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# PROCESS FOR THE PRODUCTION OF PURE ALUMINUM

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13 Claims. (Cl. 75—68)

This invention relates to a process for the production of pure aluminium.

For the production of particularly pure aluminium, the processes hitherto proposed have been mainly electrolytic processes, such as so-called three-layer processes with an electrolyte which consists substantially of cryolite, or more recently a process using electrolytes consisting of certain molten or dissolved complexes of organic aluminium compounds. The first process has a very high current consumption of about 20 kwh. per kg. of aluminium. It is true that the current consumption with the second process is substantially lower, but the deposition cannot be effected with any current density substantially higher than 1 amp./dm.$^2$, as a result of which the plant to be installed assumes a relatively large size per kg. of aluminium per hour. In addition, the electrolytes used in this latter process are sensitive to air, and therefore the removal of the cathodes covered with pure aluminium from the electrolyte baths presents certain difficulties when this electrolytic process is used for refining aluminium.

The present invention provides a process for the production of pure aluminium, which comprises decomposing an aluminium compound of the general formula $(RR'CH—CH_2)_2AlX$, in which R and R' represent the same or different saturated aliphatic radicals, and X represents either the group $RR'CH—CH_2—$ or a hydrogen atom, under the action of heat to form pure aluminium, olefines and hydrogen.

The form of the process of the invention which is most favourable from an economic point of view is that in which R and R' both represent $CH_3$ groups, i. e. when the aluminium compound decomposed is aluminium triisobutyl. If aluminium triisobutyl is heated to a temperature between 200 and 300° C., it is decomposed in accordance with the equation:

$$(C_4H_9)_3Al=3C_4H_8+1\frac{1}{2}H_2+Al$$

The metallic aluminium in this case is deposited in a very pure form. The aluminium triisobutyl can be decomposed in a single stage, but it is frequently more advantageous to carry out the decomposition in two stages. In this case, one mol of isobutylene is initially split off at about 150° C. with formation of diisobutyl aluminium hydride $(C_4H_9)_2AlH$ and then the diisobutyl aluminium hydride splits up at higher temperatures according to the equation $(C_4H_9)_2AlH=2C_4H_8+1\frac{1}{2}H_2+Al$.

Depending on the manner in which the thermal decomposition is carried out, the aluminium can be deposited in very different forms. When the said aluminium compounds are simply heated in a vessel, it is mainly a pulverous aluminium which is obtained, and this aluminium has subsequently to be melted to convert it into a compact form. The aluminium also frequently separates out at heated surfaces in the form of films and, by carrying out the reaction in a suitable manner, it is very readily possible to achieve the result that a thin metal film formed in this manner progressively increases in thickness and finally can be stripped from the surface



in the form of a compact aluminium sheet of appreciable thickness. Such a deposition in the form of aluminium films takes place mainly when the heated surfaces are brought into contact with the vapours of the said aluminium compounds, and, since the liquids have a relatively high boiling point, it is preferable to work in vacuo or even with hydrogen as a carrier gas. Metal films are, however, also formed if the heated surfaces, especially metal surfaces, are brought into contact with liquid aluminium compounds of this nature, especially with liquid diisobutyl aluminium hydride, and the temperature is kept at the lower limit of the temperature range at which decomposition takes place with deposition of aluminium. Depending on the nature of the surface on which the aluminium is deposited, the limit varies between about 200 and 300° C., and can in each case be determined very easily by a few experiments.

When pure aluminium is to be produced on a relatively large scale, the thermal decomposition of the said aluminium compounds is only the final stage of a process. In combination with the production of aluminium triisobutyl from isobutylene, aluminium and hydrogen by the process of the Belgian specification No. 535,235, the process of the invention also renders possible a purely chemical refining aluminium with an ordinary degree of purity. According to this purely chemical refining process, crude aluminium is initially dissolved in a mixture of isobutylene and hydrogen to give a mixture of aluminium triisobutyl. The aluminium triisobutyl is separated by filtration or by vacuum distillation from those impurities of aluminium which remain undissolved and then subjected to the decomposition treatment. During this treatment, isobutylene and hydrogen are recovered, and can be returned to the first stage of the process. The entire cycle of the reactions is thus equivalent to the known method of manufacturing particularly pure so-called carbonyl iron, in which iron is initially reacted with carbon monoxide to give liquid iron carbonyl, and the latter is decomposed back into carbon monoxide and iron again under the action of heat. The part of the carbon monoxide in the refining of iron is, in the process of the present invention, played by the isobutylene, or other olefine used, together with the hydrogen.

The process of the invention can be carried out economically with the highest possible efficiency when isobutylene and hydrogen are used. It can readily be seen that the highest possible yield per unit of volume and time must be produced in the stage of dissolving the crude aluminium when isobutylene is used, since isobutylene is the olefine with the lowest possible molecular weight among the group of olefines which can be used. In addition, of all organic aluminium compounds which can be considered as suitable, aluminium triisobutyl, or the diisobutyl aluminium hydride which can be split off from the latter, have the highest aluminium content. The amount of substance flowing through the entire circulation system is consequently lowest when isobutylene is used for the refining operation. In addition, isobutylene is a very cheap olefine produced in very large amounts in the refining of petroleum.

