Nov. 3, 1925. 1,560,335

J. CZOCHRALSKI

PROCESS OF IMPROVING ALLOYS AND METALS

Filed March 27, 1924

Fig. 1.

Prior Art

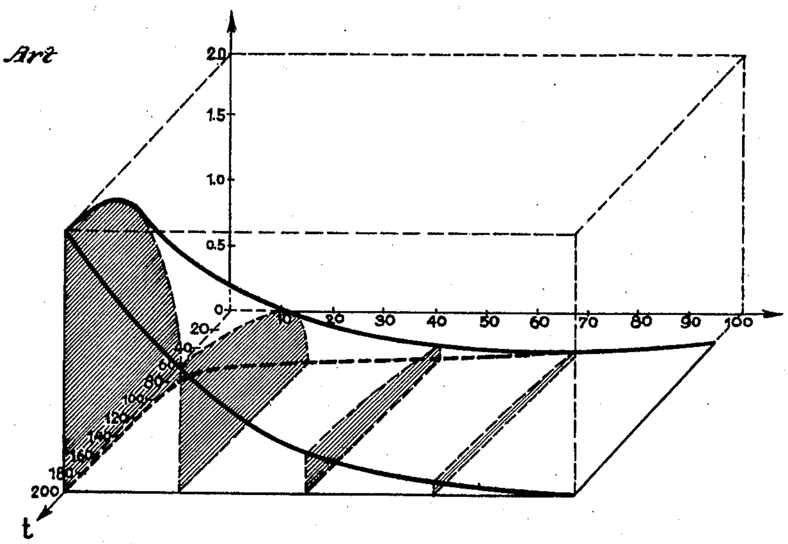
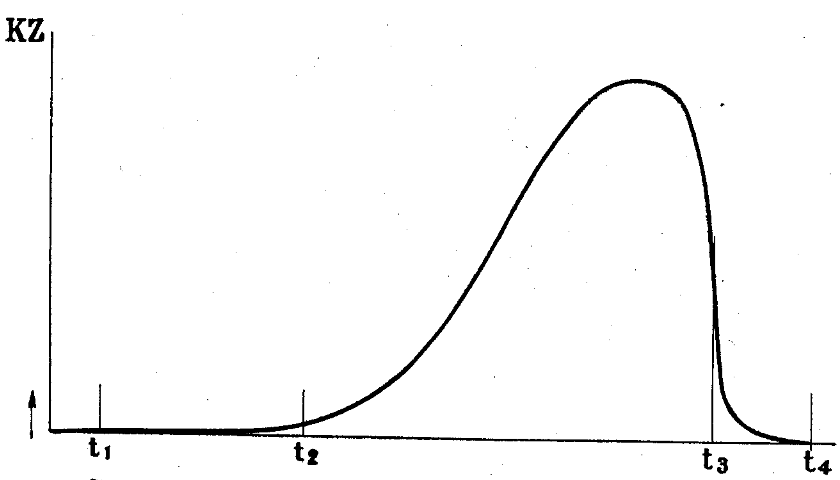


Fig. 2.



Johann Czochralski
Inventor:

by Byrnes Townsend & Brickenstein
Attys.

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UNITED STATES PATENT OFFICE.

JOHANN CZOCHRALSKI, OF FRANKFORT-ON-THE-MAIN, GERMANY, ASSIGNOR TO AMERICAN LURGI CORPORATION, OF NEW YORK, N. Y., A CORPORATION.

PROCESS OF IMPROVING ALLOYS AND METALS.

Application filed March 27, 1924. Serial No. 702,439.

To all whom it may concern:

Be it known that I, JOHANN CZOCHRALSKI, a citizen of the German Republic, residing at Frankfort-on-the-Main, Parkstrasse 18, Germany, have invented certain new and useful Improvements in Processes of Improving Alloys and Metals, of which the following is a specification.

This invention relates to a process of improving alloys and metals.

Single crystals of technical alloys and metals have up to the present been employed in only a single branch of industry, that is for the production of metal-filament lamps. As starting material, metallic tungsten was used almost exclusively.

In all other branches of the technical industry the tendencies are of an opposite nature. All technological processes of improving metals aim at obtaining materials in as finely granular a form as possible.

Thus whilst coarse-grained metals have proved inferior or even entirely useless for technological purposes, the remarkable fact has been found that the individual grains from which coarse-grained metals are built up, possess particularly valuable technical properties. The problem to be solved by technology is to produce such crystals of sufficient size. From the so-called re-crystallization diagrams, represented by Fig. 1 which is a reproduction from page 30, Internationale Zeitschrift für Metallographie, vol. 8, it is known that the granular size of a recrystallized metal has proved to be a function of the degree of cold working and the glowing temperature. This treatment can be repeated as often as desired with the same success. In this manner it is also possible to produce single crystals of any desired size, especially if quite definitely limited working conditions are adhered to. These conditions are based firstly upon a critical degree of cold working as can be seen from the construction of the diagram, Fig. 1. The glowing temperature may however, vary within wider limits; nevertheless it is important that the glowing temperature be increased very slowly.

