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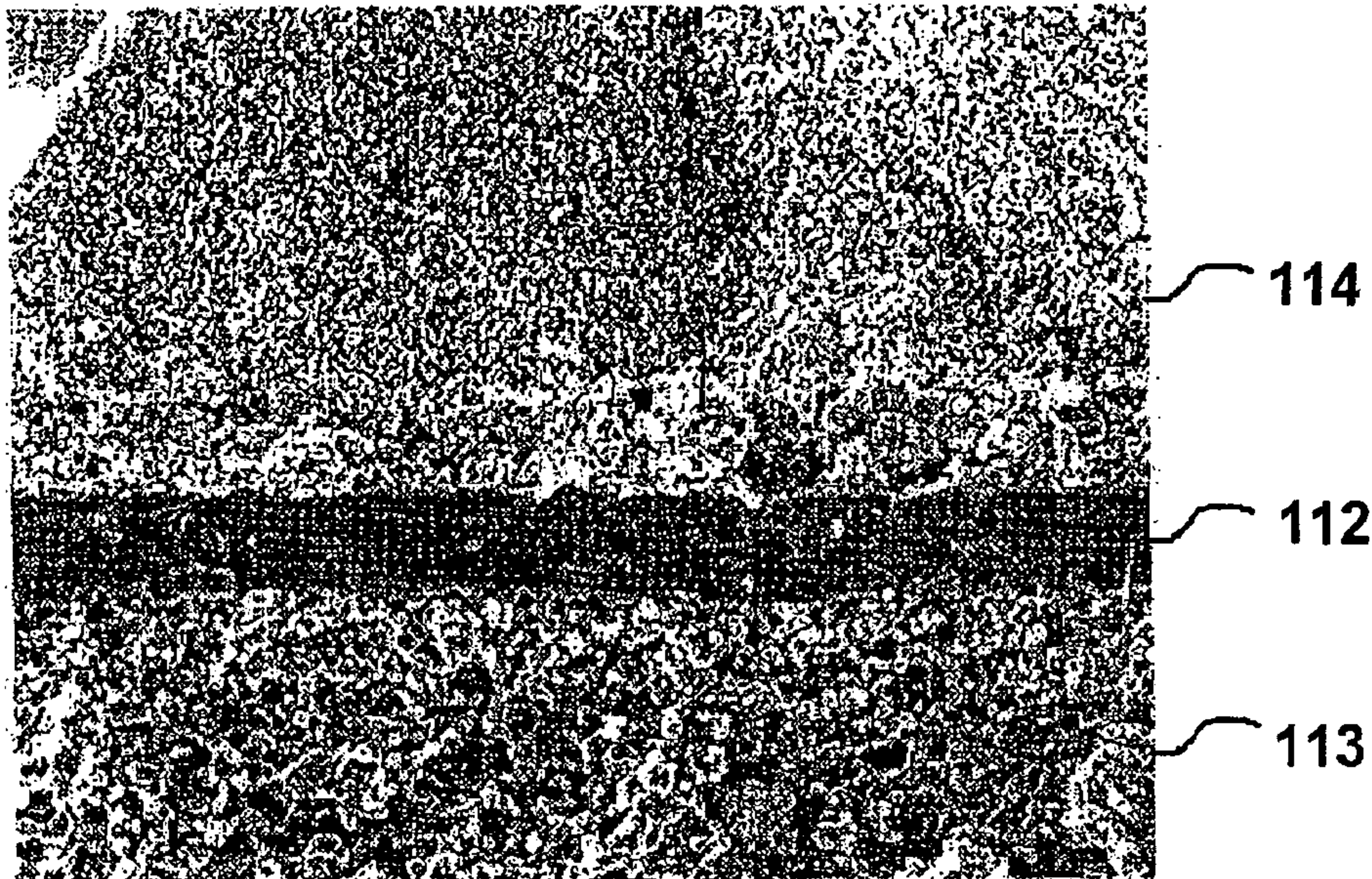
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(54) **Titre : CATHODE A HAUTE PERFORMANCE A PLAGE DE TEMPERATURE DE FONCTIONNEMENT CONTROLEE**
(54) **Title: HIGH PERFORMANCE CATHODE WITH CONTROLLED OPERATING TEMPERATURE RANGE**



(57) **Abrégé/Abstract:**

In a solid oxide fuel cell having an anode, a cathode, and a dense electrolyte disposed between the anode and the cathode, the cathode having a ceramic-ionic conducting phase of a plurality of ionic conducting particles and a metallic phase of a plurality of metallic particles. The metallic phase includes a metal alloy having an oxide-to-metal transition temperature in the range of about 600EC to about 800EC. With this cathode, solid oxide fuel cell operating temperatures as low as about 600EC may be possible.

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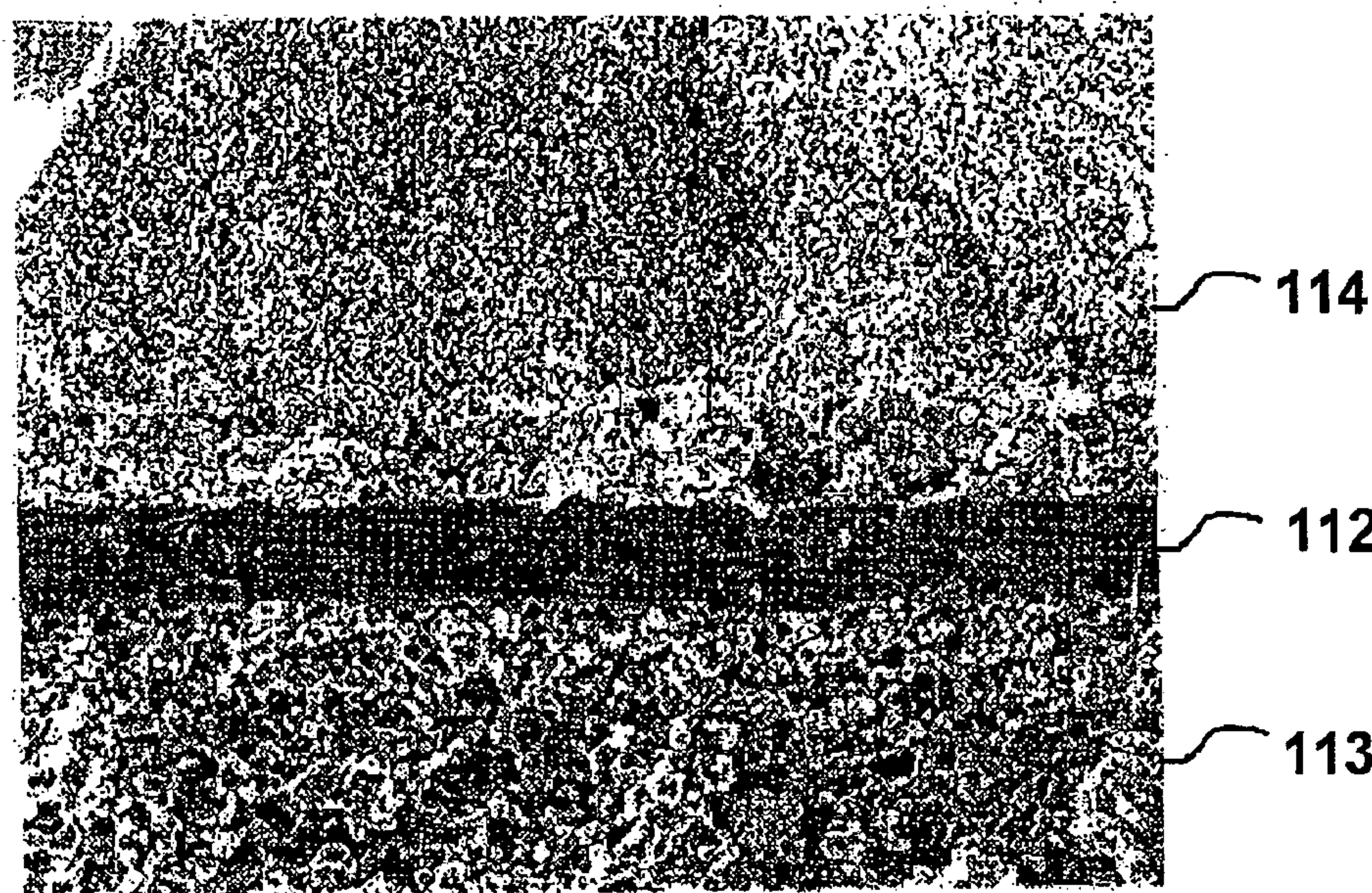
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(54) Title: HIGH PERFORMANCE CATHODE WITH CONTROLLED OPERATING TEMPERATURE RANGE



(57) Abstract: In a solid oxide fuel cell having an anode, a cathode, and a dense electrolyte disposed between the anode and the cathode, the cathode having a ceramic-ionic conducting phase of a plurality of ionic conducting particles and a metallic phase of a plurality of metallic particles. The metallic phase includes a metal alloy having an oxide-to-metal transition temperature in the range of about 600EC to about 800EC. With this cathode, solid oxide fuel cell operating temperatures as low as about 600EC may be possible.

