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Technology for the Reduction of Iron Oxides in Fluidized Bed Furnaces

Khasanov A.S.[1], Eshonqulov U.X.[2], Khojiev Sh.T.[3]

[1]Deputy Chief Engineer for Science at "Almalyk MMC" JSC, doctor of technical Science, Professor, Uzbekistan,
[2]Senior lecturer of the Department of Mining, Karshi Institute of Engineering and Economics, Karshi city, Uzbekistan,
[3]Associate Professor of the Department of Metallurgy, Tashkent State Technical University, Tashkent, Uzbekistan
E-mail: hojiyevshohruh@yandex.ru

Abstract— *The article examines the burning of pyrite concentrates in a heated bed furnace, and then the recovery of the obtained iron soot in this furnace using natural gas. The purpose of the research is to develop a blast furnace-free technology for obtaining ferrous metal. As a result of this, there will be an opportunity to involve in the production of pyrite enrichments, which are additionally extracted in copper beneficiation factories. This increases the level of complex use of raw materials in metallurgy. For this purpose, it is important to study the conditions of return of burnt pyrite enrichments with natural gas. This research is aimed at fulfilling this task, and the optimal conditions of the recovery process have been studied. The granulometric composition, activity, temperature, chemical and material composition of the product obtained as a result of the research were analyzed. Based on the discussion of the results obtained from the study, relevant conclusions are given.*

Keywords— metallurgy, pyrite, fluidized bed furnace, incineration, oxidation-reduction process, natural gas, granulometric analysis, iron.

INTRODUCTION

In non-ferrous metallurgy, oxidative roasting of materials rich in iron sulfides is used in relation to pyrrhotite gold-bearing concentrates, pyrite flotation tailings containing lead and precious metals to obtain iron flux, to obtain sulfuric acid, etc. in the chemical industry of Russia, about two thirds of sulfur pyrite, consumed for the production of sulfuric acid, is roasted in mechanical multi-hearth kilns and about one third in flash kilns [1-4]. Experience in pyrites roasting in a fluidized bed, accumulated at the Voskresensky chemical plant, showed its significant advantages over previously existing methods; the content of sulfur dioxide in roasting gases increased to 14%, and sulfur in the cinder decreased to 1% or less; the concentration of arsenic in the roasting gases has stabilized at a level not exceeding 0.1 g/m^3; the production of steam increased to 1.1 g per 1 g of pyrite; when fired in a suspended state, 0.5-0.6 tons of steam is obtained [5-7].

Calculations made about the Giprokhim Institute showed that the selling price of the produced steam exceeded by 12-15 rubles. the cost of firing 1 ton of pyrites. The consumption of metal and refractories for the construction of furnaces for firing in a fluidized bed is close to the consumption for the construction of furnaces in a suspended state and is 3-5 times less than for multi-hearth furnaces. The energy consumption per 1 ton of pyrites during firing in a fluidized bed is 40-50 kWh, and when firing in a suspended state and in mechanical furnaces - 28 kWh. The specific capital investments in the new furnace department per 1 tonne of annual pyrite production for all types of firing turned out to be the same (about 100 rubles). but with the reconstruction of the shop with the transfer to firing in a fluidized bed - 40% lower [8-15].

In terms of intensity, pyrite roasting in a fluidized bed is close to roasting in a suspended state (about 1 t/m^3 * duck or about 1.5 million kcal / m^3 * day) and is three times higher than in multi-hearth furnaces; the specific productivity of the furnace hearth area during pyrite roasting in a fluidized bed is about 9 t/m^2*day [16-17].

Abroad, the firing of pyrites in a fluidized bed, since 1951, has been widely developed, the number of such furnaces in almost all countries is about 150 pieces.

The possibility of firing in a fluidized bed sulfur-poor materials (up to 12% 5) significantly expands the natural resources of sulfur raw materials [18-19].

In 1958, at the pilot plant of the Sredneuralsk copper smelter, successful experiments were carried out on roasting in a fluidized bed of Gubakhinsky carbonaceous pyrite with 15% carbon, and a gas containing 8% sulfur dioxide was obtained. The firing was carried out at a temperature of 700-800° [20].

MATERIALS AND METHODS

As a result of the integrated laboratory experiments, an effective way was found to prevent sintering of reduced iron, which occurs at temperatures above 800-850 ºC. This method consists in the addition of granulated coke dust, the size of which was from

2 to 0.5 mm with a granule size of 0.8 mm. Coke acted as a baking powder and did not participate in the process, since the reducing gas did not contain oxygen, and the carbon dioxide content was significant (from 0.6 to 1.2%).

The optimal height of the fluidized bed under laboratory conditions was also determined, which was 1500 mm; air consumption 6 nm^3/h; natural gas consumption -1.7 nm^3/h, pressure of the gas-air mixture at the entrance to the conversion chamber - 1300-1500 mm water column [21].

