UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING SILICON NITRID.

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To all whom it may concern:

Be it known that I, ALF SINDING-LARSEN, a subject of the King of Norway, residing at Christiania, Norway, have invented certain new and useful Improvements in Processes of Manufacturing Silicon Nitrid; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

The present invention has for its object a process of manufacturing silicon nitrid, according to which this latter product is obtained in a finely divided state at the same time as a complete transformation of the silicon into nitrid is secured.

As is well known pure silicon at high temperatures very easily combine with nitrogen, but it is very difficult to obtain a quantitative transformation into nitrid, as only the surface of the silicon will be acted upon owing to the fact, that the nitrid already formed to a certain extent protects the subjacent part of the silicon against further action. If the silicon however is dissolved in a metal or the like, with which it forms an alloy or compound, which is fluid at the temperature, at which the reaction takes place between silicon and nitrogen, for instance silico-iron, aluminium silicid or another silicid, and this alloy or compound at a suitable temperature is treated with a current of nitrogen, the entire amount of silicon will be transformed into nitrid, which is produced in such a finely divided state, that it is capable of being directly employed as a manure, or if desired it could be employed for the manufacture of oxids of nitrogen and through this latter into other nitrogen compounds, while the silica obtained by the oxidation process can again be worked on nitrid. By this process a silicid of a metal could also be freed from its content of silicon, so that for instance from aluminium silicid such as obtained by totally reducing aluminium silicates, in addition to the valuable nitrid of silicon could be obtained metallic aluminium.

This process, which may be carried out in an electric induction- or resistance-furnace of such a construction as to allow of the supply and pre-heating of the current of nitrogen and also if desired of employing pressure, may be conducted in such a manner that the other component of the silicid for instance aluminium is distilled over into a suitable vessel.

The following is an example of how the process may be carried out. An alloy of aluminium and silicon, containing equal parts of each component and which may have been produced by totally reducing an aluminium silicate, is heated in a converter shaped furnace lined with silicon nitrid and kept at a temperature of about 2000° centigrade. Through the fused alloy or melt is then drawn a current of nitrogen, which keeps the bath violently stirred at the same time as the silicon content of the bath gradually combines with the nitrogen forming silicon nitrid, for instance of the formula Si_3N_4. When by this treatment the bath after some time has assumed a pulverulent or pasty consistency on account of the silicon nitrid formed, the temperature is lowered to about 900° centigrade and a mixture of chlorids of sodium and potassium is added as a flux, into which the aluminium sinks to the bottom and melts together while the nitrid remains suspended in the slag or flux. If it is found that the aluminium still contains silicon and it is desired to obtain it in a pure state the above described treatment is repeated. The silicon nitrid is then separated from the flux, in which it is suspended, either by lixiviating the chlorids and then filtering the obtained solution or by volatilizing the chlorids. The fluxing materials may of course be recovered and repeatedly employed. If the aluminium silicate employed in producing the aluminium silicon alloy is not quite pure or if the nitrogen treatment is not carried very far, the resulting aluminium will contain a quantity of iron and silicon as impurities. To be obtained in quite a pure state the aluminium may in this case be subjected to a distilling process (optionally a fractional distillation process) at a temperature of about 2500° centigrade, vacuum being then preferably employed. This distillation process is then carried out in immediate connection with the above described nitrogen treatment in order to obtain the greatest possible heat economy.

Most other silicids may be treated in the same manner as described above only that the temperature will vary in each case.

I claim:

1. The process of manufacturing silicon nitrid which comprises heating a compound

or alloy of a metal and silicon to a temperature sufficient to react on nitrogen and introducing a current of nitrogen in contact therewith, said compound being liquid at the temperature at which the reaction takes place.

2. The process of manufacturing silicon nitrid which comprises heating a compound or alloy of a metal and silicon that becomes liquid at the reacting temperature of nitrogen and silicon, and conducting nitrogen in contact with said liquid compound or alloy and evaporating the metal.

3. The process of manufacturing silicon nitrid which comprises conducting heated nitrogen into contact with a melt of a compound or alloy of a metal and silicon.

4. The process of manufacturing silicon nitrid which comprises conducting nitrogen into contact with a melt of aluminium silicid and distilling the aluminium therefrom.

5. The process of manufacturing silicon nitrid which comprises conducting heated nitrogen into contact with a melt of aluminium silicid and distilling the aluminium therefrom.

In testimony that I claim the foregoing as my invention, I have signed my name in presence of two subscribing witnesses.

ALF SINDING-LARSEN.

Witnesses:
HENRY BORDEWICH,
M. ALGER.