Comp-1 can be explained by the high content of the crystalline phase in neoplasms.

According to the data of crystal optical analysis, the main mass of the hardened composition is rounded grains of α-Al_2O_3 solid solution with Cr_2O_3, colored gray-green. The main particle sizes are 5-10 μm, the refractive index (total) is 1.685-1.750 ± 0.002.

The composition of phosphate neoplasms in both compositions is similar, only the composition on Comp-2 differs in a high content of the glass phase. The following phosphate neoplasms have been identified:

After heat treatment at 300 °C:

Glass of variable composition n= 1,525-1,538;

$Al(PO_3)_3$ most likely a mixture of forms B and A c n = 1,495-1,540;

$AlH_3(PO_4)_2$•nH_2O prismatic, well polarizing elongated (7-10 microns) crystals, n = 1,523-1,550;

$AlPO_4$•nH_2O polarizing grains (5-8 microns), n= 1,530-1,580

$AlPO_4$ - berlinite, n_p= 1,542; $n_д$= 1,566;

As a result, the study can suggest the presence of a mixture of cristobalite and tridymite forms of $AlPO_4$ with n < 1.500.

After heat treatment at 1100 °C, only well-polarizing $AlPO_4$ crystals of orthorhombic modification with n_p = 1.548 and n_d = 1.576 and a mixture of phosphotridymite and phosphocristobalite with n = 1.465-1.485 were found. The sample at Comp-2 may contain β-$CrPO_4$, small (3-5 μm) prismatic crystals with n_p = 1.812; n_d = 1.846.

The studies of the macrostructure and crystal-optical analysis have shown that the high-temperature phase composition of adhesive compositions is represented by thermally stable compounds, which ensures high performance properties when gluing refractory materials.

V. CONCLUSION

As a result of determining the physical-mechanical and technological properties of the developed refractory adhesive compositions, it satisfies the requirements of the processed ones. As a result of petrographic studies of aluminophosphate adhesive compositions with the addition of clay components fired at different temperatures, it was found that polarizing phosphocrystobolite and phosphotridymite AlPO4 crystals of rhombic modification are observed in the phase composition of fired samples. These crystals are presented in the form of thermally stable compounds, which provides high performance properties when bonding refractory materials.

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