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**Method of making a coated cutting tool**
- Verfahren zur Herstellung eines beschichteten Schneidwerkzeugs
- Procédé de fabrication d’un outil de découpe revêtu

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**References cited:**
- WO-A1-99/29921

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The present invention relates to a metal cutting tool comprising a substrate and a wear resistant coating including a multilayered structure, and a method comprising a PVD Bipolar Pulsed Dual Magnetron Sputtering technique for producing such a tool.

Modern high productivity chip forming machining of metals requires reliable tools with high wear resistance, good toughness properties and excellent resistance to plastic deformation.

This has so far been achieved by applying a suitable coating to the surface of a tool substrate. As a result, the tool may be used at considerably higher cutting speed and feed. The coating is preferably hard, wear resistant and stable at high temperatures. The tool substrate is generally in the shape of an insert clamped in a tool holder, but can also be in the form of a solid drill or a milling cutter.

Since the mid 1980:s Physical Vapor Deposition (PVD) technology has been developed to the point where not only stable coating compounds like TiN and Ti(C,N) may be grown, but also e.g. a meta-stable TiN/AlN/TiN/AlN structure, which in itself offers unique properties like high hardness, high oxidation resistance and high hot hardness.

PVD methods for depositing multilayered MX/LX/MX/LX structures, where the alternating layers MX and LX are carbides or nitrides with the elements M and L selected from the group consisting of Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si and W, comprise vacuum technologies like arc evaporation or magnetron sputtering. Evaporation of metal from targets is accomplished by electrical arc or ion bombardment in a reactive gas containing nitrogen or carbon. Very often the targets have the same metal composition as the final layer. For a multilayer arc coating, having a periodic and constant sublayer thickness throughout the coating, the average composition will depend on the compositions of the two targets and weighted with the individual sublayer thickness as measured in the direction of the layer thickness/growth direction.

Magnetron sputtering yields layers with significantly lower number of surface defects than arc evaporation, but there arise problems when a material like AlN is deposited, inasmuch the coating is an insulator. These problems are often less pronounced when using arc evaporation technology. Magnetron sputtered multilayers are usually comprising individual sublayer coatings that are conducting. There is a problem of using elemental Al targets since insulating AlN will form at the surface. The average coating composition is defined by the relative evaporation rates from the respective target and the composition of these, which compositions set the limits for the obtained coating composition.

It is known from JP-A-08-209333 that the hardness and oxidation temperature of the TiAlN layer continue to improve when the Al content increases beyond 56 at-%. It is also disclosed that as the Al composition is increased above a critical number, approximately above 75 at-%, the resulting layer again becomes soft due to formation of hexagonal AlN.

US 6,103,357 discloses a coating comprising a multilayered structure of refractory compounds in polycrystalline, non-repetitive form, MX/LX/MX/LX where the alternating layers MX and LX are metal nitrides or carbides with the metal elements M and L selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al and W.

It is an object of the present invention to provide a method for producing a cutting tool comprising depositing a refractory multilayer consisting of nitride or carbide layers or mixtures thereof, of which at least one is electrically isolating.

Fig. 1 shows a Transmission Electron Microscope micrograph of an exemplary refractory coating in which

A) = TiN and
B) = AlN

Fig. 2 shows a SEM-image of an exemplary TiN/AlN/TiN/AlN-multilayer in which

S) = substrate and
L) = multilayer.

Fig. 3 shows schematically an exemplary target configuration of the BPDMS equipment according to the present invention in which

a) = Titanium target
b) = Aluminum target
c) = Etching/bias supply
d) = Sputtering supply
e) = Heater supply

A) = TiN and
B) = AlN
5 [0014] By way of example, the cutting tool for metal machining such as turning, milling and drilling comprises a substrate of e.g. cermet, ceramic, cemented carbide or cubic boron nitride onto which a hard and wear resistant refractory coating is deposited by PVD technique. The wear resistant coating is composed of one or more layers of refractory compounds of which at least one layer consists of a multilayered MX/LX/MX/LX laminar structure where the alternating layers MX and LX are carbides or nitrides with the elements M and L selected from the group consisting of Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si or W and mixtures thereof deposited by PVD-technique, of which at least one of MX or LX are electrically isolating, and that the other layer(s), if any at all, comprise(s) wear resistant nitrides, carbides, oxides and/or carbonitrides as known in the art. The sequence of individual MX and LX layers have thicknesses that are aperiodic throughout the entire multilayer structure. Furthermore, the minimum individual layer thickness is larger than 0.1 nm but the sum of any 10 consecutive layers in the structure is smaller than 300 nm. The thickness of one individual layer does not depend on the thickness of an individual layer immediately beneath nor does it bear any relation to a individual layer above the said one individual layer. Hence, the aperiodic multilayers do not have any repeat period. The total thickness of the multilayer is 0.5 to 20 μm, preferably 1 to 10 μm, most preferably 2 to 6 μm.

