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(54) **METHOD, APPARATUS AND SYSTEM FOR ELECTRO-DEPOSITION OF A PLURALITY OF THIN LAYERS ON A SUBSTRATE**

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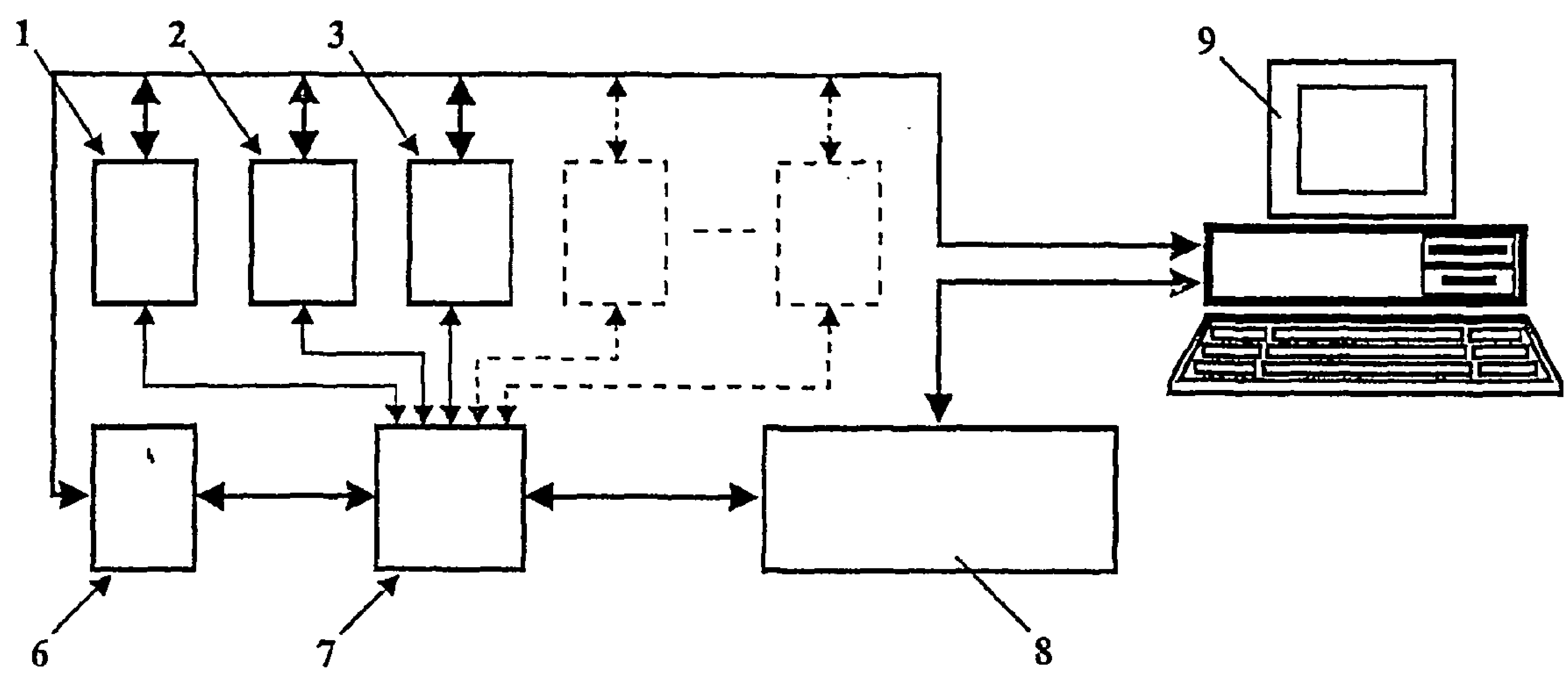
(57) **ABSTRACT**

A method, apparatus and system for fabricating nanometer range, thin multilayers, multilayered nanowires and nonocomposites by multiple bath electro-deposition of a plurality of thin lazers on a substrate, with the purpose to complete the actually developing nanotechnologies, are described. A closed electrochemical system with multiple baths, protected by inert gas, where the chemical solutions are transferred there and back between different chemical tanks and an electrochemical cell, are used to produce thin, in nanometerrange, multiple layer depositions on a substrate which stays on a fix position for all the deposition cycles inside the electrochemical cell. The transfer method protects the system against the cross contamination between different chemical solutions and the risk of contamination of the chemical solutions on their way to and from the chemical tanks. The cleaning method protects the deposited layers aginst undersirable surface reactions.