WO 2008/040660 A1

WO 2008/040660 A1



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HIGH PERFORMANCE CATHODE WITH CONTROLLED OPERATING TEMPERATURE RANGE

BACKGROUND OF THE INVENTION

Field of the Invention

5 **[0001]** This invention relates to composite electrodes for use with solid-state ionic devices. More particularly, this invention relates to composite electrodes for use with solid oxide fuel cells. More particularly yet, this invention relates to composite cathode electrodes for use with solid oxide fuel cells.

Description of Related Art

10 **[0002]** Solid state ionic devices typically consist of a fully dense electrolyte sandwiched between thin electrode layers. It is well known that the principal losses in most solid state ionic devices occur in the electrodes or the electrode/electrolyte interfaces. Thus, minimization of these losses is critical to efficient operation of these devices.

15 **[0003]** A solid oxide fuel cell is a solid electrochemical cell comprising a solid gas-impervious electrolyte sandwiched between a porous anode and porous cathode. Oxygen is transported through the cathode to the cathode/electrolyte interface where it is reduced to oxygen ions, which migrate through the electrolyte to the anode. At the anode, the ionic oxygen reacts with fuels such as hydrogen or
20 methane and releases electrons. The electrons travel back to the cathode through an external circuit to generate electric power.

[0004] Conventional solid oxide fuel cells, although operable at temperatures in the range of about 600°C to about 1000°C, generally exhibit high performance at operating temperatures only in the range of about 800°C to about
25 1000°C. However, operation at such high temperatures causes physical or chemical degradation of the fuel cell construction materials. Thus, reducing the operating temperature of a solid oxide fuel cell to reduce such physical or chemical degradation and still maintain high performance levels is highly desirable. However, at reduced operating temperatures, e.g. 700°C, electrode

reaction rates of conventional solid oxide fuel cells decrease significantly, substantially reducing cell performance.

[0005] It is well known to provide activated components on and/or within the fuel cell electrodes to support the electrochemical process. On the anode side, nickel is commonly used as a catalyst for oxidation of the fuel. On the cathode side, ceramic cathode materials, such as perovskites, typically employed in solid oxide fuel cells have a high activation energy for oxygen reduction. The activation energy for the oxygen reduction reaction may be reduced by adding noble metals such as Au, Ag, Pt, Pd, Ir, Ru, and other metals or alloys of the Pt group.

[0006] Efforts to increase electrode reactivity at lower temperatures have focused on optimizing the electrode microstructure and by introducing catalytic materials into the electrode structure. One such effort has resulted in the development of the electrode described and claimed in U.S. Patent 6,420,064 B1 to Ghosh et al. The Ghosh et al. patent teaches an electrode for a solid oxide fuel cell, which electrode comprises a porous three-dimensional solid phase comprising an electrocatalytic phase comprising a plurality of electrocatalytic particles and an ionic conducting phase comprising a plurality of ionic conducting particles, wherein the phases are interspersed, and the mean or median size of the electrocatalytic particles is substantially equal to or larger than the mean or median size of the ionic conducting particles. In accordance with one embodiment, the electrode is a cathode comprising palladium (Pd) and yttria-stabilized zirconia (YSZ). This cathode is said to significantly improve the cell performance in the operating temperature range of about 725°C to about 850°C compared with conventional ceramic cathodes. However, at these temperatures, ferritic stainless steels that are low cost and commercially available have excessive corrosion rates that limit the solid oxide fuel cell device lifetime. Thus, it is desirable to be able to operate the cell at even lower temperatures, where corrosion rates are lower. In particular, operation in the 600°C to 800°C operating temperature range is desirable. Unfortunately, below about 700°C, the cathode has low electrochemical activity.

SUMMARY OF THE INVENTION

[0007] It is, thus, one object of this invention to provide an electrode for a solid oxide fuel cell which is capable of operating with higher electrochemical activity than conventional solid oxide fuel cell electrodes in the temperature range of about 600°C to about 800°C.

[0008] It is another object of this invention to provide a solid oxide fuel cell and solid oxide fuel cell stack able to operate at higher performance levels than conventional solid oxide fuel cells and conventional solid oxide fuel cell stacks in the temperature range of about 600°C to about 800°C.

[0009] It is yet another object of this invention to provide a solid oxide fuel cell and solid oxide fuel cell stack able to operate at higher performance levels than conventional solid oxide fuel cells and conventional solid oxide fuel cell stacks at temperatures less than about 725°C.

[0010] Accordingly, this invention is directed to a solid oxide fuel cell electrode comprising means for enabling long term operation of the electrode and the solid oxide fuel cell at higher performance levels than conventional solid oxide fuel cells in a temperature range of about 600°C to about 800°C, and more particularly in a temperature range of about 600°C to about 725°C.

[0011] This invention also is directed to an electrode having a microstructure which achieves a high density of active electrochemical reaction sites between the electrolyte and the electrode, which incorporates electrocatalytic materials such as noble metals into the electrode in an intimate fashion, and which includes means for enabling long term operation of the electrode with higher electrochemical activity than conventional electrodes in the temperature range of about 600°C to about 800°C, and more particularly in a temperature range of about 600°C to about 725°C.

[0012] Although not wishing to be bound by any particular theory of cathode operation, we believe that, due to a metal(Me)-metal(MeO) oxide transition in the cathode at solid oxide fuel cell operating conditions, metal particles at the cathode effectively grab oxygen from the oxidant (air) and form a chemisorbed bond, Me-O, at the cathode surface. Compared with traditional cathode materials, dissociating molecular oxygen from the air to form physically absorbed atomic oxygen at the surface of the catalyst such as strontium doped lanthanum manganite (LSM) is usually the rate limiting step for the cathodic half cell reaction. We also believe that the oxide-to-metal transition temperature of the metal (and its oxide) is a key factor affecting the electrochemical performance of the cathode. We have discovered that by using alloys, the oxide transition temperature can be lowered or, if desired, raised, by means of which the optimum operating conditions, such as temperature and pressure, of the solid oxide fuel cell can be tailored.

15 [0013] Accordingly, in one aspect of this invention, the invention comprises an electrode forming part of a solid oxide fuel cell comprising a dense electrolyte layer, which electrode comprises a porous three-dimensional solid phase comprising an electrocatalytic phase comprising a plurality of electrocatalytic particles, a ceramic ionic conducting phase comprising a plurality of ionic conducting particles, and operates at a temperature in the range of about 600°C to about 20 800°C.

[0014] In another aspect of the invention, the invention comprises a solid oxide fuel cell having an anode, a cathode, and a dense electrolyte disposed between the anode and the cathode, wherein the cathode comprises a ceramic-ionic conducting phase comprising a plurality of ionic conducting particles and a 25 metallic phase comprising a plurality of metallic particles, and wherein the metallic phase comprises at least one of a metal and a metal alloy having an oxide-to-metal transition temperature in the range of about 600°C to about 800°C.

[0015] The electrode of this invention may be prepared by any method

known to those skilled in the art including conventional ceramic processing followed by firing, chemical vapor deposition (CVD), plasma spraying, etc. The electrode of this invention may be formed by mixing ceramic ionic conducting particles and metallic electrocatalyst particles into a composite electrode which is then applied to a dense electrolyte substrate by screen printing or by similar well known methods. The resulting electrode microstructure is highly porous and includes very long three-phase boundaries, direct ion conducting channels from the catalytic sites to the electrolyte and direct electron conducting channels through the electrode to the catalytic sites. Preferably, the electrocatalyst particles are larger than the ionic conducting particles.

[0016] The metallic electrocatalyst particles in accordance with one embodiment of this invention may comprise substantially pure metal which, when exposed to certain conditions, such as elevated pressures, has an oxide-to-metal transition temperature in the range of about 600°C to about 800°C. In accordance with one particularly preferred embodiment of this invention, the metallic electrocatalyst particles comprise a metal alloy having an oxide-to-metal transition temperature in the range of about 600°C to about 800°C.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] These and other objects and features of this invention will be better understood from the following detailed description taken in conjunction with the drawings wherein:

[0018] Fig. 1 is a diagram showing a thermogravimetric (TG) analysis of palladium powder at various oxygen partial pressures;

[0019] Fig. 2 is a diagram showing the metal oxide-to-metal transition temperature as a function of Pd content of Pd/Ag alloys;

[0020] Fig. 3 is a diagram showing cell voltage versus time at 700°C with 4A current steps at two (2) minute intervals;

[0021] Fig. 4 is a diagram showing cell voltage versus time at 650°C with 4A current steps at two (2) minute intervals;

[0022] Fig. 5 is a diagram showing cell voltage versus time at 600°C with 4A current steps at two (2) minute intervals;

[0023] Fig. 6 is a diagram showing current-voltage curves at temperatures in the range of about 600°C to about 700°C;

5 [0024] Fig. 7 is a diagram showing the steady state voltage and current for a solid oxide fuel cell having a Pd-YSZ cathode operating at a temperature of about 700°C;

[0025] Fig. 8 is a diagram showing current-voltage curves for a cell at temperatures in the range of about 650°C to about 800°C with a Pg/Ag cathode
10 (70:30 %w/w in alloy powder);

[0026] Fig. 9 is a schematic representation of a cross-sectional view of a cathode in accordance with one embodiment of this invention; and

[0027] Fig. 10 is a scanning electron micrograph of a solid oxide fuel cell in accordance with one embodiment of this invention.

