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THE STUDYING OF PYRITE AND ARSENOPYRITE SPECIFIC SURFACE AND POROSITY AT ROASTING CONDITION

Abstract. The paper presents the results of studying the modification of the specific surface area and porosity of pyrite and arsenopyrite during their thermal decomposition. It has been established that the decomposition of pyrite and arsenopyrite is accompanied by an increase in the pore volume from $0.303 \cdot 10^{-3}$ to $1.23 \cdot 10^{-3}$ with an increase in the degree of pyrite decomposition by about 41%, and then a decrease by about $0.131 \cdot 10^{-3} \text{ m}^3/\text{g}$, at a degree of decomposition of about 100%. The surface area of the pores increases from 0.31 to $1.085 \text{ m}^2/\text{g}$ with a subsequent decrease due to the coarsening and merging of small pores. An increase in porosity is accompanied by an increase in the aerodynamic resistance of particles resulting from increased dust formation of the furnace.

Keywords: pyrite, arsenopyrite, sulfur, surface area, porosity, thermal decomposition, degree of decomposition, furnace reaction zone.

Пирит пен арсенипириттің меншікті беті мен кеуектілігін зерттеу

Аннотация. Мақалада пирит пен арсенипириттің термиялық ыдырауындағы меншікті бетінің өзгеруі мен кеуектілігін зерттеу нәтижелері келтірілген. Пирит пен арсенипириттің ыдырауы кеуек көлемінің $0,303 \cdot 10^{-3}$ -тен $1,23 \cdot 10^{-3}$ -ке дейін ұлғаюымен, пириттің ыдырау дәрежесінің шамамен 41% жоғарылауымен, содан кейін ыдырау дәрежесі шамамен $0,131 \cdot 10^{-3} \text{ м}^3/\text{г}$ төмендеуімен бірге жүретіні анықталды. шамамен 100%. Кеуектердің беткі қабаты $0,31$ -ден $1,085 \text{ м}^2/\text{г}$ -ға дейін артады, содан кейін ұсақ кеуектердің кеңеюі мен бірігуіне байланысты төмендейді. Кеуектіліктің жоғарылауы пештің шанының жоғарылауы нәтижесінде пайда болатын бөлшектердің жел жылдамдығының жоғарылауымен, пирит пен арсенипириттің бөлшектерін пештің реакциялық аймағынан ауа ағынымен ішінара ыдыратуымен бірге жүреді.

Түйінді сөздер: пирит, арсенипирит, күкірт, меншікті бет, кеуектілік, термиялық ыдырау, ыдырау дәрежесі, пештің реакциялық аймағы.

Исследование удельной поверхности и пористости пирита и арсенипирита

Аннотация. В статье приведены результаты изучения модификации удельной поверхности и пористости пирита и арсенипирита при их термическом разложении. Установлено, что разложение пирита и арсенипирита сопровождается увеличением объема пор с $0,303 \cdot 10^{-3}$ до $1,23 \cdot 10^{-3}$ при увеличении степени разложения пирита примерно на 41%, а затем уменьшением примерно на $0,131 \cdot 10^{-3} \text{ м}^3/\text{г}$ при степени разложения около 100%. Площадь поверхности пор увеличивается с 0,31 до $1,085 \text{ м}^2/\text{г}$ с последующим снижением за счет укрупнения и слияния мелких пор. Повышение пористости сопровождается увеличением аэродинамического сопротивления частиц, возникающим в результате повышенного пылеобразования печи.

Ключевые слова: пирит, арсенипирит, сера, удельная поверхность, пористость, термическое разложение, степень разложения, реакционная зона печи.

Introduction

The mining industry is experiencing difficulties associated with an increase in demand for metals. Discovery rates are slowing, grades are declining, and undeveloped deposits tend to be complex, presenting us with unique environmental and technological challenges. Careful study of deposits and the use of acquired knowledge in the field of metal recovery is a key aspect of the successful implementation of new mining projects and extending the life of old ones. The prospect of processing arsenopyrite ores is to extract gold, but the problem lies in the refractory of such ores, and often even in double refractory, the study of the structure of arsenopyrite, specific surface, porosity will make it possible to more accurately determine the conditions for the technological opening of gold [1].