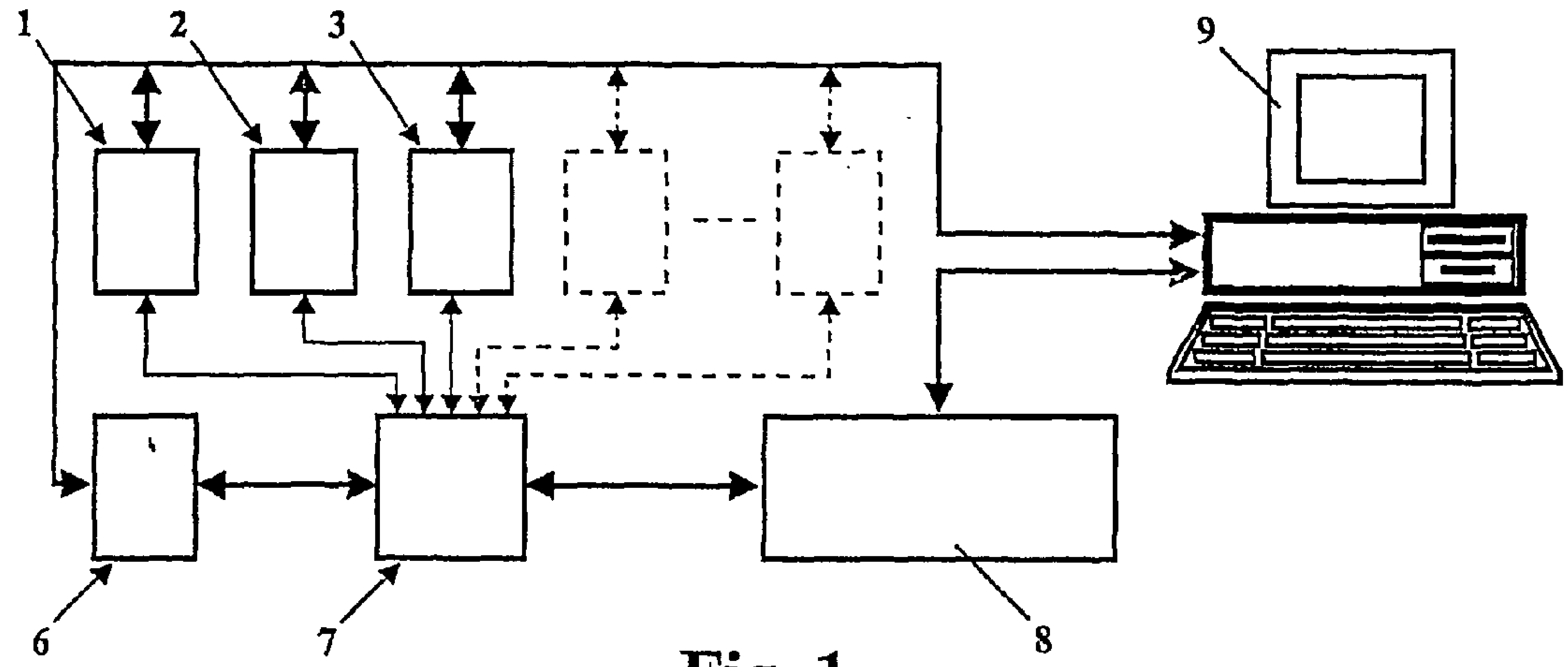

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Fig. 1.

