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THE HYDROPHILIC-HYDROPHOBIC TRANSITIONS ON CHALCOPYRITE: ELECTROCHEMICAL STUDY

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The properties of chalcopyrite were studied by the methods of cyclic voltammetry, potentiometry, X-ray photoelectron spectroscopy and various techniques of the evaluation of wettability. Results of an experimental study of the potential and pH dependencies of the various characteristics of chalcopyrite hydrophobicity: detachment force, contact angle, gas evolution pressure and floatability, are discussed. It is shown that the detachment force/potential polarization curves are similar in shape for the case of the absence of xanthate and for the presence of all xanthates studied. The electrochemical polarization leads to generation of hydrophilic compounds and increases wettability of the mineral. A formation of the hydrophilic layer of iron hydroxide is a reason for decrease of hydrophobicity and floatability under oxidative conditions. Xanthate desorption leads to hydrophilisation of the mineral surface and depression under flotation in the cathodic area.

Key words:chalcopyrite, flotation, induction time, wettability cyclic, voltammetry

INTRODUCTION

Chalcopyrite is one of the most important for industry and therefore well-studied sulfide mineral [Gardner, Woods, 1979; Trahar, 1983; Finkelstein et al., 1975; Heyes, Trahar, 1977; Ammou-Chokroum et al. 1979; Peters, 1977; Eddington, 1977]. Electrochemical reactions of chalcopyrite are important for its processing by a wet mechanical dressing, flotation, and hydrometallurgy. Common practice of the operating with ores includes regulations of its redox-conditions for an optimal treatment. Fig.1 [Chanturiya, Vigdengrauz, 1993] illustrates changes of the immersed in slurry platinum electrode potential under the influence of air dissolution, sodium sulfide and potassium xanthate additions.

Irreversible changes of chalcopyrite surface composition during its polarization are well documented. In the presence of water and oxygen, chalcopyrite is thermodynamically unstable, but its oxidation is a rather slow process. This is the

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reason for the existence of chalcopyrite in ore bodies and even in flotation tails stored for many years. There has been general agreement regarding the species responsible for rendering chalcopyrite hydrophobic and floatable. In the case of natural floatability, it is elemental sulfur and for the case of xanthate-induced flotation, it is dixanthogen and copper xanthate [Gardner, Woods, 1979; Trahar, 1983; Finkelstein et al., 1975; Heyes, Trahar, 1977; Eddington, 1977; Chanturiya, Vigdergauz, 1993].

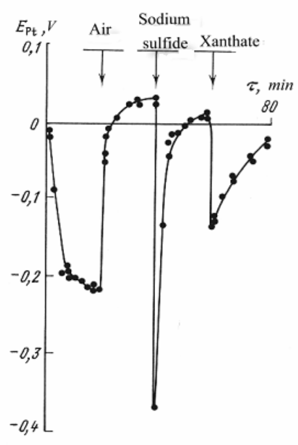

Fig. 1. Influence of reagents on the pulp potential of copper sulfide flotation

Polarization of solid electrodes changes the surface tension or surface free energy at the solid-liquid interface (γ_{sl}) that leads to the alteration of the angle of wettability. A method for the determination of the point of zero charge for metals that is based on this phenomenon is widely known [Frumkin et al., 1952]. Metal wettability has a minimum at this point. It is reasonable to assume that for the mineral-solution interface, γ_{sl} and therefore contact angle will also depend on the surface charge. As long as the charge on the mineral surface depends on the H^+ and OH^- ion concentrations, it is attractive to study pH dependences of minerals' wettability. In this paper, a summary is presented of the pH and potential dependencies of chalcopyrite wettability and possible reasons for these phenomena are proposed. There are discussed results of electrochemical studies of redox-transitions, sorption activity, wettability and flotation response of a chalcopyrite.

EXPERIMENTAL

REAGENTS PREPARATION

For the preparation of borate and phosphate buffer solutions with ionic strength $5 \cdot 10^{-2}$M, commercial salts of analytical purity were used. Potassium xanthates were obtained as commercial products from Hoechst. They were purified according to the standard procedure of two precipitations from acetone by addition of hexane.

