METHOD OF MAKING SYNTHETIC MINERALS FOR POLISHED SURFACE STUDY*

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Introduction

In carrying on experimental work involving argentite and chalcocite it was found necessary to make synthetic silver sulphides because of the scarcity of even approximately pure argentite. It was highly desirable that the synthetic argentite be obtained in a form permitting study of the material on polished surface previous to experimental work. It occurred to the writer that ordinary chemically precipitated silver sulphide could be compressed into a massive substance resembling natural argentite. The method described below resulted from experiments in making argentite and it was then applied to several of the common soft sulphides. The desirability of checking etch tests on chemically pure material is evident and the method is presented here in the hope that others will find it useful. For experimental work on some of the rarer minerals it furnishes a source of material which would otherwise be unobtainable.

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Preparation of the Sulphides

The sulphides of lead, mercury, antimony and bismuth were precipitated with hydrogen sulphide from aqueous solutions of the chlorides, slightly acidified with HCl, at a temperature of about 90°C. The sulphides of silver and copper were precipitated from hot solutions of the sulphates, slightly acidified with sulphuric acid. Except in the case of silver sulphate the concentrations of the solutions used were such as to yield ten grams of sulphide from a liter of solution. With silver sulphate a greater dilution was necessary, owing to the low solubility of silver sulphate. The solutions were well stirred during precipitation and in all cases hydrogen sulphide was passed in until precipitation was complete. The precipitates were well washed, first by decantation, and then on the Büch-

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ner funnel used for filtering. Hot water containing a little hydrogen sulphide was used for washing the lead and copper sulphides; for the others hot water alone was used. The sulphides obtained in this manner were partially dried in lump form. Following this they were powdered in a mortar and dried at 105°C for 12 hours.

Apparatus

Pressure was applied by a hydraulic press manufactured by Fred S. Carver of New York, and capable of supplying pressure up to 16,000 pounds per square inch.

For a mold, a cylinder manufactured by the same firm was adapted as shown in Fig. 1. A smaller and somewhat lighter cylinder was also used but this failed to withstand the pressures without troublesome distortion. A cylinder with a smaller inside diameter would be preferable; about 3/4 inch would seem a desirable size.

For heating the cylinder while the pressure was being applied the outside was insulated with asbestos paper and wound with nichrome wire embedded in a mixture of alundum cement and asbestos. With suitable rheostats the desired temperature may be obtained. Very high temperatures could not be used without a specially designed mold because of the effect of high temperature and stress on steel.

In the case of minerals treated with pressure alone the compression was continued from two to six hours. When heat was used both heat and pressure were applied from one to two hours.

Synthetic Minerals Produced

The following synthetic minerals were produced: argentite, covellite, galena, bismuthinite, metacinnabarite, metastibnite, and stibnite. It is evident that the softer, more sectile minerals which lack pronounced cleavage give the best results at moderate pressures and temperatures. Thus, of the minerals noted above, argentite gave the best results with pressure alone. Galena, covellite, bismuthinite and stibnite gave poor results with only pressure but yielded very satisfactory material by combined heat and pressure.

Most of the synthetic minerals are very fine grained but grains of covellite up to four tenths of a millimeter were measured. The synthetic galena is fine grained like natural steel galena. Stibnite had a well defined granular structure just visible at a magnification of eighty diameters.

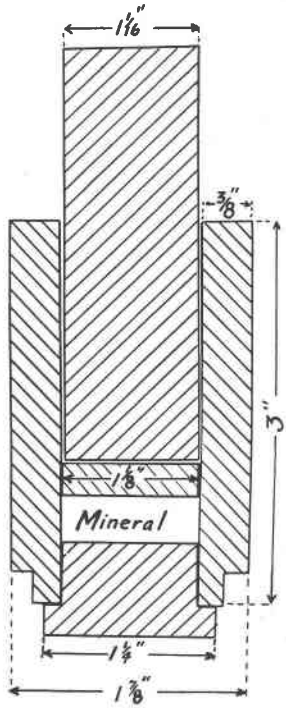

FIG. 1. Sketch of steel cylinder used for compressing chemically precipitated sulphides into solids. Two-thirds natural size.

ARGENTITE

Argentite produced the most successful synthetic mineral presumably because of its soft and sectile nature. Compression at 16,000 pounds produced a material which is remarkably like the natural material in general appearance. The briquet is strong and will stand rough handling and takes an excellent polish.

The etch reactions on the polished surface are compared below with those given by Short in United States Geological Survey, *Bulletin* **825**.

Light etch.[1] In four seconds the material forms the characteristic spots seen in natural argentite. On longer exposure, turns black and a brown to milky white sublimate is formed on the objective.

This synthetic argentite has been used in a great number of experiments and has proven a perfectly satisfactory substitute for the natural material.

[1] Stephens, Maynard M., Effect of light on polished surfaces of silver minerals: *Am. Mineral.*, vol. **16**, pp. 532–549, 1931.

Etch Tests on Argentite

Synthetic mineral	Natural Mineral (Short)
HNO_3, slight brownish fume reaction.	Fumes tarnish.
HCl, strong fume reaction.	Fumes tarnish; halo does not wash off.
KCN, quickly darkens, brings out fine structure.	Stains differentially black and brings out structure.
$FeCl_3$, quickly iridescent.	Quickly stains black.
Polarized light, isotropic.	Isotropic or anomalous anisotrophism.