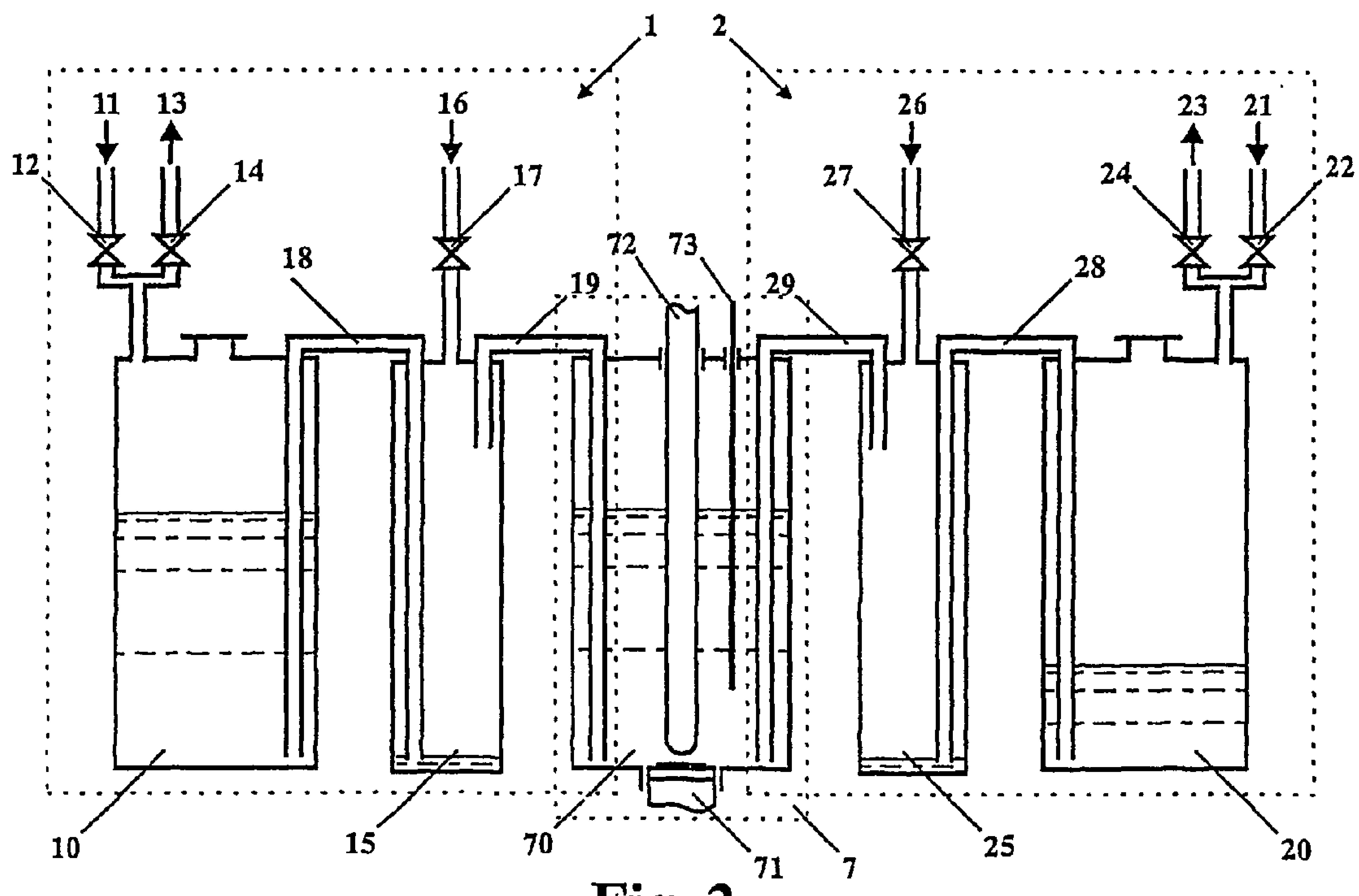
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Fig. 2.

