Activation of titanium slag by attrition grinding

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Abstract – *The transformation of a low-grade titanium slag into synthetic rutile or synthetic anatase can be enhanced by attrition grinding.*

Introduction

When an ore or a concentrate is finely ground, the degree of liberation usually increases, and the chances of being able to separate its components by physical methods will also increase. However, when the material is extremely fine, electrostatic forces between the particles become so great that all the particles adhere together and separation fails. Therefore, in mineral dressing, there is always an optimum particle size for effective separation. In pyrometallurgical processes, extremely fine ore or concentrate particles will possess a large surface area and therefore high reactivity. However, fine particles are undesirable because of their handling problems such as excessive dust formation. In hydrometallurgical processes, on the other hand, extremely fine particles are highly desirable because of their high reactivity; handling is not a problem because the material is usually pumped as a slurry. It seems, therefore, that it is only the hydrometallurgist who benefits from any grinding method that can produce extremely fine particles.

Grinding is an expensive operation; the cost usually increases with decreasing final particle size. Further, for each grinding machine, there is a limit for the particle size to be obtained no matter how long grinding has been done. Most grinding machines yield a product that is in the range of 10 to 20 μm. Recently, a less known technique called attrition grinding has been introduced in the mineral industry. It claims to produce an extremely fine particle size distribution of the product at a lower cost (Feld et al., 1960; Stanczyk and Feld, 1963, 1965, 1968, 1972; Lin et al., 1975; Hansen et al., 1981). This technique employs intense agitation of a mixture of the material to be ground, a grinding medium, and, but not necessarily, a suspending fluid. It is usually used in the paint industry and for preparing fillers and coatings in the paper industry.

Recent studies by Stanley at al. (1973, 1973a) have demonstrated that the attrition grinding process was more efficient than other fine grinding processes. For example, it was shown in the ceramic industry that a considerably higher rate of size reduction of barium ferrite was obtained in the attrition grinder than in any other mill tested. Specifically, particles 1 μm in size were produced in two hours in attrition grinding, in 10 hours in planetary milling, in 30 hours in vibration milling, and over 30 hours in ball milling.

Davis et al. (1973, 1981) showed that attrition grinding of kaolin for 30 minutes increased the amount of material less than 2 μm from 15% to 75% while a colloid mill increased the same fraction to only 18%. Autogenous attrition grinding has also been used successfully. In this case, large particles of the material to be ground are used as the grinding media.

Although the advantages of attrition grinding have been known long ago to the paint and ceramic industries, application to hydrometallurgy has been limited. Paudert et al. (1967) reported that a sample of normally acid-insoluble cassiterite dissolved completely after prolonged milling. Gerlach and coworkers (1973) and Pawlek (1976) reported that chalcopyrite subjected to attrition grinding showed remarkable reactivity toward leaching with dilute sulfuric acid and attributed this to lattice distortion.

Agar et al. (1974), however, refuted this concept by measuring the surface area of the comminuted chalcopyrite and also by studying the lattice distortion. Both were found to be proportional to grinding time. Leaching strain-free (annealed) and strained (unannealed) chalcopyrite with H_2SO_4 in presence of oxygen showed that the leaching rate was directly proportional to the specific surface area and is independent of lattice distortion. Later, Beckstead et al. (1976) confirmed these findings.

The attrition grinding of chalcopyrite concentrate prior to leaching has been tested in two pilot plants — one by the Lurgi-Mitterberg process in Austria (Anon., 1973) and the other by Envirotech in Utah (Anon., 1980). In both cases high leaching rates were achieved.

The foreign literature also contains many references to the role of attrition grinding in enhancing chemical reactions (Paudert et al., 1979; Kulebakin and Khalitova; Gock and Szantho, 1980; Arkhipenko et al., 1980; Huhn and Muller, 1980; Ziegler, 1981; Zelikman et al., 1981; Kulebakin et al., 1981; Avvakumov et al., 1981; Kolbanev and Butyagin (1982); Grizina, 1983; Berger et al., 1983; Lapteva et al., 1983; Chaikina et al., 1983; Vorobeichik et al., 1983, 1983a), and a book on the subject was recently published (Juhasz and Opoczky, 1982). The present investigation was undertaken to examine the effect of attrition grinding on the leaching properties of a standard grade titanium slag as a possible way for enhancing the transformation of the slag into an upgraded product equivalent to synthetic rutile (Toromanoff and Habashi, 1983, 1985). The slag studied was mainly composed of iron magnesium titanate and a silicate phase (Toromanoff and Habashi, 1984).

Experimental

Reactor

Tests were conducted in two stainless autoclaves heated externally by an electric furnace. Both apparatus were supplied by Parr Instrument Co. of Moline, IL. Agitation was conducted by shaking the whole setup by a motor with an eccentric shaft. One autoclave has a capacity of 25 mL and the other 200 mL.

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The slag sample was mixed with an acid in a Teflon container that fits inside the autoclave. Tests in the large autoclave were conducted to prepare large samples suitable for separating the unreacted slag from the product.

Sample

The slag sample used had the analysis shown in Table 1. It is the commercial product of QIT Fer et Titane Inc. in Sorel, Quebec, and known under the trade name Sorelslag. The effect of grinding time in the Shatterbox on particle size distribution is shown in Table 2. It can be seen that after 45 minutes, there was no more change in the granulometry, i.e., the maximum limit of grinding was achieved in that particular machine. The product obtained by attrition grinding for two hours was found to pass 400 mesh and was mainly 2.5 to 5.5 μm in size. Table 3 compares the granulometry of the different samples used — the regular slag passing 400 mesh, the quenched and ground slag passing 400 mesh, the slag ground for 45 minutes in the Shatterbox, and the attrition slag for two hours. It can be seen that:

- The quenched and ground slag passing 400 mesh has the same granulometry, specific surface area, and mean diameter as the regular (unquenched) slag passing 400 mesh.
- Slag ground for 45 minutes has a large specific surface area (1.29 m^2/cm^2) and a small mean particle diameter (6.69 μm).
- The attrition ground slag has a mean diameter of about 4.5 μm.

