

UNITED STATES PATENT OFFICE.

CHARLES MORRIS JOHNSON, OF AVALON, PENNSYLVANIA, ASSIGNOR TO CRUCIBLE STEEL COMPANY OF AMERICA, OF PITTSBURG, PENNSYLVANIA, A CORPORATION OF NEW JERSEY.

MANUFACTURE OF STEEL.

964,869.

Specification of Letters Patent.

Patented July 19, 1910.

No Drawing.

Application filed November 15, 1909. Serial No. 528,156.

To all whom it may concern:

Be it known that I, CHARLES MORRIS JOHNSON, residing at Avalon, in the county of Allegheny and State of Pennsylvania, a citizen of the United States, have invented or discovered certain new and useful Improvements in the Manufacture of Steel, of which improvements the following is a specification.

My invention relates to improvements in the manufacture of steel, and more specifically in the manufacture of tool steel in which the iron is combined or alloyed with tungsten or like metal; and the object of my improvement is economy in manufacture.

Heretofore in the production of tungsten steel, it has been considered necessary to charge into the crucible with the other steel-making ingredients, tungsten in metallic state, practically free from impurity or adulteration, and heretofore the common practice has been to employ tungsten about 95 to 98% pure. The production of metallic tungsten of such purity is accomplished at a very considerable expense, which adds appreciably to the price of the resulting tungsten steel.

I have found that it is unnecessary to bring the tungsten ingredient of the crucible charge to the degree of purity which has heretofore been thought necessary, and I have found on the contrary that tungsten adulterated with other substances contained in the native rock, and retained after such refining operation as I have found to be sufficient, notably with silica, the material containing about 80% of tungsten, may be employed in the crucible charge, and tungsten steel of good marketable quality produced, the dross or adulteration charged with the tungsten slagging off during the melting of the crucible charge. To this end I take one of the well known commercial ores of tungsten, either wolframite, (MnFe)WO₄, or sheelite, CaWO₄, or iron tungsten ore, FeWO₄. I take these minerals in their concentrated form, that is the form in which they are known to the trade as "concentrates", and I charge a quantity of such ore into a retort, preferably a vitrified clay kettle, together with a chlorin-affording substance. This chlorin-affording substance will preferably be a relatively small quantity of an alkaline chlorate (chlorate of sodium or chlorate of potassium),

the ratio of chlorate to ore being about 1 to 100. To this charge I add hydrochloric acid, preferably in the commercial form known as muriatic acid, in the ratio of about 125 to 150 pounds of mineral to 111 pounds of 20° acid. While the ensuing reaction is going on I maintain the temperature of the kettle and its content at about 90° centigrade. This I preferably accomplish by surrounding the kettle with a wooden jacket, and supplying the intervening chamber with injected steam. I preferably also cover the kettle during the operation with a wooden cover. The desired reaction will be completed after an interval of from 10 to 18 hours, at the end of which period the liquid is decanted from the solid residue, and that residue is washed with water and dried. The washing is continued until the soluble constituents are removed. That is to say, in the case of wolframite, until the wash water contains but a slight trace of manganese, or in the case of sheelite, until the wash water contains but a small trace of calcium.

After the residual substance of the process thus far described has been dried, I submit it in the presence of a reducing agent to a temperature sufficient to effect the reduction of the oxid of the particular metal with which I may be dealing. That is to say, this residue from the acid reaction will contain the metal, tungsten for example, in the form of tungsten oxid; and the object of the reducing step of the process is to effect a reduction of that oxid to metallic tungsten. Accordingly, the residual substance of the acid reaction is brought to a temperature sufficient to effect the reduction of tungsten oxid, and in the presence of a reducing agent. This step of my operation will preferably be effected in a furnace and according to the method of operation specifically described in a co-pending application of mine, filed June 19, 1909, Ser. No. 503,112, for method of reducing metallic oxids. When this operation of reduction shall have been carried out, and the charge of the reducing furnace shall have been cooled down and withdrawn, that charge will consist of metallic tungsten in finely divided form, mechanically combined with dross or adulteration, which will to a large extent consist of silica, present in the concentrated ores with which the operation began. The

tungsten content of the resulting product will ordinarily be about 80% of the whole. The silica present is not part of the original ore, but is derived from the native rock
 5 mined with the ore, and from which complete separation has not been practically accomplished. If the particular ore treated be sheelite in commercial form, the silica content of the resulting product will be particularly great; if the ore treated be wolframite or iron tungsten ore, the dross will consist in part of silica, and there will be a very appreciable quantity of iron—in the case of the iron tungsten ore, as much as
 10 7%; if the ore be wolframite, there will be some small quantity of manganese in the dross. It will be understood that the iron, when present in the dross, will be brought to metallic state at the same time with the reduction of the tungsten, and that such iron content is clear gain in the crucible charge. This resulting product, containing about 80% tungsten, and 20% dross, of silica, etc., I have found (as hereinbefore stated) adequate as an ingredient of the crucible charge
 20 in making crucible steel, for the purpose of introducing into the finished metal the desired tungsten element.

The dross or adulteration, so far as it may
 30 consist of iron, will advantageously form part of the finished product; so far as it may consist of silica, it is slagged out in the melting operation; other impurities, so far as they may be present, are either refined
 35 out or are negligible.

It will thus be observed that the silicon present and forming an adulterant with the commercial ore, instead of being separated from the tungsten before the latter is
 40 charged into the crucible (which separation can be effected only at relatively great expense) is allowed to remain and to be

charged with the tungsten into the crucible, where it is removed as an incident to the steel-melting operation, and at relatively
 45 small expense. The eradication of silicon in this manner is possible, because it is present in the crucible charge in the form of the oxid, silica, mechanically mixed with the metallic tungsten, and in this form it is
 50 slagged out unchanged.

By the method herein described, practically all of the tungsten contained in the original ore is rendered available for the crucible charge; such is not the case if the
 55 preliminary process involves bringing the tungsten to approximately pure condition by any fusion process.

I have particularly referred in describing my process to the manufacture of tungsten
 60 steel, and to the preparation of minerals in which tungsten occurs. It will of course be understood that I do not limit myself to the specific ores named, nor to a process which involves the use of tungsten only; for my
 65 invention is applicable to steel-making processes in which the metal combined with the steel is another metal than tungsten though of like character.

I claim herein as my invention:

The herein described process of steel-making which consists in charging into the steel furnace, together with the other steel-making ingredients, tungsten or like metal
 75 from which the silica of the native rock has not been refined away, and slagging out the silica so introduced as the steel-melting operation progresses.

In testimony whereof, I have hereunto set my hand.

CHARLES MORRIS JOHNSON.

Witnesses:

BAYARD H. CHRISTY,
 ALICE A. TRILL.