

DESCRIPTION

SPUTTERING TARGET

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TECHNICAL FIELD

The present invention relates to a sputtering target capable of shortening the burn-in time and performing deposition on a wafer or a substrate at a stable speed during sputtering.

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BACKGROUND ART

Heretofore, the reduction of particles and improvement of uniformity (uniformization of the film thickness and film resistance) were sought by controlling the crystal grain size and crystalline orientation of high-purity tantalum materials in a tantalum sputtering target. Examples of this are described below.

For instance, proposed is technology of providing a tantalum sputtering target, wherein the average crystal grain size is 0.1 to 300 μ m, variation of the average crystal grain size according to location is within $\pm 20\%$, the oxygen concentration is 50ppm or less, and, with respect to the impurity concentration, Na $\leq$ 0.1ppm, K $\leq$ 0.1ppm, U $\leq$ 1ppb, Th $\leq$ 1ppb, Fe $\leq$ 5ppm, Cr $\leq$ 5ppm, Ni $\leq$ 5ppm, and the total content of high-melting metal elements (Hf, Nb, Mo, W, Ti and Zr) is 50ppm or less. This tantalum sputtering target is able to improve the deposition speed by selectively increasing the plane orientation of {110},

{200} and {211} having a high atom density as the sputtering surface, and improve the uniformity by inhibiting the variation of the plane orientation (Patent Document 1).

Further, proposed is a tantalum sputtering target comprising a
5 crystal structure in which the (222) orientation was preferential from a position of 30% of the target thickness toward the center plane of the target for improving the uniformity throughout the sputtering life (Patent Document 2).

Patent Document 3 proposes a metal product and a sputtering
10 target comprising at least substantially 99.95wt% of tantalum and a (100) cubic texture of that the surface is substantially uniform, and having a maximum grain size of 50 microns or less. This technology is useful as a metal product having a fine and uniform texture, particularly as a sputtering target.

15 Further, in order to reduce the particles in the initial stage of sputtering and obtain a uniform film (uniformity), it is important to reduce the surface roughness and eliminate a work-affected layer (residual stress layer) on the surface of the sputtering target. An example of this is shown in Patent Document 4.

20 Patent Document 4 describes that it is possible to reduce the generation of particles by substantially eliminating the machining defect layer (crushed layer) resulting from minute cracks and missing portions generated during the machining process at least on the surface portion of a sputtering target for a high-melting metal alloy. In order to realize this,
25 Patent Document 4 describes that it is important to refine the finish surface roughness (Ra of $0.05\mu\text{m}$ or less), and give consideration to

reducing the working steps in relation to the distribution of material defects in order to substantially eliminate the machining defect layer with cracks and dropout holes.

The surface treatment method reduces the abrasive grains and
5 refines the finish surface roughness through the steps of lapping, polishing and mechano-chemical polishing, and thereby reduces the residual stress caused by grinding.

Further, Patent Document 5 describes a sputtering target, wherein the surface roughness (R_a is $1.0\mu\text{m}$ or less), the total content of Si, Al,
10 Co, Ni, B and high-melting metal elements excluding the primary components and alloy components as contaminants is 500ppm or less, the hydrogen content on the surface is 50ppm or less, and the thickness of the work-affected layer is $50\mu\text{m}$ or less. Patent Document 5 further describes that, when necessary, the target is manufactured by precision
15 cutting with a diamond tool.

As described above, cutting work (in particular, diamond finishing-cut) and polishing processing (wet polishing or chemical processing) are performed in manufacturing a sputtering target, and it may be difficult to prevent the generation of nodules even if the surface roughness is adjusted when high deformation is
20 performed. This is assumed to be because when high deformation is performed, the atomic arrangement is disturbed and the angle of the particles discharged during sputtering becomes lower, and even if the surface roughness is low, namely, even if the asperity of the surface is small, it eases adherence of particles to the surface. Thus, it was
25 necessary to keep the thickness of the work-affected layer of the surface $50\mu\text{m}$ or less.

If high deformation is performed so that the thickness of the work-affected layer exceeds $50\mu\text{m}$, it is not possible to effectively reduce the number of nodules and particles.

This technology itself is effective. Nevertheless, it requires an
5 extremely long time to completely eliminate the work-affected layer (residual stress layer) of the target surface, and there are problems in low productivity and a waste of materials through cutting and grinding the target thickly.