Galena

Precipitated PbS under compression of 16,000 pounds per square inch forms a compact gray briquet which remains very soft and rubs off on handling, about like graphite. When heated to about 200°C under compression a briquet is formed which closely resembles steel galena. It takes an excellent polish and the color tests compare with Short's tests as follows:

Etch Tests on Galena

Synthetic mineral	Natural Mineral (Short)
HNO_3, effervescence; turns black.	Effervesces and turns black.
HCl, quickly turns dark; iridescent around edge.	Tarnishes iridescent.
KCN, Negative.	Negative.
$FeCl_3$, turns gray to iridescent.	Stains iridescent.
Polarized light, isotropic.	Isotropic.

Covellite

Chemically precipitated CuS is a soft, dirty green powder which does not much resemble natural covellite. Compression of this material at 16,000 pounds per square inch for several hours yielded a rather brittle briquet which has the typical covellite blue on the outside, but on the inside remains somewhat greenish. Application of compression and heat (16,000 pounds per square inch and temperature of 200°C) to this material results in a much more compact cake which is blue throughout and resembles natural cavellite in all external appearances. The etch tests compare with Short's tests as follows:

ETCH TESTS ON COVELLITE

Synthetic mineral	Natural mineral (Short)
HNO_3, negative, effervescence in pores.	Negative.
HCl, negative.	Negative.
KCN, quickly dark brown to black.	Stains black.
$FeCl_3$, negative.	Negative.
Polarized light, strong anisotrophism.	Strong anisotrophism.

STIBNITE AND METASTIBNITE

Sb_2S_3 when precipitated with hydrogen sulphide from an aqueous solution of antimony chloride has an orange red color and the corresponding natural mineral, which is very rare, is known as metastibnite. This material when compressed at 16,000 pounds pressure assumes a dull red color, is very porous and scales to such an extent when moistened that it cannot be polished. When metastibnite in powdered form is compressed by 16,000 pounds pressure and heated to about 200°C at the same time, it turns black and is compressed into a dense metallic material which is somewhat brittle. This takes an excellent polish, has low porosity with some peculiar curved cracks, evidently due to expansion when the pressure is released. The material is obviously stibnite and compares with Short's tests as follows:

ETCH TESTS ON STIBNITE

Synthetic mineral	Natural mineral (Short)
HNO_3, quickly dark to iridescent around edge; very slow effervescence.	Stains differentially iridescent, some varieties effervesce slowly.
HCl, slight fume reaction.	Fumes tarnish; some varieties negative.
KCN, slight gray tarnish.	Brings out scratches and sometimes stains slightly.
$FeCl_3$, negative; if left long time iridescent fume reaction.	Negative.
KOH, instantly brown and heavy yellow coat.	Instantly brown and gives yellow coating.
Polarized light, strong anisotrophism.	Very strong anisotrophism.

Metacinnabarite

When HgS is precipitated with hydrogen sulphide from an aqueous solution of the chloride a black amorphous material is formed. This material, when compressed at 16,000 pounds pressure, forms a compact, heavy, but brittle cake. This material corresponds exactly with the properties of meta-cinnabarite. If the precipitated material is subjected to the same pressure but at a temperature of 200°C, a material with a high metallic luster is produced which is somewhat harder than that formed by pressure alone.

This compares with Short's tests for meta-cinnabarite as follows:

Etch Tests on Metacinnabarite

Synthetic mineral	Natural mineral (Short)
HNO_3, negative.	Negative.
HCl, negative.	Negative.
KCN, negative.	Negative.
$FeCl_3$, negative.	Negative.
Aqua regia, effervesces and turns black.	Effervesces and turns black.

The work of Allen and Crenshaw[2] indicates that cinnabar could be produced from metacinnabarite, but the temperatures required (400° to 500°C) are considerably higher than were used in the experiments reported here.

Bismuthinite

Bismuth sulphide when precipitated with hydrogen sulphide from an aqueous solution of the chloride is a black amorphous material, which on drying and grinding in a motar forms a granular powder. When compressed at 16,000 pounds for several hours the material forms a rather hard, but very porous briquet. Because of the porosity this material does not take a very good polish. The material when heated to 200°C, while under pressure, is very much less porous and more crystalline in appearance, and the polish is correspondingly better.

The etch tests are identical for both types and correspond with Short's tests as follows:

[2] Allen, E. T., and Crenshaw, J. L. The sulphides of zinc, cadmium and mercury; their crystalline forms and modifications: *Am. Jour. Sci.*, vol. **34**, 4th ser., p. 378, 1912.

Etch Tests on Bismuthinite

Synthetic mineral	Natural mineral (Short)
HNO_3, effervescence; quickly black.	Slow effervescence and stains black.
HCl, fume reaction and drop turns slight gray with suggestion of structure.	Fumes tarnish.
HCN, negative.	Negative.
$FeCl_3$, negative.	Negative.
$HgCl_2$, slight brown.	Stains light brown.
Slightly anisotropic	Strong anisotrophism.

Conclusion

The results described indicate that many synthetic minerals may be made by the method described, or by slight modifications. This method provides a source of chemically pure compounds which may be studied on polished surface and thus slight changes induced by natural or added impurities may be detected. The same method may be used to make intimate mixtures of two minerals as may often be desirable in experimental work. Possibly the cause of many variations in etch reactions may be discovered by work on material which is known to be chemically pure.