

UNITED STATES PATENT OFFICE.

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PROCESS FOR THE MANUFACTURE OF DRAWN TUNGSTEN WIRES.

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(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

It is known that tungsten wire, immediately after the mechanical treatment by which it was produced, has an extremely long grained structure, which, on the application of intense heat, changes into a structure that is crystalline in the ordinary sense of the word. This transformation takes place at different speeds and in various ways. The "permanent structure" of the heavily annealed wire—if this term be employed to designate the particular structure of the wire which either remains perfectly constant or still keeps altering only very gradually—varies very considerably and sometimes exhibits fine or coarse crystals which are coaxial or stretched out longitudinally, sometimes individual crystals that extend radially, and also crystals which, to a smaller or greater extent, take up the whole section of the wire and extend over a length several times greater than the diameter of the wire. Sometimes the crystals at the center of the wire differ from those at the periphery. The different structural varieties often alternate irregularly, or more or less regularly with each other in the wire. The structure of the wire is also irregularly affected by manifold circumstances, such as the properties or quality of the material from which it is produced, and the mechanical, chemical, and thermal effects to which it is subjected during manufacture. The physical properties of the wire, and hence its utility depends to a large extent upon its structure.

The object of the present invention is to produce from a body of tungsten a drawn tungsten wire which, after being heat treated until its crystal structure no longer changes to any appreciable extent, has a pre-determined substantially regular and definite crystal or grain structure of a character that occurs only irregularly and at random if the usual processes of making drawn tungsten wire are employed. In accordance with my invention the initial body from which the wire is drawn is intentionally made non-uniform in accordance with a pre-deter-

mined plan so that at many points distributed throughout the body in a pre-determined and regular way or in accordance with a pre-determined plan the material of the body differs from the adjacent material in such a way that crystal growth begins more readily at such points than at other points, whereby there is obtained a pre-determined crystalline structure of a desirable kind. The crystals of this structure may be substantially uniform or may, if desired, vary in size and shape in a regular manner along the wire. The invention is based primarily on the following facts and assumptions published in metallographic literature.

In metallic bodies or structures which are not produced by deforming the crystals by mechanical means, the crystals commence to grow at certain temperatures which generally decrease as the sizes of the crystals decrease. During this process the larger crystals generally absorb the smaller ones, and the irregularities in the material from which the structures are produced, particularly irregularities in the size of its particles, but also other irregularities of a physical or chemical nature, may give rise to a very considerable local or general increase of the size of the grains. An abnormal growth of the grains may be caused by the coincidence of suitable circumstances or conditions at certain temperatures, whilst at a higher or lower temperature the grains that form themselves will be smaller. Abnormal crystalline formations are particularly apt to occur at boundary layers. But, on the whole, the re-crystallization depends more upon the temperature than upon other factors.

In mechanically worked metals the commencement and the speed of the so-called re-crystallization depends in a great measure upon the degree to which the metal has been worked, re-crystallization taking place at a temperature which is the lower, the more the metal has been worked. Every body or piece thus worked consists of an aggregation of

crystals comprising irregularly distributed parts which have been deformed to greater and lesser extents, and the re-crystallization of these parts commences at different temperatures and proceeds at different speeds accordingly. In order to comprehend this, the long, stretched fibers of a drawn wire may be considered in regard to their tendency to re-crystallize as series of small crystals placed end to end, whose diameter corresponds to that of the fiber at the particular position considered, and which are re-crystallized at different temperatures according to their sizes. Or, in accordance with opinions, it may be assumed that the smallest particles are displaced to a different extent according to their degrees of deformation, or in other words that their positions in space as defined by coordinate lattices are irregular and that they consequently exert efforts of different strengths to regain regular positions in the said space by re-crystallization.

These known facts or hypotheses form the basis of the opinions set forth herein-after as to the reasons why the efforts to always obtain a structure of a desired kind in tungsten wires have hitherto been unsuccessful. It is, however, not averred that these opinions represent the only and correct explanation of the effect of the invention.