MINERAL USED AND SURFACE PREPARATION

All experiments were carried out with n-type chalcopyrite from Kafan (Armenia) that was described earlier [Chanturiya, Vigdergauz, 1993]. Some characteristics are: Cu content 22.6; Fe content 39.0; conductivity – $3.7 \cdot 10^{-3}\ \Omega m^{-1}$. For flotation experiments, chalcopyrite was hand-selected, ground dry with an agate mortar and pestle, sized to -0.16+0.08 mm, rinsed with water to remove fines, dried, and stored in an dessicator. For cyclic voltammetry (CV), potentiometric and rotating disk (RD) experiments, a chalcopyrite disc with a diameter of 5 mm was mounted in a fluoroplast holder. The working surface was dry-polished in stages using alumina down to 0.05 μm and rinsed with distilled water or sometimes with ethanol between polishing stages. The surface was repolished immediately before using.

APPARATUS AND PROCEDURE

The mineral surface was studied by X-ray photoelectron spectroscopy (XPS) and electrochemical techniques (potentiometry, electrophoretic mobility measurements, CV and RD techniques). The X-ray photoelectron spectra were recorded with an X-ray photoelectron spectrometer IE-1 "Varian" at room temperature and pressure of 10^8 Pa with Al Kα (1486.6 eV) as photon source, at 20 eV electron analyzer pass energies. Binding energies were calculated using the C ls peak as a reference. Quantitative estimations of surface atomic ratios of Cu, Fe and S were made with integrated peak areas.

Electrochemical cells with a three-electrode system were used in CV, air bubble detachment force (DF), air bubble induction time (IT), air bubble evolution pressure (EP) and potentially controlled flotation experiments. Potentials were measured and reported versus a silver-silver chloride reference electrode, which had a potential of +0.22V against the standard hydrogen electrode. The potential of platinum mesh or chalcopyrite disc electrodes was controlled by a PI 50 potentiostat programmed with a sweep generator PR-8 (Izmeritel, Gomel, Byelorussia). A wire connected the working electrode to the external electrical circuit. The reference electrode was connected to the main compartment through a Lugging probe capillary. Current passed between the working electrode and a platinum counter electrode, which was housed in the compartment, separated from the main cell by a sintered glass disc. An X-Y recorder was used for recording cyclic voltammograms. To study xanthate sorption, spectral measurements were used [Chanturiya, Vigdengrauz, 1993].

For the evaluation of surface wettability under conditions of pH change and potentiostatic polarization the measurements of contact angle, IT, DF and EP were used. Experimental details of the measurements of an air bubble induction time, detachment force and contact angles were described earlier [Chanturiya, Vigdengrauz, 1993; Vigdergauz, Nedosekina, 1998; Vigdengrauz et al., 1996]. A torsion balance and a modified Sven-Nilsson device were used in DF and IT experiments. Induction time was defined as the minimum time necessary for the attachment of an air bubble to the chalcopyrite surface. Air bubbles for measurements were deposited on the holder from a small diameter flat-nosed needle positioned below the holder. The bubble formation and size were controlled using a combination of plug and needle. The movement of the bubble was observed through the wall of the cell, the latter being illuminated by an electric lamp. The DF measurements were made a few seconds after the gas front had ceased to advance across the mineral surface. A freshly polished surface was used for each new series. The standard deviations of the DF values with five repeated measurements were confirmed to be less than 0.2 dyne.

For the studies of the beginning of air bubbles evolution under decompression was used the set-up that is schematically illustrated in Fig.1.

