



(22) Date de dépôt/Filing Date: 1990/08/09

(41) Mise à la disp. pub./Open to Public Insp.: 1991/02/22

(45) Date de délivrance/Issue Date: 2002/01/15

(30) Priorité/Priority: 1989/08/21 (3 037/89-7) CH

(51) Cl.Int.⁵/Int.Cl.⁵ C23C 14/06, C23C 14/34, C23C 14/24

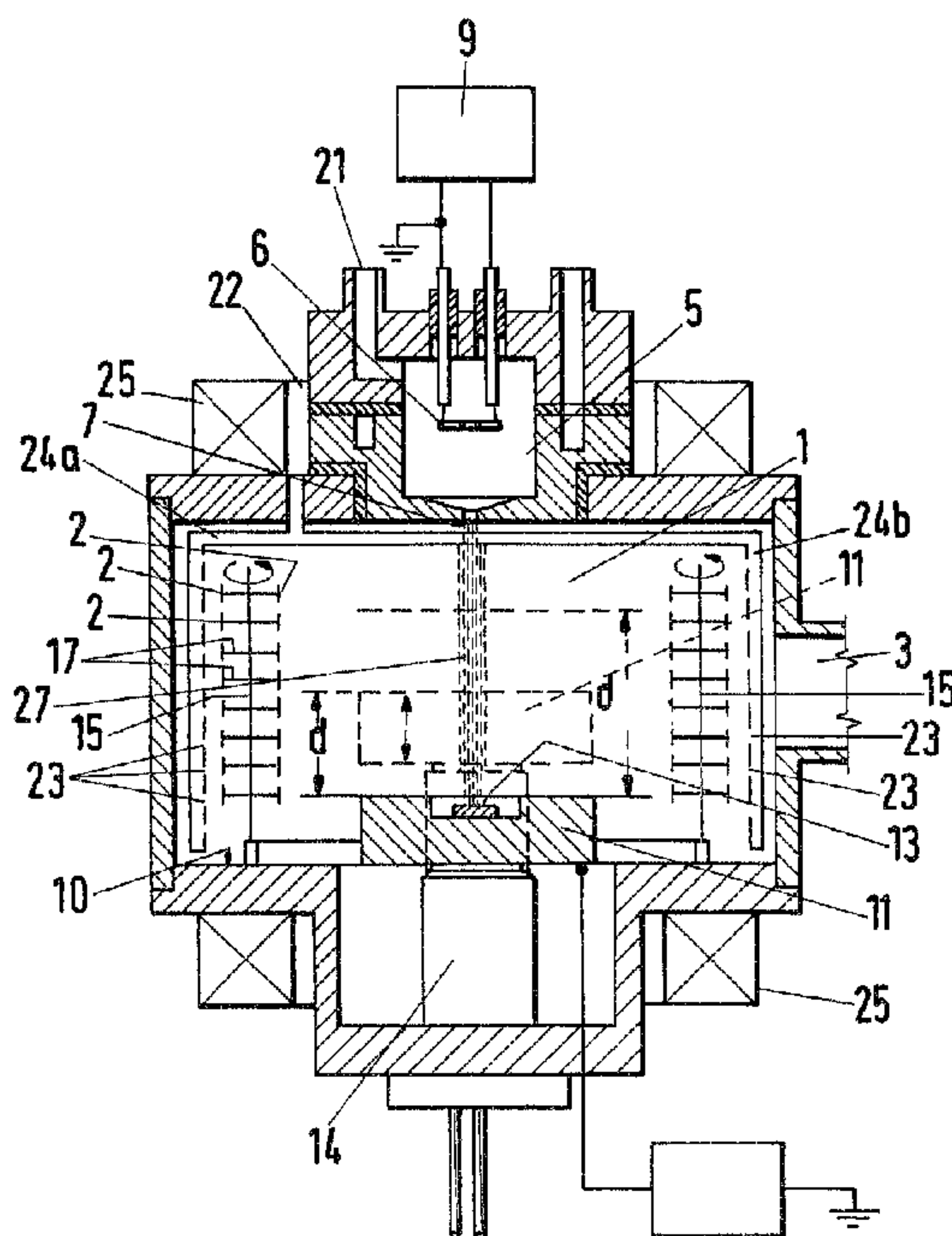
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(54) Titre : PIECE A USINER ENDUITE D'UNE COUCHE DE SOLUTION SOLIDE, METHODE ET APPAREIL DE PRODUCTION CONNEXES, ET UTILISATION DE LADITE PIECE

(54) Title: WORKPIECE COATED WITH A SOLID SOLUTION LAYER, METHOD FOR ITS PRODUCTION, USE OF THE WORKPIECE, AND APPARATUS FOR CARRYING OUT THE METHOD



(57) Abrégé/Abstract:

A solid solution layer (C, D) of metalloids is applied on a workpiece by a reactive physical coating technique and has an inclusion ratio of the metalloids which varies continuously over the thickness of the layer. The solid solution layer (C, D) lies on a separating layer (B) on the workpiece. To produce the mixed layer (C, D) titanium is vaporized in a vacuum chamber in a crucible which is moved back and forth in front of the workpiece surfaces to be coated, and two gases are introduced which have different affinity to the vaporized titanium. For the creation of a first partial layer (C) of the solid solution layer (C, D) the inclusion ratio of the gases is varied steadily. During the coating operation the workpieces rotate, so that sometimes their surface is turned toward the crucible and sometimes away from it. The coated workpieces are distinguished by a high resistance to flank wear and cratering (Fig. 3).

