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FLOTATION OF MINERALS BY CONTROLLING THE SURFACE
TENSION OF THE MEDIUM: MOLYBDENUM DISULFIDE

by

Jules Kaoma

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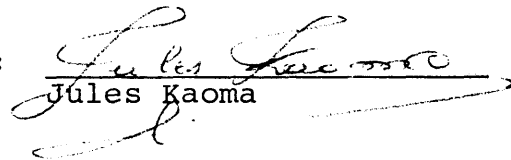
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A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science (Metallurgy).

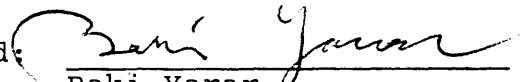
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ABSTRACT

The flotability of molybdenite without added flotation reagents was studied using synthetic and natural molybdenites in the composition range $\text{Mo}_{1+x}\text{S}_2$, $\text{Mo}_{1-x}\text{S}_2$, where $x = 0$ to 0.14 .

The critical surface tension of wetting of the solids, was monitored as a measure of wettability and flotability. Non-stoichiometry, particle size, shape, and oxygen concentration of the medium were found to affect the value of the critical surface tension of wetting and the flotability. The critical surface tension of molybdenite was found to vary from 26 to 31 dynes/cm.

The mechanochemical phenomena in a molybdenite-quartz and molybdenite-chalcopyrite grinding systems were found to determine the degree of separability of molybdenite, quartz, and chalcopyrite from their mixtures. The critical surface tension of the MoS_2 - SiO_2 and MoS_2 - CuFeS_2 mixtures obtained by prolonged mutual grinding, approaches that of molybdenite.

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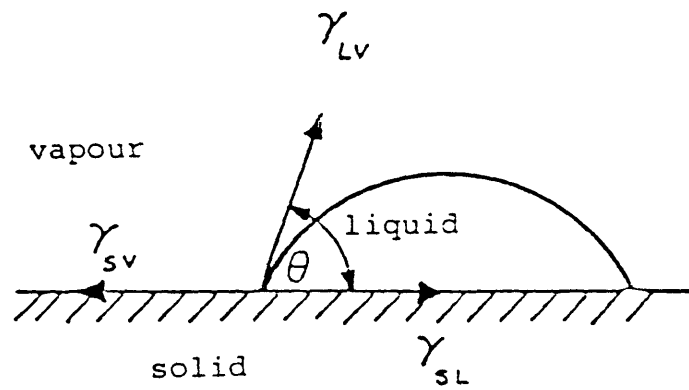
I. INTRODUCTION

The physical and chemical properties of surfaces play a major role in virtually all aspects of mineral exploitation. The wetting/non-wetting behavior of solid surfaces forms the basis of the froth flotation process. This involves the collection of hydrophobic solids in a froth layer upon introduction of gas bubbles in a mineral slurry.

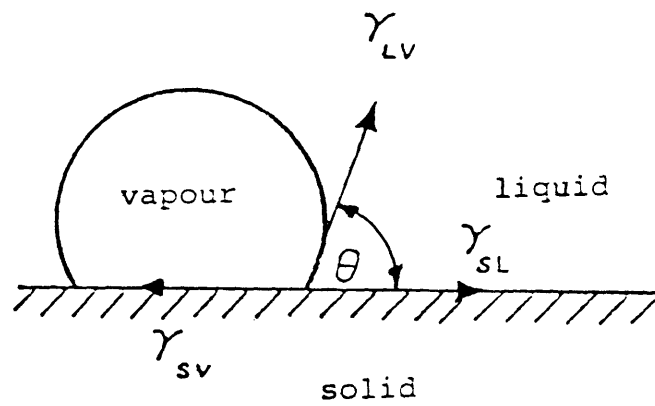
Hydrophobicity is either induced by selective adsorption of reagents at the solid/liquid interface or is inherent in some materials, such as waxes, tars, sulfur, graphite, talc, or molybdenum disulfide. Their behavior is attributed to their chemical composition and crystal structure¹. A hydrophobic solid can be characterized by a finite contact angle (measured through the aqueous phase), or by the strength of attachment of solid particles to gas bubbles.

1.1 EQUILIBRIUM CONTACT ANGLE AND FLOTABILITY

Figure 1 illustrates the classical three phase line of contact between liquid (l), vapor (v), and an undeformable solid surface (s). Equilibrium is assumed so that all phases are mutually saturated and the equilibrium contact angle θ ,



(a) LIQUID DROP



(b) AIR BUBBLE

Figure 1 - Equilibrium contact angle.

(measured in the liquid phase) is established. The relationship between the three interfacial tensions or energies, γ_{sv} , γ_{sl} , and γ_{lv} is given by the Young-Dupre equation.

$$\gamma_{lv} \cos \theta = \gamma_{sv} - \gamma_{sl} \quad (1)$$

where γ_{sv} , γ_{sl} , and γ_{lv} , are interfacial tensions between solid/vapor, solid/liquid, and liquid/vapor, respectively.

From Equation (1), two important conditions for flotability can be deduced:

i) A three-phase line of contact is established if:

$$\gamma_{sv} - \gamma_{sl} > \gamma_{lv} \quad (1a)$$

ii) No vapor/solid contact is established if:

$$\gamma_{sv} - \gamma_{sl} < \gamma_{lv} \quad (1b)$$

These represent flutable and non-flutable conditions, respectively.

Where the adsorption of surfactants is essential for the induction of hydrophobic properties, a relationship between the Young-Dupre's equation and the Gibbs's adsorption equation, can be derived to show that an excess quantity of surfactant does exist at the solid/liquid interface².

As the contact angle is measured into the water phase, establishment of a finite θ , and the larger the better, is

an apparent prerequisite for flotation. Periodic attempts have been made to correlate contact angle with flotation recovery^{3,4,5}, which is one of the oldest trends in mineral processing. However, Equation (1) introduces the notion that a high γ_{sl} does not favor a large θ . A high θ results from a solid having a high surface energy, γ_s .

1.2 OTHER WETTING TERMS RELATED TO FLOTABILITY

There are numerous equations and expressions to describe wetting phenomena, although the Young-Dupre equation has been considered to be quite adequate. Inasmuch as some of the other terms have been used in flotation research, they are described briefly below.

THE INITIAL SPREADING COEFFICIENT of Harkins⁶ applied to an aqueous system, for Figure 1, would be defined as:

$$S_{sl} = \gamma_{sv} - (\gamma_{sl} + \gamma_{lv}) \quad (2)$$

If $S_{sl} > 0$, the water spreads or advances.

If $S_{sl} < 0$, the liquid recedes. The latter condition corresponds to flotability. Observations on the relative magnitude of γ_{sv} , γ_{lv} , and γ_{sl} to achieve a flotabile condition clearly are the same as in inequality (1a).

The γ_{sv} and γ_{sl} components of Equation (1), cannot be directly measured; only their difference is measured given

by $\gamma_{1c} \cos \theta$. This quantity, therefore, assumes some significance. It is referred to as *the adhesion tension* or *the wetting energy* and for flotation, the adhesion tension should be less than γ_{1v} and has been experimentally verified^{7,8}.

SURFACE PRESSURE, Π_s , is the name given to the difference between the surface tension of the clean surface and surface tension resulting from the adsorption of surface tension lowering solute. Surface pressure of surfactant solutions has been used for correlating with flotation behavior^{9,10}.

THE WORK OF ADHESION between two phases, 1 and 2, is given by:

$$W_{I2} = \gamma_I + \gamma_2 - \gamma_{I2} \quad (3)$$

An analogous expression is THE WORK OF COHESION,

$$W_{II} = 2\gamma_1 \quad (4)$$

and corresponds to the work required to pull apart a unit area of single phase. If $W_{11} > W_{12}$, adhesion of the two phases is not favored. With 1 as the liquid and 2 as the solid, then the work of cohesion of water is greater than the work of solid/liquid adhesion and dewetting is favored¹¹. This corresponds to a floatable condition. Expressions can

be derived for vapor/solid and water/solid work of adhesion, viz.:

$$W_{sv} = \gamma_{lv} (1 - \cos \theta)$$

and

$$W_{sl} = \gamma_{lv} (1 + \cos \theta)$$

Correlation between flotation and increasing W_{sv} ^{12,13} and decreasing W_{sl} ¹⁴ can also be sought.

1.3 THE GOOD-GIRIFALCO-FOWKES-YOUNG EQUATION

The above title is quoted from Dann¹⁵ who appears to be the first to use it as recognition of the contribution of each.

Based on molecular interactions at interfaces in combination with the Young-Dupre equation, Good and Girifalco¹⁶ derived the following for the solid-liquid-gas system:

$$\cos \theta = -1 + 2 \phi \cdot \gamma_s^{0.5} / \gamma_{lv} - \Pi_s / \gamma_{lv} \quad (5)$$

where ϕ = the surface property of the particular solid/liquid combination

γ_s = the solid surface energy in vacuum

$\Pi_s = \gamma_s - \gamma_{sv}$, allows for surface energy lowering owing to adsorption of gas and if no specific solid-liquid interaction is assumed.

For low surface energy solids, it is usual to assume that $\Pi_s = 0$ and hence to derive:

$$\cos \theta = -1 + 2 \phi \cdot \gamma_s^{0.5} / \gamma_{lv} \quad (6)$$

Assuming ϕ is constant for a particular series of liquids on a single solid, $\cos \theta$ is predicted to be linear with $1/\gamma_{lv}$ with an intercept of -1 . Such plots have been demonstrated using the data of Zisman and co-workers for homologous liquid against plastics which usually exhibit natural hydrophobicity¹⁷.

Fowkes¹⁷ modified this theory, and for solid-liquid combinations, interacting by London-Van der Waals dispersion forces, only derived:

$$\cos \theta = -1 + 2 (\gamma_s^d \cdot \gamma_{lv}^d)^{0.5} / \gamma_{lv} - \gamma_s / \gamma_{lv} \quad (7)$$

where the superscript d refers to the dispersion force contribution to the respective surface energies. Again, assuming $\Pi_s = 0$, which would be the case for low surface energy solids, then a plot of $\cos \theta$ versus $(\gamma_{lv}^d)^{0.5} / \gamma_{lv}$, should be linear with an intercept of -1 ¹⁷.

All these models can be used to predict the contact angle values for a given system and a means of estimating γ_s ¹⁸ and γ_s^d ¹⁷.

Bargeman and van Voorst Vader¹⁹ rearranged Equation (7), to give for $\Pi_3 = 0$:

$$\gamma_{1V} \cos \theta = \gamma_{1V} + 2 (\gamma_S^d \cdot \gamma_{1V}^d)^{0.5} \quad (8)$$

or

$$\gamma_{1V} (\cos \theta + 1) = 2 (\gamma_S^d \cdot \gamma_{1V}^d)^{0.5} = W_{sl} \quad (9)$$

They demonstrated the linearity of the plot of $\gamma_{1V} \cos \theta$ versus γ_{1V}^d ¹⁹.

In general terms, Equation (8) can be written as:

$$\gamma_{1V} \cos \theta = -\gamma_{1V} + C \quad (10)$$

where C is a constant. Where γ_S^d values are available, the calculated C values agree with those measured¹⁹.

The linearity found in this context, augurs well for contact angle values. However, several ramifications arose from the observations as Bargeman and van Voorst Vader showed the slope of -1 means that the difference in surfactant adsorption density, between the solid/liquid and solid/vapor interfaces, must equal the adsorption density at the liquid/vapor interface.

