Chapter

The Design and Development of Control System for High Vacuum Deoxygenated and Water-Removal Glove Box with Cycling Cleaning and Regeneration

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Abstract

This study proposed a high vacuum deoxygenated and water removal glove box control system. Through parameter setting, the system can automatically perform various glove box cleaning operations and quickly reach the micro-oxygen and micro-water concentration requirements. In addition, two sets of reaction tanks are built in the system, and the hardware pipeline switching design and monitoring software control are used to provide two sets of reaction tanks to execute the cycling cleaning and cycling regeneration operation procedures synchronously, which can effectively solve the problem of interruption of the experimental process, improve the efficiency of its cleaning operations, and greatly reduce the manpower and material costs of the glove box operation. In addition, the system can automatically record the relevant data during various operations for the analysis of glove box monitoring effectiveness.

Keywords: glove box, control system, cycling cleaning, cycling regeneration, monitoring software, system integration, performance analysis

1. Introduction

A glove box, also known as an anaerobic station, is a vacuum environment without any water (H_2O), oxygen (O_2), and organic gas, where high purity inert gas is filled, and the active substances are filtered out, which can prevent the external personnel from direct contact, so that the materials placed in the glove box can be stored or tested in the vacuum environment free of water, gas, and oxygen, and the material is operated by people in a safe and contactless manner [1]. The glove box is used in a wide range of applications such as the scientific research of chemical/chemical engineering/material/drug, and so on; the research and manufacturing of organic optoelectronic OLED/polymer PLED light emitting displays; the manufacturing and research of lithium ion batteries/lithium polymer batteries/solar cells/high capacity capacitors; the research and manufacturing of special (HID) light bulbs; metal welding or laser metal welding process; the preservation or research and manufacturing of noble sensitive drugs and nuclear energy research; and so on [2–4].

The glove box is mainly composed of a glove box body, a vacuum system, a gas circulation exchange system, and a control system [5]. The glove box body is provided with an antechamber (also called a transfer box) and an isolation glove. Usually a viewing window is arranged on the front of the body, so that the operator can clearly observe the operating conditions inside the box, and the operation process can be intuitively displayed in front of the operator. The glove box facilitates operators with isolation gloves in an anhydrous, oxygen free, and vacuum environment. Basically, the vacuum glove box is an important application of vacuum technology [6–9]. What degree of vacuum the glove box can reach not only depends on quite different manufacturing costs, but also affects the effectiveness of vacuum preservation of sensitive items, and vacuum level is also an important key to effectively remove moisture and oxygen in the box. Usually, a high efficiency glove box must be the high vacuum box (transfer box) [10–12].

For high vacuum deoxygenation and water removal glove box, the oxygen content and water content in the box must usually reach the ppm level (i.e., up to 10^{-4}%) of the micro-oxygen and micro-water concentration [13–15]. However, it is difficult to achieve effectively such micro-oxygen and micro-water concentration. Usually, the glove box must be vacuumed, and then the box is filled with an inert gas (such as nitrogen or helium), and this type of evacuation and nitrogen (or helium) filling operation procedure must be performed multiple times. Thus, the deconcentration of water and oxygen can be accelerated, and the concentration of water and oxygen in the ppm range can be achieved more efficiently. In addition, the micro-water analyzer and micro-oxygen analyzer that detect ppm levels of water and oxygen concentration also have a detection range of ppm (e.g., 0–1000 ppm). To ensure the normal use of such analyzers, the input of water and oxygen concentration values of this type of analyzer should also fall within its detection range to avoid damage to the analyzer. If water and oxygen of high concentration (e.g., percentage level) are input for a long time, these analyzers will be easily damaged and cannot be used any more.

On the other hand, the transfer box of the glove box system is mainly for the user to put the materials into the glove box body or take out the materials from the box body. However, when the glove box body has reached the micro-oxygen and micro-water concentration, the material should be placed through the transfer box. The material must be first put into the transfer box, and then the transfer box is sealed and evacuated to make the water and oxygen content similar to the glove box. But the transfer box in the vacuum state will suck, making it difficult to operate, so the inert gas in the glove box must be introduced into the transfer box so that the pressure is balanced, and then the isolation glove can be used to place the material into the box. On the contrary, if the material is to be taken out of the glove box and placed back in the transfer box (to be removed from the transfer box), the transfer box must be evacuated first, and then the inert gas must be introduced from the glove box to balance the pressure.

Although the glove boxes commonly used in various industries currently have a control system, they mainly monitor the vacuum system and the gas circulation exchange system through PLC in a semi-automatic manner [15, 16], and they must be operated by people by starting and setting the parameter of the vacuum system or gas cycling exchange system, so that the vacuum operation or gas cycle cleaning operations of such a glove box often need to wait for the completion of the parameter setting for the next stage, which tends to cause inconvenience in use and operation; on the other hand, to analyze the effectiveness of the vacuum operation and cleaning operation of the glove box system, it is necessary to automatically record the required measurement information for processing during each operation. However, no major glove box system is currently available with automatic recording of measurement data. The PLC control system is also very inconvenient for the instantaneous recording and processing of measurement data. Therefore, the existing glove box systems do not provide users with efficiency analysis function for vacuum operation and cleaning operation.

Furthermore, since the commonly used glove boxes are equipped with only one gas circulation exchange system, namely, a reaction tank capable of removing moisture and oxygen through the reagent [15, 16], when the chemical reagent in the reaction tank is used up, the operation must be stopped. After the cleaning reagent is replenished, replaced, or regenerated, the cleaning operation can then resume. The regeneration of the reaction tank is usually by burning a low concentration of hydrogen to restore the cleaning function of the reagent [16]. However, during the experiment or operation in the glove box, due to the slight leakage of the glove box or some gas generated by the experimental operation, the internal environment of the box will constantly change, so that the box must be continuously monitored and the cleaning operation must be performed when the concentration of water or oxygen exceeds the specified value [11, 17]. Therefore, when the glove box system is only configured with one reaction tank, except for the inconvenience of use and operation, it may easily lead to the problem of interruption of the experiment or the operation process, thereby affecting the quality and efficiency of the glove box. Although there are currently a small number of larger glove box systems that can be configured with two or more sets of reaction tanks [16], the configured redundant reaction tanks are usually only alternative replacement devices. When the reagent of the reaction tank is used up and the cleaning function is lost, it must be switched to other reaction tank to continue the cleaning work by manual operations. In addition, the operation of replacing or regenerating the reagent is performed on the reaction tanks that have lost the cleaning function. However, this operation method still cannot solve the problem of interruption in the experiment or operation process.

In view of the defect of experimental or operational interruptions in the use of the glove box, a cycling cleaning regeneration mechanism was proposed in this study [18, 19]. This mechanism is mainly to build two reaction tanks A and B. At the beginning, the reaction tank A performs the cleaning work, and the reaction tank B takes the rest. When the reaction tank A loses the cleaning function due to the use up of the reagent, it will automatically switch to the tank B to continue to work, and the regeneration operation of the tank A starts at the same time. When the tank B loses the cleaning function due to the use up of the reagent, it immediately switches to the operation of tank A and simultaneously performs the regeneration of tank B. Thus, the two reaction tanks are used alternately to perform the cycle cleaning and recycling operation, which can effectively solve the problem of interruption in the experiment or the operation process of the glove box system.

Based on this cycle cleaning regeneration mechanism, this study developed an advanced control system for high vacuum deoxygenation and water removal glove box. Through the design of switching by the hardware pipeline and control by the monitoring software, the system provides transfer box cleaning, glove box cleaning, vacuum preservation, end of preservation, cycling cleaning regeneration, and other basic operating functions on the one hand, and each operating function can be automatically executed through the setting of the control parameters and the instrument parameters; on the other hand, during the execution of each operating function, the system can automatically record all the measured data and elastically display the vacuum test curves and the cleaning test curves. This can provide the user with the analysis of the efficiency of cleaning operation and vacuum operation of the glove box. The results of this kind of performance analysis can be used as a quality parameter to determine the quality of the glove box and the basis for the user of the glove box to plan experiments or operations.

To verify the function of the deoxygenation and dehydration of the glove box system developed, a hydrogen storage and cleaning mechanism was built in the glove box of this study to serve as a specific application of the glove box system, and the application functions of hydrogen storage and hydrogen cell cleaning were provided in the monitoring software. This kind of design mainly considers that hydrogen is the main fuel for hydrogen vehicles in industry, and usually needs to be stored in a hydrogen storage cell. The hydrogen storage operation of the hydrogen storage cell must be in an oxygen free environment, including prior vacuumizing to remove the air in the hydrogen storage cell, then filling the hydrogen storage cell with hydrogen in an oxygen free environment, so as to avoid the danger of explosion due to the combination of hydrogen and oxygen. Therefore, we use this micro-oxygen and micro-water glove box as the operating environment of the hydrogen storage cell, providing operators with the ability to safely and smoothly perform the operation of evacuating the hydrogen storage cell and storing hydrogen for hydrogen storage purpose.

