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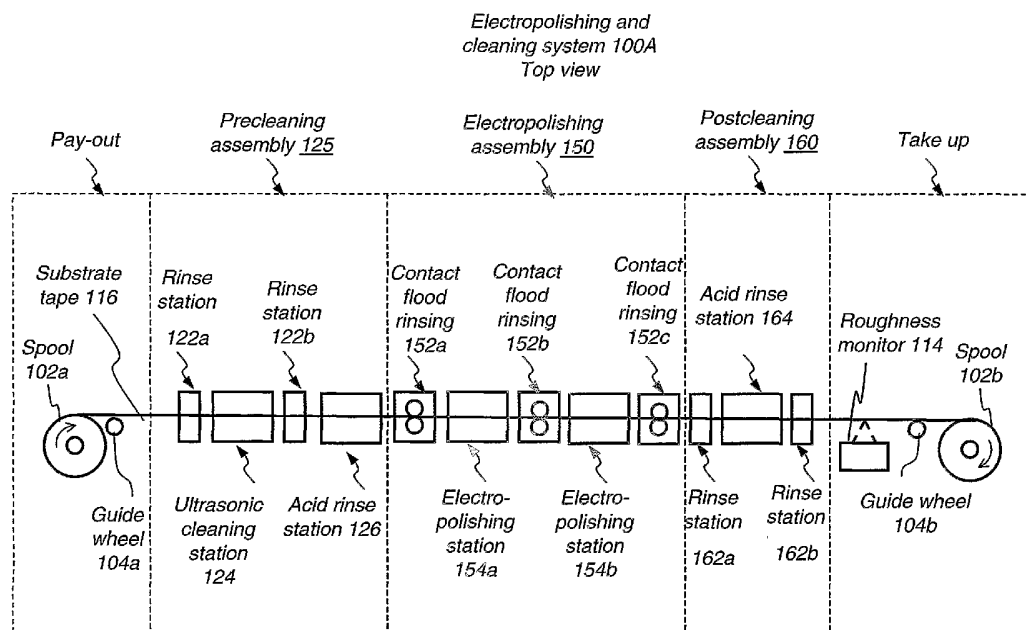
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(54) Title: PROCESS CONTROL METHODS OF ELECTROPOLISHING FOR METAL SUBSTRATE PREPARATION IN PRODUCING YBCO COATED CONDUCTORS



(57) Abstract: Disclosed is a reel-to-reel substrate tape polishing and cleaning system including precleaning, electropolishing section and post cleaning sections and which is suitable for polishing and cleaning long lengths of metal substrate tape used in the manufacture of HTS-coated tape.

Process Control Methods of Electropolishing for Metal Substrate Preparation in Producing YBCO Coated Conductors

Background

Metal substrate preparation is a critical process step in the production of commercially suitable YBCO coated conductors. The surface smoothness, uniformity and cleanliness of the metal substrate has a large influence on the texture of the buffer and the current carrying capability of the YBCO superconducting layer deposited on the buffered metal substrate.

Metal substrates up to 100 meters long can be prepared routinely by mechanical/chemical mechanical polishing. Although high current has been achieved in such lengths of mechanically polished YBCO coated conductors, it is difficult to produce conductors with such performance characteristics over long lengths due to defects and deformation generated by contact and the low speed of mechanical polishing.

As stated in Kreiskott, Sascha et al., Continuous electropolishing of Hastelloy substrates for ion-beam assisted deposition of MgO, Supercond. Sci. Technol. 16 (2003) 613-616, commercial applications of high temperature superconducting wires require fast and cost effective production processes. Electropolishing as an alternative approach of substrate preparation has a speed of at least an order of magnitude higher than mechanical polishing. Surface smoothness comparable to mechanical polishing has been achieved in prototype reel-to-reel electro polishing. This invention addresses methods for process control in electro polishing to produce suitable substrates for commercial YBCO coated conductor production.

Theoretically, the uniformity of electropolishing is much better than that of mechanical polishing. Electropolishing can polish metal substrates at speeds in the range of tens of meters per hour. Multiple tapes can be run in parallel or wide tapes may be utilized. Conventional electropolishing processes utilize external mechanical contacts which impose a limit on the scalability of the process. Nonetheless electropolishing appears to be the

preparation technique of the future. However, metal tapes thus polished still have microscopic defects that affect the performance of YBCO coated conductors.

Two major defects are observed. The first is the presence of residual particles on the surface of the polished substrate due to residual lubricant on the tape and contamination in the electropolishing process solution and rinsing solutions. The second is the presence of a columnar structure across the tape width due to the vertically circulating whirl flow of the electropolishing solution.

It is important to develop new methods that utilize a modified electropolishing process in order to achieve residue free tapes and to minimize the existence of a columnar structure across the width of the tape.

It is an object of this invention to reduce the defects present on metal substrates to be used to produce superconducting tapes.

It is another object of this invention to greatly increase the performance of YBCO coated conductors.

It is an object of the invention to provide a system and method for surface preparation of long lengths of metal substrates in a continuous manner.

It is another object of the invention to provide a cleaning process for producing a highly clean substrate surface that can, after a subsequent film deposition process, achieve high current densities in long lengths of HTS-coated tape.

It is yet another object of the invention to provide a polishing process for producing a substrate having a smooth surface that is of very high quality in a single polishing pass.

It is yet another object of the invention to provide a substrate for high current density superconducting tapes.

Summary

These objects and other benefits are obtained by the inventive method of utilizing flow control for water, air and acids in each process cell/bath. Each cell/bath has its own dedicated reservoir with its own filter, pump, bypass valve, pressure gauge, flow indicator and regulator to control process parameters and residual particles.

Surfactant is utilized in the ultrasonic cleaning step to eliminate oil residue before electro polishing.

Submerged multiple electrodes/electrode pairs are utilized to maximize solution life and to obtain better surface finish.

Fluid circulation is configured by manifolds in the electropolishing cell to direct the electropolishing solution flow along tape length direction instead of across tape width.

Prior to electropolishing, an in-line electrocleaning (pickling) process is provided to descale the substrate, removing oxide particle or oxide layer and exposing a fresh surface in preparation for electropolishing.

To minimize the environmental impact of the process and to increase productivity, in-line deionized water regeneration is provided to collect waste water from each rinse tank and transfer it to a reservoir. The waste water is treated to convert the water to qualified deionized water thereby reducing consumption of deionized water, minimizing down time and permitting maximum water usage in the process while minimizing overall usage for environmental reasons.

A counter flow mechanism is provided to feed fresh deionized water at the last rinse station. The over flow of rinse water from this station is introduced into the other rinse stations in a direction counter to tape movement and is allowed to overflow or is pumped to a reservoir where it is held for deionized water regeneration.

Brief Description of the Drawings

Figure 1A illustrates a top view of a first embodiment of an electropolishing and cleaning system of the present invention suitable for polishing long lengths of metal substrate tape used in the manufacture of HTS-coated tape.

Figure 1B illustrates a side view of a first embodiment of an electropolishing and cleaning system with individual reservoirs, particle control, flow control and regulating devices of the present invention suitable for polishing long lengths of metal substrate tape used in the manufacture of HTS-coated tape.

Figure 1C illustrates a top view of an alternative embodiment of an electropolishing and cleaning system with bipolar electrocleaning and bipolar electropolishing suitable for polishing long lengths of metal substrate tape used in the manufacture of HTS-coated tape.

