

[Document Name] SPECIFICATION

[Title of the Invention]

5 SURFACE-TREATED ALUMINUM MATERIAL, METHOD OF  
MANUFACTURING THE SAME, AND MATERIAL FOR SEMICONDUCTOR-  
MANUFACTURING APPARATUS

[TECHNICAL FIELD]

10 **[0001]** The present invention relates to a surface-treated aluminum material, a method  
of manufacturing the same, and a material for a semiconductor-manufacturing apparatus.

[BACKGROUND ART]

15 **[0002]** Aluminum materials composed of aluminum or an aluminum alloy are utilized  
in various applications. An anodic oxide film can be provided on the surface(s) of these  
aluminum materials for purposes such as surface protection.

20 **[0003]** For example, a component for a substrate-processing apparatus that performs  
plasma treatment on a substrate is disclosed in Patent Document 1, characterized by having a  
film that has been formed on a surface of the component by an anodizing treatment, in which  
the component is connected to an anode of a DC power supply and is immersed in a solution  
that contains an organic acid as a main component, wherein a semi-sealing process using  
boiling water is performed on the film.

[PRIOR ART LITERATURE]

25 [Patent Documents]

**[0004]** [Patent Document 1] Japanese Laid-open Patent Publication 2008-81815

[SUMMARY OF THE INVENTION]

[PROBLEMS TO BE SOLVED BY THE INVENTION]

30 **[0005]** However, the component of Patent Document 1 has the problem that, because  
pores in the anodic oxide film are not completely closed up, durability with respect to  
corrosive gases and plasma is low.

**[0006]** Meanwhile, a method that completely closes up the pores in the anodic oxide film on the component of Patent Document 1 is conceivable in order to increase durability with respect to corrosive gases and plasma. However, in this situation, cracks are more likely to form in the anodic oxide film when the temperature rises, and there is a risk that debris composed of small pieces of the anodic oxide film might be formed. To curtail the formation of such debris, there is demand to further increase the heat resistance of aluminum materials comprising anodic oxide films on their surfaces.

**[0007]** The present invention was conceived in view of this background, and it is an object is to provide: a surface-treated aluminum material that excels in corrosion resistance with respect to corrosive gases and plasma and is capable of curtailing the formation of cracks even when the temperature rises; a method of manufacturing the same; and a material for a semiconductor-manufacturing apparatus.

#### **[MEANS FOR SOLVING THE PROBLEMS]**

**[0008]** One aspect of the present invention is a surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu (copper) content is 0 mass% or more and 1.8 mass% or less, and a protective film, which is formed on the base material, wherein:

the protective film comprises: an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

in the situation in which cathodic polarization measurements are performed—using a measurement solution obtained by mixing a solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% such that the volumetric ratio of the NaCl solution to the acetic acid = 1000:1—on the base material and on the surface-treated aluminum material after it had been heated at a temperature of 200°C for 4 hours, and the current density at the electric potential at the center of an electric-potential region of the base material that exhibits the diffusion-limited current of hydrogen ions was measured, the ratio  $J1/J2$  of the current density  $J1$  of the surface-treated aluminum material to the current density  $J2$  of the base material is  $150 \times 10^{-5}$  or less.

**[0009]** A second aspect of the present invention is a surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less, and a protective film, which is formed on the base material, wherein:

5 the protective film comprises:

an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

10 a sample having the protective film on one surface of the base material was prepared from the surface-treated aluminum material and measured in a state in which a strain gauge is mounted on the sample on a rear surface of the surface having the protective film, the difference  $\varepsilon_1 - \varepsilon_2$  between the amount  $\varepsilon_1$  of strain of the sample at a temperature of 200°C and the amount  $\varepsilon_2$  of strain of the base material at a temperature of 200°C is  $100 \times 10^{-6}$  or  
15 less.

**[0010]** A third aspect of the present invention is a material for a semiconductor-manufacturing apparatus composed of the surface-treated aluminum material according to the above-mentioned aspects.

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**[0011]** A fourth aspect of the present invention is a method of manufacturing the surface-treated aluminum material according to the aforementioned aspects, comprising:

forming the oxide layer, which has pores, on the base material by performing an anodizing treatment on the base material;

25 thereafter, heating the base material and the oxide layer at a temperature of 50°C or higher and 350°C or lower; and

thereafter, contacting the oxide layer with a sealing agent, and forming the hydrated oxide layer on the oxide layer while sealing the pores.

30 **[EFFECTS OF THE INVENTION]**

**[0012]** A surface-treated aluminum material (referred to as "aluminum material" below) according to the first aspect has a protective film having the oxide layer and the hydrated oxide layer on a surface of the base material. In addition, in the situation in which cathodic polarization measurements are performed according to the above-mentioned specific

method on the base material and on the surface-treated aluminum material after it was heated for 4 hours at a temperature of 200°C, the ratio  $J1/J2$  of the current density  $J1$  of the surface-treated aluminum material to the current density  $J2$  of the base material is  $150 \times 10^{-5}$  or less. Aluminum materials having such a characteristic excel in corrosion resistance with respect to corrosive gas and plasma, excel in heat resistance, and can curtail the formation of cracks even in situations in which the temperature has risen.

**[0013]** A surface-treated aluminum material according to the second aspect has a protective film, which comprises the oxide layer and the hydrated oxide layer on a surface of the base material. In addition, the difference  $\epsilon1 - \epsilon2$  between the amount  $\epsilon1$  of strain of the sample at a temperature of 200°C and the amount  $\epsilon2$  of strain of the base material at a temperature of 200°C, measured using the above-mentioned specific method, is  $100 \times 10^{-6}$  or less. Aluminum materials having such a characteristic excel in corrosion resistance with respect to corrosive gas and plasma, excel in heat resistance, and can curtail the formation of cracks even in situations in which the temperature has risen.

**[0014]** Because it is constituted from the above-mentioned aluminum material, a material for a semiconductor-manufacturing apparatus according to the third aspect excels in corrosion resistance with respect to corrosive gas and plasma, excels in heat resistance, and can curtail the formation of cracks even in situations in which the temperature has risen.

**[0015]** In addition, in the method of manufacturing the aluminum material according to the fourth aspect, after having performed an anodizing treatment on the base material, the oxide layer, which had formed owing to the anodizing treatment, is heated at a temperature within the above-mentioned specific range. Thus, by heating the oxide layer before sealing the pores in the oxide layer, internal stresses created when the oxide layer was formed can be relaxed. Then, after the internal stresses in the oxide layer have been relaxed, by contacting the oxide layer with a sealing agent and forming the hydrated oxide layer on the oxide layer and sealing the pores, corrosion resistance with respect to corrosive gas and plasma increases, heat resistance increases, and the formation of cracks can be curtailed even in situations in which the temperature has risen.