[0015] In another example the multilayered structure has a content gradient in the elements M and L, due to a shift in the relation of the average MX and LX layer thicknesses throughout the multilayer. The average M-content may e.g. be close to zero percent in the innermost part of the multilayer, i.e., the average MX layer thickness is close to zero, hence the average L-content being close to 100 percent. The average M-content may increase to a content near 100 percent towards the outermost part of the multilayer due to a gradually increased average MX layer thickness towards the outermost part of the multilayer.

[0016] In yet another example the multilayer has the structure TiN/AlN/TiN/AlN/... which, when written in the form Ti1-xAlxN, has an average Al-content 0.10 < x < 0.85 and a nitrogen content of about 50 at-%. The exemplary multilayer has further an intensity ratio for diffraction peak [200] of I200/(I111+I200+I220) > 0.33, or intensity ratio for diffraction peak [111] of I111/(I111+I200+I220) > 0.33, or a mixture thereof, with both I200/(I111+I200+I220) > 0.33 and I111/(I111+I200+I220) > 0.33, as determined by using XRD with Cu Kα radiation and the intensity ratios calculated as 1/\(k(I_{111}+2I_{200}+I_{220})\). The hardness of said multilayer is in the range 23 to 32 GPa.

[0017] In another example the above described TiN/AlN/TiN/AlN/... structured multilayer, the multilayer is XRD amorphous.

[0018] In a further example of the above described TiN/AlN/TiN/AlN/... structured multilayer, particularly suitable for depositing an aluminumoxide layer onto, the multilayer has an Al content gradient such that the average Al content increases from the innermost part of the multilayer towards the outermost part of the multilayer.

[0019] In a further example of the above described TiN/AlN/TiN/AlN/... structured multilayer, the Al-content is 0.10 < x < 0.30, resulting in a cutting tool particularly useful in applications having high demands on abrasive wear resistance due to very high hardness of the multilayer.

[0020] In a further example of the above described TiN/AlN/TiN/AlN/... structured multilayer, the Al-content is 0.40 < x < 0.55, resulting in a versatile cutting tool combining high hardness and high temperature properties.

[0021] In a further example of the above described TiN/AlN/TiN/AlN/... structured multilayer, the Al-content is 0.60 < x < 0.80, resulting in cutting tool with particularly high resistance against crater wear.

[0022] According to the present invention there is provided a method for manufacturing a metal cutting tool comprising depositing a hard and wear resistant refractory coating comprising compounds of which at least one layer consists of a multilayered MX/LX/MX/LX laminar structure where the alternating layers MX and LX are carbides or nitrides with the elements M and L selected from the group consisting of Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si or W and mixtures thereof, of which at least one of MX or LX are electrically isolating, and that the other layer(s), if any at all, comprise(s) wear resistant nitrides, carbides, oxides and/or carbonitrides as known in the art, onto a substrate of e.g. cermet, ceramic, cemented carbide or cubic boron nitride by PVD Bipolar Pulsed Dual Magnetron Sputtering (BPDMs) technique. In the BPDMs process magnetron pairs are used comprising either one M element target and one L element target using preferably two magnetron pairs, or using magnetron pairs with two targets of the same element, arranged so that one target in the magnetron pair acts as anode and the other target acts as cathode and vice versa. A voltage of 400 to 1000 V is applied to each bipolar switch and the supply power is limited to 2x10 up to 2x70 kW. The base pressure prior to deposition should be <2mPa and the Ar sputtering gas pressure is in the range 0.1 to 1 Pa. The deposition process is performed at substrate temperatures between 300 to 700 °C. Preferably a 20 to 100 V bipolar pulsed bias; bias is known to have effect on the intensity ratio of the coating and it is hence possible to achieve different intensity ratios by choosing different bias values; and a current of 4 to 30 A is applied to the substrates using the wall as counter electrode. The substrate rotation is employed in order to ensure homogeneous coating deposition. The sputtering pulse times on the M (tM) and L (tL) targets are set in the range from 2 to 100 μs and the pulse off times, (tOFF), between the M and L sputtering pulses in the range 2 to 10 μs. The reactive gas such as N2 or methane or other nitrogen or carbon containing gases, is let in through a common inlet and the reactive gas pressure is preferably in the range 0.01 to 0.25 Pa.