Further, it is difficult to uniformly polish a large area (for instance,
10 up to around $\phi 450\text{mm}$) of a target or the like by chemical polishing such as etching, and the surface gloss often deteriorates by selectively etching the crystal grain boundary. In particular, tantalum having high chemical resistance must be polished with powerful hydrofluoric acid or sulfuric acid, and there is a problem in that the removal of the residual
15 liquid is difficult.

In the sputtering process, a conditioning process referred to as a burn-in is performed while subjecting a dummy wafer to a flow until the initial deposition becomes stable. As with this burn-in process, proposals have been made to perform reverse sputtering after the final
20 processing of the target in order to eliminate the work-affected layer so as to shorten the burn-in time when the target is used in the actual deposition process.

Nevertheless, these various methods require the same level of equipment as a sputtering device for manufacturing semiconductors.
25 Moreover, in these methods, the burn-in time is merely shifted to the target manufacturing process and it does not provide the comprehensive

solution for shortening the overall processing time.

[Patent Document 1] Japanese Patent Laid-Open Publication No.
H11-080942

5 [Patent Document 2] Japanese Patent Laid-Open Publication No.
2004-107758

[Patent Document 3] National Publication of Translated Version
2002-518593

[Patent Document 4] Japanese Patent Laid-Open Publication No.
10 H3-257158

[Patent Document 5] Japanese Patent Laid-Open Publication No.
H11-1766

DISCLOSURE OF THE INVENTION

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Thus, an object of the present invention is to provide a tantalum or a tantalum-based alloy target capable of shortening the burn-in time of a sputtering target and minimizing the fluctuation in the deposition speed throughout the target life, whereby the production efficiency of
20 semiconductors in the sputtering process can be improved and stabilized, and the production cost can be reduced.

In order to achieve the foregoing object, as a result of intense study concerning the influence that the various target materials has on the deposition speed, it was found that the {200} plane, which is the
25 crystalline orientation of the outermost surface (initial surface) of the target, significantly affects the burn-in time and the fluctuation in the

subsequent deposition speed.

Based on the foregoing discovery, the present invention provides:

1) A tantalum or a tantalum-based alloy sputtering target, wherein FWHM of a {200} crystal plane measured by X-ray diffraction of the sputtered outermost surface is 0.1 to 0.6. It enables to significantly shorten the burn-in time and inhibit changes in the subsequent deposition speed.

The foregoing conditions are the optimal conditions for achieving the present invention, which aims at significantly shortening the burn-in time and inhibiting changes in the subsequent deposition speed. Although there are preferable processing and heat treatment conditions for achieving these conditions, such conditions can be selected arbitrarily, and it should be understood that there is no reason for the present invention to be bound by these conditions. Further, as shown below, there are additional preferable conditions. Each of these preferable conditions is in itself a novel invention and is an improvement of the foregoing invention, and it should be understood that these additional preferable conditions do not limit the scope of the invention described in paragraph 1) above.

2) The sputtering target according to paragraph 1) above, wherein FWHM is 0.15 to 0.45. More preferable results can be obtained when FWHM is within the foregoing range.

3) The sputtering target according to paragraph 1) or paragraph 2) above, wherein the variation of FWHM is within ± 0.05 . It is desirable to keep the variation of FWHM as small as possible, and the fluctuation in the deposition speed can thereby be effectively inhibited.

4) The sputtering target according to any one of paragraphs 1) to 3) above, wherein FWHM of a {110} crystal plane is 0.25 to 0.4, and the variation of FWHM is within ± 0.05 . By adjusting FWHM of the {110} crystal plane, it is possible to further improve the effect of the present invention.

5) The sputtering target according to any one of paragraphs 1) to 4) above, wherein the depth of the processing strain of the tantalum or tantalum-based alloy sputtering target is a depth of $15\mu\text{m}$ from the target surface; and

6) The sputtering target according to paragraph 5) above, wherein the depth of the processing strain of the tantalum or tantalum-based alloy sputtering target is a depth of $10\mu\text{m}$ from the target surface.

The present invention yields superior effects of significantly reducing the initial burn-in time of a tantalum or a tantalum-based alloy sputtering target and minimizing the fluctuation in the deposition speed throughout the target life, whereby the production efficiency of semiconductors in the sputtering process can be improved and stabilized, and the production cost can be significantly reduced.