**[0016]** As described above, according to the above-mentioned aspects, a surface-treated aluminum material that excels in corrosion resistance with respect to corrosive gases

and plasma, excels in heat resistance, and is capable of curtailing the formation of cracks even in situations in which the temperature has risen, a method of manufacturing the same, and a material for a semiconductor-manufacturing apparatus can be provided.

## 5 [BRIEF DESCRIPTION OF THE DRAWINGS]

**[0017]** [FIG. 1] FIG. 1 is a cross-sectional view of a surface-treated aluminum material of Working Example 1.

[FIG. 2] FIG. 2 is a cross-sectional view of a base material on which an oxide layer is formed during a process of manufacturing the surface-treated aluminum material of Working Example 1.

[FIG. 3] FIG. 3 is an explanatory graph showing a cathodic polarization curve of the base material of Working Example 1.

[FIG. 4] FIG. 4 is an enlarged view of a step portion in the cathodic polarization curve of the base material.

[FIG. 5] FIG. 5 is an explanatory diagram for describing a method of measuring the amount of strain in the surface-treated aluminum material of Working Example 3.

[FIG. 6] FIG. 6 is an explanatory graph showing results of measuring the amount of strain in the surface-treated aluminum material of Working Example 3.

## 20 [MODES FOR CARRYING OUT THE INVENTION]

### **[0018]** (Aluminum Material)

The base material of the aluminum material is constituted from aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less. The shape of the base material is not particularly limited, and various shapes may be used in accordance with the application of the aluminum material.

**[0019]** The material of the base material in the aluminum material can be selected as appropriate, in accordance with the application of the aluminum alloy, from a group consisting of aluminum and an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less. For example, in situations in which it is attempted to reduce outgassing from the aluminum material, the base material is preferably constituted from a 1000-series aluminum or a 3000-series aluminum alloy.

**[0020]** Aluminum having a chemical composition indicated by, for example, alloy numbers AA1100, AA1100A, AA1200, AA1230, AA1230A, AA1235, AA1145, AA1345, AA1350, AA1199, AA1050, AA1060, AA1085, AA1060EC, or AA1070 can be used as the 1000-series aluminum constituting the base material of the aluminum material.

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**[0021]** In addition, an aluminum alloy having a chemical composition that contains, for example, Mn (manganese): 1.0 mass% or more and 1.5 mass% or less and includes one or two or more elements selected from the group consisting of Si (silicon), Fe (iron), Cu (copper), Mg, Cr (chromium), Zn (zinc), and Ti (titanium) as (an) optional component(s), the remainder being composed of Al and unavoidable impurities, can be used as the 3000-series aluminum alloy constituting the base material of the aluminum material.

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**[0022]** More specifically, an aluminum alloy having a chemical composition indicated by, for example, alloy numbers AA3003, AA3203, AA3004, AA3104, AA3005, AA3105, or AA3021 can be used as the 3000-series aluminum alloy.

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**[0023]** In addition, in situations in which it is attempted to increase the strength of the aluminum material, the base material is preferably constituted from a 5000-series aluminum alloy or a 6000-series aluminum alloy. For example, an aluminum alloy having a chemical composition that contains 0.5 mass% or more and 5.0 mass% or less of Mg (magnesium) and contains one or two or more elements selected from the group consisting of Si, Fe, Cu, Mn, Cr, Zn, and Ti as (an) optional component(s), the remainder being composed of Al and unavoidable impurities, can be used as the 5000-series aluminum alloy.

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**[0024]** More specifically, aluminum having a chemical composition indicated by, for example, alloy numbers AA5182, AA5005, AA5110A, AA5021, AA5041, AA5042, AA5050, AA5151, AA5251, AA5052, AA5252, AA5154, AA5154C, AA5254, AA5454, AA5554, AA5654, AA5754, AA5356, AA5456, AA5556, AA5657, AA5083, AA5183, AA5086, AA5457, AA5082, AA5006, or AA5652 can be used as the 5000-series aluminum alloy.

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**[0025]** In addition, an aluminum alloy having a chemical composition that contains, for example, 0.3 mass% or more and 1.5 mass% or less of Mg and 0.2 mass% or more and 1.2 mass% or less of Si, and contains one or two or more elements selected from the group

consisting of Fe, Cu, Mn, Cr, Zn, and Ti as (an) optional component(s), the remainder being composed of Al and unavoidable impurities, can be used as the 6000-series aluminum alloy constituting the base material of the aluminum material.

5    **[0026]**        More specifically, an aluminum alloy having a chemical composition indicated by, for example, alloy numbers AA6101, AA6201, AA6003, AA6005, AA6005A, AA6005C, AA6105, AA6110, AA6111, AA6016, AA6151, AA6351, AA6951, AA6053, AA6060, AA6061, AA6162, AA6262, AA6463, AA6066, AA6070, AA6181, AA6063, AA6082, or AA6253 can be used as the 6000-series aluminum alloy.

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**[0027]**        More specifically, an aluminum alloy having a chemical composition indicated by, for example, alloy numbers AA8021, AA8079, AA8017, AA1385, AA8030, or AA8176 can be used as the 8000-series aluminum alloy constituting the base material of the aluminum material.

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**[0028]**        A protective film that contains an oxide layer, which is composed of an oxide or oxides of aluminum and has been laminated on the base material, and a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and has been laminated on the oxide layer, is provided on the base material. More specifically, the

20    hydrated oxide layer may be constituted from the hydrated oxide(s) of aluminum. In addition, the hydrated oxide layer may contain a hydrated oxide or hydrated oxides and a metal salt or metal salts. After having formed the oxide layer, which has numerous pores, on the surface of the base material by performing an anodizing treatment on the base material, the protective film can be obtained, for example, by performing a sealing treatment and thereby closing up  
25    the pores in the oxide layer with the hydrated oxide layer. Such a protective film excels in corrosion resistance with respect to corrosive gas and plasma. For this reason, the corrosion resistance of the aluminum material can be increased by forming the protective film on the base material.

30    **[0029]**        The thickness of the protective film preferably is 2  $\mu\text{m}$  or more. Thereby, the corrosion resistance of the aluminum material can be further increased. From the viewpoint of corrosion resistance, the upper limit of the thickness of the protective film is not particularly limited; the thicker the protective film is made, the more the corrosion resistance of the aluminum material can be increased. From this viewpoint, the thickness of the

protective film is more preferably 5  $\mu\text{m}$  or more and yet more preferably 10  $\mu\text{m}$  or more. It is noted that, from the viewpoint of manufacturing, the upper limit of the thickness of the protective film is, for example, 200  $\mu\text{m}$ . From the viewpoint of curtailing the formation of cracks in the protective film, the thickness of the protective film is preferably 100  $\mu\text{m}$  or less.