2. System design

The system architecture of the high vacuum deoxygenation and water removal glove box control system developed in this study is shown in **Figure 1**. In the figure, the solid blue line represents the gas flow path, the green dotted line represents the

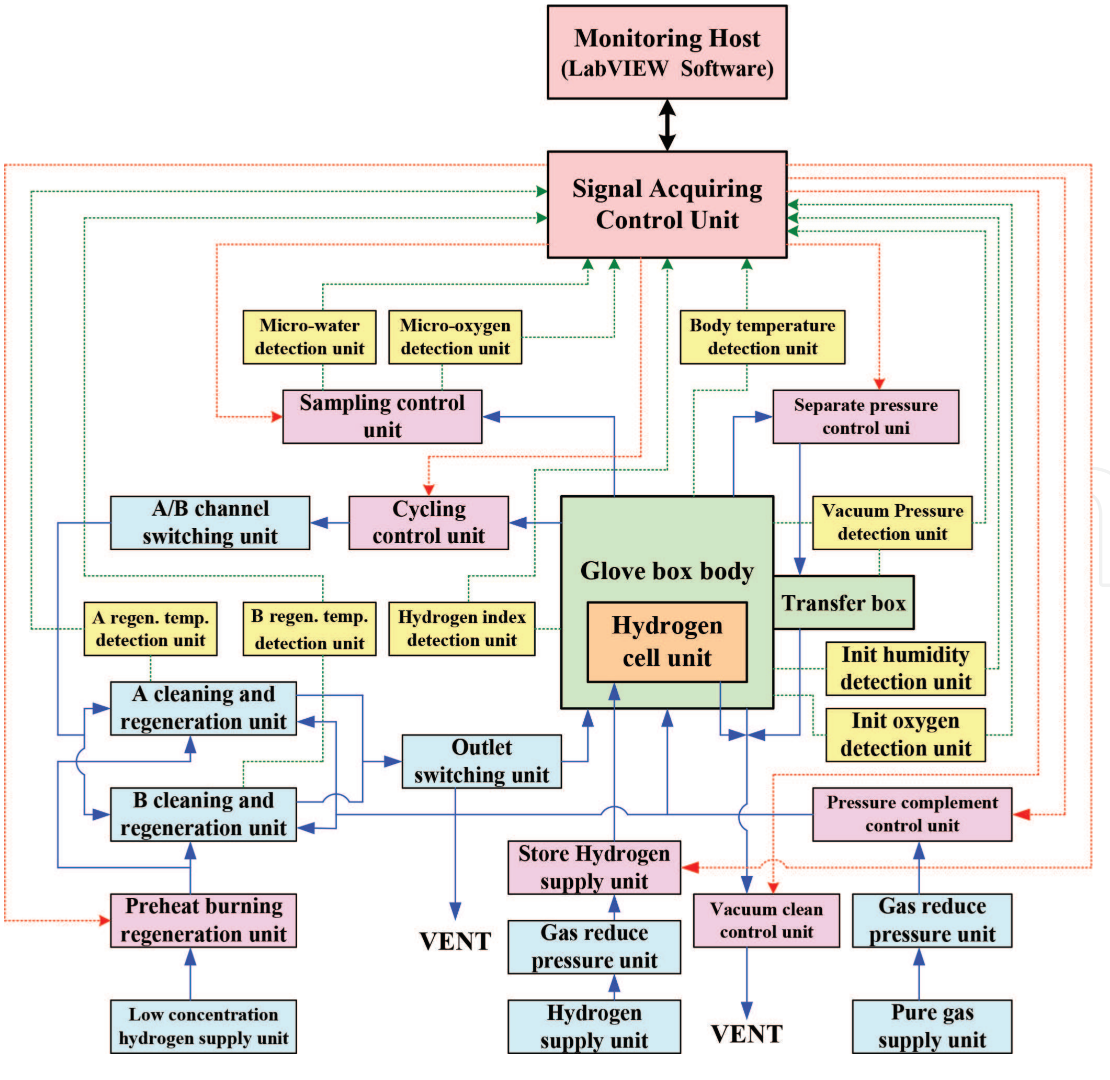


Figure 1.
Architecture of the glove box control system.

detection signal transmission path, and the red dotted line represents the control signal line.

The functions of each component unit in **Figure 1** are described as follows:

1. Glove box body: a box body of oxygen free and water free environment that provides a high vacuum for the user to perform material handling or storage. The body contains a set of hydrogen cell units that can store hydrogen or remove hydrogen.

2. Transfer box: it is used for users to put the material into the glove box or remove the material from the box.

3. Body temperature detection unit: the temperature sensor used to detect the temperature of the glove box body, and the detected temperature can be transmitted to the monitoring host.

4. Primary humidity detection unit: it can detect the humidity of the body in the atmospheric state, and it is a humidity sensor of the percentage range detection.

5. Primary oxygen detection unit: it can detect the oxygen concentration of the body in the atmospheric state, and it is an oxygen sensor of the percentage range detection.

6. Partial pressure control unit: it is a control module used to introduce the inert gas from the body into the transfer box to achieve a pressure balance.

7. Vacuum pressure detection unit: the pressure sensor used to detect the gas pressure (i.e., vacuum level) in the body, and the detected pressure can be transmitted to the monitoring host.

8. Pure gas unit: source of purified inert gas supply for high pressure nitrogen or helium.

9. Gas decompression unit: since the high pressure inert gas or high pressure hydrogen provided by the pure gas unit and the hydrogen supply unit are of very high pressure and cannot be directly used, the decompression unit must be used to decompress the high pressure gas before the gas enters the glove box system.

10. Gas supplement control unit: the control module to receive the command from the monitoring host, which is used to supplement the decompressed pure gas to the body or supply it to the regeneration reaction tank.

11. Vacuum removal control unit: the vacuum pump controlled by the monitoring host to remove the gas in the body or the transfer box and bring it to a vacuum state.

12. Micro-oxygen detection unit: a micro-oxygen detector that can detect ppm oxygen concentration. The detected oxygen concentration can be transmitted to the monitoring host.

13. Micro-water detection unit: a micro-water detector that can detect ppm level moisture concentration, and the detected water concentration can be transmitted to the monitoring host.

14. Sampling control unit: the sampling box controlled by the monitoring host may be used only for capturing gas in the body during the cycling cleaning operation for the micro-oxygen detector and the micro-water detector to detect the water and oxygen concentration so as to prevent such an expensive detector from being damaged by excessive concentrations of moisture or oxygen.

15. Cleaning regeneration unit: it consists of two reaction tanks that can carry out cleaning and regeneration functions. The cleaning function is to use the cleaning reagent to adsorb oxygen and water vapor that flows through the reaction tank. When the regeneration function is performed, low concentration hydrogen gas is introduced to regenerate its cleaning agent.

16. Cycle control unit: it can be controlled to perform the cycling cleaning and the cycling regeneration operations alternately of the two reaction tanks A and B.

17. A/B channel switching unit: it is used to switch the pipeline channel of reaction tanks A and B.

18. Preheated burning regeneration control unit: it can receive the command from the monitoring host for heating of the reaction tanks A or B and to remove oxygen and water vapor adsorbed on the chemicals by burning low concentration hydrogen to achieve regeneration.

19. Regeneration temperature detection unit: a temperature sensor that can be used to detect the heating temperature when the reaction tank performs a regeneration function. Each of the reaction tanks A and B has a temperature sensor. The detected heating temperature can be transmitted to the monitoring host.

20. Low concentration hydrogen supply unit: it provides 3% concentration of hydrogen and nitrogen mixed gas, and this low concentration of hydrogen can be burnt to activate the regeneration function of the cleaning reagent (catalyst) in the reaction tank.

21. Outlet switching unit: it is used to switch the flow path of the gas from the outlet of the reaction tank A or reaction tank B. When the reaction tank performs the cleaning operation, the outlet gas flows back to the glove box body, and the outlet gas is directly discharged when the regeneration operation is performed.

22. Hydrogen supply unit: the hydrogen source module for hydrogen supply.

23. Hydrogen storage control unit: the module unit is controlled by the monitoring host to store and release hydrogen into the hydrogen storage cell.

24. Hydrogen index detection unit: it is used to detect the hydrogen index in the box body and send it to the monitoring host. If the hydrogen index is too high, there is a danger of explosion. The monitoring host will stop the hydrogen storage operation.

DOI: http://dx.doi.org/10.5772/intechopen.80423

25. Signal acquiring control unit: the Advantech USB-4711A multi-function data acquisition card [20] is used as a signal capture unit to convert the analog and digital signals captured by each sensor into digital data, which are then transmitted to the monitoring host for processing. In addition, the Advantech USB-4750 digital output control card [21] is used as a signal control unit to transmit the control signals generated by the monitoring host to each control unit to control each valve switch.

26. Monitoring host: it is a personal computer that can execute LabVIEW monitoring software and configures a touch screen as the user interface for the user to operate the system. The LabVIEW monitoring software provides users with various manipulation functions, and at the same time, it can automatically record the relevant data of various operations, for drawing and flexible display of various test curves.