[0023] By varying the reactive gas pressure it is possible to shift the sputtering power between the M an L targets hence changing the current relation between the targets and thereby varying the multilayer composition, i.e., varying...
the average MX and LX layer thicknesses, respectively.

In the deposition process it is also possible to vary the multilayer composition, i.e., vary the average MX and LX layer thicknesses, respectively, by varying the sputtering pulse times in the applied pulse train, defined as \[ t_M + t_L + 2\times t_{OFF}. \]

**Example 1**

A TiN/AlN/TiN/AlN/… multilayer, denoted Ti\(_{1-x}Al_xN\), was deposited on a cemented carbide substrate using BPDMs using two magnetron pairs, each comprising of one Al and one Ti target, arranged so that Al acted as anode when Ti was cathode and vice versa. A voltage of 900 V was applied to each bipolar switch and the supply power was limited to 2x35 kW. The base pressure prior to deposition was <2mPa and the Ar sputtering gas pressure was 0.4 Pa. The coatings that were deposited are presented together with characteristic process parameters in Table I below. All coatings were deposited at a substrate temperature of 500°C. A constant reactive gas pressure was maintained using pressure feed-back control. A 40 V bipolar pulsed bias and a current of about 15 A was applied to the substrates using the wall as counter electrode. Parameters that were varied were sputtering pulse times on the Ti (\( t_{Ti} \)) and the Al (\( t_{Al} \)) targets, respectively, as well as \( N_2 \) pressure. The \( N_2 \) was let in through a common diffuse inlet. The pulse-off times between the Ti and Al sputtering pulses were kept at 5 \( \mu \)s during all experiments and the pulse train is written as \( t_{Al}/t_{Ti} \), e.g. 7/30 \( \mu \)s. The three different pulse conditions studied are 7/30 \( \mu \)s (total pulse train length 47 \( \mu \)s), 12/25 \( \mu \)s (total pulse train length 47 \( \mu \)s) and 10/10 \( \mu \)s (total pulse train length 30 \( \mu \)s). The sputtering currents for the Ti and Al targets were resolved by measuring the branch currents during the respective sputtering pulses. Not only the nitrogen flow but also the individual Ti and Al sputtering currents varied with the set \( N_2 \) pressure. For the sputtering pulse 7/30 \( \mu \)s it was found that the Al sputtering currents increased from values close to zero at a low pressure, to a magnitude close to that of the Ti sputtering current at high pressure. Threefold substrate rotation was employed in order to ensure homogeneous coating deposition of the cemented carbide cutting tools and the deposition time was set to 120 min.

### Table I

<table>
<thead>
<tr>
<th>Sample</th>
<th>( t_{Al}/t_{Ti} ) [( \mu )s]</th>
<th>( P_{IM} ) [Pa]</th>
<th>( P_{N2} ) [Pa]</th>
<th>( I_{N2} ) [sccm]</th>
<th>( I_{Ti} ) [A]</th>
<th>( I_{Al} ) [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>7/30_1.2</td>
<td>7/30</td>
<td>0.52</td>
<td>0.12</td>
<td>184</td>
<td>31</td>
<td>26</td>
</tr>
<tr>
<td>12/25_0.5</td>
<td>12/25</td>
<td>0.45</td>
<td>0.05</td>
<td>153</td>
<td>22</td>
<td>10</td>
</tr>
<tr>
<td>12/25_0.7</td>
<td>12/25</td>
<td>0.47</td>
<td>0.07</td>
<td>154</td>
<td>19</td>
<td>18</td>
</tr>
<tr>
<td>12/25_0.8</td>
<td>12/25</td>
<td>0.48</td>
<td>0.08</td>
<td>166</td>
<td>21</td>
<td>28</td>
</tr>
<tr>
<td>12/25_0.9</td>
<td>12/25</td>
<td>0.49</td>
<td>0.09</td>
<td>158</td>
<td>17</td>
<td>35</td>
</tr>
<tr>
<td>10/10_0.3</td>
<td>10/10</td>
<td>0.43</td>
<td>0.03</td>
<td>71</td>
<td>6</td>
<td>5</td>
</tr>
<tr>
<td>10/10_0.6</td>
<td>10/10</td>
<td>0.46</td>
<td>0.06</td>
<td>108</td>
<td>5</td>
<td>20</td>
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</table>

