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BACKGROUND OF THE INVENTION

field of the invention

the present invention relates to a hydrogen storage body, and more particularly to a hydrogen storage body capable of storing hydrogen in a short time thus, the present invention provides a hydrogen purification. The present invention is widely applicable to a recovery device, a heat pump, a hydrogen storage device, an actuator, a normal temperature nuclear fusion device, and the like

description of the related art

in recent years, a hydrogen storage body such as a hydrogen storage alloy has not only been used as a storage tank for hydrogen (light hydrogen) fuel serving as a future clean energy source, but also has attracted attention to practical use to a storage battery, an automobile, a household goods and the like. As the use form of the hydrogen storage body, a method such as filling the powder with a container, subjecting the powder to a pre-treatment such as microencapsulation or pelletization by a binder, or the like, or attaching metal hydride particles to the porous metal body is adopted

the reason for such a form is that the crystal lattice of the hydrogen storage body is generally in the range of 10 to 25% to expand the volume by the adsorption and desorption of hydrogen (light hydrogen). This is because shrinkage occurs. In other words, the bulk hydrogen storage alloy contains hydrogen ions. To provide a charging. To solve the problem that when discharging is repeated. To solve a problem that a fine crack, a pulverizing phenomenon, a shape change. This is because the desorption characteristics are deteriorated a hydrogen storage. To provide a hydrogen storage. It is shown that a hydrogen storage alloy having a particle size of a limit degree (about several μ) of powdering due to release is used

on the other hand, as an example in which a hydrogen storage body is used, a group of martin flischmann taught by the university of the united states university and the university stley pons of united states, and the so-called normal temperature nuclear fusion published by the university steven E Jones teaching of the united states are attracting attention (for example, "E Jones et. Al Nature 338 (1989) 737") the normal temperature nuclear fusion is a method in which an anode is immersed in a solution in which heavy water and a small amount of a metal salt are dissolved, and an electrode is immersed in a solution in which a positive electrode is a white bacterium or a bacterium, and a cathode is made to have hydrogen storage capacity, and electrolysis is performed. This method is very simple in comparison with the conventional nuclear fusion method for stopping the high temperature plasma, and has attracted attention. A plate-like or rod-like palladium or titanium is mainly used as a cathode for generating normal temperature nuclear fusion, and as a result of electric separation, deuterium is stored in Pd or Ti as the electrode, and nuclear fusion is considered to occur in order to cause such a reaction, a large amount of hydrogen storage must be performed in the electrode. A hydrogen storage body used in normal temperature nuclear fusion is different from a conventional hydrogen storage body, and a bulk type is used. To provide a hydrogen storage. To prevent the use of a general hydrogen storage body for hydrogen storage such as repeating emission

however, when fine powder is used as a hydrogen storage body, hydrogen storage. To provide a method for deforming a hydrogen storage alloy structure due to release. To prevent collapse, but not to sufficiently improve time and efficiency required for hydrogen storage. To solve the problem that, when

excessive hydrogen is stored in a hydrogen storage body by using a bulk hydrogen storage body as an electrode as in the case of causing a normal temperature nuclear fusion reaction, a considerable time is required until a practical hydrogen storage capacity is obtained because the storage speed of deuterium is slow, and the pretreatment of the normal temperature nuclear fusion electrode is very troublesome

therefore, there is a need for a hydrogen storage body capable of efficiently performing hydrogen storage in a shorter time and in a high concentration [problem to be solved by the invention]

to provide a hydrogen storage body capable of efficiently storing hydrogen in a hydrogen storage body in a short time in a short time

the present invention also provides a hydrogen storage body capable of performing hydrogen storage in a high concentration in a short time as a normal temperature nuclear fusion electrode

the hydrogen in the present invention also indicates light hydrogen, deuterium, tritium, and mixed gas thereof unless otherwise specified

the present invention is to solve the problem

the present invention is made to solve such problems, and in the first aspect, pd ultrafine particles, which are hydrogen storage materials having an average particle size of 200 Å or less, are deposited on a base material made of pd, which is a hydrogen storage material

in a second embodiment of the present invention, ti ultra-fine particles, which are hydrogen storage materials having an average particle size of 200 Å or less, are deposited on a base material comprising lani5 as a hydrogen storage material in the first and second embodiments, the deposition thickness of the ultrafine particles deposited on the base material may be 0.2 μm or more and 100 μm or less