In the hardware design part, the P&ID (Process and Instrument Diagram) of the glove box control system is shown in **Figure 2**, where R-A and R-B represent the reaction tanks A and B, respectively; PI is the pressure indicator; PT, TT, HT,

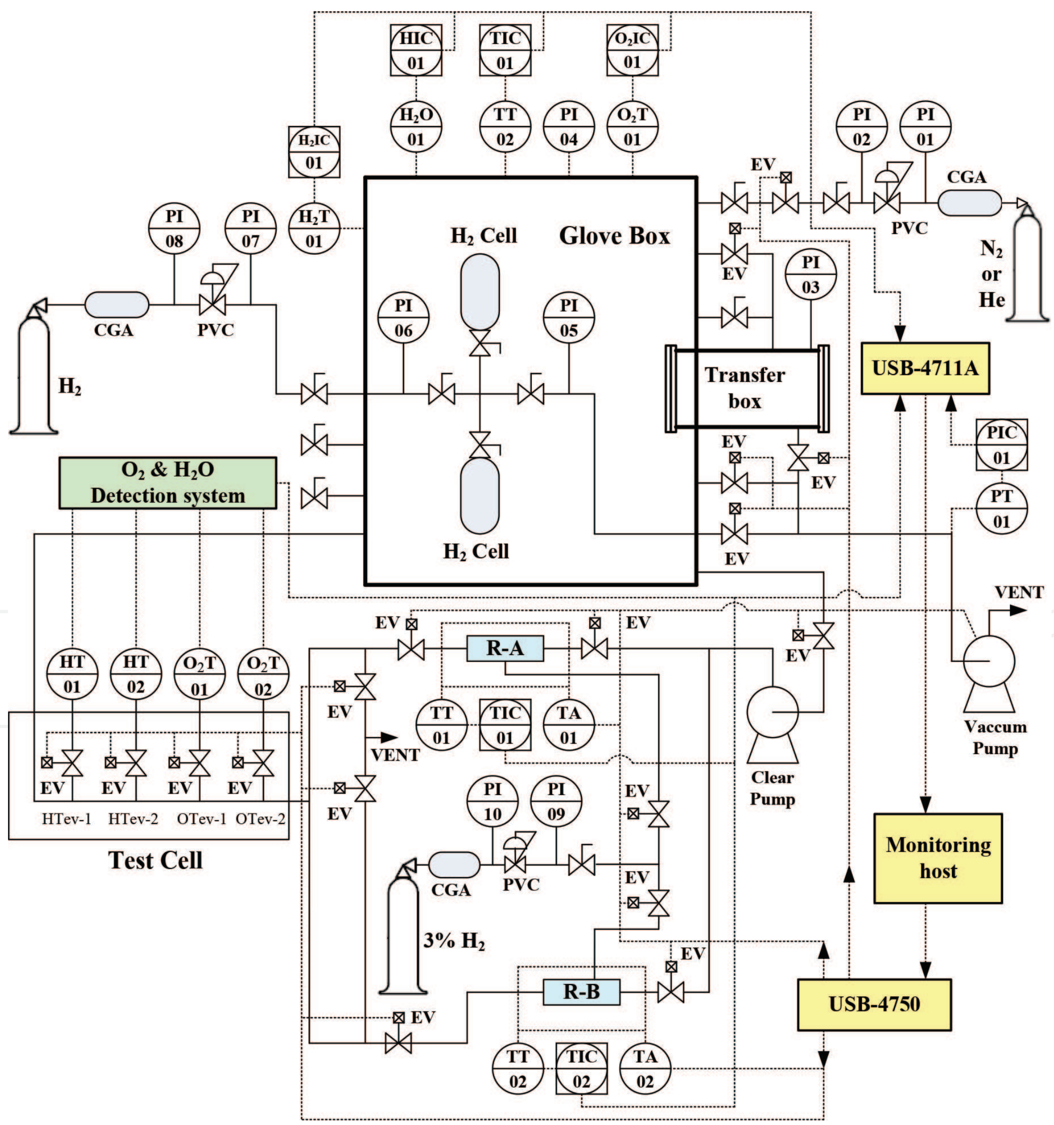


Figure 2.
Hardware design P&ID diagram.

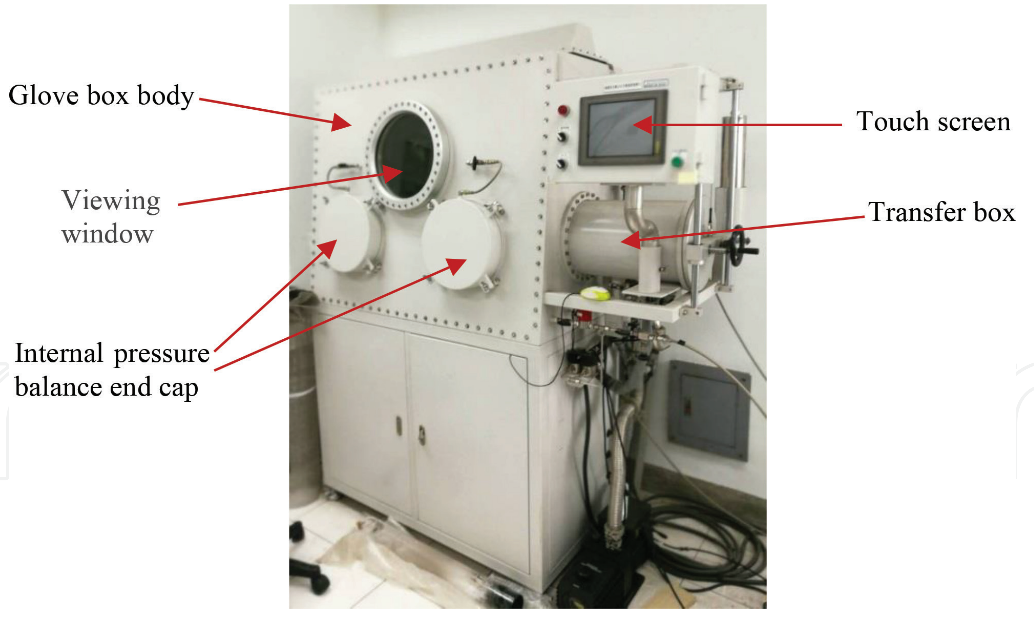


Figure 3.
Glove box control system entity.

O_2T, and H_2T indicate the detecting transducers of pressure, temperature, humidity, oxygen concentration, and hydrogen concentration; PIC, TIC, HIC, and H_2IC, respectively, indicate the indication controllers of pressure, temperature, humidity, and hydrogen concentration. All indication controllers inside the box can send signals to the USB-4711A adapter card and be processed by the monitoring host. TA and HA indicate the temperature and humidity actuators, respectively, and PVC indicates the pressure regulating valve. Each EV is an electric valve that can be controlled by the monitoring host via a USB-4750 adapter card.

To obtain high measurement quality of signals, such as temperature, pressure, water/oxygen concentration, and hydrogen concentration, etc., in terms of hardware, this study removed excessively large external signals through the masking technique and filtered the high frequency noises by virtue of short circuit filter capacitance. While in terms of software, this study employed the method of mean value to remove the influence of a small number of surges. For example, in a sampling period, the system captured 10 signals, calculated their mean, and used it as the measurement value, so that accurate and precise measurement signals can be obtained.

The glove box control system entity developed in this study is shown in **Figure 3**. We installed an internal pressure balanced end cap on the outside of the isolation gloves of the glove box to seal the isolation gloves inside the end cap. During vacuum operation of the box body, vacuum is applied together with the inside of the end cap to maintain the pressure balance. This avoids the pressure difference between the inside and the outside of the body, which may cause the isolation glove to inflate or even burst.

3. Software development

In this study, the automatic monitoring software for this glove box control system was developed using the LabVIEW graphical language [22–24]. The module hierarchy is shown in **Figure 4**. The monitoring software includes three major functional modules of parameter settings, program monitoring, and curve drawing. The parameter setting module can provide control parameter setting and instrument parameter setting functions. The program monitoring module can provide six

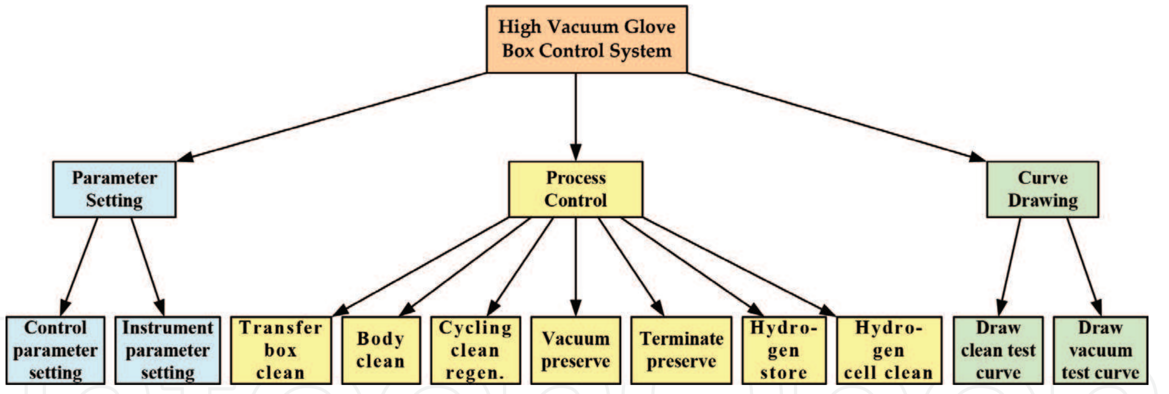


Figure 4.
The hierarchy of automatic monitoring software modules.

functions of monitoring, including transfer box cleaning, box body cleaning, cycling cleaning and regeneration, vacuum preservation, hydrogen storage tank cleaning, and hydrogen storage. And the curve drawing module can perform the function of drawing and displaying the cleaning test curve and the vacuum test curve.

