

UNITED STATES PATENT OFFICE.

GUILLAUME DE CHALMOT, OF HOLCOMB ROCK, VIRGINIA.

METHOD OF OBTAINING FREE AMORPHOUS SILICON.

SPECIFICATION forming part of Letters Patent No. 602,632, dated April 19, 1898.

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To all whom it may concern:

Be it known that I, GUILLAUME DE CHALMOT, a citizen of the United States, residing at Holcomb Rock, county of Bedford, and State of Virginia, have invented a new and useful Improvement in Methods of Obtaining Free Amorphous Silicon, of which the following is a specification.

In the process heretofore practiced by me of reducing silica or silicates in the presence of copper I have obtained crystalline alloys which consist of free copper, free silicon, and copper silicid, represented by the formula Cu_2Si . By treating these crystalline alloys with acids a large proportion or all of the copper is dissolved, while the silicon that is combined with the copper is oxidized and forms silica. The crystalline silicon remains unaltered by this treatment, and it can be purified of the silica by dissolving the same in hydrofluoric acid. In this method of obtaining free silicon a part of the silicon that had been reduced from the silica could not be obtained in a free condition, and to this extent the former method was defective. This portion was that united to the copper and oxidized under the treatment with acids.

A copper-silicon alloy of average quality was found by chemical analysis to contain: copper, 70.05 per cent.; iron, 0.87 per cent.; free silicon, 19.21 per cent.; combined silicon, 10.10 per cent.

The object of my present invention is to reclaim as large a portion as possible of the 10.10 per cent. of combined silicon and convert the same into free silicon, as it is apparent that heretofore more than one-third of the total percentage of silicon in the alloy was lost in the operation.

In carrying out the present process the alloy is finely ground and mixed with sulfur, the quantity of sulfur being determined by the percentage composition of the alloy. This finely-ground mixture is heated in a closed vessel to a temperature which preferably ranges between 250° and 300° centigrade. Under this heat the copper unites with the sulfur and the larger part of the silicon of the silicid is made free in the form of an amorphous brownish-black powder.

If there is not enough sulfur employed, some of the copper will remain in its union

with the silicon, and if there is too much sulfur some of it will unite with the silicon, especially at places where the sulfur has run together into drops.

I have found that silicon is liberated if the amount of sulfur employed is just sufficient to form with all the copper the compound Cu_2S , and that it is also liberated if twice as much sulfur is used, so that the compound CuS is formed; but in the latter case more of the silicon will combine with sulfur. I have also found that the higher the temperature is raised the more the silicon will combine with sulfur, but that at the temperatures named the reaction obtained is the most desirable.

Operating with a high-grade silicon alloy I obtained the following results: Before treatment: free silicon, 19.21 per cent.; combined silicon, 10.10 per cent. After treatment with sulfur: 25.77 per cent. of original alloy and 3.54 per cent. of original alloy.

The following formula shows the result of treating a low-grade silicid with just enough sulfur to obtain the compound Cu_2S : Before treatment: free silicon, 1.22 per cent.; combined silicon, 11.74 per cent. After treatment with sulfur: 7.84 per cent. of original alloy and 5.12 per cent. of original alloy.

The following formula shows the result of treating a low-grade silicid with enough sulfur to produce the compound CuS : Before treatment: free silicon, 1.22 per cent.; combined silicon, 11.74 per cent. After treatment with sulfur: 7.27 per cent. of original alloy and 5.69 per cent. of original alloy.

The mixture of sulfids and free silicon is treated with acids. I prefer boiling with diluted nitric acid. By these means all the copper is dissolved and most of the sulfur of the copper sulfid liberated. The silicon sulfid that may be present is decomposed at the same time, yielding mainly silica and hydrogen sulfid. The copper salts are washed out and the residue is dried and extracted with carbon disulfid, which removes the sulfur. The remainder is treated with hydrofluoric acid, that removes all the silica. These operations may be repeated. The product in most cases is a mixture of crystalline and of amorphous silicon, for the crystalline silicon of the original alloy passes unaltered through all the operations.

I claim as my invention—

1. In the process of reducing silica or silicates in the presence of copper, the method herein specified of reclaiming the silicon in a free condition, consisting in finely pulverizing the silicon-copper alloy and mixing the same with sulfur, heating the same in a closed vessel to a temperature preferably between 250° and 300° centigrade, in which the copper unites with the sulfur and the silicon of the silicid is set free as an amorphous powder, substantially as set forth.

2. In the process of reducing silica or silicates in the presence of copper, the method herein specified of reclaiming the silicon in a free condition consisting in finely pulverizing the silicon-copper alloy and mixing the same with sulfur, heating the same in a closed vessel to a temperature preferably between 250° and 300° centigrade, in which the copper unites with the sulfur and the silicon of the silicid is set free as an amorphous powder, the amount of sulfur being regulated so as to obtain the compound Cu_2S or the compound CuS , substantially as set forth.

3. In the process of reducing silica or silicates in the presence of copper, the method herein specified of reclaiming the silicon in a free condition, consisting in finely pulverizing the silicon-copper alloy and mixing the same with sulfur, heating the same in a closed vessel to a temperature preferably between 250° and 300° centigrade, in which the copper unites with the sulfur and the silicon of the silicid is set free as an amorphous powder, treating the mixture of sulfids and free silicon with acids to dissolve the copper and liberate the sulfur, decomposing the sulfids, washing out the copper salts, drying the residue and extracting the sulfur by carbon disulfid and treating the remainder with hydrofluoric acid to remove the silica, substantially as set forth.

Signed by me this 15th day of September, 1897.

G. DE CHALMOT.

Witnesses:

GEO. T. LANCASTER,
JOHN C. TEMPLE.