The above noted models appear to be interesting in the sense that they can be used in controlling surface phenomena to promote flotability.

1.4 CRITICAL SURFACE TENSION AND FLOTABILITY

Zisman and co-workers^{20,21} introduced the concept of *the critical surface tension of wetting of solid* while studying the contact angles of homologous series of organic liquids on low surface energy solids, $\cos \theta$ was found usually to be a linear function of γ_{1V} , as shown in Figure 2. Extrapolating $\cos \theta$ to 1, i.e., $\theta = 0$, gives γ_{1V} at which the liquid just wets the solid. This value of γ_{1V} is referred to as *the critical surface tension of wetting* of the solid and is expressed as follows:

$$\cos \theta = 1 + b (\gamma_c - \gamma_{1V}) \quad (11)$$

where b is the slope of the line exemplified in Figure 2.

The flotability criteria are:

- i)* $\gamma_{1V} > \gamma_c$, non-wetting or partial wetting of the solid. There is an establishment of contact angle, i.e., the solid is flutable.
- ii)* $\gamma_{1V} < \gamma_c$, wetting of the solid surface, i.e., spreading of the liquid on the solid leading to non-flotability.

Zisman considered the critical surface tension of wetting as an empirical parameter to predict the

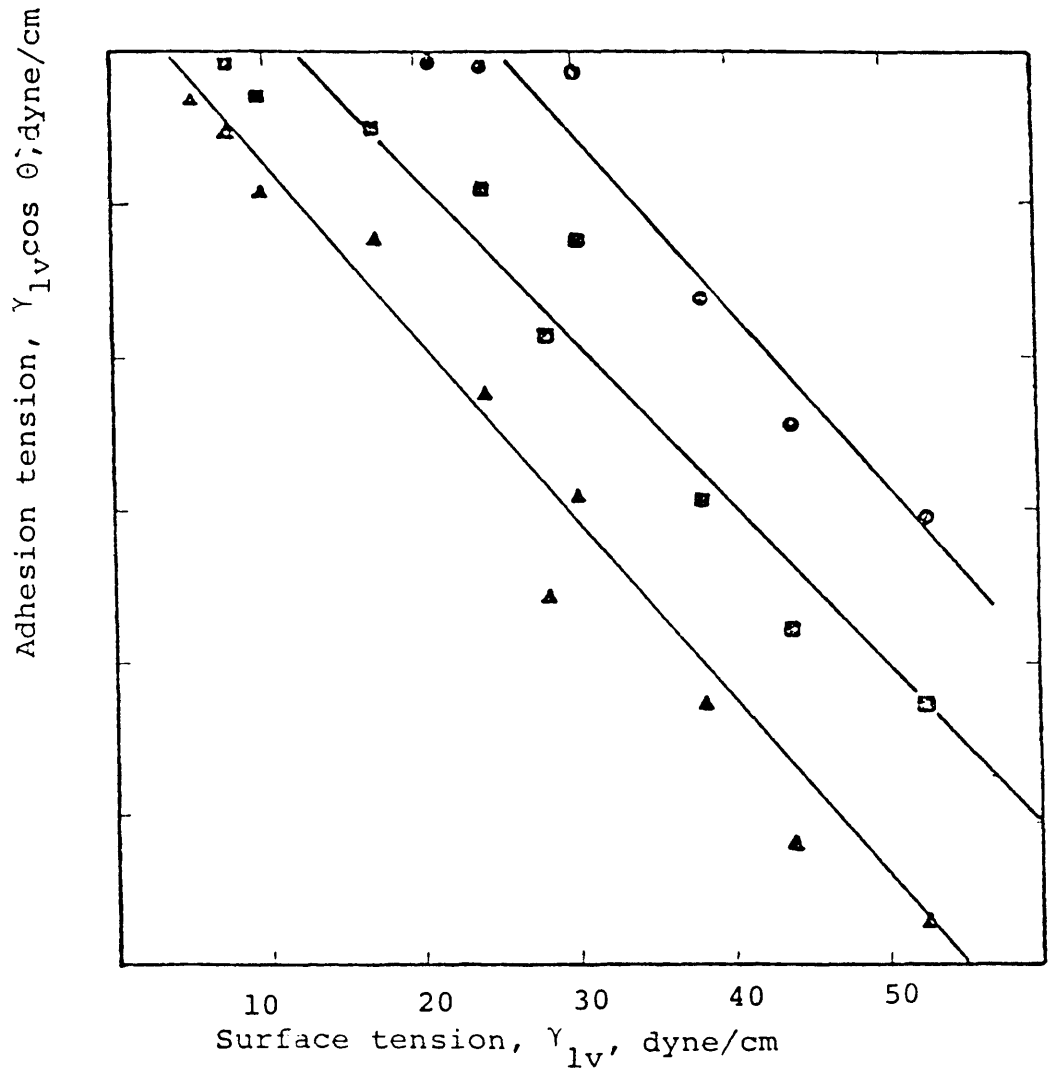


Figure 2 - Wettability diagram for PbS-Xanthate system, (after Aplan and Parehk²⁵).

non-wettability of a solid, i.e., hydrophobicity. Attempts have been made to equate γ_c directly with γ_s (or γ_s^d)¹⁸.

Dann¹⁶ has shown that the Zisman equation is a limiting case of Equation (9) with $\Pi_s = 0$ as $\cos \theta$ approaches unity. Adamson²² stresses that γ_c is an empirical parameter, noting that to an extent, γ_c depends on the liquid used as well as the solid. Any interaction between solid and liquid must be expected to alter γ_c ²³.

The critical surface tension of the wetting model has been shown to be handy in characterizing the wetting behavior of hydrophobic substrates. Parekh and Aplan^{24,25} used the critical surface tension of wetting concept in characterizing the degree of hydrophobicity of minerals.

The control of the critical surface tension of wetting as a means of separating solids of different degrees of hydrophobicity has been exploited in a limited number of cases. For example, it should be possible to selectively separate two inherently hydrophobic (low energy) solids by flotation if they have significantly different critical surface tensions (estimated with solutions of the same surfactant)^{26,27}. The necessary conditions are illustrated in Figures 3 and 4, the *wettability* and *flotability* diagrams.

Lines 1 and 2 in Figure 3, represent the linear relationship between $\gamma_{lv} \cos \theta$ and γ_{lv} for two hydrophobic solids

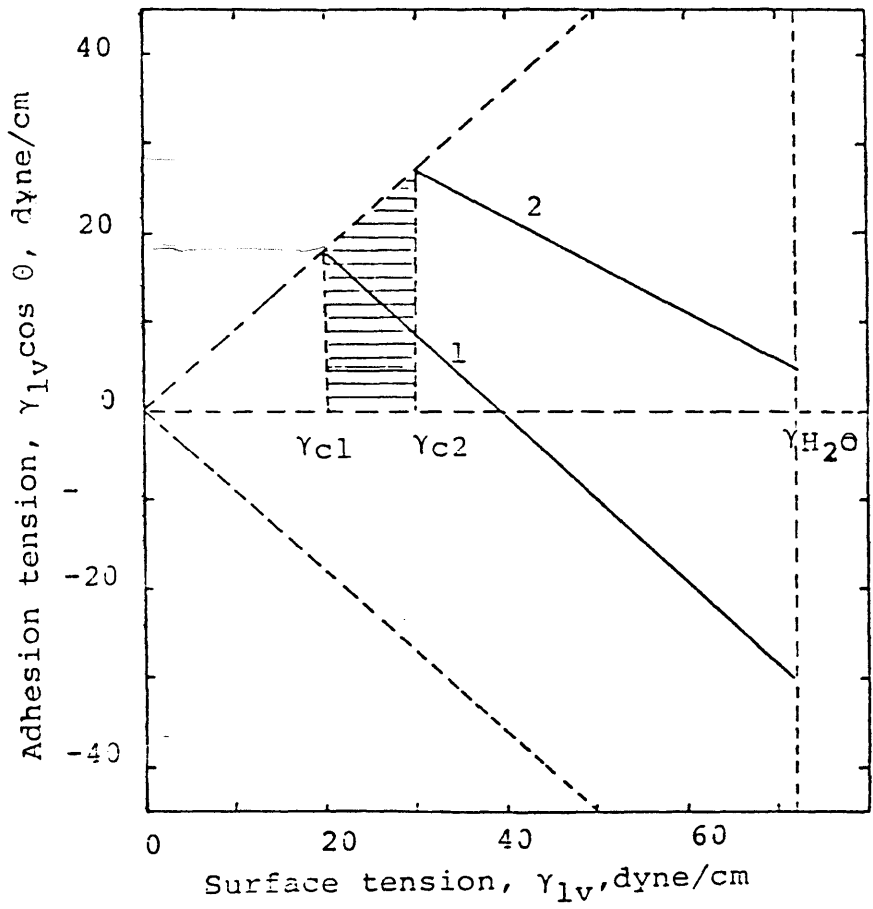


Figure 3 - Region of separability of two hydrophobic materials on the wettability diagram.

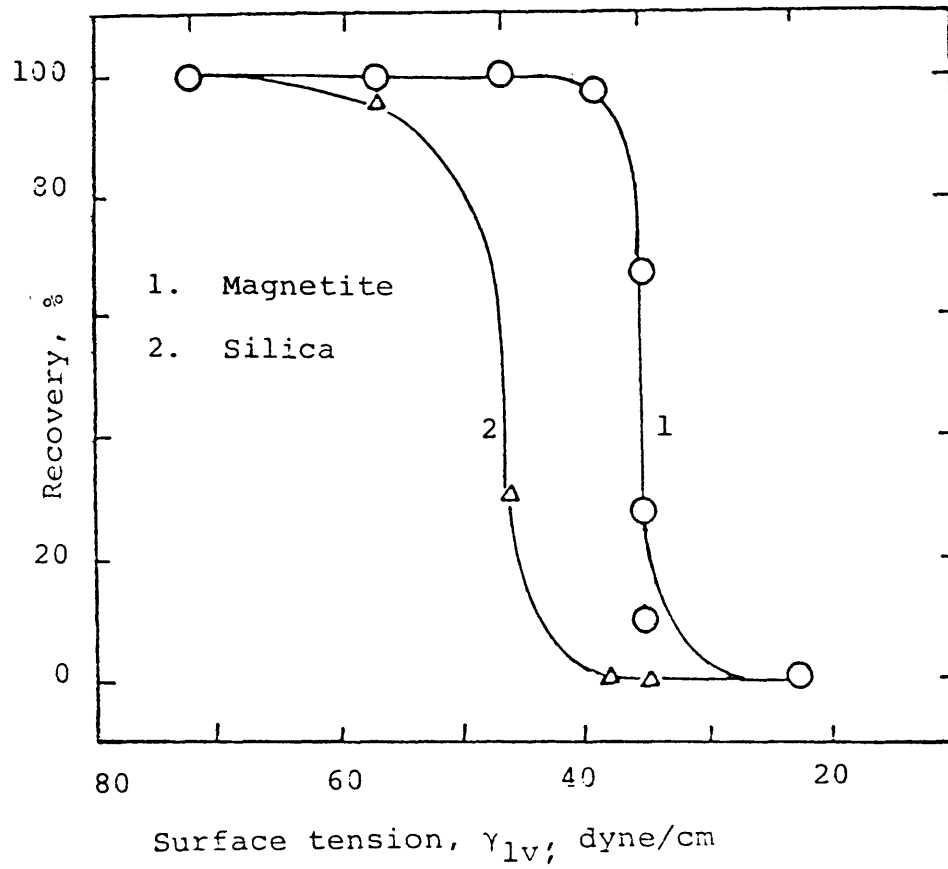


Figure 4 - Separation of two hydrophobic materials on the flotability diagram (after Finch and Smith¹⁰).

in the same liquid. Solid 1 becomes completely wetted in solutions with surface tension below its critical surface tension γ_{c1} . Solid 2 becomes completely wetted below γ_{c2} . The shaded area represents the region where Solid 1 is only partially wetted and selective separation should be possible. (A solution surface tension close to should be used to give maximum contact angle and particle/bubble adhesion.) The selective separation of two hydrophobic materials by controlling the surface tension is well illustrated in Figure 4.