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ABSTRACT OF THE DISCLOSURE

A solid solution layer (C, D) of metalloids is applied on a workpiece by a reactive physical coating technique and has an inclusion ratio of the metalloids which varies continuously over the thickness of the layer. The solid solution layer (C, D) lies on a separating layer (B) on the workpiece. To produce the mixed layer (C, D) titanium is vaporized in a vacuum chamber in a crucible which is moved back and forth in front of the workpiece surfaces to be coated, and two gases are introduced which have different affinity to the vaporized titanium. For the creation of a first partial layer (C) of the solid solution layer (C, D) the inclusion ratio of the gases is varied steadily. During the coating operation the workpieces rotate, so that sometimes their surface is turned toward the crucible and sometimes away from it. The coated workpieces are distinguished by a high resistance to flank wear and cratering (Fig. 3).

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WORKPIECE COATED WITH A SOLID SOLUTION LAYER,
METHOD FOR ITS PRODUCTION, USE OF THE WORKPIECE
AND APPARATUS FOR CARRYING OUT THE METHOD

FIELD AND BACKGROUND OF THE INVENTION

The present invention relates in general to the field of vapor deposition and in particular to a new and useful coated workpiece, a method for its production, uses for the coated workpiece and an apparatus for carrying out the method.

A method of this type and a workpiece coated by the method in order to increase its hardness and toughness are known from European Patent document A 0191554, issued August 20, 1986 to Tsukada et al. Such workpieces are used e.g. as cutting tools. The known coating is carried out by means of a PVD process at temperatures between 200 and 700°C. Up to four discrete layers of titanium carbide, titanium nitride and titanium carbonitride are applied, a titanium nitride layer being always applied directly over the surface to be coated.

SUMMARY OF THE INVENTION

It has now been found that during application of the carbon-containing titanium compounds as coating layers, pure carbon is incorporated into the respective layer as well. These carbon inclusions significantly reduce the adhesivity of the layer on its substrate, as well as the toughness of the coating.

An object of the present invention is to remedy this situation. By the invention, the problem is solved by providing a coated workpiece, as well as a method for the production of the coated workpiece, in which the applied layers adhere well to the workpiece surface and have a high toughness.

The invention includes various preferred forms of the coated workpiece and preferred embodiments of the method for

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toughness.

The invention includes various preferred forms of the coated workpiece and preferred embodiments of the method for producing the coated workpiece.

Accordingly, a further object of the present invention is to provide a coated workpiece having a solid solution coating of metalloids which are applied by means of a reactive physical coating process, wherein the inclusion ratio of the metalloids in the solid solution coating vary continuously over a majority of a thickness of the layer.

According to an aspect of the invention, there is provided a coated workpiece with a firmly adhering, tough solid solution coating which is applied by a reactive physical coating process and which comprises at least two metalloids whose concentration ratio relative to each other, at least in a predominant solid solution coating sub-region, continuously increases and decreases with a periodic component of change in the direction of the surface normal.

Another object of the present invention is to provide a method of producing a coated workpiece wherein a material to form a component of the coating is vaporized in a vacuum chamber and condensed in the form of a solid solution coating on the workpiece, a first gas being supplied to the vacuum chamber during the condensation of the vapor, with the flow of the first gas steadily decreasing.

According to a further aspect of the invention there is provided a reactive physical coating process for the production of the aforesaid coated workpiece wherein a metal which is converted into the gaseous state by means of vapor source in a vacuum chamber is deposited on a surface of a moved workpiece, with chemical reaction with a gaseous reactant introduced into the vacuum chamber characterized in

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that as the reactants in a first gas is introduced at a continuously reduced feed flow, for the formation of a first and a second metalloid, and the workpiece is relatively so moved in relation to the vapor source that the spacing thereof and/or the orientation thereof with respect to the vapor source periodically alters at such a speed that deposited on the workpiece is a solid solution coating with said metalloids whose concentration ratio relative to each other is made up only in the direction of the surface normal from superimposition of the periodic changes in concentration corresponding to the changes in spacing and/or orientation and the change in concentration corresponding to the change in feed flow.

The coated workpiece may be used as a cutting or forming tool.

Another object of the present invention is to provide an apparatus for carrying out the method which includes a vapor source for depositing one component of the coating on the workpiece, and means for moving the vapor source past the workpiece.

According to another aspect of the invention, there is provided an apparatus for producing a coated workpiece made of a workpiece portion having a surface and a solid solution coating of at least two metalloids on the surface, the layer being deposited by a reactive physical coating process and having a concentration ratio of the at least two metalloids in a predominant portion of the layer thickness which varies continuously over the predominant portion of the layer thickness, the apparatus comprising means for forming a vapor source for vaporizing a metal for use in reactions with gases to form the at least two metalloids, and means for moving the vapor source with respect to a workpiece portion for producing the coated workpiece.

