

UNITED STATES PATENT OFFICE.

HENRY SPENCER BLACKMORE, OF MOUNT VERNON, NEW YORK, ASSIGNOR
TO THE PURE ALUMINIUM AND CHEMICAL COMPANY, OF WEST VIRGINIA.

PROCESS OF PRODUCING ALUMINIUM SULFID AND REDUCING SAME TO METALLIC STATE.

SPECIFICATION forming part of Letters Patent No. 605,380, dated June 7, 1898.

Application filed July 22, 1896. Renewed April 21, 1898. Serial No. 678,434. (No specimens.)

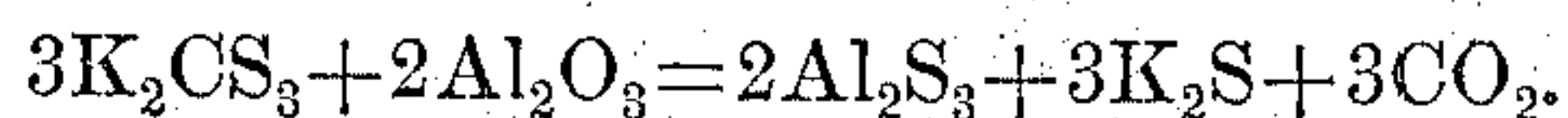
To all whom it may concern:

Be it known that I, HENRY SPENCER BLACKMORE, a citizen of the United States, and a resident of the city of Mount Vernon, in the county of Westchester and State of New York, have invented a new and useful Improvement in Processes of Producing Aluminium Sulfid and Reduction of the Same to Metallic State, of which the following is a specification.

The object of my invention is to convert aluminium oxid into sulfid and reduce the sulfid to metallic aluminium, with simultaneous production of alkali thiocarbonates by electrolysis, in a rapid and efficient manner at a reduced cost as compared with the present electrolytic processes; and it consists in transforming the alumina into aluminium sulfid by chemical action of alkali thiocarbonates and final reduction of sulfid to metallic state, together with the simultaneous production of alkali thiocarbonate by electrolytic action, using carbon anodes.

In carrying out my invention on a practical basis I prefer to employ a molten bath of sodium and potassium sulfids in about equal portions, by weight, on account of their low melting-point when mixed. In this bath I either introduce or produce thiocarbonates (sulfocarbonates) of the alkalis named, which becomes a part of the molten bath. The thiocarbonate may be produced directly in the bath by action of carbon bisulfid either introduced from a foreign source or produced electrolytically in the bath by employing carbon anodes. In this molten bath of alkali sulfid containing alkali thiocarbonate I introduce alumina in pulverulent form. This alumina not being soluble in the bath remains suspended by circulation of the bath, and as it becomes heated reacts with the thiocarbonate, producing aluminium sulfid, which assumes a molten condition and then becomes a part of the bath, at the same time eliminating carbonic anhydrid, the excess of alumina remaining in the bath to be converted into aluminium sulfid by action of new alkali thiocarbonate formed or produced during electrolysis, as herein set forth. The reaction which takes place may be illustrated

by the following chemical formula or equation:

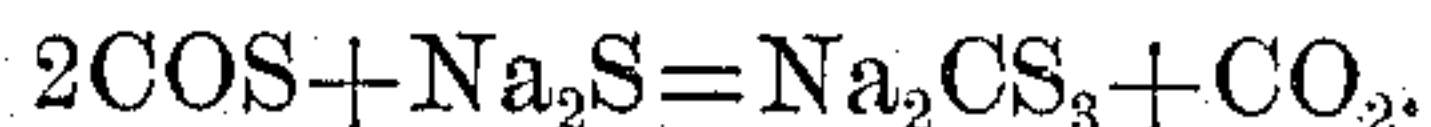


This represents the transformation under most favorable circumstances. It is obvious, however, that under certain circumstances more or less carbonyl sulfid (COS) will be produced and a more complicated reaction take place, producing, however, the same result, which may be illustrated by the following chemical formula or equation:

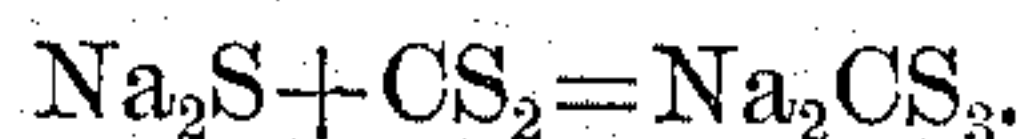
Production of aluminium sulfid:



Decomposition of carbonyl sulfid and re-production of thiocarbonates:



These latter complicated reactions take place when the temperature is not high enough for complete reaction and the thiocarbonate is largely in excess. By means of electrolytic action, employing carbon anodes, this process may be conducted continuously, the thiocarbonate being produced in the bath, as the sulfur liberated at the anode combines with the same, producing carbon bisulfid, which is almost immediately absorbed by the alkali sulfid present in the bath, producing alkali thiocarbonate, which reacts chemically on the suspended alumina, converting it into aluminium sulfid, as before described. The combination which takes place may be illustrated by the following chemical formula or equation:



It is therefore seen that the product produced is a definite chemical compound known as "sodium thiocarbonate." This is the product which transforms the aluminium oxid into sulfid during the process of electrolysis and not carbon bisulfid in a gaseous or vaporous state. Simultaneously with the production of thiocarbonates by the double reaction at the anode during electrolytic action metallic aluminium is liberated at the cathode.

I am aware that alumina has been dissolved in molten bath of fluorids and dis-

sociated by electrolysis direct; but in that case the resistance of the bath and dissolved alumina (aluminium oxid) considerably exceeds that required to reduce the sulfid of aluminium electrolytically.

In my process alumina (aluminium oxid) is not reduced or dissociated by electrolysis, as it is not soluble in the bath until transformed into sulfid, and consequently in its solid state is not in condition to admit of being dissociated by electrolysis. Hence no action toward the separation of aluminium takes place until after the alumina is chemically transformed into aluminium sulfid by action of the thiocarbonate or absorbed or occluded carbon bisulfid in the bath. The wide difference between these processes can therefore be readily seen.

I am also aware that processes have been devised for subjecting aluminium sulfid to electrolytic action in a molten bath of alkali chlorids and fluorids; but in that case no reaction took place producing alkali thiocarbonate, and thus the negative products of decomposition distilled off, such as free sulfur or carbon bisulfid, without change at the high temperature employed, and no aluminium sulfid was simultaneously produced to recuperate the bath as the aluminium was separated; also, aluminium oxid was not suspended in the bath for the purpose of simultaneously transforming the same into aluminium sulfid during electrolytical action on the aluminium sulfid.

Having now described my process, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of producing aluminium sulfid and reduction of the same to metallic state which consists in simultaneously converting insoluble aluminium oxid into soluble aluminium sulfid while in a molten bath and dissociating the soluble aluminium sulfid by electrolytical action substantially as described.

2. The process of producing aluminium sulfid and reducing the same to metallic state which consists in dissolving aluminium sulfid in a molten bath not capable of dissolving aluminium oxid, and mixing aluminium oxid in the same, then subjecting the mass to electrolytic action whereby the aluminium sulfid is dissociated, metallic aluminium being produced and insoluble aluminium oxid converted into soluble aluminium sulfid by secondary reaction substantially as described.

3. The process of producing aluminium sulfid and reducing the same to metallic state which consists in exposing aluminium oxid to the action of a molten bath of alkali salts capable of dissolving aluminium sulfid, having dissolved therein sulfids-of-alkali bases containing the combined elements of carbon bisulfid and subjecting the same to electrolytic action substantially as described.