[image formation]

in the present invention, hydrogen storage speed or storage efficiency can be improved by forming a hydrogen storage body using ultrafine particles of a hydrogen storage material having an average particle size of 200 Å or less when such ultrafine particles are produced, the particle diameter control is easy, and the reproducibility is excellent even at 100 Å or less, and therefore, a gas evaporation method and a sputtering method are preferably used

the present invention relates to a method for producing a steel sheet

the present invention is obtained as a result of intensive studies. When the present invention is completed, the hydrogen storage state of the hydrogen storage body electrode used in the conventional normal temperature nuclear fusion reaction is examined. As a result, it has been found that the deuterium is not sufficiently stored unless the hydrogen storage is performed by the electric segregation over a considerable time. A second drawing is an electrolysis cell according to an example of a normal temperature nuclear fusion device, and an anode 7 and a cathode 6 consisting of a hydrogen storage body are immersed in an electrolytic solution 5 composed of heavy water in a container 4 and an additive of a small amount of metal salt by a constant voltage power source 3, and electrolysis is performed by applying a constant voltage of 5 v to both electrodes at this time, a palladium rod of 4 mmφ is used for the cathode 6, and 0.1 mol of LIOD and heavy water are used as a small amount of metal salt in the electrolyte solution 5. Hydrogen storage materials such as pd and lani5 are known to transition from alpha to beta in association with storage of hydrogen. Thus, the state of hydrogen storage can be known by examining the transition state of alpha → beta a hydrogen storage body in which deuterium is stored by electrolysis of heavy water is measured by x-ray diffraction. When the surface of the palladium rod is a mixed phase of (alpha + beta) after a lapse of

150 hours, the peak of the beta phase which is the basis for sufficiently storing deuterium after 450 hours has been confirmed. That is, it has been found that the storage of deuterium is still insufficient at about 150 hours. It has been found that a very long time is required for the hydrogen storage body to be in a state in which hydrogen is sufficiently stored as a result of intensive studies in order to improve the hydrogen storage efficiency in the present invention, it has been found that the storage efficiency can be improved by using a hydrogen storage body containing ultrafine particles comprising a hydrogen storage material having an average particle size of 200 Å or less the substance has various characteristics (magnetic properties, electric characteristics, etc.), and it is known that when the substance is made small, characteristics different from those of the original solid substance are exhibited. The particles giving such a state are referred to as "ultrafine particles", and are distinguished from typical particles. In general, a particle having a size of about 0.1 μm does not significantly change the characteristics of the solid substance, but it is considered that there is a limit in which the property different from that of the large particle becomes remarkable because of the fact that the particle becomes finer particles the change of the properties is considered to occur due to the volume effect and the surface effect accompanied by an increase in the surface area, and the phenomenon in which the storage efficiency of hydrogen is greatly improved in the present invention is considered not to be involved in these effects

in particular, when the hydrogen storage body according to the present invention is used as an electrode, the overvoltage of the electrolytic electrode can be reduced by the effect of the ultrafine particles, and excessive sub-reactions other than hydrogen generation can be suppressed, and hydrogen can be efficiently generated. Further, it is considered that hydrogen storage can be performed more efficiently because the probability of hydrogen storage reaction on the surface is increased by the surface effect

the present invention is described below with reference to the drawings the third and fourth figures show an outline of a typical device configuration for obtaining ultrafine particles used as a hydrogen storage body of the present invention. The apparatus of the third drawing uses a gas evaporation method, and the apparatus is composed of an ultrafine particle generation chamber 12, an ultrafine particle volume chamber 14, and a reduction expansion nozzle 13 for connecting the two chambers. Reference numeral 11 denotes an introduction system of an Ar gas which is an inert gas, and 16 denotes an exhaust system. Reference numeral 10 denotes an evaporation source of a hydrogen storage material serving as a raw material for the ultrafine particles arranged in the ultrafine particle generation chamber 12, and reference numeral 9 denotes a sample stage for placing and rotating a base material as a sample 9 further, 15 denotes the start of the deposition of the ultrafine particles/ and to control the deposition thickness

