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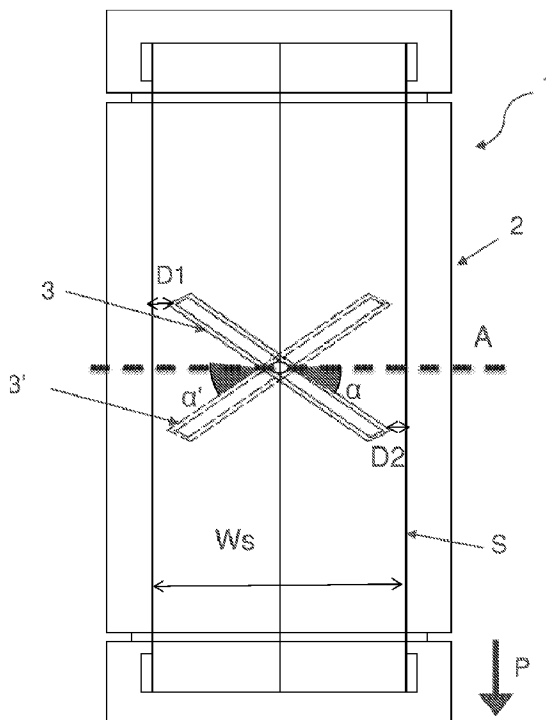
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(54) Titre : INSTALLATION DE DEPOT SOUS VIDE ET PROCEDE DE REVETEMENT D'UN SUBSTRAT  
(54) Title: VACUUM DEPOSITION FACILITY AND METHOD FOR COATING A SUBSTRATE

Figure 1



(57) **Abrégé/Abstract:**

The present invention relates to A Method for continuously depositing, on a running substrate, coatings formed from at least one metal inside a Vacuum deposition facility comprising a vacuum chamber; a substrate coated with at least one metal on both sides

(57) **Abrégé(suite)/Abstract(continued):**

of the substrate having an average thickness, wherein the coating is deposited homogenously such that the maximum thickness of the coating can exceed the average thickness of 15% maximum and a vacuum deposition facility.

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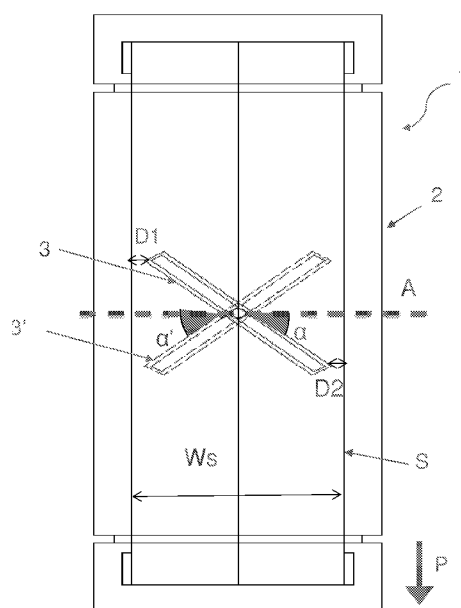
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## (54) Title: VACUUM DEPOSITION FACILITY AND METHOD FOR COATING A SUBSTRATE

Figure 1



(57) Abstract: The present invention relates to A Method for continuously depositing, on a running substrate, coatings formed from at least one metal inside a Vacuum deposition facility comprising a vacuum chamber; a substrate coated with at least one metal on both sides of the substrate having an average thickness, wherein the coating is deposited homogeneously such that the maximum thickness of the coating can exceed the average thickness of 15% maximum and a vacuum deposition facility.

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## Vacuum deposition facility and method for coating a substrate

5 The present invention relates to a method for continuously depositing, on a substrate, coatings formed from metal or metal alloys. The present invention also relates to a vacuum deposition facility used in this method.

10 Various processes for depositing metal coatings, eventually composed of alloys, on a substrate, such as a steel strip, are known. Among these, mention may be made of hot-dip coating, electrodeposition and also the various vacuum deposition processes, such as vacuum evaporation and magnetron sputtering.