BEST MODE FOR CARRYING OUT THE INVENTION

The present invention is now explained in detail below.

FWHM (full width of half maximum) of a crystal plane measured by X-ray diffraction of the target only represents the internal stress contained in such crystal plane, and this results from the deformation processing such as forging or rolling during the target manufacture and

the processing strain during the machining process such as when cutting the target.

The larger FWHM, the larger the residual strain. Thus, a target has conventionally been manufactured so that residual strain formed on the surface can be reduced as much as possible by alleviating residual strain, which results from molding of the target shape through forging or rolling and texture control for refining and uniformizing the crystal grain size, with heat treatment, and by subjecting the target surface to precision machining.

The crystal structure of tantalum, as with vanadium, niobium, molybdenum, tungsten and the like, is of a body-centered cubicle (BCC) structure. Nevertheless, particularly with high-purity tantalum of 3N5 to 6N, since it is a soft material unlike tungsten, it is easy to perform deformation processing such as forging and rolling in a non-oxygenated atmosphere.

Like this, since it is possible to control the crystalline orientation through deformation processing, targets with various orientations have been proposed.

With a BCC structure having few equivalent crystal planes, even an inclination of a minor eroded surface will become a significant fluctuation as the plane orientation, and it is considered that the internal strain of the crystal plane will be largely affected.

For instance, Japanese Patent Laid-Open Publication No. 2003-49264 proposes a tungsten target wherein FWHM of the peak of the crystal plane {110} that is a close-packed plane obtained by X-ray diffraction in order to improve the uniformity of the tungsten film and

reduce the generation of particles is 0.35 or less.

Nevertheless, unlike this kind of tungsten target, in the case of a tantalum or a tantalum-based alloy, since the plastic deformation capacity is high as described above and rotation of the crystal plane will occur, FWHM of the close-packed plane {110} will not exert much influence, and it has been discovered that rather the half-width value of the {200} plane exerts significant influence.

Conventionally, processing strain on the surface was eliminated by performing pre-sputtering known as burn-in in the initial stage. Nevertheless, since conventional burn-in required much time, it caused the sputtering efficiency to deteriorate. This is because a method of adequately evaluating the processing strain of the target has not been established, and consequently a specific method of constantly reducing the processing strain of the target has not been established.

As described above, variation in FWHM of the crystal plane {200} of a tantalum or a tantalum-based alloy is caused by deformation processing such as forging and rolling and processing strain resulting from machining of the surface.

Therefore, it has been discovered that it is possible to reduce the processing strain of the target and shorten the burn-in time by appropriately controlling FWHM by reference to FWHM of the crystal plane {200} as an index, and further minimize the fluctuation in the deposition speed. This was conventionally unthinkable.

In order to stably control FWHM of the {200} plane of a tantalum or a tantalum-based alloy, it has been discovered that performing heat treatment at a relatively low temperature in a vacuum is especially

effective.

The reason FWHM of the {200} plane was made to be 0.1 to 0.6 (preferably 0.15 to 0.45) is because there is a problem in that the processing strain will become significant when FWHM exceeds 0.6.

5 This will affect and deteriorate the uniformity of the sputtered film. In order to improve the uniformity, there is a problem in that the burn-in must be performed for a long period of time when the processing strain is concentrated on the surface.

Tantalum is used as a barrier film by forming a tantalum nitride film
10 through reactive sputtering, and inferior film uniformity is considered to increase the residual stress (film stress) in the nitride film and must be avoided.

Contrarily, if FWHM is less than 0.1, the redeposition film will peel easily and increase the generation of particles when a film (redeposition
15 film) to be reattached to the target is formed. Further, when FWHM is less than 0.1, processing will become extremely difficult, and this is not realistic since an enormous amount of time will be required for the processing of finishing and the like.

Therefore, it is evident that controlling FWHM of the {200} plane of
20 a tantalum or a tantalum-based alloy to be 0.1 to 0.6 (preferably 0.15 to 0.45) is effective in reducing the burn-in time in the initial stage, minimizing the fluctuation in the deposition speed throughout the target life, and facilitating the processing of the target.