All coatings were studied using a Hitachi S-4300 field emission gun scanning electron microscope (FEG-SEM) at 10 kV and a JEOL 2010 transmission electron microscope (TEM) with selected area electron diffraction (SAED) to enable phase analysis. The chemical composition was determined by Electron Probe Micro Analysis (EPMA) using a JEOL JXA-8900R at an acceleration voltage of 10 kV and a 10 nA probe current. Ti and Al were analyzed by their x-ray intensities while the N stoichiometry was estimated by the difference between the coating x-ray intensity and the intensity of a metallic sample. The phase composition was determined using x-ray diffraction with Cu K\(_a\) radiation. The intensity ratios were calculated as \( I_{hk}/(I_{111}+I_{200}+I_{220}) \). A CSEM nano hardness tester was used to determine the hardness of the coatings. By using a load of 25 mN the contribution from the substrate was regarded as very small or none for all coatings.

When comparing coatings deposited using a pulse train of 12/25 \( \mu \)s, it was found that the growth rate was highest for the lowest \( N_2 \) pressure, 0.05 Pa. This is most likely due to the fact that sputtering at a lower \( N_2 \) pressure equals sputtering at a more metallic mode. The depositions at 0.07-0.09 Pa were deposited in the reactive regime. Despite the increasing \( N_2 \) pressure the deposition rates were equal at around 18 nm/min.

For a pulse of 7/30 \( \mu \)s the coating metal composition corresponded to Ti\(_{0.64}Al_{0.16}\) and for a pulse train of 12/25 \( \mu \)s \( x \) ranged from 0.36 - 0.58 and for a pulse train of 10/10 \( \mu \)s \( x \) varied between 0.72 - 0.84. This means that by varying the pulse times in the present range in combination with a variation of the \( N_2 \) pressure as 0.08p\(_{Ar}\) \( \leq \) p\(_{nitrogen}\) \( \leq \) 0.3p\(_{Ar}\), \( x \) in Ti\(_{1-x}Al_xN\) can be controlled in the range 0.16 to 0.84. The nitrogen composition was calculated to around 50 at-%. Coatings with a low Al content, \( x \leq 0.49 \), were columnar and the surface were cauliflower-like. A higher Al content, \( x \geq 0.58 \), resulted in a more glassy structure. The columnar coatings exhibit a bronze-like colour whereas the glassy coatings exhibit a blue or even transparent colour.
[111] type for the coatings with higher Al content.

Table II

<table>
<thead>
<tr>
<th>Sample</th>
<th>r [nm/min]</th>
<th>t_c [μm]</th>
<th>Colour</th>
<th>x in Th_{1-x}Al_xN</th>
<th>XRD I_{111}/I_{tot} *</th>
<th>XRD I_{200}/I_{tot}</th>
<th>XRD I_{220}/I_{tot}</th>
<th>NH_{25mN} [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>7/30_1.2</td>
<td>12</td>
<td>1.4</td>
<td>Brown-bronze</td>
<td>0.16</td>
<td>0.17</td>
<td>0.73</td>
<td>0.10</td>
<td>31.0</td>
</tr>
<tr>
<td>12/25_0.5</td>
<td>23</td>
<td>2.8</td>
<td>Purple-bronze</td>
<td>0.36</td>
<td>0.58</td>
<td>0.30</td>
<td>0.12</td>
<td>23.0</td>
</tr>
<tr>
<td>12/25_0.7</td>
<td>18</td>
<td>2.1</td>
<td>Purple-bronze</td>
<td>0.41</td>
<td>0.68</td>
<td>0.29</td>
<td>0.03</td>
<td>30.9</td>
</tr>
<tr>
<td>12/25_0.8</td>
<td>18</td>
<td>2.1</td>
<td>Purple-blue</td>
<td>0.49</td>
<td>0.85</td>
<td>0.13</td>
<td>0.03</td>
<td>31.6</td>
</tr>
<tr>
<td>12/25_0.9</td>
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<td>2.0</td>
<td>Blue</td>
<td>0.58</td>
<td>-**</td>
<td>-**</td>
<td>-**</td>
<td>28.7</td>
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<td>9</td>
<td>1.1</td>
<td>Transparent</td>
<td>0.72</td>
<td>-**</td>
<td>-**</td>
<td>-**</td>
<td>25.6</td>
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<tr>
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<td>13</td>
<td>1.5</td>
<td>Transparent</td>
<td>0.84</td>
<td>-**</td>
<td>-**</td>
<td>-**</td>
<td>25.2</td>
</tr>
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</table>