15 DETAILED DESCRIPTION OF THE PRESENTLY
PREFERRED EMBODIMENTS

[0028] The invention disclosed herein is a solid oxide fuel cell and solid oxide fuel cell stack operating at a temperature in the range of about 600°C to about 800°C.

20 [0029] The invention disclosed herein is a composite electrode for a solid oxide fuel cell, which electrode, by virtue of its composition, enables operation of the solid oxide fuel cell and solid oxide fuel cell stack at high performance levels at temperatures as low as about 600°C.

[0030] The solid oxide fuel cell, as shown in Fig. 10, comprises a dense
25 electrolyte layer 112 sandwiched between an anode electrode layer 113 and a cathode electrode layer 114. The cathode layer 10, as depicted in Fig. 9, shown bonded to an electrolyte 12, comprises a ceramic-ionic conducting phase comprising a plurality of ionic conducting particles 16 and a metallic phase comprising a plurality of metallic particles 14. The ionic conducting particles

combine to form ionic conducting paths from the electrolyte 12 to the electrochemically active sites 18. The metallic phase forms electronic conducting paths (E) through the electrode 10 to a contact paste (not shown) and cathode electronic conducting strip (not shown). The electrochemically active area
5 coincides with a three phase boundary 18 which extends along the common boundary of the gaseous pore phase, the ceramic phase 16 and the metallic phase 14.

[0031] The metallic phase, in accordance with one embodiment of this invention, comprises at least one of a metal and a metal alloy having an oxide-to-
10 metal transition temperature in a range of about 600°C to about 800°C. In accordance with one embodiment of this invention, the metal or metal alloy comprises a noble metal. In accordance with one preferred embodiment of this invention, the noble metal is selected from the group consisting of silver (Ag), gold (Au), iridium (Ir), osmium (Os), palladium (Pd), ruthenium (Ru), rhodium
15 (Rh) and platinum (Pt). In accordance with one particularly preferred embodiment of this invention, the noble metal is palladium.

[0032] More particularly, the invention disclosed herein is a cathode electrode comprising an alloyed metal and YSZ or other ceramic oxide ionic conductor such as gadolinia-doped ceria as the key components whereby the
20 alloyed metal has a lower oxide-to-metal transition temperature at 0.21 atm pO₂ than the metal components individually, in the range of about 600°C to about 800°C. This includes Pd/Ag alloys where the Pd content is greater than about 50% by weight of the alloy, Pd/Au alloys where the Pd content is greater than about 70% by weight of the alloy, Pd/Pt alloys where the Pd content is greater than
25 about 70% by weight of the alloy, and Pd/Cr alloys or Pd/Nb alloys wherein the Pd content is greater than about 80% by weight of the alloy.

[0033] As previously indicated, the electrode of this invention is formed by mixing ceramic ionic conducting particles and metallic electrocatalyst particles into a composite electrode which is then applied to a dense electrolyte substrate.

In accordance with one embodiment of this invention, the ionic conducting particles comprise ceramic particles which may be yttria stabilized zirconia and the metallic electrocatalyst particles are particles of a metal alloy comprising at least one noble metal. In accordance with one embodiment, the mixture comprises
5 metallic particles ranging in size from about 1-2 microns in diameter and 8 mol% yttria stabilized zirconia (8YSZ) particles ranging in size from about 0.1 to about 0.3 microns in diameter. The preferred microstructure in accordance with one embodiment of this invention comprises about 1-10 vol % Pd and 40-80 vol % YSZ balanced with about 20-50 vol % porosity. It should be noted that all
10 references herein to volume percentages of the electrocatalyst phase and the ionic conducting phase are of the volume of the entire solid phase.

[0034] The cathode layer in accordance with one embodiment of this invention has a thickness of less than about 10 microns, preferably less than about 5 microns. It is screen-printed and co-sintered with a screen-printed electrolyte
15 layer (8YSZ), screen-printed anode functional layer and tape-casted anode substrate. A layer of ionic conducting ceramic materials (perovskites) is printed on the other surface after co-firing. The perovskite particles enter the cathode porous structure during the printing process. The layer is then fired *in-situ* at the operating temperature of the tri-layer, about 600°C to about 850°C. This layer
20 provides electrical contact from the cathode to the bipolar plates of the fuel cell stack (interconnects).

[0035] Due to its materials composition, the majority of the cathode materials (YSZ) is the same as the electrolyte materials. During the co-firing, sufficient sintering between the cathode and electrolyte occurs, creating a strong
25 interface that is less prone to mechanical and thermal mechanical failure during stack assembly and operation.

[0036] Fig. 1 is a diagram showing a thermogravimetric (TG) analysis of palladium powder at various oxygen partial pressures. As shown therein, as the cathode is polarized, the oxygen partial pressure falls in the cathode in accordance

with the Nernst equation. Within the solid oxide fuel cell operating temperature range of about 725°C to about 850°C, approximately every 50 mV polarization produces an order of magnitude lower oxygen partial pressure (effectively) in the cathode, although the exact value varies with temperature. Fig. 1 shows a significant lowering of the oxide-to-metal transition temperature (dashed lines) as pO_2 is lowered over 2 orders of magnitude (approximately equivalent to about 100 mV cathode polarization). Based upon these results, it is reasonable to expect similar performance in a solid oxide fuel cell stack operation and it establishes a lower temperature limit of about 700°C to about 725°C required for metallic palladium.

[0037] We have discovered that even lower operating temperatures, in the range of about 600°C to about 650°C, may be achieved by alloying the palladium or choosing other precious metal alloys for use as an electrocatalyst in the cathode. Fig. 2 shows that by alloying palladium with silver (Ag), the oxide-to-metal transition temperature can be significantly lowered. For an alloy comprising 70 wt % Pd and 30 wt % Ag, the oxide-to-metal transition temperature is reduced from 800°C for Pd metal alone to about 650°C for the alloy.

[0038] A single cell test was conducted using a cathode contact of perovskites and 10% v/v silver addition for the dual purpose of a sinter aid for the perovskite powder (silver has a low melting point of 962°C) and potentially to modify the oxide transition temperature of palladium in the cathode, allowing the cell to operate at lower temperatures. Figs. 3-5 show cell voltage stepped by 4A every two minutes at temperatures of 700, 650 and 600°C, respectively. The large increases in cell voltage with current increase is due to increases in overpotential at the cathode, lowering the pO_2 and causing an oxide-to-metal transition which decreases overvoltage, thereby increasing cell voltage. Over time, oxide reforms, again lowering cell voltage, and the cycle is repeated at the next current load step. Fig. 6 shows current-voltage data for the temperature range of about 600°C to about 700°C. These tests, together with TG measurements demonstrating a lowering of oxide transition temperature for a Pd/Ag alloy cathode, suggest that any method of lowering the oxide-to-metal

transition temperature while maintaining cathode micro structure will result in an improved low temperature cathode, which, in turn, should lead to lower degradation rates due to operation at lower temperatures.

[0039] Fig. 7 shows a cell with a Pd-YSZ cathode operating at 100A
 5 (1.23A/cm²) and 700°C. As can be seen, cell voltage increased from about 591mV to about 605mV in about two days, and one week later was running at 607mV with no degradation. Unexpectedly, the degradation rate at 100A and 700°C is lower than at 40.5A (0.5A/cm²) and 750°C (not shown) despite operating at about 2.5 times the current density. This suggests that oxide transition is a limiting factor in reducing
 10 cell operation of a Pd cathode and that the oxide-to-metal transition temperature of Pd can be reduced to 700°C by imposing > 100mV polarization at the cathode. At such high current density, the cathode polarization exceeds this value and, thus, the cell is stable. At this temperature and 0.5A/cm², the cell degrades rapidly.

[0040] Cell tests have shown reversible degradation below 700°C due to the
 15 cathode. In one test, a cell was cycled between 650°C and 750°C at 0.5A/cm² and showed very high degradation at 650°C, but on returning to 750°C, the cell voltage was within 1 mV of the voltage previously at 750°C, indicating reversible degradation at 650°C. This is due to the palladium-to-palladium oxide reversible transition and data from this test is summarized in Table 1.

TABLE 1

Steady State

	Voltage	Shunt	Time Elapsed	%/1000hr
Start 650°C	0.646	40.50	N/A	N/A
End	0.597	40.50	96	79.0
Start 750°C	0.855	40.50	N/A	N/A
End	0.835	40.50	24	97.5
Start 650°C	0.648	40.50	N/A	N/A
End	0.600	40.50	48	154.3
Start 750°C	0.836	40.50	N/A	N/A
End	0.832	40.50	24	19.9
Start 650°C	0.647	40.50	N/A	N/A
End	0.586	40.50	48	196.4
Start 750°C	0.831	40.50	N/A	N/A
End	0.827	40.50	24	20.1
Start 650°C	0.641	40.50	N/A	N/A
End	0.576	40.50	48	211.3
Start 750°C	0.911	40.50	N/A	N/A
End	0.906	40.50	72	7.6

Note: Time is calculated in 24 hr increments

- 5 **[0041]** Fig. 8 shows V-I curves for a cell with a Pd-Ag cathode (70:30 %w/w in alloy powder). The cell has a voltage of 801mV at 700°C and 0.74A/cm². The cell became unstable when run long term at low temperature (650°C), after which time power curves were repeated and cell voltage dropped to 715mV at the same condition, indicating that the electrode is unstable. But, when
- 10 the alloy is stable and its oxide transition temperature is as low as for Pd-Ag (70:30), i.e. 650°C, low temperature cell performance is possible.