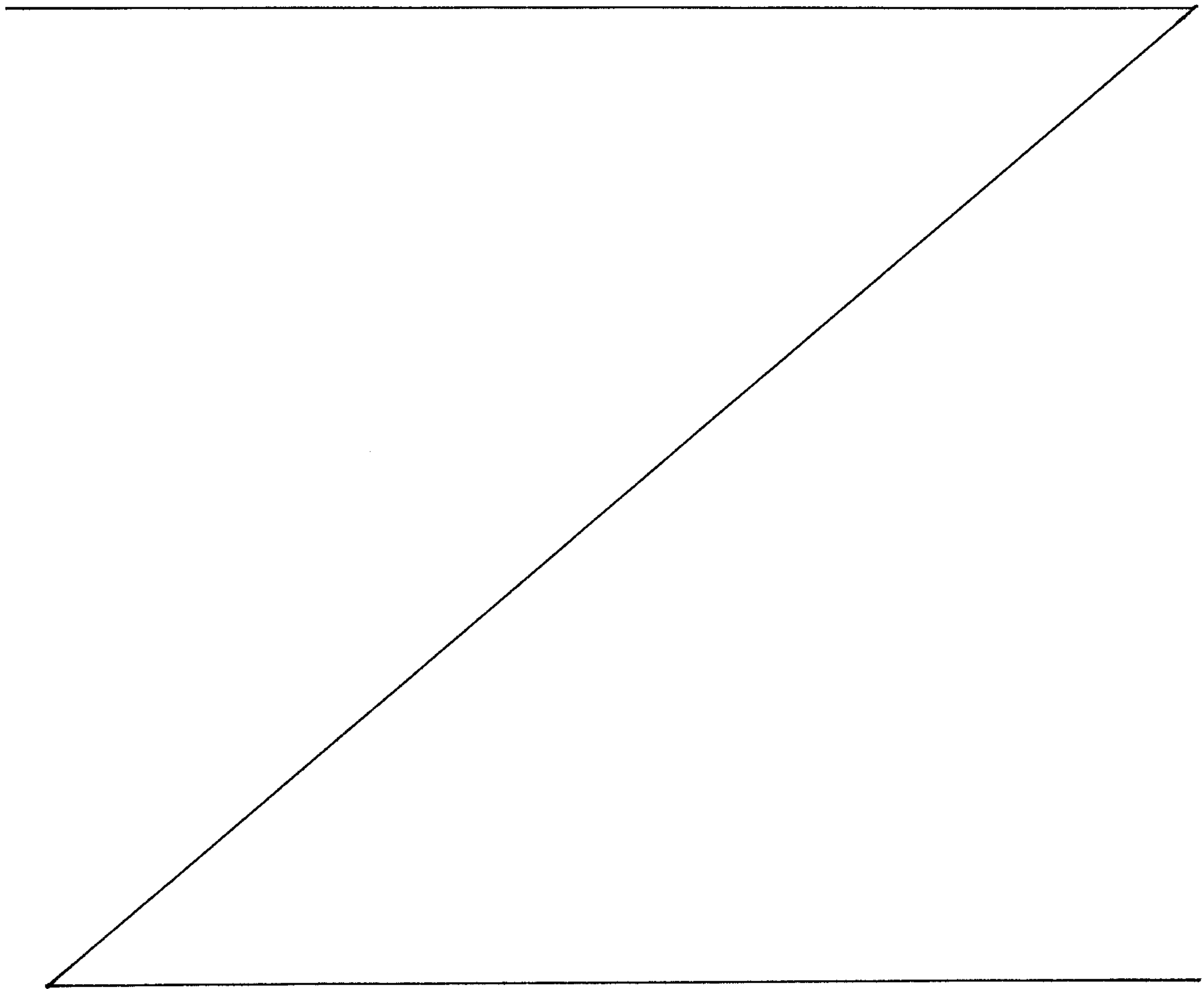
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In the following, an example of the method according to the invention and workpieces coated according to the invention are explained more specifically with reference to drawings, in which:

FIG. 1 is a schematic representation of a vapor deposition installation;

FIG. 2 is a composite illustration showing the time response of various parameters during application of layers on workpieces according to the invention; and

FIG. 3 is a composite illustration showing percentages of the vapor-deposited material over the layer thickness.



DESCRIPTION OF THE PREFERRED EMBODIMENTS

Fig. 1 shows an example of a vapor deposition installation for carrying out the method of the invention for the production of coated workpieces 2. The system has a vacuum chamber 1 with an evacuation connection 3 and a glow cathode chamber 5 with a glow cathode 6 connected to the vacuum chamber 1 via an opening 7. The glow cathode 6 is powered by a current supply unit 9. Approximately over the center of the bottom 10 of the vacuum chamber 1 is a coolable, vertically displacable crucible 11, in which titanium 13 is brought into its gaseous state by vaporization. The crucible 11 is shown in broken lines in Fig. 1 raised by a distance d from its lowest position which is shown in solid lines. The displacement takes place through a vertically displacable movement system shown at 14. System 14 consists of three cylinders telescoping by a spindle mechanism (not shown). In the vacuum chamber 1 are twelve supports 15 rotatable about the longitudinal axis of the chamber, of which two are shown, and on which the workpieces 2 to be coated are held on a mount 17 for each. The glow cathode chamber 5 further has a coolant duct, to cool its walls during operation. Gas feed lines 21 and 22 lead into the glow cathode chamber 5 and into the vacuum chamber 1 respectively. Line 22 divides in the vacuum chamber 1 into several branches 24a, 24b provided with openings 23. Two branches are shown. Inside the vacuum chamber 1, the branches and openings 23 produce a uniform distribution of the gas or gas mixture which is admitted through the gas feed line 22. Two schematically represented magnet coils 25 are located below the bottom 10 and above a cover part of the vacuum

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chamber 1 in rotational symmetry to the crucible 11 to create an approximately parallel vertical magnetic field therein.

To produce the coated workpieces 2, the workpieces are fastened on the mounts 17 of the supports 15, and titanium 13 is placed in the crucible 11. In a first process step the vacuum chamber 1 is closed, evacuated, and through the gas feed line 21 the rare gas argon is admitted until a partial pressure of 200 mPa is reached. For heating and ion etching the workpiece surfaces, a low-voltage arc burns in the argon atmosphere from the glow cathode 6 to the surfaces of the workpieces 2. For uniform heating and cleaning of the workpieces 2 arranged around the support 15, the supports rotate at about one revolution in five seconds.