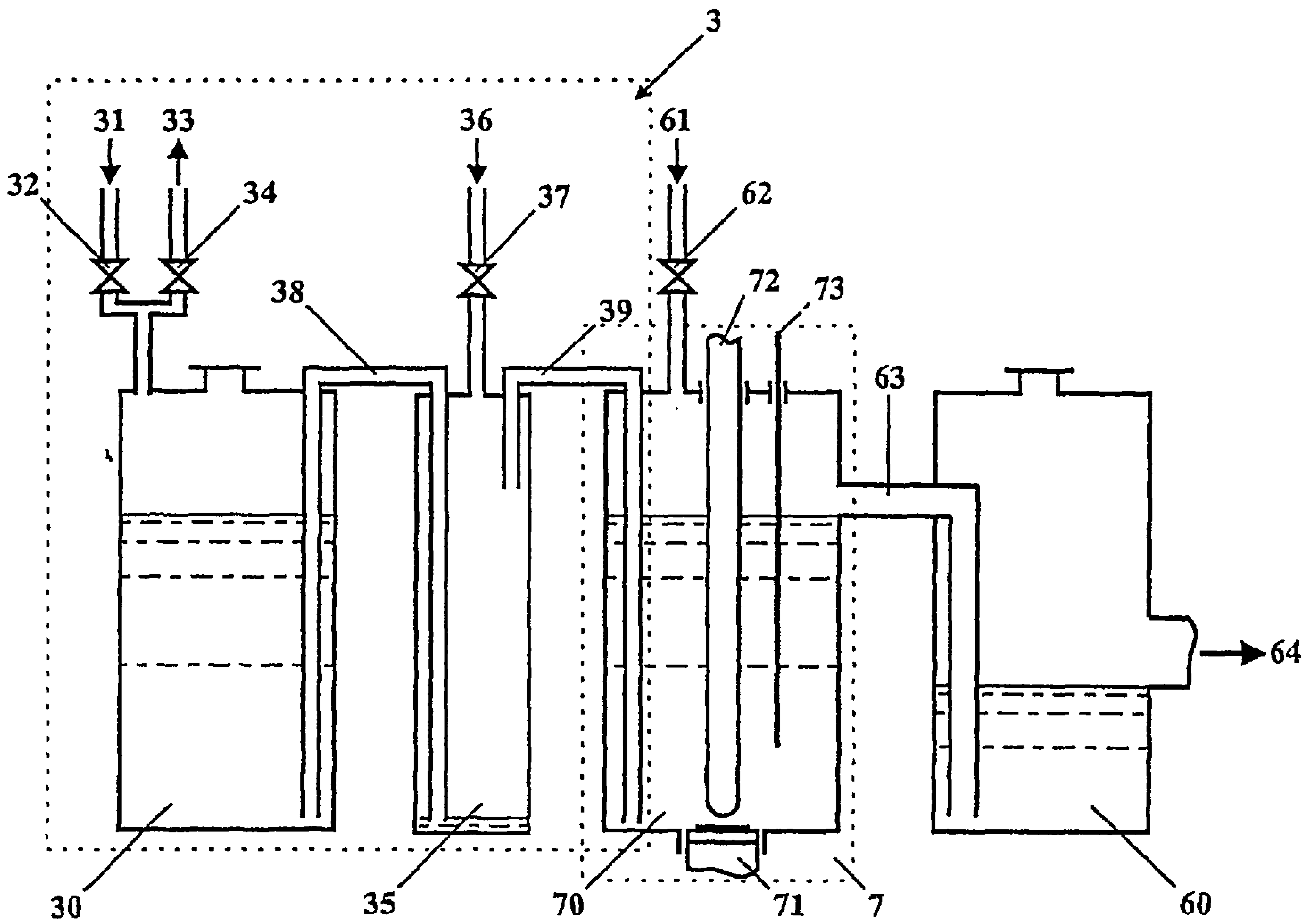

Fig. 3.

METHOD, APPARATUS AND SYSTEM FOR ELECTRO-DEPOSITION OF A PLURALITY OF THIN LAYERS ON A SUBSTRATE

[0001] The invention relates generally to a method, an apparatus and a system for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by electro-deposition of a plurality of thin layers on a substrate, in particular for the fabrication of nanostructures with the purpose to complete the actually developing nanotechnologies.

[0002] As known in the state of the art, an atomistic deposition process represents an essential requirement for producing multilayered structures with the individual layers a few nanometers in thickness. Several techniques, such as molecular beam epitaxy and sputter deposition satisfy quite well this requirement. Some disadvantage of these techniques, such as cost, low deposition rates or diffusion problems between the deposited layers due to relatively high temperature of these processes, limits their applicability. In the last few years an alternative atomistic deposition and cost saving technique was developed for producing thin multilayers, namely the single bath electro-deposition technique. The electrochemical deposition of thin multilayers opens a new way to realise compositionally modulated nanowires in nanoporous materials like polymer and alumina membranes, which was nearly impossible to do previously.

[0003] The single bath electro-deposition technique makes it possible to fabricate periodic stacking of metal and alloy layers. For this technique, the electrochemical bath contains ions of two or more metals separable in two groups, more noble (more electro positive) and less noble metal ions. Deposition in the controlled way of the more noble group (metal or alloy) or of the less noble group alloy are realized usually by varying the deposition potential or current. The concentration of the nobler ions in the bath usually is much lower than the concentration of the less noble ions, to reduce undesirable co deposition effect. This single bath technique permit to realize up to thousands of above mentioned double layers with thickness down to a few nanometers. The main inconvenience of this single bath technique are the impossibility to deposit two different metals for which the electrochemical reduction potentials are too close to each other and also the fact that the less noble deposited metal or alloy always contains a definite amount of nobler metal due to co deposition.

[0004] In contrast, the multiple bath electro-deposition technique does not suffer from these limitations as to the deposited materials. It permits to stack different types of metals, semimetals, alloys, semiconductors, and thin oxide layers. This technique requires the use of a different bath for each layer to be deposited. The inconveniences, like much longer processing time due to cleaning and transfer between baths after each deposit, limit the maximal number of the deposited layers to a few hundreds. This technique is widely used in industrial processes, for deposition of thick (micrometer-range) layers. Unfortunately undesirable surface reactions appear during the cleaning and transfer of the substrate between the different baths, which are unacceptable for nanometer-range thin layer deposition. The attempts to solve these problems were unsuccessful.

[0005] It is an object of the invention to provide a method, apparatus and system for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by multiple bath electro-deposition which is improved with respect to the above-mentioned problems in particular by reducing contamination of the chemical solutions during the deposition process, by reducing the undesirable surface reactions during electro-deposition, and enabling sensible handling of the substrate throughout the electro-deposition process.

[0006] According to the invention, a method for electro-deposition of a plurality of thin layers on a substrate is provided, wherein the electro-deposition is carried out inside a closed electrochemical cell while the substrate is positioned, for all the deposition steps, in the electrochemical cell, and wherein multiple chemical solutions stored in respective tanks are transferred back and forth between the tanks and the electrochemical cell.

[0007] In method of the invention, electro-deposition takes place inside on a closed electrochemical cell to eliminate the undesirable surface reactions, while the substrate stays in the electrochemical cell for all the deposition steps the safeguard the substrate taking into account the mechanical sensibility of the nanostructures. In this case the substrate is not transferred between different baths, it rest in the electrochemical cell, and different solutions are transferred, from different chemical tanks to the electrochemical cell.