[Examples]

25 Examples and Comparative Examples of the present invention are now explained. Incidentally, these examples are merely illustrative,

and the present invention shall in no way be limited thereby. In other words, the present invention shall include the various modes and modifications covered by the technical spirit of this invention.

(Examples 1 to 16)

5 An EB molten tantalum ingot ($\phi 190 \times 60\text{mmh}$) having a purity level of 4N5 was subject to deformation processing and heat treatment and used as the raw material. Foremost, the ingot was subject to cold tightening forging up to $\phi 100 \times 100\text{mmh}$, and thereafter subject to upset forging up to $\phi 160 \times \text{xt}$.

10 After performing heat treatment for 2 hours at 1000 to 1200°C, cold rolling was performed up to a thickness of 10mm. The degree of rolling processing is as per Table 1.

 Further, the degree of rolling processing was adjusted according to the thickness of the preform during upset forging. After cold rolling,
15 heat treatment was performed at a low temperature of 250°C or less in Example 1 to Example 5. In particular, heat treatment was performed in a temperature range of 100 to 200°C in Example 1 to Example 4, and at a temperature of 250°C in Example 5. Vacuum heat treatment was not performed at a low temperature in Example 6 to Example 16. Table 1
20 shows a list of conditions of the heat treatment temperature. The obtained samples were diffusion-bonded to a copper alloy backing plate, and thereafter machined into a target shape.

 As the surface machining method, lathe was used for crude processing, precision lathe was used for cutting work, and wet polishing
25 was performed thereafter to inhibit the formation of a work-affected layer as much as possible. Specifically, after forming a target surface to a certain degree with crude processing, the target was cut with a precision

lathe to a depth of $100\mu\text{m}$.

Since it has become known that the work-affected layer resulting from this cutting was roughly $1/2$ to $1/3$ of the cut amount, the work-affected layer was cut with a precision lathe or diamond cutter at a
5 depth of $50\mu\text{m}$ in order to eliminate this work-affected layer. The work-affected layer that was newly formed as a result of this cutting process was $25\mu\text{m}$ or less. Cutting was further performed at a depth of $25\mu\text{m}$. The work-affected layer that was formed as a result of this cutting process was $15\mu\text{m}$ or less. Like this, since it is difficult to
10 further eliminate the work-affected layer merely by repeating lathe processing, wet polishing was subsequently performed. It was thereby possible to stably form a preferable work-affected layer of $10\mu\text{m}$ or less.

The target surface roughness was uniformized so that the center-line surface roughness R_a will be roughly 0.2 to $0.3\mu\text{m}$ (in reality,
15 R_a 0.16 to $0.33\mu\text{m}$) for the sake of comparison. Further, vacuum heat treatment was performed at a low temperature to certain samples. Before sputtering, XRD measurement was conducted on a plane that is parallel with the target sputtered face at 9 locations in a cross shape. The burn-in time was calculated as the time (integral power) until the
20 deposition speed stabilized.

The XRD measurement conditions are as follows.

X-ray generator: 3kW

Radiation source: Cu

Wavelength: 1.54056 angstrom

25 Tube voltage: 40.0kV

Tube current: 30.0mA

Scan speed: 15.000deg/min

Sampling interval: 0.020deg

Scan axis: $2\theta/\theta$

The target thus obtained was sputtered at a sputtering power of
5 30kW. The results are shown in Table 1.

[Table 1]