Gold that is adsorbed onto the surface of other minerals during primary ore formation, oxidation, and/or processing is generally referred to as surface-bound gold. It is this kind of gold that is often found in pyrite-arsenopyrite ores. [2] In refractory ores, the gold is strongly isolated from the cyanide solution, and some oxidative pre-treatment is necessary to open it, this may be roasting, oxidation (biological or autoclave) or ultrafine grinding followed by oxidative leaching to make the ores suitable for cyanidation.

Thus, it is relevant to study the structure of arsenopyrite, specific surface area and porosity for the subsequent technological opening of gold.

As a result of ore base exhaustion of ferrous and nonferrous metallurgy at metallurgical treatment are involving more complicated and less qualitative ores. Magnetizing ores content considerable quantity of pyrite complicate the technology

of iron extraction. At the same time the pyrite concentrates processing at dressing content different nonferrous metals in industrial quantities [3-4].

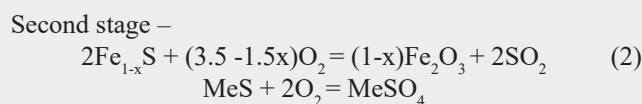
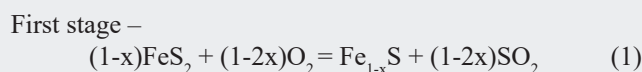
Extracting ores of nonferrous metals are contenting considerable quantity of arsenic at the present time. Extraction of base metals from complicated raw materials demands new technologies. Arsenic contenting in ores is necessary to utilize (waste burial) in more ecological compatible form. Arsenic on acting metallurgical and chemical enterprises from technological cycle is withdrawing in the form of arsenate – arsenite cakes, burial of their may lead to seriously ecological problems as times goes by.

It was carried out the cycle of researching by pyrite, arsenopyrite and arsenate – arsenite cakes treatment with the object of arsenic withdrawal in sulfide form – in more ecological and safe form and with the object of preparation of initial ore raw materials to following pyro- and hydrometallurgical treatment [5]. As a result of sulfidizing of arsenate – arsenite cakes by pyrite are forming the volatile arsenic forms and pyrrhotite of variable structure.

The experience of one-stage sulfatizing roast carrying out of pyrite concentrates in fluidized bed furnaces is show that more higher results by sulfurizing (the degree of cobalt sulfatizing – till 75%) achieve at carrying out of process in furnaces with return cyclone.

The positive influence on return cyclone degree of sulfatizing may be explain by that underdecomposed substances of higher sulfides return on roast from cyclone the time of sulfatizing at that is increase. This circumstance is increase the duration of process and decrease the productivity of roast.

We propose to conduct roasting in two stages: pyrrhotizing roasting in the atmosphere with limited oxygen quantity at the first stage and oxidizing-sulfatizing roasting of pyrrhotizing cinder at the second stage according to the next equations:



Using two-stage process of roasting of pyrite concentrates instead of one-stage roasting allows to decrease by 40 % the necessity heart of the furnaces, to increase the efficiency of use of blast oxygen, to decrease more than twice the quantity of the flush acid, to improve labor conditions and to stabilize the technological parameters of the process.

The pilot scale testing of the technology of oxidizing-sulphatizing roasting of pyrite-cobalt concentrates from Sokolovsk-Sarybai Integrated Industrial Plant gave extraction into solution of 90.0% (2).

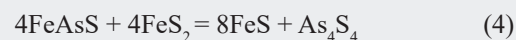
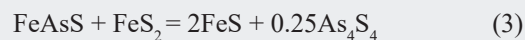
On the basis of the results of laboratory and pilot scale tests of the two-stage roasting technology (pyrrhotizing-sulphatizing) and their comparison with the results of the one stage sulphatizing we give recommendations on improving the two stages process of sulphatizing roasting of pyrite concentrates with the aim of increasing cobalt and nickel sulphatization degree with simultaneous decrease of iron sulphatization, increase of roasting efficiency, increase of the oxygen blast utilization coefficient, increase of sulfur dioxide and decrease of sulfur trioxide in the escaping gases, etc [6-7].

