

User Manual



Please read the manual before installation and operation.

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1.Introduction

X-ray Diffractometer (XRD) is analytical instrument which exploits x-ray diffraction principle to do analysis on substance's internal structure. DW-XRD-Y3000 adopts multi CPU system to control x-ray generator and goniometer and collect data. Equipping with high performance PC and software, it can precisely measure the crystal structure, lattice parameter, qualitative & quantitative analysis of the substances. When it is equipped with relative accessories, it can do structure and stress measurement. XRD is widely used in industry, agriculture, national defense and scientific research. It has features, including integration level, rich software and low failure rate, etc. These features improve the analysis speed, measurement accuracy. Using computer to control the fluctuation of x-ray generator's tube current & voltage and shutter's switching, this improve the XRD's automation level.

1.1 XRD Working Condition

Working temperature: 10~30℃ ;

Working humidity:30%~80%;

Power supply: Single-phase, 220~230V, 50 Hz. The range of voltage fluctuation must not exceed -12%~+10% of the rated voltage; the power capacity can't be lower than 8 kW

Grounding device: ground impedance can't be larger then 4Ω; The power supply circuit cannot be interfered by welding machine and high-frequency furnace, etc.

Cooling water: Water pressure: 1.8kg/cm²~2.3kg/cm², water flow-rate ≥3.5 L per minute; the water quality must be equal to drinking water; the water inlet temperature ≤30℃ and it cannot be lower than 0℃.

1.2 Main Technical Index

Output power: Tube voltage, tube current and output power must be set according to the selected load, the maximum design values are 60kV, 80mA, 3kW;

Stability: Power supply: 220V, circumstance temperature:20±2℃, warming up for 1 hour, stability between ±0.3% within 8 hours.

Tube voltage setting: 10~60kV; 1kV continuous setting;

Tube current setting: 5~80mA; 1mA continuous setting;

Protection function: have or no water, over current, over voltage and over power;

2θangle measuring precision:≤0.01°;

Goniometer repeatability: ≤0.003°

Angular accuracy: $\leq 0.01^\circ$

Angle setting speed: $100^\circ/\text{minute}$;

Scanning mode: continuous or step scanning;

Continuous scanning speed: $0.001^\circ\sim 0.127^\circ/\text{S}$ or $0.01^\circ\sim 1.27^\circ/\text{S}$;

Step width of step scanning: $0.001^\circ\sim 160^\circ$;

Mini stepping angle: 0.001° ;

Maximum counting rate: 50000cps/s ;

Complex stability: power supply is 220V, when the circumstance temperature is $20\pm 2^\circ\text{C}$, the stability $< 1\%$;

2. Installation

2.1 Installation condition

- (1)The installation site is well ventilated and free of dust;
- (2)The installation site is well ventilated, no dust, flat floor, no vibration around, no strong electromagnetic field;
- (3)Power supply and water;
- (4)The installation site area is $4.0\text{m} \times 3.0\text{m}$, and should be equipped with low noise air conditioning and dehumidifier;

2.2 Installation method and operation condition

Installation method and operation condition must be in accordance with 2.1;

2.2.1 Preparation before installation

After opening the box, clean the surface of the instrument, then check whether the screw and nut are loose, whether the components are damaged. The X-ray tube and the high voltage cable must be clean by alcohol, but the carbon tetrachloride cannot be used. After cleaning the contact part between the high voltage cable head and X-ray tube should be coated with silicone grease, the machine platform should be flat and firm.

2.2.2 Installation of x-ray tube

The water inlet and outlet of the cleaned x-ray tube and the water inlet and outlet of the pipe sleeve (the inlet is at the bottom left corner of the pipe sleeve) should be correspond. The line focus of the tube should be corresponding to the tube sleeve window, then put the sealing washer well and put the tube into the tube sleeve gently. Alternately tighten the two screws of the X-ray tube base. See whether there is water leakage phenomenon by water test. Check the correctness of the water connection.

2.2.3 Installation of high voltage transformer and cable

Remove the left door panel and push the transformer forward. Insert the cleaned cable into the transformer socket, then connect the high voltage cable with the X-ray tube and tighten the screw cap. Be careful in installation to avoid damaging the tube.

2.2.4 Installation of water pump

According to the requirements of cooling water pump, connect the water and check the leakage.

2.2.5 Cable connection

According to the overall connection diagram, connect the cable, including power cable, water pump cable, high-voltage transformer cable, etc., especially pay attention to the connection of grounding wire.

A. Connection of detector cable

Plug the rectangular plug into the socket of the cable plate; plug the round plug into the control power; the other end of the cable should be plug into the socket of the scintillation detector.

B. Connection of the interface cable

Connect RS232 interface 1 with the serial port 1 of the PC system, RS232 interface 2 should connect with the serial port of LGPLC x-ray generator control panel.

C. Connection of the goniometer

There are two stepping motors behind the goniometer. Connect the upper cable (2θ driving motor) to the socket of the cable panel of the 2θ control unit; the lower cable (θ driving motor) should connect to the socket of the θ cable panel.

3. Sample making method

Sample making and operating condition are the key characters to ensure the accuracy of the measuring result.

Natural or artificially synthesized Organic and inorganic material are all powder or solid, no matter what structure, XRD can measure them. The methods of sample making are not exactly same. Operator should choose method according to the actual situation. Whatever which method you use, it must meet the one precondition---since the sample is ready to the trail ended, the composition and the physicochemical property must be the same as the original sample. Operator must guarantee the sample's reliability.