Figure 1D illustrates a side view of an alternative embodiment of an electropolishing and cleaning system with bipolar electrocleaning and bipolar electropolishing with individual reservoirs, particle control, flow control and regulating devices suitable for polishing long lengths of metal substrate tape used in the manufacture of HTS-coated tape.

Figures 2A, 2B and 2C illustrate a top view, side view and an end view, respectively, of an electropolishing station bath/cell suitable for use as a polishing station within the polishing and cleaning system of the present disclosure.

Figures 3A, 3B, and 3C illustrate a top view, side view and an end view, respectively, of an alternative embodiment of an electropolishing station bath/cell suitable for use as a polishing station within the polishing and cleaning system of the present disclosure.

Detailed Description of the Invention

The present invention is a reel-to-reel substrate tape polishing and cleaning system suitable for polishing and cleaning long lengths of metal substrate tape used in the manufacture of HTS-coated tape. The polishing and cleaning system of the present invention includes

precleaning, electropolishing and post cleaning sections. The electropolishing section further includes contact flood rinsing stations and multiple polishing stations in combination with multiple bipolar/DC contacts and a subsequent drag out rinsing station, each with an individual liquid supply reservoir, filters and flow control devices arranged along the axis of the translating metal substrate tape.

The inventive methods disclosed include a new configuration of the process steps, sequencing the cleaning and polishing of the substrate in a manner such that the final product contains a greatly diminished level of defects.

The new configuration includes precleaning, electropolishing and post cleaning sections. The precleaning section includes water rinsing, ultrasonic cleaning and acid rinsing.

In one preferred embodiment, the precleaning section includes water rinsing, ultrasonic cleaning preferably utilizing non-foaming surfactant and electrocleaning with bipolar electrodes in light acid. Electrocleaning with bipolar electrodes is accomplished by translating the submerged substrate tape which is not connected to the power supply, between an anode and a cathode. At least one pair of anode and cathode is used for electrocleaning. The configuration of the bipolar anode/cathode pair can be arranged in any polarity order or by adding extra anode/cathode[s] in additional to the bipolar pairs. These anodes and cathodes are connected to the power supply and are placed in the longitudinal direction of the to-be-polished substrate metal tape. At low current, the surface of the substrate closest to the anode becomes cathodic and the surface of the substrate nearest the cathode becomes anodic. The anodic surface is scrubbed by evolving hydrogen while the cathodic surface is scrubbed by oxygen evolution. As the substrate passes through the series of cells of reversed polarity, the alternative hydrogen and oxygen scrubbing removes irregularities in the surface and results in a surface, after rinsing, ready for electropolishing.

The electropolishing section includes at least one cell with at least one multiple bipolar/DC contact disposed in juxtaposition to each cell so as to reduce the current resistance and to limit the heating of the metal tape.

The bipolar/DC contacts are submerged in the solutions. In the bipolar configuration, pairs of anodes and cathodes are disposed next to each other along the length of to be polished metal tape. There is no physical contact between the anodes/cathodes and the to be polished metal tape; electric current is passed from the anodes to the metal tape through the electropolishing solution.

In one embodiment of the invention contact flood rinsing stations are interposed between each of multiple electropolishing stations. The contact flood rinse stations contain anode or anode pairs that contact with the to-be-polished metal tape. The metal tape is polished as it moves through electropolishing station bath/cell[s] that contain cathodes connected to the negative terminal of the power supply. The contact flood rinsing also removes any nonadherent deposits of debris removed during the electropolishing step.

In a second embodiment utilizing bipolar electrodes, the electropolishing assembly includes multiple electrolytic cells with adjacent cells containing electrodes of reversed polarity.

In one embodiment a cathode is used in the last step of the electropolishing process. The benefit derived from this variation is better efficiency of polishing (no current leaking), better cleanliness of tape (diminishment of the amount of residue resulting from the products of the high temperature reaction between the metal and the electropolishing solution) and longer solution life.

The electrodes utilized in the electropolishing cells may vary in length (the dimension parallel to the longitudinal direction of to-be-polished substrate tape) from about 0.5 inch to about 4 inches, preferably from about 0.5 to about 2 and most preferably from about 0.75 to about 1.5 inches.

In a preferred configuration of the invention, the electrodes are no longer than 4 inches or are partially shielded to obtain optimum surface smoothness. In general, the shield may cover from about 1 to about 99 percent of the electrode. The shield is utilized most desirably where the electrodes are longer than 4 inches and is utilized to achieve the same smoothness with such long electrodes as is achieved with 4 inch or shorter electrodes.

The post cleaning section includes drag out rinsing, light acid rinsing and final DI rinsing.

In the process, the substrate is first subjected to cleaning in precleaning assembly **120** or **125**. It then moves through the electropolishing assembly **140** or **150**, and the post cleaning DI rinse assembly **160**.

By translating through the precleaning stations **122a**, **122b**, **124** and **126** and multiple polishing stations **152a**, **152b**, **152c**, **154a**, **154b** in a first embodiment or precleaning stations **122a**, **122b**, **124** and **128** and multiple polishing stations **146a**, **146b**, **146c**, **146d**, **148a**, **148b**, **148c**, and **148d** in a second embodiment, in combination with the subsequent cleaning stations **162a**, **162b**, and **164**, the metal substrate tape **116** experiences a series of cleaning, polishing and cleaning events to progressively diminish its surface contamination and roughness. More specifically, the metal substrate tape **116** experiences, via progressive stages, first a water rinsing, surfactant bath and acid dip, then electropolishing, then a drag out rinsing and acid dip event in combination with a respective rinsing event as it translates through the polishing section.

The pre and post cleaning assemblies **125** and **160** may include an ultrasonic cleaning station **124** and at least one rinsing bath **122a**, preferably two rinsing baths **122a** and **122b**, with individual liquid supply reservoirs, filters **106** and flow control devices **108**. The cleaning and rinsing baths act to control particle contamination and remove any surface contaminants that are detrimental to the surface quality of the substrate tape and which may limit the achievable current density in the finished HTS-coated tape. Thus, the metal substrate tape **116** achieves, in a single pass through the polishing and cleaning system of the present invention, a surface smoothness and cleanliness that is suitable for the subsequent deposition of a buffer layer.

Figures 1A and **1B** illustrate a polishing and cleaning system **100A** for the surface preparation of a substrate tape used in the manufacture of HTS-coated tape in accordance with the invention. As illustrated in **Figure 1A**, the polishing and cleaning system **100A** includes a precleaning section **125** that performs the substrate tape cleaning function and feeds a polishing section **150** that performs the substrate tape polishing function and which

feeds a post cleaning section **160** that performs a subsequent substrate tape cleaning function.

The polishing and cleaning system **100A** includes at least one pair of spools **102**, i.e., a spool **102a** and a spool **102b** (**Figure 1A**). The spool **102a** serves as a payout spool located at the entry point of the polishing and cleaning system **100**. A length of substrate tape **116** that is formed of a metal such as stainless steel or a nickel alloy such as Inconel/Hastelloy is wound upon the spool **102a**. The substrate tape **116** may pay out from the top of the spool **102a** as shown in **Figure 1A** or, alternatively, from the underside of the spool **102a**. The substrate tape **116** is capable of withstanding high temperatures and vacuum conditions, and is typically between 3 mm and 15 cm in width and may be upwards of several kilometers in length. The substrate tape **116** typically has several meters of "leader" at both ends to aid in handling. The substrate tape **116** is laced from the spool **102a** through the precleaning section **125**, the polishing section **150** and the post cleaning section **160** of the polishing and cleaning system **100A** and wound onto the spool **102b**, which serves as a take-up spool, at the exit point of the polishing and cleaning system **100A**.