15 It is known from WO97/47782 a method for the continuous coating of a steel substrate in which a metallic vapor spray, propelled at a speed greater than 500m/s, comes in contact with the substrate. The deposition method is called jet vapor deposition.

20 EP2048261 discloses a vapor generator for depositing a coating on a metallic substrate, and comprises a vacuum chamber in the form of an enclosure provided with a unit to ensure a state of depression with respect to the external environment and a unit allowing entry and exit of the substrate. The enclosure comprises a head for vapor deposition, and an ejector for creating a metal vapor jet at the sonic speed in the direction of and perpendicular to the substrate surface. The ejector is sealably connected with a crucible by a supply pipe. The crucible contains a mixture of metals in liquid form, and is located outside the vacuum chamber and fed by pumping or by barometric effect of the melt obtained from a melting furnace placed at atmospheric pressure. A unit is arranged to regulate flow, pressure and/or speed of the metal vapor in the ejector. The regulation unit comprises a butterfly type proportional valve and/or a pressure drop device arranged in the pipe. The ejector comprises a longitudinal slit as sonic collar for vapor exit extending on the whole width of the substrate, and a sintered filter medium or a pressure loss body for standardizing and correcting the velocity of the vapor exiting from the ejector.

30 In EP2048261, preferably, the generator comprises a means for adjusting the length of the longitudinal slit of the ejector to the width of the substrate. In particular, a simple system for adjusting the vapor jet slot to the width of the strip by rotation of

the ejector around its axis is disclosed. Thus, the edges of the vapor jet and the edges of the substrate are in same plans, i.e. the distances between edges of the vapor jet and the edges of the substrate are equal to 0mm. The generator can comprise two ejectors located on both side of the metallic substrate.

5           Nevertheless, by using such generator, there is a risk that during the deposition process, metal vapors are heterogeneously deposited on the metallic substrate. Indeed, it has been observed that the vapors tend also to accumulate in some areas of the metallic substrate for example on the edges of the substrate.

10           The aim of the present invention is therefore to provide a method for depositing coatings on a running substrate wherein metal vapors are homogeneously deposited on both sides of the metallic substrate.

          This is achieved by providing a method for depositing coatings on a running substrate according to claim 1. The method can also comprise any characteristic of claims 2 to 13.

15           The invention also covers a coated substrate according to claims 14 to 16.

          The invention also covers a vacuum facility according to claims 17 or 18.

          To illustrate the invention, various embodiments and trials of non-limiting examples will be described, particularly with reference to the following Figure:

20           Figure 1 illustrates a top view of a substrate coated with two vapor ejectors inside a vacuum deposition facility according to the present invention.

          Figure 2 illustrates a top view of a substrate coated with two vapor ejectors inside a vacuum deposition facility according to the prior art.

          Figure 3 illustrates a side view of a substrate coated with two vapor ejectors inside a vacuum deposition facility according to the present invention.

25           Figure 4 illustrates an example of a vapor ejector ejecting a metallic vapor according to the present invention.

          Figure 5 illustrates a side view of a substrate coated with two vapor ejectors inside a vacuum deposition facility according to the preferred embodiment.

30           Other characteristics and advantages of the invention will become apparent from the following detailed description of the invention.