It is of course possible for the process of the invention also to be carried out with other olefines, for example with 2-methyl-pentene-(1) and other analogously constructed unsymmetrically disubstituted ethylenes.

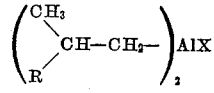
The amounts of substances constantly used up in the process of the invention are very small. Losses only occur during the dissolution of the aluminium with the isobutylene and hydrogen and during the preparation of the aluminium alkyl compounds, since here a certain proportion of the isobutylene is hydrogenated to form isobutane. Even this loss can be reduced to a minimum

if the isobutane which is formed as a side product is supplied to a separate small plant for catalytic dehydrogenation, where it is split up into isobutylene and hydrogen.

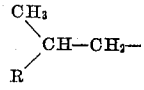
The temperature range in which decomposition of the aluminium triisobutyl or of the diisobutyl aluminium hydride takes place with deposition of aluminium has been stated above to be 200–300° C. It is nevertheless possible to use higher temperatures, if desired. The decomposition can also be readily carried out on a surface of molten aluminium, the vapours of the compounds being caused to sweep past such a surface. In this way, isobutylene and hydrogen are constantly removed, while the aluminium which is formed is readily precipitated in a liquid condition in the molten aluminium. The increased amount of aluminium can then be continuously extracted in a manner known per se from the bath of liquefied aluminium. All processes should be carried out in closed equipment since the substances formed in the course of the process of the invention ignite spontaneously and are readily combustible. However, operation in completely closed equipment in this manner does not present any particular difficulties. It is also quite common practice with many other modern chemical or metallurgical processes. The advantages of the process of the invention are obvious. It is not necessary to install any separate electrical equipment and the apparatus used can be quite small in relation to the amount of material converted.

What we claim is:

1. A process for the production of pure aluminum from crude aluminum containing impurities which comprises forming an aluminum compound of the general formula



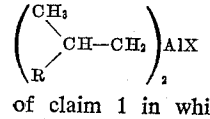
in which R represents a saturated aliphatic radical and X represents a substituent selected from the group consisting of the



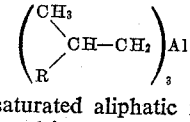
radical and a hydrogen atom from the crude aluminum, separating said compound from the impurities of the crude aluminum and then decomposing said compound under the action of heat to form pure aluminum, an olefine and hydrogen.

2. The process of claim 1 in which R represents a methyl radical.

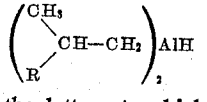
3. The process of claim 1 in which the olefine and hydrogen formed are reacted with further quantities of crude aluminum to form further quantities of the aluminum compound of the general formula



4. The process of claim 1 in which the aluminum compound is



in which R is a saturated aliphatic radical and the decomposition is effected in two stages by first heating said compound to form



and then heating the latter at a higher temperature to form pure aluminum.



5. The process of claim 1 in which said aluminum compound is contacted with a heated surface to effect decomposition of the aluminum compound with the formation of an aluminum film on said surface.

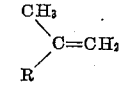
6. The process of claim 5 in which said heated surface is a heated metal surface.

7. The process of claim 5 in which said heated surface is that of molten aluminum.

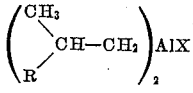
8. The process of claim 1 in which said aluminum compound is contacted with a heated surface to effect decomposition of the aluminum compound with the formation of a compact coating of aluminum on such heated surface and stripping the coating from said surface to form an aluminum sheet.

9. The process of claim 1 in which said aluminum compound is decomposed at a temperature between 200 and 300° C.

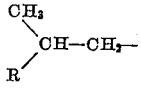
10. In a process for the production of pure aluminum the steps which comprise dissolving crude aluminum containing impurities to be refined in a mixture of an olefine of the formula



in which R is a saturated aliphatic radical and hydrogen to form an aluminum compound of the general formula



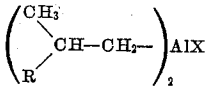
in which R is a saturated aliphatic radical and X is a substituent selected from the group consisting of the



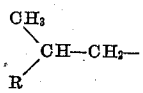
radical and a hydrogen atom, separating said compound from the impurities of the crude aluminum and then decomposing said aluminum compound to form pure aluminum, an olefine and hydrogen.

11. The process of claim 10 in which said olefine is isobutylene and said aluminum compound is aluminum triisobutyl.

12. A process for the production of pure aluminum from crude aluminum containing impurities which comprises forming an aluminum compound of the formula



in which R represents a saturated aliphatic radical and X represents a substituent selected from the group consisting of the



radical and a hydrogen atom from the crude aluminum separating said compound from the impurities of the crude aluminum and then flowing a vapor of said compound past a surface of molten aluminum to decompose such aluminum compound with the formation of aluminum, an olefine and hydrogen, continuously removing the hydrogen and olefine formed, and recovering the aluminum formed from the molten aluminum.

13. The process of claim 1 in which the decomposition takes place in closed apparatus.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