With reference to the portion of **Figure 1A** and **Figure 1B** which illustrates the tape precleaning function, the precleaning section **125** further includes at least one rinse, ultrasonic and/or solution rinse/bath and acid rinse station(s). Each rinse/bath station has a separate process cell located along the path of substrate tape **116**. Each rinse/bath station has a separate solution container or reservoir located at the lower part of the section. The solution of each rinse/bath station is pumped to the process cell through a particulate control device, a filter **106** for example, and a flow control device, for example a flow meter/regulator **108**.

Upon exit from the precleaning section the substrate enters the electropolishing section. The electropolishing section incorporates multiple cells with additional DC contacts among each cell. In the conventional electropolishing procedure, single cells are used and each cell contains two DC contacts. The provision of multiple cells, preferably two or more cells with each cell containing a minimum of one DC contacts provides the following benefits: evenly

distributed current along the tape and a lesser amount of heat generated due to the resistance of the metal tape. These benefits and the superior results obtained are caused by diminishment of the amount of residue resulting from the products of the high temperature reaction between the metal and the electropolishing solution.

With reference to the portion of **Figure 1A** and **Figure 1B**, which illustrates the substrate tape polishing function, the polishing section **150** further includes at least two contact rinse/electropolishing station(s). Each rinse station has a separate process cell located along the path of substrate tape **116**. A current is applied to the substrate tape through DC contacts in contact rinse stations while it passes through the polishing stations to activate the polishing process. Each rinse/bath station has a separate solution container or reservoir located at the lower part of the section. The solution of each rinse/bath station is pumped to the process cell through a particulate control device, a filter for example, and a flow control device, for example a flow meter/regulator.

With reference to the portion of **Figure 1A** and **Figure 1B**, which illustrates the substrate tape post cleaning function, the post cleaning section **160** further includes at least one rinse, ultrasonic and/or solution rinse/bath station(s). Each rinse/bath station has a separate process cell located along the path of substrate tape **116**. Each rinse/bath station has a separate solution container or reservoir located at the lower part of the section. The solution of each rinse/bath station is pumped to the process cell through a particulate control device, a filter for example, and a flow control device, for example a flow meter/regulator.

Figures 1C and **1D** illustrate a polishing and cleaning system **100B** for the surface preparation of a substrate tape used in the manufacture of HTS-coated tape in accordance with the invention. As illustrated in **Figure 1C**, the polishing and cleaning system **100B** includes a precleaning section **120** that performs the substrate tape cleaning function and feeds a polishing section **140** that performs the substrate tape polishing function and which feeds a post cleaning section **160** that performs a subsequent substrate tape cleaning function.

With reference to **Figures 1C** and **1D**, the polishing and cleaning system **100B** includes at least one pair of spools **102**, i.e., a spool **102a** and a spool **102b** (**Figure 1C**). The spool **102a** serves as a payout spool located at the entry point of the polishing and cleaning system **100B**. A length of substrate tape **116** that is formed of a metal such as stainless steel or a nickel alloy such as Inconel/Hastelloy is wound upon the spool **102a**. The substrate tape **116** may pay out from the top of the spool **102a** as shown in **Figure 1C** or, alternatively, from the underside of the spool **102a**. The substrate tape **116** is capable of withstanding high temperatures and vacuum conditions, and is typically between 3 mm and 15 cm in width and may be upwards of several kilometers in length. The substrate tape **116** typically has several meters of "leader" at both ends to aid in handling. The substrate tape **116** is laced from the spool **102a** through the precleaning section **120**, the polishing section **140** and the post cleaning section **160** of the polishing and cleaning system **100B** and wound onto the spool **102b**, which serves as a take-up spool, at the exit point of the polishing and cleaning system **100B**.

With reference to the portion of **Figures 1C** and **1D** which illustrates the tape precleaning function, the precleaning section **120** further includes at least one rinse, ultrasonic and/or solution rinse/bath and electrocleaning in solution station(s) **128**. Each rinse/bath station has a separate process cell located along the path of substrate tape **116**. Each rinse/bath station has a separate solution container or reservoir located at the lower part of the section. The solution of each rinse/bath station is pumped to the process cell through a particulate control device, a filter **106** for example, and a flow control device, for example a flow meter/regulator **108**.

Upon exit from the precleaning section the substrate enters the electropolishing section. The electropolishing section incorporates multiple cells with additional bipolar contacts in each cell. In the conventional electropolishing procedure, single cells are used and each cell contains two bipolar electrodes. The provision of multiple cells, preferably two or more cells with each cell containing a minimum of one pair of bipolar electrodes provides the following benefits: evenly distributed current along the tape and a lesser amount of heat generated due to the resistance of the metal tape. These benefits and the superior results

obtained are caused by diminishment of the amount of residue resulting from the products of the high temperature reaction between the metal and the electropolishing solution.

With reference to the portion of **Figure 1C** and **Figure 1D** that illustrate the substrate tape polishing function, the polishing section **140** includes at least two electropolishing station(s) containing bipolar electrodes. In the embodiment of the figures four electropolishing cells, **142a**, **142b**, **142c**, and **142d** are shown with alternating polarities.

With reference to the portion of **Figure 1C** and **Figure 1D**, which illustrates the substrate tape post cleaning function, the post cleaning section **160** further includes at least one rinse, ultrasonic and/or solution rinse/bath station(s). Each rinse/bath station has a separate process cell located along the path of substrate tape **116**. Each rinse/bath station has a separate solution container or reservoir located at the lower part of the section. The solution of each rinse/bath station is pumped to the process cell through a particulate control device, a filter for example, and a flow control device, for example a flow meter/regulator.

Upon exit from the polishing section the substrate enters the post cleaning section. The post cleaning section includes drag out rinsing, light acid rinsing and final DI rinsing. In the drag out rinsing step reaction products and the electropolishing solution are removed. Light acid rinsing is accomplished utilizing an acid mixture of sulfuric acid and water and is preferably conducted at a temperature in the range of from about 15 C to about 29 C. The acid concentration is in the range of 0.5 to about 20%. Final DI rinsing occurs at room temperature.

Each process step is separated from the other steps by conventional means, such as shields, partitions and air blanket to prevent water or acid drag out. All utilities are metered in the conventional manner by flow meters and pressure regulating valves as required.

Figures 2A, 2B and **2C** respectively show top side and end views of one embodiment of an electropolishing section, **142/154**, of the process. The substrate tape **116** enters the electropolishing station and is contacted by flowing electropolishing solution injected into

the electropolishing section EP cell **154a** and **154b** in a first embodiment or **142a**, **142b**, **142c**, or **142d** in a second embodiment through the electropolishing solution inlet manifold assembly **210** comprising multiple inlet manifolds **210a**, **210b**, **210c**, **210d**, **210e** and **210f** which inject EP solution **313** constantly to maintain tape **116** submerged in EP solution **313**. In a preferred embodiment, the inlet manifold assembly **210** comprises 6 inlet manifolds, three located so as to impinge on opposite sides of the translating substrate tape **116**. The electropolishing solution flows over the tape and is recirculated through electropolishing solution drain outlets **212a** and **212b**. The tape exits the first EP cell and moves on to the next EP cell in the electropolishing section until it has passed through all the cells present and then moves to the next section, the postcleaning section **160**.