Figure 5 shows the initial screen of the glove box automatic monitoring system designed by LabVIEW. Click the "Parameter Setting" button on the upper left of the screen to set the control parameters and instrument parameters. The seven green buttons on the right side of the screen are for the user to perform seven monitoring functions such as transfer box cleaning, box body cleaning, cycling cleaning and regeneration, vacuum preservation, end preservation, hydrogen storage tank cleaning, and hydrogen storage. Click the "Curve Drawing" button at the bottom left to draw and display the clear test curve and vacuum test curve.

3.1 Control parameter and instrument parameter setting

Before executing various control functions, the user can first set various control parameters and instrument parameters. The setting screen of control parameters is shown in **Figure 6(a)**. There are 16 control parameters that can be set in this screen. The control parameters set by users include the vacuum pressures of box body, transfer box and hydrogen cell, the set temperature of reaction tanks for cycling regeneration, the concentrations of water and oxygen set to stop cycling cleaning, and the times T1–T9 set to control various operation functions.

The instrument parameter setting can mainly provide the system designers to use the sensing instrument based on actual conditions to establish the software and hardware interface of the system, that is, the detection range of the sensor and the signal level relationship of the corresponding adapter card pin. The instrument parameter setting screen is as shown in **Figure 6(b)**. In the setting of instrument parameters, nine sensing ranges (for the regenerative temperature of reaction tanks A and B, the internal temperature of box body, the primary humidity and primary oxygen of box body, the concentrations of micro-water and micro-water, the vacuum pressure of vacuum pump, and the hydrogen explosion index) are given, and the corresponding signal levels of analog input pin AI0–AI8 are selected.

3.2 Real-time control process

After completing the control parameter setting, the user can perform various operation functions such as transfer box cleaning, box body cleaning, cycling cleaning regeneration, vacuum preservation, end of preservation, storing hydrogen and hydrogen cell cleaning, etc. The state transition diagram as shown in **Figure 7**

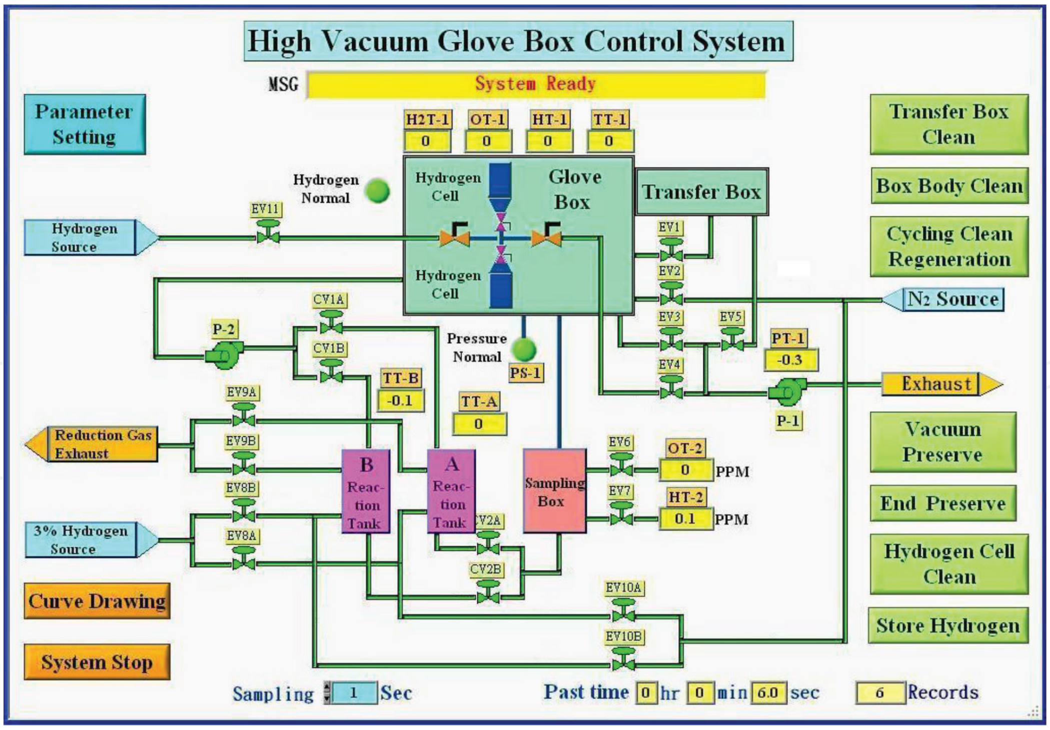
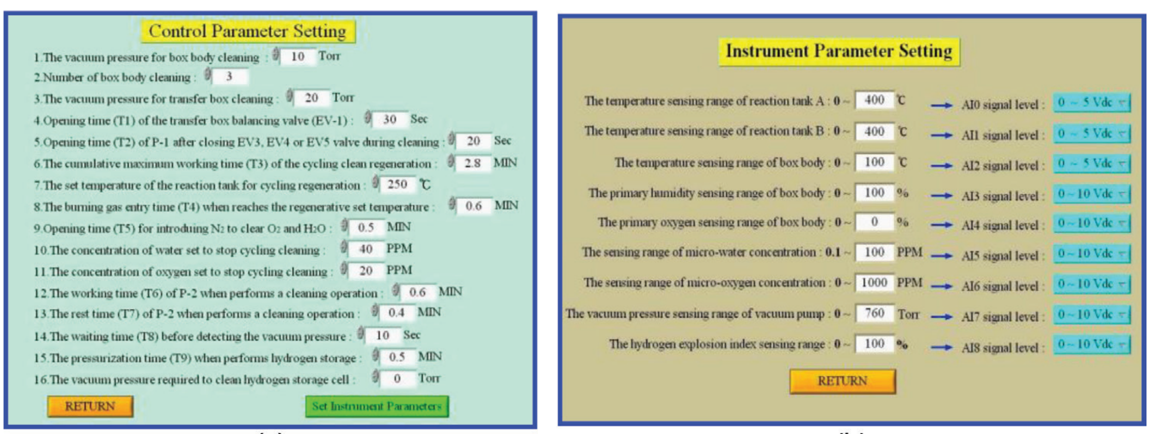


Figure 5.
Initial screen of the glove box automatic monitoring system.



(a) (b)

Figure 6.
Parameter setting screens. (a) Control parameter setting and (b) instrument parameter setting.

was used to represent the real-time control process for various operational functions of the glove box. The ellipses in the figure represent the states of the system, and the thick ellipses represent the final states. The double-circle ellipse represents the composite state, which is used to represent another state transition diagram. The line connecting the ellipses represents the transition of the state, and an event is attached next to each of the state transition connection line. This means that the transition of the system state is due to the occurrence of this event. In addition, a horizontal line can be added below the event. The action below the horizontal line is the action that accompanies the event, as shown below.

In the state transition diagram of **Figure 7**, when the system starts up, it enters a "Wait" state and enters the initial screen as shown in **Figure 5**, and the user can click "Transfer box clean", "Box body clean", "Cycling clean regen", "Vacuum preserve", "End preserve", "Hydrogen cell clean" or "Store Hydrogen" buttons to start the desired operation function. The system will enter the state corresponding