using such a device, a base material 8 is set on a stage 9, and the inside of the apparatus is evacuated to a vacuum degree of 10 to 7 torr by an exhaust system 16, and an inert gas is introduced into the ultrafine particle generating chamber 12. It is desirable that the diameter of the reduction expansion nozzle 13 is about 5 mm, and the distance between the reduction expansion nozzle 13 and the base material 8 is about 200 mm

a hydrogen storage material such as palladium is evaporated from an evaporation source 10 such as a carbon crucible, and the generated ultrafine particles are blown out from a nozzle 13, and deposited on the base material 8

the fourth figure shows the outline of a vacuum apparatus for deposition of ultrafine particles for sputtering using a sputtering method. And the structure

of the apparatus is a target of a hydrogen storage material serving as a raw material for the ultrafine particles for sputtering in place of the evaporation source 10 of the crucible which is in the ultra-fine particle generator 12 of the apparatus of the third drawing, and the same numerals are assigned to the same parts as those in the third drawing. In the deposition method, an exhaust system 16 exhausts the vacuum in the apparatus to 10^{-7} torr, and introduces argon gas 11 as an inert gas into the ultra-fine particle generation chamber 12 in this case, it is desirable that the diameter of the reduction expansion nozzle 13 is about 4 mm, and the distance between the reduction expansion nozzle 13 and the base material 8 is about 150 mm. a voltage is applied to a target 17 of 4 inches by DC sputtering, and the generated ultrafine particles are deposited on a base material 8

it is possible to form a hydrogen storage body having ultrafine particles by using these devices and methods, and any device and method capable of forming ultrafine particles with good reproducibility even in these devices and methods, and capable of easily controlling the particle size can be used. The control of the particle size of the ultrafine particles produced in the third and fourth figures can be performed by the gas flow rate introduced into the ultrafine particle generation chamber 12. In this case, the particle size can be made smaller as the gas flow rate is lowered. The gas to be introduced may be other than argon as long as it is an inert gas

a hydrogen storage material used for a base material for depositing ultrafine particles and ultra-fine particles includes: a single metal such as Pd, Ti, Fe, Ni, Pt, Mg, La; and other metals such as Lanthanum, Lanthanum-x-cobalt (a: Ti, Y, Ce); rare earth alloys TiFe, TiNi, Ti₂Ni, TiNi such as Lanthanum-yttrium (b: Al, Cr, Mn, Co, Cu), and Lanthanum-y-cobalt (b: Al, In). A method for producing Ti₂Ni, TiNi (3) it is possible to use a titanium alloy such as Ti₂-x-cobalt x-iron, Ti₁-x-niobium (M: V, Cr, Mo, Mn), CaNi₅, Ca₁-x-cobalt, MgNi, Mg₂Cu, Pd-diameter alloy, or the like the shape of the base material is not limited to a needle-like shape, a rod shape, a plate-like shape, and the like. The shape of the base material is not limited to a needle-like shape, a rod shape, a plate-like shape, and the like. The shape of the base material is not limited to a needle-like shape, a rod shape, a plate-like shape, and the like. The shape of the base material may be a shape in which ultrafine particles. Further, the bulk-like part of the hydrogen storage body and the material of the deposited body of the ultra-fine particles may be the same or different materials. When a compound hydrogen storage material such as Lanthanum having a large amount of hydrogen storage is used as ultra-fine particles, the composition of the particles is deviated from the stoichiometric composition. In particular, it is effective to use super-particles as another kind of hydrogen storage material. The base material may be a combination of a hydrogen storage material and another material the deposition thickness of the ultrafine particles is required to be a thickness sufficient to cover the surface of the base material and not to be peeled off from the base material, and the thickness thereof is preferably 0.2 μm or more and 100 μm or less, and more preferably 1 μm to 10 μm the ultrafine particles of the hydrogen storage body are deposited on the surface of the hydrogen storage body of the bulk-like base material, but the present invention is not limited to this, and it is of course possible to appropriately determine the use mode of such ultra-fine particles. When used as a hydrogen generator, for example, a substrate made of glass, Si, GaAs, or a metal body, or an electrode configuration in which ultrafine particles are formed on a substrate formed by forming a metal film can be used. As the hydrogen reservoir, the ultrafine particle powder may be dispersed in a polymeric binder, micro-encapsulated or pelletized, and can be used in a

