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(54) **BAINS D'EVAPORATION EN CERAMIQUE AYANT UNE
PERFORMANCE INITIALE AMELIOREE CONTRE
L'HUMIDITE ET SES PROPRIETES**

(54) **CERAMIC EVAPORATION BOATS HAVING IMPROVED
INITIAL WETTING PERFORMANCE AND PROPERTIES**

(57) The invention relates to ceramic evaporation boats for the evaporation of metal comprising a conductive component and a nonconductive component, wherein the conductive component of the ceramic material is concentrated at the surface of the evaporation boat from which the evaporation of the metal occurs. The evaporation boats have an improved initial wetting performance.

**CERAMIC EVAPORATION BOATS HAVING IMPROVED INITIAL WETTING
PERFORMANCE AND PROPERTIES**

Abstract

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10 the ceramic material is concentrated at the surface of the evaporation boat from which the evaporation of the metal occurs. The evaporation boats have an improved initial wetting performance.

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**CERAMIC EVAPORATION BOATS HAVING IMPROVED INITIAL WETTING
PERFORMANCE AND PROPERTIES**

BACKGROUND OF THE INVENTION

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The invention relates to ceramic evaporation boats having improved initial wetting performance and properties and processes for producing such products.

10 The most widely used method of coating flexible substrates with metals, in particular with aluminum, is high vacuum tape coating. The substrate to be coated is passed over a cooled roller while being exposed to aluminum vapor which deposits on the substrate surface as a thin metal layer.

15 To generate the constant vapor stream required, ceramic evaporators known as evaporation boats, are heated to about 1450°C by direct passage of electric current through the evaporation boat. Aluminum wire is continuously fed to the boat, liquefied on the ceramic surface and evaporated in a vacuum of about 10^{-4} mbar. In metallization units, a series of
20 evaporation boats are arranged in such a way that a uniformly thick aluminum layer is deposited across the entire width of the substrate.

Evaporation boats are generally made of hot-pressed titanium diboride (TiB_2) and boron nitride (BN) and/or
25 aluminum nitride (AlN). In such evaporation boats, TiB_2 is the electrically conductive component which allows the evaporator to be heated like an ohmic resistance.

30 One of the main problems in the operation of web coating units is the initial wetting of the evaporation boats with the metal to be vapor-deposited. In practice, the operator has to have a great deal of experience in order to be able to carry out the initial wetting of the evaporation boats in an optimal way. Thus, the term "break-in procedure" has

become established to describe this initial wetting of the evaporation boat which illustrates the complexity of this step. Thus, during the running-up phase, the cavity of the evaporator can be incompletely wetted. This results in
5 increased deposits on the side opposite that where the wire is fed which forces the operator to run the evaporation boat "hot" for a given evaporation rate, i.e., to heat it to a very high temperature. This leads to a drastic decrease in the life of the evaporation boats.

10 In addition, incomplete wetting corresponds to non-uniform wetting of the cavity of the evaporation boat. As a result, uniform, continuous evaporation of the metal to be evaporated is not possible later. This forces the operator to adjust the evaporator heating continually. As a result, the
15 evaporation boat is, on average, run too hot. This greatly reduces the life of the evaporation boats, as already mentioned.

In the case of evaporation boats having a very high electric resistance, the voltage of the vapor deposition unit
20 is generally insufficient to heat it to the wetting temperature. If such an evaporation boat were to be wetted more readily than conventional evaporation boats, some wetting would take place even before the full wetting temperature is reached. As a result, the system evaporation boat-aluminum
25 bath has a lower electric resistance. This immediately produces a higher current which in turn leads to better heating of the evaporation boat and consequently also to even better wetting.

This also makes the problem of the difference in the
30 resistance from evaporation boat to evaporation boat significantly less critical and stoppage of the vapor deposition units due to high-resistance evaporation boats does not occur.

The better the wetting of the evaporator material by the metal to be evaporated, the lower the risk of the evaporation boat being overheated and thus the life of the evaporation boat being drastically reduced. In addition, good wetting leads to optimum metal bath formation in the cavity of the evaporation boat and thus to improved evaporation conditions and more uniform stressing of the evaporation boat which, in turn, increases the life of the evaporation boat.