Raw materials with strictly limited arsenic content are used in smelting processes. Direct hydrometallurgical processing of raw materials with high arsenic content is used in a limited number of cases. Forecasting of the roasting process parameters is possible from the study of behavior of arsenic-bearing materials under high temperatures. The behavior of arsenopyrite under roasting is of theoretical and practical interest. The mechanism of pyrite and arsenopyrite decomposition at their thermal treatment has an influence on properties and behavior.

The research has shown that during the pyrite roasting in the autogenous mode with limited oxygen content in the gaseous phase the quantity of the sulfide anhydride and elemental sulfur formed in course of the reaction depends on the blast oxygen content (1). Presence in the roasting gaseous phase of high concentrations of sulfur dioxide and elemental sulfur will influence the mechanism of arsenopyrite roasting and the composition of the products formed under roasting.

Behavior of roasted arsenopyrite was studied by many authors. In [8] it was determined that arsenopyrite begins to dissociate at 550°C and at 700-900°C the process goes on with high velocity. Two stages of the process are observed: first arsenic vapor and high sulfur pyrrhotite are formed, and then the high pyrrhotite changes into low sulfur pyrrhotite and troilite. Thermodynamic analyses has shown that under thermal processing of arsenopyrite in the presence of pyrite and limited quantities of oxygen in the gaseous phase forma-

tion of arsenic sulfides is possible according to the following reactions:



The process of arsenopyrite thermal treatment in faintly oxidizing atmosphere also accomplish by arsenic isolation to gas phase with pyrrhotites of variable structure forming, contenting some quantity of arsenic [9-11].

The arsenopyrite dissociation velocity depends on the grain size of the initial material. It is established that the velocity of arsenopyrite dissociation process limited by diffusion factor. In the presence of pyrite the arsenopyrite dissociates at lower temperatures and with higher velocities.

In this work introduce the results of studying of pyrite and arsenopyrite specific surface modification and porosity at their thermal decomposition at depends of decomposition degree [12].

Initial materials

The structure of arsenopyrite %: iron – 34.8, arsenic – 44.3, sulfur 20.5 and monomineral pyrite (99.05% pyrite), taking from industrial iron-magnetite ore, contenting 53.9% of iron and 45.25% sulfur.

Methods and equipment

The decomposition of initial materials was realized in fixed layer in alumina tube. We studied the initial materials influence of decomposition degree on specific surface and porosity of cinders. The different decomposition degree was provided by variation of temperature from 650 until 850° C, and duration from 15 until 60 minutes.

Initial materials and decomposition products analyzed by chemical and X-ray methods, carried out electron – microscopic researching and defined their specific surface and porosity.

The electron – microscopic researching were carried out on electron scanning microscope JEOL M – 25 S3.

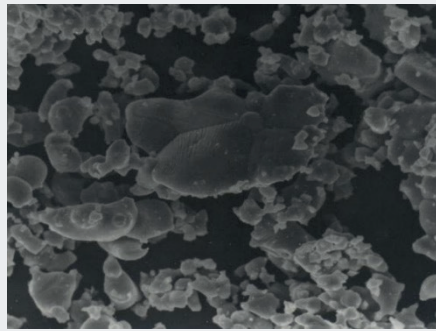
Electron – microscopic researching show that surface of initial pyrite is too much developing. Were defined the pores, splits in pyrite grains (figures a, b).

At the beginning of pyrite decomposition is observing the increasing of particles porosity, their some loosening making increasing their specific surface. At the end of decomposition the quantity of pores are decrease, the separate pores interflow, the size of pores increase and surface of materials smoothing (c, d). Reaction descends from surface to depth of particle. The form of initial particles are not change, the considerable particle destruction is absent.

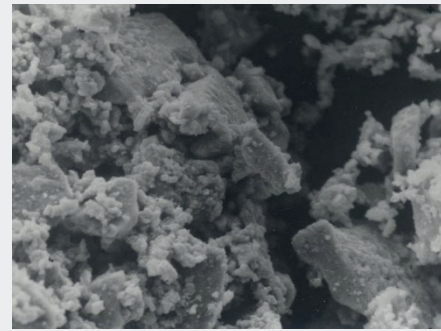
Photomicrographs of forming pyrrhotites at pyrite decomposition show the forming of layer-like structure (fig. 1c), this mechanism of appearance such structure demand additional studying. The results of pyrrhotites samples show that solution is occur anisotropically by layer in acids.