DOI: http://dx.doi.org/10.5772/intechopen.80423

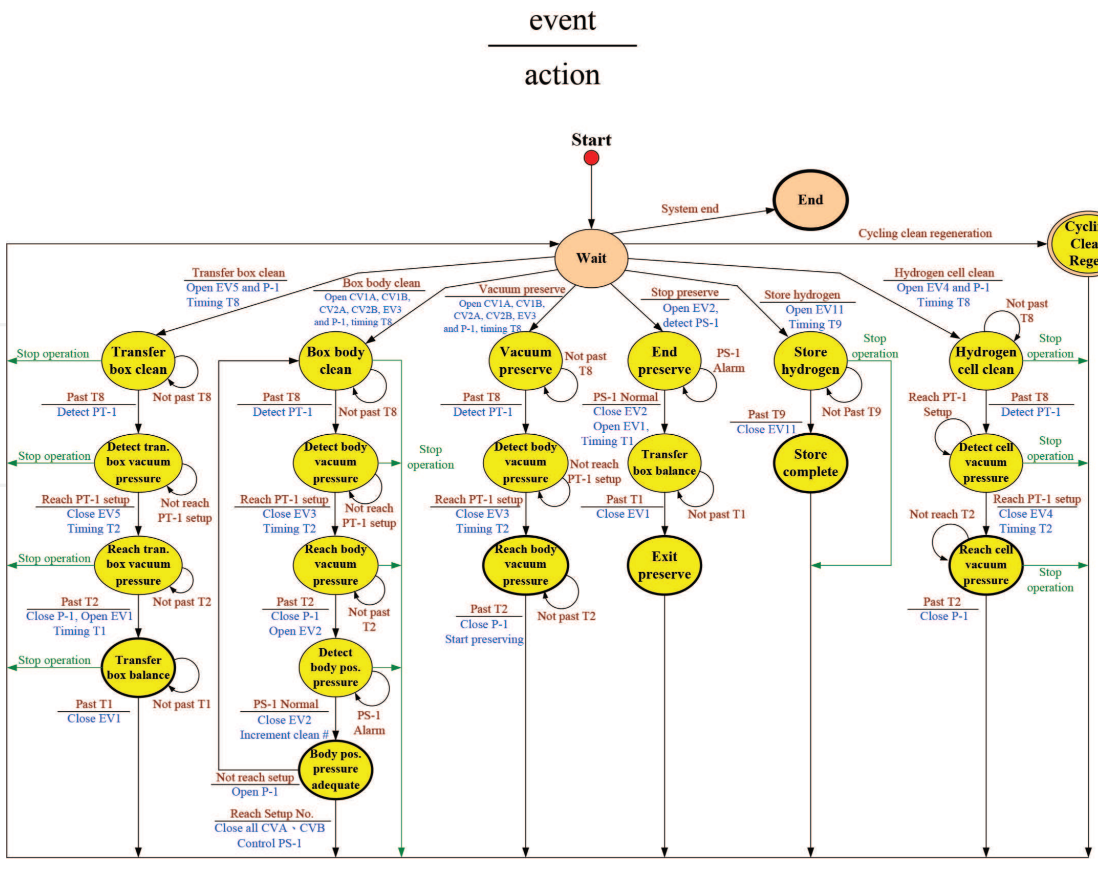
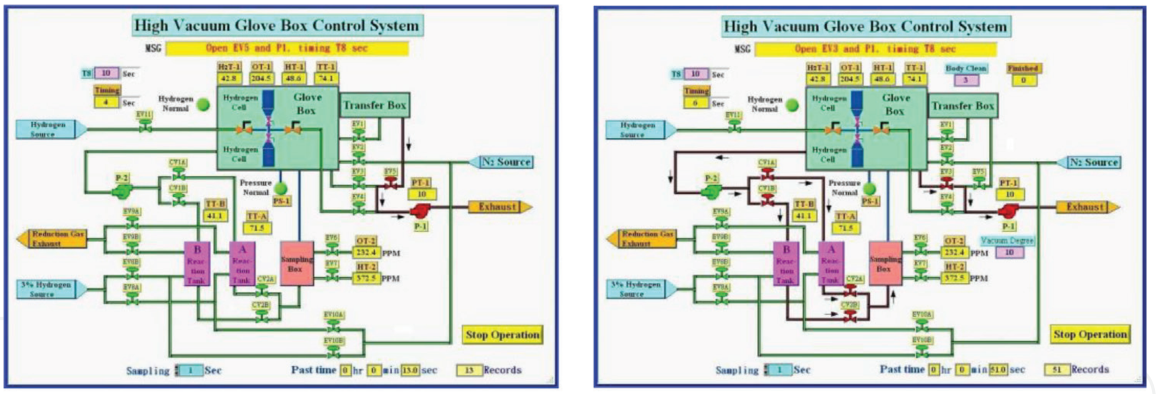


Figure 7.
State transition diagram of the glove box real-time control process.

to this operation function and start to execute its state transition diagram. The operating function of "Cycling clean regen" corresponds to a double-loop composite state. Entering this state will transfer to perform the "Cycling clean regen" as shown in **Figure 12**. After each state change graph of each operation function in the state transition diagram of **Figure 7** is executed, it will return to the "Wait" state for the user to select the next operation function, or the user may click the "System end" button to end the execution of this system.

3.3 Transfer box cleaning and box body cleaning operations

The transfer box cleaning operation removes moisture and oxygen from the transfer box of the glove box to a set vacuum level. When this operation is performed, the system first turns on the transfer box vacuum valve EV-5 and starts the vacuum pump P-1 to extract the air, and then enters the "*Transport box clean*" state, as shown in **Figure 8(a)**, which is the screen to execute the transfer box cleaning. In this screen, the red path represents the opening of the electric valve. Then it waits for T8 time, enters the state of "*Detect transfer box vacuum pressure*", starts measuring the vacuum pressure PT-1 (representing the transfer box pressure), and waits for the pressure to reach the set transfer box vacuum level (determined by the control parameters set in **Figure 6(a)**). When the vacuum pressure of the PT-1 is reached, it enters the "*Reach transfer box vacuum pressure*" state. At this time, the EV-5 shutoff action is started first, the T2 time is waited for, then the P-1 is closed, and then the "*Transfer box balance*" state is entered, this will open the transfer box balance valve EV-1 and time T1 will be counted, in order to introduce the inert gas in the box body into the transfer box, so that it can achieve pressure balance, thus completing the transfer box cleaning work.



(a) (b)

Figure 8.
Execution screen of transfer box cleaning and box body cleaning. (a) Transfer box cleaning procedure and (b) body cleaning procedure.

The box body cleaning operation is used to remove the moisture and oxygen of the glove box itself so that it reaches the set vacuum level. When this operation is performed, the system first opens the box vacuum valve EV-3 and activates the vacuum pump P-1, and simultaneously opens the inlet valves CV1A, CV2A, CV1B, and CV2B of the regeneration system, to eliminate the oxygen and moisture in the pipeline. Then it enters the "*Box body clean*" state, as shown in **Figure 8(b)**, which is the screen to perform the box body cleaning. Then it waits for the T8 time, enters the next state of "*Detect body vacuum pressure*", starts to measure the vacuum pressure PT-1 (representing the body pressure), and waits for the pressure to reach the set vacuum level of the box body. When the vacuum pressure of PT-1 is reached, it will enter the "*Reach body vacuum pressure*" state. At this time, the EV-3 shutdown will be started first, the time will be waited for T2, then the P-1 will be closed, then the "*Detect box positive pressure*" state is entered, the glove box air supply replenishment valve EV-2 will be started, and N_2 gas will be added. When the positive pressure of the box body detected is sufficient, the system will enter the state of "*Body positive pressure adequate*" and close the EV-2 to stop the replenishment of the N_2 gas source and completes the box body cleaning work. The system will check if the set number of body cleaning has been reached. If it has not, it will return to the "*Box body clean*" state and start the next round of box cleaning until the set number of box body cleaning has been completed. Then it will close the EV-3, CV1A, CV2A, CV1B, and CV2B valves and end the entire box body cleaning work.

3.4 Vacuum preservation and end preservation operations

The vacuum preservation operation is used to start the procedure for storing sensitive materials in the glove box. To perform a vacuum preservation operation, the user can activate the vacuum preservation control process after the material to be stored is placed in the glove box. The system first opens the box body vacuum valve EV-3 and activates the vacuum pump P-1 and simultaneously turns on the inlet valves of the regeneration system, CV1A, CV2A, CV1B, and CV2B to remove the oxygen and water in the pipeline, and then enters the "*Vacuum preserve*" state. The execution screen for vacuum preservation is similar to the body cleaning screen, as shown in **Figure 8(a)**, which is the screen to perform vacuum pumping. Then it waits for the T8 time, then enters the "*Detect body vacuum pressure*" state, and starts to measure the vacuum pressure PT-1 to wait for this pressure to reach the set box body vacuum level. Then it enters the "*Reach body vacuum pressure*" state. At this time, the EV-3 is turned off, and the T2 timing is started, when the vacuum

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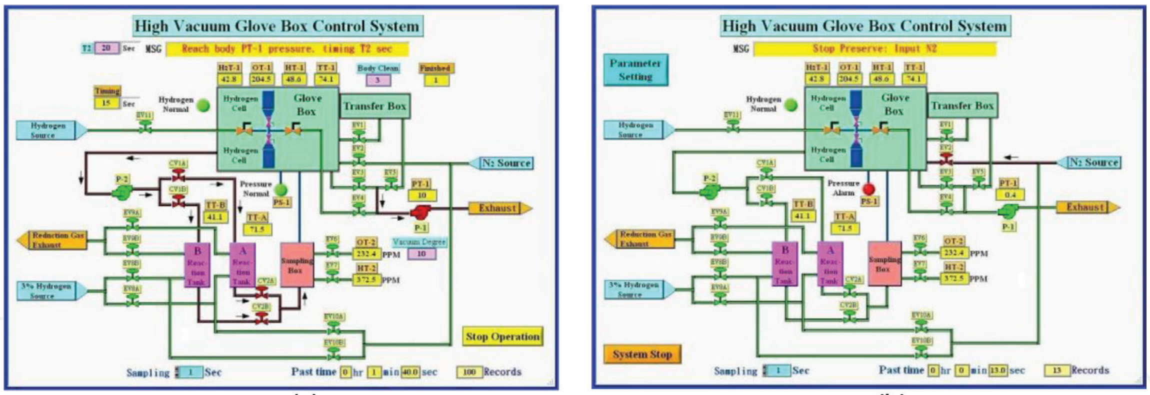


(a) (b)

Figure 9.
Execution screen for vacuum preservation and end preservation. (a) Closing the vacuum valve of the box body at the vacuum preservation procedure and (b) supplementing the box body with positive pressure at the end preservation procedure.