Since in the present study the reduction experiments were carried out without prior pelletization of the material, it was necessary to create a fluidized bed bath from a larger material than the one being reduced in order to check the possibility of retaining more ionic material in a fluidized bed bath from a larger one. Coke was chosen as a large material.

It was found that the required coke size was minus 1.0-1.5 mm. The air consumption in the experiments was 3.9-4.2 Nm^3/hour; natural gas consumption 1.3-1.5 nm^3/hour.

Changing the composition and size of the catalyst in the conversion chamber (GIAP-3 catalyst) made it possible to lower the temperature of the conversion process to 900-950 ºC and the pressure of the gas-air mixture at the inlet to the conversion chamber, which amounted to 950-1000 mm of water column.

Since the coke size for the experiments was 1.0-1.5 mm, and the recovered material - 0.6 mm, -99%, the samples of the reduced cinder were sieved through a sieve - 0.6 mm in order to separate the coke, which was used as a circulating product.

Cinder samples (-0.6), dust from the dust chamber and bag filter dust were subjected to manual magnetic separation and the magnetic fraction after grinding was analyzed to determine Fe_{tot}; Fe_{met}; Fe bound in the form of FeO and other components.

RESULTS AND DISCUSSION

The composition of the charge in all experiments was as follows: 40% coke and 60% of the recovered material, a mixture of pyrite cinder and dust.

The temperature in the reaction zone in the first four experiments ranged from 850 to 920 º, in the next three experiments from 940 to 980 ºC.

The composition of the reducing gas phase in terms of the content of carbon monoxide and carbon dioxide varied from 15.4 to 17.2% and from 0.7 to 1.6%, respectively. Hydrogen concentration fluctuated within 31.35%. There was practically no oxygen in the reaction zone.

The loading of material into the reduction reactor ranged from 2.5 to 4 kg/h. The duration of the experiments was from 5 to 20 hours in the optimal mode.

In experiments 1 and 2, the temperature in the reaction zone ranged from 870-850, the material loading was 2.8 kg/hour. The compositions of the reducing gas differ slightly from each other. The recovery results in both experiments are very close to each other. The degree of recovery in both experiments is low due to the low firing temperature.

In experiments 3 and 4, the firing temperature is approximately the same, but higher than in the previous two. Ceteris paribus, the content of metallic iron and the degree of reduction in experiments 3 and 4 are higher than in experiments 1 and 2, which is explained by a slightly higher firing temperature (9000), despite even a slightly higher productivity in experiments 3 and 4.

The composition of the gas phase and the performance of experience No. 5 are the same as those of experience No. 1, but the temperature of experience No. 5 is 50-70 ºC higher. On the example of these two experiments, the effect of temperature on the reduction process is especially clearly seen: an increase in temperature to 940°C significantly increased the content of metallic iron in the samples of cinder and dust.

In experiments No. 6, No. 7, the composition of the gas phase, the temperature were the same, but with productivity they differ from each other, it is 3, 7 in the sixth experiment, and 3 kg / hour in the seventh.

A decrease in productivity by 23% made it possible to increase the content of metallic iron in cinders from 56.5% to 57.2%, i.e. by 5%.

From the table above (No.3) it can be seen that the decisive conditions for the reduction process are the temperature in the reaction zone and productivity, i.e. residence time of the material in the fluidized bed. The composition of the gas phase, which was optimal under the adopted conversion mode, varied within narrow limits in all experiments, so its effect on the reduction process could not be studied. The optimum process temperature lies in the range of 960-980 ºC, and the productivity is from 3 to 4 kg/h of charge, which corresponds to from 3.64 to 4.25 t/m^2 of iron powder with an active iron content of 55-60%.

Tables 1 and 2 present the chemical and phase analyzes of the recovery products.

Table 1

Chemical composition of iron powder (magnetic fraction)

Components	Content, %
Iron total	65,1
Copper	0,60
Zinc	0,29
Sulfur total	0,55

Carbon	0,70
Silica	8,52
Alumina	0,83
calcium oxide	1,55
magnesium oxide	0,97
Total:	79,11

Table 2

Phase composition of iron powder

Components	Content, %
Iron metal (active)	60,0
Iron oxide	not
ferrous iron	3,4
Iron sulfide	not
Total	63,4

Tables 1-5 show sieve analyzes of coke, charge, cinder, mixture of cinder and dust, dust from the dust chamber and from the bag filter.

As can be seen from a comparison of the data in tables 3, the cinders are larger than the dust from the chamber, which is explained by the presence of some coarsening of the material during the firing process, which can provide some increase in the productivity of the process.

