UNITED STATES PATENT OFFICE.

JOHANNES SCHILLING, OF COLONIE GRUNEWALD, NEAR BERLIN, GERMANY.

PROCESS OF PRODUCING AN AMORPHOUS TUNGSTEN POWDER.

950,869. Specification of Letters Patent. Patented Mar. 1, 1910.

No Drawing. Application filed March 23, 1909. Serial No. 485,306.

To all whom it may concern:

Be it known that I, JOHANNES SCHILLING, a subject of the King of Prussia, and resident of 1 Winklerstrasse, Colonie Grunewald, near Berlin, German Empire, have invented a new and useful Process for Producing an Amorphous Tungsten Powder, of which the following is a specification.

The present invention relates to a process for the production of a finely divided amorphous powder of tungsten or compounds of tungsten which are poor or comparatively poor in oxygen. Such finely divided metallic powder is of special use in the manufacture of metallic filaments for incandescent electric lamps.

The present invention depends on the property of tungsten compounds to break up into small particles when brought to a high temperature in the presence of ammonia. When this process is carried out in the air or in any oxidizing medium the ordinary trioxid of tungsten (WO_3) is formed. If however the process is carried on in a non-oxidizing medium or atmosphere an exceedingly fine soft amorphous metallic powder is obtained as the result of the disruption on heating. This powder may be easily pressed and is specially suited for the manufacture of metallic filaments with any suitable binding agent in any known manner. With this fine powder the filaments may be manufactured without encountering the usual difficulties of clogging or damaging of the nozzle through which the material is forced when forming filaments. Further in consequence of the fine state of division of the powder it forms a much more intimate mixture with the binding agent and consequently produces a stronger filament which may be handled much more easily. The tungsten powder produced is either pure metallic tungsten or a mixture of the metallic tungsten with lower oxids of tungsten.

In carrying the invention into effect, the substance containing tungsten which is preferably employed, is one of the various meta- or para-tungstates of ammonium. The salt is heated in a closed tube or retort to a little above the temperature at which the disruption of the tungstate takes place. Simultaneously a current of hydrogen and nitrogen or ammonia gas may be led over the mass.

When tungsten trioxid (WO_3) is dissolved in ammonia water (NH_3) at a moderate temperature, a salt is obtained which is approximately

$$(NH_4)_{10},W_{12}O_{41}$$

or—

$$(NH_4)_6,W_7O_{24}$$

or—

$$(NH_4)_2,W_2O_7.$$

On heating this salt as described above while leading ammonia gas over it, the ammonia is driven out of the salt and at a sufficiently high temperature this ammonia is wholly or partly dissociated into hydrogen and nitrogen. The hydrogen in *statu nascendi* is exceedingly active as a reducing agent and reduces the oxid of tungsten from a higher oxid into a lower oxid or to the metallic tungsten. The amount of ammonia led over the heated mass while stirring is determined by the composition of the salt and the required degree of reduction of the tungsten oxids. It is also advisable to draw off the water of crystallization or the water formed by the reduction of the oxids of tungsten, and this may be done by leading a current of non-oxidizing gas, conveniently a mixture of nitrogen and hydrogen or dried ammonia gas over the mass. With care it is also possible to employ hydrogen gas for the purpose but the objection here is that a hard rough granular gray powder is obtained as discovered by Berzelius, and this substance is not suited for use in the formation of metallic filaments for lamps.

The powder produced according to the present invention may be further treated in any known manner as for instance in the manner described in my United States application Serial No. 349336 filed 24th. of December, 1906.

I claim:—

1. The process for the production of a fine amorphous tungsten powder, consisting in heating a tungsten compound adapted to be dissociated by heat in a non-oxidizing atmosphere to a temperature above the disruption temperature whereby the reducing agents separated from the said tungsten compound act upon the oxids of tungsten present.

2. The process for producing a fine amorphous tungsten powder, consisting in heating an ammonium salt of tungsten in a non-oxidizing atmosphere to a temperature slightly above the disruption temperature to

cause the ammonia gas separated out by the heating to be itself dissociated and thereby act as a reducing agent on the oxids of tungsten remaining in the mass.

3. The process for producing a fine amorphous tungsten powder, consisting in heating an ammonium salt of tungsten in a non-oxidizing atmosphere to a temperature slightly above the disruption temperature to cause the ammonia gas separated out by the heating to be itself dissociated and thereby act as a reducing agent on the oxids of tungsten remaining in the mass, passing a non-oxidizing gas over said heated mass to assist the reduction and draw off the moisture present, substantially as described.

4. In process of making metallic tungsten filaments for incandescent electric lamps the production of a fine amorphous tungsten powder consisting in heating a tungsten compound adapted to be dissociated by heat in a non-oxidizing atmosphere to a temperature slightly above the disruption temperature, separating thereby reducing agents from the dissociating compound and allowing same to act on the oxid of tungsten obtained by the dissociation whereby fine amorphous tungsten powder is obtained.

Signed at Berlin, Germany this 3rd day of March 1909.

JOHANNES SCHILLING.

Witnesses:

HENRY HASPER,

WOLDEMAR HAUPT.