1.5 NON-STOICHIOMETRY

So far, most mineral technology researchers have been concerned with the type of interaction taking place at the various interfaces of the flotation system. The nature of the surface state plays a major role in mineral beneficiation. It is known that the lattice and electron fine structure of minerals can influence on the collector adsorption and the flotability.

Changes made in any of the phases concerned, i.e., gas, liquid, or solid phase, are reflected through the changes occurring in two of the surface energy terms. Thus, a change in the liquid composition would be noted in γ_{s1} and γ_{lv} , and it is this phase, the liquid, which is not frequently altered in flotation studies.

Changes in the solid phase must be reflected through the changes occurring in the γ_{sv} and γ_{lv} terms. This means that changes in the surface properties of the solid phase will alter the interfacial tensions, which, in turn will be reflected in flotation results.

In many compounds, the proportion of elements differ appreciably, though rarely by more than 1%, from those demanded by simple chemical formulae. This deviation from stoichiometry, has important consequences in flotation, photochemistry, catalysis, sintering, and other related processes^{28,29}. In principle, four different types of non-stoichiometry are possible, since either the metallic or the electronegative component may be present in excess, and either kind of excess may be associated with interstitials or with vacancies, as illustrated in Figure 5.

Type I -- excess metal with anion vacancies.

Type II -- excess metal with interstitial cations.

Type III -- excess electronegative constituent with
cation vacancies.

Type IV -- excess electronegative constituent with
interstitial anions.

Electroneutrality is secured in all types by the presence in the vicinity of anions (or cations) vacancy

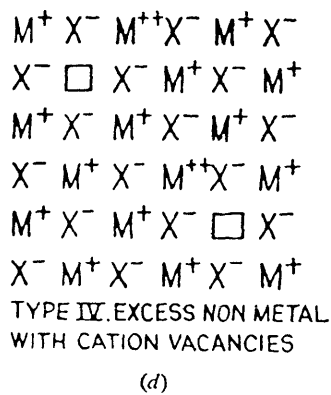
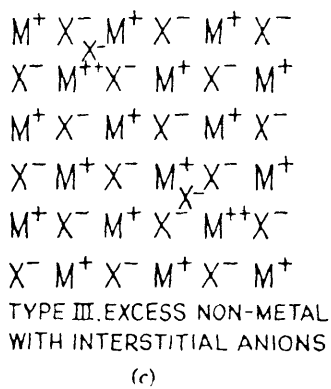
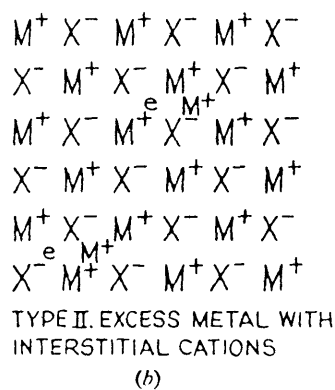
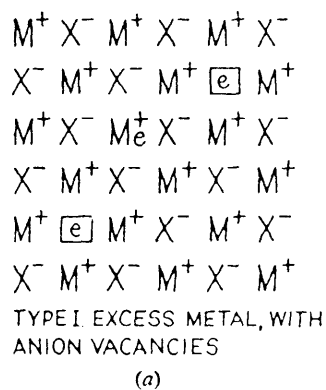


Figure 5. Different types of non-stoichiometry

(Type I and III) or interstitial cation (anion) (Type II and IV) of electrons or holes, respectively.

Investigators have studied the influence of point defects on the flotation of minerals. Simkovich³⁰, for example, found out that Bi_2S_3 -doped galena floats better.

Besides the change in the surface properties of solids, the nature of the gaseous phase may affect the flotation of minerals. In the following section, discussion will be concentrated on the role of oxygen in flotation.

1.6 OXYGEN AND FLOTABILITY

It is well known in mineral concentration practice using froth flotation that the presence of oxygen in the pulp affects the adsorption of collector on the mineral surface³¹⁻³⁵. It reacts either with the collector or the mineral or both at the surface, thus modifying the flotation process.

The change in the nature of the electric charge carriers is caused not only by oxygen but also by other species having oxidation-reduction properties. These substances when arriving at the electron transition zone may change the position of the electrochemical potential levels on the surface layers of the mineral.

The difference in electrochemical potential of the surface and the liquid phase components determines the intensity and the direction of electron transition band, which, in turn, determines the condition for adsorption of reagents.

Figure 6 illustrates the action of oxygen on n-type sulfide mineral surface. The adsorption of free electrons from the conduction band creates conditions for an increase in energy to the top of the energy zone. At some point, a, the Fermi level of the sulfide crosses the Fermi level of the intrinsic conductivity of the mineral. Furthermore, at this point, the inversion of the conductivity of the surface layers of the mineral from n-type to p-type occurs.

Scientists in the Soviet Union, have shown that an increase in oxygen concentration on the surface of the sulfide minerals reduces the contact time between the mineral particles and air bubbles. They derived a relationship between the change in concentration of the electric charge carrier and the oxygen concentration³⁴ in the form:

for the change in concentration of electrons:

$$D_{ne} = -A \cdot C_{O_2}^{0.25} \quad (12)$$

and the change in the concentration of free holes is:

$$D_{np} = A \cdot C_{O_2}^{-0.25} \quad (13)$$

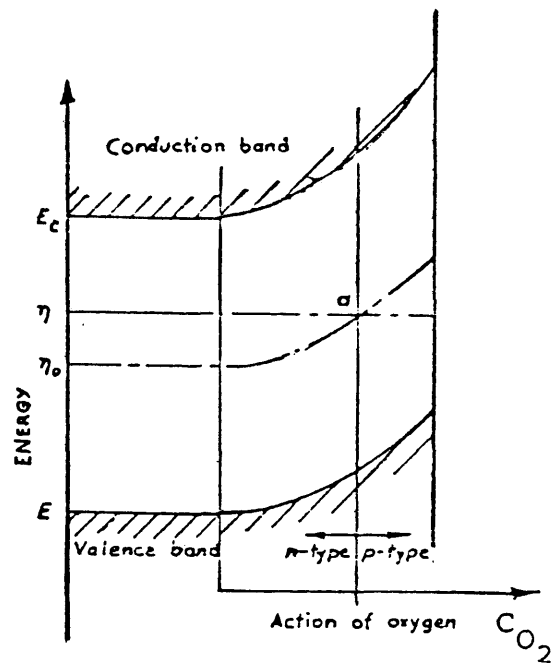


Figure 6 - Action of oxygen on n-type sulfide mineral surface (after Plaksin and Shafeev³¹).

where $D_{n,e,p}$ = the electric charge carrier concentration

C_{O_2} = the concentration of oxygen in the liquid
phase of the pulp (or time of action)

A = a constant

From the above equations, it can be seen that oxygen plays a role in flotation. Besides oxygen, other effects, such as mechanochemical effects, may alter the crystal structure as well as the space charge-layer at the mineral surface.

1.7 THE MECHANOCHEMICAL PHENOMENA

Mechanical treatment of organic and inorganic solids has become extremely important in many processes in chemical technology, material science, ore dressing, and various other fields. For instance, the application of comminution processes, such as crushing, grinding, pulverizing, thermal shock, ultrasonic grinding, sawing, machining, cutting, polishing, hot pressing, drilling, etc., to break materials, such as minerals, rocks, and metals, is common.

Mechanochemistry includes the following:

- Polymorphic transformations and alterations in physical properties of the bulk phase.
- Recrystallization, decrystallization, and amorphization.

- Mechanochemical solid state reactions either at the surface or in the bulk.

For instance, comminution is a highly active mechanical process which consists of grinding through impact, compression, and attrition. The strain energy, shear energy, thermal energy, and kinetic energy transform the solids involved from one phase or compound into another through polymorphic or solid state reactions³⁶⁻⁴¹.

When the surface of two solid bodies are brought sufficiently close together, their fields of forces overlap and thus cause adhesion wherever the two solids are nearly or completely in contact. Adhesion of solids manifest itself indirectly by such effects as friction if the two solids are made to slide relative to one another, in the seizure of the two solids, in the aggregation of powders and in sintering, resulting in point defect formation in the mineral.

It is recognized in mineral technology, that during the process of comminution, some radical changes in the crystal structure of the mineral occur. The extent of deformation of the solid depends not only on its dimensions, but also on its elasticity, as measured, for example by the Young's modulus. The smaller the value of this modulus the softer the material and the greater will be the deformation

produced by a given deforming force (determined by the surface energy and the geometry around the area of contact) in a solid of given dimensions. For this reason, adhesion is promoted if one of the solids is soft, or better still, if both are soft.

The application of force can bring about a change in the chemistry of the substance. Fine grinding breaks the particle mechanically, but the desintegration is opposed by the tendency of the fine particles to adhere to one another under the joint action of surface forces and mechanical pressure exerted in the mill. It has been found that fine grinding increases the surface activity of a solid by distorting lattice structures. For instance, quartz is insoluble in water, but upon grinding it became soluble. Yarar and Kitchener⁴² found out that a mixture of silica and calcite powders can be easily separated by selective flocculation and upon being ground together, they became totally inseparable which provides an example of mechanochemical phenomena mentioned.

Some mechanochemical effects in molybdenite containing systems are reported in this study.

II. EXPERIMENTAL

The experimental work includes the determination of the critical surface tension of wetting of molybdenite and the assessment of the effects of non-stoichiometry, the oxygen content of the pulp, particle size, and the mechanochemical phenomena on molybdenite flotability.

Molybdenite was chosen because of its natural hydrophobicity coupled with the problems caused for its separation from other minerals.

The flotation technique was used as an alternative method for the determination of γ_c for powders, since the Zisman technique requires the measurement of the contact angle θ , which in turn, is not easily applied to finely powdered material.

In this study, methanol solutions of various concentrations were used for flotation experiments. Methanol was chosen because it has been reported in the literature that it does not adsorb on mineral surfaces²⁷, due to the strong hydrogen bonding between CH_3OH and H_2O in solution.

2.1 REAGENTS

All the reagents used in the synthesis of molybdenite were of analytical grade (Fischer Scientific Company). These were molybdenum trioxide, sulfur, sodium carbonate, ammonium molybdate, potassium thiocyanate, aqueous ammonia, and hydrochloric acid.