The time sequence of the individual process steps for the production of the coating after the cleaning and heating step is shown in Fig. 2. The layers produced during one of the process steps, namely a bottom layer A, a separating layer B, a first partial and predominant portion or layer C of a solid solution layer and a second partial and subordinant portion or layer D of the solid solution layer, which forms the topmost layer of the coating, are plotted on the abscissa. In Fig. 2, graph a indicates the time-current curve I_{arc} of the current strength of the low voltage arc, graph b is the response of the negative bias U_{sub} at the workpieces 2, graph c is the distance d of Fig. 1 for crucible 11 from its lowest position on the bottom 10 of the vacuum chamber 1, graph d is the gas flow of argon Ar into the vacuum chamber 1 through gas feed line 21, graph e is the gas flow of acetylene C_2H_2 through the gas inlet 22, and graph f is the nitrogen gas flow N_2 . Each of the graphs is correlated in time (t) with each other.

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Following the heating and cleaning of the surfaces of the workpieces 2, in a next process step a titanium bottom layer A of approximately 1000 Å is applied directly onto the surfaces of the workpieces 2.

The materials deposited on the surface of the workpieces 2 during the entire process are plotted in Fig. 3 in their percent composition by weight at a selected distance a from the original workpiece surface. In Fig. 3, graph a, the titanium nitride content TiN is plotted, in Fig. 3, graph b, the titanium carbide TiC content is plotted, and in Fig. 3, graph c, the content of pure titanium Ti is plotted.

For applying the pure titanium layer A (see Fig. 3, graph c, and Fig. 2), a low-voltage arc 27 from the glow cathode 6 to the crucible 11 burns with a current (Fig. 2, graph a) of approximately 80 A. The workpieces 2 are at a potential with respect to the crucible 11 (Fig. 2, graph b) of about -100 V. The vaporizing titanium Ti is ionized in the gas discharge and attracted by the surfaces of the workpieces 2. For obtaining a uniform titanium layer, the crucible 11 is moved toward the glow cathode 6 and away from the bottom 10 of vacuum chamber 1 to a maximum distance $d_{\max}$ along the workpieces 2 attached to the supports 15 (see Fig. 2, graph c). The time for the production of this titanium bottom layer A is chosen so that the surfaces of the rotation workpieces 2 face the crucible 11 several times as the supports rotate.

The titanium bottom layer having been vapor deposited in a thickness approximately 1000 Å, in a next process step nitrogen N₂ is admitted through the

gas inlet 22 with the low-voltage arc 27 burning (Fig. 2, graph f), a nitrogen partial pressure of 50 mPa being adjusted so as to have a sufficient supply of nitrogen atoms and ions available for complete reaction with the vaporized titanium Ti, the vaporization being increased by increasing the current (Fig. 2, graph a) of the low voltage arc 27 to about 200 A. Through the branches 24a, 24b, etc. the nitrogen N_2 distributes itself uniformly in the vacuum chamber 1 and forms with the titanium vapor Ti titanium nitride TiN, which deposits on the surfaces of the workpieces 2. From the workpieces 2 a current of approximately 20 A flows after reduction of the negative bias (Fig. 2, graph b) to -50V. The workpieces 2 rotate also during this process step. The crucible 11 (Fig. 2, graph c) is moved from its topmost position downward toward the bottom 10 and toward the end of this process step once more upward and again downward, to obtain a uniform coating with titanium nitride TiN.

After vapor deposition of the separating layer B of titanium nitride TiN of a thickness of about one micrometer (the thickness can be varied depending on the purpose of use for the workpieces 2), acetylene C_2H_2 as a carbon-releasing gas, and as shown in Fig. 2, graph e, is admitted together with the nitrogen N_2 through the gas inlet 22 in a further process step and distributed evenly in the vacuum chamber 1 through the branches 24a, 24b etc. In proportion as the percentage of acetylene C_2H_2 increases in the vacuum chamber 1, the nitrogen percentage N_2 is reduced as seen in Fig. 2, graphs e and f. The acetylene C_2H_2 is dissociates and the dissociates carbon is ionized in the vacuum chamber. The ionized carbon as well as the ionized nitrogen combine

with the titanium vapor Ti to titanium nitride TiN and titanium carbide TiC respectively. The inflow of acetylene is increased and the inflow of nitrogen decreased until there is 70% nitrogen N_2 and 30% acetylene C_2H_2 in the vacuum chamber 1. The inflow of argon continues (see Fig. 2, graph d). During the increase of the acetylene inflow, the crucible 11 (see Fig. 2, graphs c) is moved back and forth twice. During the back and forth movement a solid solution layer forms the partial layer C, with a layer thickness of about two micrometers of titanium carbide TiC and titanium nitride TiN. Depending on the purpose of use for the workpieces 2, the layer thickness is preferably 1.2 to 2 times the layer thickness of the separating layer B.

After the acetylene inflow has reached 30% compared to the nitrogen inflow, in a further process step a partial layer D about one micrometer thick (thinner partial layer D of the solid solution layer) forms with this constant nitrogen-acetylene ratio and forms $TiC_{0.3}N_{0.7}$. Depending on the purpose of use for the workpieces 2, this layer thickness is between one fifth and one half of the preceding layer C.

Surprisingly, due to the above described rotation of the workpieces 2, as well as the up and down movement of the crucible 11, the carbon inclusions that impair adhesivity and toughness disappeared in the applied layers C and D. Also it was observed on the basis of prepared microsections on coated workpieces 2 that superposed on a steady increase of the titanium carbide content of the applied layer C were concentration fluctuations k of titanium carbide TiC to titanium nitride TiN which are shown in Fig. 3 and are in agreement with the movements of the crucible 11. Superposed on these fluctuations were additional concentration fluctuations s which correlate with the rotation of the supports 15.