Figures 3A, 3B and 3C respectively show top side and end views of an alternate embodiment of the electropolishing section, **142/154**, of the process. The substrate tape **116** enters the electropolishing station and is contacted by flowing electropolishing solution injected into the electropolishing section tank --- through the electropolishing solution inlet manifold assembly **310** comprising multiple inlet manifolds **310a** and **310b** which inject EP solution **313** constantly to maintain tape **116** to be submerged by EP solution. In a preferred embodiment the inlet manifold assembly **310** comprises 2 inlet manifolds, one located at approximately the longitudinal midpoint of the electropolishing tank **142/154** with one manifold injecting solution **313** toward the entering tape **116** and the other positioned to inject electropolishing solution toward the leaving tape. An electropolishing solution agitation assembly **312** comprising a multitude of low shear mixers **312a**, **312b**, **312c**, **312d**, **312e**, **312f** are positioned on either side of the translating tape and serve to bring fresh electropolishing solution into contact with the tape as it moves through the electroplating station. The electropolishing solution flows over the tape and is recirculated through electropolishing solution drain outlets **314a** and **314b**. The tape exits the electropolishing section and moves to the next section, the postcleaning section.

Claims

1. A method of preparing a substrate for superconductors comprising:

precleaning at least one substrate in at least one first solution, said at least one first solution in fluid communication with at least one first particle control mechanism;

electropolishing said at least one substrate in at least one second solution in an electropolishing section comprising at least one electropolishing cell containing at least one electrode, said at least one second solution in fluid communication with at least one second particle control mechanism; and

post cleaning said at least one substrate in at least one third solution, said at least one third solution in fluid communication with at least one third particle control mechanism.

2. The method of claim 1 further comprising controlling a flow of at least one of said first, second and third solutions wherein with at least one flow control device in fluid communication with at least one of said solutions.
3. The method of claim 1 wherein the electropolishing section comprises at least one bipolar electrode disposed between at least two electropolishing cells.
4. The method of claim 3 wherein the bipolar electrode in the last electropolishing cell is a cathode.
5. The method of claim 4 wherein the at least one bipolar electrode is shielded such that no more than 4 inches of the width of the electrode is exposed.
6. The method of claim 4 wherein the at least one bipolar electrode is shielded such that from about 1 to about 99 percent of the electrode is exposed.

7. The method of claim 1 wherein the at least one electrode is less than about 4 inches long.
8. The method of claim 1 wherein the at least one electrode is from about 0.5 inch to about 4 inches long.
9. The method of claim 1 wherein the at least one electrode is from about 1.5 inches to about 2.5 inches long.
10. The method of claim 1 wherein the at least one electrode is from about 1 to about 3 inches long.
11. The method of claim 1 further comprising applying a current to said substrate.
12. The method of claim 1 wherein the current density in the electrocleaning section is in the range of 20 to 300 A/ft².
13. The method of claim 1 wherein the current density in the electropolishing section is in the range of 200 to 1200 A/ft².
14. The method of claim 1 wherein the current density in the electropolishing section is in the range of 200 to 900 A/ft².
15. A method of preparing a substrate for superconductors comprising:

precleaning at least one substrate in at least one first solution, said at least one first solution in fluid communication with at least one first particle control mechanism,

wherein said at least one solution is in fluid communication with at least one flow control device;

electropolishing said at least one substrate in at least one second solution, said at least one second solution in fluid communication with at least one second particle control mechanism,

wherein said at least one solution is in fluid communication with at least one flow control device; and

post cleaning said at least one substrate in at least one third solution, said at least one third solution in fluid communication with at least one third particle control mechanism,

wherein said at least one third solution is in fluid communication with at least one third flow control device.

11. The method of claim 1 wherein in-line deionized water is regenerated in-line.
12. The method of claim 1 wherein counter current flow with a flow control mechanism utilized for regeneration.
13. The product of the process of claim 1.