METHANOL

The solvent was a synthetic purified Fischer Scientific product. Solutions of different concentrations were prepared and their surface tensions determined by the drop-weight method⁴³. The results are reported in Appendix 1. The pH of the water-methanol mixture was 7.5.

WATER

The water used throughout the experiments was double distilled in an all glass apparatus. The electrical conductivity of water was 10 umhos.

2.2 MATERIALS

MOLYBDENITE SAMPLES

Two samples of different stoichiometry were synthesized as per Bell and Herfert procedure⁴⁴. Natural molybdenite on the other hand was hand-sorted from a sample of AMAX/Climax operations ore. The molybdenum content of the three samples

was determined by atomic absorption spectrophotometry using the Perkin-Elmer A, a spectrophotometer model 107. Sulfur was determined on the LECO sulfur determinator model IR 58. The chemical results are compiled in Appendix 2.

QUARTZ

This was a natural hand-sorted mineral from Socorro, New Mexico. The sample was ground and screened before being leached in hydrochloric acid to remove impurities and washed with double distilled water.

CHALCOPYRITE

The Butte, Montana sample was hand-sorted, ground to 100% passing 147 μ m, and used in experiments.

2.3 EXPERIMENTAL PROCEDURES

2.3.1 Flotation Cell

A 150cc Partridge-Smith cell⁴⁵ was used throughout the experiments. The set-up of the cell is shown in Figure 7.

2.3.2 Flotation Test Procedure

One gram sample at a time was used in all experiments. Solutions of varying surface tensions were used as flotation reagents. The flow of the gas in the cell was controlled, such as to avoid overbubbling. The slurry was conditioned for 1 minute, then floated for 3 minutes (the optimum

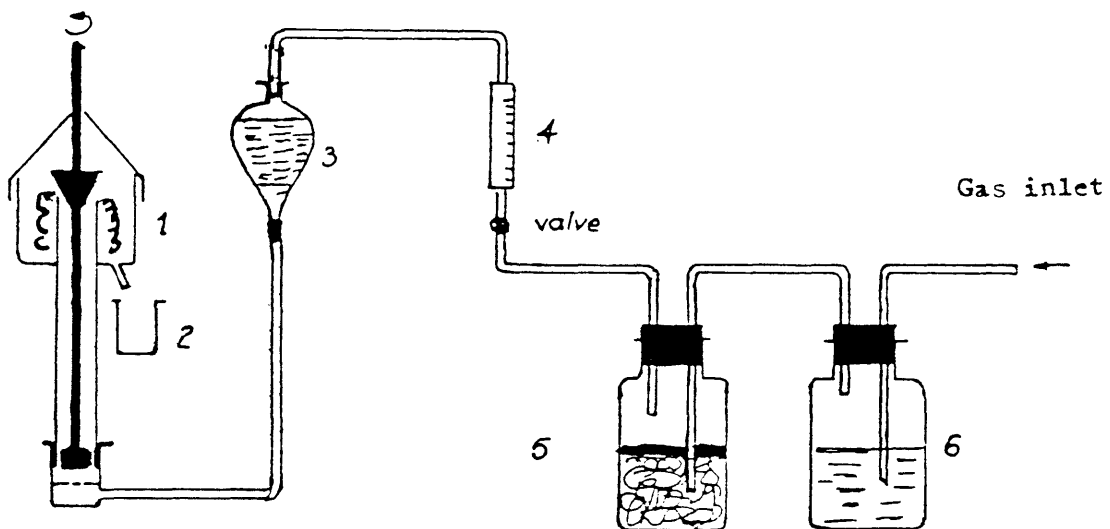


Figure 7 - Schematic of the experimental flotation set-up.

- | | |
|-----------------------|--------------------|
| 1. Flotation Cell | 4. Flow Meter |
| 2. Collection Beaker | 5. Desiccation |
| 3. Solution Reservoir | 6. Washing Bottles |

flotation time is shown in Appendix 3. The floated material was collected in a beaker, dried in an oven at $75 \pm 5^\circ\text{C}$ to constant weight. The recovery was computed on a weight difference basis.

To test the role of gases on the flotability of molybdenite, nitrogen, oxygen, and air were used. Before being introduced in the flotation cell, these were freed of possible contaminations by passing them through a train of washing bottles containing 50% sodium hydroxide solution followed by distilled water. These were then dried by passing through a desiccator containing magnesium perchlorate as a desiccant.

THE EFFECT OF PARTICLE SIZE

The influence of particle sizes was investigated as follows: the synthetic molybdenites were screened and the -38, 38/48, 48/74 μm size fractions were separated. These were subsequently used in flotation experiments.

MECHANOCHEMICAL EFFECTS

The mechanochemical effects were studied as follows: 100% -74 μm natural molybdenite and 100% 100-150 μm silica were mixed on 1:1 ratio basis. This mixture was ground for a period ranging from 30 seconds to 10 minutes, then these products were floated as above with air. The same was repeated in case of chalcopyrite.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The data of the flotation tests were plotted as % *recovery* versus surface tension of the solution γ_{lv} . These are shown in Figures 8 to 21.

The flotation curves for hydrophobic particles approximate the shape of the curve given in Figure 8 and consist of four regions:

Region I is the zero recovery range.

Region II lies between γ_c and γ_{lv} . In this range, the recovery varies with the change in solution surface tension, γ_{lv} . This is the linear part of the curve.

Region III is the maximum recovery range.

Region IV shows the decrease in recovery in water.

The critical surface tension was determined from the extrapolation of the linear part of the curve to 0% recovery. The critical surface tensions of wetting are 26, 28, and 31 dynes/cm for natural molybdenite, sulfur-excess, and molybdenum-excess molybdenite, respectively, as found from Figures 9, 10, and 11.

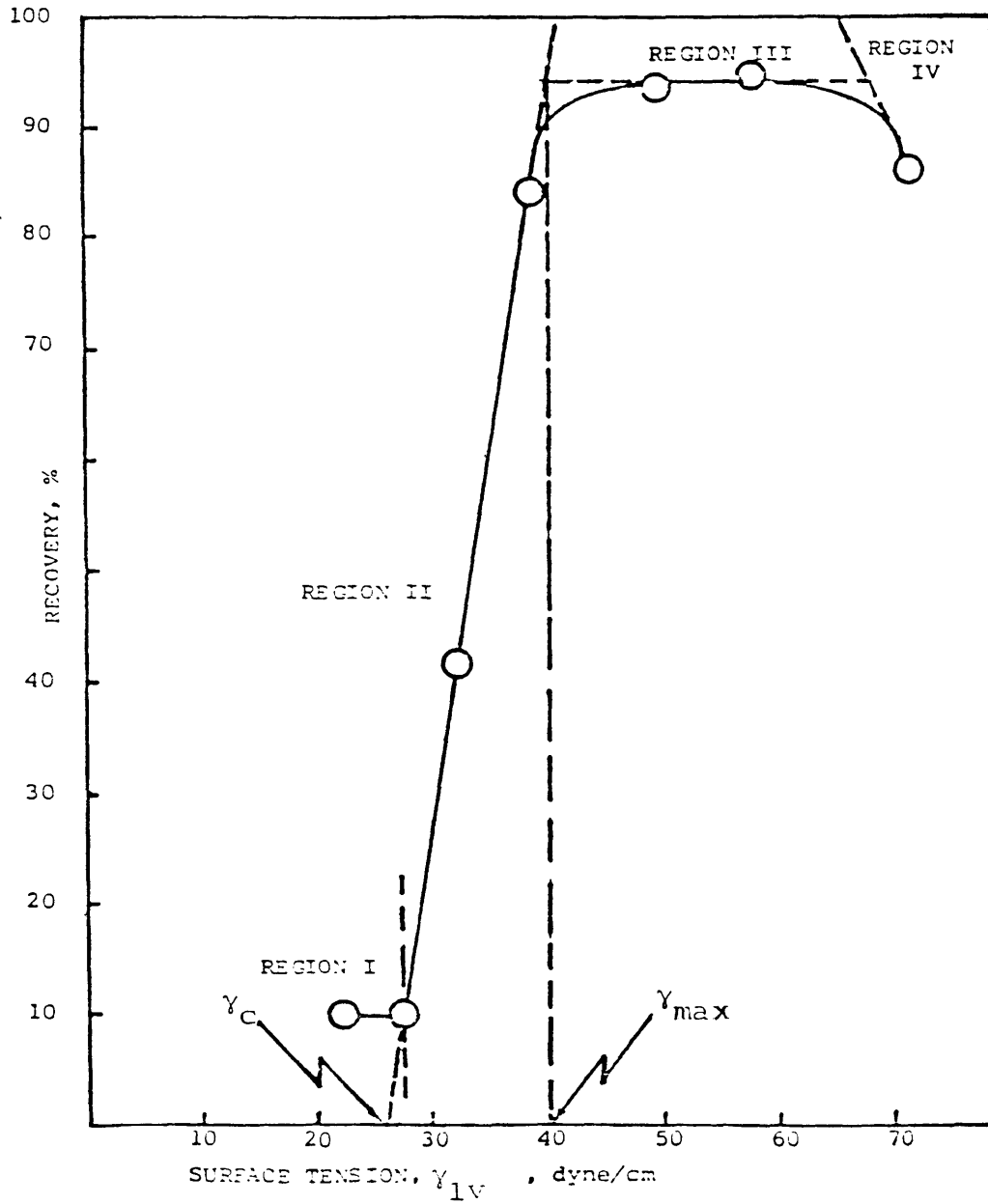


Figure 8 - Typical flotability diagram for molybdenite.