Fig. 3 shows the concentration of pure titanium Ti (graph c), titanium nitride TiN (graph a) and titanium carbide TiC (graph b) according to a microsection of a coated workpiece. The fluctuation of small amplitude s and small period in the thickness zone C and D (see also Fig. 2) are due to the rotation of the workpieces 2 on the supports 15. The two fluctuations of large amplitude k and large period in zone C, as well as the one fluctuation in zone D are attributable to the two up and down movements of the crucible 11 during the vaporization of the layer in zone C and the one up and down movement in zone D.

The broken lines g_1 and g_2 in fig. 3, graph a and respectively j_1 and j_2 in Fig. 3, graph b indicate the percent titanium carbide TiC and respectively titanium nitride content TiN of the vapor-deposited layer if no crucible movements and no workpiece movements had taken place. The course of lines m_1 to m_4 for the titanium nitride TiN and n_1 to n_4 for the titanium carbide TiC indicates the response of the percent titanium nitride TiN and titanium carbide content TiC of a vapor-deposited layer if the rotation of the workpieces 2 around support 15 had been dispensed with. The indices used are identical with the indices of the movement of the crucible 11 in Fig. 2, graph c. The curves shown in Fig. 3 apply to workpieces 2 near the bottom 10. For workpieces 2 near the cover parts 26 the line m_1 is flapped up symmetrically to line g_1 , and the line g_1 down symmetrically to line j_1 . the equivalent applies to the lines m_2 and n_2 . Line m_3 then lies above line g_1 or g_2 and line n_3 below line j_1 and j_2 , respectively.

As known already from European Patent document A 0191554, workpieces which are coated only with titanium nitride TiN are, due to their reduced hardness, less resistant to flank wear than workpieces which are coated only with titanium carbide TiC. However, a workpiece coated only with titanium carbide TiC has, because of the low chemical resistance, greater crater wear. In European Patent document A 0191554 the attempt was made to combine both advantages by applying on a titanium nitride layer TiN a titanium carbide TiC or titanium carbonitride layer TiCN.

It has now been found that the coating made according to the method of the invention, in which there are no longer any discrete individual layers but only layers whose inclusion ratio fluctuates continuously over the layer thickness, have a significantly higher adhesivity on the workpiece and the workpieces have a significantly higher toughness than those coated according to the known method.

In long-term tests it was possible to cut 7000 threads with an uncoated M8 tap. With a tap coated with TiN 25,000 threads could be cut, and with a tap with a coating according to the invention 75,000 threads were cut. With a punch of 1.0334 material, 20,000 stampings were obtained for uncoated material, 62,000 for TiN-coated, and 140,000 for material coated according to the invention.

The concentration fluctuations within the coating can very likely be explained by the different affinity of ionized carbon and nitrogen to titanium vapor. As long as only nitrogen is present, the nitrogen combines with the titanium. If ionized carbon is present, the carbon combines preferably with the titanium, that is, the zone

directly around the crucible 11 is depleted of carbon, as the carbon has combined with the titanium. Therefore, with decreasing distance of a workpiece surface from the crucible 11 more titanium nitride TiN precipitates on the surface thereof.

Instead of using acetylene C_2H_2 as the carbon-releasing gas, ethylene C_2H_4 or other carbon-releasing gases can be used.

Instead of the two fluctuations in the first partial layer C of the solid solution layer, several fluctuations can be produced by a greater number of crucible movements; preferably one to five fluctuations per two micrometers of layer thickness are produced. Also the rotation of the workpieces 2 around the supports 15 can be varied in the range of from 5 to 100 period per crucible movement cycle.

Instead of transforming the titanium 13 in crucible 11 into the gaseous state with a low-voltage arc, cathode sputtering, plasma-supported vaporization, or cathodic arc vaporization may be used.

While specific embodiments of the invention have been shown and described in detail to illustrate the application of the principles of the invention, it will be understood that the invention may be embodied otherwise without departing from such principles.

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The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows.

1 1. A coated workpiece with a firmly adhering, tough solid
2 solution coating which is applied by a reactive physical
3 coating process and which comprises at least two metalloids
4 whose concentration ratio relative to each other, at least in
5 a predominant solid solution coating sub-region, continuously
6 increases and decreases with a periodic component of change in
7 the direction of the surface normal.

1 2. A coated workpiece according to claim 1 including a
2 separating layer consisting essentially of only one of the
3 metalloids and connected between the surface of the workpiece
4 portion and the predominant portion of the layer thickness of
5 the solid solution coating, the concentration ratio of the at
6 least two metalloids in the predominant portion of the layer
7 thickness changing over steadily from the separating layer to
8 the predominant portion of the layer thickness in the solid
9 solution coating, the solid solution coating being essentially
10 1.2 to 2 times as thick as the separating layer.

1 3. A coated workpiece according to claim 1, wherein the
2 solid solution coating includes the predominant portion of the
3 layer thickness which is adjacent the workpiece portion, and a
4 subordinate portion of the layer thickness spaced away from
5 the workpiece portion, the concentration ratio of the at least
6 two metalloids being at least substantially constant over the
7 subordinate portion of the layer thickness, the concentration
8 ratio of the predominant portion of the layer thickness
9 changing over smoothly to the subordinate portion of the layer
10 thickness, the predominant portion of the layer thickness
11 being 2 to 5 times as thick as the subordinate portion of the
12 layer thickness.