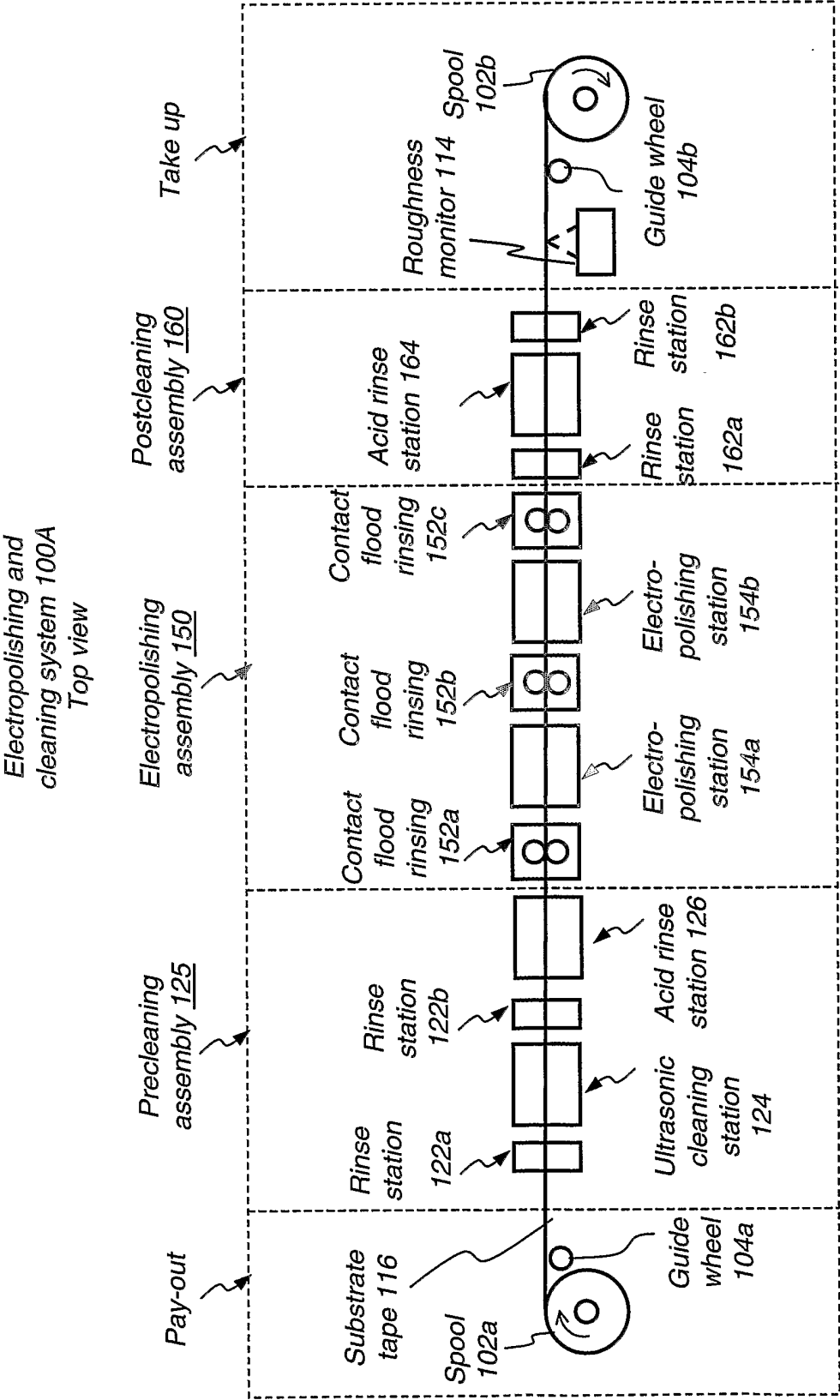


FIG. 1A

Electropolishing and
cleaning system 100A
Side view

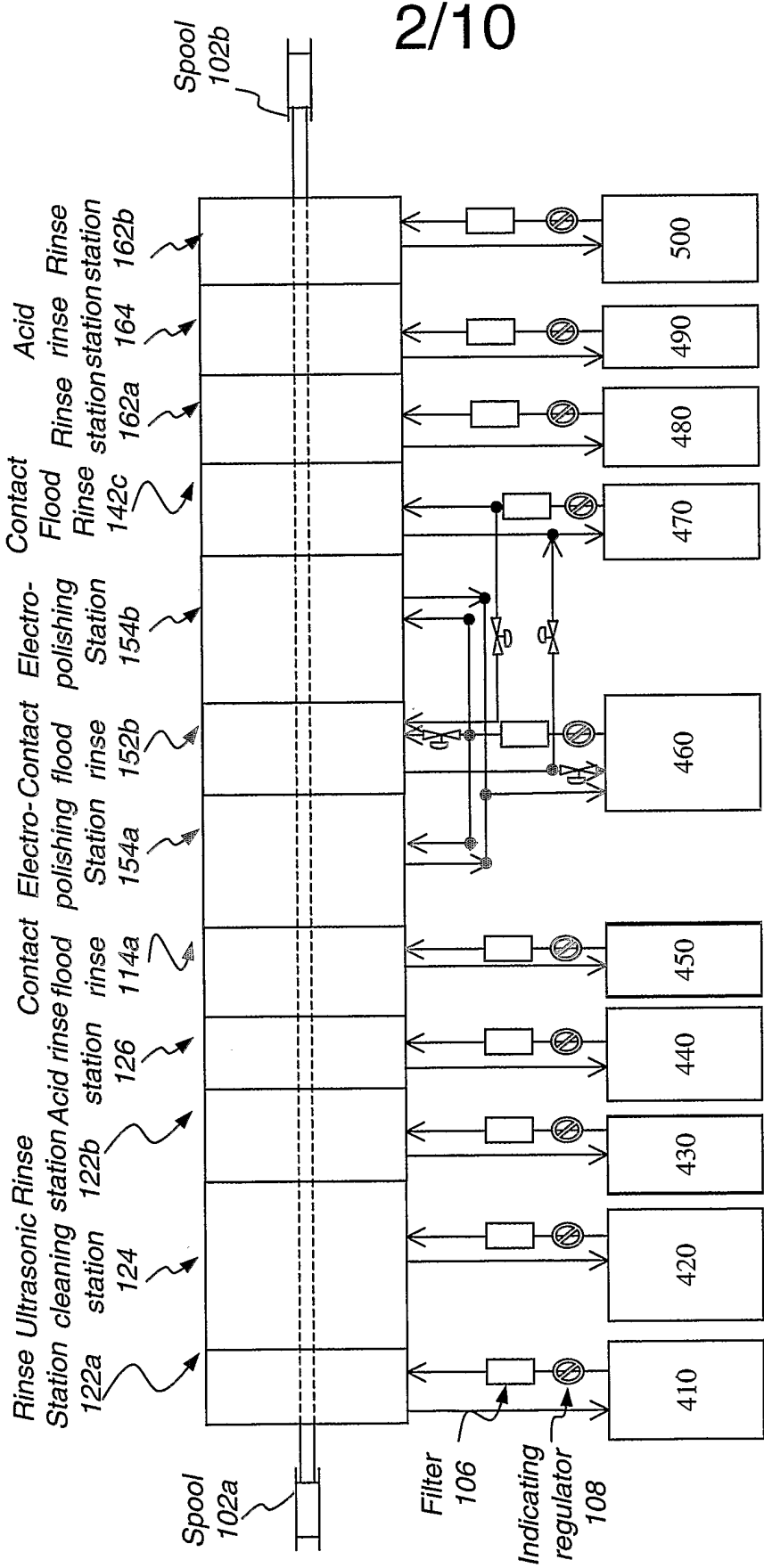


FIG. 1B



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Electropolishing and
cleaning system 100B
Top view

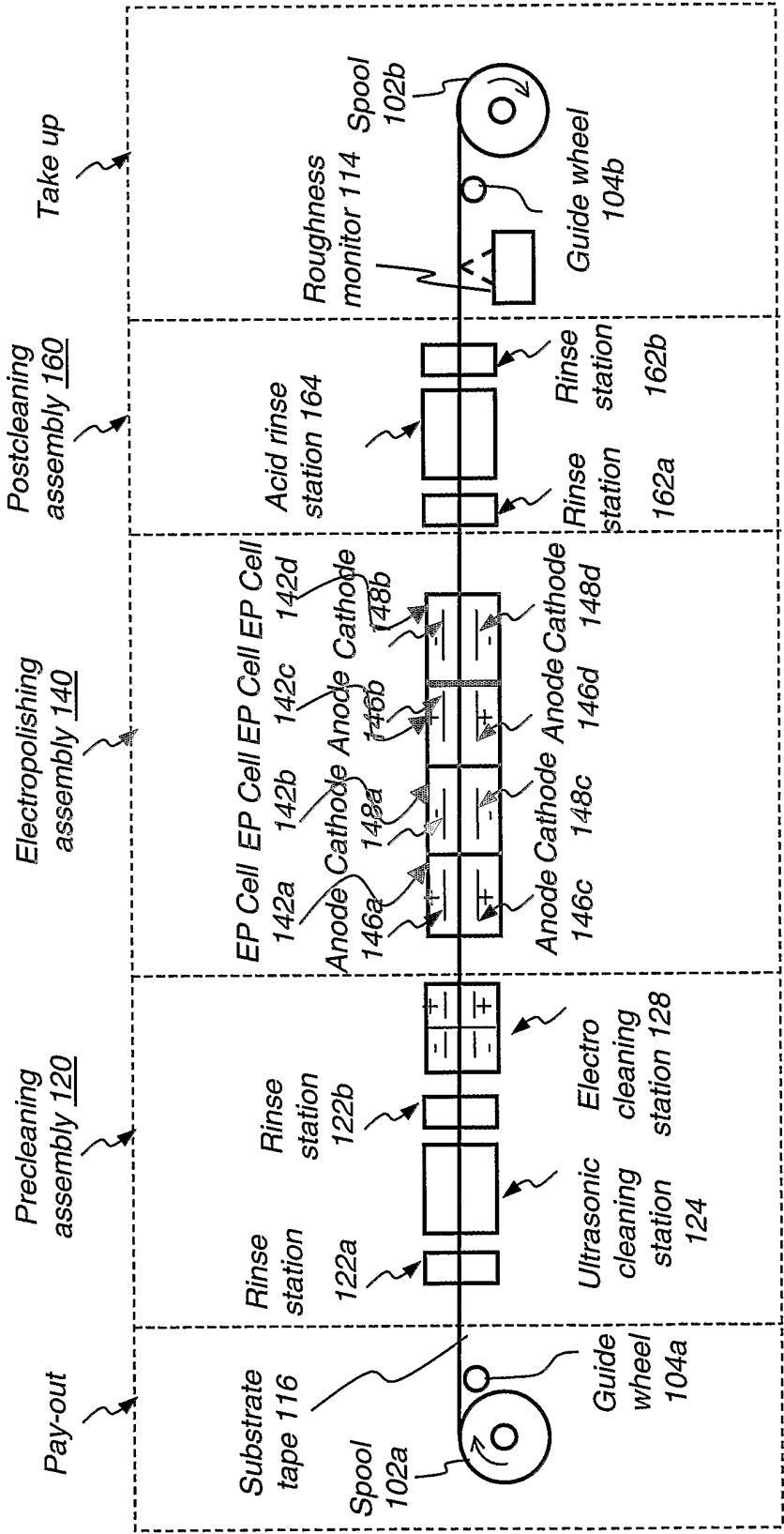


FIG. 1C



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Electropolishing andB
cleaning system 100B
Side view