[0008] In a preferred embodiment of the method of the invention, the chemical solutions transfer process is realized using pressure differences between the chemical tank and the electrochemical cell. In particular, the pressure differences are realized applying a pressure between 1 to 1000 mbar to the chemical tank and to the electrochemical cell. This substantially reduces the risk of contamination of the chemical solutions which are transferred Multiple times to and from the electromechanical cell during the deposition process as the solutions are not flowing through pumps and are not passing sealing device, valves and the like. Furthermore, the transfer process is simplified and the lifetime of the equipment is increased.

[0009] In a further preferred embodiment of the method of the invention, the chemical solution is transferred between the chemical tank and the electrochemical cell through a separation chamber. The cross contamination between different chemical solutions are solved using these separation chambers.

[0010] In a further preferred embodiment of the method of the invention, the elctrochemical cell and the tanks are part of a closed electrochemical system in order to avoid contamination from the environment.

[0011] In a further preferred embodiment of the method of the invention, the closed electrochemical system is protected by inert gas to further reduce contamination of the system.

[0012] In a further preferred embodiment of the method of the invention, the cleaning process is performed using ultra pure water. The cleaning procedure between two consecutive electro-depositions is made using ultra pure water including no dissolved oxygen or other chemically active gas, in order to eliminate the undesirable surface reactions.

[0013] In a further preferred embodiment of the method of the invention, the cleaning process is performed by letting the ultra pure water flow through the electrochemical cell and by transferring the water after cleaning from the electrochemical cell to the wastewater tank. During this step, the substrate remains also in the cell and the entire cell is cleaned with ultra pure water. The cleaning process is performed allowing the ultra pure water to flow through the electrochemical cell, flowing out to evacuation. At the end, the water is removed from the cell, using one of the same transfer subsystems, dedicated for cleaning process, transferring the water from the cell to the wastewater tank.

[0014] In a further preferred embodiment of the method of the invention, a) the substrate is placed and fixed to the working electrode inside the electrochemical cell; b) the chemical solution is transferred from the a respective tank to the electrochemical cell; c) a computer controlled electro-deposition process is carried out on the substrate; d) the chemical solution is transferred back from the electrochemical cell to the chemical tank for future reuse; e) the electrochemical cell is cleaned by ultra pure water; f) steps b) to e) are repeated until all the desired layers are deposited; g) the substrate is removed from the electrochemical cell.

[0015] In a further preferred embodiment of the method of the invention an automatic mode of the entire electro-deposition cycle is provided under the control of a computer. Due to the computer control in automatic mode of the entire electro-deposition cycle, the method is easy to use. The computer control allows also to precisely control the thickness parameters of the deposited layers.

[0016] A multiple bath electro-deposition apparatus of the invention for fabrication of a plurality of thin layers on a substrate, comprises a closed system of an electrochemical deposition system and a multiple transfer system, the electrochemical deposition system comprising an electrochemical cell adapted to receive the substrate for all the deposition steps.

[0017] The substrate reside for all the deposition steps attached to the working electrode inside the electrochemical cell, which is closed and the deposited layers are protected from the undesirable surface reactions which can occur during the transfer process of different chemical solutions. The volume of the electrochemical cell is minimized given to the small quantity of chemical solution needed to deposit thin layers.

[0018] According to a preferred embodiment the invention provides an apparatus wherein the electrochemical cell includes working-, counter- and reference-electrodes such as to enable an electrochemical process on the working electrode on a the substrate and access means for transferring fluids to and from the electrochemical cell.

[0019] According to a further preferred embodiment the invention provides an apparatus wherein the substrate is attached to the working electrode to be supported on the same place in the electrochemical cell.

[0020] According to a further preferred embodiment the invention provides an apparatus wherein the transfer system includes a chemical solution tank; a separation chamber; means to transfer the solution through the separation chamber between the chemical tank and the electrochemical cell.

[0021] According to a further preferred embodiment the invention provides an apparatus comprising a cleaning system, wherein the cleaning system comprises one of the transfer subsystems where the tank is used as wastewater tank; an evacuation chamber; means to allow the access of pure water to the electrochemical cell; and means to evacuate different fluids from the electrochemical cell.

[0022] According to a further preferred embodiment the invention provides an apparatus wherein means to transfer the solution comprise transfer pipes between the chemical tank and the separation chamber; transfer pipes between the separation chamber and the electrochemical cell, gas-in and vacuum-out pipes and electrovalves on them connected to the chemical tank to control the transfer process.

[0023] According to a further preferred embodiment the invention provides an apparatus wherein the means to transfer the solution comprise a gas-in pipe and an electrovalve on it connected to the separation chamber.

[0024] According to a further preferred embodiment the invention provides an apparatus wherein the evacuation chamber comprises a separation vessel between the electrochemical cell and an evacuation duct, permitting to keep the electro-deposition system under inert gas protection for all the deposition steps.