1 4. A coated workpiece according to claim 1, wherein the

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2 concentration ratio of the two metalloids in the predominant
3 portion of the layer thickness of the solid solution coating
4 either increases or decreases over the thickness of the
5 predominant portion of the layer thickness.

1 5. A coated workpiece according to claim 1, where the
2 concentration ratio fluctuates periodically along the
3 thickness of the solid solution coating essentially 1 to 5
4 fluctuations per two micrometers of layer thickness.

1 6. A coated workpiece according to claim 5, wherein the
2 essentially 1 to 5 fluctuations per two micrometers of layer
3 thickness comprises a first periodic fluctuation, the
4 concentration ratio of the at least two metalloids fluctuating
5 along the solid solution coating at a second periodic
6 fluctuation which varies 5 to 100 times per fluctuation of the
7 first periodic fluctuation.

1 7. A coated workpiece according to claim 2 including a
2 bottom layer made of the same material as the workpiece
3 portion, connected between the surface of the workpiece
4 portion and the separating layer, the bottom layer being 0.01
5 to 0.5 micrometers thick.

1 8. A coated workpiece according to claim 1, wherein the at
2 least two metalloids comprise titanium nitride and titanium
3 carbide forming the solid solution coating.

1 9. A coated workpiece according to claim 2, wherein the
2 separating layer consists essentially of titanium nitride.

1 10. A coated workpiece according to claim 7, wherein the
2 bottom layer consists essentially of titanium.

1 11. A coated workpiece according to claim 3, wherein the
2 subordinate portion of the layer thickness of the solid
3 solution coating consists essentially of a solid solution

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4 containing up to 30% by weight titanium carbide and down to
5 70% by weight titanium nitride.

1 12. A reactive physical coating process for the production of
2 the coated workpiece according to one of claims 1 to 11,
3 wherein a metal which is converted into the gaseous state by
4 means of vapor source in a vacuum chamber is deposited on a
5 surface of a moved workpiece, with chemical reaction with a
6 gaseous reactant introduced into the vacuum chamber
7 characterized in that as the reactants in a first gas is
8 introduced at a continuously reduced feed flow, for the
9 formation of a first and a second metalloid, and the workpiece
10 is relatively so moved in relation to the vapor source that
11 the spacing thereof and/or the orientation thereof with
12 respect to the vapor source periodically alters at such a
13 speed that deposited on the workpiece is a solid solution
14 coating with said metalloids whose concentration ratio
15 relative to each other is made up only in the direction of the
16 surface normal from superimposition of the periodic changes in
17 concentration corresponding to the changes in spacing and/or
18 orientation and the change in concentration corresponding to
19 the change in feed flow.

1 13. The method according to claim 12, wherein the at least
2 two gases are selected to have different affinities for the
3 vaporized metal.

1 14. The method according to claim 12, wherein the metal is
2 vaporized by means of arc vaporizing.

1 15. The method according to claim 12, wherein the vaporized
2 metal is supplied from a vapor source in the vacuum chamber,
3 and including periodically moving the vapor source along the
4 surface of the workpiece portion for changing the
5 concentration ratio of the at least two metalloids in the
6 solid solution coating and in the direction of movement of the

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7 vapor source with respect to the workpiece portion.

1 16. The method according to claim 12, wherein the vapor
2 source is moved at a lower speed than the workpiece portion.

1 17. The method according to claim 12 including forming a
2 separating layer consisting essentially of only one metalloid
3 formed by only one of the gases and the metal, by vaporizing
4 the metal in the vacuum chamber and initially supplying only
5 one of the gases to the vacuum chamber, the solid solution
6 coating being formed subsequently by introducing the other of
7 the at least two gases into the vacuum chamber.

1 18. The method according to claim 16 including forming the
2 predominant portion of the layer thickness of the solid
3 solution coating by reducing the flow of one of the at least
4 two gases with respect the other of the at least two gases
5 during formation of the predominant portion of the thickness
6 of the solid solution coating, and subsequently forming a
7 SUBORDINANT portion of the thickness of the solid solution
8 coating by admitting both of the at least two gases into the
9 vacuum chamber and at a constant ratio.

1 19. The method according to claim 12, wherein one of the at
2 least two gases is nitrogen and the other of the at least two
3 gases is a carbon releasing gas, the metal comprising
4 titanium.

1 20. A method according to claim 18, wherein the inflow of
2 nitrogen and carbon releasing gas into the vacuum chamber is
3 adjusted so that the subordinate portion of the thickness of
4 the solid solution coating comprises $\text{TiC}_{0.3}\text{N}_{0.7}$.

1 21. The method according to claim 12 wherein the coated
2 workpiece is used as one of a cutting and forming tool.