Table 3

Sieve analysis of used coke

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	-	-
-14+35	-1+0,5	38,6	38,6
-35+60	-0,5+0,246	28,6	57,20
-60+90	-0,246+0,16	15,25	82,45
-90+150	-0,16+0,104	6,0	88,45
-150+200	-0,104+0,074	4,45	92,90
-200+250	-0,074+0,052	2,15	95,05
-250	-0,052	4,85	99,90
		99,9	

The weighted average grain diameter is 0.44 mm.

Table 4

Sieve analysis of charge

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	0,45	0,45
-14+35	-1+0,5	16,80	17,85
-35+60	-0,5+0,246	15,20	32,24
-60+90	-0,246+0,16	15,35	47,80
-90+150	-0,16+0,104	13,40	61,20
-150+200	-0,104+0,074	16,70	77,90
-200+250	-0,074+0,052	11,75	89,65
-250	-0,052	10,20	99,85
		99,85	

Table 5

Sieve Analysis of Recovered Screened Cinder Before Magnetic Separation (Experiment number 7)

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	-	-
-14+35	-1+0,5	9,8	9,8
-35+60	-0,5+0,246	66,7	76,5
-60+90	-0,246+0,16	20,75	97,25

-90+150	-0,16+0,104	2,25	99,50
-150+200	-0,104+0,074	0,25	99,75
-200+250	-0,074+0,052	0,10	99,85
-250	-0,052	0,10	99,95
		99,95	

Average grain diameter =0.37mm,
Average-weighted grain diameter = 0.26mm.

Table 6

Sieve analysis of cinder (Experiment No. 7) (magnetic fraction)

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	-	-
-14+35	-1+0,5	8,85	8,85
-35+60	-0,5+0,246	70,40	79,25
-60+90	-0,246+0,16	19,40	96,65
-90+150	-0,16+0,104	1,10	99,75
-150+200	-0,104+0,074	0,10	99,85
-200+250	-0,074+0,052	0,10	99,95
-250	-0,052	0,05	100,0
		100,0	

Average-weighted grain diameter = 0.37mm

Table 8

Sieve analysis of a mixture of cinder and dust from the chamber (magnetic fraction, Experiment No. 6)

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	0,70	0,70
-14+35	-1+0,5	13,40	14,10
-35+60	-0,5+0,246	31,85	45,25
-60+90	-0,246+0,16	19,40	65,35
-90+150	-0,16+0,104	11,55	76,90
-150+200	-0,104+0,074	11,55	88,75
-200+250	-0,074+0,052	6,15	94,90
-250	-0,052	5,0	99,9
		100,0	

The average grain diameter is 0.29mm,

As can be seen from tables 8, 9, mixing the cinder with dust from the chamber reduces the average grain diameter from 0.37 mm (cinder) to 0.29 mm.

The sieve analyzes in Tables 8 and 9 are given as averages after the sieve composition has been stabilized based on a large number of analyzes of intermediate samples of cinder and dust from the chamber.

Table 9

Sieve analysis of dust from the dust chamber (magnetic fraction)

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	-	-
-14+35	-1+0,5	1,0	1,0
-35+60	-0,5+0,246	4,9	5,9
-60+90	-0,246+0,16	13,5	19,4
-90+150	-0,16+0,104	25,4	44,8
-150+200	-0,104+0,074	27,8	72,6
-200+250	-0,074+0,052	13,2	85,8
-250	-0,052	14,2	100,0
		100,0	

Average weighted grain diameter - 0.12 mm.

Table 10

Sieve analysis of bag filter dust (non-demagnetized)

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
+14	+1	-	-
-14+35	-1+0,5	-	-
-35+60	-0,5+0,246	5,8	5,8
-60+90	-0,246+0,16	15,1	20,9
-90+150	-0,16+0,104	18,4	39,3
-150+200	-0,104+0,074	19,2	58,5
-200+250	-0,074+0,052	19,6	78,1
-250	-0,052	21,9	100,0
		100,0	

Average-weighted grain diameter - 0.14 mm.

The output of the magnetic fraction from the loaded iron-containing material was in all experiments 75-78%.

Table 11 shows the material balance of experience 7.

Table 11

Material balance experience 7

Loaded, kg		Received, kg		Yield of solid roasting products, %
Iron material	17,4	cinder end	17,62	75,5
Coke	11,4	Dust from chamber		20,08
		Dust from bag filter	1,0	4,42
Total	29,0	Total	23,31	100%

The balance discrepancy is 5.69 kg. The large discrepancy is explained by the fact that about 3.5-4 kg of iron-bound oxygen is carried away from the reduction zone along with the exhaust gases.

Table 12 shows the iron balance.