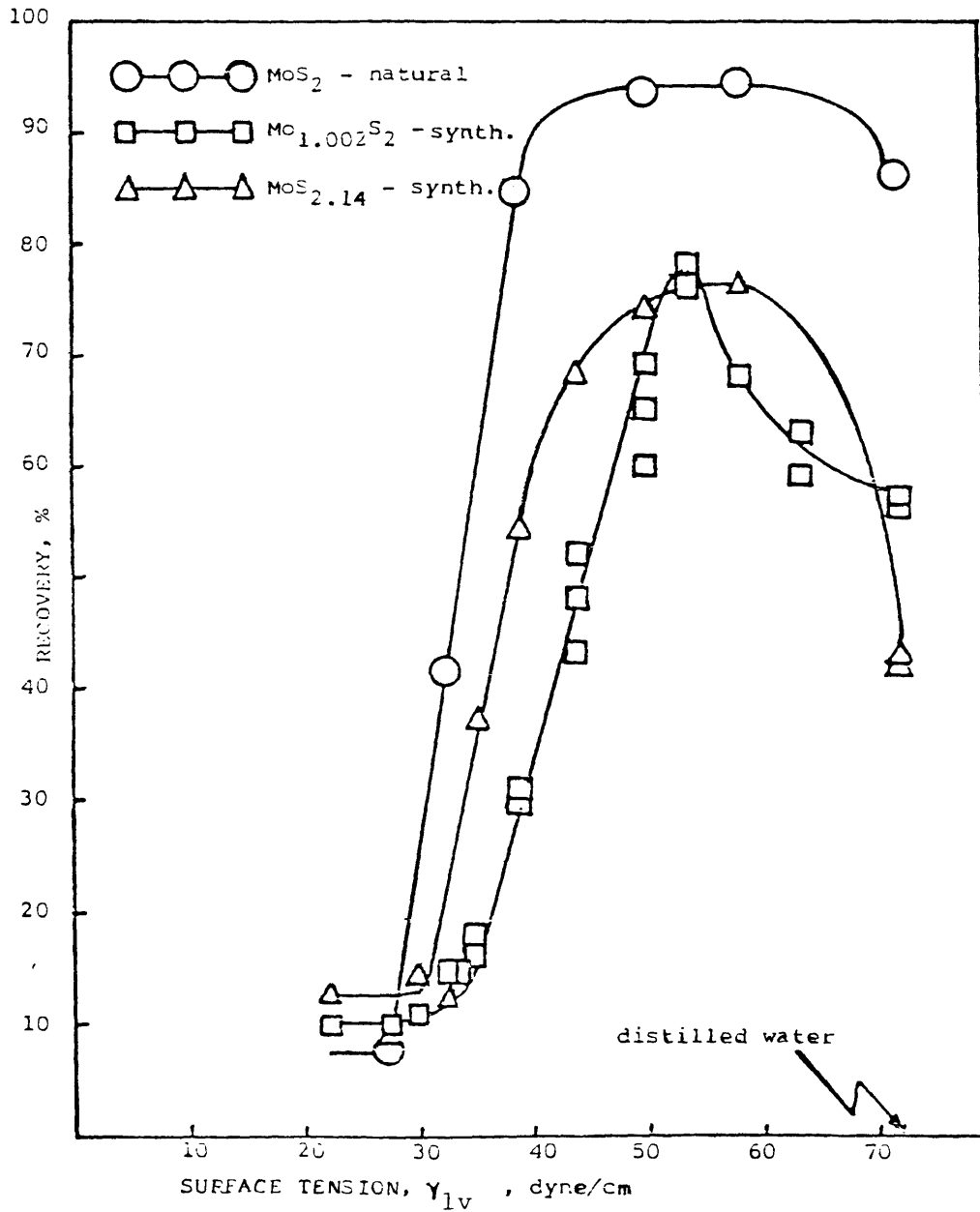


Figure 9 - Flotability diagram for stoichiometric and non-stoichiometric molybdenite in presence of nitrogen.

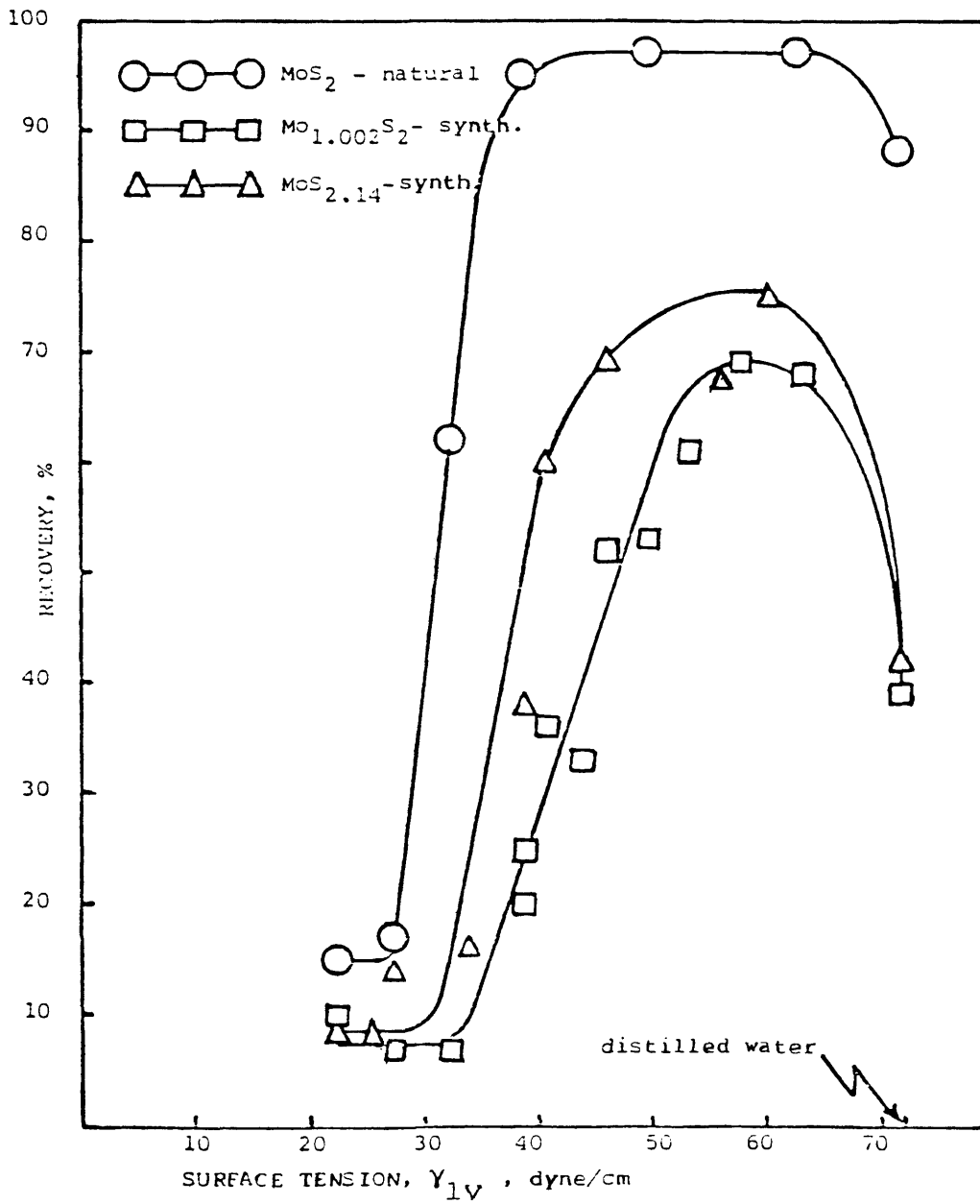


Figure 10 - Flotability diagram for stoichiometric and non-stoichiometric molybdenites in presence of air.

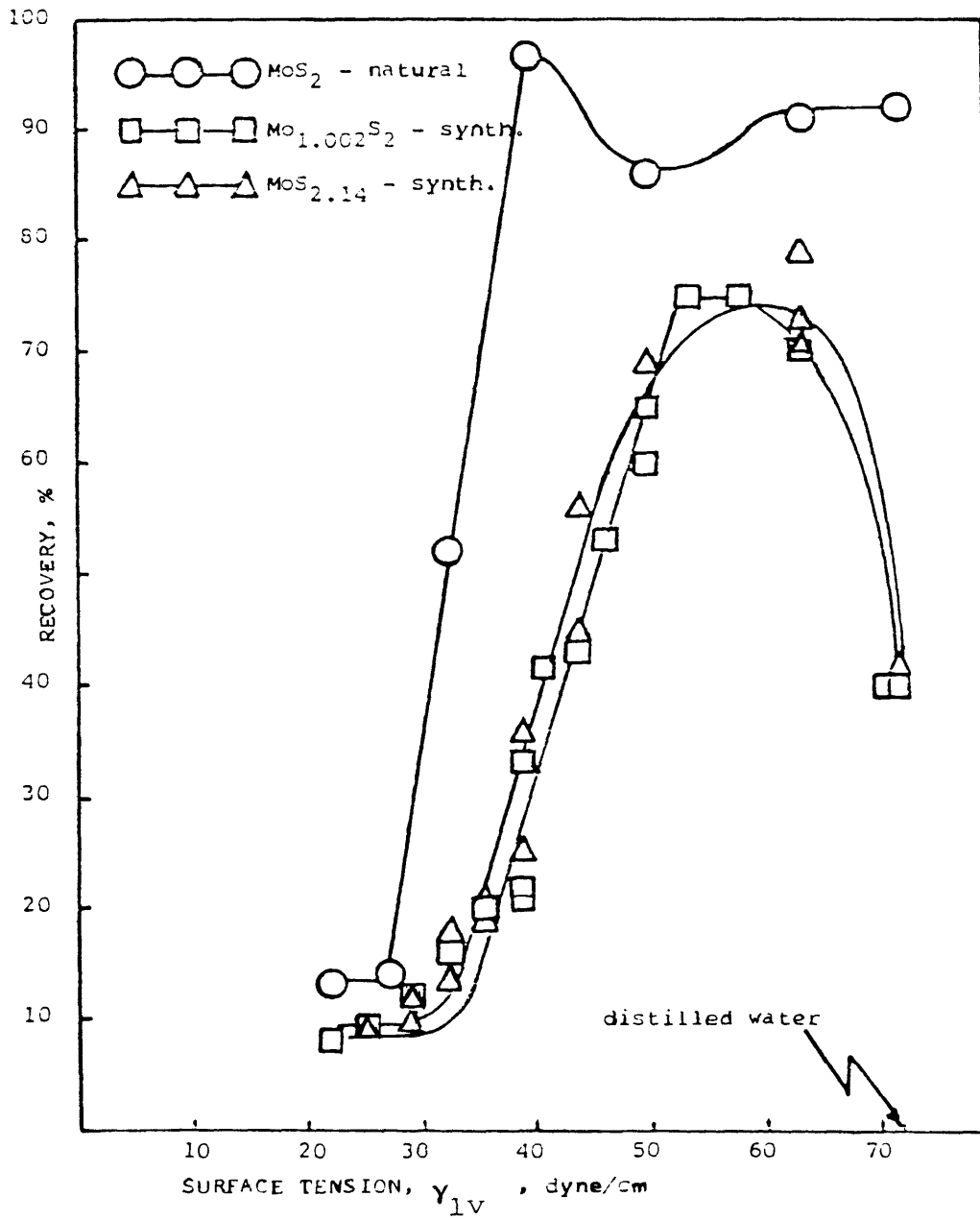


Figure 11 - Flotability diagram for stoichiometric and non-stoichiometric molybdenites in presence of oxygen.

A mathematical expression in terms of γ_c , γ_{\max} , and $dR/d\gamma$, describes the Region II of the curve.

Thus, for $R_c > 0$,

$$R_{\max} = dR/d\gamma (\gamma_m - \gamma_c) + R_c \quad (14)$$

For the corrected form of the curve, $R_c = 0$ and R_γ , % recovery for any given value of γ_{1v} , Equation (14), can be written as:

$$R_\gamma = dR/d\gamma (\gamma_{1v} - \gamma_c) \quad (15)$$

or

$$\gamma_c = -\gamma_{1v} \cdot dR/d\gamma + R_\gamma \quad (16)$$

where γ_m = the surface tension at maximum % recovery;

γ_c = the critical surface tension of wetting; and

$dR/d\gamma$ = the flotability gradient.

The critical surface tension of the molybdenite sample as well as their flotability gradient as function composition and oxygen content of the slurry are listed in Table I.

The critical surface tension of wetting of the molybdenite sample studied follows this sequence:

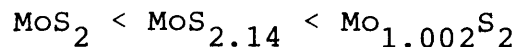


TABLE I - γ_c and γ_m and Flotability Gradient in Range II
As A Function of Solids Composition and
Oxygen Content of the Flotation Pulp

	γ_c dyne/cm	γ_{max} dyne/cm			$dR/d\gamma$		
		N ₂	Air	O ₂	N ₂	Air	O ₂
Molybdenite							
MoS ₂	26	41	37	40	6.8	8.8	6.0
MoS _{2.14}	28	48	48	58	4.8	5.2	3.5
Mo _{1.002} S ₂	31	58	63.5	58	3.7	3.0	3.5

The flotability gradient follows the same sequence, but the flotability gradient, as a function of gases used, follows the order, air > nitrogen > oxygen.