Electron-microscopic studying of arsenopyrite and products of arsenopyrite decomposition show that at arsenopyrite decomposition is forming the friable, porous structure with very developing surface. The decipitation of arsenopyrite grains is absent, geometrical sizes and particles form is differ slightly.

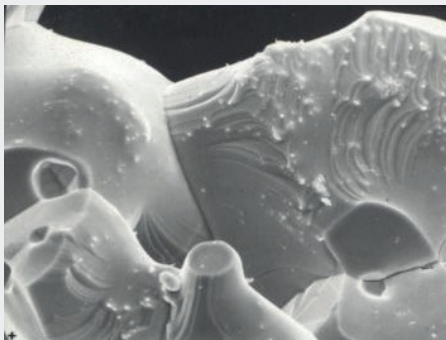
Specific surface and porosity were defined by BET methods by quantity of adsorbing nitrogen at his boiling-point.



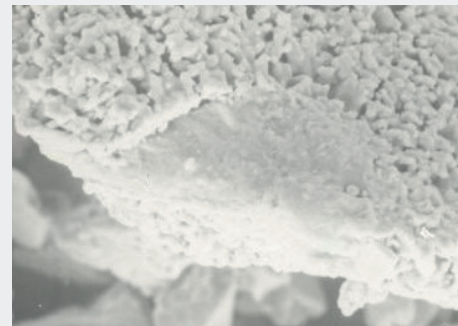
a) x300



b) x1000



c) x1500

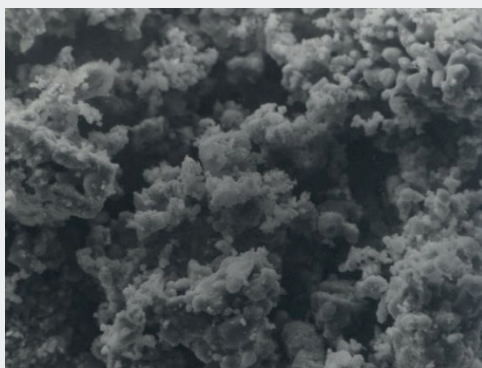


d) x1500

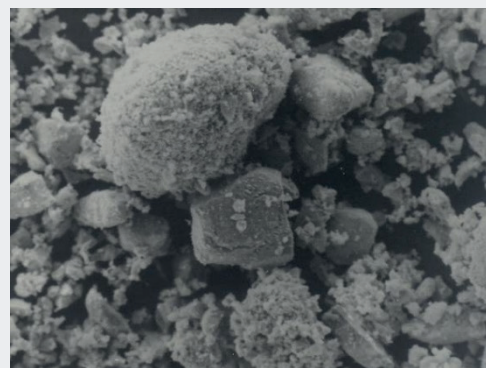
Figure 1. Electron photomicrography of initial pyrite and products of pyrite decomposition: a – initial pyrite concentrate; b – products of pyrite decomposition on 50%; c, d – products of pyrite decomposition on 95%.

Сурет 1. Бастапқы пирит пен оның ыдырау өнімдерінің электронды фотомикрографиясы: а – пириттің бастапқы концентраты; б – пириттің 50-ге ыдырау өнімдері %; с, д – пириттің 95-ке ыдырау өнімдері %.

Рис. 1. Электронная фотомикрография исходного пирита и продуктов его разложения: а – исходный концентрат пирита; б – продукты разложения пирита на 50%; с, д – продукты разложения пирита на 95%.



a) x200



b) x4500

Figure 2. Electron photomicrography of initial arsenopyrite and products of arsenopyrite decomposition: a – initial arsenopyrite; b – products of arsenopyrite decomposition on 97%.

Сурет 2. Бастапқы арсенопирит пен арсенопириттің ыдырау өнімдерінің электронды фотомикрографиясы: а – бастапқы арсенопирит; б – арсенопириттің 97-ге ыдырау өнімдері %.

Рис. 2. Электронная фотомикрография исходного арсенопирита и продуктов разложения арсенопирита: а – исходный арсенопирит; б – продукты разложения арсенопирита на 97%.

BET (Brunauer – Emmett – Taylor) analysis – analysis of measurement of the specific surface of dispersed materials by the method of low-temperature gas adsorption.