[0025] The cleaning process is performed allowing the introduction of pure water into the electrochemical cell through a pipe and an electrovalve. The water flowing through the cell is evacuated through pipes and an evacuation chamber. This evacuation chamber represent a separation vessel between the electrochemical cell and an evacuation duct, permitting to keep the electro-deposition system for all the deposition steps under inert gas protection, designed in the manner such that it permits only one direction of flowing through of different kind of fluids, from the electrochemical cell to the evacuation duct. After that the electrovalve is closed, the remaining water on the electrochemical cell is transferred to the wastewater tank by the dedicated transfer subsystem for cleaning.

[0026] According to a further preferred embodiment the invention provides an apparatus wherein it is designed in the manner such that it permits only one direction of flow of different kinds of fluids from the electrochemical cell to the evacuation duct.

[0027] According to a further preferred embodiment the invention provides an apparatus wherein means to allow the access of pure water to the electrochemical cell and means to evacuate different fluids from the electrochemical cell comprise pipes and an electrovalve to control the cleaning process.

[0028] According to a further preferred embodiment the invention provides an apparatus comprises a computer controlling the apparatus to perform an in automatic mode of the entire electro-deposition cycle. Due to the computer control the apparatus is easy to use even if it seems to be complex.

[0029] The invention furthermore provides a modular multiple bath electro-deposition system for fabrication of a plurality of thin layers on a substrate, in particular a variety of multilayered nanostructures, comprising at least one electrochemical subsystem as claimed in claims, at least one transfer subsystem as claimed in claims and at least one cleaning subsystem as claimed in claims, wherein the subsystems are integrated to form a closed electro-deposition system. Preferably, the system is protected by inert gas.

Further perferred is a system comprising a computer controlling the apparatus to perform an in automatic mode of the entire electro-deposition cycle.

[0030] According to the invention a modular designed multiple bath electro-deposition system for fabrication of multiple different thin layers on a substrate, comprise: an electrochemical subsystem, multiple transfer subsystems and a cleaning subsystem, everything controlled by computer. The electrochemical subsystem includes: an electrochemical cell; working, counter and reference electrodes such as to enable an electrochemical process on the working electrode on a substrate; access for different fluids therein to. One of the transfer subsystems (each the same) includes: a chemical solution tank; a separation chamber; means to transfer the solution there and back, through the separation chamber, between the chemical tank and the electrochemical cell. The cleaning subsystem is composed by: one of the transfer subsystems where the tank is used as wastewater tank; an evacuation chamber; means to allow the access of pure water to the electrochemical cell; and means to evacuate different fluids from the electrochemical cell.

[0031] In the preferred embodiments of the invention, the fixed position on the electrochemical cell for the substrate for all the electro-deposition steps allows the possibility of fabrication of multilayered nanostructures and nanowires using the multiple bath technique, solving the mechanical sensibility problems of this structures. Due to ultra pure water use, without any dissolved oxygen or other chemically active gas; the undesirable surface reactions are eliminated during the cleaning process. Small volume for the electrochemical cell allows small quantity of wastewater production and better cleaning due to use for each cleaning step pure water. The inert gas protection in the closed electrochemical system of the substrate also eliminates undesirable surface reactions during transfer process of different chemical solutions.

[0032] The method and apparatus of the present invention provide a large improvement in electro-deposition process of the nanometer-range multilayers. One of the most important improvement is the reduction of the undesirable surface reactions during cleaning and transfer between different baths. Another important improvement is he reduction of cross contamination between baths and the quantity of wastewater. A further important improvement resides in that no delicate manipulation of the substrate during transfer between baths is necessary taking in consideration the sensibility of the nanostructures and nanowires to eventually mechanical shocks. Due to the modularity of the system, to the low quantity of wastewater production and other previously presented advantages are suitable for industrial application.

[0033] The preferred embodiments of the method, the apparatus and the system for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by multiple bath electro-deposition of a plurality of thin layers on a substrate, with the purpose to complete the actually developing nanotechnologies, comprise a closed electrochemical system with multiple baths, protected by inert gas, where the chemical solutions are transferred there and back between different chemical tanks and an electrochemical cell. Multiple layer depositions in nanometer-range, are produced on a substrate which stays on a fix position for all the deposition cycles inside the electrochemical cell. The transfer method protects the system against the cross contamination between different chemical solutions and the risk of contamination of the chemical solutions on their way to and from the chemical tanks. The cleaning method protects the deposited layers against undesirable surface reactions.

[0034] In order that the present invention may be more fully understood, it will now be described by way of example and with reference to the accompanying drawings in which:

[0035] **FIG. 1** shows a block diagram of the modular designed multiple bath electro-deposition system;

[0036] **FIG. 2** shows a schematic diagram of the system in which are showed only two of the transfer subsystems to be easily understandable; **FIG. 3** shows a schematic diagram of the cleaning part of the system.