* I_{tot} = (I_{111} + I_{200} + I_{220})

** XRD amorphous

Claims

1. Method for manufacturing a metal cutting tool comprising depositing a hard and wear resistant refractory coating comprising compounds of which at least one layer consists of a multilayered MX/LX/MX/LX laminar structure where the alternating layers MX and LX are carbides or nitrides with the elements M and L selected from the group consisting of Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si or W and mixtures thereof, of which at least one of MX or LX are electrically isolating, and that the other layer(s), if any at all, comprise(s) wear resistant nitrides, carbides, oxides and/or carbonitrides as known in the art, onto a substrate characterized in using PVD Bipolar Pulsed Dual Magnetron Sputtering (BPDMS) technique, using magnetron pairs comprising either one M element target and one L element target or using magnetron pairs with two targets of the same element, arranged so that one target in the magnetron pair acts as anode and the other target acts as cathode and vice versa, applying a voltage of 400 to 1000 V to each bipolar switch and limiting the supply power to 2x10 up to 2x70 kW, using a base pressure prior to deposition <2mPa and a reactive gas pressure between 0.01 to 0.25 Pa.

2. The method according to claim 1 characterized in that the composition of multilayer is varied by altering the relation between the sputtering pulse times on the M (t_M) and L (t_L) targets.

3. The method according to claims 1-2 characterized in that the composition of multilayer is varied by altering the reactive gas pressure.

4. The method according to claim 3 characterized in that the pressure of reactive gas is varied during the deposition process.

5. The method according to claims 1-4 characterized in the relation between the sputtering pulse times on the M (t_M) and L (t_L) targets is varied during the deposition process.

6. The method according to any of claims 1-5 characterized by using magnetron pairs comprising one M element target and one L element target using two magnetron pairs.

7. The method according to any of claims 1-6 characterized by applying a 20 to 100 V bipolar pulsed bias and a current of 4 to 30 A to the substrates using the...
1. Verfahren zum Herstellen eines Metallschneidwerkzeuges, wobei das Verfahren das auf einem Substrat Abscheiden einer harten und verschleißfesten, hitzebeständigen Beschichtung aufweist, welche Verbindungen enthält, von denen zumindest eine Schicht aus einer laminaren, mehrlagigen MX/LX/MX/LX-Struktur besteht, wobei die abwechselnden Schichten MX und LX Karbide oder Nitride mit den Elementen M und L sind, welche ausgewählt werden aus der Gruppe, die besteht aus: Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si oder W oder Mischungen hieraus, wobei mindestens MX oder LX elektrisch isolierend sind und die andere Schicht (bzw. Schichten), soweit vorhanden, verschleißbeständige Nitride, Karbide, Oxide und/oder Carbonitride enthalten, wie es im Stand der Technik bekannt ist, dadurch gekennzeichnet, dass eine PVD-Technik mit bipolaren, gepulsten Doppelmagnetronsputtern verwendet wird, wobei Magnetronpaare verwendet werden, die entweder ein M-Element-Target und ein L-Element-Target aufweisen oder durch Verbinden von Magnetronpaaren, zwei Targets des selben Elemente aufweisen und welche so angeordnet sind, dass das eine Target in dem Magnetronpaar als Anode und das andere Target als Kathode wirkt oder umgekehrt, Anlegen einer Spannung von 400 bis 1000 Volt an jedem bipolaren Schalter und Begrenzen der Zufuhrleitung auf 2x10 bis zu 2x70 KW unter Verwendung eines Basisdruckes vor der Abscheidung von weniger als 2mPa und einem Ar-Sputtergasdruck im Bereich von 0,1 bis 1 Pa, mit Substrattemperaturen zwischen 300 und 700°C und unter Verwendung einer dreifachen Substratrotation, Einstellen der Sputterpulszeiten auf den M (tM)- und L (tL)-Targets im Bereich von 2 bis 100 μs und Auszeiten der Pulse (tOFF) zwischen den M- und L-Sputterimpulsen im Bereich von 2 bis 10 μs und Verwenden eines Reaktionsgasdruckes zwischen 0,01 bis 0,25 Pa.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die Zusammensetzung der Mehrfachschicht variiert wird durch Ändern des Verhältnisses zwischen den Sputterimpulszeiten auf den M (tM)- und L (tL)-Targets.