Table 1 — Analysis of Slag Sample Used After Grinding and Sieving to −400 Mesh

	%
TiO_2	71.35
Ti_2O_3 (expressed as TiO_2)	10.8
Fe (total)	9.2
Fe (metallic)	0.05
Al_2O_3	5.42
SiO_2	5.26
CaO	1.12
MgO	5.24
MnO	0.26
V_2O_5	0.58
Cr_2O_3	0.18

Table 3 — Particle Size Distribution of Differently Ground Slag

Particle size, μm	Regular −400		Quenched −400		45 min wet grinding		Attrition grinding 2 hr scanning microscope reading
	% cumul.	%	% cumul.	%	% cumul.	%	
176	100		100		100		
125	100		100		100		
88	100		100	1.1	100		
62	100	3.0	98.8	2.1	100		
44	96.9	12.0	96.7	10.8	100		
31	84.9	17.5	85.9	16.0	100	0.2	
22	67.3	14.9	69.9	15.3	99.7	4.6	
16	52.3	12.0	54.5	12.2	95.0	10.7	
11	40.3	10.3	42.2	12.3	84.2	14.8	
7.8	30.0	7.8	29.8	9.4	69.4	15.2	
5.5	22.1	8.8	20.3	8.5	54.1	20.0	
3.9	13.3	7.1	11.8	6.6	34.0	17.6	majority
2.8	6.1	6.1	5.1	5.1	16.4	16.4	2.5 - 5.5
specific surface area	0.702		0.688		1.29		
mean diameter	17.4		17.3		6.69		~4.5

Samples were ground and sieved to different fractions to study the effect of particle size. Grinding was conducted in two machines:

- A tungsten carbide grinder Shatterbox No. 8504 supplied by SPEX in Metuchen, NJ. This grinder is known to be a very effective machine. It is a short steel cylindrical container 152.4 mm inner diameter and 76.2 mm high, 100 mL capacity, with a loose tungsten carbide liner, a massive loose tungsten carbide disk, and a steel cover. When mounted on a vibrating machine, the UC disk grinds the material while bombarding the loose liner.
- An Attritor Model 1S of Union Process Inc. of Akron, OH. This is a cylindrical water-jacketed tank equipped with three horizontal pedals mounted on a spindle driven by a variable speed motor to agitate the batch. A large number of 6-mm steel balls were added to the batch as a grinding media. Grinding was done dry but a few drops of methyl alcohol were added to avoid agglomeration.

A regular slag sample (about 2 mm size) ground dry in the Shatterbox for 20 minutes and sieved to pass 400 mesh was taken as a standard for comparison with

Table 2 — Effect of Grinding Time of a Slag Fraction −325 +400 Mesh on Particle Size Distribution

Particle size, μm	slag, original fraction, −325 +400		Time of wet grinding									
			15 min		30 min		45 min		60 min		90 min	
	% cumul.	%	% cumul.	%	% cumul.	%	% cumul.	%	% cumul.	%	% cumul.	%
176	100	0	100		100		100		100		100	
125	100	0.3	100		100		100		100		100	
88	99.6	12.6	100		100		100		100		100	
62	80.9	24.8	100	2.6	100		100		100		100	
44	56.1	17.2	97.3	18.9	100	7.8	100		100		100	
31	38.9	13.1	78.4	18.4	92.1	18.1	100	0.2	100		100	1.2
22	25.7	7.0	59.9	14.2	73.9	16.7	99.7	4.6	100	4.3	98.8	4.0
16	18.7	6.2	45.7	13.4	57.2	14.8	95.0	10.7	95.6	9.5	94.7	10.9
11	12.4	2.8	32.2	9.1	42.4	14.7	84.2	14.8	86.1	13.3	83.7	12.3
7.8	9.6	1.9	23.1	6.0	27.6	8.7	69.4	15.2	72.8	13.2	71.3	10.5
5.5	7.7	2.7	17.1	6.5	18.8	7.6	54.1	20.0	59.5	20.0	60.8	19.2
3.9	4.9	2.0	10.6	5.6	11.2	5.1	34.0	17.6	39.5	18.9	41.5	18.9
2.8	2.9	2.9	4.9	4.9	6.0	6.0	16.4	16.4	20.5	20.5	22.6	22.6
specific surface area, m^2/cm^3	0.333		0.607		0.695		1.29		1.38		1.40	
mean diameter, μm	40.3		19.7		15.5		6.69		6.25		6.45	

samples prepared by other methods as follows:

- A slag sample was sized between −325 and −400 mesh then wet ground for different periods of time in the same mill and the particle size of the product was determined.
- A regular slag sample was melted in a crucible by an oxyacetylene torch then quenched while molten in ice cold water. The quenched product was then ground and sieved to pass 400 mesh.
- A regular slag sample was ground for two hours in the attritor under argon atmosphere, with 6.35-mm (¼-in.) carbon steel balls, at 320 rpm spindle speed.

Test procedure

Hydrochloric and sulfuric acids of different concentrations were used. For the small autoclave, the volume was kept constant at 15 mL and different weights of slag were treated to study the effect of acid concentration and solid-liquid ratio. For the large autoclave, 150 mL acid were used. A small amount of Lignosol was added to prevent caking. Tests were conducted at different times and at the end of each test the product was filtered, washed, dried, weighed, and then analyzed by X-ray diffraction. The solution from the small autoclave was adjusted to 100 mL and analyzed by atomic absorption for Ti, V, Fe, Al, and Mg. That from the large autoclave was adjusted to 500 mL and analyzed in the same way.

Evaluation of product

The product of reaction was examined by:

Chemical analysis — X-ray fluorescence was used to determine Fe, Al, Mg, Ca and Si, and wet methods were used to determine Ti (III).

Phase analysis — For tests conducted in HCl, the percentage of rutile in the product was determined quantitatively by X-ray diffraction using a synthetic mixture of slag and pure crystals of naturally occurring rutile supplied by the US Bureau of Standards, as shown in Fig. 1. In this diagram, X_i is the area of the rutile peak for a mixture of rutile and slag, and R equals the area of rutile peak for pure rutile.