[0042] While in the foregoing detailed description this invention has been described in relation to certain preferred embodiments thereof, and many details have been set forth for purposes of illustration, it will be apparent to those skilled

in the art that certain of the details described herein can be varied. The scope of the claims should not be limited by the preferred embodiments set forth in the examples, but should be given the broadest interpretation consistent with the description as a whole.

CLAIMS:

1. In a solid oxide fuel cell having an anode, a cathode, and a dense electrolyte disposed between said anode and said cathode, the improvement comprising:

said cathode comprising a ceramic-ionic conducting phase comprising a plurality of ionic conducting particles and a metallic phase comprising a plurality of metallic particles, said metallic particles comprising a metal alloy having an oxide-to-metal transition temperature in a range of about 600°C to about 800°C, wherein said metal alloy is selected from the group consisting of:

- a Pd-Ag metal alloy comprising 50% or more by weight of Pd;
- a Pd-Au metal alloy comprising 70% or more by weight of Pd;
- a Pd-Pt metal alloy comprising 70% or more by weight of Pd;
- a Pd-Cr metal alloy comprising 80% or more by weight of Pd; and
- a Pd-Nb metal alloy comprising 80% or more by weight of Pd.

2. A solid oxide fuel cell in accordance with Claim 1, wherein said ionic conducting particles and said metallic particles are interspersed and the mean size of the metallic particles is one of substantially equal to or greater than the mean size of said ionic conducting particles.

3. A solid oxide fuel cell in accordance with Claim 1, wherein said cathode has a thickness less than about 10 μm .

4. A solid oxide fuel cell in accordance with Claim 1, wherein said metal alloy has an oxide-to-metal transition at a $p\text{O}_2$ equal to about 0.21 atm. within an operating temperature range of said solid oxide fuel cell.

5. A cathode forming part of a solid oxide fuel cell comprising a dense electrolyte layer, said cathode comprising a porous three-dimensional solid phase comprising:

an electrocatalytic phase comprising a plurality of metallic electrocatalytic particles comprising a metal alloy having an oxide-to-metal transition temperature in a range of about 600°C to about 800°C; and

a ceramic-ionic conducting phase comprising a plurality of ionic conducting particles; and

wherein said metal alloy is selected from the group consisting of:

a Pd-Ag metal alloy comprising 50% or more by weight of Pd;

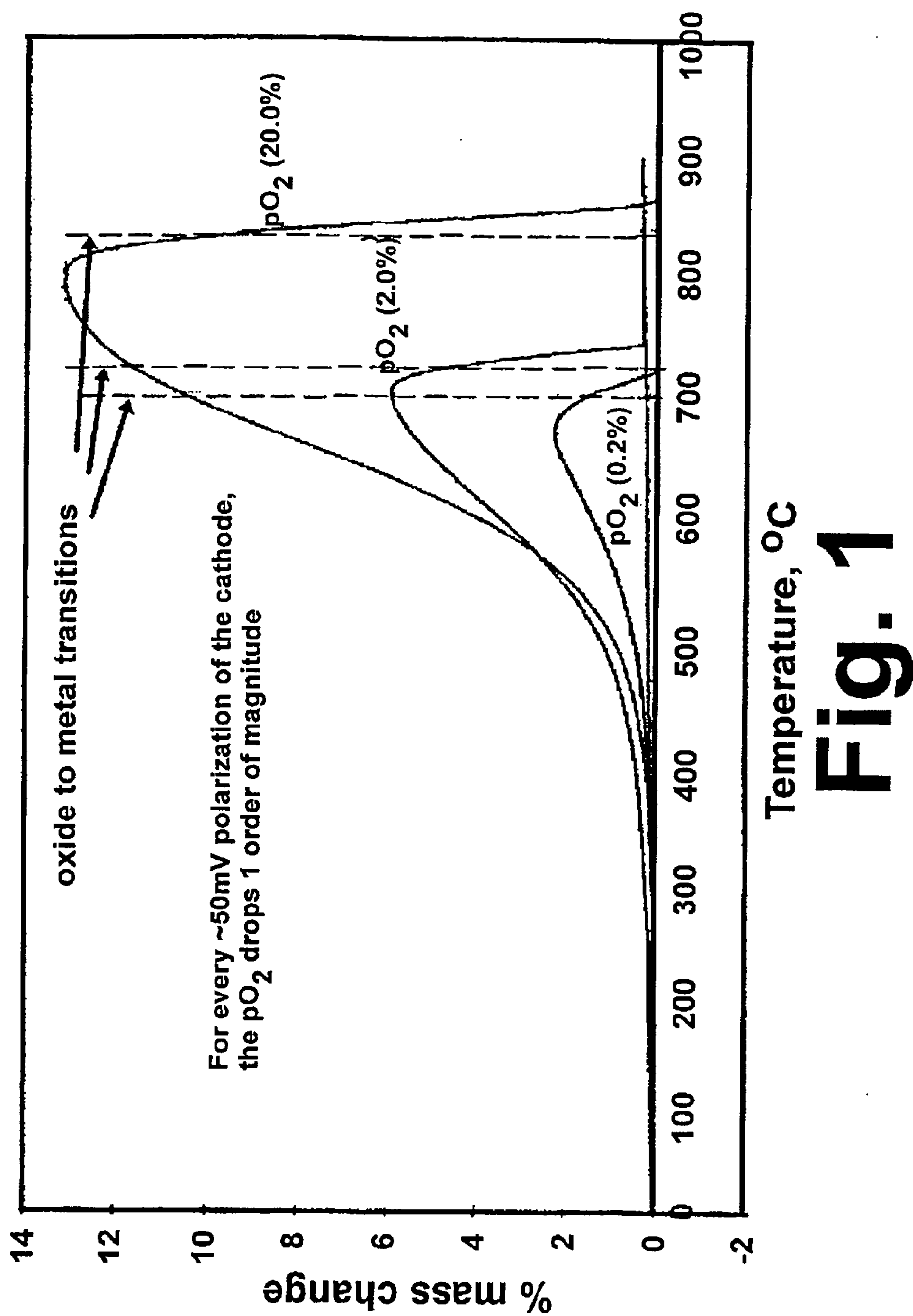
a Pd-Au metal alloy comprising 70% or more by weight of Pd;

a Pd-Pt metal alloy comprising 70% or more by weight of Pd;

a Pd-Cr metal alloy comprising 80% or more by weight of Pd; and

a Pd-Nb metal alloy comprising 80% or more by weight of Pd.