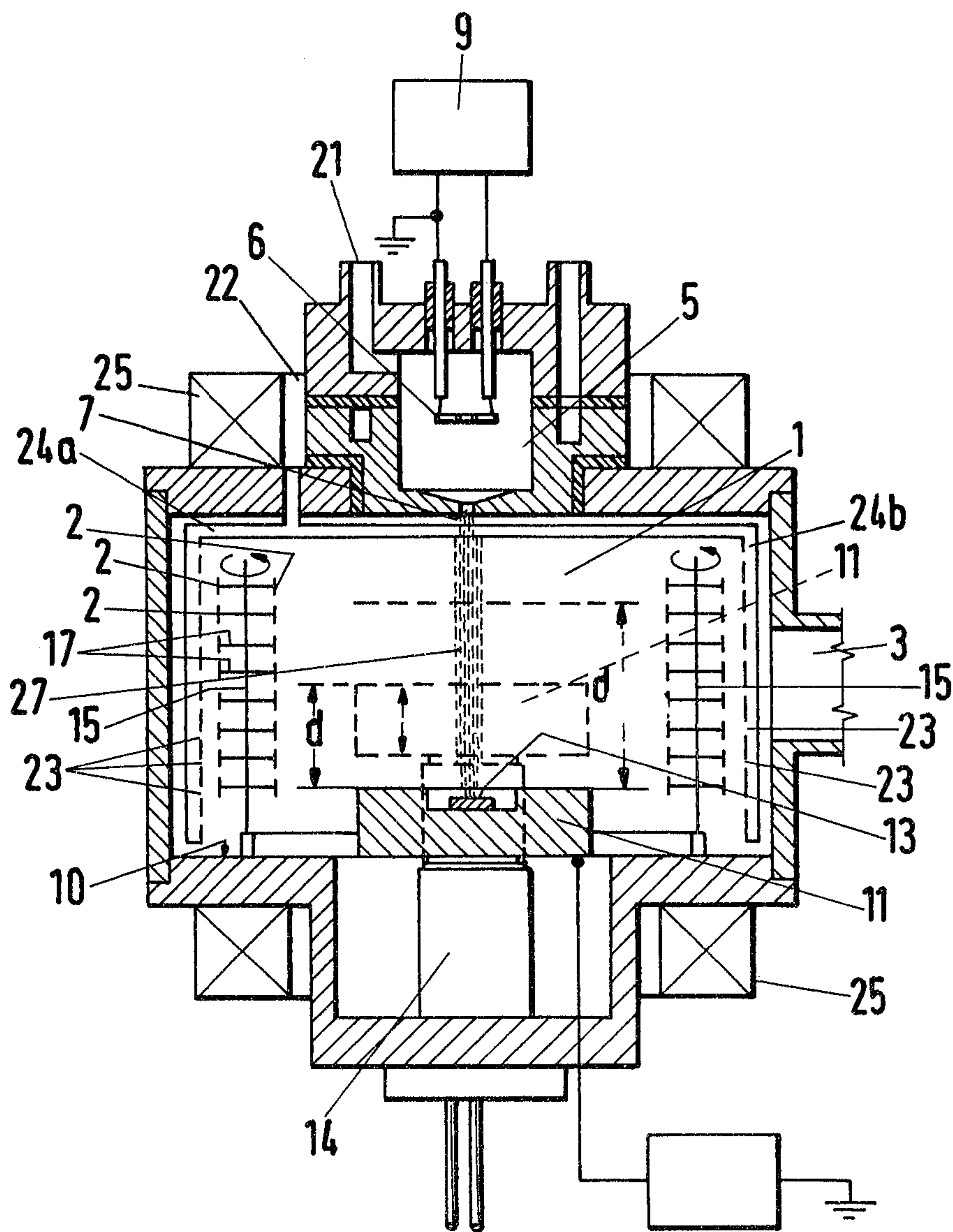
1 22. An apparatus for producing a coated workpiece made of a

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2 workpiece portion having a surface and a solid solution
3 coating of at least two metalloids on the surface, the layer
4 being deposited by a reactive physical coating process and
5 having a concentration ratio of the at least two metalloids in
6 a predominant portion of the layer thickness which varies
7 continuously over the predominant portion of the layer
8 thickness, the apparatus comprising means for forming a vapor
9 source for vaporizing a metal for use in reactions with gases
10 to form the at least two metalloids, and means for moving the
11 vapor source with respect to a workpiece portion for producing
12 the coated workpiece.