For tests conducted in H_2SO_4, the product of reaction was only anatase, whose peak in the X-ray diffraction pattern coincides with that of the slag. Therefore, phase analysis by this method was not possible. It was thought, however, that if the sample were heated at 800° for two hours to effect the transformation of anatase to rutile, it might be possible to calculate the amount of anatase originally formed. But, it was found that even heating at 1100° for five hours was not enough to completely effect the transformation. Therefore, this procedure was abandoned, and the evaluation of these tests was conducted by determining the TiO_2 content in the residue.

Physical methods — The difference in particle size and color of the precipitated TiO_2 as compared to the slag suggested separating one from the other by a physical method. Two methods were used:

- Gravity method — In this method, separation was conducted by slurrying the product in water and separating the suspended fine particles from the settled heavy fraction.
- Magnetic method — This method is based on the fact that titanium slag contains a small amount of tiny metallic iron droplets that solidified before settling during the production of the slag. These particles render the slag magnetic. A CHARPO MWL 3465-131 machine was used (supplied by Research & Engineering Co., Jacksonville, FL).

The final test was *examination by scanning electron microscopy.*

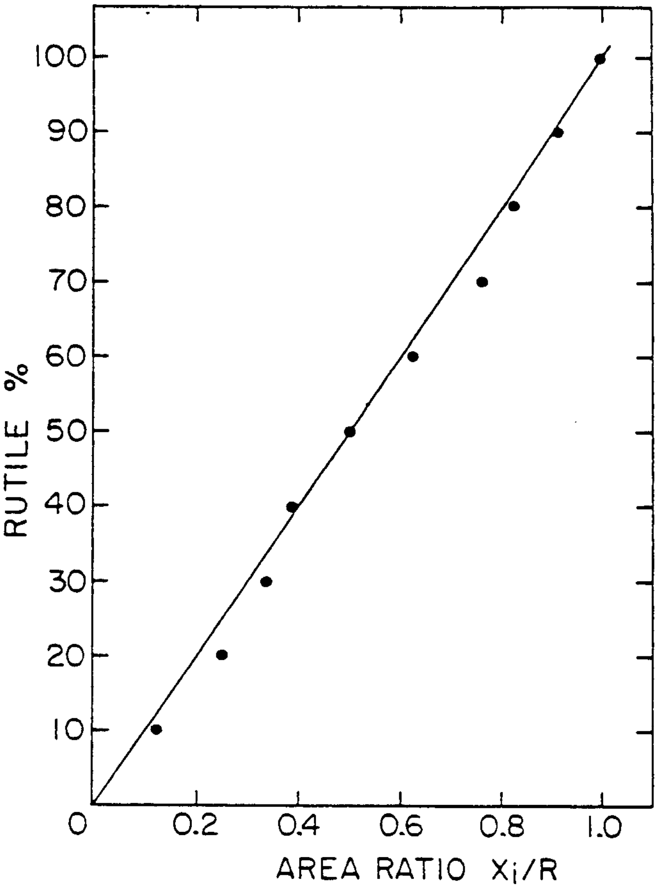

Fig. 1 — Standard curve for the determination of rutile in product by X-ray diffraction

Method of calculation

Knowing the weight of the residue and the percentage of rutile, one can calculate the fraction transformed into rutile. Knowing the amount of rutile and titanium in solution as determined by atomic absorption, it was possible to calculate the amount of reacted slag and TiO_2 in the unreacted slag. From the amount of dissolved slag, it was possible to calculate the amount of SiO_2 liberated. For example,

$$\text{Weight of rutile in residue, g} = \frac{\text{weight of residue (g)} \times \text{\% rutile}}{100}$$

$$\text{\% } TiO_2 \text{ in solution (by atomic absorption)} = \frac{TiO_2 \text{ in solution (g)}}{\text{total } TiO_2 \text{ (g)}} \times 100$$

$$\text{\% } TiO_2 \text{ in residue as unreacted slag} = \frac{\text{total } TiO_2 \text{ (g)} - (\text{rutile (g)} + TiO_2 \text{ in solution (g)})}{\text{total } TiO_2 \text{ (g)}} \times 100$$

$$\text{Percentage of slag reacted} = \frac{\text{rutile (g)} + TiO_2 \text{ in solution (g)}}{\text{total } TiO_2 \text{ (g)}}$$

Weight slag unreacted (g) = slag feed (g) − slag reacted (g)

$$\text{\% unreacted slag in residue} = \frac{\text{weight slag unreacted (g)} \times 100}{\text{weight residue (g)}}$$

$$\mathrm{SiO_2\ liberated\ (g)} = \frac{\mathrm{slag\ reacted\ (g)} \times \mathrm{total\ SiO_2\ (\%)}}{100}$$

$$\mathrm{\%\ SiO_2\ liberated} = \frac{\mathrm{SiO_2\ liberated\ (g)}}{\mathrm{weight\ of\ residue\ (g)}} \times 100$$

Characterization of slag samples

The granulometry of the ground particles was determined by a Leeds and Northrup Microtrac particle size monitor. This apparatus combines a laser beam with optical technology and a microcomputer to convert the light-scattering effect of particles in slurries to measured and calculated numerical values. The machine reports:

- A 13-channel histogram. Successive channel widths vary as $\sqrt{2}$ with the smallest from 1.9 to 2.8 μm and the largest from 125 to 176 μm.
- A 13-channel cumulative "% passing" number for 176, 125, 88, 62, 44, 31, 22, 16, 11, 7.8, 5.5, 3.9, or 2.8 μm (in volume percent).
- The average particle diameter (mean) of the volume distribution in microns.
- The specific surface which is the scaled reciprocal of average particle diameter (mean) of the area distribution.

Figure 2 shows the granulometric curve for slag ground to −400 mesh. Figure 3 shows the effect of time of grinding in the Shatterbox on the mean particle size of the slag.