conventionally used form further, an electrode having a high degree of freedom in form can be produced by applying the polymer dispersed in the high-molecular binder onto a flexible substrate. Further, hydrogen purification with high hydrogen storage amount is achieved by filling the container with the ultrafine particle powder. To provide a hydrogen storage body for a recovery device an embodiment of the present invention in which a hydrogen storage body is manufactured by using the above-described apparatus is described below

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in this embodiment, the apparatus for forming ultrafine particles is performed as follows using the gas in-gas evaporation apparatus of the third figure a palladium rod 8 having a length of 4 mm and a length of 10 cm is used as a base material and is set on a stage 9. Further, palladium is set in the evaporation source 10. Thereafter, the inside of the apparatus is evacuated to a vacuum degree of 10 to 7 Torr by an exhaust system 16, and an ar gas is introduced into the ultra-fine particle generating chamber 12 by 80 sccm. At this time, the pressure in the ultrafine particle generation chamber 12 is 8×10^{-2} Torr, the pressure in the ultrafine particle deposition chamber 14 is 3×10^{-4} Torr, the diameter of the reduction expansion nozzle 13 is 5 mm ϕ , and the distance between the reduction expansion nozzle 13 and the sample 8 is 200 mm next, ultra-fine particles of pd generated by evaporating palladium pd from an evaporation source 10 are blown out from a nozzle 13, and deposited on a base material

the pd ultrafine particles at this time have an average particle size of 100 to 400 Å and an average particle diameter of 200 Å, which are confirmed by an electrolytic electron emission type scanning electron microscope. The deposition thickness is set to about 3 μm. The obtained sample is subjected to heat treatment at 400 to 500°C in a vacuum to improve adhesion of the ultrafine particles

the hydrogen storage body thus prepared has a structure shown in FIG. 1, comprises a base material 1 of pd which is a bulk hydrogen storage material, and ultrafine particles 2 of pd, which is a hydrogen storage material deposited on the base material 1, and is used as an electrode

the electrode is installed as a cathode 6 in a second figure, and electrolysis is performed. A 0.5 mm ϕ platinum wire is used as the anode 7, and the application voltage is fixed to 5 v, and electrolysis is performed for 150 hours under the condition of a current density of about 90 m3/cm2

after electrolysis, concentration analysis of absorbed deuterium is performed by structural analysis and quadrupole mass spectrometry of a bulk hydrogen storage body by x-ray analysis. A sample is placed in a quartz cell for analysis of deuterium concentration, and the inside of the cell is evacuated to 1×10^{-6} torr at room temperature. Thereafter, the sample is heated up to 550°C by a heater at a temperature rise rate of 4° c./min. then, the temperature is maintained for two hours, and the desorption gas is analyzed. In the deuterium, a peak of m/e = 4 is measured, and the integrated amount is obtained

comparative examples 1 and gt;

the same processing as in the first embodiment is performed except that the deposition of the palladium ultrafine particles is not performed in the first embodiment. Analysis is performed. This is referred to as comparative example 1 thus, the integrated amount of the ion peak can be normalized by using the comparative example 1 as 1.0, and the accumulated amount of the ion peak per unit volume can be compared with the integrated amount of the example. The first table to be described later is the result thereof, and by laminating a deposit made of ultra-fine particles of palladium from the table, the storage of deuterium is sufficiently performed in a short time of about 1/3 of the

conventional one, and it is found that the beta phase indicating that the deuterium is stored is sufficiently detected in the x-ray analysis even in the x-ray analysis