          The invention relates to a method for continuously depositing, on a running substrate, coatings formed from at least one metal inside a Vacuum deposition facility, wherein the method comprises:

- a step in which in the said vacuum chamber, a metallic vapor is ejected through at least two vapor ejectors, towards both side of the running substrate and a layer of at least one metal is formed on each side by condensation of ejected vapors, the at least two vapor ejectors facing each other being located on both sides of the substrate and being positioned respectively with an angle  $\alpha$  and  $\alpha'$ , being between the vapor ejector and the axis A perpendicular to the running direction of the substrate, the axis being in the plane of the substrate,  $\alpha$  and  $\alpha'$  both satisfying the following equation:

$$(D1 + D2) + Le \sin \alpha + We \cos \alpha = W_s, \text{ and}$$

$$(D1 + D2) + Le \sin \alpha' + We \cos \alpha' = W_s$$

$\alpha$  and  $\alpha'$   $\alpha$  in absolute value being above  $0^\circ$  and

D1 and D2 being the lower distance between ejectors and each substrate edge along the axis (A),  $W_s$  being the substrate width, D1 and D2 being above 0mm and

- said vapor ejectors having an elongated shape and comprising a slot and being defined by a slot length  $Le$  and a slot width  $We$ , said vapor ejectors having the same rotation axis.

Without willing to be bound by any theory, it is believed that with the method according to the present invention, it is possible to obtain a coating having a homogeneous thickness. Indeed, the inventors have found that the at least two vapor ejectors have to be positioned respectively with a specific angle  $\alpha$  and  $\alpha'$  so that metal vapors are ejected almost without any lost. When  $\alpha$  and  $\alpha'$  satisfy the equation, the trajectory of the ejected metal vapor is well controlled to be deposited in the entire surface of both sides of the metallic substrate. Thus, the yield of the metal vapor deposited is highly improved. Moreover, the metal vapor is homogenously deposited on both sides of the running substrate leading to a coating having a constant thickness.

With reference to Figure 1, the facility 1 according to the invention first comprises a vacuum chamber 2 and a means for running the substrate through the chamber. This vacuum chamber 2 is a hermetically-sealable box preferably kept at a pressure of between  $10^{-8}$  and  $10^{-3}$  bar. It has an entry lock and an exit lock (these

not being shown) between which a substrate S, such as for example a steel strip, can run along a given path P in a running direction.

The at least two vapor ejectors 3, 3' eject metallic vapors at sonic speed on both sides of the running substrate. Both vapor ejectors are positioned respectively  
5 with an angle  $\alpha$  and  $\alpha'$  between the vapor ejector and the axis A perpendicular to the running direction of the substrate, the axis being in the plane of the substrate,  $\alpha$  and  $\alpha'$  both satisfying the following equations:

$$(D1 + D2) + L_e \sin \alpha + W_e \cos \alpha = W_s, \text{ and}$$

$$(D1 + D2) + L_e \sin \alpha' + W_e \cos \alpha' = W_s.$$

10 The vapor ejectors can have different shapes, such as a rectangular shape or trapezoidal shape. Different distances values of D1 and D2 are possible as illustrated in Figure 1. Preferably, D1 and D2 represent the lowest distance between the ejector edges and the substrate edges along the axis A.

According to the present invention, D1 and D2 are above 0mm, i.e. the ejector  
15 edges do not go beyond the substrate edges. Without willing to be bound by any theory, it is believed that if D1 and D2 are equal or below to 0mm, there is a risk that the trajectory of the metallic vapor ejected through the vapor ejectors is not controlled leading to heterogeneous coating deposition. When D1 and D2 are below zero, it means that the edges of the vapor ejector extend beyond the substrate  
20 edges as illustrated in Figure 2.

Preferably, D1 and D2 are independently from each other and above 1 mm, advantageously between 5 and 100mm and more preferably between 30 and 70mm.

In a preferred embodiment, D1 is identical to D2.

25 Preferably, the length of the ejector split  $L_e$  is between 5 and 50 mm.

Preferably, the substrate width  $W_s$  is maximum of 2200mm. Advantageously,  $W_s$  is minimum of 200mm. For example,  $W_s$  is between 1000 and 1500mm.

Preferably,  $W_e$  is maximum of 2400mm. Advantageously,  $W_e$  is minimum of 400mm.