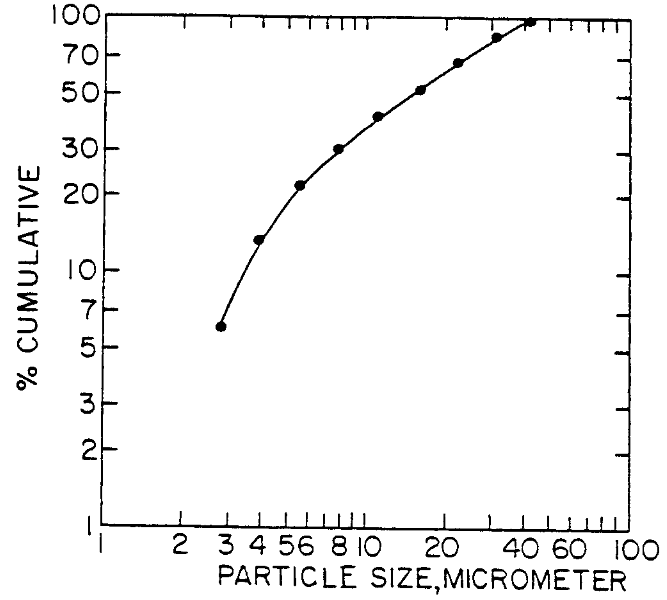

Fig. 2 — Granulometric curve for slag ground to −400 mesh

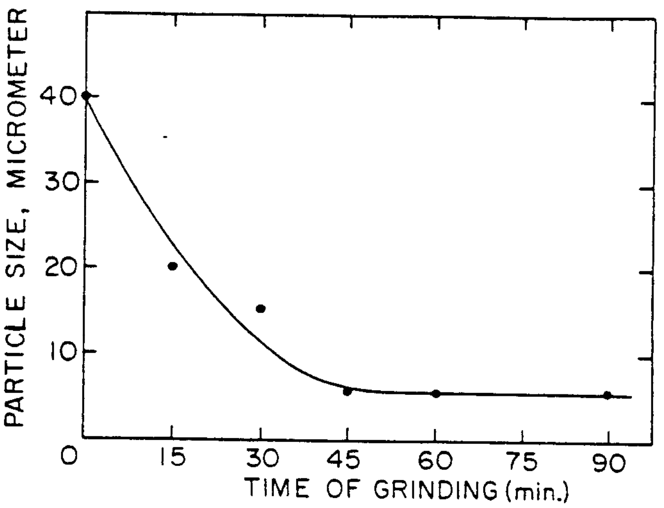

Fig. 3 — Effect of time of grinding in the Shatterbox on the mean particle diameter of product

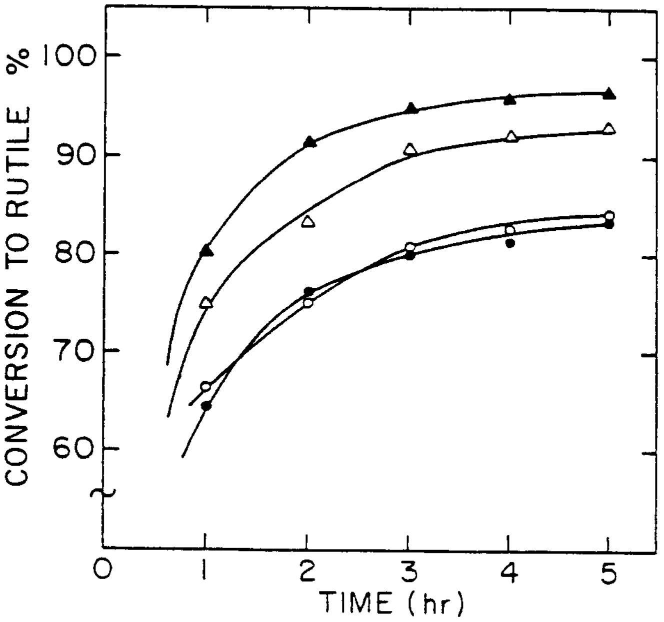

Fig. 4 — Effect of time on the conversion of activated slag to rutile at 180° C, 10 N HCl

▲ Attrition
△ 45-min grinding
○ −400 mesh quenched
● −400 mesh regular

Results and discussions

Figure 4 shows the effect of method of activation of slag on its transformation to synthetic rutile. It can be seen that the samples ground to −400 mesh in the Shatterbox machine behave similarly whether the sample was quenched or not. On the other hand, the sample ground for 45 minutes showed a faster rate of reaction than the previous samples. The attrition ground sample reacted even faster. Figure 5 shows also the effect of acid concentration and temperature. The rate of transformation increases with increased acid concentration and increased temperature.

As a result of increased reaction rate of the attrition ground slag, more rutile is formed and the product contains a high rutile content. Figure 6 shows that after five hours reaction, the −400-mesh slag was transformed into a product containing 72% rutile, while an attrition ground sample was transformed into a product containing 88% rutile. Also, in the attrition ground sample, it was observed that the rate of dissolution is faster than the rate of precipitation, as shown in Fig. 7 by the increased titanium ion in solution. A scanning micrograph of the reaction product in 10 N HCl is shown in Fig. 8; it can be seen that rutile is deposited in the form of needles about 2 μm long.

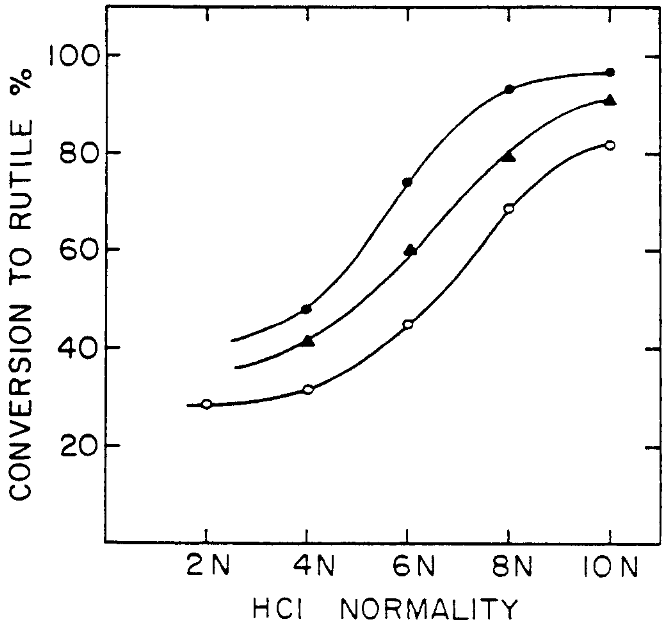

Fig. 5 — Effect of acid concentration on the conversion of regular slag, −400 mesh, to rutile at 180°C, 200°C, and attrition ground slag at 200°C; 4 hrs retention time
● Attrition, 200°C
△ Regular, 200°C
○ Regular, 180°C