[0037] **FIG. 1** illustrates different functional blocks of the multiple bath electro-deposition apparatus according to the present invention. The computer **9** using the electrochemical process control equipment **8**, for example a potentiostat and/or galvanostat, controls the electrochemical deposition process. The future role of the computer **9** is to control the different steps of the multilayer electro-deposition. The main parts of the system are the electrochemical subsystem **7** and the transfer subsystems **1**, **2**, **3** etc. The electro-deposition of the thin multilayers on a substrate takes place in the electrochemical subsystem **7**. The number of the transfer subsystems is not limited, such that the system is modular. Their number is a function of the number of different types of desired layers in the multilayer structure. After each deposited layer, the electrochemical subsystem is cleaned and rinsed with ultra pure water using the cleaning part of the system composed by evacuation and cleaning part **6** and one of the transfer subsystems for instance **3**, which has the role to remove the water from the electrochemical cell.

[0038] The operation sequences for one electro-deposition cycle consist on the following steps: the chemical solution is transferred from the desired chemical tank to the electrochemical cell; then, the electro-deposition process takes place controlled by computer; next, the solution from the electrochemical cell is transferred back to the chemical tank for future reuse; next, the cleaning process of the electrochemical cell is carried out. The multilayer-electro-deposition consists of several cycles, as described above, the cycles being controlled in an automatic mode by computer.

[0039] The principle of the transfer operation is illustrated in **FIG. 2** which shows only two of the transfer subsystems **1**, **2** and the electrochemical subsystem **7**. For example the transfer sequence from the chemical tank **10** to the electrochemical cell **70** consist in starting to increase the inert gas pressure in the chemical tank **10**, opening a gas-in electrovalve **12** whereupon a chemical solution starts to flow through the transfer pipe **18** first to the separation chamber **15** than when it is filed, through the next pipe **19** to the electrochemical cell **70**; next, when the level of the liquid is sufficient in the electrochemical cell **70**, the gas-in electrovalve **12** is closed; next, opening the gas-in electro valve **17** of the separation chamber **15** and the vacuum-out electrovalve **14** of the chemical tank **10** the solution from the separation chamber **15** is transferred back to the chemical tank **10** and in parallel the transfer pipe **19** between the separation chamber **15** and the electrochemical cell **70** it becomes empty. During the transfer from the chemical tank **10** to the electrochemical cell **70**, in all the rest of the transfer subsystems the gas-in electrovalves for the separation chambers are opened for instance the electrovalve **27**, **37** respectively **FIGS. 2,3**. The inert gas flowing through the transfer pipe **29**, **39** respectively (**FIGS. 2,3**) on the electrochemical cell **70** keeps the purity of the different chemical solutions preventing the cross contamination between the newly transferred solution on the electrochemical cell **70** and the solutions in different chemical tanks for instance **20**, **30** respectively (**FIGS. 2,3**). The excess inert gas from the electrochemical cell **70** flows out through the evacuation pipe **63**, the evacuation chamber **60** and the evacuation duct **64**. The role of the evacuation chamber **60** is to keep the electrochemical system closed and under protective inert gas. When this transfer operation is finished, one step of the electro-deposition process can take place.

[**0040**] A step of the electro-deposition process consists in deposition of one layer on the substrate using normal electro-deposition technique or multiple layers using the single bath electro-deposition technique. By carrying out both techniques in the same electrochemical system, the possibilities to realize different kinds of multi layered structures become virtually unlimited.

[**0041**] When the electro-deposition step is finished, the chemical solution is transferred from the electrochemical cell **70** back to the chemical tank **10** for future reuse. The transfer sequence consist in keeping an inert gas overpressure in the electrochemical cell **70** in all the. transfer subsystems, except this one, opening the gas-in electrovalves for the separation chambers, for instance the electro valve **27**, **37** respectively in **FIGS. 2,3**; then opening the vacuum-out electrovalve **14** whereby the solution starts to flow through the transfer pipe **19** at first to the separation chamber **15** and then through the next pipe **18** to the chemical tank **10**; next, when the electrochemical cell **70** is empty, closing the vacuum-out electrovalve **14** and also closing, in all the rest of the transfer subsystems, the gas-in electrovalves for the separation chambers, for instance closing the electro valve **27**, **37** respectively in **FIGS. 2,3**.

[**0042**] The transfer procedures on all other transfer subsystems between different chemical tanks and the electrochemical cell **70** follow the same above described sequences. When the transfer is finished and the electrochemical cell is empty, the next step is the cleaning procedure. This step is illustrated in **FIG. 3** and starts with opening the electrovalve **62** whereby the ultra pure water starts to flow through pipe **61** filing the electrochemical cell **70** and; in parallel, the previously used water from the waste water tank **30** is transferred also to the electrochemical cell **70** using the previously described transfer sequences. The excess water from the electrochemical cell **70** flows out through the evacuation pipe **63**, the evacuation chamber **60** and the evacuation duct **64**; where the duration of flowing the pure water is in agreement to the volume of the electrochemical cell **70**. After a certain time, the electrovalve **62** is closed and the water from the electrochemical cell **70** is transferred to the wastewater tank **30** using the previously described transfer sequences. In order to increase the efficiency of the cleaning process some of above described cleaning sequences can be repeated. When the cleaning process is finished, the next electro-deposition cycle can follow.