Different size fractions (irrespective of sizes) are also influenced by the gases in the same sequence as noted above, i.e., air > nitrogen > oxygen, as can be deduced from the slopes of Region II of Figures 12, 13, and 14.

On the other hand, individual size fractions float more readily in presence of either gases than their mixtures.

It is reported in the literature, that fine particles have an effect on flotability^{46,47}. From this investigation, it was found that fines float as well as coarse particles do. The slight drop in flotability of the mixed size sample may be due to adhesion of fines to coarse particles by hydrophobic bonding. Figure 15 shows the adhesion of fines on coarse particles even after screening.

The difference in maximum recovery of the various particle size fractions may be attributed to the increase in surface area or in the surface reactivity resulting from grinding.

The difference in flotability between the non-stoichiometric molybdenites may also be due to the difference in particles shape as well as due to the high electron exchange

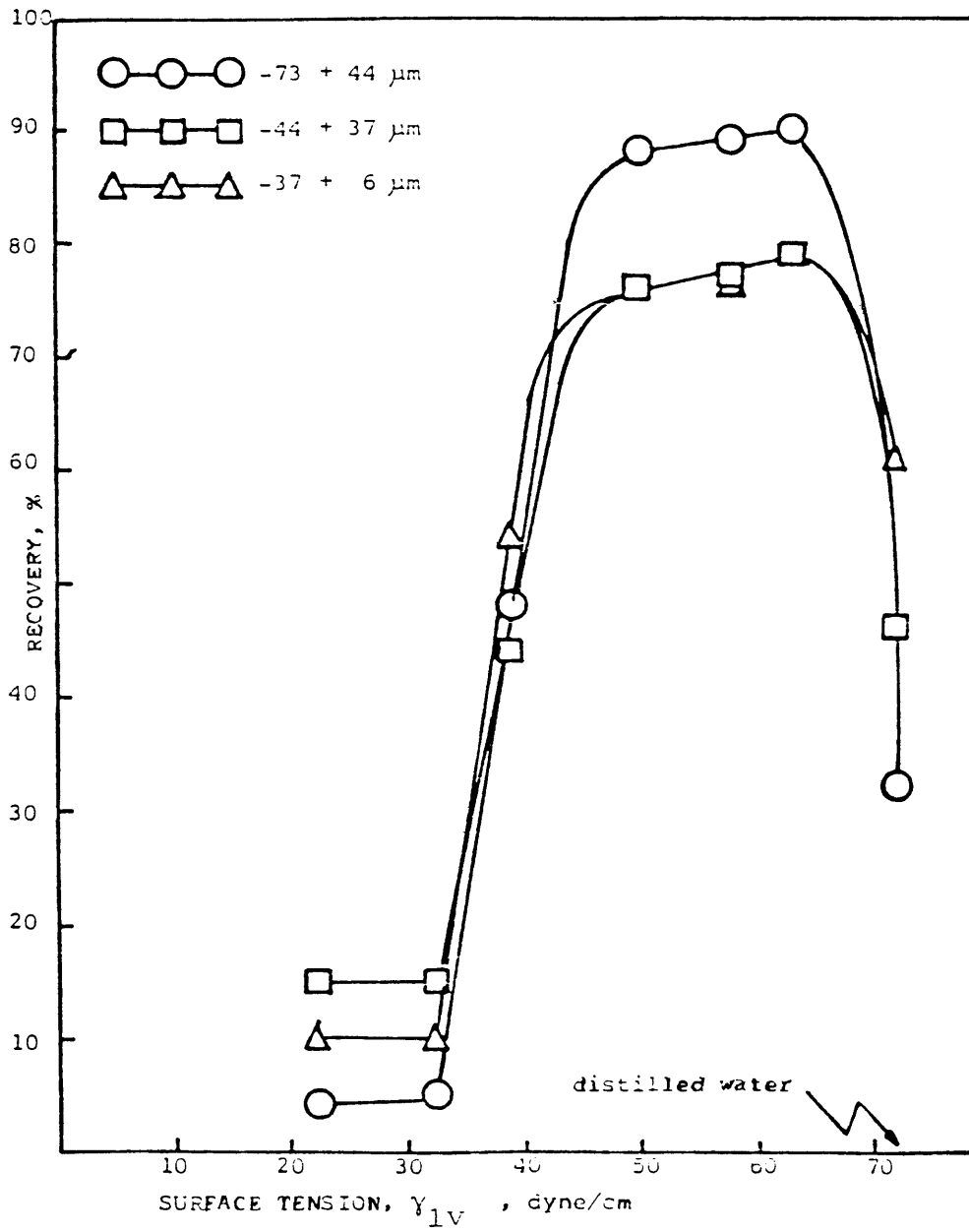


Figure 12 - Particle size effect on the flotability of molybdenum-excess molybdenite, $\text{Mo}_{1.002}\text{S}_2$.

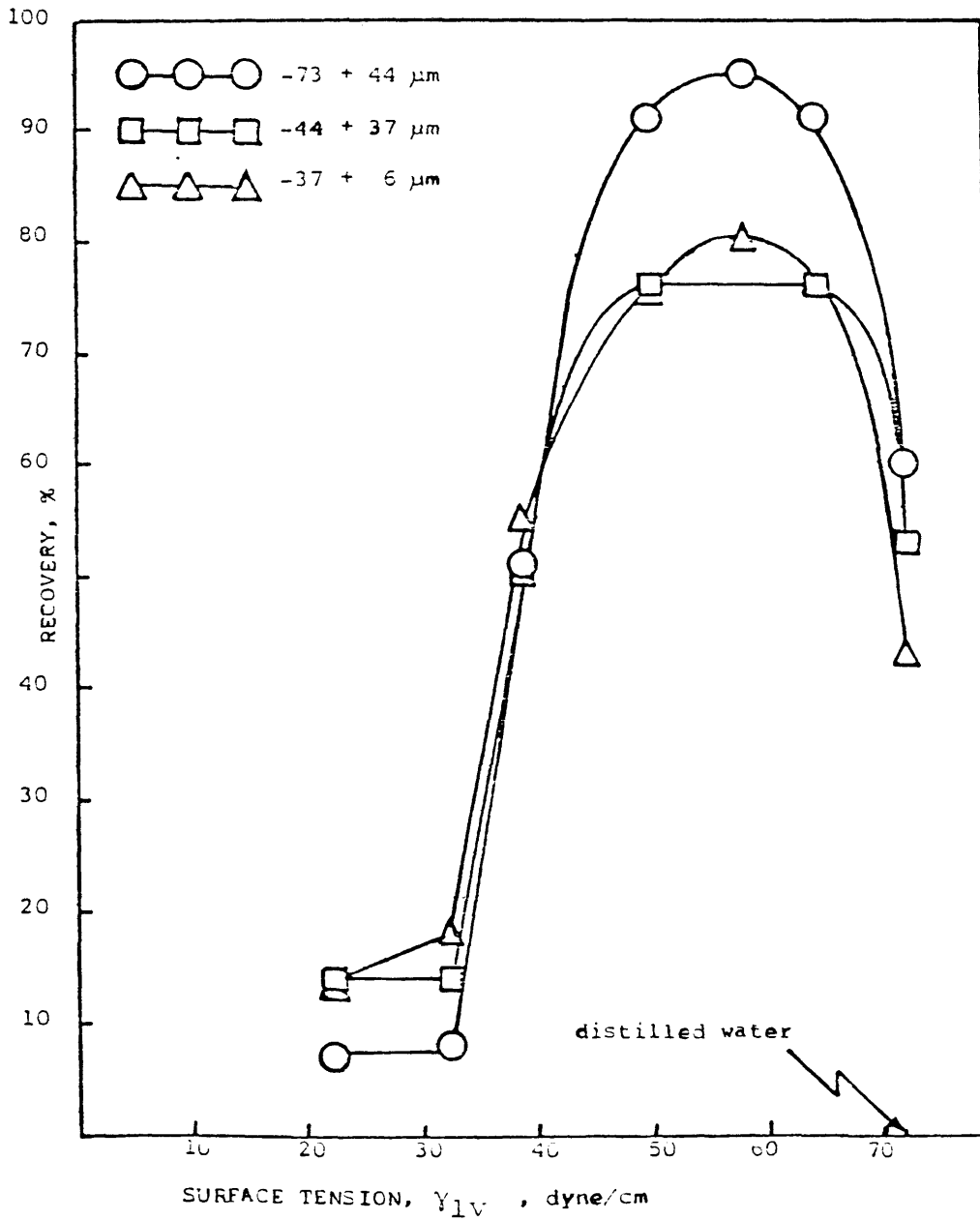


Figure 13 - Particle size effect on the flotability of sulfur-excess molybdenite, $\text{MoS}_{2.14}$.

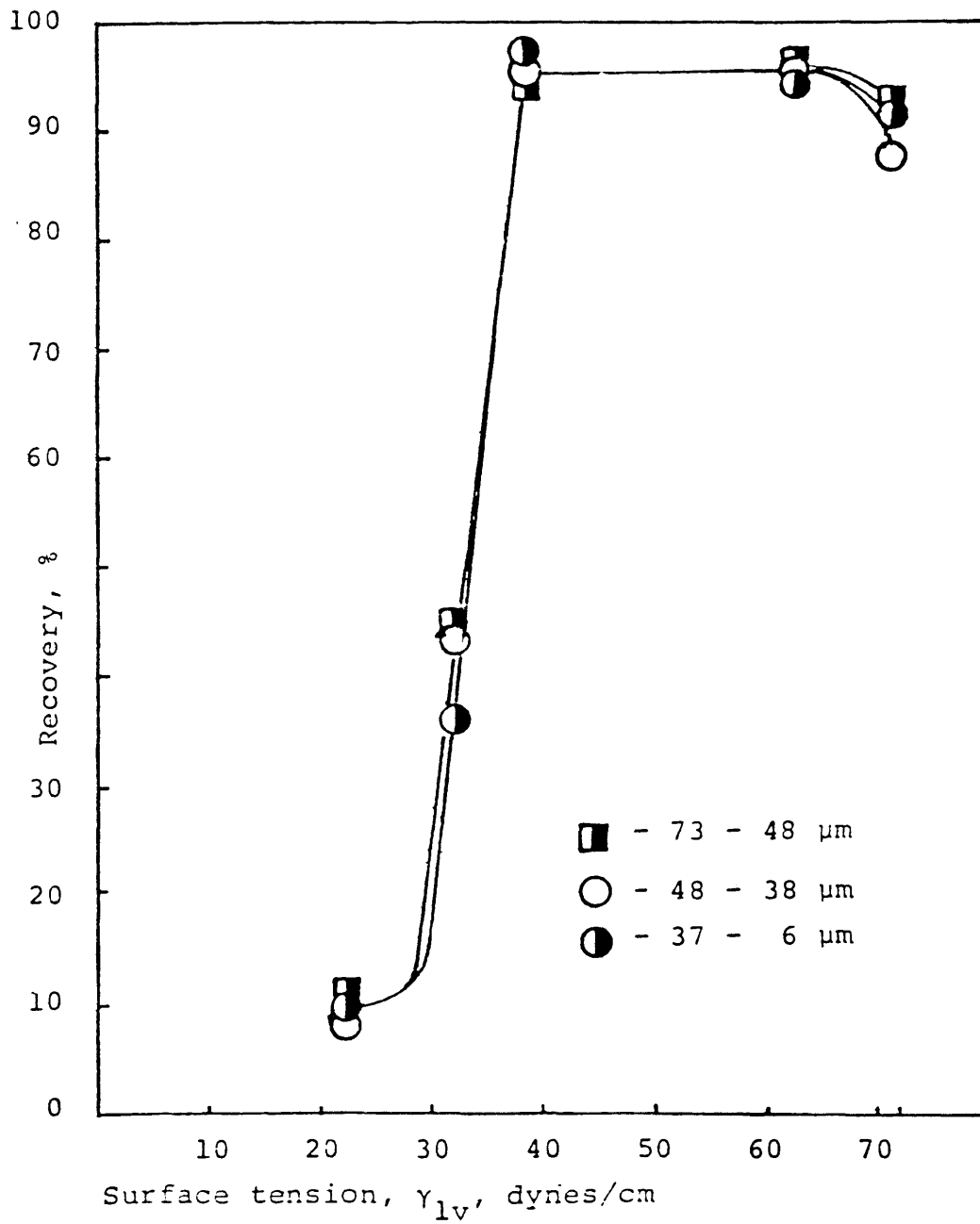


Figure 14 - Particle size effect on the flotability of natural molybdenite, MoS_2 .