It is an object of the invention to provide an evaporation boat of ceramic material for the evaporation of metal comprising a conductive component and a nonconductive component, which boat is initially wetted more readily by the metal to be evaporated.

15 **BRIEF SUMMARY OF THE INVENTION**

The object is achieved by an evaporation boat in which the conductive component of the ceramic material is concentrated at the surface of the evaporation boat at which evaporation of the metal occurs.

Preferably, the content of conductive component at the surface of the evaporation boat at which evaporation of the metal occurs is at least two % (2%) (relative) higher than in the remaining material of the evaporation boat.

25 Preferably, a layer of the conductive component of the ceramic material is located on the surface of the evaporation boat at which the evaporation of the metal occurs.

The layer of conductive component should preferably be in electrical contact with the remaining evaporator boat material. As a result, this layer becomes self-conducting and, owing to the low resistivity of the material, becomes hotter than the remaining evaporator boat material. This again leads to an improvement in the wettability of the surface of the

evaporator boat.

The concentration of the conductive component at the surface of the evaporation boat is preferably achieved starting from an evaporation boat known from the prior art by means of one of the three methods described below:

- 1) The surface of the evaporation boat from which the evaporation of the metal is to take place in normal operation is heated by means of a high-energy beam so that the nonconductive components [in general BN (melting point: 2300°C) and AlN (melting point: 2300°C)] evaporate and at the same time the conductive component [in general TiB₂ (melting point: 2900°C)] is only melted. The energy content of the high-energy beam is therefore preferably selected such that it heats the surface of the evaporation boat to more than 2900°C but not less than 2700°C. This results, after cooling, in a layer enriched in the conductive ceramic component (in general TiB₂) on the surface of the evaporation boat. Briefer heating gives a layer which is less enriched in the conductive ceramic component on the surface of the evaporation boat.
- 2) Powder comprising the conductive ceramic component can be applied to the surface of the evaporation boat and welded on by means of a high-energy beam so as to form an electrically conductive layer of the conductive ceramic material. This can be achieved, for example, in the case of TiB₂ as a conductive ceramic component, using a method analogous to a TiB₂ powder coating process known per se.
- 3) Powder comprising the conductive ceramic component can be processed with an organic or inorganic binder to form a paste and the paste applied to the surface of the evaporation boat. The binder is selected so that it evaporates during the heating of the evaporation boat.

Thus, when the evaporation boat is heated, the binder evaporates and the desired electrically conductive layer which can subsequently be wetted by aluminum is formed. The binder used can be, for example, glycerol. This layer can be additionally treated by means of a high-energy beam as described in 2) to obtain better contact between the electrically conductive layer and the remaining evaporator material.

10 **DETAILED DESCRIPTION OF THE INVENTION**

As a rule, the electrically conductive component of the ceramic material is TiB_2 .

For this reason, a TiB_2 -containing powder is generally used as a powder comprising the conductive ceramic component. Preference is given to using TiB_2 powder.

As the high-energy beam, it is possible to use, for example, a laser beam. The laser used can be, for example, a gas, solid-state or semi-conductor laser.

20 The heating of the surface of the ceramic evaporation boat by means of a high-energy beam is preferably carried out under inert gas conditions. Examples of inert gases are helium and argon.

The evaporation boats of the present invention have the following advantages over known evaporation boats:

- 1) From the commencement of use, they display good uniform wetting which leads to a constant (over time) and uniform (in space) evaporation rate without scattering.
- 2) They run in a steady manner from the beginning and the power does not have to be continually adjusted.

3) The scattering in the resistance of the evaporation boats which results from the scarcely avoidable scatter in the resistance of the sintered body from which the evaporation boats are produced does not have the adverse effect of the higher-resistance evaporation boats no longer being wettable and the vapor deposition units therefore having to be stopped.

The following examples illustrate the invention.