	No.	Degree of Rolling Processing Temperature (%)	Heat Treatment Temperature (°C)	Target Processing	Vacuum Heat Treatment (°C)	FWHM of Surface (200)	FWHM Variation of Surface (200)	FWHM of Surface (110)	FWHM Variation of Surface (110)	Work-Affected Layer (Approx. μm)	Burn-in (kWh)	Particles
Examples	1	80	1000	Lathe, Precision Lathe, Wet Grinding	100	0.41	± 0.03	0.30	± 0.07	8	38	Few
	2	75	1050	Lathe, Precision Lathe, Wet Grinding	125	0.45	± 0.03	0.35	± 0.03	7	28	Few
	3	90	1000	Lathe, Precision Lathe, Wet Grinding	150	0.34	± 0.03	0.30	± 0.03	5	30	Few
	4	65	1050	Lathe, Precision Lathe, Wet Grinding	200	0.31	± 0.03	0.30	± 0.03	5	32	Few
	5	70	1100	Lathe, Precision Lathe, Wet Grinding	250	0.22	± 0.04	0.21	± 0.02	8	55	Normal
	6	70	1050	Lathe, Precision Lathe, Wet Grinding	-	0.12	± 0.03	0.25	± 0.02	5	60	Normal
	7	75	1050	Lathe, Precision Lathe, Wet Grinding	-	0.15	± 0.03	0.25	± 0.02	5	25	Few
	8	75	1200	Lathe, Precision Lathe, Wet Grinding	-	0.35	± 0.03	0.30	± 0.04	10	30	Few
	9	85	1150	Lathe, Precision Lathe, Wet Grinding	-	0.31	± 0.03	0.30	± 0.03	10	33	Few
	10	70	1200	Lathe, Precision Lathe, Wet Grinding	-	0.45	± 0.03	0.42	± 0.06	10	38	Few
	11	85	1150	Lathe, Precision Lathe, Wet Grinding	-	0.30	± 0.08	0.30	± 0.04	12	45	Few
	12	70	1200	Lathe, Precision Lathe, Wet Grinding	-	0.43	± 0.07	0.35	± 1.00	13	45	Normal
	13	75	1050	Lathe, Precision Lathe	-	0.44	± 0.09	0.43	± 1.02	17	59	Few
	14	89	1100	Lathe, Precision Lathe, Wet Grinding	-	0.55	± 0.04	0.45	± 0.06	17	75	Few
	15	92	1150	Lathe, Precision Lathe, Wet Grinding	-	0.46	± 0.03	0.29	± 0.06	18	80	Few
	16	80	1000	Lathe, Precision Lathe	-	0.53	± 0.08	0.42	± 0.04	18	70	Few
Comparative Examples	1	80	850	Lathe, Precision Lathe, Wet Grinding	-	0.91	± 0.05	0.96	± 0.04	25	180	Normal
	2	75	1300	Lathe, Precision Lathe, Wet Grinding	-	0.78	± 1.01	0.81	± 0.95	20	170	Normal
	3	80	1000	Lathe, Precision Lathe, Wet Grinding	800	0.07	± 0.03	0.17	± 0.03	5	55	Many
	4	80	1000	Lathe, Precision Lathe, Etching	-	0.08	± 0.12	0.31	± 0.09	8	60	Many
	5	89	1100	Lathe, Precision Lathe	-	0.75	± 0.08	0.35	± 0.07	18	130	Many
	6	92	1150	Lathe, Precision Lathe	-	0.65	± 0.03	0.40	± 0.05	17	120	Normal

∴ No Vacuum Heat Treatment

(Comparative Examples 1 to 6)

As with the Examples, an EB molten tantalum ingot ($\phi 190 \times 60\text{mmh}$) having a purity level of 4N5 was subject to deformation processing and heat treatment and used as the raw material. Foremost, 5 the ingot was subject to cold tightening forging up to $\phi 100 \times 100\text{mmh}$, and thereafter subject to upset forging up to $\phi 160 \times \text{xt}$.

After performing heat treatment for 2 hours at 850 to 1300°C, cold rolling was performed up to a thickness of 10mm. The degree of rolling processing is as per Table 1.

10 Further, the degree of rolling processing was adjusted according to the thickness of the preform during upset forging. As shown in Table 1, heat treatment was performed at a high temperature of 800°C after cold rolling in Comparative Example 3, and the obtained samples were machined into a target shape. Vacuum heat treatment was not 15 performed in other Comparative Examples.

As the surface machining method, as with the Examples, lathe was used for crude processing, and precision lathe was used for cutting work to obtain the same degree of surface roughness in one or two processes. Certain samples were subject to wet polishing or etching treatment after 20 the cutting work. In other words, wet polishing was performed in Comparative Examples 1 to 3, and etching treatment was performed in Comparative Example 4 to remove the surface. In Comparative Examples 5 and 6, neither wet polishing nor etching treatment was performed.

25 Before sputtering, XRD measurement was conducted on a plane that is parallel with the target sputtered face at 9 locations in a cross shape. The burn-in time was calculated as the time until the deposition

speed stabilized. The results are similarly shown in Table 1.