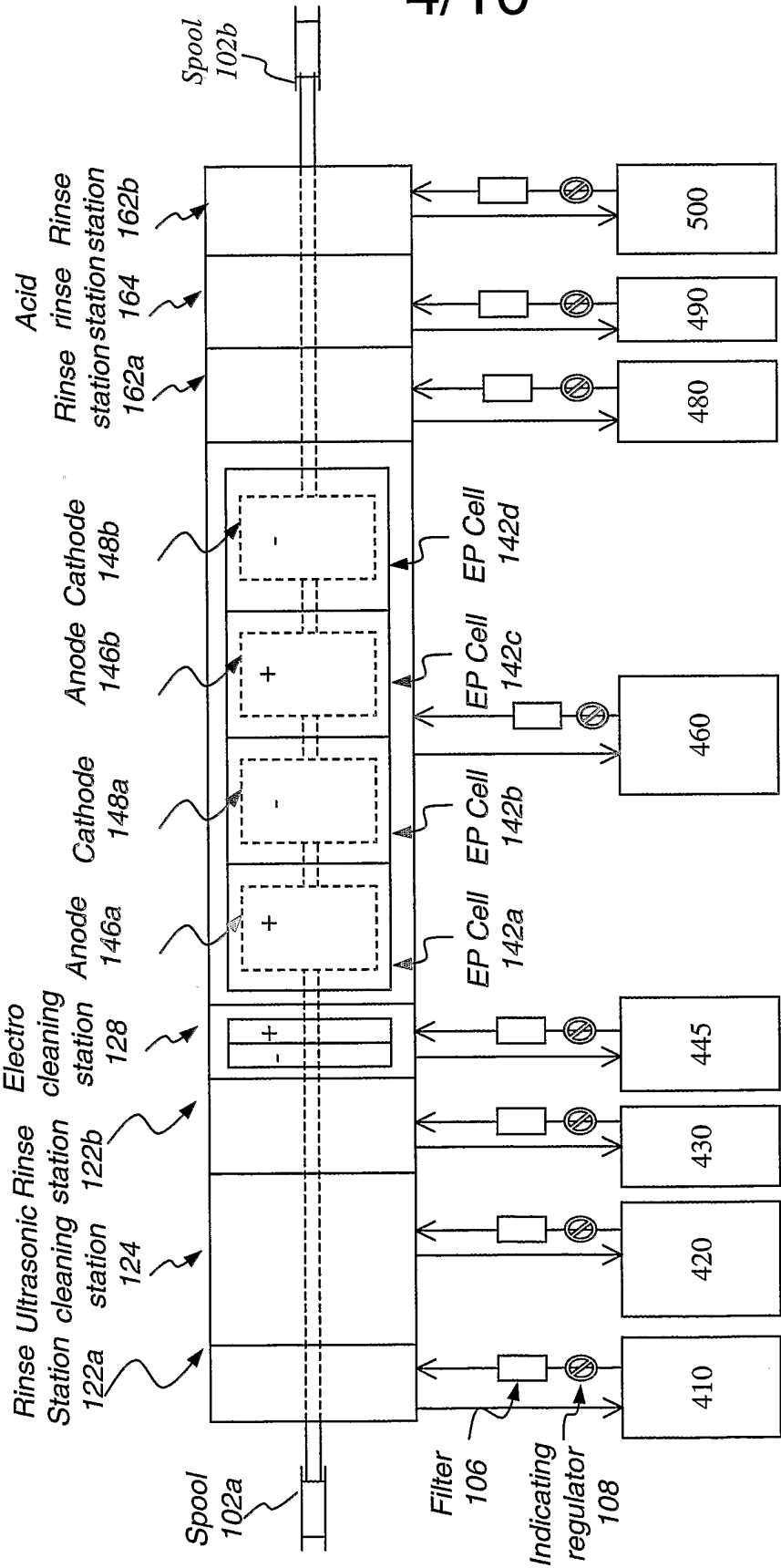


FIG. 1D

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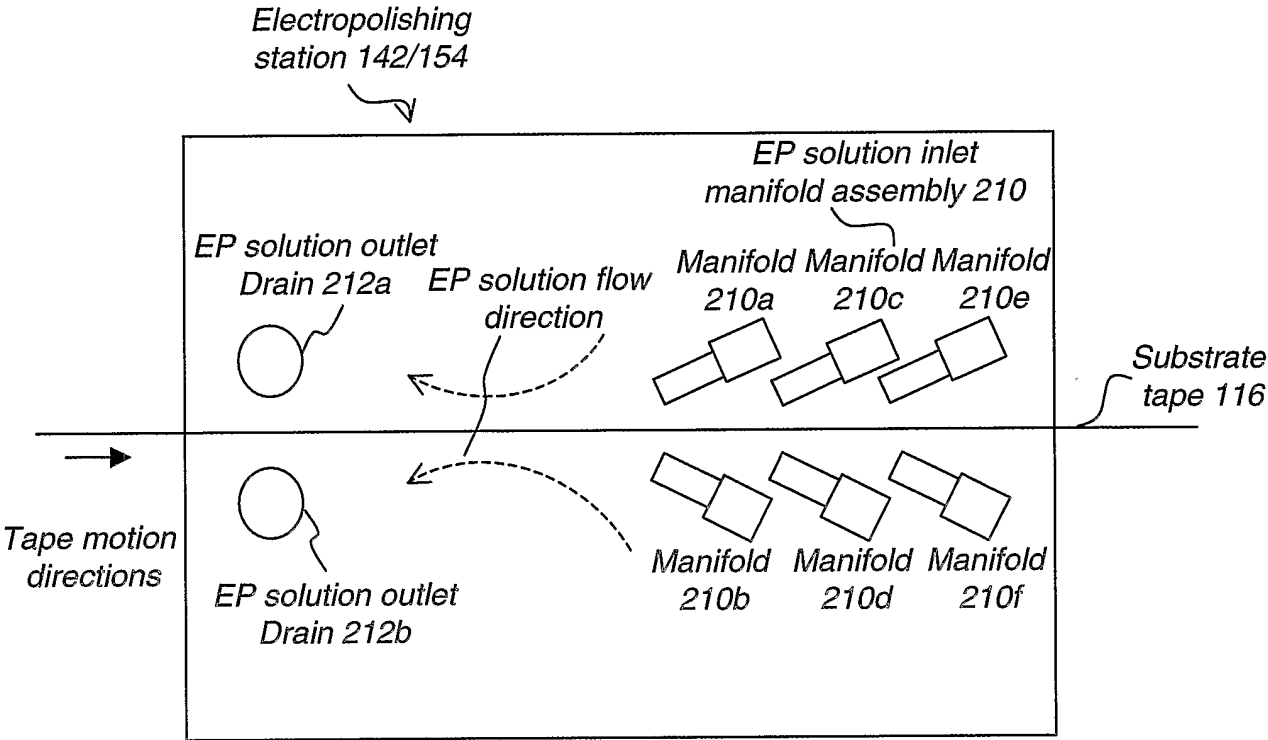