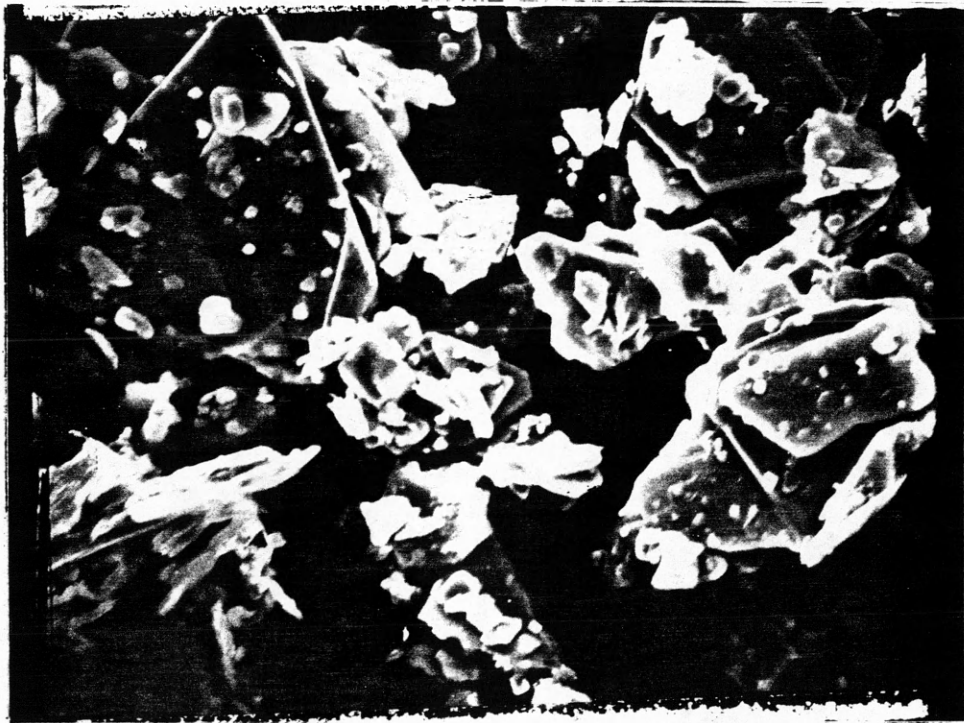
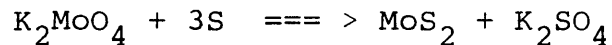
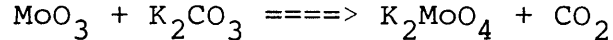


Figure 15 - Adhesion of fines to coarse particles.

barrier presented by sulfur⁴⁸. Figures 16 and 17, electron micrographs taken on the AMR 1200 scanning electron microscope, show the round and angular shapes for molybdenum and sulfur-excess molybdenite, respectively.

It has also been reported that sulfides of older geologic formations are more stable than those of the recent dates and that mineral mixtures oxidize more rapidly than any one mineral alone³². The synthetic molybdenites were prepared from the following ingredients; molybdenum trioxide (or ammonium molybdate), sulfur, and sodium carbonate. The following reactions may be assumed to have taken place, viz:



But, in reality, the molybdenites produced were ternary compounds of the following composition K_xMoS_2 (with $x = 0 - 1$)⁴⁹, because these were synthesized in an alkali flux. The latter compound has proven to be reactive. Hence, upon leaching with aqueous ammonia, some of the structure may have collapsed resulting in a more or less distorted lattice, i.e., weakening of the chemical bonds.



Figure 16 - Angular shape of molybdenum-excess molybdenite particles.

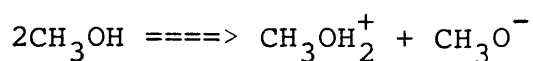


Figure 17 - Round shape of sulfur-excess molybdenite particles.

METHANOL ADSORPTION

Methanol was used as a reagent with the assumption that it would only affect the surface tension of the solution and not adsorb appreciably at the solid/liquid interface²⁷. Thus, the curves presented in Figures 8-21 would be expected in the absence of methanol adsorption at the s/l interface. The fact that flotation recovery is lower in pure water ($\gamma_{lv} = 72$ dynes/cm) than at $\gamma_{lv} < \gamma_{H_2O}$ (in Region III of Figure 8) which is obtained upon the introduction of methanol into the solution, indicates that this reagent (CH₃OH) does adsorb at s/l interface with the methyl group CH₃⁻ oriented towards the aqueous phase.

Zisman²⁰ indicated that the CH₃⁻ should exhibit a γ_c of 22-24 dynes/cm. It is known, on the other hand, that methanol does collect at the water/vapor interface⁵⁰ and that hydrogen-bond competition plays a role in this process. This may also explain the preferential adsorption of methanol on molybdenite. Furthermore, methanol can be viewed in terms of the Bronsted acid-base theory⁵¹; and therefore, could adsorb at the solid/liquid interface. Methanol is a polar solvent of which the constant of dissociation is 10^{-17} ⁵¹. Methanol dissociate as follows, viz:



Both dissociated species may adsorb on molybdenite.

Experimental data given in Figure 11, for example, shows that water is adsorbed much stronger than methanol on sulphur-excess molybdenite in the presence of nitrogen.

While stoichiometric molybdenum disulphide is less affected by water, thus the order of water uptake in the presence of nitrogen being: $\text{MoS}_2 > \text{Mo}_{1.002}\text{S}_2 > \text{MoS}_{2.14}$.

Introduction of air or oxygen to the medium brings molybdenum-excess molybdenite to a higher level of hydration as can be seen in Figures 10 and 11.

MECHANOCHEMICAL EFFECTS

One of the major variables affecting separation in mineral processing technology relates to the liberation of mineral particles in ore. It has been recognized, however, that comminution does, in fact, create problems by leading to interparticle contamination. Figures 18 and 19 show the effect of dry grinding on the recovery of both molybdenite and silica.

About 60% of silica is rendered hydrophobic in a grinding period as short as 30 seconds. For periods longer than 3 minutes, the critical surface tension of the mixture equals that of molybdenite as shown in Figures 18 and 19. Figure 20 shows the coating of silica with molybdenite specs after

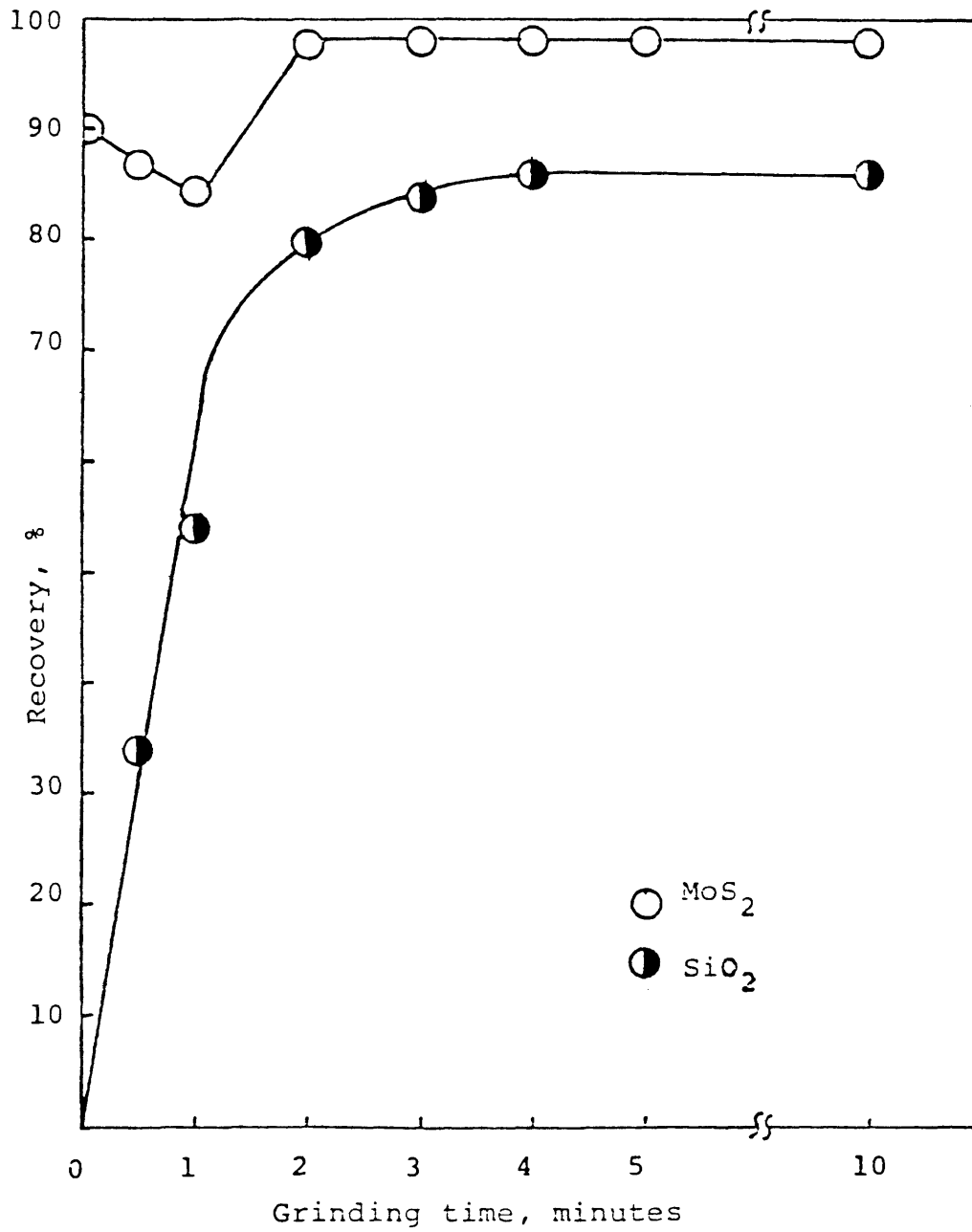


Figure 18 - Mechanochemical effect on the flotability of molybdenite and silica as a function of grinding time.