30 In a preferred embodiment,  $W_s$  is smaller or equal to  $W_e$ .

Preferably,  $\alpha'$  is such that  $\alpha - \alpha' < 10^\circ$ , more preferably  $\alpha - \alpha' < 5^\circ$  and advantageously,  $\alpha - \alpha' < 3^\circ$  in absolute terms. For example,  $\alpha - \alpha'$  are equal to  $0^\circ$ .

Preferably,  $\alpha$  is between 5 and 80°, advantageously between 20 and 60° in absolute terms and for example between 35 and 55° in absolute terms.

The vacuum chamber can comprise three or several vapor ejectors positioned on both sides of the running substrate. For example, the vacuum chamber can comprise two vapor ejectors positioned on each side of the metallic substrate.

As illustrated in Figure 3, the substrate S may be made to run by any suitable means, depending on the nature and the shape of said substrate. A rotary support roller 4 on which a steel strip can bear may in particular be used.

With reference to Figure 4, the two vapor ejectors 3, 3' according to the present invention ejects a metallic vapor jet 5 at sonic speed on the running substrate (not represented). The at least two vapor ejectors have an elongated shape and comprises a slot and is defined by a slot length  $L_e$ , a slot width  $W_e$ .

As illustrated in Figure 5, the vacuum chamber 2 can further comprise a central casing 6. This is a box surrounding the substrate path P on a given length in the running direction, typically 2 to 8 m long in the case of one ejector per side. Its walls delimit a cavity. It comprises two apertures, i.e. a substrate entry 7 and a substrate exit 8 located on two opposite sides of the central casing. Preferably the central casing is a parallelepiped which width is slightly larger than the substrates to be coated.

Preferably, the inner walls of the central casing are suited to be heated at a temperature above the condensation temperature of the metal or metal alloy vapors. The heating may be made by any suitable means, such as for example an induction heater, heating resistors, electron beam. The heating means are suited to heat the inner walls of the central casing at a temperature high enough to avoid condensation of metal or metal alloy vapors on them. Preferably, the walls of the central casing are suited to be heated above the condensation temperatures of the metal elements forming the coating to be deposited, typically above 500°C, for example between 500°C and 700°C so as to avoid the condensation of zinc vapors or zinc-magnesium alloy vapors. Thanks to these heating means, the inner walls of the central casing do not become clogged and the facility does not have to be frequently stopped for cleaning. Moreover, the condensation of metal or metal alloys vapors on the inner walls is avoided.

In particular, with the method according to the present invention, it is possible to obtain a metallic substrate coated with at least one metal on both sides of the substrate having an average thickness, wherein the coating is deposited homogeneously such that the maximum thickness of the coating can exceed the average thickness of 15% maximum.

In the present invention, the at least one metal is preferably chosen among: zinc, chromium, nickel, titanium, manganese, magnesium, silicon, aluminum or a mixture thereof. Preferably, the metal is zinc with optionally magnesium.

Preferably, the metallic substrate is a steel substrate. Indeed, without wishing to be bound by any theory, it is believed that the flatness is further improved when using steel substrate.

The thickness of the coating will preferably be between 0.1 and 20  $\mu\text{m}$ . On one hand, below 0.1  $\mu\text{m}$ , there would be a risk that the corrosion protection of the substrate would be insufficient. On the other hand, it is unnecessary to go beyond 20  $\mu\text{m}$  in order to have the level of corrosion resistance which is required, in particular, in the automotive or construction field. In general, the thickness may be limited to 10  $\mu\text{m}$  for automotive applications.