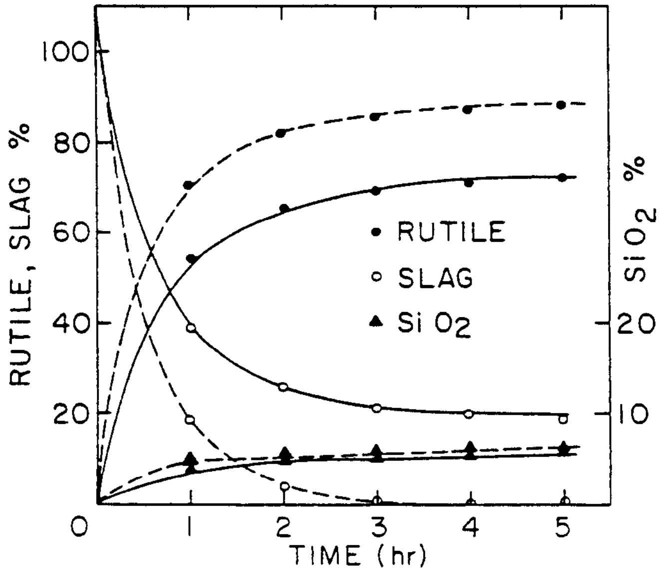

Fig. 6 — Phase distribution in the residue obtained by conversion of regular slag, −400 mesh, as compared to attrition ground slag; 180°C, 10 N HCl
— — — Attrition
——— −400 mesh

Behavior of impurities

Figure 9 shows the concentration of TiO_2 as well as the impurities Fe, MgO, Al_2O_3, and V_2O_5 in solution as a function of HCl concentration. It can be seen that the attrition ground material produces solutions containing high Fe, MgO, and Al_2O_3 concentrations. The amount of vanadium and titanium in solution is negligible except at 10 N HCl.

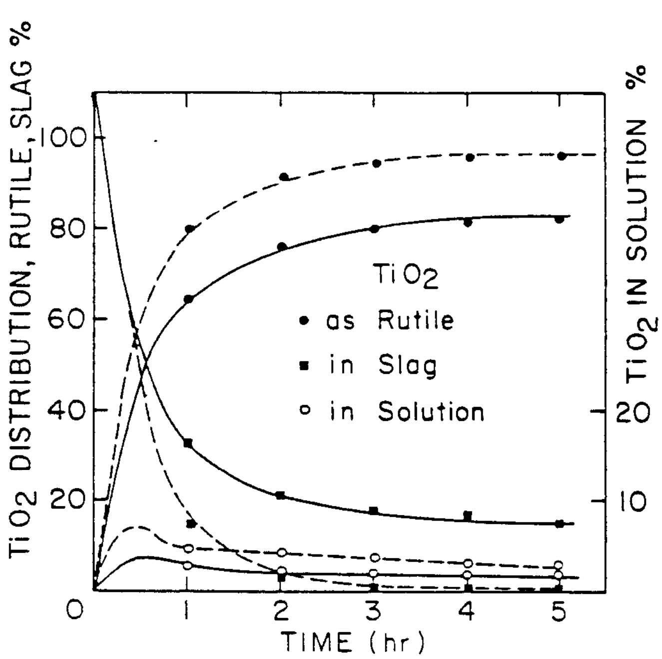

Fig. 7 — Phase distribution of TiO_2 for regular slag, −400 mesh, as compared to attrition ground; 180°C, 10 N HCl
— — — Attrition
——— −400 mesh

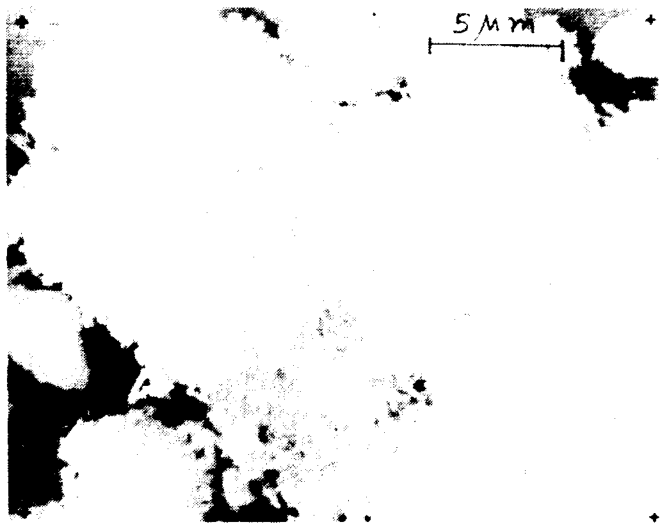

Fig. 8 — Scanning micrograph of reaction product in 10 N HCl showing rutile in form of needles about 2 μm long (X 4500)

Separation of rutile and anatase from unreacted slag

X-ray diffraction analysis has shown that the product of reaction of HCl with the slag is TiO_2 in the form of rutile, and that produced by treatment of the slag with H_2SO_4 is TiO_2 in the form of anatase. Any unreacted slag had the same X-ray diffraction pattern as the original slag, i.e., no intermediate phase was produced. It was not possible to identify the silicate or SiO_2 phases in the reaction product by X-ray diffraction, although chemical analysis showed that in most cases SiO_2 content became higher than the original slag treated.