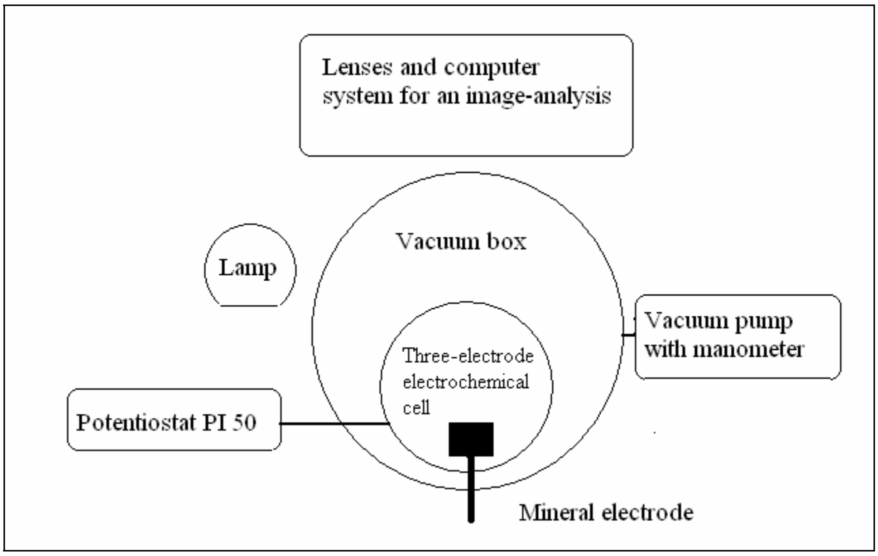

Fig. 2. Set-up for the EP-experiments

This set-up included vacuum box with vacuum pump and manometer, potentiostat, programmed with a sweep generator, electrochemical cell with a three-electrode system and photo lens with computer for an image analysis. Under potentiostatic control, the current passed between the mineral disc and a platinum counter electrode, which was housed in the compartment, separated from the main cell by a sintered glass disc. The wetting behavior can therefore be monitored over a range of anodic as well as cathodic polarization. An air bubbles evolution under decompression could be used for an evaluation of differences of surface properties of minerals. Device for the study of an electrochemical polarization influence on air bubbles evolution under decompression is a unique observatory that enables a direct view of the wetting process.

Flotation experiments were carried out in an electrochemical cell based on the Hallimond tube, Fig.3 [Chanturiya, Vigdergauz, 1993].

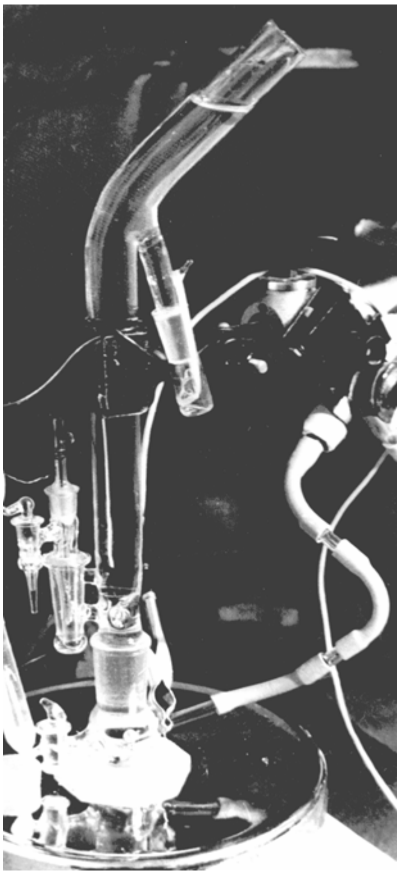

Fig. 3. The Hallimond tube cell for flotation with electrochemical treatment

The potential of the platinum mesh electrode was controlled with a potentiostat. The Hallimond tube cell design permits flotation without any frother. The usual procedure was to fill the cell with 7 ml of borate solution, load 250 mg of mineral, and condition the mineral suspension for 10 min at the desired potential prior to adding the collector. Floatability was determined by weighing the floated fraction. All flotation experiments were done with the new portions of chalcopyrite.

RESULTS AND DISCUSSION

REDOX STATE, SURFACE COMPOSITION AND REACTIVITY

X-ray photoelectron spectra of air-ground chalcopyrite show that the Fe(2p)3/2 spectra includes the major peak positioned at 711.7 eV associated with Fe^{3+} in a sulfide phase. The S(2p) spectra includes an intense peak arising at 162.33 eV that is usually attributable to sulfide species. One can conclude when comparing the Fe(2p) Cu(2p), and S(2p) spectra obtained with literature data [Putnis, McConnel, 1976] that the oxidation state of the iron is 3+, that of copper is 1+, and that of sulfur is 2-. Thus, $CuFeS_2$ is a double sulfide of Fe_2S_3 and Cu_2S. It must also be pointed out that the position of the S(2p) maximum (about 162 eV) in the spectrum implies that elemental sulfur and sulfate are not present on the surface, even in the case of $CuFeS_2$ not polished.