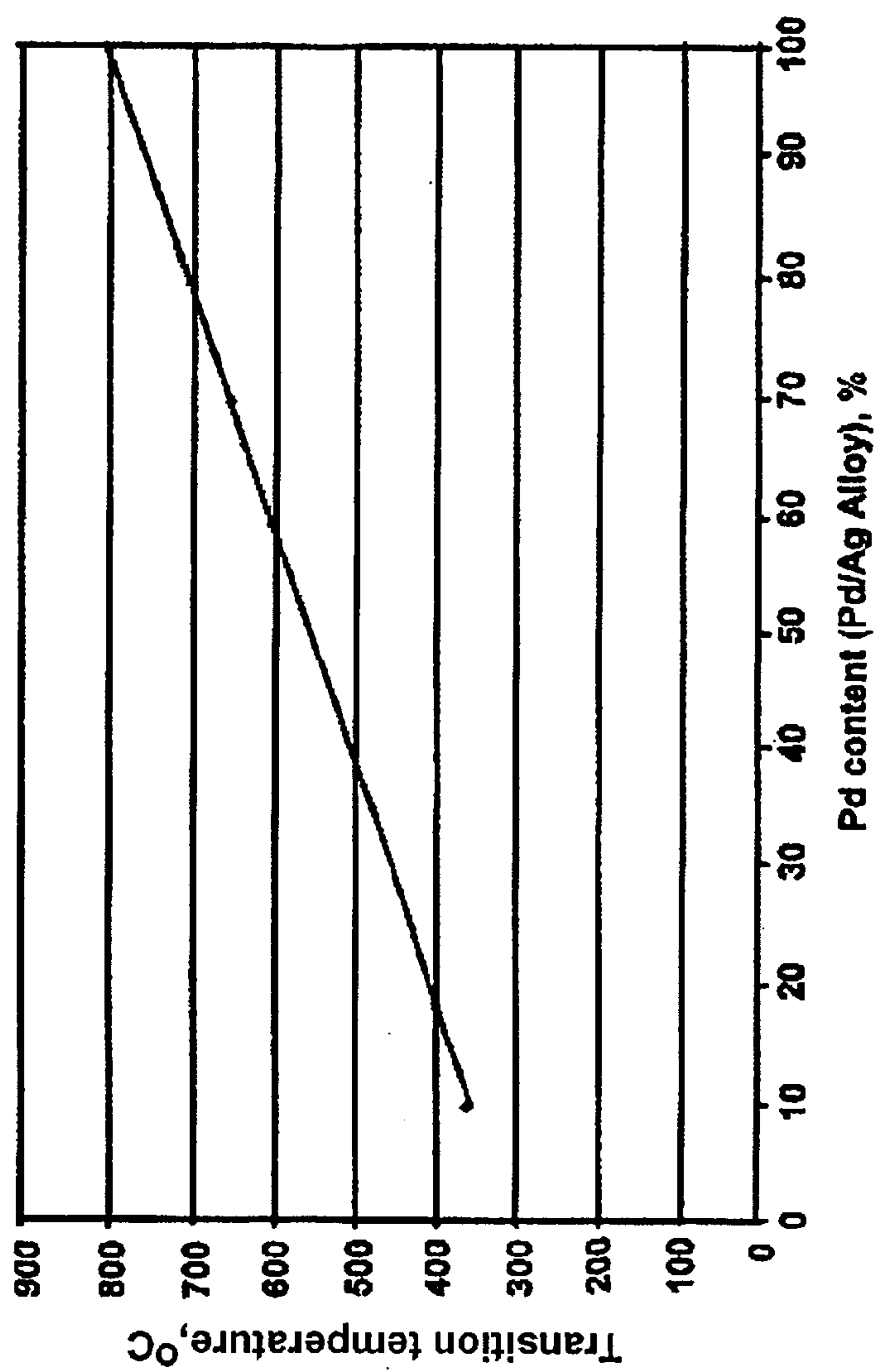
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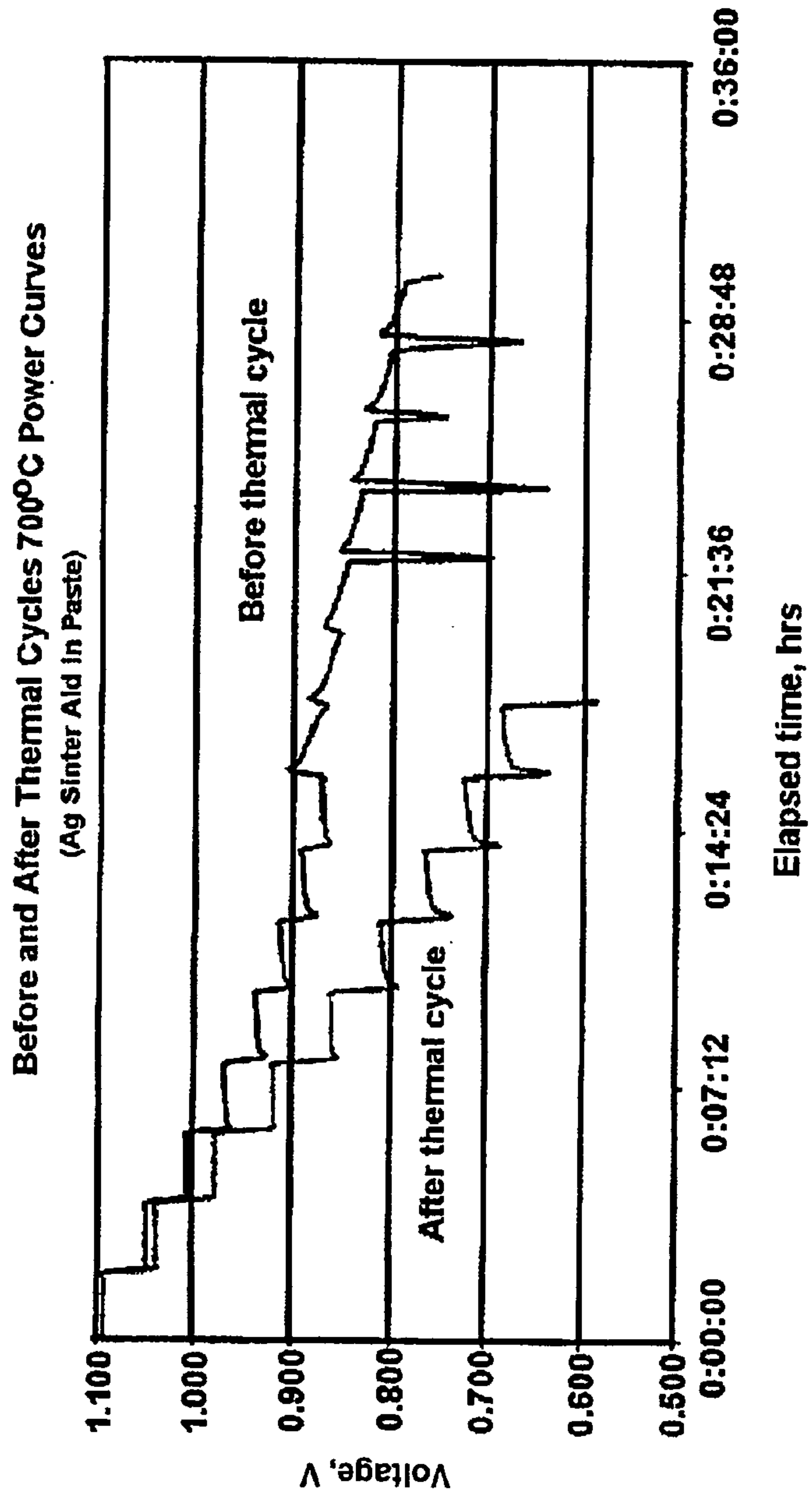
Temperature, °C

Fig. 1

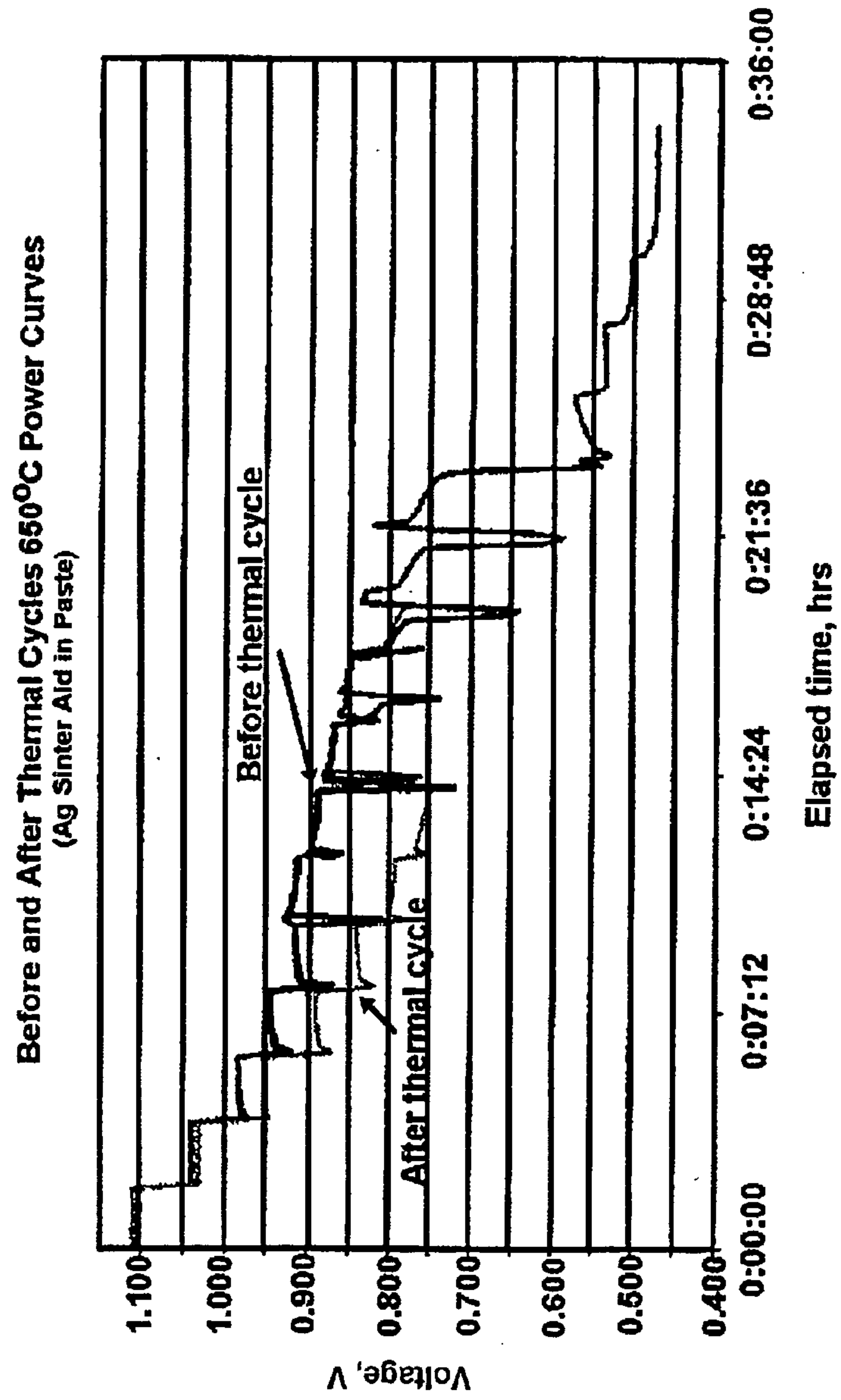
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**Fig. 2**