In annealing tungsten wires, such as those used for making the illuminating bodies of electric glowlamps, the wires are either evenly heated throughout their whole lengths as described in United States patent to Pacz No. 1,410,499, March 21, 1922, or gradually passed through a hot zone in accordance with a known process as described in United States patent to Schaller No. 1,256,929, February 9, 1918.

When the entire wires are annealed large parts of the same are heated at the same time to the same temperature. Crystals will commence growing here and there, one crystal originating quicker than another according to the thickness of the fibers and their position at the particular spot and the properties of adjacent fibers. It will depend on chance whether numerous crystals attain fairly even sizes in a certain section, or whether a few of them will preponderate and absorb the others so that here and there crystals of an extraordinary size will develop. In passing the wire through a hot zone a surface of uniform temperature will be produced which is generally similar to a paraboloid with its concave side facing the direction in which the wire is moved or the opposite direction depending upon whether the greater part of the heat is supplied to the wire from an external heating coil or from the interior of the wire by passing a heavy current through it. These different kinds of surfaces may also occur in different

temperature zones in a wire. If, for example, a structure or body is to be produced that consists of a long crystal that occupies the whole sectional area of the structure it will be found that, in the case of a squirted filament which, in its preliminary stages of development, consists of parts of approximately equal size, or in any case of parts whose degrees of displacement are equally small, either the incipient crystal located in the line of the central axis of the wire, or a crystal that commences to grow near the periphery, will preponderate, according to the nature of the heating of the structure, because the particles situated next to the growing crystal in the same sectional plane as the crystal can all be absorbed by it on account of the fact that the temperature of the said particles is lower than that of the crystal so that they are, as yet, not crystallized or re-crystallized and are therefore smaller than the growing crystal and hence prone to absorption. However, it will often happen that a growing crystal will here and there strike upon another crystal, which, as a result of its previous history has already assumed a stable form, at a lower temperature, has increased in size, and will then continue to grow independently and to form a dividing surface between itself and the crystal that encounters it. Thus only very irregular long crystals will, as a rule, develop in drawn wires.

In accordance with this invention and the process hereinafter described with greater particularity the crystallization in drawn tungsten wire begins, or progresses more rapidly at or in certain points or layers distributed throughout the wire in accordance with a pre-determined plan as hereinafter set forth; or if hinderances to crystallization are present at such well distributed points or layers, the crystal growth proceeds more rapidly elsewhere in the wire; but in any event there is obtained in the wire the formation of a crystal structure in accordance with a definite and pre-determined plan.

In accordance with the novel process the bodies from which the wire is developed are bodies whose particles have been caused to cohere by the application of pressure in accordance with United States patent to Coolidge No. 1,082,933, December 30, 1913, or by any other suitable means, and whose cross sections contain zones which differ from the main substance and, if necessary, from each other, in regard to their chemical or physical properties, or the composition of their component parts, their nature, proportion or characteristics. As a rule all sections of the body are similar to each other throughout its length. The initial piece, i. e. the piece from which the wire is developed, may consist of a homogenous

principal or ground substance into which longitudinal bodies that possess the above-mentioned qualities and which are of circular, rectangular, or any form of cross section or size, are introduced. The composition of the initial body may differ in the direction from its axis towards its periphery, or its composition may be different at various points along a line extending from one part of its periphery to a diametrically opposite part, or the wire may contain sectors differing in their compositions. The layers or other forms of elements of different compositions of which the wire consists may be continuous, or they may abruptly or gradually merge into each other.