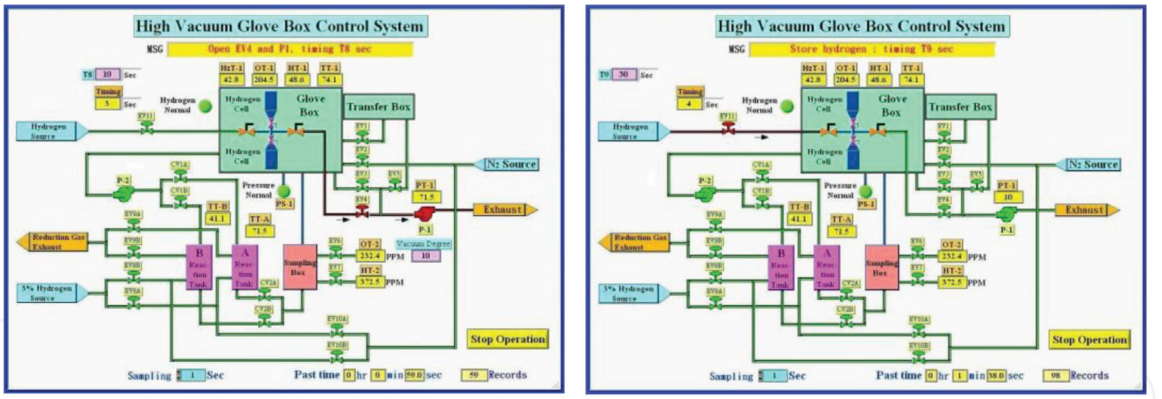
pump P-1 continues to run, waiting for the EV-3 to complete the closing action, as shown in **Figure 9(a)**, which is the screen where the EV-3 is turned off first, and the P-1 continues to run. When the T2 time expires, the system will close the valves of CV1A, CV2A, CV1B, and CV2B. At this time, the box body can maintain a vacuum environment and the material storage operation can begin.

The end preservation operation is used to start the procedure for ending the material storage. When the user finishes the preservation operation, the system will first enter the "*End preserve*" state, open the glove box gas source replenishment valve EV-2, and start to input the N_2 gas to supplement the pressure of the box body and detect the box positive pressure PS-1, as shown in **Figure 9(b)**, which is the screen to supplement the box body positive pressure. When the detected positive pressure in the box body is sufficient, the system will first turn off the EV-2, stop the replenishment of the N2 gas source, then enter the "*Transfer box balance*" state, and the transfer box balancing valve EV-1 will be turned on. The gas in the body is introduced into the transfer box to bring it into pressure balance, then the time is counted to perform T1 time, and then the system enters the "*Exit preserve*" state. At this point, the user can open the inside and outside doors of the transfer box to take out the stored material.

3.5 Hydrogen cell cleaning and hydrogen storage operations

The hydrogen cell cleaning operation is used to remove the gas in the hydrogen storage cell to reach the set vacuum level, so as to facilitate the subsequent operation of storing and releasing hydrogen. When this operation is performed, the system will first turn on the hydrogen cell vacuum valve EV-4 and start the vacuum pump P-1 to enter the "*Hydrogen cell clean*" state, and start the T8 timing, as shown in **Figure 10(a)**, which is the hydrogen cell cleaning screen. After waiting for the T8 timer to complete, the system will enter the "*Detect cell vacuum pressure*" state. At this time, the pressure PT-1 (representing the vacuum pressure of the hydrogen cell) will be measured. It is necessary to wait for the pressure to reach the set vacuum level. When the pressure of PT-1 reaches the set vacuum level, the system enters the "*Reach cell vacuum pressure*" state. At this time, the closing action of EV-4 is started first, and the time is waited for T2 time. When the time of T2 is reached, the hydrogen cell cleaning operation can be ended.

Hydrogen storage operation can store and release hydrogen in the hydrogen cell in the glove box. When this operation is performed, the system first enters the "*Store*



(a) (b)

Figure 10.
Execution screen for hydrogen storage tank cleaning and hydrogen storage. (a) Hydrogen cell cleaning procedure and (b) hydrogen storage procedure.

hydrogen" state, opens the hydrogen storage shut-off valve EV-11, and inputs hydrogen into the hydrogen cell of the glove box, as shown in **Figure 10(b)**, which is the initial screen for hydrogen storage. Then waits T9 time to finish the hydrogen storage operation and enters the "*Store complete*" state. In this process of hydrogen storage, if the hydrogen concentration is detected too high, the green "Hydrogen normal" light (as shown in **Figure 10(b)**) will be changed to flashing "Hydrogen alarm" red light, and the hydrogen storage operation will be immediately stopped.

4. Cycling cleaning regeneration control process

The glove box system contains two reaction tanks A and B. The cycling cleaning regeneration function can cycle between the two reaction tanks. When one reaction tank loses the cleaning capacity due to the run out of reagent, it can switch automatically to another reaction tank to work, and the reaction tank that losing the cleaning capacity performs the regeneration operation at the same time before joining the cycling work after they resume the cleaning capacity. In other words, this system can perform the cycle cleaning and cycle regeneration between reaction tanks A and B synchronously. The synchronization process is shown in **Figure 11**. In the figure, T3–T7 are the time parameters set by the user in **Figure 6(a)**, where T3 is the cumulative maximum working time of the reaction tanks (and the tanks can no longer work beyond this time), T4 is the time to regenerate the reagent by introducing 3% hydrogen, T5 is the time for the introduction of nitrogen to remove oxygen and water, T6 and T7 are the working time and the rest time, respectively, of the reaction tanks for each operation.

Figure 12 shows the state transition diagram of the glove box cycling cleaning regeneration control process. The dashed circles (states) and the arrow lines represent the parts that can be executed synchronously. Reaction tank A is preset to be the tank for the first time execution by the system, and the reaction tank B is in standby state.

The control system first enters the "*A works*" state, executes the cycling cleaning procedure of reaction tank A, it opens the cycling inlet valve CV1A of tank A, the cycling outlet valve CV2A, the oxygen sampling valve EV-6, the humidity sampling valve EV-7, etc., opens the clean pump P-2, and starts the timing of T3 and T6 at the same time, as shown in **Figure 13(a)**, which is the screen for tank A to perform the cleaning. At this point, the clean pump P-2 starts to send the oxygen and moisture in the glove box continuously to the reaction tank A for adsorption, so as to achieve

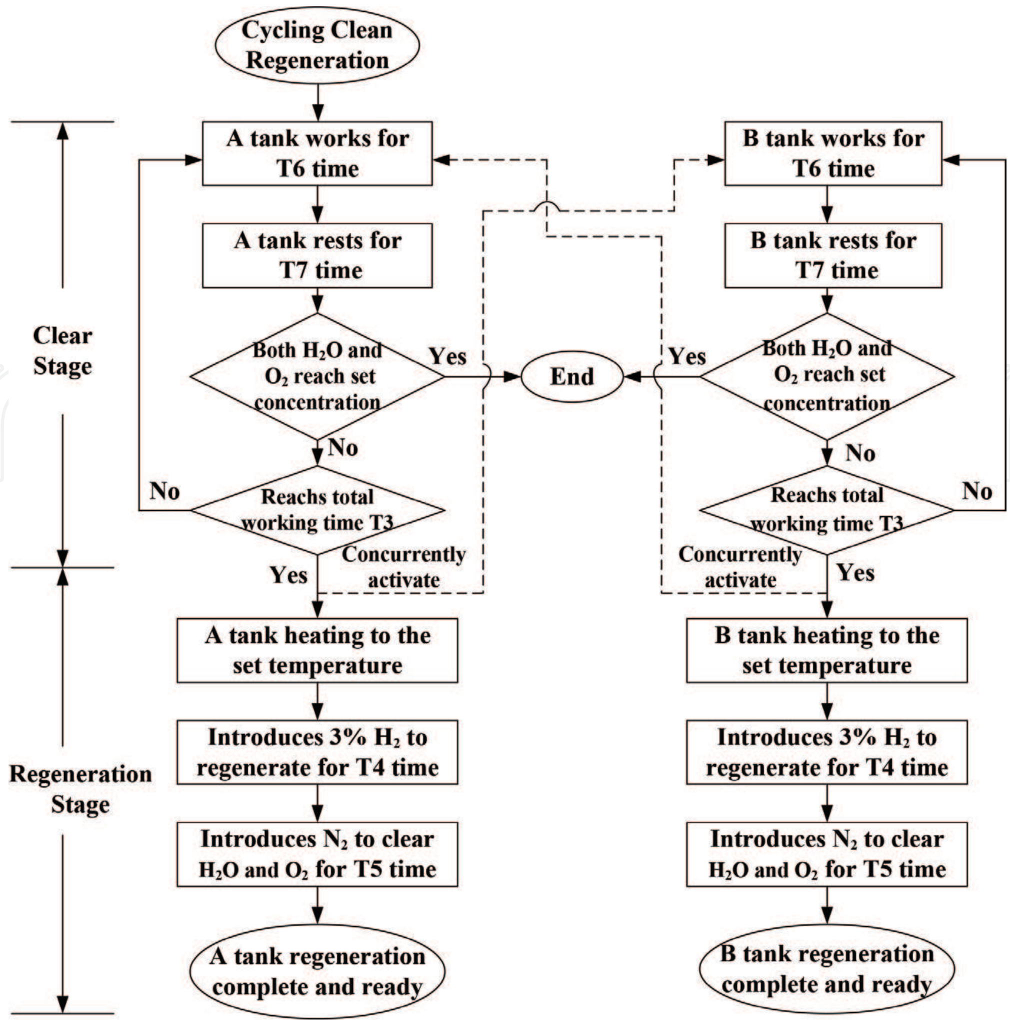


Figure 11.
Synchronization process flow of cycling cleaning regeneration.