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**[0030]** In a situation in which a sealing test has been performed according to the method stipulated in JIS H8683-2:2013, the mass loss per unit of area of the aluminum material is preferably 0.3  $\text{g}/\text{dm}^2$  or less. Because the pores in the oxide layer are sufficiently sealed by the hydrated oxide layer, such an aluminum material can more reliably increase the corrosion resistance of the aluminum material.

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**[0031]** It is noted that a specific method of the sealing test is as follows. First, 35 mL of phosphoric acid and 20 g of anhydrous chromic acid are dissolved in water to prepare 1 L of a test solution. Next, a test piece, which includes the protective film, is extracted from the aluminum material, and the surface area of the protective film on the test piece is measured. After contamination on the surface of this test piece has been removed, the mass of the test piece is measured. Subsequently, the test piece is immersed for 15 min  $\pm$  5 s in the test solution, which is held at a temperature of 38°C  $\pm$  1°C.

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**[0032]** After immersion of the test piece in the test solution has been completed, the test piece is rinsed with running water and then further rinsed with deionized water or distilled water. Following the rinsing and after the test piece has been thoroughly dried, the mass of the test piece is measured.

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**[0033]** The surface area  $A$  (unit:  $\text{dm}^2$ ) of the protective film of the test piece obtained as described above, the mass  $m_1$  (unit: g) of the test piece before immersion in the test solution, and the mass  $m_2$  (unit: g) of the test piece after immersion in the test solution can be used to calculate the mass loss  $\delta_A$  per unit of area (unit:  $\text{g}/\text{dm}^2$ ) based on Equation (1) below.

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$$\delta_A = (m_1 - m_2)/A \quad (1)$$

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**[0034]** Surface-treated aluminum materials according to the first aspect have the characteristic that, in the situation in which cathodic polarization measurements are performed—using a measurement solution obtained by mixing a solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% such that the

volumetric ratio of NaCl solution:acetic acid = 1000:1—on the base material and on the surface-treated aluminum material after it had been heated at a temperature of 200°C for 4 hours, and the current density at the electric potential at the center of an electric-potential region of the base material that exhibits the diffusion-limited current of hydrogen ions was measured, the ratio  $J1/J2$  of the current density  $J1$  of the surface-treated aluminum material to the current density  $J2$  of the base material is  $150 \times 10^{-5}$  or less. Aluminum materials having a ratio  $J1/J2$  of the current density  $J1$  within the above-mentioned specific range have the characteristic that cracks form less readily when the temperature has risen. For this reason, aluminum materials provided with the protective film and having the current-density ratio  $J1/J2$  within the above-mentioned specific range excel in both corrosion resistance and heat resistance. From the viewpoint of increasing the heat resistance of the aluminum material, there is no lower limit for the current-density ratio  $J1/J2$ ; thus, given that definition, the current-density ratio  $J1/J2$  is definitely a value greater than 0.

**[0035]** In addition, surface-treated aluminum materials according to the second aspect have the characteristic that a sample having the protective film on one surface of the base material was prepared from the surface-treated aluminum material and measured in a state in which a strain gauge is mounted on the sample on a rear surface of the protective film, the difference  $\varepsilon1 - \varepsilon2$  between the amount  $\varepsilon1$  of strain of the sample at a temperature of 200°C and the amount  $\varepsilon2$  of strain of the base material at a temperature of 200°C is  $100 \times 10^{-6}$  or less. Aluminum materials in which the difference  $\varepsilon1 - \varepsilon2$  of the amounts of strain are within the above-mentioned specific range have the characteristic that cracks form less readily when the temperature has risen. For this reason, aluminum materials provided with the protective film and having a difference in the amounts of strain within the above-mentioned specific range excel in both corrosion resistance and heat resistance. It is noted that the lower limit of the difference  $\varepsilon1 - \varepsilon2$  in the amounts of strain of the sample prepared from the aluminum material usually becomes  $-100 \times 10^{-6}$  or more.

**[0036]** The method of preparing the sample from the surface-treated aluminum material is not particularly limited, and various methods can be employed as long as the method chosen does not affect the amount of strain in the sample. In the situation in which the surface-treated aluminum material is, for example, a sheet having the protective film on only one surface of the base material, a small piece of the surface-treated aluminum material that is cut to an appropriate size can be used as the sample. In addition, in the situation in

which the surface-treated aluminum material is, for example, a sheet that has the protective film on both surfaces of the base material, the sample can be obtained by cutting the surface-treated aluminum material to an appropriate size to prepare a small piece and then removing one of the two protective films from the small piece. A method of, for example, dissolving the protective film using an acid or an alkali, or the like can be employed as a method of removing the protective film.

**[0037]** As described above, the aluminum materials excel in corrosion resistance with regard to corrosive gases, plasma, and the like, and the formation of cracks in the protective film can be curtailed even in the situation in which the temperature has risen. For this reason, the aluminum materials are suitable for applications such as materials for covers provided around fans of heating and cooking equipment, materials for semiconductor-manufacturing apparatuses, and the like. More specifically, the aluminum materials are suitable for use in, for example: chambers in semiconductor-manufacturing apparatuses, such as film-forming apparatuses and etching apparatuses; components disposed within such chambers; and the like. Examples of film-forming apparatuses include physical vapor deposition (PVD) apparatuses and chemical vapor deposition (CVD) apparatuses. In addition, examples of etching apparatuses include dry-etching apparatuses and the like.

#### **[0038]** (Method of Manufacturing the Aluminum Material)

To manufacture the surface-treated aluminum material, first, a base material composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less is prepared. The method of manufacturing the base material is not particularly limited, and known methods can be employed. The base material may be manufactured, for example, by a method in which casting, rolling, and heat treatment(s) are combined as appropriate. In addition, after the base material has been manufactured and before performing the anodizing treatment, pretreatments of the anodizing treatment, such as degreasing, acid cleaning, and grinding, may be performed, as necessary. Next, the oxide layer, which has pores, is formed on the base material by performing the anodizing treatment on the base material. The oxide layer can be formed on the surface(s) of the base material during the anodizing treatment by flowing a direct current between the base material and a counter electrode in a state in which the base material and the counter electrode are immersed in an electrolyte solution. The oxide layer thus formed is constituted from an oxide or oxides of aluminum, such as alumina, and has numerous pores.