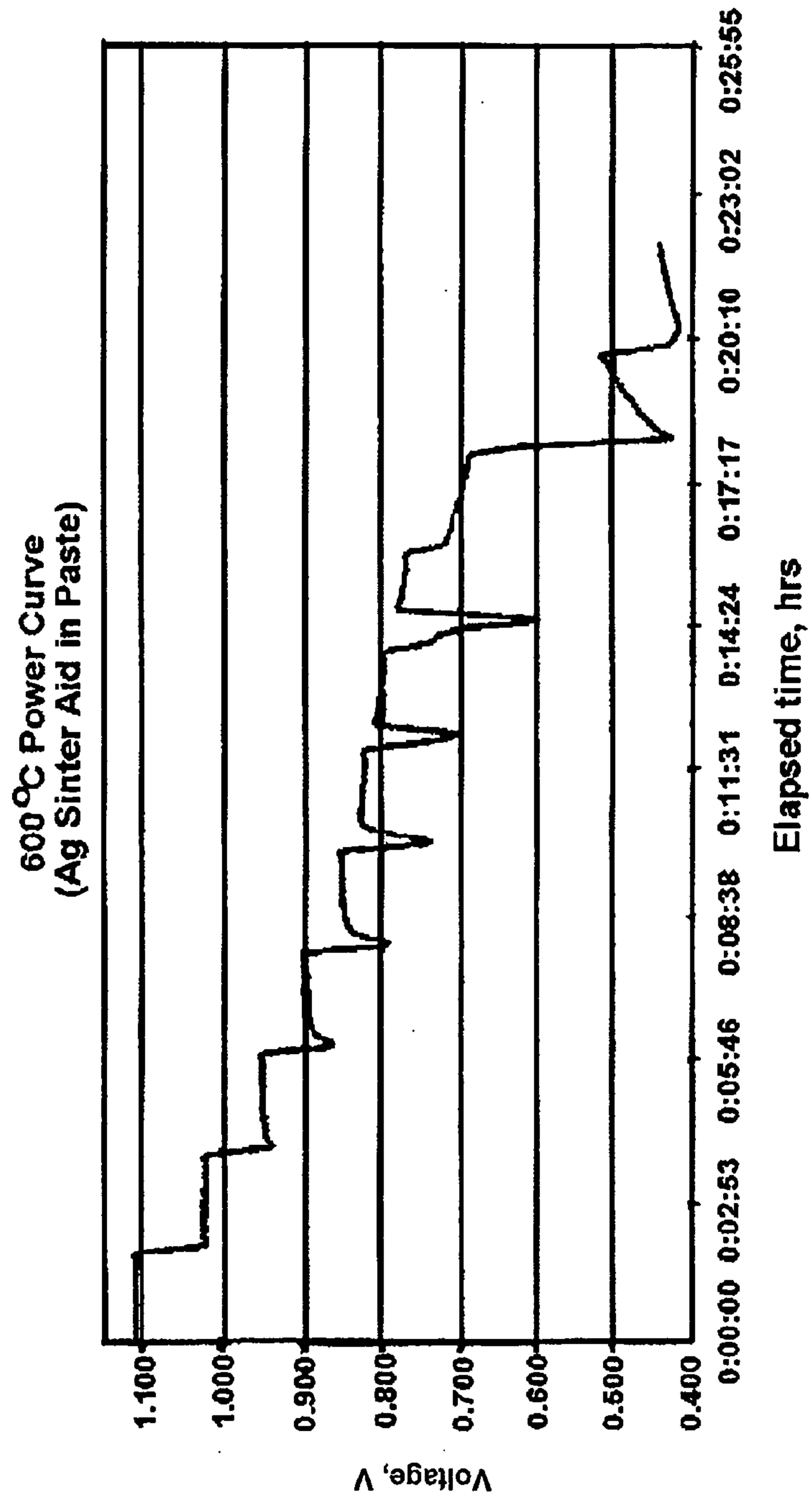
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**Fig. 3**

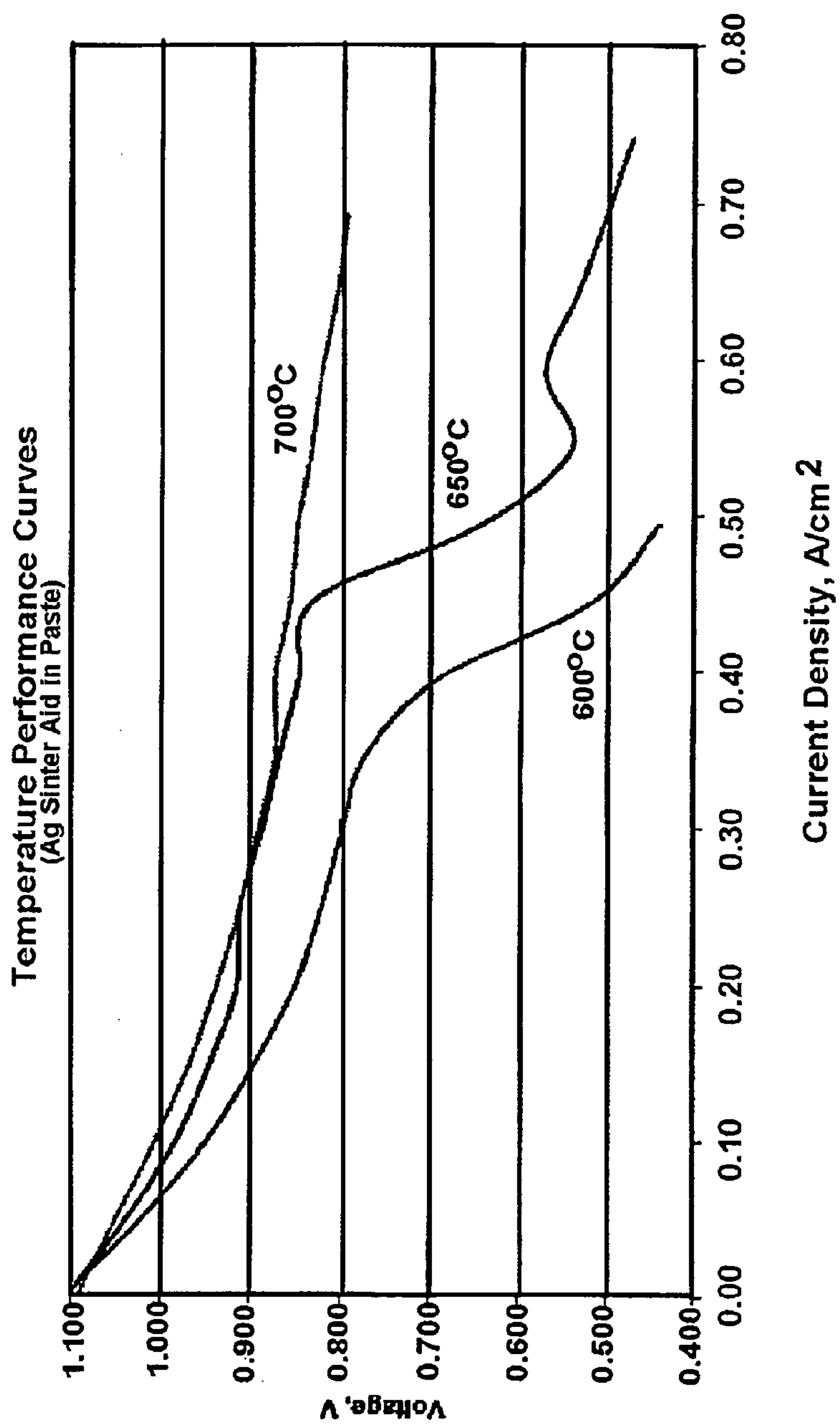
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**Fig. 4**