CV study of redox-transitions on the chalcopyrite surface under cathodic and anodic polarization leads to the conclusion that both cathionic and anionic sites participate in them simultaneously. It could be clearly demonstrated by a comparison of the CV-curves on the Fig. 4 [Radyushkina et al., 1986]. Rotation of the electrode leads to the removing of the maximums attributed to the reduction of copper ions and to the oxidation of hydrogen sulfide.

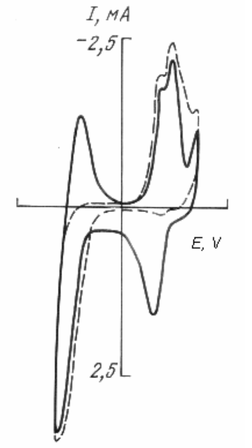

Fig. 4. Influence of rotation on the cyclic voltammograms of chalcopyrite in acid solution (pH 1.3) under potential sweep 25 mV/sec: 1 – solid curve without rotation; 2 – dotted curve under rotation 60 rev/sec

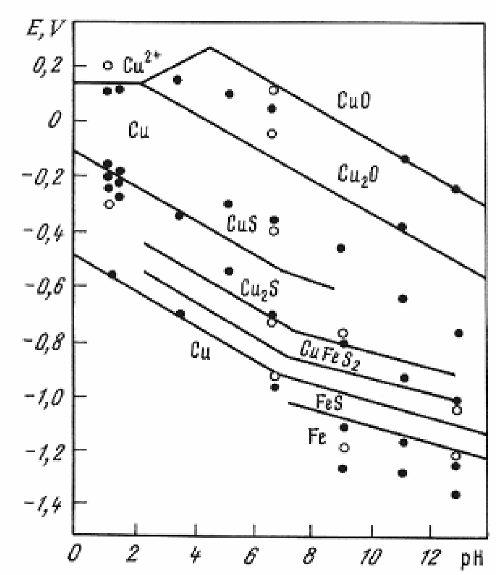

Fig. 5. Some lines of the Cu-Fe-S-H_2O diagram and potentials of the cathodic maxima of CV curves of chalcopyrite(•) and chalcocite (o)

Figure 5 shows the open-circuit potentials and the potentials of the cathodic maxima of $CuFeS_2$, and Cu_2S as functions of solution pH. The same figure shows some lines from the potential-solution pH diagram of the copper-iron-sulfur system [Chanturiya, Vigdergauz, 1993].

With increasing pH the hydroxides of the metals become ever more important in the surface redox reactions of chalcopyrite. Fig.6 shows a shift of the stationary potential of the chalcopyrite electrode during long time conservation under water.

Impulse electrochemical methods (CV and impedance measurements) reflect an existence of the cationic and anionic centers on mineral surfaces. Surface of sulfide minerals is electrochemically heterogeneous and there are could be simultaneously represented cathodic and anodic areas. It was clearly shown by Plaksin and Shafeev with the help of microradiographic technique [Plaksin, Shafeev, 1959]. Moreover these surfaces are chemically heterogeneous and wettability measurements could reflect this heterogeneity. On the fresh non-oxidized surface of chalcopyrite, there are sulfur and metall sites with non-compensated negative and positive charges successively. These charges could be compensated both by regulation of pH and electrochemical potential according to the follow simplified schemes [Vigdergauz, 2001]:

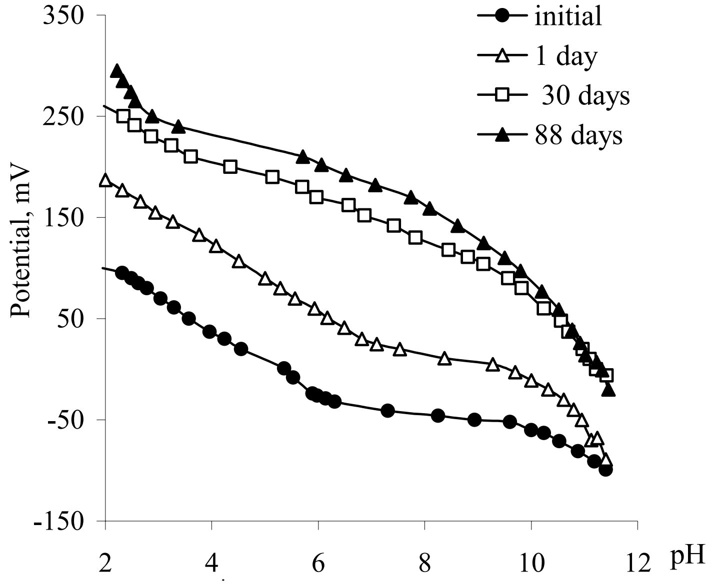

Fig. 6. Time dependence of the electrochemical potential – pH curves for chalcopyrite:
- initial; - 1 day; - 30 days; - 88 days

I. By regulation of pH:

$$-S^{\delta-} + H_2O = -SH + OH^- \quad (1)$$

$$-Me^{\delta+} + H_2O = -MeOH + H^+ \quad (2)$$

II. By regulation of an electrochemical potential:

$$-S^{\delta-} - \delta e = -S \quad (3)$$

$$-Me^{\delta+} + \delta e = -Me \quad (4)$$

EVALUATION OF WETTABILITY

Self-induced wettability was determined and also wettability induced by xanthates of different alkyl chain length. A combination of wettability measurements with electrochemical techniques has the advantage of controlling the surface oxidation state and the nature and amount of surface species present. Study of chalcopyrite wettability was carried out at various potentials of electrochemical polarization in buffer solutions with various pH.

Induction time measurements. Table 1 presents the dependence of induction time on the potential of electrochemical polarization in borate buffer solutions.

TABLE 1 The influence of the potential of chalkopyrite electrode and the length of xanthate carbon chain on the air bubble-mineral attachment and the induction time values[a]

	Potential of chalcopyrite electrode, V																			
	-0.7	-0.6	-0.5	-0.4	-0.3	-0.2	-0.1	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0	1.1	1.2
Xanthate	IT,sec																			
Ethyl	no[b]	no	no	no	no	no	no	2.5	0.7	0.8	0.8	0.8	0.8	2.0	2.0	no	no	no	no	no
Butyl	no	no	no	no	no	5.0	5.0	3.0	0.02	0.02	1.5	3.5	4.5	4.5	4.5	4.5	no	no	no	no
Amyl	no	no	no	no	no	no	4.0	2.0	2.0	2.0	2.5	1.5	2.0	2.0	1.5	0.4	1.5	1.5	2.5	1.5
Hexyl	no	5.0	3.5	3.5	2.5	2.0	1.5	1.5	1.5	0.1	0.45	1.5	1.5	0.6	0.02	0.02	0.004	0.004	0.004	0.5

[a] Xanthate concentration, - 50 mg /dm^3 or 312, 265, 248 and 231μM for ethyl, butyl, amyl, and hexyl xanthates, respectively.

[b] No attachment

The result of increasing the carbon chain of xanthate was as a rule a decreasing IT and an expanding of the area of adhesion potentials. These data are in accordance with the well known fact that in increase in the alkyl chain of xanthate collectors leads to more rapid flotation [Vigdergauz, 2001]. In the experiments with hexyl-xanthate, we observed a cathodic shift of adhesion area of about 500 mV in comparison with the amyl-xanthate experiment. Due to the narrow adhesion interval, ethyl and butyl xanthates as collectors have an advantage for the electrochemical regulation of selective flotation processes.

From the above results, it is clearly seen that a process of bubble attachment to a planar surface is a rather slow one. It was shown that an induction time in the experiments with flat surfaces is two orders of magnitude bigger in comparison with the values determined in the experiments with powders [Sutherland, Wark, 1955]. So IT results could only be used for the approximate comparison of surface wettability and preliminary evaluation of its influence on flotation response.

Air bubble detachment force and calculated contact angles. Detachment force/potential polarization curves are similar in shape for the absence of xanthate and for the presence of all the xanthates studied, Fig.7. The qualitatively similar character of the dependences of hydrophobicity functions with and without collector leads to the conclusion that xanthate ions block metal sites that give an input to the total hydrophobicity, but the basic influence is the self-induced hydrophobicity of chalcopyrite due to the influence of S-sites.