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in place of the pd rod in example 1, a plate of 10 mm × 10 mm × 3 mm of lani5 is used as a bulk hydrogen storage alloy. Further, titanium ultrafine particles are deposited on a lani5 plate by using a vacuum apparatus for sputtering of a fourth figure on a lani5 plate instead of the deposit of the palladium ultrafine particles

in the ultra-fine particle molding, ti is used as a target for sputtering in the deposition method, the degree of vacuum in the apparatus is set to 10 to 7 Torr by an exhaust system 16, and an ar gas 11 is introduced into the ultra-fine particle generating chamber 12 by 100 sccm. At this time, the pressure in the ultra-fine particle generation chamber 12 is 0.8 Torr, the pressure in the ultra-fine particle deposition chamber 14 is 8×10^{-4} Torr, the diameter of the reduction expansion nozzle 13 is 4 mmφ, and the distance between the reduction expansion nozzle 13 and the sample 8 is 150 mm sputtering is performed on a ti target 17 of 4 inches by DC sputtering under a condition of an applied voltage of - 900 V and an input power of 360 w, and the generated ti ultrafine particles are deposited on a sample 8 by about 5 μm. It is confirmed by the FE-SEM that the ti ultrafine particles at this time have a particle size of 40 to 200 Å and an average particle diameter of 80 Å. The sample is heat treated in a vacuum at 500-700°C

comparative examples 2 and gt;

the same processing as in example 2 is performed except that the deposition of titanium ultrafine particles is not performed in example 2. Analysis is performed. This is referred to as a comparative example 2

the integrated amounts of the ion peaks of example 2 and comparative example 2 are relatively compared with each other in the same manner as in example 1 and comparative example 1. The results are shown in table 1. However, as compared with the relative comparison between example 1 and comparative example 1, in comparison with the relative comparison between example 1 and comparative example 1, the storage of deuterium is sufficiently performed in a shorter time than in comparative example 2, and it is found that a large amount of beta phase indicating that deuterium is sufficiently stored in x-ray analysis is detected

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the difference in hydrogen storage efficiency due to the size of the ultrafine particles to be deposited is shown

the same experiment as in example 1 is performed except that an ar gas introduction amount to the ultrafine particle generation chamber 12 in example 1 is changed to 20,50,100, 150, 200 sccm. The average particle size of the pd ultrafine particles at this time is 100 Å, 170 Å, 200 Å, 250 Å, and 280 Å after electrolysis, a deuterium ion peak integration amount is obtained by a method similar to that obtained by performing concentration analysis of deuterium absorbed by a quadrupole mass spectrometry method in a &T. the total amount of deuterium ion peak in each average particle diameter is normalized to 1.0 as the result of example 1, and shown in table 2. As can be seen from the table, the average particle size of the ultrafine particles is 200 Å or less, and the storage amount of deuterium is rapidly increased

as described above, by laminating a deposit made of ultrafine particles of a hydrogen storage material on a bulk hydrogen storage material (base material), and providing an electrode for accelerating deuterium storage by electrolysis, an electrode having a practical hydrogen storage capacity capable of using the bulk body itself can be obtained in a short period of processing without

requiring much labor
in the above embodiment, the case where deuterium is occluded has been described. However, the present invention is also effective as a light hydrogen storage body
the present invention has been described in detail with reference to the accompanying drawings
as described above, according to the present invention, it is possible to obtain a hydrogen storage body capable of efficiently storing hydrogen in a high concentration in a short time
the present invention is not limited to the above embodiments and various changes and modifications may be made without departing from the

the first figure is a schematic cross-sectional view showing one embodiment of the hydrogen storage body of the present invention
FIG. 2 is a schematic diagram of an electrolytic device to which the present embodiment is applicable
FIG. 3 is a schematic diagram of a vacuum apparatus for showing a production principle of ultrafine particles and a deposition method
FIG. 4 is a schematic diagram of a vacuum apparatus for showing another production principle of ultrafine particles and a deposition method

one-bulk hydrogen storage material
ultrafine particle of two-hydrogen storage material
three-constant voltage power supply
four-container,
five electrolyte solution
six-cathode,
7-anode,
eight samples
9-sample stage
10 evaporation source
11 there is provided a method for producing an introduction gas (R),
12 ultrafine particle generation chamber
13 nozzle and nozzle
14 ultrafine particle deposition chamber
15 shutter
16 exhaust system
17