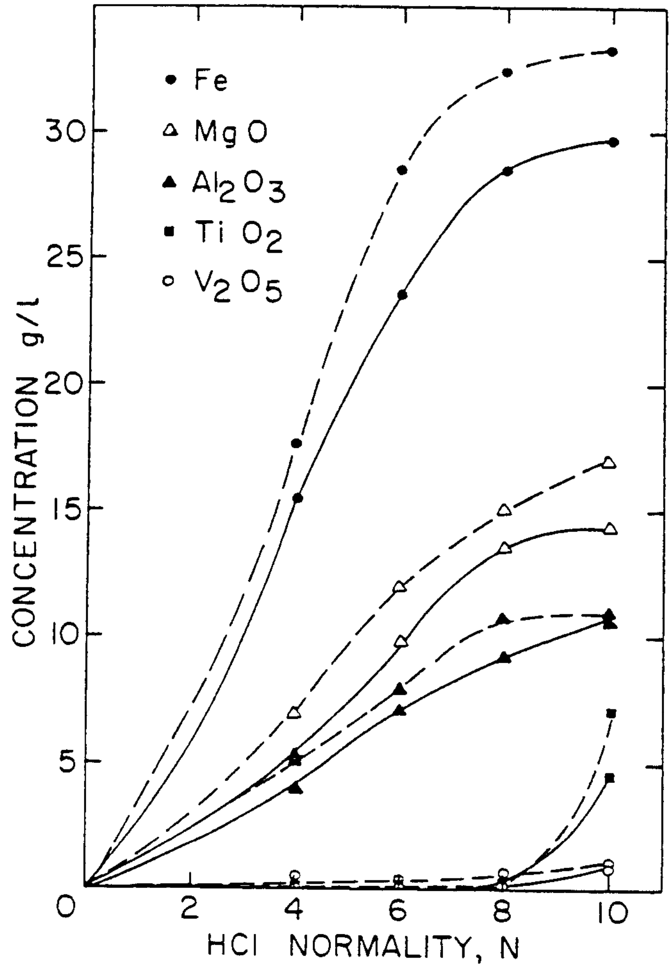

Fig. 9 — Impurities of Fe, MgO, Al_2O_3, V_2O_5, and TiO_2 in solution as a function of acid concentration for regular slag, −400 mesh, as compared to attrition ground slag at 200°C; 4 hrs

— — — Attrition

——— −400 mesh

Table 4 shows the results of gravity separation of the components of the product obtained by treatment of slag with 6 N HCl, while Table 5 is that for a treatment at 8 N HCl. It can be seen that the TiO_2 content of the separated products (rutile and unreacted slag) is not much different from the unseparated product. However, the rutile fraction is lower in iron, Al_2O_3, and MgO but higher in SiO_2 than the unreacted slag. Separation was not sharp due to the presence of fine particles of slag that contaminated the rutile (light fraction) and some rutile particles that remained sticking on the slag particles. However, it seems that SiO_2 was always associated with the rutile fraction because of the similarity in particle size. Table 6 shows more data on the chemical composition of the rutile fraction separated from products obtained at different acid concentrations. Table 7 shows the results of separating the products obtained from the attrition ground standard slag treated at 200°C with 6 N HCl for four hours.

Table 8 shows results of tests conducted using 10 N HCl and 10 N H_2SO_4 at 180°C for the same solid-liquid ratio. As mentioned earlier, it was not possible to determine quantitatively the amount of anatase formed during treatment with H_2SO_4. The data, however, show that HCl reacts faster than H_2SO_4 as can be judged from the high TiO_2 content in the residue.

Table 4 — Analysis of Products Obtained After Separation of Rutile from Unreacted Slag by Gravity*

	Composition, %			
	Original slag	**Unseparated product**	**Rutile fraction**	**Unreacted slag**
Weight, g	180.0	163.1	104.9	50.2
TiO_2	71.35	77.5	76.25	77.3
Fe (total)	9.2	4.99	3.5	7.0
Al_2O_3	5.42	4.0	3.45	4.5
SiO_2	5.26	5.80	(8.43)	3.68
CaO	1.12	0.68	0.71	0.51
MgO	5.24	—	2.65	4.82
MnO	0.26	—	0.15	0.21
V_2O_5	0.58	—	0.49	0.60
Cr_2O_3	0.18	—	0.18	0.49
TiO_2/SiO_2	13.56	13.36	9.06	21.01

* *Product obtained at 180°C, 4 hr, 6 N HCl*

Table 5 — Analysis of Product Obtained After Separation of Rutile from Unreacted Slag by Gravity*

	Composition, %			
	Original slag	**Unseparated product**	**Rutile fraction**	**Unreacted slag**
Weight, g	180.0	158.2	124.3	31.1
TiO_2	71.35	79.2	80.3	79.6
Fe (total)	9.2	2.4	2.5	5.3
Al_2O_3	5.42	2.9	2.65	3.97
SiO_2	5.26	6.87	7.5	4.15
CaO	1.12	0.66	0.63	0.5
MgO	5.24	—	1.75	3.97
MnO	0.26	—	0.11	0.18
V_2O_5	0.58	—	0.40	0.55
Cr_2O_3	0.18	—	0.19	0.19
TiO_2/SiO_2	13.56	11.53	10.71	19.18

* *Product obtained at 180°C, 4 hr, 8 N HCl*

Table 6 — Analysis of Products Obtained After Separation of Rutile from Unreacted Slag by Gravity*

	Original slag	**4 N HCl**	**6 N HCl**	**8 N HCl**	**10 N HCl**
Weight, g	180.0	11.6	112.9	141.3	147.1
TiO_2	71.35	79.8	81.1	83.75	85.5
Fe (total)	9.2	2.5	2.5	1.35	0.63
Al_2O_3	5.42	3.7	3.45	3.05	2.50
SiO_2	5.26	7.4	5.85	6.8	7.1
CaO	1.12	0.69	0.67	0.59	0.52
MgO	5.24	2.22	2.17	1.46	0.94
MnO	0.26	0.13	0.12	0.09	0.07
V_2O_5	0.58	0.60	0.64	0.57	0.40
Cr_2O_3	0.18	0.20	0.20	0.19	0.15
TiO_2/SiO_2	13.56	10.78	11.84	12.32	12.04

* *Composition of the rutile fraction.*
Product obtained at 200°C; 4 hr; 4, 5, 6, and 10 N HCl.