**[0039]** The electrolyte solution employed in the anodizing treatment may be, for example, an acidic electrolyte solution containing an electrolyte, such as sulfuric acid, phosphoric acid, or the like, or may be an alkaline electrolyte solution containing an electrolyte, such as sodium metaborate. The electrolyte solution employed in the anodizing treatment preferably contains an inorganic electrolyte composed of an inorganic cation and one or two or more anions selected from the group consisting of a sulfate ion, a phosphate ion, an ammonium ion, and a borate ion. An oxide layer having a desired structure can be more easily formed by performing the anodizing treatment using an electrolyte solution that contains an inorganic electrolyte.

**[0040]** The current density of the direct current in the anodizing treatment can be set, for example, in a range of 1 mA/cm<sup>2</sup> or more and 100 mA/cm or less, as appropriate. In addition, the temperature of the electrolyte solution in the anodizing treatment can be set, for example, in a range of 0°C or higher and 40°C or lower, as appropriate.

**[0041]** The thickness of the oxide layer formed in the anodizing treatment is preferably 2 μm or more. By making the thickness of the oxide layer 2 μm or more, the thickness of the protective film obtained after sealing can be made sufficiently thick, and an aluminum material that excels in corrosion resistance and in heat resistance can be more easily obtained.

**[0042]** In the above-mentioned method of manufacturing, after the anodizing process has been performed, the base material and the oxide layer are heated at a temperature of 50°C or higher and 350°C or lower. By heating the oxide layer at a temperature within the above-mentioned specific range after the anodizing process has been performed and before sealing the pores in the oxide layer, internal stresses in the oxide layer can be relaxed. Then, by sealing the pores after the internal stresses of the oxide layer were relaxed, the internal stresses in the protective film after sealing can be reduced. As a result, the formation of cracks in the protective film when heated can be curtailed, and aluminum materials that excel in heat resistance can be obtained.

**[0043]** In the situation in which the heating temperature of the oxide layer is lower than 50°C, there is a risk that the relaxation of the internal stresses in the oxide layer might be

insufficient, and cracks might form in the protective layer more readily when the temperature of the aluminum material rises. On the other hand, in the situation in which the heating temperature of the oxide layer exceeds 350°C, there is a risk that the oxide layer might be unable to follow the thermal expansion of the base material, and cracks might form in the protective film. When heating the oxide layer, the heating may be ended immediately after the temperature of the oxide layer has reached a desired temperature, or that temperature may be maintained for a certain extent of time after the desired temperature of the oxide layer has been reached. From the viewpoint of sufficiently relaxing the internal stresses in the oxide layer and more reliably increasing the heat resistance of the aluminum material, the heating time from when heating of the oxide layer is started to when heating is ended is preferably 1 min or more and less than 12 hours.

**[0044]** After the oxide layer has been heated, the oxide layer is contacted with a sealing agent. Thereby, a hydrated oxide layer is formed on the oxide layer, and the pores in the oxide layer are sealed by the hydrated oxide layer. For example, a substance, such as hot water, that can react with an oxide or oxides of aluminum and form a hydrated oxide or hydrated oxides can be used as the sealing agent. In the situation in which the sealing process is performed using hot water, a hydrated oxide layer composed of the hydrated oxide(s) of aluminum can be formed on the oxide layer.

**[0045]** In addition, for example, a substance, such as aqueous nickel acetate, aqueous cobalt acetate, aqueous chromate, and aqueous silicate, which can react with an oxide or oxides of aluminum and form a hydrated oxide or hydrated oxides and a metal salt or metal salts, can be used as the sealing agent. In the situation in which sealing is performed using such a sealing agent, a hydrated oxide layer containing the hydrated oxide(s) of aluminum and the metal salt(s) can be formed on the oxide layer.

**[0046]** From the viewpoint of more easily obtaining an aluminum material that excels in corrosion resistance and heat resistance, the sealing agent is preferably hot water. In addition, by sealing the pores in the oxide layer with hot water, a hydrated oxide layer that does not contain a metal salt or metal salts can be formed on the oxide layer. In the situation in which, for example, the aluminum material is used as a material for a semiconductor-manufacturing apparatus, a metal salt in the hydrated oxide layer might become the cause for contamination of the interior of the apparatus. Accordingly, by using hot water as the sealing

agent and forming a hydrated oxide layer that does not contain a metal salt, an aluminum material that is suitable for use as a material for a semiconductor-manufacturing apparatus can be easily obtained. In the situation in which hot water is used as the sealing agent, the pores in the oxide layer are more preferably sealed by contacting the oxide layer with hot water that is 95°C or higher for 10 min or more and less than 120 min.

[Working Examples]

**[0047]** (Working Example 1)

Working examples of a surface-treated aluminum material and a method of manufacturing the same will be described with reference to FIG. 1 to FIG. 2. As shown in FIG. 1, the surface-treated aluminum material 1 of the present example comprises: a base material 2, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less; and a protective film 3, which is formed on the base material. The protective film 3 comprises: an oxide layer 31, which is composed of an oxide or oxides of aluminum and covers the base material 2; and a hydrated oxide layer 32, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer 31. In the situation in which cathodic polarization measurements are performed—using a measurement solution obtained by mixing a solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% such that the volumetric ratio of the NaCl solution:acetic acid = 1000:1—on the base material 2 and on the surface-treated aluminum material 1 after it had been heated for 4 hours at a temperature of 200°C, and the current density at the electric potential at the center of an electric-potential region of the base material 2 that exhibits the diffusion-limited current of hydrogen ions was measured, the ratio  $J1/J2$  of the current density  $J1$  of the surface-treated aluminum material 1 to the current density  $J2$  of the base material 2 is  $150 \times 10^{-5}$  or less.

**[0048]** To manufacture the aluminum material 1 of the present example, an oxide layer 31, which has pores 311, is first formed on the base material 2, as shown in FIG. 2, by performing an anodizing treatment on the base material 2. Thereafter, the base material 2 and the oxide layer 31 are heated at a temperature of 50°C or higher and 350°C or lower and internal stresses in the oxide layer 31 are relaxed. Thereafter, the aluminum material 1 can be obtained by contacting the oxide layer 31 with the sealing agent, and forming a hydrated oxide layer 32 on the oxide layer 31 while sealing the pores 311.