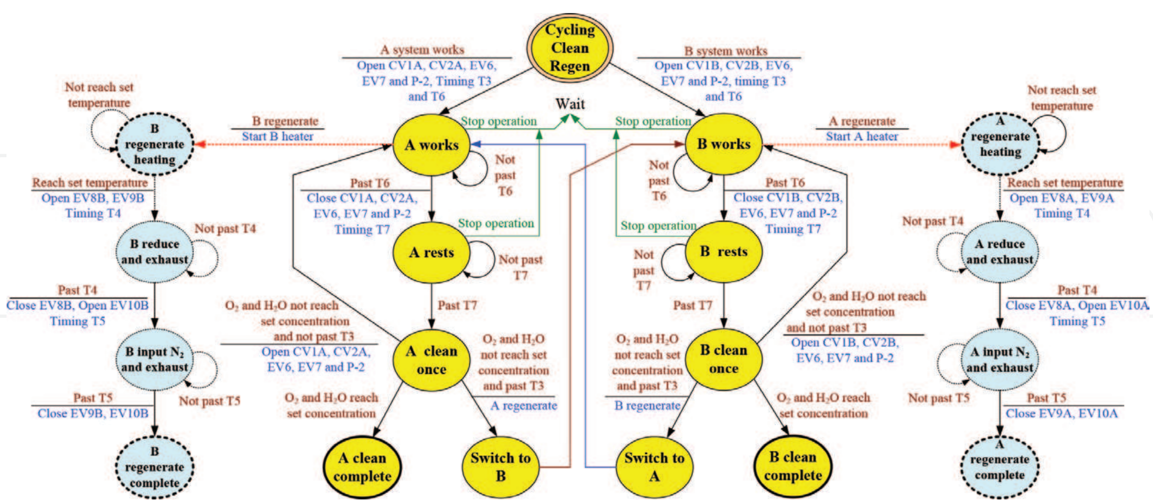
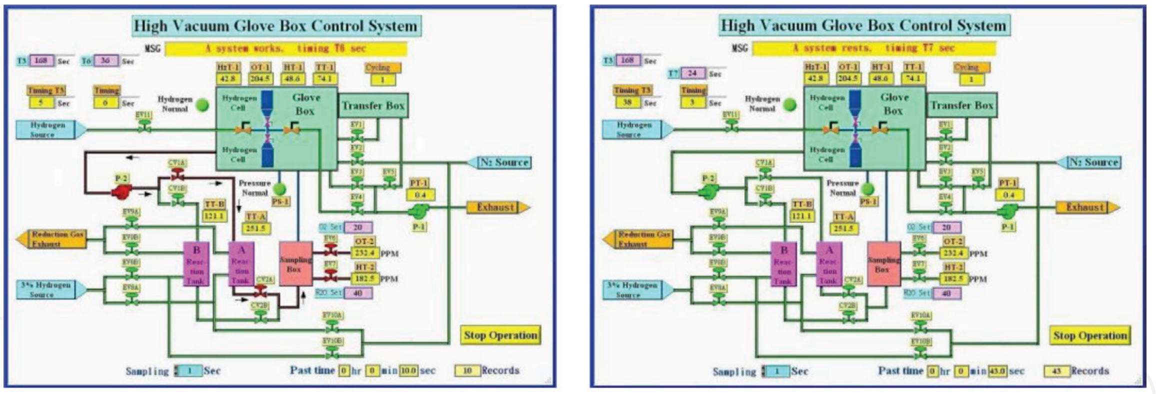


Figure 12.
State transition diagram of cycling cleaning regeneration control process.

the purpose of removing oxygen and moisture. When the T6 time is completed, the system enters the "*A rests*" state. At this time, the valves CV1A, CV2A, EV-6, EV-7, and the clean pump P-2 are all closed, and the T7 timing for rest starts, as shown in **Figure 13(b)**, which is the screen of the reaction tank A taking rest for the time of T7.

When the T7 timing is completed, the system will enter the status of "*A clean once*". At this time, the system will check whether both the detected oxygen



(a) (b)

Figure 13.
Screen for reaction tank A to perform cleaning operation. (a) Cleaning operation of reaction tank A and (b) reaction tank A taking rest for T7 time.

concentration OT-2 and the moisture concentration HT-2 have reached the set concentrations. If yes, the system will enter "*A clean complete*" state and automatically return to the "*Wait*" state of **Figure 6**. If OT-2 and HT-2 cannot reach the set concentrations at the same time, the system will check whether the cumulative working time T3 has been reached. If the T3 time has not yet been reached, it will return to the "*A works*" state and continue to perform the cycling procedure of tank A working first for T6 time and then resting for T7 time.

If T3 time has been reached and OT-2 and HT-2 have not yet reached the set concentrations, it means that the reaction tank A has lost the cleaning ability due to the run out of the reagent, and the system will enter the state of "*Switch to B*". This state will start the reaction tank A regeneration function first, and then move to the "*B works*" state for reaction tank B to perform the cleaning work, while tank A performs the regeneration process at the same time, as shown in **Figure 13(a)**, which is the screen for tank B to work and tank A in regeneration. When entering the "*B works*" state, the system will first open the CV1B, CV2B, EV-6, and EV-7 valves and the clean pump P-2 and start the timing of T6 and T3. Then it performs the cycling cleaning procedure of working for T6 time and then resting for T7. This control process is similar to the monitoring process when tank A is working.

When tank B performs the cleaning operation, tank A executes the regeneration process synchronously, and the state transition diagram thereof is shown in the rightmost blue dotted state area of **Figure 12**. Reaction tank A first enters the "*A regenerate heating*" state. The system controls the heaters to heat the reaction tank A [25–27], and detects whether the temperature TT-A has reached the set regeneration temperature. When the reaction tank A's temperature TT-A reaches the set temperature, the system will enter the "*A reduce and exhaust*" state. At this time, the 3% hydrogen inlet valve EV-8A and exhaust valve EV-9A will be turned on for 3% hydrogen to enter the reaction tank A to regenerate its cleaning agent, for the reduced waste gas to be discharged via the EV-9A valve, and the timing of T4 starts at the same time, as shown in **Figure 14(a)**, which is the screen for tank B to perform the cleaning work (which is now in the T7 rest phase) and for tank A to enter 3% hydrogen to regenerate its cleaning reagent.

When the T4 time expires, the system will enter the "*A inputs N_2 and exhaust*" state. At this time, the 3% hydrogen gas inlet valve EV-8A will be closed first, but the cleaning valve EV-10A will be opened to introduce dry N_2 gas in the gas source, so that the oxygen and moisture in the reaction tank A are taken away and discharged through the outlet valve EV-9A. And the timing of T5 starts, as shown in

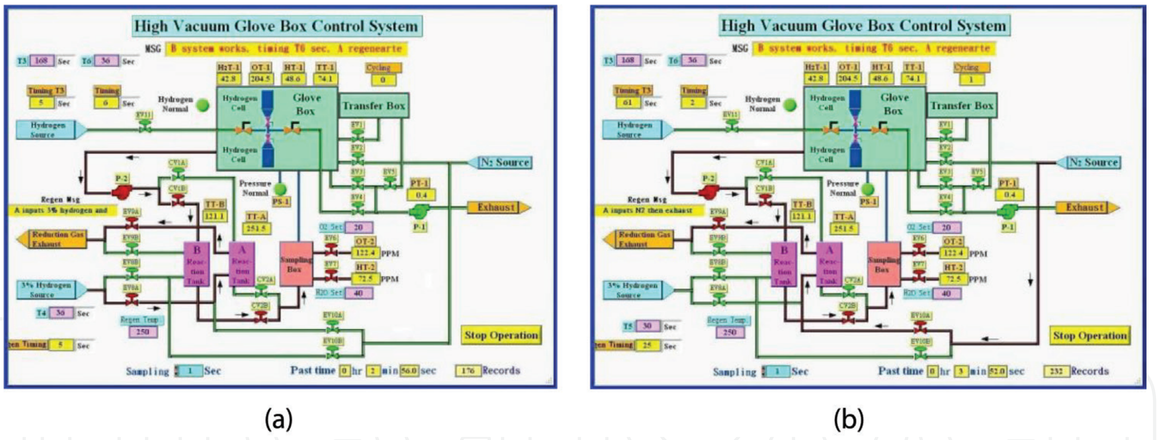


Figure 14.
Screen of tank B performing cleaning operation and tank A in regeneration. (a) Tank B resting for T7 and tank A entering 3% hydrogen to regenerate the reagent and (b) tank B operating and tank A introducing dry gas to take away oxygen and moisture.

Figure 14(b), which is the screen where tank B operates and tank A introduces dry gas to take away the oxygen and moisture.

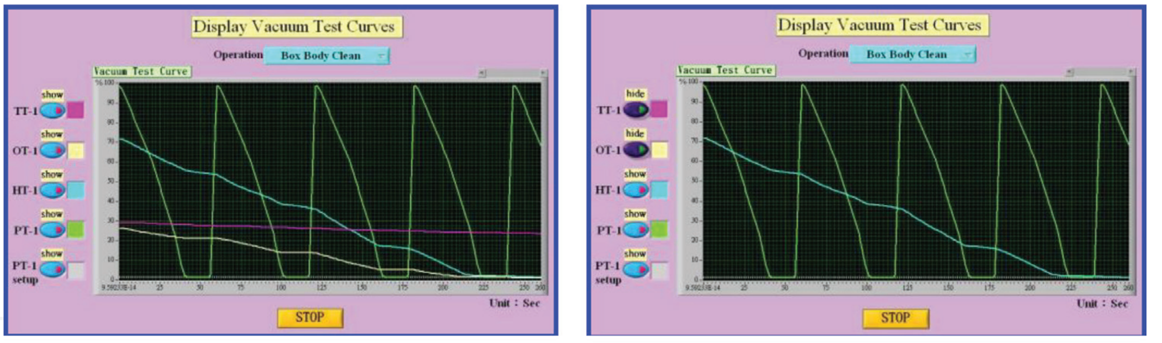
When the T5 time expires, the system enters the "*A regenerate complete*" state. At this time, EV-9A and EV-10A are turned off to end tank A regeneration operation. When tank B has several cycling of operation by working for T6 time and resting for T7 time several times and the detected oxygen and water concentration still fail to reach the set values, this means the cleaning agent has also been used up. At this time, as long as the T3 time has been reached, it will also start tank B regeneration and then enter the state of "*Switch to A*", to give the cleaning work back to tank A, and tank B will perform the regeneration work.

