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RECOVERY FROM IRON ORE OF SECONDARY METAL

John L. Young, Pittsburgh, Pa., assignor, by mesne assignments, to Plastic Metals, Inc., a corporation of New York

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This invention relates to the recovery, from ores and oxides derived from industrial operations in which both iron and another metal are present, the two metals.

In a companion application for Letters Patent filed by me October 22, 1938, Serial No. 236,589, for an invention described in an earlier and forfeited application, filed March 13, 1937, Serial No. 130,715, I have described the preparation of an anode for an electrolytic cell. The anode may be prepared from granulated sponge iron. A body of such material is shaped and compressed, and in compressed condition is brought while in a reducing atmosphere to bright-annealing temperature. Under such conditions surface oxides are reduced, metallic bridges are formed between the grains, and the result is a porous anode of high conductivity throughout its mass. An anode of sponge iron is described also in Letters Patent 2,043,823 granted June 9, 1936, on my application.

In Letters Patent of the United States No. 2,102,249, the joint inventors, of whom I am one, have described the production of an anode by the compression of discrete bodies of iron coated with another metal, and by heating the compressed mass until the coating metal forms bridges between the constituent bodies. In this case, too, a porous anode is formed of high conductivity. In the electrolytic operation it was found that the iron of the anode of the last-named patent could be taken into solution by the electrolyte, while the coating metal (typically tin) would fall away and would collect with the slime in the bottom of the cell, and from that slime the coating metal manifestly could be recovered.

I now have discovered that, beginning with native ore (or, indeed, with oxide derived from other industrial operations) in which iron is found in association with another metal, and proceeding along lines pursued in such other connection, I may not only recover the iron in pure state, but I may effect a substantially clean cut, between the iron and the other metal present in the ore; and I may recover from the slime deposited in the electrolytic cell such other metal, in reduced or combined state according to circumstances. For instance, I may so recover in metallic state copper present with iron in the ore (or oxide), or, again, I may so recover titanium, in the state of oxide.

I have found it to be characteristic of my procedure that the degree of pressure may be empirically determined, and the degree of porosity of the anode correspondingly fixed, so that the iron will pass freely and fully into solution in the electrolyte, to be delivered thence as a cathode deposit, while the remaining constitutents of the substance of the anode will slough freely off and be deposited as slime in the bottom of the cell, and the electrolytic operation will go forward continuously and without choking.

Beginning with ore (or equivalent oxide) in finely divided or granular condition, I first bring it in a reducing atmosphere to iron-oxide reducing temperature, and in so doing I convert the iron oxide or substantial portions thereof to metallic iron. I thin, while maintaining the temperature and the reducing atmosphere, compress the granular material and form of it a coherent but still porous mass. Conveniently the mass is pressed upon a core of clean-surfaced metal, and the adjacent grains are thus brought into electric union with the core, while under the same pressure contiguous particles within the mass are brought into electric union one with another. The core serves, of course, as a pole piece in the assembly of the anode in an electrolytic cell. A core is not, however, necessary; the granular material may be compressed to a coherent anode body without it.

The so completed anode is allowed to cool and is then brought into the open air. It is assembled with a suitable cathode in an electrolytic cell, and an electrolyte, preferably a solution of ferrous and ammonium sulphate, is provided.

Current being then applied, the iron of the anode will progressively go into solution; a pure deposit of iron will progressively be laid on the cathode; the other constitutents of the anode body will progressively slough away and will collect in the slime at the bottom of the cell; and the anode will continuously present a fresh surface, uncoated by residual material, to the continued vigorous action of the electrolyte.

The slime collected from the bottom of the cell will contain the other components of the ore (or oxide) remaining after the iron-oxide reduction operation, such as sand and other material of stony nature, commonly called gangue, and it will also contain either in reduced or combined state such other metallic component or components as may have existed in the ore; as, for example copper (in reduced and metallic condition) or titanium (still unreduced and in the condition of oxide). Such metallic content of the slime may then by subsequent known procedure be recovered.

I have specified an electrolyte of ferrous and

ammonium sulphate. There are iron ores that will admit of the use of an electrolyte of a solution of iron chloride; but it is only in exceptional cases that an iron chloride bath will serve. Ordinarly, the use of iron chloride will result in the appearance at the cathode not of iron alone, but of other metal also.

I have said that the degree of compression must, for a particular ore, be empirically determined. If the compression be carried too far and the anode be too densely compacted, it will as electrolysis progresses become coated with refuse material, to the embarrassment of the electrolytic operation; if, on the other hand, it be insufficiently compressed, an excessive amount of iron will be carried into the slime.

I am aware that it has been proposed to use as an anode loose sponge iron held in a suitable container, but in such case as operation progresses the coating over with refuse material of the grains of sponge iron is an embarrassment that has not been successfully overcome. And no attempt has ever been made in such manner to effect separation from the iron of a secondary metal present in the anode material. I am aware that the bonding of sponge iron particles with iron sulphide to produce a coherent anode has been proposed, but in that case also the anode becomes fouled in a sulphate bath. Furthermore, undissolved sulphide gathers in the slime in quantities such as would become an embarrassment to recovery of any secondary metal therefrom. No attempt has heretofore been made in such manner to separate from the iron a secondary metal present in the anode material. If sponge iron be melted and cast or rolled, any secondary metal present would be so far alloyed with the iron as to be inseparable by such methods of electrolysis as here are contemplated. No attempt has heretofore been made to effect separation from the iron of a secondary metal present in an anode so formed.

I believe myself to be the first to discover the possibility of separating from the iron a secondary metallic component of an iron ore, or equivalent oxide, by granulating the ore, reducing its iron content to metallic state, consolidating the sponge to a coherent and porous body, and employing that body as an anode in an electrolytic cell, with the result that the iron goes to the cathode and the secondary metal passes with a free-sloughing residue to the slime at the bottom of the cell. I believe I am the first to find that the combination of a sulphate bath with a suitable sponge iron anode will give a pure cathode deposit and a second component, recoverable from the slime.

I claim as my invention:

1. The method herein described of separating two metallic components of an iron ore, which consists in bringing the ore in finely divided condition into a reducing atmosphere and there subjecting it to reducing temperature; then, when under such conditions the iron content has been brought to metallic state and while the conditions of an inert atmosphere and an elevated temperature are maintained, compressing the material to a coherent porous body within which the iron continues in free and pure metallic state; and, finally, conducting an electrolytic operation with such coherent porous body as the anode and with a solution of ferrous and ammonium sulphates as the electrolyte, in which operation the iron goes progressively into solution and is deposited on the cathode, while the other constituents of the anode body are progressively sloughed away and collect in the slime at the bottom of the cell.

2. The method herein described of recovering from an iron ore or equivalent oxide that contains also a secondary metallic content the two metals, separate each from the other, which consists in subjecting the raw material in finely divided condition to iron oxide reducing conditions and producing thereby within the mass pure metallic iron, compressing the material so treated while the conditions of an inert atmosphere and an elevated temperature are maintained to a coherent porous mass, conducting an electrolytic operation with such coherent porous mass serving as the anode, whereby the iron taken into solution in the electrolyte is deposited at the cathode and the secondary metal is sloughed off and carried with the residue to the slime in the bottom of the cell, and recovering from the slime the secondary metal.

JOHN L. YOUNG.