Example 1: Production of an evaporation boat according to the invention

The surface from which metal evaporation is to take place of an evaporation boat having the dimensions 10x20x120 mm and produced from a ceramic material consisting of 47.5% by weight of TiB_2 and 52.5% by weight of BN was irradiated by means of a YAG laser (wavelength = 1.06 μm /beam diameter = 6 mm/power = 100 W) in a plurality of traces (80 mm long) in a stream of argon.

Example 2: Production of an evaporation boat according to the invention

Grooves having a depth of about 0.5 mm were scratched mechanically into the surface of an evaporation boat from which metal evaporation is to take place (10x20x120 mm/47.5% by weight of TiB_2 and 52.5% by weight of BN). TiB_2 powder was sprinkled into these grooves. This powder was irradiated by means of a YAG laser as described in Example 1. Unbound powder residues were subsequently blown off using compressed air.

Example 3: Production of an evaporation boat according to the invention

A paste of TiB_2 powder and glycerol as binder was applied by means of a spatula to the surface of an evaporation boat from which metal evaporation is to take place (10x20x120 mm/47.5% by weight of TiB_2 and 52.5% by weight of BN).

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Example 4: Comparison of the evaporation boats of the invention with a known evaporation boat

As a comparison evaporation boat, a further evaporation boat having the same dimensions as described above was produced from the same batch of sintered material (47.5% by weight of TiB_2 and 52.5% by weight of BN) from which the evaporation boats described in Examples 1 to 3 had been produced.

This comparison evaporation boat and the evaporation boats of the invention produced in Examples 1 to 3 were compared under defined conditions.

The evaporators were clamped in place at the end faces and, before heating, 2 g of Al wire was laid on the middle of the surface of the evaporation boat from which metal evaporation is to occur. A vacuum of $< 1 \times 10^{-4}$ mbar was applied. In this high vacuum, the evaporation boats were heated linearly over a period of 10 minutes to a power of 3.96 KW by means of a power ramping software program.

The evaporator voltage was measured directly at the end faces of the evaporators. At the same time, the current flowing through the evaporation boat was measured at a different point in the circuit. The measured values for voltage and current were recorded at one second intervals. They were available as a data file after the experiment.

The wetting of the evaporator surface by the aluminum was generally indicated by a disproportionate increase in the current at a given voltage compared to the increase in the current through an evaporator without aluminum placed on it,

since the wetting aluminum makes possible an additional flow of current compared to an evaporator without aluminum.

If the heating power at which this increase in current takes place is calculated for the glowing surface, a wetting
5 power per unit area of glowing surface of $44\text{W}/\text{cm}^2$ is obtained for the comparison evaporation boat while that for the evaporation boats from Examples 1 to 3 is $< 40\text{W}/\text{cm}^2$.

This result demonstrates that the evaporation boats of
the invention were wetted at lower temperatures than the
10 comparison evaporation boat and thus have a better initial wetting performance than known evaporators.

Claims:

1. An evaporation boat of ceramic material for the evaporation of metal comprising a conductive component and a nonconductive component, wherein the conductive component of the ceramic material is concentrated at the surface of the evaporation boat from which the evaporation of the metal occurs.
2. The evaporation boat as claimed in claim 1, wherein the content of conductive component at the surface of the evaporation boat from which the evaporation of the metal occurs is at least two % (relative) higher than in the remaining material of the evaporation boat.
3. The evaporation boat as claimed in claim 1, wherein a layer of the conductive component is located on the surface of the evaporation boat from which the evaporation of the metal occurs.
4. The evaporation boat as claimed in claim 1, wherein the conductive component is TiB_2 .
5. The process for producing an evaporation boat as claimed in claim 1, wherein the surface of an evaporation boat from which the evaporation of the metal occurs in normal operation is heated by means of a high-energy beam so that the non-conductive components of the evaporation boat evaporate and at the same time the conductive component of the evaporation boat is only melted.
6. The process for producing an evaporation boat as claimed in claim 1, wherein a powder comprising the conductive component of the ceramic material is applied to the surface of an evaporation boat from which evaporation of the metal occurs and the powder is welded to the surface by means of a high-energy beam so as to form an electrically conductive layer of the conductive material on the surface from the evaporation of the metal occurs.