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**Fig. 5**

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**Fig. 6**

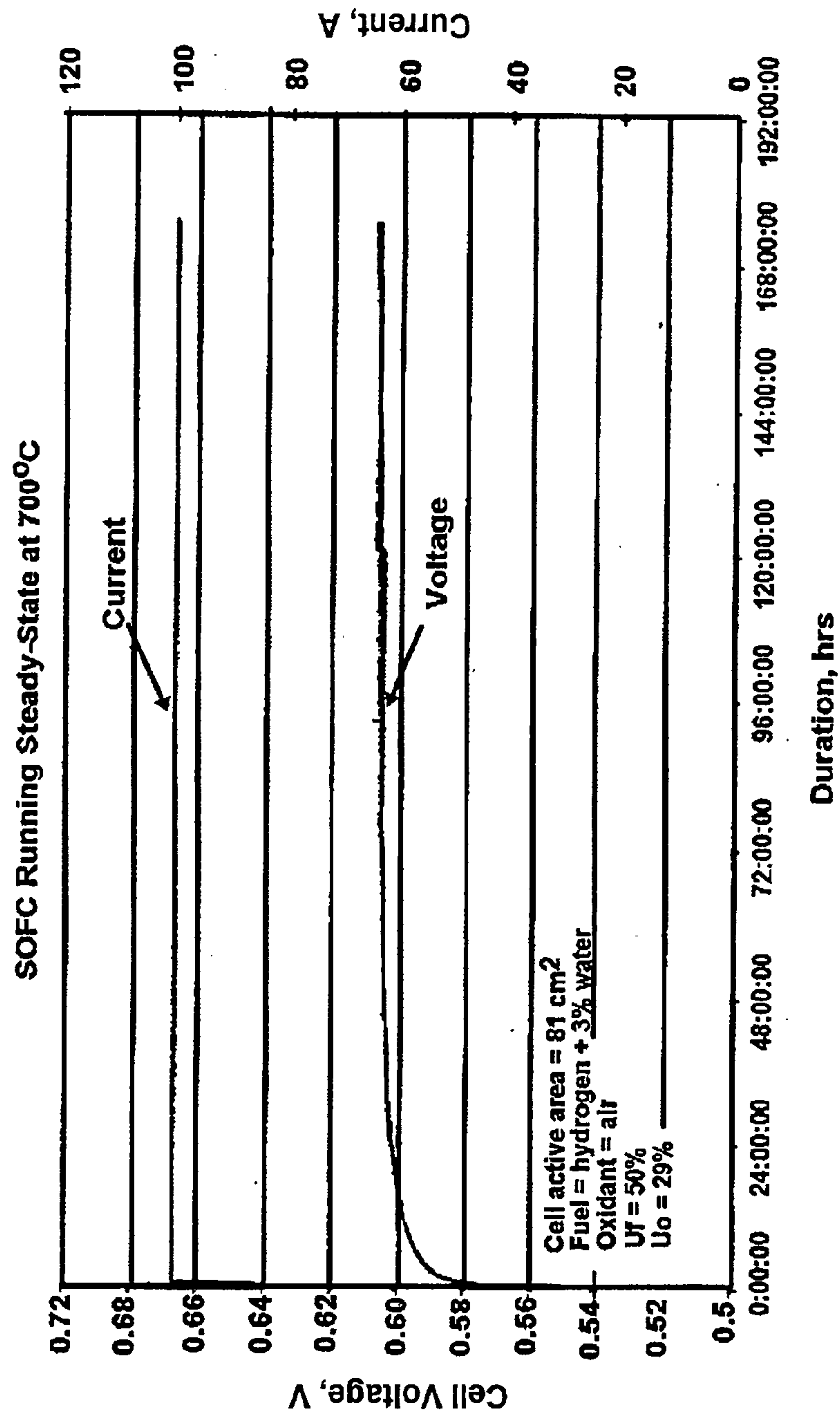


Fig. 7

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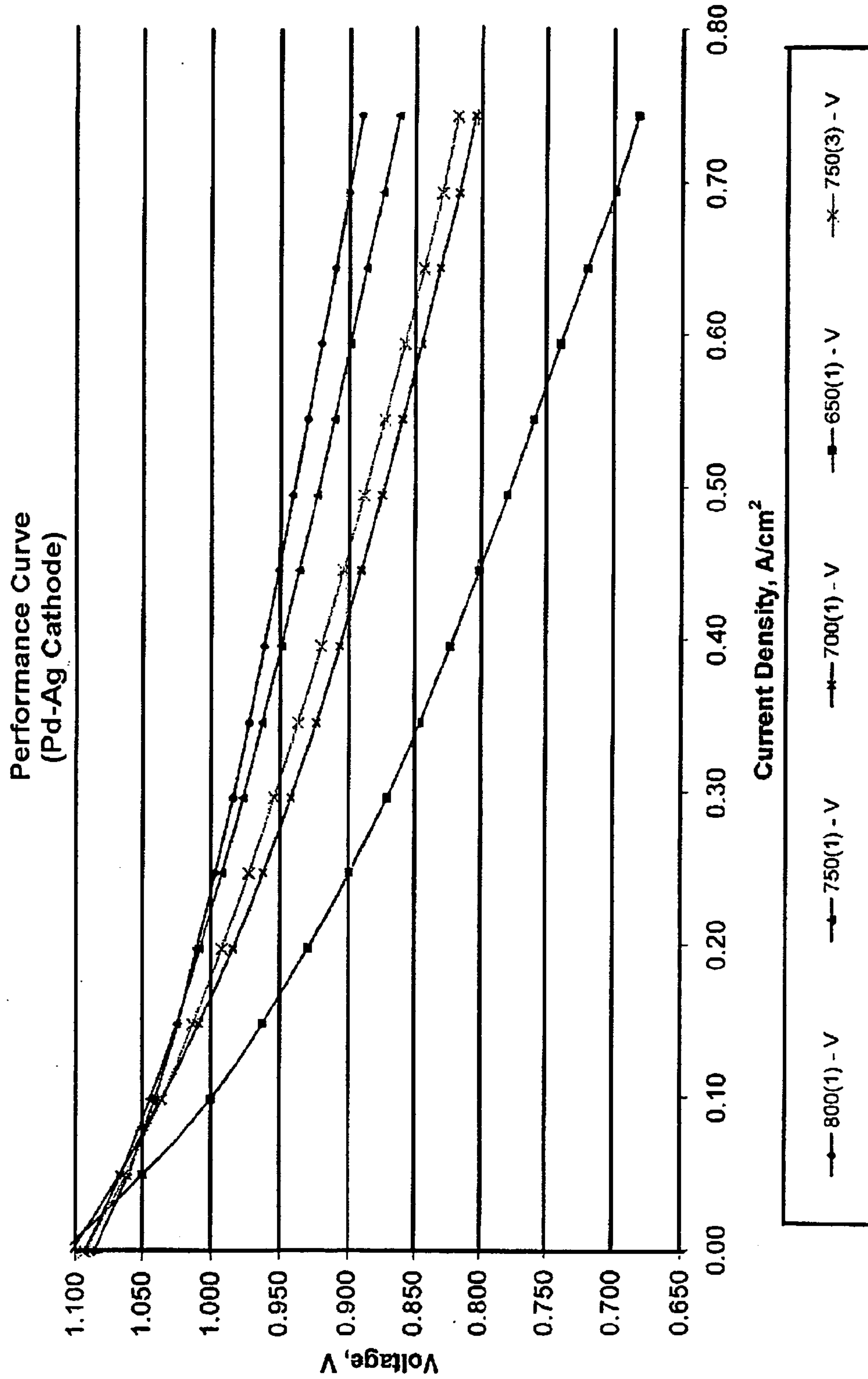
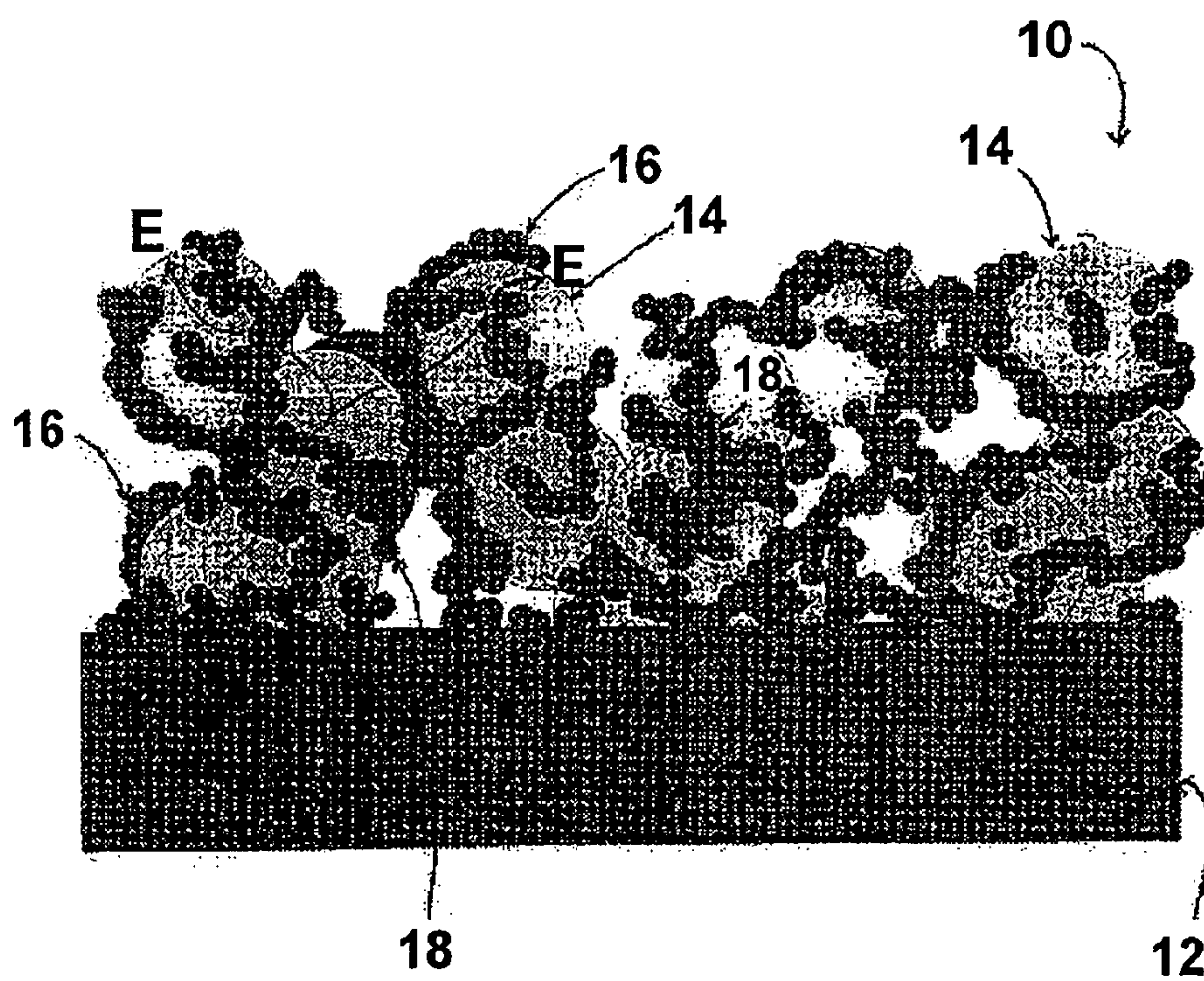