**[0049]** In Table 1, specific examples of the aluminum material 1 (Test Materials A1–A16) are shown. The method of manufacturing Test Materials A1–A16 is, for example, as follows. First, aluminum sheets having the chemical compositions indicated by each of the alloy numbers listed in Table 1 and having a thickness of 1.1 mm are prepared as the base material 2. Pretreatments of the anodizing treatment are performed on these base materials 2. Specifically, as the pretreatments, an alkaline etching treatment is first performed in which the base material 2 is immersed in an aqueous solution of sodium hydroxide having a concentration of 5 mass% and a temperature of 55°C. Subsequently, a desmutting treatment is performed by immersing the base material 2 in nitric acid having a concentration of 30 mass%. Subsequently, a chemical polishing process is performed by immersing the base material 2 in a mixed solution in which phosphoric acid and sulfuric acid are mixed at a temperature of 85°C and a volumetric ratio of phosphoric acid:sulfuric acid = 7:3. After the chemical polishing process, a desmutting treatment is performed again under the same conditions described above.

**[0050]** After the pretreatments have been performed on the base material 2 as described above, an anodizing treatment is performed on the base material 2, and the oxide layer 31 forms on the surface of the base material 2. The electrolyte solution used in the anodizing treatment is an aqueous solution of sulfuric acid having a concentration of 15 mass%, and the temperature of the electrolyte solution is 5°C. In addition, the current density in the anodizing treatment is 10 mA/cm<sup>2</sup>, and the treatment time is 60 min. The oxide layer 31 thus formed is a so-called porous-type alumite film and, as shown in FIG. 2, has numerous pores 311. It is noted that the thickness of the oxide layer 31 formed by performing the anodizing treatment under the conditions described above is approximately 15 μm.

**[0051]** After having performed the anodizing treatment, the base material 2 is heated inside a heating furnace and the internal stresses in the oxide layer 31 are relaxed. The set temperature of the heating furnace is the value shown in the "Heating Temperature" column in Table 1, the residence time of the base material inside the furnace, that is, the time from the start of heating to the end of heating, is the value shown in the "Heating Time" column in Table 1.

**[0052]** Thereafter, by immersing the base material 2, which has the oxide layer 31, in hot water as the sealing agent at a temperature of 100°C for 60 min, the hydrated oxide layer

32, which is composed of the hydrated oxide(s) of aluminum, forms on the oxide layer 31, and the pores 311 in the oxide layer 31 are sealed by the hydrated oxide layer 32. Test Materials A1–A16 shown in Table 1 could thereby be obtained based on the above. It is noted that, in the situation in which the pores 311 in the oxide layer 31 are sealed under such conditions, the mass loss per unit of area of the aluminum material 1 became 0.3 g/dm<sup>2</sup> or less in the situation in which a sealing test was performed using the method stipulated in JIS H8683-2:2013.

**[0053]** It is noted that Test Materials B1–B7 shown in Table 1 are test materials for comparison with Test Materials A1–A16. The method of manufacturing Test Materials B1–B6 is the same as the method of manufacturing Test Materials A1–A16 except that, after the oxide layer 31 was formed on the base material 2, the oxide layer 31 is contacted with the sealing agent without being heated. In addition, the method of manufacturing Test Material B7 is the same as the method of manufacturing Test Material A1 except that the heating conditions in the heating furnace were modified as shown in Table 1.

**[0054]** Next, the method of measuring the cathodic polarization of Test Materials A1–A16 and Test Materials B1–B7 will be explained.

#### **[0055]** (Cathodic Polarization Measurement)

Using the following method, cathodic polarization measurements are performed on the base material and on the test material that had been heated for 4 hours at a temperature of 200°C; based on the resulting cathodic polarization curves, the ratio J1/J2 of the current density J1 of the surface-treated aluminum material to the current density J2 of the base material is calculated. First, the test material is heated for 4 hours in an oven set to a temperature of 200°C. After removing the test material from the oven and cooling to room temperature, an evaluation region was established on the protective film, and portions other than the evaluation region on the surface of the test material are covered with silicone resin.

**[0056]** Next, an aqueous solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% are prepared, and a measurement solution is prepared by adding the acetic acid to the aqueous solution of NaCl such that the volumetric ratio of aqueous solution of NaCl to the acetic acid is an aqueous solution of NaCl:acetic acid = 1000:1. A test piece, a counter electrode, and a reference electrode, which are electrically

connected to a potentiostat, are immersed in this measurement solution and left standing for 30 min to stabilize the electric potential of the measurement area. It is noted that degassing of the measurement solution is not performed. In addition, for example, an Ag/AgCl electrode can be used as the reference electrode.

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**[0057]** After the electric potential of the measurement area had stabilized, a voltage is applied between the test piece and the counter electrode using the potentiostat, and the electric potentials of the measurement area are swept at a sweep rate of 20 mV/min until the electric potentials of the measurement area reached  $-2,000$  mV relative to the reference electrode. A cathodic polarization curve for the post-heated test material is obtained by measuring the current density flowing in the measurement area at this time. In addition, a cathodic polarization curve for the base material is obtained by performing a similar measurement using the base material after the pretreatment of the anodizing treatment had been performed using the method described above. It is noted that the cathodic polarization measurements of both the test materials and the base materials are performed in the state in which the temperature of the measurement solution was maintained at  $25^{\circ}\text{C}$  in ambient atmosphere. In addition, the cathodic polarization measurements of the test materials and the base materials are performed in the state in which the measurement solution was not agitated and the measurement solution was substantially not flowing.

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**[0058]** One example of the cathodic polarization curve of the base material is shown in FIG. 3. It is noted that, in FIG. 3, the ordinate is the electric potential of the measurement area (unit: V), and the abscissa is the current density (unit:  $\mu\text{A}/\text{cm}^2$ ). In addition, the scale of the abscissa in FIG. 3 is a logarithmic scale. As shown in FIG. 3, the cathodic polarization curve of the base material has a step shape. During the cathodic polarization measurement, the change in the electric current becomes smaller relative to the change in the electric potential of the measurement areas as the electric current approaches the state in which it becomes rate limited owing to the diffusion of hydrogen ions. Accordingly, as shown in FIG. 3, in the cathodic polarization curve in which the electric potential is represented by the ordinate and the current density is represented by the abscissa, the electric-potential region that exhibits the diffusion-limited current of the hydrogen ions contains a portion in which the slope of the curve is steep at a step portion of the cathodic polarization curve.

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**[0059]** An enlarged view of the step portion in the cathodic polarization curve in FIG. 3 is shown in FIG. 4. The following is the method of determining the electric-potential region that exhibited the diffusion-limited current of the hydrogen ions in the cathodic polarization curve of the base material. First, a tangent L is drawn at the step portion of the cathodic polarization curve at which the absolute value of the slope is the largest as shown in FIG. 4. Then, region R where this tangent L and the cathodic polarization curve overlap is taken as the electric-potential region that represents the diffusion-limited current of the hydrogen ions. Current density J2 is calculated at the electric potential at the center of region R determined in this manner. In addition, in the cathodic polarization curve of the post-heated test material, current density J1 is calculated at the electric potential the same as the electric potential at the center of the electric-potential region in the cathodic polarization curve of the base material as described above.