FIG. 2A

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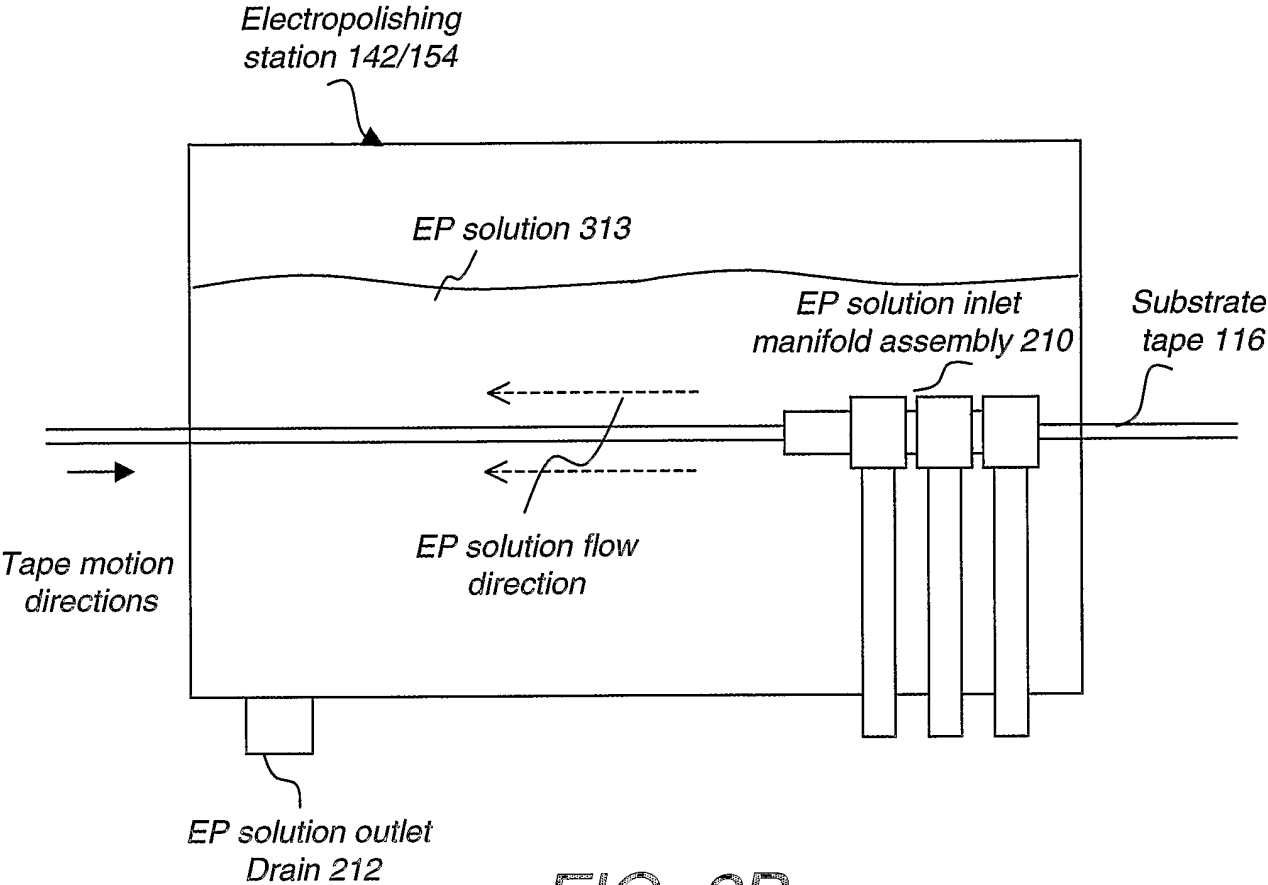


FIG. 2B



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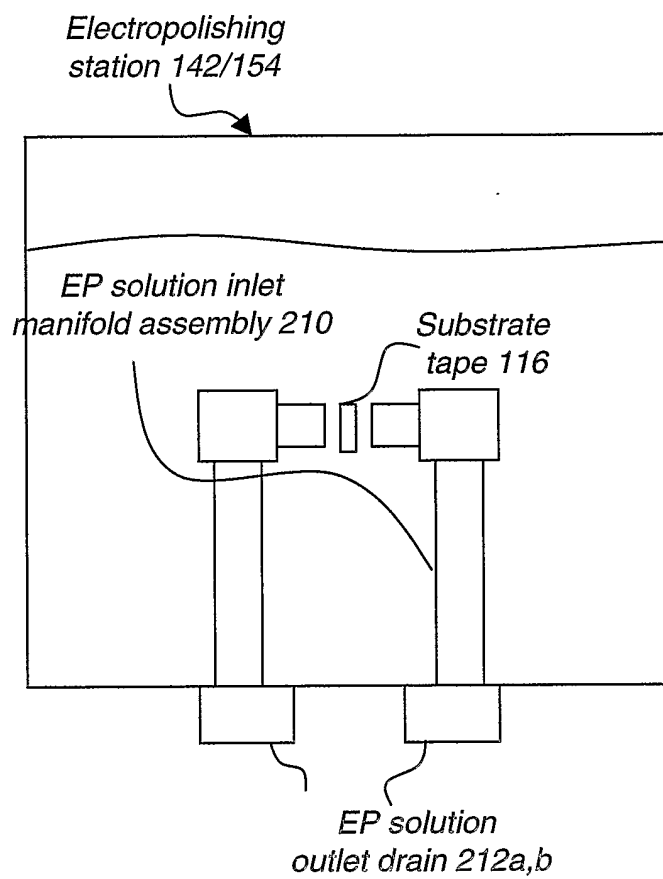


FIG. 2C

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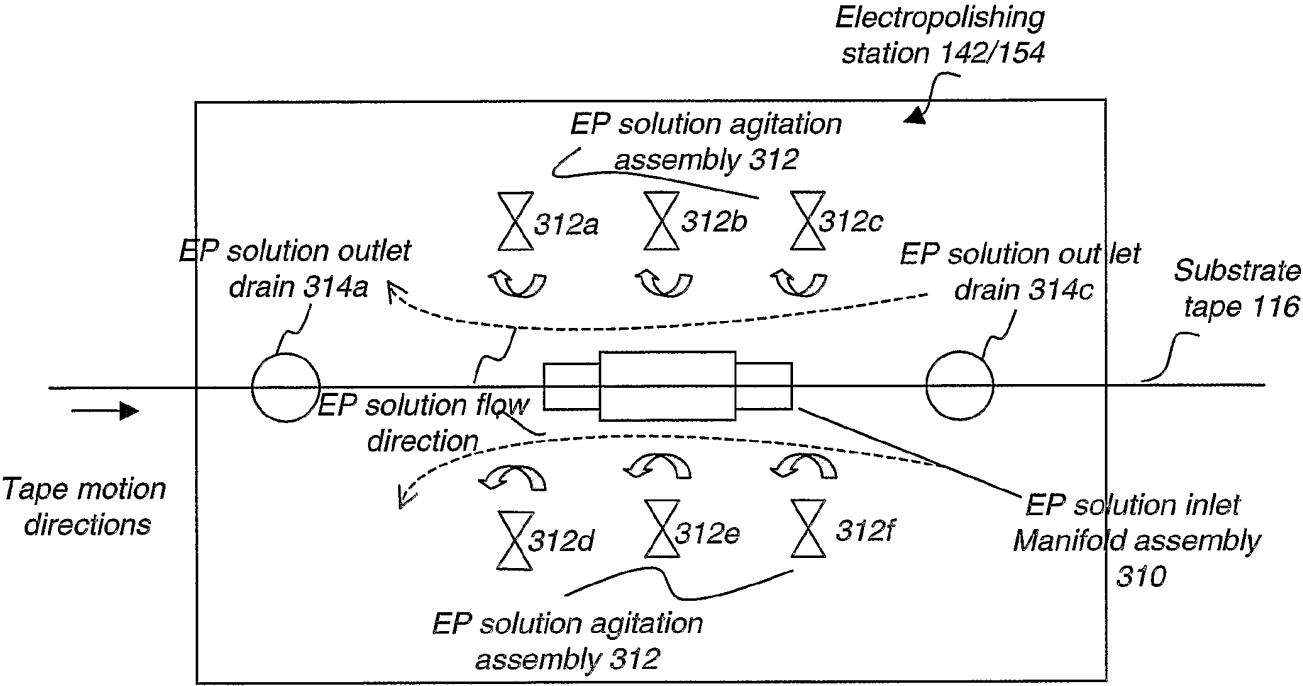