It has been found that, in spite of the considerable difficulties always encountered in the mechanical working of tungsten, bodies of the above-described kind are, in many cases, capable of being worked even with such longitudinal bodies introduced into them as are unworkable when treated by themselves, and that the diffusion in intensely heated tungsten is generally so small that the shapes of the cross sections of the introduced bodies remain practically unchanged, as in producing platinum wires of extreme fineness according to the Wellaston process, even when their diameters are reduced to the utmost. The ground substance, and the bodies introduced into the same, may consist of pure tungsten with grains of different fineness, tungsten with various admixtures such as thorium di-oxide in various proportions, degrees of fineness and distribution; tungsten containing, in solution or mixed, other metals or metalloids such as molybdenum, uranium, thorium, tantalum, vanadium, chromium, carbon, phosphor or others, or mixtures or combinations of these elements with each other or with other elements. The differences between the layers etc. may sometimes be very slight, as for instance when it is desired to decrease or increase the rapidity of crystallization in a layer by adding a metal or certain oxides.

The wires produced from the above-described initial bodies are finally subjected to a suitable heat treating which is already known in the art. In carrying out the above-described process the procedure may be as follows: The principal substance is made up of tungsten with a certain proportion of thorium di-oxide for instance, whilst the core is made up of tungsten with a smaller proportion of thorium dioxide or other addition, or of pure tungsten; or the principal substance is made up of pure tungsten and the core of tungsten whose temperature of crystallization has been reduced by additions of other materials, or tungsten having a lower specific gravity. The wires comprising a core that crystallizes easier than its surrounding substances is subjected

to a suitable heat treatment such as disclosed in Schaller Patent No. 1,256,929 during the course of which the formation of crystals will progress along the core in a regular manner, if the core of the wire is hotter or at least not colder than the principal substance. An alternative procedure is the following: For the purpose of forming coarse crystals stretched to considerable lengths and which impart a fibrous texture to the wire drawn wires are used consisting of material containing little or no thorium and comprising longitudinal columns or layers of tungsten containing a high percentage of thorium di-oxide or of an admixture which produces a similar effect. The wires are either caused to continually pass through a heating device, such as described in the patent to Schaller No. 1,256,929, whereby there is obtained a tungsten wire having in effect longitudinal strands of thoriated tungsten, or the whole of the said wires will be annealed simultaneously at the temperature of rapid grain growth.

The effect of the additions to the tungsten is as though there were distributed through the mass of tungsten in a regular and predetermined manner nuclei or points or origin for crystals. The growth of the crystals begins or is accelerated at such points and the crystals grow under conditions that are predetermined to a large extent and depend upon the distribution of such nuclei throughout the mass of tungsten. By means of these additions there is obtained a control of the distribution of these nuclei, so that they can be distributed throughout the mass of tungsten substantially in accordance with any pre-determined plan and in this way, there may be obtained a finished produce which consists of crystals or grains of a desirable kind arranged substantially in a pre-determined and planned way as distinguished from the heterogenous and uncontrollable crystal arrangement obtained in wire made in the usual way. A body of tungsten having a structure consisting essentially of large uniformly distributed crystals or grains obtained in this manner has less tendency to offset and to sag than is exhibited by a similar body of tungsten made in the usual way. The additions may be introduced into the pressed initial bodies in various ways, as by placing different layers of granules or powder one above the other, or one within the other; by introducing bodies or wires whose particles have already been brought to a more or less high degree of coherence, or have been more or less sintered or subjected to mechanical treatment; or by introducing granulated bodies, or bodies whose particles cohere more or less, into holes in the initial body; by impregnating the pressed bodies whose particles may adhere to each other more or less

strongly with limited quantities of solutions which only penetrate into the exterior layers of the said bodies; by impregnating the bodies several times with solutions that
 5 penetrate to different depths; by thus impregnating the bodies several times and by partial chemical conversion of the introduced substances by means of liquids or gases, which latter process may be combined
 10 with the extraction of the non-converted parts by means of solutions or by distillation; and in many other ways.