**[0060]** Current density J1 calculated based on the cathodic polarization curve of the post-heated test material can be used as an indicator of the contact-surface area between the base material of the post-heated test material and the measurement solution; the higher the current density value, the greater the contact-surface area between the base material and the measurement solution. Accordingly, the ratio J1/J2 of the current density J1, calculated using the post-heated test piece, to the current density J2, calculated using the base material, can be used as an indicator of the rate of increase in the surface area of the base material that was exposed by heating. More specifically, in the situation in which, for example, defects, such as cracks, are formed in the protective film of the post-heated test material, then the base material is sometimes exposed by the cracks. Accordingly, the current-density ratio J1/J2 becomes large in this situation. In Table 1, the current-density ratio J1/J2 for each test material is shown.

**[0061]** [Table 1]

(Table 1)

|                  | Base Material | Heating of Oxide Layer   |                     | Current-Density Ratio J1/J2 |
|------------------|---------------|--------------------------|---------------------|-----------------------------|
|                  |               | Heating Temperature (°C) | Heating Time (Mins) |                             |
| Test Material A1 | AA6016        | 50                       | 60                  | $59 \times 10^{-5}$         |
| Test Material A2 | AA6016        | 100                      | 60                  | $100 \times 10^{-5}$        |
| Test Material A3 | AA6016        | 200                      | 60                  | $84 \times 10^{-5}$         |

|                   | Base Material | Heating of Oxide Layer   |                     | Current-Density Ratio J1/J2 |
|-------------------|---------------|--------------------------|---------------------|-----------------------------|
|                   |               | Heating Temperature (°C) | Heating Time (Mins) |                             |
| Test Material A4  | AA6016        | 250                      | 60                  | $9 \times 10^{-5}$          |
| Test Material A5  | AA6016        | 300                      | 60                  | $2 \times 10^{-5}$          |
| Test Material A6  | AA6016        | 250                      | 1                   | $38 \times 10^{-5}$         |
| Test Material A7  | AA6016        | 250                      | 5                   | $12 \times 10^{-5}$         |
| Test Material A8  | AA6016        | 250                      | 10                  | $7 \times 10^{-5}$          |
| Test Material A9  | AA6016        | 250                      | 20                  | $5 \times 10^{-5}$          |
| Test Material A10 | AA6016        | 250                      | 30                  | $5 \times 10^{-5}$          |
| Test Material A11 | AA6016        | 250                      | 45                  | $7 \times 10^{-5}$          |
| Test Material A12 | AA1050        | 250                      | 30                  | $1 \times 10^{-5}$          |
| Test Material A13 | AA3003        | 250                      | 30                  | $29 \times 10^{-5}$         |
| Test Material A14 | AA5052        | 250                      | 30                  | $36 \times 10^{-5}$         |
| Test Material A15 | AA6061        | 250                      | 30                  | $1 \times 10^{-5}$          |
| Test Material A16 | AA8021        | 250                      | 30                  | $18 \times 10^{-5}$         |
| Test Material B1  | AA6016        | No heating               |                     | $555 \times 10^{-5}$        |
| Test Material B2  | AA1050        | No heating               |                     | $1893 \times 10^{-5}$       |
| Test Material B3  | AA3003        | No heating               |                     | $1858 \times 10^{-5}$       |
| Test Material B4  | AA5052        | No heating               |                     | $1806 \times 10^{-5}$       |
| Test Material B5  | AA6061        | No heating               |                     | $908 \times 10^{-5}$        |
| Test Material B6  | AA8021        | No heating               |                     | $153 \times 10^{-5}$        |
| Test Material B7  | AA6016        | 400                      | 30                  | $2079 \times 10^{-5}$       |

**[0062]** As shown in Table 1, when Test Materials A1–A16 are being manufactured, after the oxide layer had formed on the base material, the oxide layer is heated at a temperature within the above-mentioned specific range before sealing the pores in the oxide layer. Consequently, the current-density ratio J1/J2 of each of these test materials is in the above-mentioned specific range, and the formation of cracks in the protective film could be curtailed even in the situation in which the temperature had risen. In addition, these test materials excelled in corrosion resistance with respect to corrosive gas and plasma because the oxide layer of the protective film was sealed by the hydrated oxide layer.

**[0063]** In contrast, when Test Materials B1–B6 are being manufactured, after the oxide layer had formed on the base material, the pores are sealed without heating the oxide layer. Consequently, the current-density ratio J1/J2 of each of these test materials is higher than the above-mentioned specific range, and therefore cracks tend to form in the situation in which the temperature has risen.

**[0064]** When Test Material B7 was being manufactured, the heating temperature when the oxide layer was heated was too high. Consequently, the current-density ratio  $J1/J2$  of Test Material B7 is higher than the above-mentioned specific range, and cracks tend to form in the situation in which the temperature has risen.

**[0065]** (Working Example 2)

In the present example, an example of a situation in which the oxide layer is heated at an even higher temperature during the process of manufacturing the aluminum material will be described. Although not shown in the drawings, the aluminum material of the present example comprises: the base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less; and a protective film, which is formed on the base material. The protective film comprises: an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and the hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer. In Table 2, specific examples of the aluminum material of the present example (Test Material A17 and Test Material A18) are shown. The method of manufacturing Test Material A17 and Test Material A18 is the same as the method of manufacturing Test Materials A1–A16 in Working Example 1 except that, when heating the oxide layer, the set temperature of the heating furnace and the in-furnace residence time of the base material are modified as shown in Table 2.

**[0066]** In Table 2, the current-density ratios  $J1/J2$  of Test Material A17 and Test Material A18, respectively, are shown, each of which was based on cathodic polarization measurements. It is noted that the measuring method of the cathodic polarization measurement in the present example is the same as the measuring method of the cathodic polarization measurement in Working Example 1 except that the heating temperature for the test materials employed in the measurement was modified to 250°C.