3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, dass die Zusammensetzung der mehrschichtigen Schicht durch Verändern des Reaktionsgasdruckes variiert wird.


5. Verfahren nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, dass das Verhältnis zwischen den Sputterimpulszeiten auf den M (tM)- und L (tL)-Targets während des Abscheidevorganges variiert wird.

6. Verfahren nach einem der Ansprüche 1 bis 5, gekennzeichnet durch die Verwendung von Magnetronpaaren, welche ein M-Element-Target und ein L-Element-Target aufweisen, unter Verwendung zweier Magnetronpaaren.

7. Verfahren nach einem der Ansprüche 1 bis 6, gekennzeichnet durch Anlegen einer bipolaren Pulsivorspannung von 20 bis 100 V und eines Stromes von 4 bis 30 A an den Substraten, wobei die Wand als Gegenelektrode verwendet wird.

Revidications

1. Procédé de fabrication d’un outil de coupe de métaux comprenant le dépôt d’un revêtement réfractaire dur et résistant à l’usure comprenant des composés dont au moins une couche est constituée par une structure laminaire multicouche MX/LX/MX/LX où les couches alternées MX et LX sont des carbures ou des nitrures avec les éléments M et L choisis dans le groupe constitué par Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, Al, Si ou W et leurs mélanges, dont au moins l’un de MX ou LX est isolant électriquement, et la ou les autres couche(s), le cas échéant, comprend (comprendent) des nitrures, carbures, oxides et/ou carbonitrures résistants à l’usure, tels que connus dans l’art, sur un substrat caractérisé par l’utilisation de la technique de pulvérisation à magnétron double pulsé bipolaire PVD (BPDMS), l’utilisation de paires de magnétrons comprenant un élément cible M et un élément cible L ou l’utilisation de paires de magnétrons avec deux cibles du même élément, agencées de telle sorte qu’une cible dans la paire de magnétrons agit en tant qu’anode et l’autre cible agit en tant que cathode et vice versa, l’application d’une tension de 400 à 1 000 V à chaque commutateur bipolaire et la limitation de l’alimentation en puissance à 2 x 10 jusqu’à 2 x 70 KW, l’utilisation d’une pression de base avant le dépôt < 2 mPa et d’une pression de gaz de pulvérisation Ar dans la plage de 0,1 à 1 Pa, de températures de substrat entre 300 et 700 °C, et l’utilisation d’une triple rotation des substrats, l’établissement
des durées d’impulsion de pulvérisation sur les cibles M \((t_M)\) et L \((t_L)\) dans la plage de 2 à 100 \(\mu\)s et des durées sans pulsation \((t_{OFF})\) entre les pulsations de pulvérisation M et L dans la plage de 2 à 10 \(\mu\)s, et l’utilisation d’une pression de gaz réactif entre 0,01 et 0,25 Pa.

2. Procédé selon la revendication 1, caractérisé en ce que la composition de la multicouche est modifiée en changeant la relation entre les durées de pulsation de pulvérisation sur les cibles M \((t_M)\) et L \((t_L)\).

3. Procédé selon les revendications 1 et 2, caractérisé en ce que la composition de la multicouche est modifiée en changeant la pression de gaz réactif.

4. Procédé selon la revendication 3, caractérisé en ce que la pression de gaz réactif est modifiée pendant le procédé de dépôt.

5. Procédé selon les revendications 1 à 4, caractérisé en ce que la relation entre les durées de pulsation de pulvérisation sur les cibles M \((t_M)\) et L \((t_L)\) est modifiée pendant le procédé de dépôt.

6. Procédé selon l’une quelconque des revendications 1 à 5, caractérisé par l’utilisation de paires de magnétrons comprenant un élément cible M et un élément cible L en utilisant deux paires de magnétrons.

7. Procédé selon l’une quelconque des revendications 1 à 6, caractérisé par l’application d’une polarisation pulsée bipolaire de 20 à 100 V et d’un courant de 4 à 30 A sur les substrats en utilisant la paroi comme contre-électrode.
Fig. 2
REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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