Table 12

Iron balance

Loaded, kg	Received, kg
Iron contained in the source material 17,4*0,504=8,76	In the cinder end 6,092 In the dust from the chamber 1,655 In bag dust 0.605
Total 8,76	Total 8,352
Discrepancy - 0.408 kg	

In order to elucidate the carburization behavior of copper reduced in a fluidized bed of iron powder, the enrichment laboratory conducted carburation experiments, for which cinders with an active iron content of 58 and 60% and dust from a dust chamber containing 50% active iron were used. The results of experiments on cementation are given in Table 13.

Table 13

Results of experiments on cementation of copper with reduced iron powder

№	Name of products	Exit, %	Copper content, %	Extraction of copper, %	Note
1.	Copper concentrate Industrial product Tails	15,5 7,2 77,3	6,45 0,55 0,20	83,7 3,4 12,9	Iron shavings act.85%
Ore		100,0	1,19	100,0	
2.	Copper concentrate Industrial product Tails	9,9 2,8 87,3	7,35 1,30 0,48	61,4 3,1 35,5	Cinder, act.60%
Ore		100,0	1,18	100,0	
3.	Copper concentrate Industrial product Tails	10,2 5,0 84,7	7,88 1,90 0,33	68,0 8,0 24,0	Dust, act.50%
Ore		100,0	1,18	100,0	
4.	Copper concentrate Industrial product	13,2 5,3	7,9 0,44	86,6 1,9	Crushed cinder, activity 60%

	Tails	81,5	0,17	11,5	
Ore		100,0	1,20	100,0	
5.	Copper concentrate Industrial product Tails	13,1 4,2 82,7	8,0 0,68 0,16	85,4 2,4 12,2	Crushed cinder, activity 58%.
Ore		100,0	1,22	100,0	

The first three experiments were carried out with unground cinders and dust.

Their sieve analyzes are given in Tables 8 and 11. Table 15 shows that the results for carburizing with dust are worse than with iron chips, since its activity is much lower.

When carburizing with iron powder (cinder), the results are also lower than with iron chips due to the significantly larger size of the cinder compared to chips with a size of minus 0.2 mm, 100%.

Experiments 4 and 5 were carried out with crushed cinders of the same activity. Sieve analysis is given in Table 14.

Table 14

Sieve analysis of crushed cinder

Sieve size, mesh	Grain size, mm	Yield class, %	Total yield, %
-60+90	+0,16	36,8	36,8
-90+150	-0,16+0,104	21,1	57,9
-150+200	-0,104+0,074	11,7	69,6
-200+250	-0,074+0,052	9,9	79,5
-250	-0,052	20,5	100,0
Total		100%	

From Table 16 it can be seen that the results for cementation with crushed cinder are significantly better than with coarse cinder of the same activity, and even slightly better than with iron shavings.

CONCLUSION

Based on the results of experiments carried out on an enlarged-laboratory installation of continuous operation, it was established that it is possible to carry out the process of reducing enlargement of a very thin material from a mixture of pyrite cinders and dust (products of burning pyrite concentrate in furnaces of the FBF).

The mode and indicators of recovery firing of non-granulated material have been specified:

a) the coke size for creating a fluidized bed bath was minus 1.5-1.0 mm with an average weighted grain diameter of 0.5 mm; the recovered material had a weighted average grain diameter of 0.12-0.15 mm;

b) the optimal temperature in the fluidized bed, depending on the activity and fineness. The resulting iron powder is 940-980 °;

c) the productivity of the process for active iron powder, depending on the temperature, is 2.6-3.7 t/m^2 per day;

d) the yield of non-magnetized solid roasting products is: cinder -70-75.5%, dust from the chamber - 21.0 -25%, dust from the bag filter - 4.42%. The output of the magnetic fraction of the cinder is dust 75%.

As a result of experiments in the temperature range of 970-980 °, the degree of reduction achieved is 90-92%, while the content of active iron in the magnetic fraction of the cinder is 58-60%. The dust from the chamber is somewhat poorer in the content of active iron, but it is sufficient for its use in the process of copper cementation. However, in the specified temperature range, the iron powder is relatively coarser than at temperatures of 930-940 °.

The active powder obtained at a temperature of 970-980 ° (cinder and dust) was tested in the enrichment laboratory of Gintsvetmet. Tests have shown that when cementing with cinder with 60% activity and an average grain diameter of 0.37 mm, the results are worse than when cementing with iron shavings with 85% activity. Obviously, it is necessary to use a finer powder, which is confirmed by the results of cementation with a reduced finer powder in the experiments of 1959, as well as experiments on cementation with powder crushed to a fineness of minus 0.2 mm, obtained at a high temperature of 970-980 °C.

The specific gas consumption in comparison with previous experiments decreased and amounted to 1290 Nm^3 per 1 ton of iron powder.

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