The use of such single crystals is based upon the fact, that they possess quite different properties in the various directions of their axes. With metal crystals of the isometric system, for example, the strength and elasticity attain a maximum value in the direction perpendicular to the octahedral surface, whereas on the other hand the ductility is at a maximum in the directions perpendicular to the dodecahedral surface. According to whether a material of great strength or of high ductility is required, the constructional material has to be produced in the directions of these axes. However, definite relations to the direction of the crystal exist as regards also torsional strength, blowing- and bending-strength, hardness and compressive strength. The sound quality of single crystals is particularly peculiar and this opens quite new fields of application for them as construction material. Moreover, the conductivity for heat, sound and electricity varies considerably in the various directions of the crystals. Single crystal textures of all metals of importance in the technical field and metal alloys without exception, exhibit this characteristic.

By mechanical working, special properties such as increase in hardness, strength and elasticity and the like can be imparted to the single crystals.

The possibility of general technical application of single crystals has not up to the present been known, and has nowhere been described.

It has now been found that quite a special structure can be produced in single crystals by mechanical working. As can be shown by way of an etching test they then exhibit quite a different grain in accordance with the kind of working or stress to which they have been subjected. For example in the case of samples submitted to torsion a structure is obtained, which is characterized by a very regular construction, inasmuch as a crystal subjected to stress of this kind exhibits quite different reflexes in its various parts. In consequence of the regular arrangement of these reflexes at the etched plane surfaces or cross sections, decorative effects can be attained. Single crystal structures treated in such a manner, may be used for art purposes, as well as for ornamentation.

The above-described method for the production of single crystals is generally adapted for operation on a small scale as, for instance, in research work.

It has now been found that the production of single crystals of any size can be effected on a technical scale, if definite working conditions are adhered to. This is attained by first maintaining the metal, which has been previously submitted to a critical degree of stretching, at its recrystallization temperature for some time, and thereupon submitting same to the action of an essentially higher recrystallization temperature. The process is illustrated by Fig. 2. The ordinate gives the nucleus figures, whilst the abscissa the temperatures in degree centigrade. When a multicrystalline sample of a certain degree of cold working is submitted to recrystallization, then the process of nucleus formation starts within the temperatures zone t^1 and t^2 (lower temperature of recrystallization). The velocity of formation of the nuclei is infinitesimally small within lower limits of temperature. The less nuclei formed, the more favourable will this be for the subsequent process of recrystallization. It is therefore necessary to select the conditions in such a way, that only a single nucleus is formed. The nucleus thus formed is thereupon further developed at the same temperature, until it can be made visible at the surface, if desired, by etching. The thus grown crystal nucleus is free of tension to a high degree and is highly superior to all adjacent crystals in respect of stability. It is therefore best preconditioned and capable for further growing by gradually assimilating all adjacent crystals. Hitherto the opinion prevailed that this process of further growth proceeds the more favourably, the more slowly the temperature of the sample is increased. Investigations on which the invention is based have shown that, contrary to the older views, a rapid increase in temperature is of the greatest advantage. In the case of aluminum the temperature range t^1 to t^2 is approximately between 250–500° C. the temperature range t^3 to t^4 between about 600–654° C. In the case of other metals the temperature ranges lie in essentially different limits, with tin, for example, within 20–100° C. and 200–230° C. The lower recrystallization temperature may also be influenced especially by the degree of cold stretching.

By the above-described method perfect crystals can be produced in the most easy manner on a technical scale. It is entirely immaterial, whether cast metals or recrystallized metals or alloys are used as initial material. However single crystals can also be obtained with metals, which in themselves produce tensional stresses within the metals, as is the case, for example, with metals deposited by electrolysis or by compressing powder, as well as with metal deposits produced by diffusion or sublimation.

What I claim is:

1. Process of producing single-crystal metal structures, which consists in initially cold-working the metal to a suitable critical degree, then maintaining the thus treated metal at a relatively low recrystallization temperature until a nucleus is well formed and then developing the nucleus by application of a higher recrystallization temperature until conversion to a single-crystal structure is completed.

2. Process of producing single-crystal metal structures of predetermined physical characteristics, which consists in initially subjecting the metal to mechanical strain depending on the physical characteristics to be imparted, then maintaining the metal thus treated at a relatively low recrystallization temperature until a nucleus is well formed and then maintaining the metal at a higher recrystallization temperature until conversion to a single-crystal structure is completed.

3. Process of producing single-crystal metal structures of predetermined physical characteristics, which consists in initially cold-working the metal structure to impart to it definite physical qualities, then maintaining the metal thus treated for an extended period at a relatively low re-crystallization temperature favorable to nucleus formation and then raising the crystallization temperature at a relatively high rate and maintaining the crystallization temperature until the conversion to a single crystal structure is completed.

4. Process according to claim 1, wherein the cold-working includes subjecting the metal to torsional stresses.

In testimony whereof I affix my signature.

JOHANN CZOCHRALSKI.