7. The process as claimed in claim 6, wherein the electrically conductive component of the ceramic material of the evaporator is TiB_2 and the powder comprising the conductive component is a TiB_2 -containing powder.
- 5 8. The process as claimed in claim 6, wherein the high-energy beam comprises a laser beam.
9. A process for producing an evaporation boat as claimed in claim 1, wherein the surface of an evaporation boat from which evaporation of the metal occurs is coated with a paste
10 comprising the conductive component of the ceramic material and at least one binder selected from the group consisting of organic binders and inorganic binders which binders vaporise during the heating of the evaporation boat.
10. A process as claimed in claim 9, wherein the binder
15 used is glycerol.
11. The evaporation boat as claimed in claim 2, wherein the conductive component is TiB_2 .
12. The evaporation boat as claimed in claim 3, wherein the conductive component is TiB_2 .
- 20 13. The process for producing an evaporation boat as claimed in claim 2, wherein the surface of an evaporation boat from which the evaporation of the metal occurs in normal operation is heated by means of a high-energy beam so that the non-conductive components of the evaporation boat evaporate
25 and at the same time the conductive component of the evaporation boat is only melted.
14. The process for producing an evaporation boat as claimed in claim 3, wherein the surface of an evaporation boat from which the evaporation of the metal occurs in normal
30 operation is heated by means of a high-energy beam so that the non-conductive components of the evaporation boat evaporate

and at the same time the conductive component of the evaporation boat is only melted.

15. The process for producing an evaporation boat as claimed in claim 4, wherein the surface of an evaporation boat
5 from which the evaporation of the metal occurs in normal operation is heated by means of a high-energy beam so that the non-conductive components of the evaporation boat evaporate and at the same time the conductive component of the evaporation boat is only melted.

10 16. The process for producing an evaporation boat as claimed in claim 2, wherein a powder comprising the conductive component of the ceramic material is applied to the surface of an evaporation boat from which evaporation of the metal occurs and the powder is welded to the surface by means of a high-
15 energy beam so as to form an electrically conductive layer of the conductive material on the surface from the evaporation of the metal occurs.

17. The process for producing an evaporation boat as claimed in claim 3, wherein a powder comprising the conductive
20 component of the ceramic material is applied to the surface of an evaporation boat from which evaporation of the metal occurs and the powder is welded to the surface by means of a high-energy beam so as to form an electrically conductive layer of the conductive material on the surface from the evaporation of
25 the metal occurs.

18. The process for producing an evaporation boat as claimed in claim 4, wherein a powder comprising the conductive component of the ceramic material is applied to the surface of an evaporation boat from which evaporation of the metal occurs
30 and the powder is welded to the surface by means of a high-

energy beam so as to form an electrically conductive layer of the conductive material on the surface from the evaporation of the metal occurs.

19. The process as claimed in claim 7, wherein the high-
5 energy beam comprises a laser beam.

20. A process for producing an evaporation boat as claimed in claim 2, wherein the surface of an evaporation boat from which evaporation of the metal occurs is coated with a paste comprising the conductive component of the ceramic material
10 and at least one binder selected from the group consisting of organic binders and inorganic binders which binders vaporise during the heating of the evaporation boat.

21. A process for producing an evaporation boat as claimed in claim 3, wherein the surface of an evaporation boat from
15 which evaporation of the metal occurs is coated with a paste comprising the conductive component of the ceramic material and at least one binder selected from the group consisting of organic binders and inorganic binders which binders vaporise during the heating of the evaporation boat.

20 22. A process for producing an evaporation boat as claimed in claim 4, wherein the surface of an evaporation boat from which evaporation of the metal occurs is coated with a paste comprising the conductive component of the ceramic material and at least one binder selected from the group consisting of
25 organic binders and inorganic binders which binders vaporise during the heating of the evaporation boat.

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