Fig. 8

**Fig. 9**

10/10

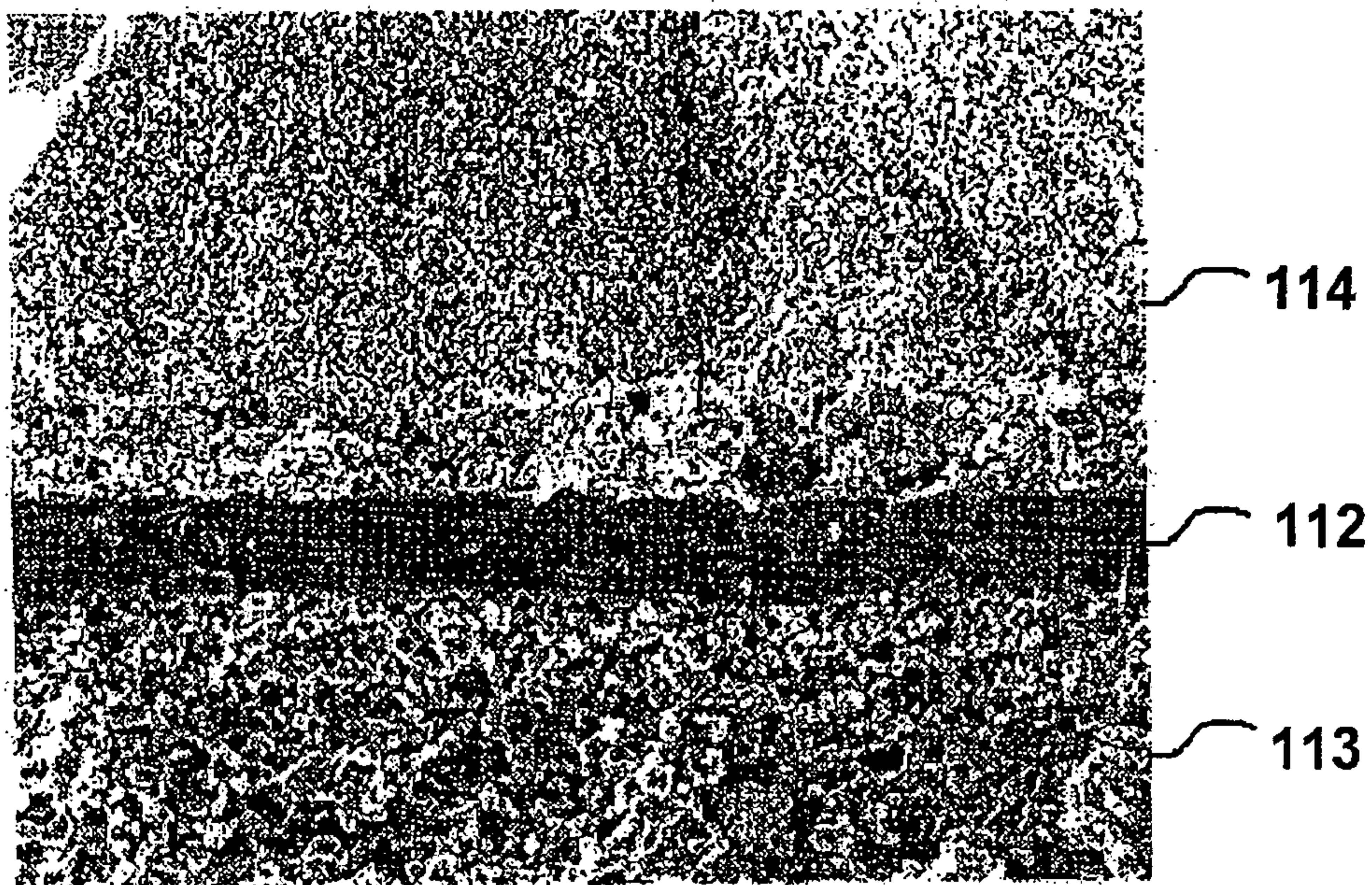
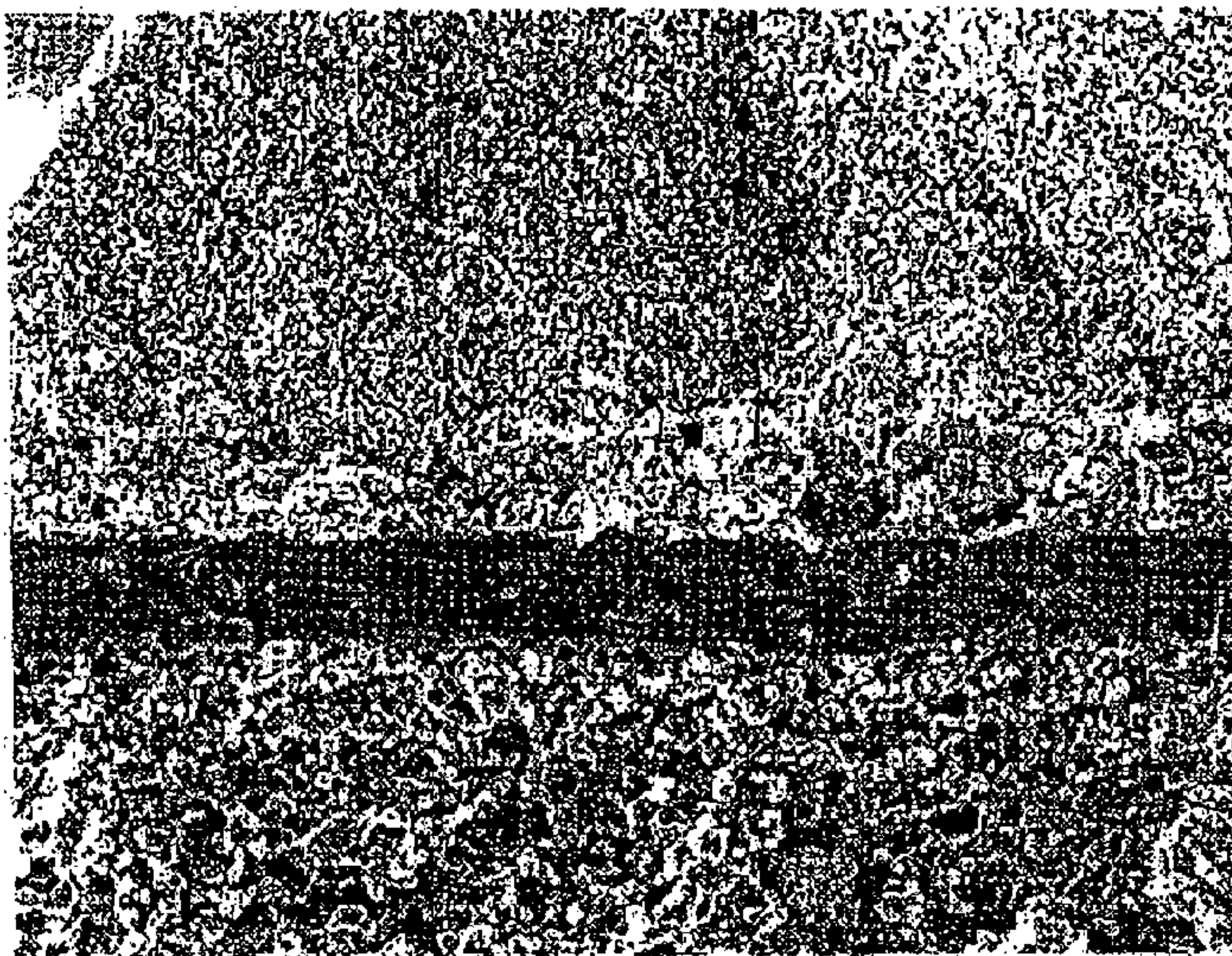


Fig. 10



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113