Finally, the invention relates to a Vacuum deposition facility for the method according to the present invention for continuously depositing, on a running substrate, coatings formed from at least one metal, the facility comprising a vacuum chamber through which the substrate can run along a given path, wherein the vacuum chamber further comprises:

- the at least two vapor ejectors facing each other being located on both sides of the substrate and being positioned respectively with an angle  $\alpha$  and  $\alpha'$ , being between the vapor ejector and the axis A perpendicular to the running direction of the substrate, the axis being in the plane of the substrate,  $\alpha$  and  $\alpha'$  both satisfying the following equation:

$$(D1 + D2) + Le \sin \alpha + We \cos \alpha = Ws, \text{ and}$$

$$(D1 + D2) + Le \sin \alpha' + We \cos \alpha' = Ws$$

$\alpha$  and  $\alpha'$  in absolute value being above  $0^\circ$  and

D1 and D2 being the lower distance between ejectors and each substrate edge along the axis (A),  $W_s$  being the substrate width, D1 and D2 being above 0mm and

said vapor ejectors having an elongated shape and comprising a slot and being defined by a slot length  $L_e$  and a slot width  $W_e$ , said vapor ejectors having the same rotation axis.

In a preferred embodiment, the at least two vapor ejectors are mounted to be able to rotate around a feeding pipe linked to a vapor source so that  $\alpha$  and  $\alpha'$  are adjusted.

### Examples

Tests have been performed on the vacuum deposition facility to assess the efficiency of the method comprising two vapor ejectors ejecting zinc vapor.

Zinc vapor was deposited on both sides of the steel substrate having a width  $W_s$  of 1300mm in the vacuum chamber comprising two vapor ejectors having  $L_e = 24$  mm and  $W_e = 1750$  mm. For the Trials, D1 and D2 were identical and were fixed to be between -10mm to +20mm. -10mm means that the edges of the vapor extend 10mm beyond the edges of the substrate.  $\alpha$  and  $\alpha'$  were calculated for each Trial with the equation according to the present invention. The vacuum pressure was of  $10^{-1}$  mBar. The desired thickness of zinc coating was 8  $\mu\text{m}$  corresponding to 100%. The thickness of the metal was measured by X-ray fluorescence spectrometry. The results are in the following Table 1:

Trials	D1 = D2 (mm)	D1 and D2 > 0 mm	$\alpha$ and $\alpha'$ (degrees)	$\alpha$ Satisfies the equation	maximum thickness on both sides (%)		
					45mm from the substrate edges	30mm from the substrate edges	15 mm from the substrate edges
1	-6	No	42.2	Yes	130	126	123
2*	+27	Yes	45.4	Yes	110	103	106
3*	+40	Yes	46.6	Yes	102	98	102

\*: according to the present invention

The coating of Trials 2 and 3 was homogeneously deposited compared to Trial 1.

**CLAIMS**

5

1) A Method for continuously depositing, on a running substrate (S), coatings formed from at least one metal inside a Vacuum deposition facility (1) comprising a vacuum chamber (2), wherein the method comprises:

10

- A step in which in the said vacuum chamber, a metallic vapor is ejected through at least two vapor ejectors (3, 3'), towards both sides of the running substrate and a layer of at least one metal is formed on each side by condensation of ejected vapors, the at least two vapor ejectors facing each other being located on both sides of the substrate and being positioned respectively with an angle  $\alpha$  and  $\alpha'$ , being between the vapor ejector and the axis (A) perpendicular to the running direction of the substrate, the axis being in the plane of the substrate,  $\alpha$  and  $\alpha'$  both satisfying the following equations:

15

$$(D1 + D2) + Le \sin \alpha + We \cos \alpha = Ws \text{ and}$$

20

$$(D1 + D2) + Le \sin \alpha' + We \cos \alpha' = Ws$$

$\alpha$  and  $\alpha'$   $\alpha$  in absolute value being above  $0^\circ$  and

25

D1 and D2 being the lower distance between ejectors and each substrate edge along the axis (A),  $W_s$  being the substrate width, D1 and D2 being above 0mm, i.e. the ejector edges do not go beyond the substrate edges, and said vapor ejectors having an elongated shape and comprising a slot and being defined by a slot length  $Le$  and a slot width  $We$ , said vapor ejectors having the same rotation axis.

30

2) A method according to claim 1, wherein the distances between the ejector and the substrate edges D1 and D2 are above 1mm.