5. Curve rendering analysis

5.1 Vacuum test curve rendering analysis

The system automatically records the detected input signals for archiving during the execution of various glove box operation functions. The detected input signals are shown in the screen of **Figure 5**. By using the internal temperature TT-1, the primary humidity HT-1, the primary oxygen concentration OT-1, the vacuum pressure PT-1, and the PT-1 set value, the system can draw the vacuum test curves of the operation functions such as the transfer box cleaning, box body cleaning, and vacuum preservation, as shown in **Figure 15**, which is the elastic display screen of the vacuum test curve corresponding to the box body cleaning. **Figure 15(a)** shows the complete vacuum test curves. There are five switch buttons and five curve color setting boxes of "TT-1," "HT-1," "OT-1," "PT-1," and "PT-1 setup" on the left of the screen. Each switch button can be used to switch between "show/hide." Each color setting box enables the user to set the display color of the corresponding curve. At present, all five buttons are in the "show" state. Therefore, the above five test curves are displayed in the vacuum test graph in the middle of the screen. All the curves are normalized so that the display range is from 0 to 100%. From **Figure 15(a)**, we can see that the rate of decrease of water vapor concentration (72 → 2%) is higher than the rate of decline of oxygen concentration (26 → 2%).

The "show/hide" status of each switch button is set appropriately, allowing the user to analyze the relationship between various curves. If you want to analyze the vacuum pressure and the change of the primary water vapor concentration in



(a) (b)

Figure 15.
Display screen of vacuum test curves of box body cleaning. (a) Complete vacuum test curve and (b) change relationship between PT-1 and HT-1.

the box, you may click "TT-1" and "OT-1" switch buttons to set them to the "hide" status, and make the other three buttons "HT-1," "PT-1," and "PT-1 setup" maintain the "show" state, as shown in **Figure 15(b)**, which is the display of the relationship between the PT-1 and HT-1 curves. From this screen, it is observed that the green PT-1 pressure curve causes the significant drop of the blue HT-1 water vapor concentration during each descent (which represents the vacuumizing stage), and whether the PT-1 pressure curve can reach the gray PT-1 setup curve (i.e., reaching the setting value of the vacuum pressure) at each descent; and the pressure curve of PT-1 during the flat bottom is the nitrogen filling phase of the box body, when the change in the HT-1 water vapor concentration curve is less obvious. From the above analysis, we can see that the effect of the vacuumizing stage on the decline of water vapor concentration is obviously higher than that of the nitrogen filling stage.

5.2 Cleaning test curve rendering analysis

By using automatically recorded box temperature TT-1, regeneration temperature TT-A of the reaction tank A, regeneration temperature TT-B of the reaction tank B, the micro-water concentration HT-2 and micro-oxygen concentration OT-2 detected by the sampling tank, and the settings of HT-2 and OT-2, the system can draw the clean test curves of the cycling cleaning regeneration operation, as shown in **Figure 16**, which is the elastic display screen of the test curve of such cleaning. **Figure 16(a)** shows the complete cleaning test curves. There are eight switching buttons left side of the screen, such as "TT-1," "TT-A," "TT-B," "HT-2," "OT-2," "Regen Temp setup," "OT-2 setup," and "HT-2 setup" and eight corresponding curve color setting blocks. Each switching button can be used by the user to switch between show/hide, and each color setting box enables the user to set the display color of the corresponding curve. Currently, all eight buttons are in the "show" state, so the above eight test curves will be displayed in the vacuum test graph in the middle of the screen. The "show/hide" status of each switch button is set appropriately, allowing the user to analyze the relationship between various curve changes.

To analyze the relationship between the change of micro-oxygen concentration and the cycling cleaning regeneration phase, users can display five test curves such as TT-A, TT-B, OT-2, regeneration temperature setting, and OT-2 setting in this screen, as shown in **Figure 16(b)**. It can be seen from the figure that the reaction tank A performs cleaning for the T6 time (OT-2 curve drop) and rests for T7 time (OT-2 curve is flat). After two such cycles, the reaction tank B is immediately switched to perform such a cleaning rest cycle (the change in the OT-2 curve from continuing to fall till becoming flat), while the reaction tank A simultaneously

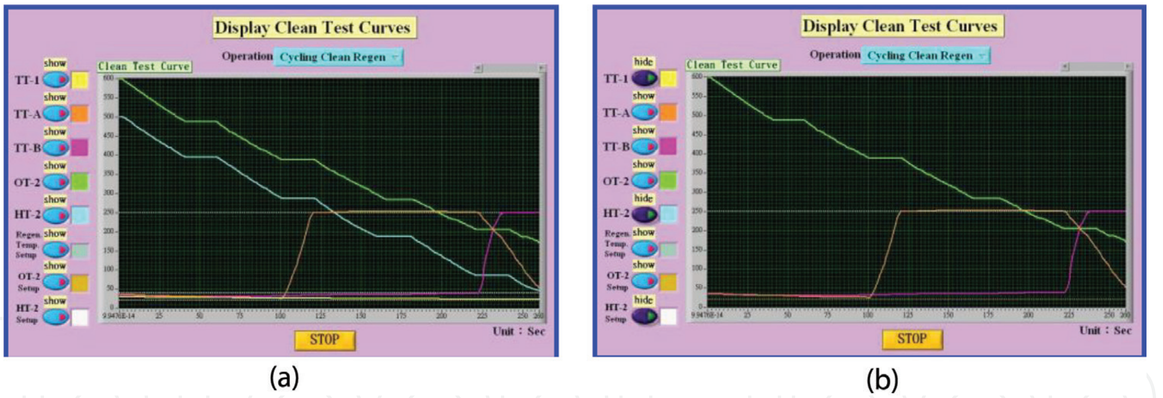


Figure 16.
Display screen of cycling cleaning test curves. (a) Complete test curve of the cycling cleaning regeneration operation and (b) relationship among TT-A, TT-B, and OT-2.

starts the regeneration operation, that is, it is heated to the regeneration temperature (the orange TT-A curve rises to the regeneration temperature setting), and then 3% hydrogen is entered to regenerate the cleaning reagent. After the reaction tank B performs two cycles of cleaning rest, it will switch back to the reaction tank A to continue the cycling cleaning operation, when the reaction tank B will start the regenerating operation synchronously, because the red TT-B curve will rise due to heating the regeneration temperature setting. From the above changes in the curves, it can be seen that the operation of removing the micro-oxygen from the glove box can be performed in turn by two reaction tanks, and no interruption occurs due to the run out of the reagent of a reaction tank. In addition, for the change of the micro-oxygen concentration, performing two cleaning cycles in the tank A can reduce the micro-oxygen concentration from 1000 ppm to about 390 ppm. And two cycling cleaning operations by the reaction tank B is followed, and the micro-oxygen concentration can be further reduced to about 210 ppm.

6. Conclusions

This article develops an advanced control system for high vacuum deoxygenation and water removal for the glove box. This system can use the switching of the hardware pipeline and the control of monitoring software to provide users with the transfer box cleaning, box body cleaning, vacuum preservation, end preservation, hydrogen storage, hydrogen cell cleaning and cycling cleaning regeneration operating functions, and each operating function can be automatically executed through the settings of control parameters and instrument parameters. This system combined sequential control with multi-conditional logic control. In the state transition diagram of control process, there are mainly the timing control (according to the timing parameters of T1, T2, … or T9) and multiple characteristic monitoring controls (such as temperature, vacuum pressure, oxygen concentration, water concentration, … and other control parameters). The system provides users to set the target values of parameters for the sake of multi-conditional monitoring and logic control. Hence, the system may decide the direction of execution when facing with various events. As a result, the control output of multi-conditional judgment may be highly tolerant and stable toward signal offset and change in other external conditions. Therefore, this control system may be high robustness.

From the control point of view, the cycling cleaning regeneration function provided by the system must be considered in terms of its control, including the comparison between the detection values of water and oxygen concentration and the set values, whether the cleaning capacity of the reaction tank during operation is already insufficient, whether the regeneration reaction tank has completed the regeneration time, and so on. The monitoring host can use these judgment results to simultaneously perform the cycling cleaning and cycling regeneration control. This kind of control mode is a double loop control mode, in which cleaning and regeneration are performed at the same time. On the other hand, during the execution of various operating functions, the system can automatically record all measured data and display the vacuum test curves and cleaning test curves flexibly. This can provide the user with the analysis of performance on the glove box cleaning operation and vacuum operation, and the analysis results can be used as a basis for determining the quality parameters of the glove box and can also be used as a basis for the glove box user to plan experiments or operating procedures.

Acknowledgements

This study was sponsored by the project of Ministry of Science and Technology, R.O.C., project number: MOST 106-2221-E-344-004.

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