4. The process of producing aluminium sulfid and reduction of the same to metallic

state which consists in transforming aluminium oxid into aluminium sulfid by action of thiocarbonates-of-alkali bases, then dissociating the sulfid by electrolysis simultaneously producing metallic aluminium at the cathode and thiocarbonate-of-alkali bases at the anode by secondary reaction, and then adding more aluminium oxid and continuing the electrolysis substantially as described.

5. The process of producing aluminium sulfid and reduction of the same to metallic state which consists in supplying aluminium oxid to a molten bath of sulfid-of-alkali bases during process of electrolysis while employing carbon anodes substantially as described.

6. The process of producing aluminium sulfid and reduction of the same to metallic state which consists in introducing aluminium oxid into a molten bath of sulfid-of-alkali bases containing the combined elements of carbon bisulfid, and electrolyzing the aluminium sulfid produced, thus producing metallic aluminium and regenerating the bath with the combined elements of carbon bisulfid substantially as described.

7. The process of producing aluminium sulfid and reduction of the same to metallic state which consists in simultaneously converting aluminium oxid into sulfid by chemical action while suspended in a molten bath of alkali salts, containing sulfid-of-alkali bases and combined elements of carbon bisulfid, and dissociating the sulfid produced by electrolytic action employing carbon anodes substantially as described.

8. The process of producing aluminium sulfid and reduction of the same to metallic state which consists in producing aluminium sulfid in a molten bath of sulfid-of-alkali bases during electrolytic action by adding insoluble aluminium oxid to said bath substantially as described.

9. In the process of producing aluminium sulfid and reduction of the same to metallic state the process for producing aluminium sulfid which consists in transforming aluminium oxid into aluminium sulfid by chemical action of thiocarbonate-of-alkali bases and reduction of aluminium and reproduction of thiocarbonate-of-alkali bases by electrolysis of aluminium sulfid substantially as described.

10. In the process of producing aluminium sulfid and reduction of the same to metallic state, the art of producing aluminium sulfid which consists in introducing aluminium oxid into a molten bath of sulfid-of-alkali bases containing thiocarbonates, and separating the aluminium from the sulfid produced, by electrolysis substantially as described.

11. In the process of producing aluminium sulfid and reduction of the same to metallic state, the process of transforming aluminium oxid into sulfid by chemical action of thiocarbonates, (or sulfid-of-alkali bases containing combined carbon bisulfid) and finally eliminating the metallic aluminium from the

product by process of electrolysis substantially as described.

12. In the process of producing aluminium sulfid and reducing the same to metallic state
5 the process of subjecting a molten bath containing dissolved aluminium sulfid to electrolytic action at a temperature sufficient to produce carbon bisulfid at the anode while in a molten bath capable of taking up the combined elements of carbon bisulfid so produced
10 at the temperature employed and suspending aluminium oxid in said bath said bath being maintained at a temperature sufficient to permit reaction substantially as described.

13. In the process of producing aluminium sulfid and reduction of same to metallic state,
15 the art of producing thiocarbonate-of-alkali bases which consists in dissociating aluminium sulfid by electrolysis while dissolved in a molten bath of sulfid-of-alkali bases at a temperature sufficient to produce carbon bisulfid
20 but not so high as to prevent the absorption

of the same by the sulfid-of-alkali bases substantially as described.

14. The herein-described process of converting metallic oxid into sulfid and effecting
25 final elimination of metallic base which consists in introducing said oxid into a molten bath containing thiocarbonate-of-alkali bases and subjecting the product to electrolytic action substantially as described.
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15. The process of producing aluminium sulfid herein described which consists in exposing aluminium oxid to the action of thiocarbonate-of-alkali bases in a heated state.
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In testimony that I claim the foregoing as my invention I have signed my name, in presence of two witnesses, this 8th day of July, 1896.

HENRY SPENCER BLACKMORE. [L. s.]

Witnesses:

ISAAC BLACKMORE,
CHARLES F. IRWIN.