- 3) A method according to claim 1 or 2, wherein the substrate width  $W_s$  is maximum of 2200mm.
- 4) A method according to anyone of claims 1 to 3, wherein  $W_s$  is minimum of 200mm.
- 5) A method according to anyone of claims 1 to 4, wherein  $\alpha'$  is such that  $\alpha - \alpha' < 10^\circ$  in absolute terms.
- 6) A method according to anyone of claims 1 to 5, wherein  $\alpha$  is between 5 and 80° in absolute terms.
- 7) A method according to claim 6, wherein  $\alpha$  is between 20 and 60° in absolute terms.
- 8) A method according to claim 7, wherein  $\alpha$  is between 35 and 55° in absolute terms.
- 9) A method according to anyone of claims 1 to 8, wherein the length of the ejector split  $L_e$  is between 5 and 50 mm.
- 10) A method according to anyone of claims 1 to 9, wherein the ejectors have a rectangular shape or a trapezoidal shape.
- 11) A Method according to anyone of claims 1 to 10, wherein  $D_1$  is identical to  $D_2$ .
- 12) A Method according to anyone of claims 1 to 11, wherein the vacuum chamber further comprises a central casing (6) surrounding the substrate, said central casing comprising a substrate entry (7) and a substrate exit (8) located on two opposite sides of the central casing and at the least two vapor ejector.

13)A Method according to of claim 12, wherein the inner walls of the central casing (6) are suited to be heated at a temperature above the condensation temperature of the metal or metal alloy vapors.

5 14)A metallic substrate obtainable from the method according to anyone of claims 1 to 13 coated with at least one metal on both sides of the substrate having an average thickness, wherein the coating is deposited homogenously such that the maximum thickness of the coating can exceed the average thickness of 15% maximum.

10 15)A metallic substrate according to claim 14, wherein the metal is chosen from: zinc, chromium, nickel, titanium, manganese, magnesium, silicon and aluminum or a mixture thereof.

15 16)A metallic substrate according to anyone of claim 14 or 15, wherein the metallic substrate is a steel substrate.

17)Vacuum deposition facility for the method according to anyone of claims 1 to 13 for continuously depositing, on a running substrate (S), coatings formed from at least one metal, the facility (1) comprising a vacuum chamber (2) through which the substrate (3) can run along a given path, wherein the vacuum chamber further comprises:

- the at least two vapor ejectors facing each other being located on both sides of the substrate and being positioned respectively with an angle  $\alpha$  and  $\alpha'$ , being between the vapor ejector and the axis (A) perpendicular to the running direction of the substrate, the axis being in the plane of the substrate,  $\alpha$  and  $\alpha'$  both satisfying the following equation:

$$(D1 + D2) + Le \sin \alpha + We \cos \alpha = W_s \text{ and}$$

$$(D1 + D2) + Le \sin \alpha' + We \cos \alpha' = W_s$$

$\alpha$  and  $\alpha'$  being above  $0^\circ$  and

D1 and D2 being the lower distance between ejectors and each substrate edge along the axis (A),  $W_s$  being the substrate width,

D1 and D2 being above 0mm, i.e. the ejector edges do not go beyond the substrate edges, and said vapor ejectors having an elongated shape and comprising a slot and being defined by a slot length  $L_e$  and a slot width  $W_e$ , said vapor ejectors having the same rotation axis.

5

18)A Vacuum deposition according to claim 17, wherein the at least two ejectors are mounted to be able to rotate around a feeding pipe linked to a vapor source so that  $\alpha$  and  $\alpha'$  are adjusted.