1. A method for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by electro-deposition of a plurality of thin layers on a substrate, wherein the electro-deposition is carried out inside a closed electrochemical cell while the substrate is positioned, for all the deposition steps, in the electrochemical cell, and wherein multiple chemical solutions stored in respective tanks are transferred back and forth between the tanks and the electrochemical cell.

2. The method of claim 1 wherein the chemical solutions transfer process is realized using pressure differences between the chemical tank and the electrochemical cell.

3. The method according to claim 2 wherein the pressure differences are realized applying a pressure between 1 to 1000 mbar to the chemical tank and to the electrochemical cell.

4. The method of claim 2 wherein the chemical solution is transferred between the chemical tank and the electrochemical cell through a separation chamber.

5. The method of claim 1, wherein the electrochemical cell and the tanks are part of a closed electrochemical system.

6. The method of claim 1, wherein the closed electrochemical system is protected by inert gas.

7. The method of claim 1, wherein the cleaning process is performed using ultra pure water.

8. The method of claim 1, wherein the cleaning process is performed by letting the ultra pure water flow through the electrochemical cell and by transferring the water after cleaning from the electrochemical cell to the wastewater tank.

9. The method of claim 1, wherein

a) the substrate is placed and fixed to the working electrode inside the electrochemical cell;

b) the chemical solution is transferred from the a respective tank to the electrochemical cell;

c) a computer controlled electro-deposition process is carried out on the substrate;

d) the chemical solution is transferred back from the electrochemical cell to the chemical tank for future reuse;

e) the electrochemical cell is cleaned by ultra pure water;

f) steps b) to e) are repeated until all the desired layers are deposited;

g) the substrate is removed from the electrochemical cell.

10. The method of claim 1, wherein an in automatic mode of the entire electro-deposition cycle is provided under the control of a computer.

11. A apparatus for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by multiple bath electro-deposition of a plurality of thin layers on a substrate, comprising a closed system of an electrochemical deposition system and a multiple transfer system, the electrochemical deposition system comprising an electrochemical cell adapted to receive the substrate for all the deposition steps.

12. The apparatus of claim 11, wherein the electrochemical cell includes working-, counter-and reference-electrodes such as to enable an electrochemical process on the working electrode on a the substrate and access means for transferring fluids to and from the electrochemical cell.

13. The apparatus of claim 11, wherein the substrate is attached to the working electrode to be supported on the same place in the electrochemical cell.

14. The apparatus of claim 11, wherein the transfer system includes a chemical solution tank; a separation chamber; means to transfer the solution through the separation chamber between the chemical tank and the electrochemical cell.

15. The apparatus of claim 11, comprising a cleaning system, wherein the cleaning system comprises one of the transfer subsystems where the tank is used as wastewater tank; an evacuation chamber; means to allow the access of pure water to the electrochemical cell; and means to evacuate different fluids from the electrochemical cell.

16. The apparatus of claim 11 wherein means to transfer the solution comprise transfer pipes between the chemical tank and the separation chamber; transfer pipes between the separation chamber and the electrochemical cell, gas-in and vacuum-out pipes and electrovalves on them connected to the chemical tank to control the transfer process.

17. The apparatus of claim 11, wherein the means to transfer the solution comprise a gas- in pipe and an electrovalve on it connected to the separation chamber.

18. The apparatus of claim 11, wherein the evacuation chamber comprises a separation vessel between the electrochemical cell and an evacuation duct, permitting to keep the electro- deposition system under inert gas protection for all the deposition steps.

19. The apparatus of claim 11, wherein it is designed in the manner such that it permits only one direction of flow of different kinds of fluids from the electrochemical cell to the evacuation duct.

20. The apparatus of claim 11, wherein means to allow the access of pure water to the electrochemical cell and means to evacuate different fluids from the electrochemical cell comprise pipes and an electrovalve to control the cleaning process.

21. The apparatus of claim 11, comprising a computer controlling the apparatus to perform an in automatic mode of the entire electro-deposition cycle.

22. The apparatus of claim 11, comprising a computer controlling the apparatus to perform an in automatic mode of the entire electro-deposition cycle.

23. A modular multiple bath electro-deposition system for fabricating nanometer range, thin multilayers, multilayered nanowires and nanocomposites by electro-deposition of a plurality of thin layers on a substrate, in particular a variety of multilayered nanostructures, comprising at least one electrochemical subsystem, at least one transfer subsystem and at least one cleaning subsystem as claimed in claim 1, wherein the subsystems are integrated to form a closed electro-deposition system.

24. The system of claim 23, wherein the system is protected by inert gas.

25. The system of claim 23, comprising a computer controlling the apparatus to perform an in automatic mode of the entire electro-deposition cycle.

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