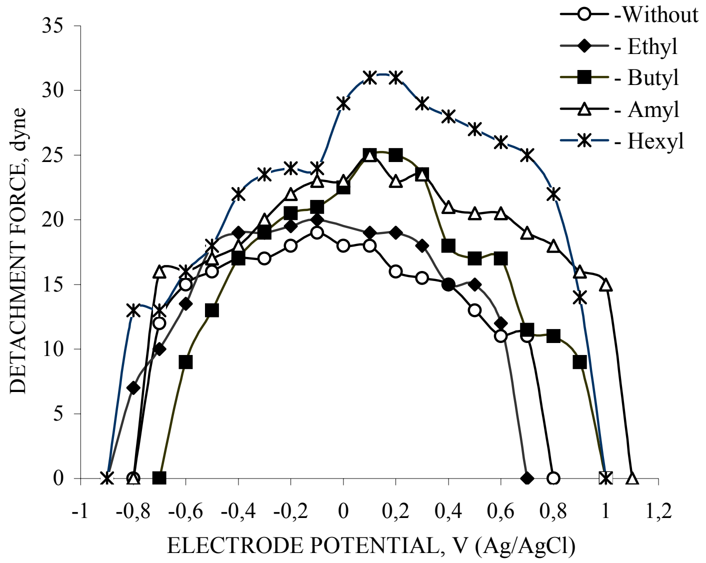

Fig. 7. DF dependences on the potential of chalcopyrite electrode

For the comparison of the observed DF results with the data on contact angles, a procedure was developed for the approximate evaluation of a contact angle from the measured force of detachment. DF data could be recalculated to give contact angles by Eq. 5 [Drzymala, Vigdergauz, 2001]:

$$F_d = 2\pi r_k \gamma_{lv} (1\text{-}\cos\theta) = 4\pi r_k \gamma_{lv} \sin^2(\theta/2) = 2\pi R \gamma_{lv} (1\text{-}\cos\theta) \sin\theta\ 1 \qquad (5)$$

Eq.5 is based on the process of detachment with the formation of new interfaces while the use of equations which describe the process of rupture of the bubble from the surface and account capillary forces only does not explain the experimental values of the detachment force. Example of contact angles calculation from the experimental DF is given earlier for pyrite [Vigdergauz, 2001].

In the presence of butyl, amyl and hexyl xanthates the main DF maximum is observed near a potential of 0.2 V. This potential corresponds to that of dixanthogen layer formation on the surface. The maximum value of the detachment force changes between 20 and 32 dyne with the increase in length of the alkyl chain of the collector. Minimum and maximum values were observed for ethyl and hexyl xanthates, respectively. Cathodic shift of DF maximums with an increasing of the length of apolar group of xanthate, Fig.7, correlates with the similar shift of IT minimums, Table 1. As it was mentioned above chalcopyrite is roughly a double sulfide of copper and iron $Cu_2S{\cdot}Fe_2S_3$ and its DF-potential curves are in first approximation a superposition of similar curves for individual sulfides of copper and iron.

Gas evolution under decompression. Micro bubbles are produced on the mineral surface under decompression and preferably they will be appear on the hydrophobic sites. Consequently, surface wettability could be estimated by a comparison of a pressure decrease that will be enough for micro bubbles production.

Fig. 8 illustrates bubbles evolution under decompression on the surfaces of pyrite and chalcopyrite.