10



Figure 2

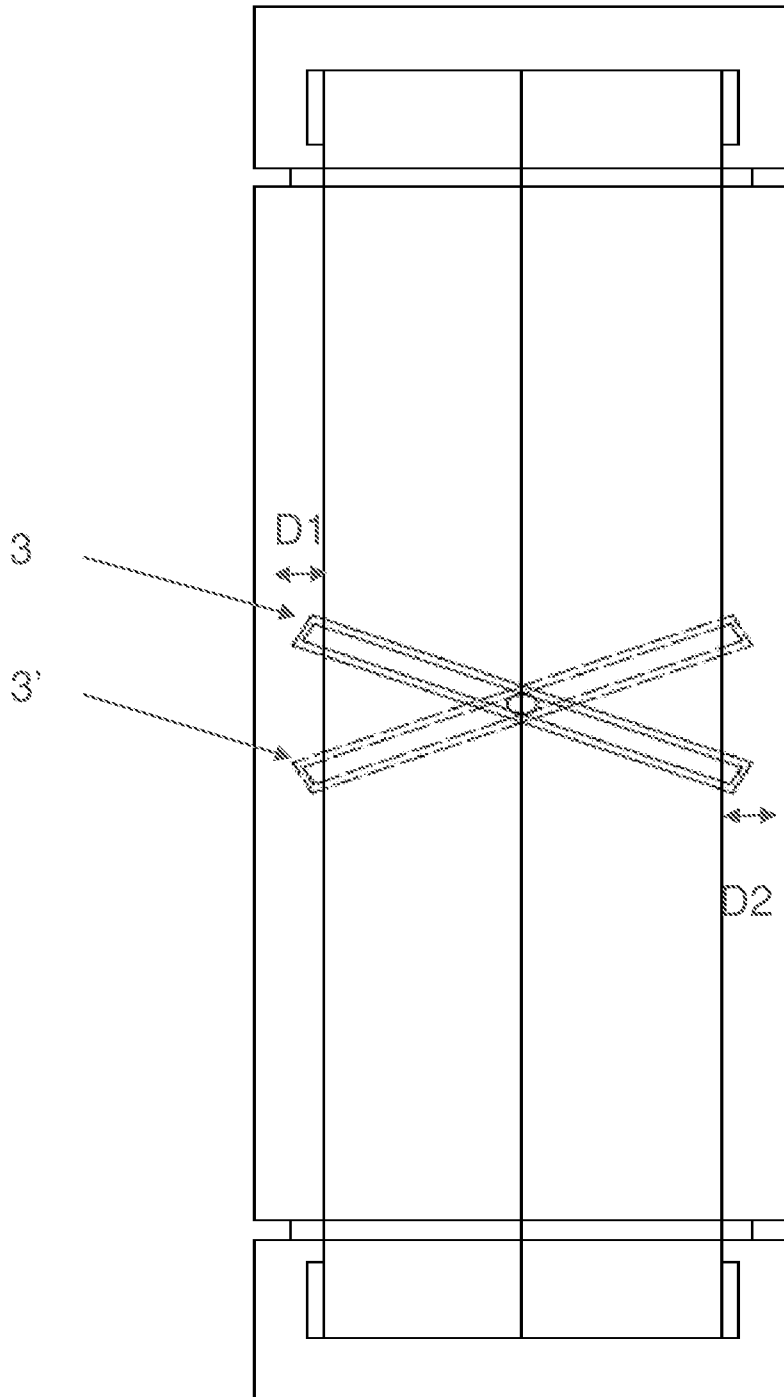


Figure 3

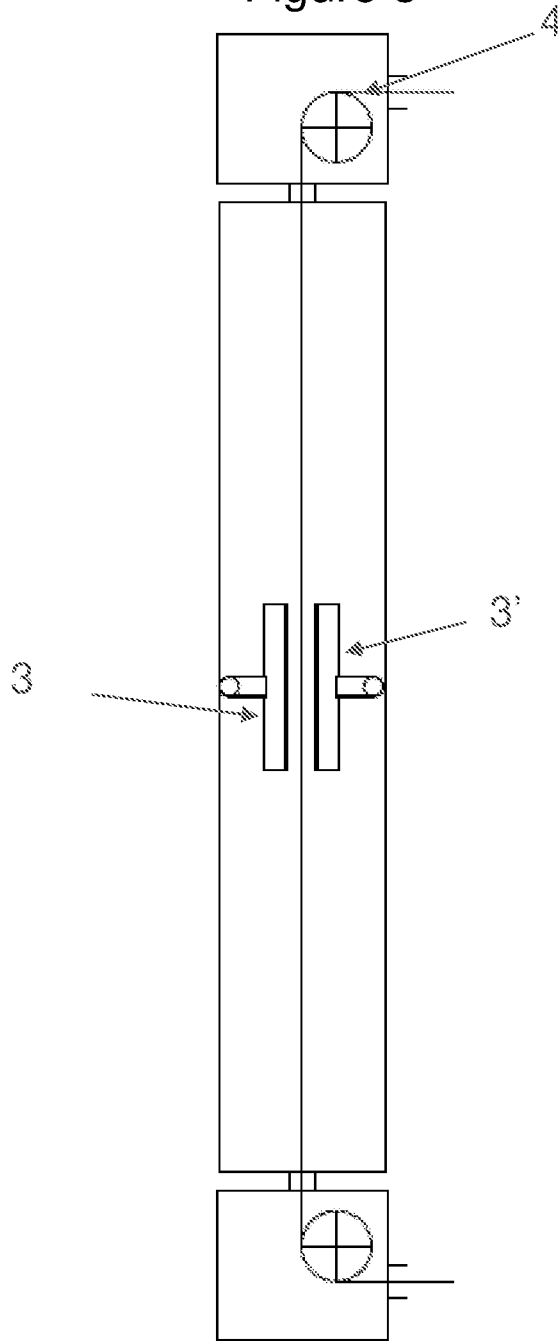