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Fig.1



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Fig.2

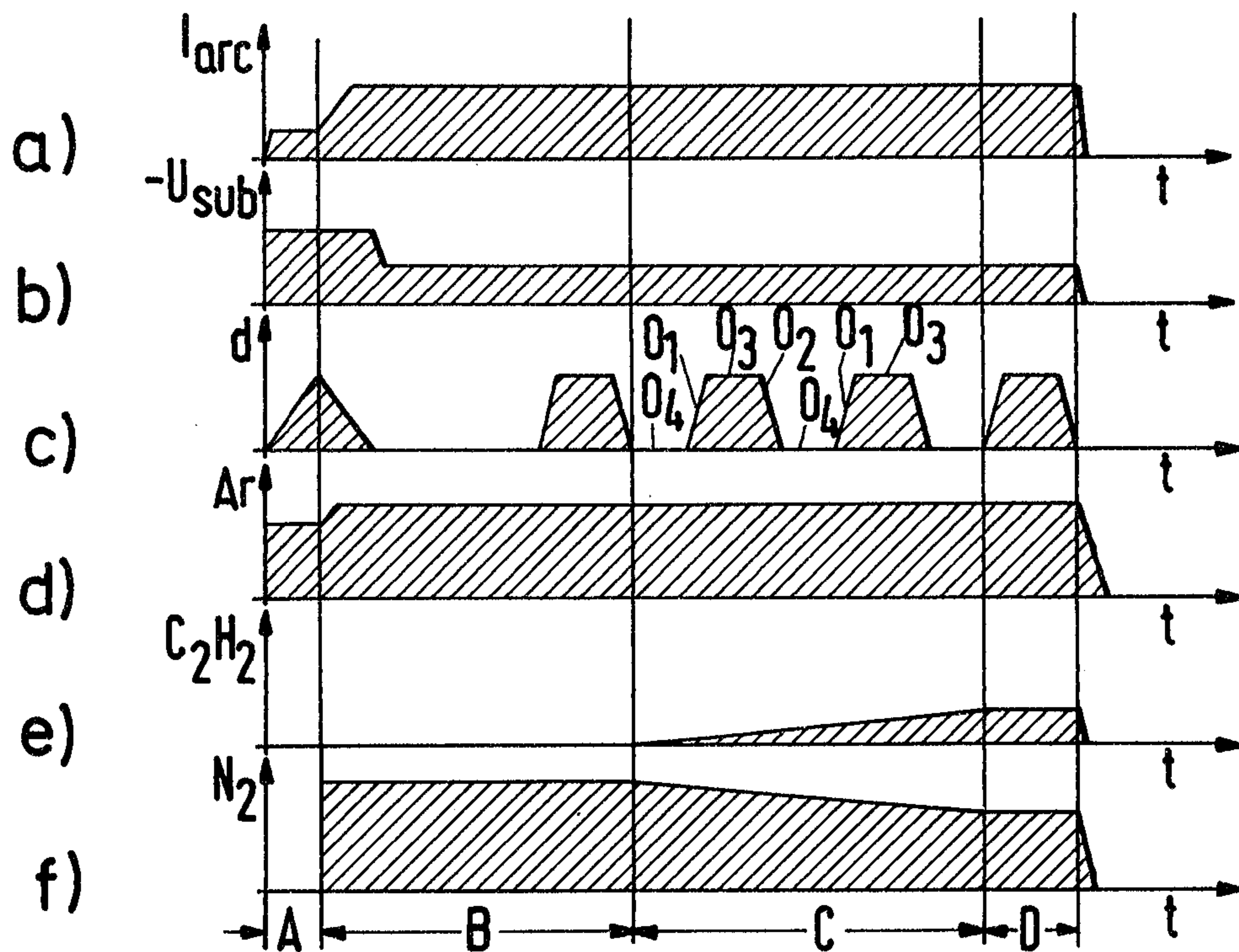
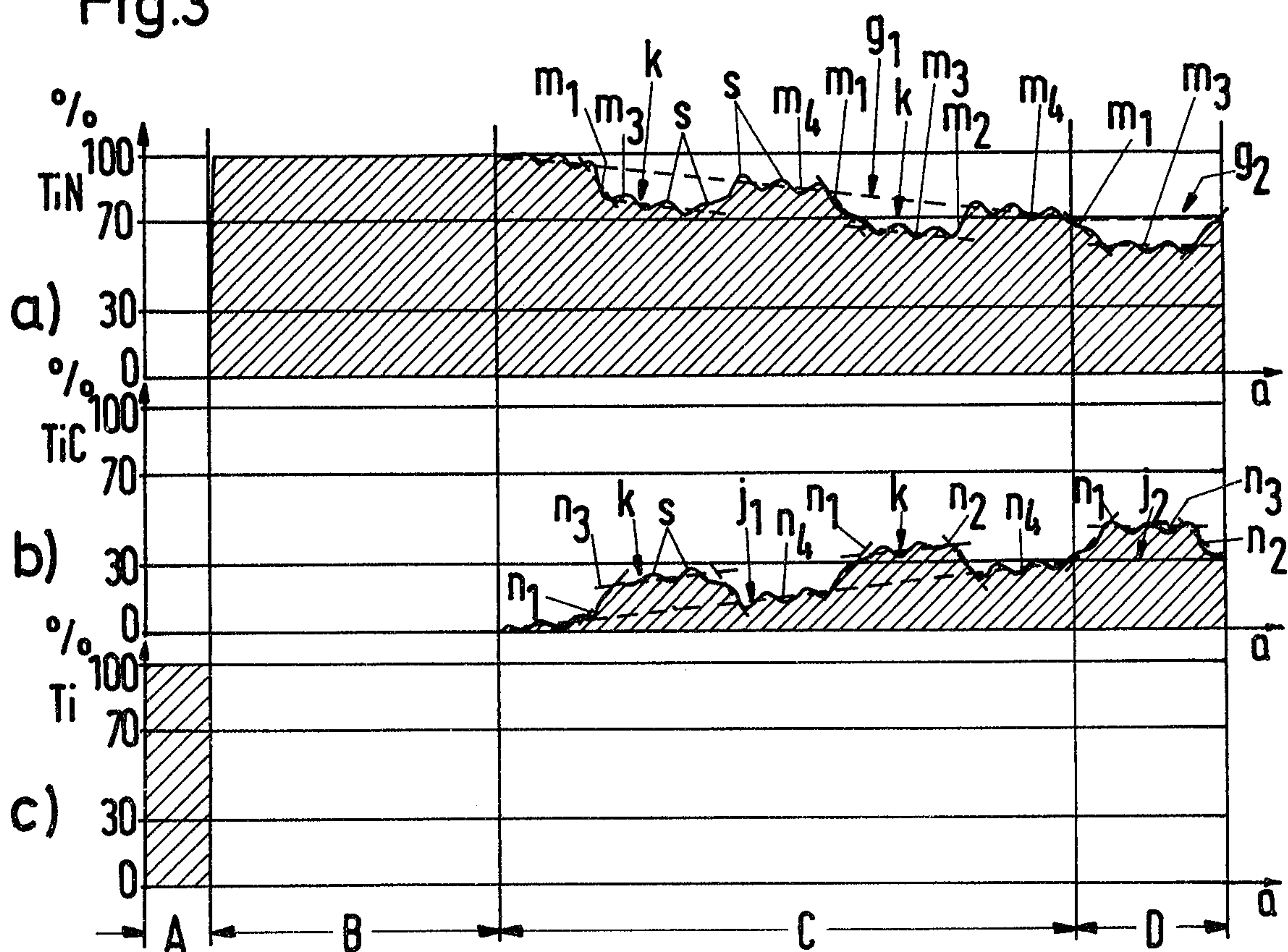


Fig.3



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