The heat treatment may consist in annealing the wire or bodies in a known manner
 15 as described in the patent to Pacz No. 1,410,499 at certain fixed temperatures or with rising temperatures. Or it may consist in passing the wire through a hot zone formed by causing an electric current of sufficient
 20 strength to pass through a short piece of a wire, or by this piece of wire being surrounded by an exterior heating body in the form of a cylinder or ring or the like as illustrated in the patent to Schaller No.
 25 1,256,929. A combination of these two heating means may be employed and, according to the details of the procedure adopted, the temperature in the interior of the filament may be higher than the tem-
 30 perature at the surface or vice versa. In another modification a short piece of the wire may be very intensely heated by an electric current and the preceding or following piece may be heated to a lower tem-
 35 perature.

In addition to the advantages set forth above, wires produced in accordance with the above-described process offer other advantages. Thus in wires in which the intro-
 40 duced bodies determine the progress of the process of crystallization, the introduced bodies are protected by their envelope against deleterious effects due to the mechanical treatment to which the wire is sub-
 45 jected and which effects would change the crystallizability. Furthermore, the composition of the wire the various zones of whose cross section consist of substances of different characters, may be such that stresses
 50 which arise in an ordinary wire when it is annealed and which result in distortions of the bodies such as incandescent bodies produced therefrom, are caused to neutralize each other by the counterbalancing effects of
 55 the suitably selected layers of which the wire is built up.

I claim:

1. In a process of manufacturing drawn wires: developing the wire from an initial
 60 body containing longitudinal bodies whose crystallizability differs from that of the rest of the substance of the initial body; treating by heat whereby the crystallization process is caused to progress along the longitu-

dinal bodies, and a union of crystals in a
 65 direction across the cross section of the wire is prevented, and a wire is obtained whose permanent structure is characterized by the majority of its crystals having a length that is several times greater than the diameter
 70 of the wire.

2. A wire consisting of different materials crystallized into longitudinal layers of different character, the same metal being
 75 present in each layer.

3. A wire consisting of different materials crystallized into longitudinal layers of different character, each layer having tung-
 80 sten.

4. A wire consisting of different materials crystallized into longitudinal layers of different character, the same metal being
 85 present in each layer, one layer consisting of said metal and an ingredient.

5. A wire consisting of different materials crystallized into longitudinal layers of different character, each layer having tung-
 90 sten, one layer consisting of tungsten and an ingredient.

6. A wire consisting of different materials crystallized into longitudinal layers of different character, the same metal being
 95 present in each layer, one layer consisting of said metal and an ingredient and another layer consisting of said metal and a different proportion of the ingredient.

7. A wire consisting of different materials crystallized into longitudinal layers of different character, each layer having tung-
 100 sten, one layer consisting of tungsten and an ingredient, and another layer consisting of tungsten and a different proportion of the ingredient.

8. A wire consisting of different materials crystallized into longitudinal layers, the crystal formation of the layers being dif-
 105 ferent from one another, each layer having a metal.

9. A wire consisting of different materials crystallized into longitudinal layers, the crystal formation of the layers being dif-
 110 ferent from one another, the same metal being present in each layer.

10. A wire consisting of different materials crystallized into longitudinal layers, the crystal formation of the layers being dif-
 115 ferent from one another, each layer having tungsten.

11. The process of making a current conducting filament for an electric device which
 120 consists in compressing different materials into longitudinal layers to form a body, drawing the body and heating to convert into permanent crystalline form.

12. The process of making a current con-
 125 ducting filament for an electric device which consists in compressing different materials into layers to form a body, each layer hav-

ing tungsten, hot working and drawing the body into wire and heating to convert the wire into permanent crystalline form. nally disposed crystals, one surrounded by the other, the crystals in one zone overlapping the crystals in the other, each zone forming a longitudinal section of the filament distinguishable from the other. 10

13. A wire consisting of different layers of macrocrystalline material, each layer being of a different character, the same metal being present in each layer. In testimony whereof I affix my signature.

14. A filament having zones of longitudi-

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