FIG. 3A

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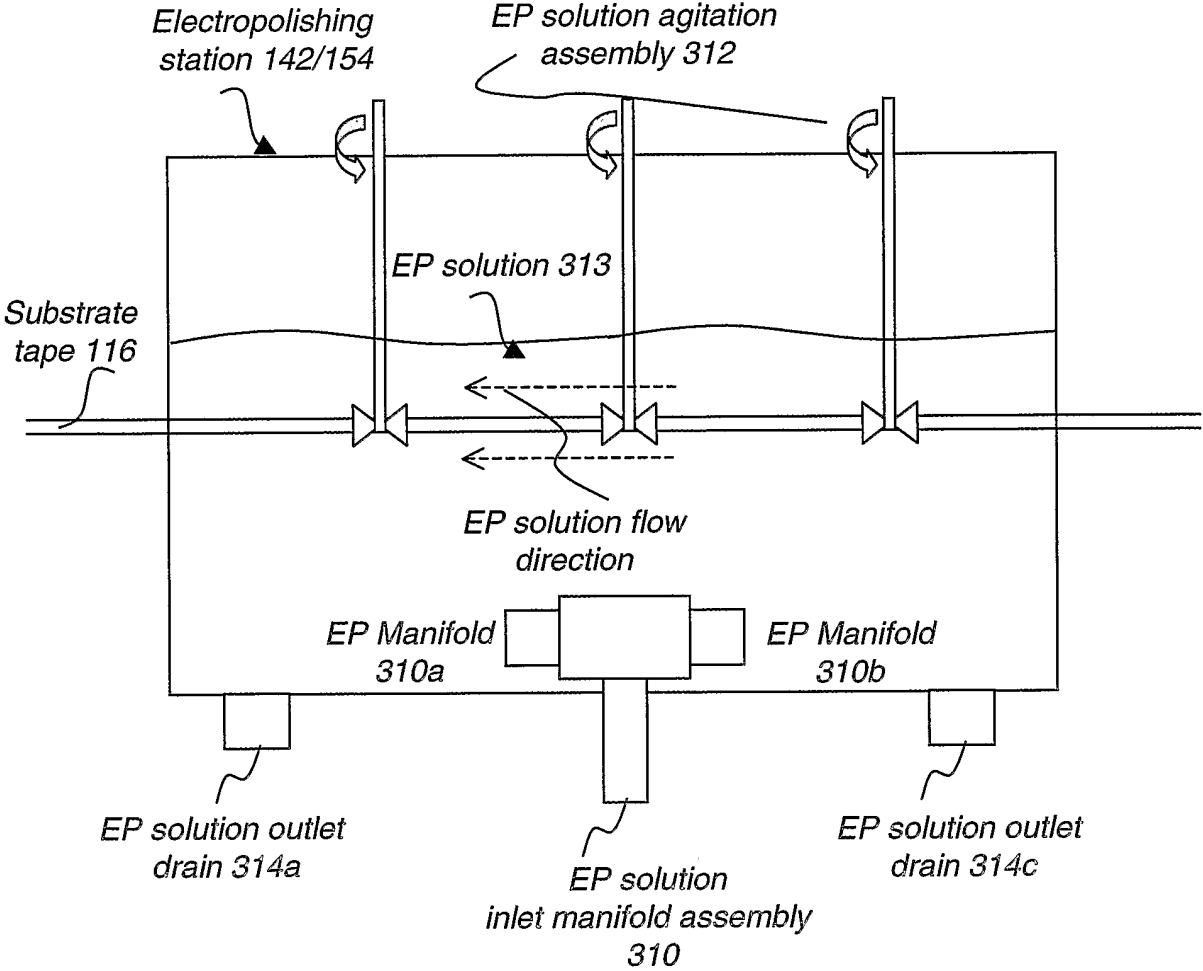


FIG. 3B

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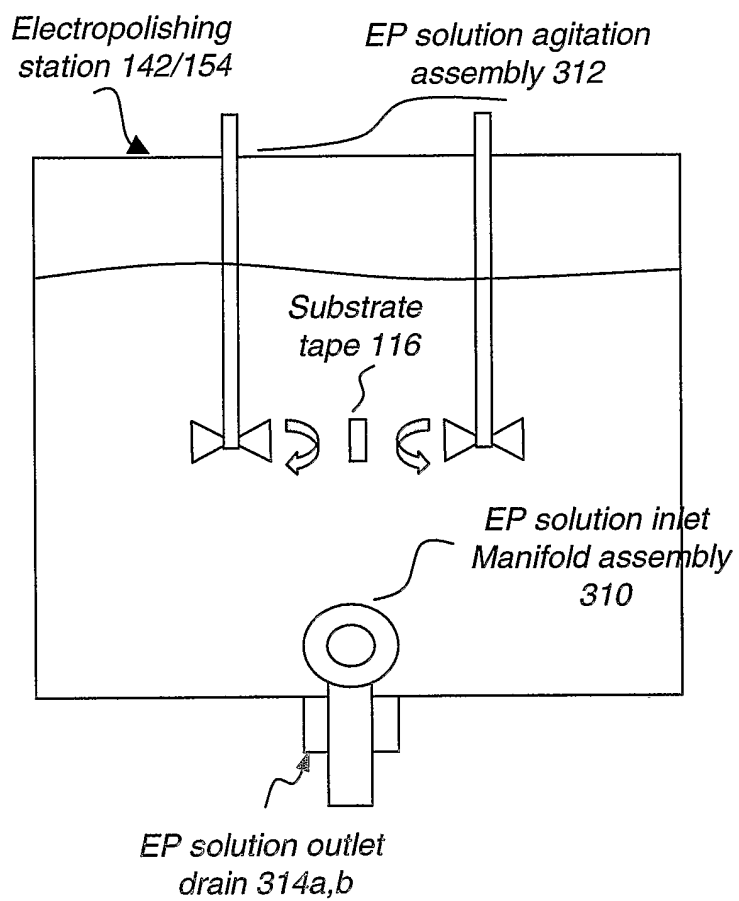


FIG. 3C

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US04/20220

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C25F 3/00
US CL : 205/660, 661

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 205/660, 661

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EAST ; preclean\$3; electropolish\$3; particle near5 control; superconductor

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6,544,402 A (GRAF) 08 April 2003 (08.04.2003)	

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

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"&" document member of the same patent family

Date of the actual completion of the international search

10 November 2004 (10.11.2004)

Date of mailing of the international search report

23 NOV 2004

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