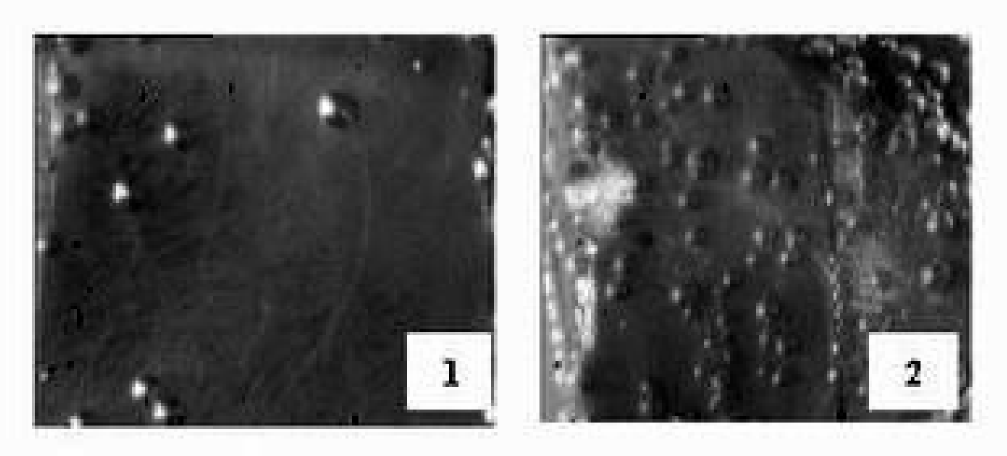

Fig. 8. Bubbles evolution under decompression (500 mm Hg) on the surfaces of pyrite (1) and chalcopyrite (2): pH 12.2 and butyl xanthate concentration 30 mg/l

Influence of an electrochemical polarization on a vacuum that leads to an emanation of bubbles on the chalcopyrite surface in weak alkaline buffer solution under butyl xanthate concentration 10 mg/l is demonstrated in Fig.9.

Area of the potentials of maxima hydrophobicity, Fig.9, corresponds with DF – potential results, Fig.7. Cathodic polarization leads to desorption of xanthate compounds from the surface of chalcopyrite electrode decreases DF and puts an end to bubble formation. Similar effects are observed under anodic polarization due the formation of hydrophilic layer of iron oxides.

Figure 2 [Vigdergauz, Panova, 2001] shows pH influence on the DF and EP values for chalcopyrite.

Comparison of Taggart's evaluation of the suction behind the blades of the impeller of an agitation-froth machine [Taggart, 1927] and EP data on chalcopyrite, Figs.7-10, shows that gas evolution will be observed in the range of 1300-2700 revolutions of impeller per minute for hydrophobic and hydrophilic surfaces successively.

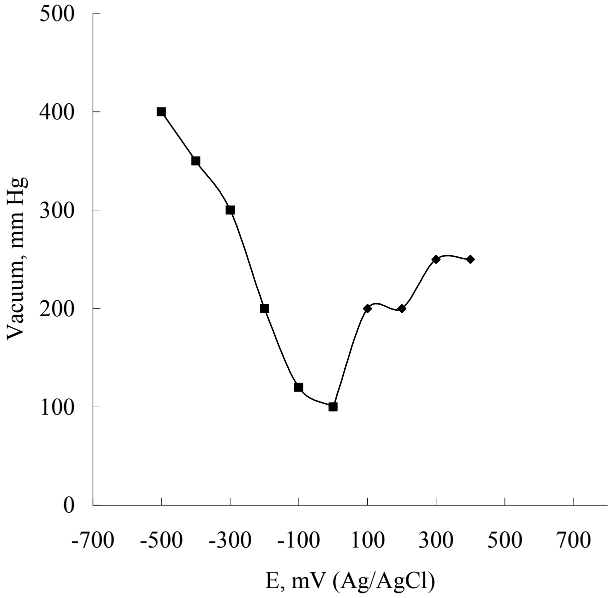

Fig. 9. Potential dependence on the decompression of the beginning of bubbles evolution on chalcopyrite: pH 9.2 and butyl xanthate concentration 10 mg/l