Specific surface was defined by following equations (5, 6):

$$S = \frac{X}{M} \cdot N \cdot A_N \cdot 10^{-20} \quad (5)$$

where: S – specific surface of sample, m^2/g ;

X – mass of nitrogen monomolecular layer, adsorbing 1 g of sample;

M – nitrogen molecular weight, $M = 28$ g;

N – number of molecules in gram-mole, $N = 6,023 \cdot 10^{23}$ molecules/mole;

A_N – area, engaging by one adsorbing nitrogen molecule $10^{-23} m^2/molecule$.

$$A_N = 1,091 \left(\frac{M}{\rho N} \right)^{\frac{2}{3}} \cdot 10^{16} m^2/molecule, \quad (6)$$

ρ – nitrogen density; $\rho = 0,80925$ g/sm³;

$A_N = 16,2 \cdot 10^{-20} m^2/molecule$.

If we should substitute the getting expressions in initial equation we get the equation for calculation of specific surface:

$$S = \frac{X}{M} \cdot 6,023 \cdot 10^{23} \cdot 16,2 \cdot 10^{-20} = \frac{1}{(m+1) \cdot M} \cdot 9,75726 \cdot 10^4 \quad (7)$$

Results and discussion

The studying of modification of pyrite and arsenopyrite specific surface and porosity at their thermal decomposition.

Were studied the pyrite decomposed in nitrogen atmosphere at 823, 873, 923 and 973 K, decomposition degree was equal 20,1, 41,0, 85,8 and 100% accordingly, and arsenopyrite was decomposed at 823, 873, 923 and 973 K, decomposition degree was equal – 19,1, 35,0, 84,8 and 97,0%. The calculation of adsorbing nitrogen weight was made according with B.E.T. (Brunauer – Emmett – Taylor) method.

Table 1

Specific surface (S_{ss}) and porosity products of pyrite decomposition in nitrogen atmosphere

Кесте 1

Азот атмосферасындагы пириттің ыдырау өнімдерінің меншікті беті (S_{ss}) және кеуектілігі

Таблица 1

Удельная поверхность (S_{ss}) и пористость продуктов разложения пирита в атмосфере азота

T, K	Decomposition degree, %	S _{ss} , m ² /g	Pore radius, 10 ⁻¹⁰ m	Void space, sm ³ /g	Pores surface area, m ² /g	Pore distribution, %	Total Pores surface, m ² /g
823	20.1	0.79	More 84.067	0.0503	0.0162	5.60	0.0162
			57.911-84.067	0.0768	0.0578	19.90	0.074
			38.239-57.911	0.0649	0.076	34.60	0.11405
			19.229-38.239	0.065	0.0806	48.0	0.21935
873	41.0	1.7	More 84.067	0.0372	0.12	5.1	0.012
			57.911-84.067	0.2345	0.0317	13.6	0.0437
			38.239-57.911	0.158	0.401	36.9	0.829
			19.229-38.239	0.2392	0.256	23.7	1.085
923	85.8	1.57	More 84.067	0.4725	0.152	14.0	0.152
			57.911-84.067	0.3660	0.276	25.4	0.428
			38.239-57.911	0.104	0.1218	24.6	0.2602
			19.229-38.239	0.145	0.2549	47.4	0.4951
973	100	1.20	More 84.067	0.1010	0.0325	6.6	0.0325
			57.911-84.067	0.1404	0.1059	21.4	0.1384
			38.239-57.911	0.0776	0.0908	24.1	0.1515
			19.229-38.239	0.1313	0.2253	58.8	0.3768

The results of studying specific surface and porosity show (table 1), that the initial pyrite concentrate has very developing surface this confirmed by the results of electron-microscopic researching.

With the increasing of decomposition degree the void space is increasing from $0.303 \cdot 10^{-3}$ to $1.23 \cdot 10^{-3}$ at increasing of pyrite degree decomposition about 41% and then decrease about $0.131 \cdot 10^{-3} m^3/g$ at degree of decomposition near 100%. The sur-

face area of pores increase from 0.31 to 1.085 m²/g with following lowering due to 0.18-0.138 m²/g, specific surface increased at the beginning from 1.088 to 1.7 m²/g, and decrease to 0.488.