As shown in Examples 1 to 16 of Table 1, it is evident that the targets having FWHM (full width of half maximum) of the {200} crystal plane of tantalum of 0.1 to 0.6 have low integral power required for the burn-in, and the burn-in time has been significantly shortened.

In particular, as shown in Examples 1 to 5 and 7 to 13, targets having FWHM of the {200} crystal plane of tantalum of 0.15 to 0.45 are superior.

Further, as shown in Examples 1 to 4 and 7 to 10, it is evident that targets in which the variation of FWHM is within ± 0.05 are stable and even more effective.

As shown in Examples 2, 3, 4, 6 and 7, it is evident that targets having FWHM of the {110} crystal plane of 0.25 to 0.4 and in which the variation of FWHM is within ± 0.05 are preferable since they are able to shorten the burn-in time even more.

Further, Examples 1 to 10 show targets in which the depth of the processing strain of the tantalum sputtering target is a depth of $15\mu\text{m}$ from the target surface.

Consequently, even though processing is performed with care, processing strain will exist within a range of $10\mu\text{m}$ or less from the surface. Nevertheless, by performing vacuum heat treatment at a low temperature of around 100 to 250°C , an effect is yielded in that FWHM of {200} can be controlled stably and the burn-in time can be shortened.

Further, even in cases where a processing strain layer of roughly $20\mu\text{m}$ exists, it has been discovered that the burn-in time can be effectively shortened.

Contrarily, with Comparative Example 1, Comparative Example 2, Comparative Example 5, and Comparative Example 6, since FWHM of the {200} crystal plane of tantalum exceeded 0.6, it is evident that the integral power required for the burn-in increased, and the burn-in time increased proportionately.

Meanwhile, with Comparative Example 3 and Comparative Example 4, since FWHM of the {200} crystal plane of tantalum is less than 0.1, the generation of particles increased. Incidentally, in Comparative Example 3 and Comparative Example 4, vacuum heat treatment was performed at a high temperature of 800°C in order to reduce the processing strain. Nevertheless, although improvement in the burn-in time was acknowledged, the level of particles contrarily became inferior.

Therefore, this shows that the reduction of FWHM of the {200} crystal plane of tantalum does not necessarily yield a favorable effect.

With Comparative Example 4, Comparative Example 5, and Comparative Example 6, FWHM of the {110} crystal plane of tantalum was adjusted to be within the range of the present invention. Nevertheless, since preference is given to FWHM of the {200} crystal plane, it is evident that controlling FWHM of the {200} crystal plane of tantalum is more important than adjusting FWHM of the {110} crystal plane.

INDUSTRIAL APPLICABILITY

The present invention yields superior effects of significantly

reducing the initial burn-in time of a tantalum or a tantalum-based alloy sputtering target and minimizing the fluctuation in the deposition speed throughout the target life, whereby the production efficiency of semiconductors in the sputtering process can be improved and stabilized,
5 and the production cost can be significantly reduced. Thus, the present invention is extremely useful as a tantalum or a tantalum-based alloy target.

It is obvious that the same effect is yielded in a tantalum-based alloy target having the same BCC structure. As the additive elements
10 of this tantalum-based alloy target, high-melting metals such as Mo and 3- or 4-group elements can be considered, and platinum group metals such as platinum can be considered as other alloy elements. The additive amount should be an amount that does not significantly change the crystal structure.

CLAIMS

1. A tantalum or a tantalum-based alloy sputtering target, wherein FWHM of a {200} crystal plane measured by X-ray diffraction of the sputtered outermost surface is 0.1 to 0.6.
2. The sputtering target according to claim 1, wherein FWHM is 0.15 to 0.45.
3. The sputtering target according to claim 1 or claim 2, wherein the variation of FWHM is within ± 0.05 .
4. The sputtering target according to any one of claims 1 to 3, wherein FWHM of a {110} crystal plane is 0.25 to 0.4, and the variation of FWHM is within ± 0.05 .
5. The sputtering target according to any one of claims 1 to 4, wherein the depth of the processing strain of the tantalum or tantalum-based alloy sputtering target is a depth of $15\mu\text{m}$ from the target surface.
6. The sputtering target according to claim 5, wherein the depth of the processing strain of the tantalum or tantalum-based alloy sputtering target is a depth of $10\mu\text{m}$ from the target surface.