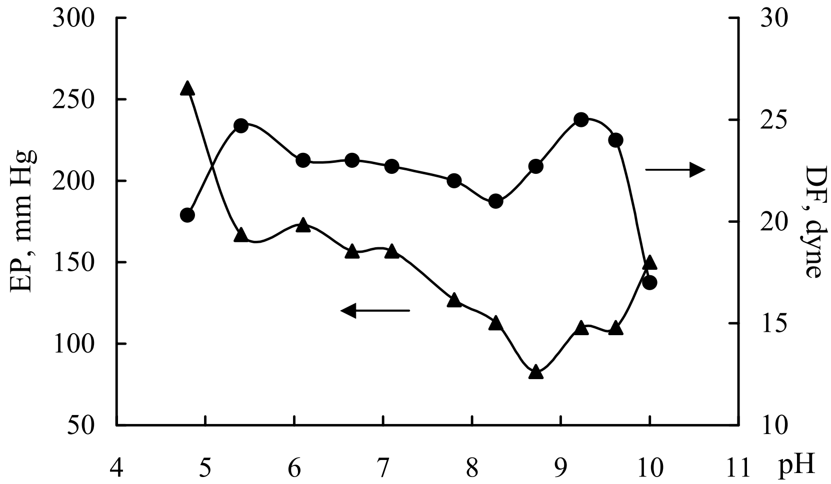

Fig. 10. DF and EP dependences on the pH for chalcopyrite

Floatability. Fig. 11 shows floatability-potential curves for chalcopyrite flotation without xanthate and in the presence of butyl xanthate [Chanturiya, Vigdergauz, 1993].

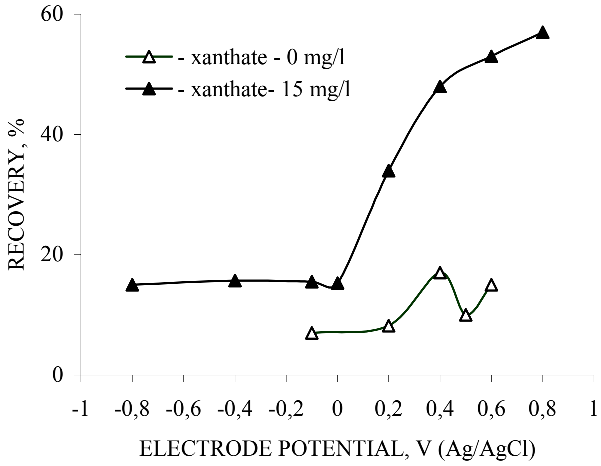

Fig. 11. Chalcopyrite floatation dependence on the potential of Pt mesh

Curves are similar in shape for the potentials of Pt mesh less than 0.4 V. Dixanthogen formation at potentials of +0.4 V and above leads to considerable increase of floatability in the experiments with xanthate.

SUMMARY

Presented results show that chalcopyrite surface is covered by cationic and anionic centres with charges that could be compensated by regulation of pH or an electrochemical potential value. Chalcopyrite surface hydrophobicity is a result of an input of hydrophilic and hydrophobic sites. Heterogeneity of the chalcopyrite surface determines the existence of pH and potential dependencies of hydrophobicity and flotation. In the case of xanthate induced hydrophobicity, a maximal hydrophobicity of chalcopyrite was observed on the stationary potential values at pH 5.5 and 9.2, and on the pH 9.2 at potentials of 0-0.2 V. A general agreement is observed between the pH of maximal hydrophobicity of bulk chalcopyrite determined by DF and EP experiments. There is also a general agreement between the potentials of maximal hydrophobicity of the bulk chalcopyrite electrode and the potentials of electrochemically-conditioned flotation of chalcopyrite. Under the conditions of froth flotation, it is necessary to account for the hydrophobicity of surface sites of major and minor influences to predict the observed phenomena. Resultant wettability reflects wettabilities of atomic or ionic sites and sorbed molecules and could be calculated through the energies of hydratation of these sites.

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Fizykochemiczne właściwości chalkopirytu, zwłaszcza zwilżalność powierzchni zostały przebadane z wykorzystaniem takich technik badawczych jak: woltamperometria cykliczna, potencjometria, spektroskopia fotoelektryczna i inne. Przeprowadzono obszerną dyskusję otrzymanych wyników, które obejmowały zależność stopnia hydrofobowości chalkopirytu od potencjału i pH, a także siłę odrywu, kąt zwilżania, zmiany ciśnienia gazu i flotowalność. Zostało wykazane, że krzywe obrazujące zależność siły odrywu od potencjału polaryzacji wykazują podobny kształt dla przypadków braku ksantogenianu jak dla przypadku obecności ksantogenianu w układzie. Elektrochemiczna polaryzacja prowadzi do powstania hydrofilnych związków i wzrostu zwilżalności powierzchni badanego minerału. Utworzenie hydrofilnej warstwy wodorotlenku żelaza jest przyczyną obniżenia stopnia hydrofobowości i flotowalności w warunkach utleniających. Desorpcja ksantogenianu prowadzi do hydrofilizacji powierzchni i depresji we flotacji prowadzonej w warunkach katodowych.