Conclusions

Titanium slag ground to −400 mesh was transformed at 180°C at an appreciable rate to synthetic rutile in 10 N HCl, but only to a negligible extent to synthetic anatase in 10 N H_2SO_4.

Titanium slag when melted and quenched in water and then ground to −400 mesh reacted in a similar manner to unquenched and ground slag.

Titanium slag ground in an attrition mill showed nearly the same reactivity toward HCl and H_2SO_4 under similar conditions of treatment.

Table 7 — Analysis of Products After Magnetic Separation of Rutile from Unreacted Regular Slag Attrition Ground*

	Composition, %		
	Original	Rutile fraction	Unreacted slag
Weight, g	120.0	71.6	28.1
TiO_2	71.35	83.2	75.75
Fe (total)	9.2	1.75	8.3
Al_2O_3	5.42	3.4	4.58
SiO_2	5.26	5.96	4.0
CaO	1.12	0.48	0.69
MgO	5.24	1.71	4.85
MnO	0.26	0.09	0.21
V_2O_5	0.58	0.63	0.63
Cr_2O_3	0.18	0.23	0.18
TiO_2/SiO_2	13.56	13.96	18.94

* *Product obtained at 200°C, 4 hr, 6 N HCl*

Table 8 — Comparison Between HCl and H_2SO_4 on the Transformation of Attrition Ground Slag Into Rutile and Anatase, Respectively*

Acid	Temp.,	Time,	Residue			Solution	
	C°	hr	weight, g	TiO_2, g	TiO_2, %	TiO_2, g	TiO_2, %
10 N HCl	180	1	9.6427	8.1552	84.57	0.4068	4.75
		2	9.5571	8.1783	85.57	0.3837	4.48
		3	9.4962	8.2194	86.50	0.3426	4.00
		4	9.4396	8.2751	87.66	0.2869	3.35
		5	9.3237	8.2685	88.68	0.2934	3.42
10 N H_2SO_4	180	1	9.9834	8.2686	82.82	0.2934	3.43
		2	9.9518	8.3338	83.74	0.2282	2.67
		3	9.9220	8.3338	83.99	0.2282	2.67
		4	9.8737	8.3552	84.62	0.2068	2.42
		5	9.8625	8.3911	85.08	0.171	2.00

* *Feed 12 g slag containing 71.35% TiO_2*

Attrition grinding is a very effective method in accelerating the rate of transformation of titanium slag into synthetic rutile or synthetic anatase.

Rutile can be separated from unreacted slag by gravity or magnetic methods. ■

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References

Ager, G.E., et al., 1974, "The Effects of Vibratory and Ball Milling on the Oxygen Pressure Leaching of Chalcopyrite," Conference of Metallurgists. abstract, *Bulletin*, Canadian Institute of Mining & Metallurgy, August.

Anon., 1973, "The Lurgi-Mitterberg Process: Pollution-Free Copper Recovery," *Bulletin*, Canadian Institute of Mining & Metallurgy, September.

Anon., 1980, "Lower-Energy Process for Copper Refining," *Chemical and Engineering News*, June 9.

Arkhipenko et al., 1980, "Model of the Mechanical Breakdown of Jarosite," *Izvestiya Akademii Nauk SSSR, Neorganische Materialy*, Vol. 16, No. 10, pp. 1837-1841.

Avvakumov, E.G., Samarin, O.J., and Kulebakin, V.G., 1981, "The Role of Mechanochemical Reactions in the Treatment of Sulfide Raw Material," *Isvestiya Sibirskogo Otdeleniya Akademii Nauk SSSR, Seriya Khimicheskikh Nauk*, Vol. 3, pp. 29-35; *Chemical Abstracts*, Vol. 95, 154419 s.

Beckstead, L.W., et al., 1976, "Acid Ferric Sulfate Leaching of Attritor-Ground Chalcopyrite Concentrates," *Extractive Metallurgy of Copper*, J.C. Yannopoulos and J.C. Agarwal, eds., AIME, New York.

Berger, A.S., et al., 1983, "Destruction of Mechanically Activated Spodumene and Phase Transformations of Activation Products Under Heating," *Isvestiya Sibirskogo Otdeleniya Akademii Nauk SSSR, Seriya Khimicheskikh Nauk*, Vol. 5, pp. 91-96; *Chemical Abstracts*, Vol. 99, 197325 h.

Chaikina, M.V., Tatarintseva, M.I., and Kolosov, A.S., 1983, "Mechanical Activation of Apatite," *Isvestiya Sibirskogo Otdeleniya Akademii Nauk SSSR, Seriya Khimicheskikh Nauk*, Vol. 5, pp. 99-104; *Chemical Abstracts*, Vol. 99, 197327 k.

Davis, E.G., Collins, E.W., and Feld, I.L., 1973, "Large Scale Continuous Attrition Grinding of Coarse Kaolin," Report of Investigations 7771, US Bureau of Mines.

Davis, E.G., Hansen, J.P., and Sullivan, G.V., 1981, "Attrition Microgrinding," *Fine Particles Processing*, P. Somasundaran, ed., AIME, New York, pp. 74-95.

Feld, I.L., et al., 1960, "Paper-Coating Clay from Coarse Georgia Kaolins by a New Attrition Grinding Process," Report of Investigations 5697, US Bureau of Mines.

Gerlach, J.K., Gock, E.D., and Ghosh, S.K., "Activation Leaching of Chalcopyrite Concentrates with Dilute Sulfuric Acid," *International Symposium on Hydrometallurgy*, D.J.I. Evans and R.S. Shoemaker, eds., AIME, New York, pp. 403-416.

Gock, E., and Szantho, E.v., 1980, "Laugung von Rutil nach mechanischer Aktivierung in einer Schwingmuehle," *Erzmetal*, Vol. 23, pp. 165-168.