Figure 4

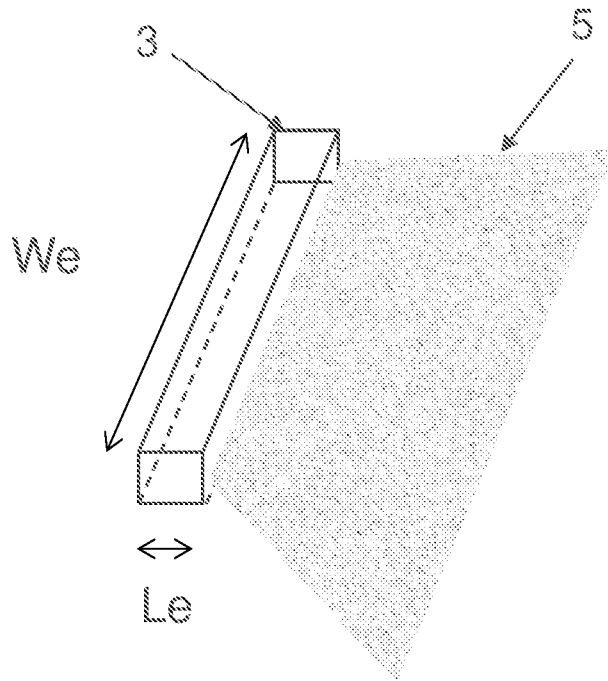


Figure 5

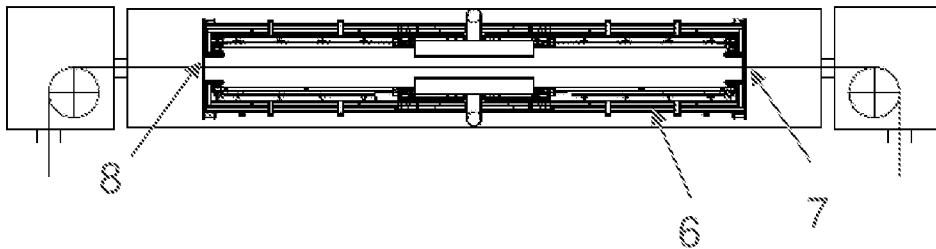


Figure 1