**[0067]** [Table 2]  
(Table 2)

|                   | Base Material | Heating of Oxide Layer   |                     | Current-Density Ratio J1/J2 |
|-------------------|---------------|--------------------------|---------------------|-----------------------------|
|                   |               | Heating Temperature (°C) | Heating Time (Mins) |                             |
| Test Material A17 | AA6016        | 350                      | 30                  | $17 \times 10^{-5}$         |
| Test Material A18 | AA6016        | 300                      | 30                  | $32 \times 10^{-5}$         |

**[0068]** As shown in Table 2, the current-density ratios J1/J2 of Test Material A17 and of Test Material A18 are within the above-mentioned specific range even in the situation in which the test materials are heated at the temperature of 250°C. Typically, cracks tend to form more readily in the protective film as the heating temperature increases; therefore, the current-density ratio J1/J2 at a temperature of 200°C would conceivably be equal to or less than the current-density ratio J1/J2 in the situation in which the protective film were heated at a temperature of 250°C. Accordingly, Test Material A17 and Test Material A18 would conceivably have current-density ratios J1/J2 within the above-mentioned specific range even in the situation in which heating was performed at a temperature of 200°C, and thus the formation of cracks in the protective film could be curtailed even in the situation in which the temperature had risen.

**[0069]** (Working Example 3)

In the present example, measurements of the amounts of strain in aluminum materials having the protective film on the base material will be explained. It is noted that, among the symbols used in the present example, unless particularly explained, the symbols that are the same as the symbols used in previously discussed examples indicate structural elements, etc. the same as structural elements, etc. in the previously discussed examples.

**[0070]** To measure the amount of strain in the aluminum material 1 having the protective film 3, the base material 2 of the aluminum material 1 is exposed on the rear surface of the surface having the protective film 3, as shown in FIG. 5. Then, a strain gauge 4 is mounted on the exposed base material 2. The strain due to thermal expansion of the base material 2 and the protective film 3 can be measured by heating the aluminum material 1 on which the strain gauge 4 was mounted in the aforementioned manner. It is noted that the structure of the protective film 3 is simplified in FIG. 5 for the sake of convenience of explanation.

**[0071]** In FIG. 6, changes in the amounts of strain in Test Material A4, Test Material A6, and Test Material B1 of Working Example 1 are shown in the situation in which the test materials were heated for 30 min using the heating furnace, which was set to a temperature of 200°C. In FIG. 6, the ordinate is the amount of strain and the abscissa is the time elapsed since the start of heating. It is noted that, for comparison, in FIG. 6 the change in the amount of strain is shown in the situation in which the base material 2, which had no protective film 3, was heated for 30 min using the heating furnace, which was set to a temperature of 200°C. Although not shown in the drawing, to measure the amount of strain in the base material 2, which has no protective film 3, after the strain gauge has been mounted on one of the surfaces in the thickness direction of the base material 2, the base material 2 should be heated.

**[0072]** Because the test materials immediately after the start of heating underwent thermal expansion commensurate with the temperature rise, as shown in FIG. 6, the respective amounts of strain increased sharply from the point in time when the test started up until several minutes elapsed thereafter. Thereafter, when the temperatures of the test material and the base material reached a roughly constant temperature, the respective amounts of strain of the test material and the base material became roughly constant values.

**[0073]** In Table 3, the maximum value of the amounts of strain while the test materials and the base materials were being heated is shown. In addition, value  $\varepsilon_1 - \varepsilon_2$  resulting from subtracting the maximum value  $\varepsilon_2$  of the amount of strain of the base material from the maximum value  $\varepsilon_1$  of the amount of strain of the corresponding test material is shown in Table 3. The difference  $\varepsilon_1 - \varepsilon_2$  between the amount  $\varepsilon_1$  of strain of the corresponding test material and the amount  $\varepsilon_2$  of strain of the base material indicates the magnitude of the internal stresses in the protective film released by the heating during the test; this means that the smaller the difference  $\varepsilon_1 - \varepsilon_2$  of the amounts of strain, the smaller the internal stresses in the protective film.

**[0074]** [Table 3]  
(Table 3)

|                  | Protective Film | Heating of Oxide Layer                     |                     | Maximum Value of Strain Amount | Difference Between Strain Amounts $\epsilon_1 - \epsilon_2$ |
|------------------|-----------------|--------------------------------------------|---------------------|--------------------------------|-------------------------------------------------------------|
|                  |                 | Heating Temperature ( $^{\circ}\text{C}$ ) | Heating Time (Mins) |                                |                                                             |
| Test Material A4 | Present         | 250                                        | 60                  | $2298 \times 10^{-6}$          | $-11 \times 10^{-6}$                                        |
| Test Material A6 | Present         | 250                                        | 1                   | $2380 \times 10^{-6}$          | $71 \times 10^{-6}$                                         |
| Test Material B1 | Present         | No heating                                 |                     | $2461 \times 10^{-6}$          | $152 \times 10^{-6}$                                        |
| Base material    | Not present     | No heating                                 |                     | $2309 \times 10^{-6}$          | —                                                           |

**[0075]** As shown in Table 3, the amounts of strain of Test Material A4 and Test Material A6, on which the hydrated oxide layer is formed after the oxide layer is heated during the manufacturing process for the test material, are smaller than the amount of strain of Test Material B1, on which the hydrated oxide layer is formed without heating the oxide layer. Accordingly, from these results, it can be understood that, by forming the hydrated oxide layer after heating the oxide layer during the process of manufacturing the aluminum material, the internal stresses in the protective film can be relaxed.

**[0076]** In addition, comparing each of Test Material A4 and Test Material A6 with Test Material B1, it can be understood that, with regard to an aluminum material in which the difference  $\epsilon_1 - \epsilon_2$  between the amount  $\epsilon_1$  of strain of the sample at a temperature of  $200^{\circ}\text{C}$  and the amount  $\epsilon_2$  of strain of the base material at a temperature of  $200^{\circ}\text{C}$ , measured in the state in which the strain gauge was mounted on the test material on a rear surface of the protective film, was  $100 \times 10^{-6}$  or less, the internal stresses in the protective film were low and thus the aluminum material excelled at heat resistance.

**[0077]** Aspects of the surface-treated aluminum material and the method of manufacturing the same according to the present invention were described above based on the working examples; however, the specific aspects of the surface-treated aluminum material and the method of manufacturing the same according to the present invention are not limited to the aspects in the working examples, and the constitutions thereof can be modified, as appropriate, within a scope that does not depart from the gist of the present invention.

**[0078]** For example, the surface-treated aluminum material according to the present invention can obtain the aspects according to [1]–[3] below.