Grizina, K., 1983, "Studies on the Dissolution Behavior in Water of Vanadium Pentoxide Activated by Grinding with Special Regard to the Dissolution Kinetics," *Chemie-Ingenieur-Technik*, Vol. 55, No. 1, pp. 70-71; *Chemical Abstracts*, Vol. 98, 96444 v.

Hansen, J.P., Davis, E.G., and Sullivan, G.V., 1981, "Autogenous Attrition Microgrinding of Calcium Carbonate Minerals," Report of Investigations 8503, US Bureau of Mines.

Huhn, H.J., and Mueller, A., 1980, "Alteration of the Thermal Behavior of Kaolinite by Mechanical Activation," *Journal of Thermal Analysis*, Vol. 19, No. 3, pp. 485-491; *Chemical Abstracts*, Vol. 95, 119713 m.

Juhasz, A.Z., and Opoczky, L., 1982, *Mechanical Activation of Silicate by Fine Grinding* (in Russian), Akademiai Kiado, Budapest, Hungary, 243 pp.; *Chemical Abstracts*, Vol. 97, 165431 g.

Kolbanev, I.V., and Butyagin, P.Yu, 1982, "Mechanochemical Reactions of Silicon with Water," *Kinetika i Kataliz*, Vol. 23, No. 2, pp. 327-333; *Chemical Abstracts*, Vol. 97, 12527 t.

Kulebakin, V.G., and Khalitova, R.S., 1980, "Transformation of Marmatite During Mechanical Treatment and at High Temperature," Deposited Document VINITI 657-81 (in Russian), 12 pp.; *Chemical Abstracts*, Vol. 96, 220994 z.

Kulebakin, V.G., et al., 1981, "Autoclave Leaching of Activated Pyrrhotite Concentrate," *Fiziko-Tekhnicheskie Problemy Razrabotki Poleznykh Iskopaemykh*, Vol. 6, pp. 96-100; *Chemical Abstracts*, Vol. 96, 220957 q.

Lapteva, E.S., Yusupov, T.S., and Stolpovskaya, V.N., 1983, "Study of Mechanically Activated Celestite," *Isvestiya Sibirskogo Otdeleniya Akademii Nauk SSSR, Seriya Khimicheskikh Nauk*, Vol. 5, pp. 96-99; *Chemical Abstracts*, Vol. 99, p. 197326 j.

Lin, I.J., Nadiv, S., and Grodzian, D.J.M., 1975, "Changes in the State of Solids and Mechano-Chemical Reactions in Prolonged Comminution Processes," *Minerals Science & Engineering*, Vol. 7, No. 4, pp. 313-336.

Paudert, R., et al., 1967, "Soluble Cassiterite through Mechanical Processing," *Chemische Technik*, Vol. 19, No. 6, pp. 356-359.

Paudert R., et al., 1979, "Increase in the Reactivity of Apatitic Natural Phosphates by Mechanical Activation," *Zeitschrift fuer Anorganische und Allgemeine Chemie*, Vol. 452, pp. 141-150; *Chemical Abstracts*, Vol. 91, 63409 q.

Pawlek, F.E., "The Influence of Grain Size and Mineralogical Composition on the Leachability of Copper Concentrates," *Extractive Metallurgy of Copper*, J.C. Yannopoulos and J.C. Agarwal, eds., AIME, New York, pp. 690-705.

Stanczyk, M.H., and Feld, I.L., 1963, 1965, "Continuous Attrition Grinding of Coarse Kaolin," Reports of Investigations 6327, 6694, US Bureau of Mines.

Stanczyk, M.H., and Feld, I.L., 1968, "Investigation of Operating Variables in the Attrition Grinding Process," Report of Investigations 7168, US Bureau of Mines.

Stanczyk, M.H., and Feld, I.L., 1972, "Ultrafine Grinding of Several Industrial Minerals by the Attrition Grinding Process," Report of Investigations 7641, US Bureau of Mines.

Stanley, D.A., et al., 1973, "Production of Submicron Silicon Carbide Powders by Attrition Milling," *Proceedings*, 2nd International Conference on Fine Particles, Electrochemical Society, pp. 331-336.

Stanley, D.A., Sadler, L.Y., and Brooks, D.R., 1973a, "Size Reduction of Ceramic Powders by Attrition Milling," *Proceedings*, 1st International Conference on Particle Technology, IIT Research Institute, pp. 280-285.

Toromanoff, I., and Habashi, F., 1983, 1985, "Transformation of a Low-Grade Titanium Slag into Synthetic Rutile," *Proceedings*, Symposium on Extraction of Steel Alloying Metals, Technical University of Lulea, Sweden, pp. 229-251; *International Journal of Mineral Processing*, Vol. 15, pp. 65-81.

Toromanoff, I., and Habashi, F., 1984, "The Composition of a Titanium Slag from Sorel," *Journal of Less Common Metals*, Vol. 97, pp. 317-329.

Vorobeichik, A.I., et al., 1983, "Mechanical Activation of Rutile and Anatase Modifications of Titanium Dioxide and the Change of Their Reactivity," *Isvestiya Sibitskogo Otdeleniya Akademii Nauk SSSR, Seriya Khimicheskikh Nauk*, Vol. 5, pp. 119-125; *Chemical Abstracts*, Vol. 99, 197328 m.

Vorobeichik, A.I., Pryakhina, T.A., and Boldyrev, V.V., 1983a, "Physicochemical Studies of Properties of Mechanically Activated Natural and Synthetic Titanates," *Isvestiya Sibitskogo Otdeleniya Akademii Nauk SSSR, Seriya Kimicheskikh Nauk*, Vol. 5, pp. 125-130; *Chemical Abstracts*, Vol. 99, 197329 h.

Zelikman, A., et al., 1981, "Study of Mechanically Activated Scheelite Concentrates," *Tsvetnye Metally*, Vol. 12, pp. 80-82; *Chemical Abstracts*, Vol. 96, 220921 y.

Ziegler, G., 1981, "Structural Changes During Milling of Alumina Powders," *Keramische Zeitschrift*, Vol. 33, No. 10, pp. 602-605; *Chemical Abstracts*, Vol. 96, 108980 r.