Figure 19 - Mechanochemical effect on the separation of molybdenite and silica.

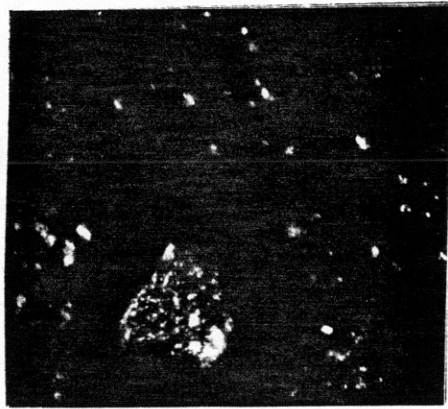


Figure 20 - Molybdenite smeared
silica.

3 minutes of grinding molybdenite-quartz mixture. From Figure 21, it can be seen that molybdenite and chalcopyrite can be separated from their mixture by setting the surface tension of the solution γ_{1v} , to be less than the critical surface tension of wetting of chalcopyrite. This is illustrated in Figure 21.

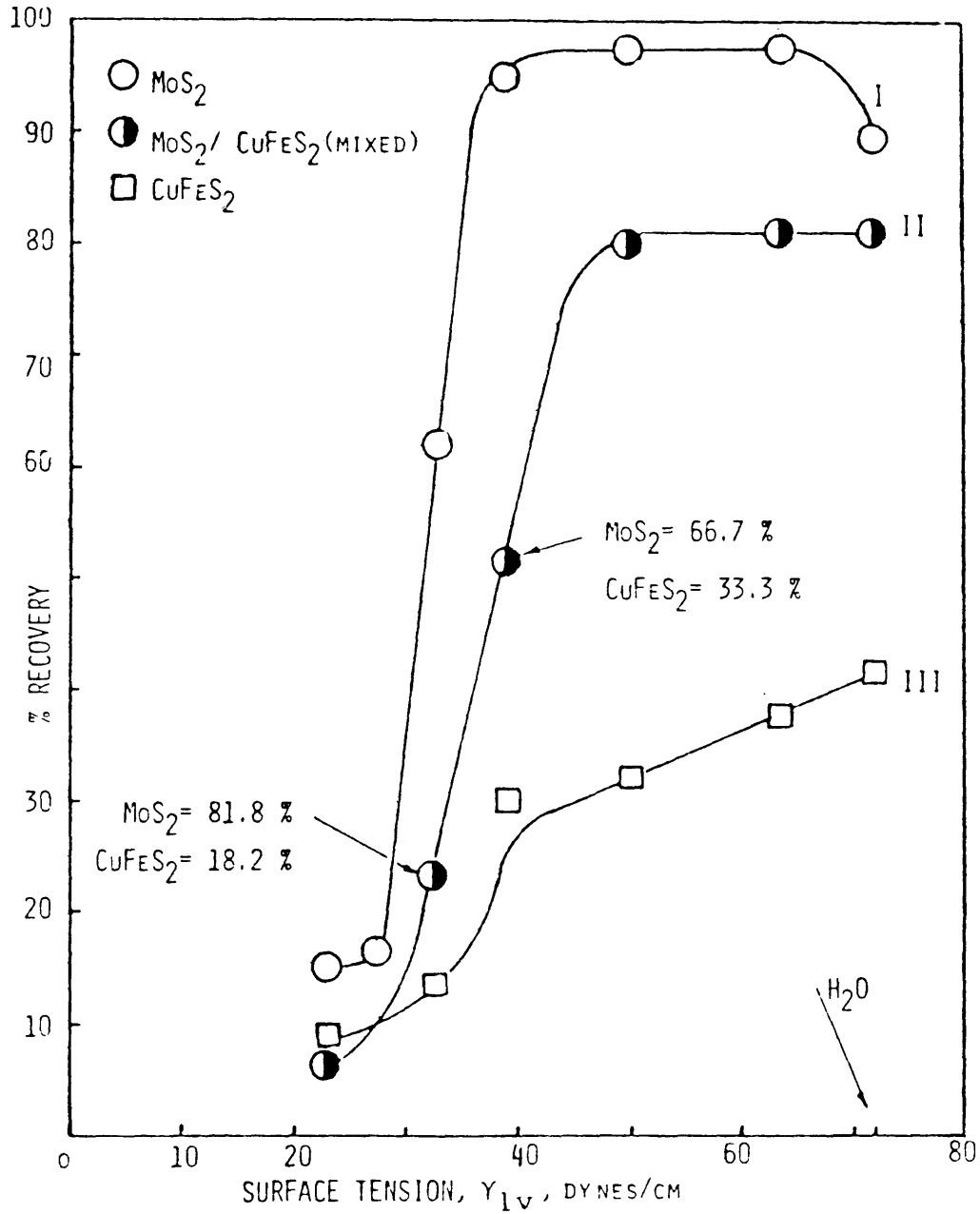


Figure 21 - Mechanochemical effect on the separation of molybdenite and chalcopyrite.

IV. CONCLUSIONS

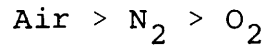
4.1 SUMMARY

From the data presented, the following conclusions were drawn:

1. Estimation of the critical surface tension of wetting by flotation, is a new alternative to the Zisman method applicable to powders where the Zisman approach is impracticable.
2. The critical surface tension of wetting, γ_c , varies with stoichiometry. The critical surface tension of the molybdenite sample studied are: natural molybdenite (stoichiometric), γ_c (MoS_2) = 26 dynes/cm; sulfur-excess molybdenum disulphide, γ_c ($\text{MoS}_{2.14}$) = 29 dynes/cm; molybdenum-excess molybdenite, γ_c ($\text{Mo}_{1.002}\text{S}_2$) = 31 dynes/cm.
3. Flotability gradient (Region II of the % recovery-curves) follows the following sequence (irrespective of gas used): $\text{MoS}_2 > \text{MoS}_{2.14} > \text{Mo}_{1.002}\text{S}_2$.
4. Dependence of the flotability gradient on the gas used for flotation. In all the cases, it appears that the

4. (continued)

sequence in flotability in various gases is as follows:



The presence of oxygen in the flotation pulp affects the solid surface of molybdenum-excess molybdenite more than the sulfur-excess or stoichiometric molybdenite.

5. Independence of the flotability gradient on particle size. The shape of particles may also contribute to the difference in flotability between $\text{MoS}_{2.14}$ and $\text{Mo}_{1.002}\text{S}_2$.
6. The flotability gradient is affected by grinding for the non-stoichiometric samples.
7. Methanol does adsorb on molybdenite, as contrary to previous contention.
8. From the mechanochemical phenomena it can be said that:
 - i*) Non-hydrophobic material ground together with hydrophobic material, become hydrophobic due to induced hydrophobicity. It will exhibit the critical surface tension of the hydrophobic material.
 - ii*) Two hydrophobic materials ground together, remain hydrophobic, but the critical surface tension

8. (continued)

- ii*) of wetting of the mixture will be the average of those materials depending on the degree of interaction.
- iii*) Loss of hydrophobicity is related to the surface reactivity of the material or presence of impurities.

4.2 RECOMMENDATIONS FOR FUTURE WORK

The problem related to the selective flotation of minerals are numerous. This study has been an attempt to elucidate some of them, such as non-stoichiometry, the presence of impurities like gangue brought about by comminution. It has also been found that minerals can be easily separated by simply controlling the surface tension of the solution and also by using unconventional reagent like methanol. To fully understand the flotation mechanism, more elaborate work, such as chemical analysis of the solid surface coupled with electrochemical studies, has to be done to correlate the surface state to adsorption of ions.

It has been shown that mechanical treatment of minerals can have a tremendous effect on flotation beside mineral liberation. As related to mechanochemistry, studies need to be done in a wet environment to see how mineral surface

properties are altered. On the same line of investigation, more work needs to be done to elucidate the role of irradiation as another activation phenomenon.

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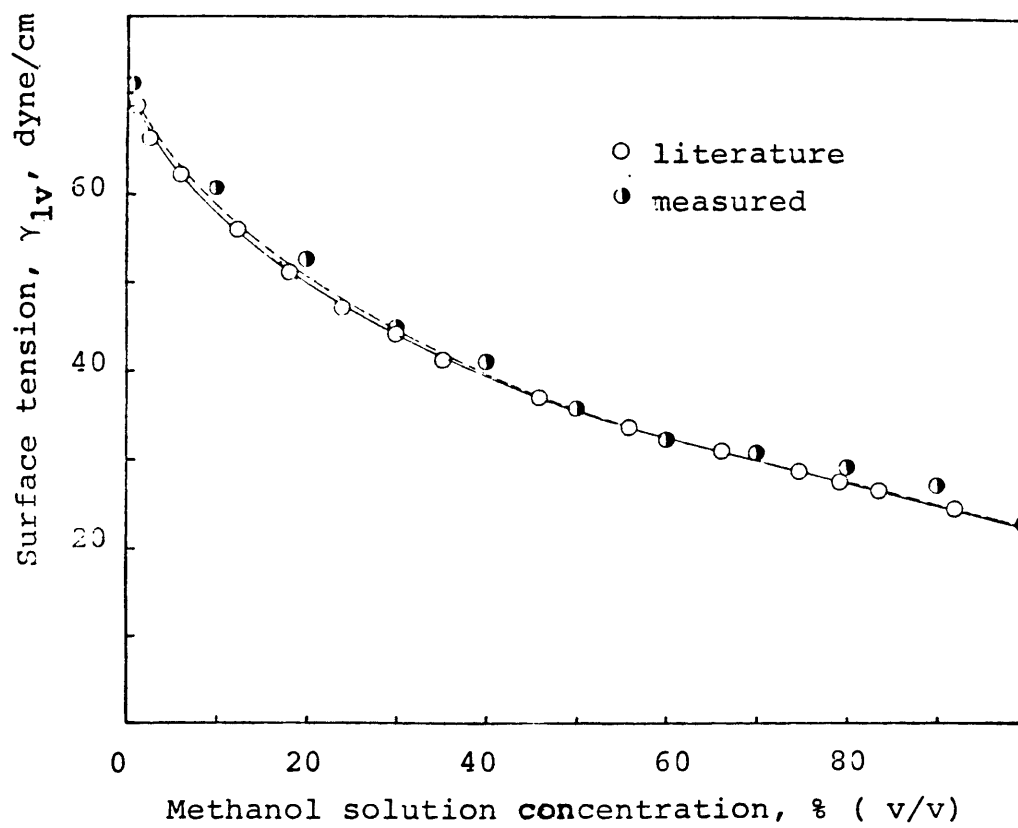
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APPENDIX 1

APPENDIX 1. MEASUREMENT OF THE SURFACE TENSION BY
THE DROP-WEIGHT METHOD.



APPENDIX 2

APPENDIX 2: MOLYBDENITE ASSAY.

Samples	Elements, %		
	S(± 0.1)	Mo(± 0.1)	MoS ₂ (± 0.1)
1	39.7	60.1	99.8
2	42.8	53.7 40.7	99.5
3	39.7	42.9	99.6

sample 1= molybdenum-excess molybdenite

sample 2= sulfur-excess molybdenite

sample 3= natural molybdenite

APPENDIX 3

Appendix 3. Optimum flotation time