**[0079]** [1] A surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less, and a protective film, which is formed on the base material, wherein: the protective film comprises:

5 an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

10 in the situation in which cathodic polarization measurements are performed—using a measurement solution obtained by mixing a solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% such that the volumetric ratio of the NaCl solution to the acetic acid = 1000:1—on the base material and on the surface-treated aluminum material after it had been heated at a temperature of 200°C for 4 hours, and the current density at the electric potential at the center of an electric-potential region of the base  
15 material that exhibits the diffusion-limited current of hydrogen ions was measured, the ratio  $J_1/J_2$  of the current density  $J_1$  of the surface-treated aluminum material to the current density  $J_2$  of the base material is  $150 \times 10^{-5}$  or less.

**[0080]** [2] A surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less, and a protective film, which is formed on the base material, wherein: the protective film comprises:

20 an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

25 a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

a sample having the protective film on one surface of the base material was prepared from the surface-treated aluminum material and measured in a state in which a strain gauge is mounted on the sample on a rear surface having the protective film, the difference  $\varepsilon_1 - \varepsilon_2$   
30 between the amount  $\varepsilon_1$  of strain of the sample at a temperature of 200°C and the amount  $\varepsilon_2$  of strain of the base material at a temperature of 200°C is  $100 \times 10^{-6}$  or less.

**[0081]** [3] The surface-treated aluminum material according to [1] or [2], wherein the mass loss per unit of area is  $0.3 \text{ g/dm}^2$  or less in the situation in which a sealing test has been performed using the method stipulated in JIS H8683-2:2013.

5 **[0082]** In addition, a material for a semiconductor-manufacturing apparatus according to the present invention can obtain the aspect according to [4] below.

[4] A material for a semiconductor-manufacturing apparatus composed of the surface-treated aluminum material according to [1] or [2].

10 **[0083]** In addition, a method of manufacturing the surface-treated aluminum material according to the present invention can obtain the aspects according to [5]–[9] below.

**[0084]** [5] A method of manufacturing the surface-treated aluminum material according to any one of [1]–[3], comprising:

15 forming the oxide layer, which has pores, on the base material by performing an anodizing treatment on the base material;  
thereafter, heating the base material and the oxide layer at a temperature of  $50^\circ\text{C}$  or higher and  $350^\circ\text{C}$  or lower; and  
thereafter, contacting the oxide layer with a sealing agent and forming the hydrated  
20 oxide layer on the oxide layer while sealing the pores.

**[0085]** [6] The method of manufacturing the surface-treated aluminum material according to [5], wherein, during the heating, the heating time from the start of heating the oxide layer to the end of heating is 1 min or more and less than 12 hours.

25 **[0086]** [7] The method of manufacturing the surface-treated aluminum material according to [5] or [6], wherein the sealing agent is hot water.

**[0086]** [8] The method of manufacturing the surface-treated aluminum material according to any one of [5]–[7], wherein, during the sealing, the oxide layer is contacted with  
30 hot water as the sealing agent that is  $95^\circ\text{C}$  or higher for 10 min or more and less than 120 min.

[9] The method of manufacturing the surface-treated aluminum material according to any one of [5]–[8], wherein the electrolyte solution employed in the anodizing process contains an inorganic electrolyte composed of an inorganic cation and one or two or more

anions selected from the group consisting of a sulfate ion, a phosphate ion, an ammonium ion, and a borate ion.

## [Document Name] SCOPE OF CLAIMS

[Claim 1] A surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass%

5 or less, and a protective film, which is formed on the base material, wherein:

the protective film comprises:

an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

10 a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

in the situation in which cathodic polarization measurements are performed—using a measurement solution obtained by mixing a solution of NaCl having a concentration of 5 mass% and acetic acid having a concentration of 99.7% such that the volumetric ratio of the NaCl solution to the acetic acid = 1000:1—on the base material and the surface-treated aluminum material after it had been heated at a temperature of 200°C for 4 hours, and the current density at the electric potential at the center of an electric-potential region of the base material that exhibits the diffusion-limited current of hydrogen ions was measured, the ratio  $J_1/J_2$  of the current density  $J_1$  of the surface-treated aluminum material to the current density  $J_2$  of the base material is  $150 \times 10^{-5}$  or less.

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[Claim 2] A surface-treated aluminum material having a base material, which is composed of aluminum or an aluminum alloy in which the Cu content is 0 mass% or more and 1.8 mass% or less, and a protective film, which is formed on the base material, wherein:

the protective film comprises:

25 an oxide layer, which is composed of an oxide or oxides of aluminum and covers the base material; and

a hydrated oxide layer, which contains a hydrated oxide or hydrated oxides of aluminum and covers the oxide layer; and

30 a sample having the protective film on one surface of the base material was prepared from the surface-treated aluminum material and measured in a state in which a strain gauge is mounted on the sample on a rear surface of the protective film, the difference  $\epsilon_1 - \epsilon_2$  between the amount  $\epsilon_1$  of strain of the sample at a temperature of 200°C and the amount  $\epsilon_2$  of strain of the base material at a temperature of 200°C is  $100 \times 10^{-6}$  or less.

[Claim 3] The surface-treated aluminum material according to claim 1 or 2, wherein the mass loss per unit of area is  $0.3 \text{ g/dm}^2$  or less in the situation in which a sealing test has been performed using a method stipulated in JIS H8683-2:2013.

- 5 [Claim 4] A material for a semiconductor-manufacturing apparatus composed of the surface-treated aluminum material according to any one of claims 1–3.

[Claim 5] A method of manufacturing the surface-treated aluminum material according to any one of claims 1–3, comprising:

- 10       forming the oxide layer, which has pores, on the base material by performing an anodizing treatment on the base material;  
          thereafter, heating the base material and the oxide layer at a temperature of  $50^\circ\text{C}$  or higher and  $350^\circ\text{C}$  or lower; and  
          thereafter, contacting the oxide layer with a sealing agent, and forming the hydrated  
15       oxide layer on the oxide layer while sealing the pores.

[Claim 6] The method of manufacturing the surface-treated aluminum material according to claim 5, wherein, during the heating, the heating time from the start of heating the oxide layer to the end of heating is 1 min or more and less than 12 hours.

- 20 [Claim 7] The method of manufacturing the surface-treated aluminum material according to claim 5 or 6, wherein the sealing agent is hot water.

- [Claim 8] The method of manufacturing the surface-treated aluminum material according to  
25 any one of claims 5–7, wherein, during the sealing, the oxide layer is contacted with hot water as the sealing agent that is  $95^\circ\text{C}$  or higher for 10 min or more and less than 120 min.

- [Claim 9] The method of manufacturing the surface-treated aluminum material according to any one of claims 5–8, wherein the electrolyte solution employed in the anodizing treatment  
30 contains an inorganic electrolyte composed of an inorganic cation and one or two or more anions selected from the group consisting of a sulfate ion, a phosphate ion, an ammonium ion, and a borate ion.