As development of dissociation process is occur the desulphurization that lead to confluence of separate micropores, forming of large pores, decreasing of specific surface. Electron-microscopic researching are confirming such modification of pyrite particles in decomposition process. Thus,

modification of pyrite porosity and specific surface at decomposition will be influence on velocity of gaseous sulfur removal forming as a result of process.

Further are introduced the results of researching specific surface and porosity products of arsenopyrite decomposition in nitrogen atmosphere (table 2).

Table 2
Specific surface (S_{ss}) and porosity products of arsenopyrite decomposition in nitrogen atmosphere

Азот атмосферасындағы арсенипириттің ыдырау өнімдерінің меншікті беті (S_{ss}) және кеуектілігі
Кесте 2
Удельная поверхность (S_{ss}) и пористость продуктов разложения арсенипирита в атмосфере азота
Таблица 2

T, K	Decomposition degree, %	S _{ss} , m ² /g	Pore radius, 10 ⁻¹⁰ m	Void space, sm ³ /g	Pores surface area, m ² /g	Pore distribution, %	Total Pores surface, m ² /g
823	19.1	0.85	More 84.07	0.0061	0.00201	0.5	0.00201
			57.91-84.07	0.0786	0.06912	13.3	0.7113
			38.24-57.91	0.1181	0.1317	29.7	0.60283
			19.23-8.24	0.140	0.2509	56.5	0.45373
873	35.0	1.89	More 84.07	0.2463	0.0776	7.3	0.0776
			57.91-84.07	0.1428	0.1068	10.0	0.1844
			38.24-57.91	0.2897	0.373	35.0	0.5574
			19.23-8.24	0.231	0.5079	47.7	1.0653
923	84.8	1.23	More 84.07	0.2372	0.0758	12.6	0.0758
			57.91-84.07	0.1406	0.1062	16.6	0.1820
			38.24-57.91	0.1276	0.1430	23.7	0.3250
			19.23-8.24	0.163	0.2783	46.1	0.6033
973	97.0	1.13	More 84.07	0.2483	0.0787	15.5	0.0787
			57.91-84.07	0.1534	0.1166	12.9	0.1947
			38.24-57.91	0.1483	1.0854	34.9	0.371
			19.23-8.24	0.0831	0.1346	26.7	0.5056

The results show that (table 2) as a developing of arsenopyrite thermal dissociation process specific surface and porosity of products of arsenopyrite decomposition is increasing at first (also windage) and at the end of decomposition are decreasing. At the degree of decomposition near 100% pores surface area increasing from 0.1166 until 1.0854 m²/g with following decreasing due to enlargement and confluence of minor pores.

From the beginning of process are forming many minor pores which for the end of decomposition enlarge. The increasing of windage partially decomposed pyrite and arsenopyrite particles could lead to their higher emission with dust obviously.

At sulfatizing roast of sulfide concentrates observe dust emission of underdecomposed pyrite particles possessing heightened windage which could be located by dust extractors. At the return of locating dust on roast from return cyclone pyrite decomposition degree increase and their windage diminish. Dust particles returning from return cyclone partially not rush out and follow to sulfatizing roast in full capacity.

Conclusion

Thus, was established that pyrite and arsenopyrite decomposition accomplish by increasing of porosity and specific surface at increasing of pyrite decomposition degree until 40-45% at following decomposition the specific surface and porosity are decreasing – occur enlargement and confluence of minor pores this also confirm by results of microscopic analysis. The increasing of porosity accomplishes by increasing of particle windage and this is lead to heightened dust emission partially decomposed pyrite and arsenopyrite particles by air stream from furnaces reaction zone. This is explaining heightened contention of sulfide sulfur in roast dusts of sulfide concentrates.

Simultaneous modification of pyrite and arsenopyrite specific surface and porosity show that for more full arsenic sulfidizing of arsenopyrite the process more effectively realize in continuous conditions at constant receipt of elemental sulfur to reaction zone.

Acknowledgements

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ВСЕРОССИЙСКАЯ НАУЧНО-ТЕХНИЧЕСКАЯ КОНФЕРЕНЦИЯ С УЧАСТИЕМ ИНОСТРАННЫХ СПЕЦИАЛИСТОВ «ЦИФРОВЫЕ ТЕХНОЛОГИИ В ГОРНОМ ДЕЛЕ»



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