3.1 Sample preparation

Although many solid samples are microcrystalline, more samples are rough and larger powder particle; big or small crystal grain crystallization texture; coarse grain which is recognizable easily. So we usually

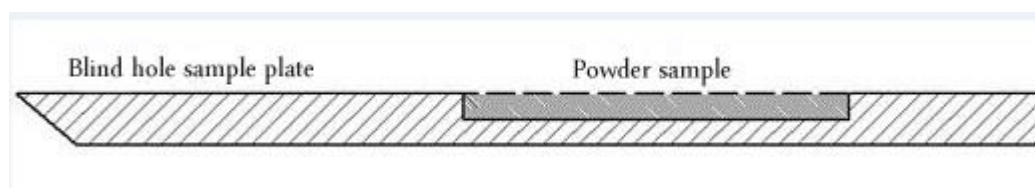
use grinding and sifting method to get very fine powder without any particles. Grind the sample in the mortar or ball-milling continuously < 360 meshes.

For some soft samples which are difficult to grinding (inorganic or organic), we can cool down the temperature of the sample by dry ice or liquefied air to make it brittle, then it is easy for grinding.

For some compound with different hardness and crystal habits, we should grind the sample fully and blend the powders completely to get the good sample.

3.2 Using blind hole sample plate to get powder sample

Powder sample without any granular sensation and prominent anisotropy, which is stability in the air, can use compression method. First, put the blind sample plate on the flat glass plane; sprinkle the powder over the sample plate's window hole. Second, use glass slide to chop the sample tight to make sure the sample is spread out evenly. Third, use the glass slide to press the powder sample tight and scrape the redundant powder by glass slide to make the surface of the powder sample in the window hole and the sample plate surface on the absolutely sample plane. Forth, carefully carry up the sample plate from the glass plane, now we will get a standard powder sample for XRD. See profile below:



3.3 Using through-hole sample plate to get powder sample

First, put the aluminum through-hole sample plate on to a smooth and clean glass plain, and use the sample spoon to sprinkle the powder evenly into the window hole of the sample plate. Second, use the glass slide to press the powder sample tight and make powder evenly spread out in the window hole. Make the surface of the powder sample and the sample plate surface basically the same. Third, use small filter paper to cover the sample, and use the left hand to press the sample plate to prevent moving, and use the right hand to press the powder sample under the filter paper hardly, especially the sample in the four angles of the window hole. Forth, when it is pressed tightly, move the filter paper and scrape the redundant powder by glass slide. Fifth, carefully carry up the sample plate from the glass plane. The sample surface is just the surface which contact with the glass plane, and it is just a sample's diffraction surface. See profile below:



3.4 Using glass slide to get powder sample

Powder sample also can get by glass slide according to the sample's physicochemical property. First, use the diluents (ethyl alcohol, acetone and distilled water) to dilute and shake. Second, use sucker to equably drop sample liquid on the glass slide. Third, dry it to get a standard x-ray diffraction sample.

When we use glass slide method, the thickness of the sample on the glass slide must be $>0.1\text{mm}$, so that the incident ray cannot shoot on the glass slide.

For example, we can get the sample of NaCl powder from its saturated solution, the measurement will be very excellent. See constructed profile below:

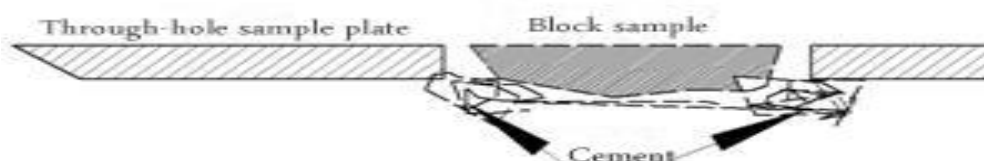


3.5 block sample making (Metal and alloy)

For some block sample such as metal and alloy, first, use at least 1200 abrasive paper to grind it on a plane. The make sure the level surface is no more than 15*15mm. Second, clean the level surface by alcohol.

First, place the aluminum through-hole sample plate on the flat and clean glass plate, and place the ground block sample plane downward in the window hole of the sample plate. Second, use the cement which is offered along with the XRD to fasten the block sample and the sample plate together. The usage amount of cement is depends on the weight of the sample. In a word, make sure the sample and the sample plate are steady and firm together.

Please see the construction profile below:



Texture usually exists in rocks, metal and alloy blocks. To get reliable measuring result, it should be ground or filed into fine particles. Use fine plastic file to get fine powder of metal. The file should be cleaned with a small file length and light strength to avoid sample heating. The obtained filings should also be treated with annealing to eliminate the dot matrix stress caused by the cold processing of the file cutting process.

In this way, the rock or metal powder is prepared by using the blind-hole sample plate, which is consistent with the method of making the powder diffraction sample by using the blind-hole sample plate.

4. Starting operation

The starting operation of the XRD is powered up to three units respectively, which are computer, circulating water cooling device and the main engine of the XRD.

The circulating water device is connected to the power supply. After the circulating water device is started, the "water protection" indicator on the front panel of the main engine of the XRD is turned on, indicating that the circulating water cooling device is working normally.

If the "water cycle" indicator is not on, it indicates that the X ray tube is blocked and the X ray tube is cleaned.

Turn off the circulating water device and cut off the power supply of the circulating water device.

The power switch of the main engine of the XRD is ON the right side of the front door of the main engine of the XRD. The power switch can be switched ON by pulling ON "ON", and the power supply of the main engine can be cut OFF by pulling ON "OFF".

5. Sample measurement

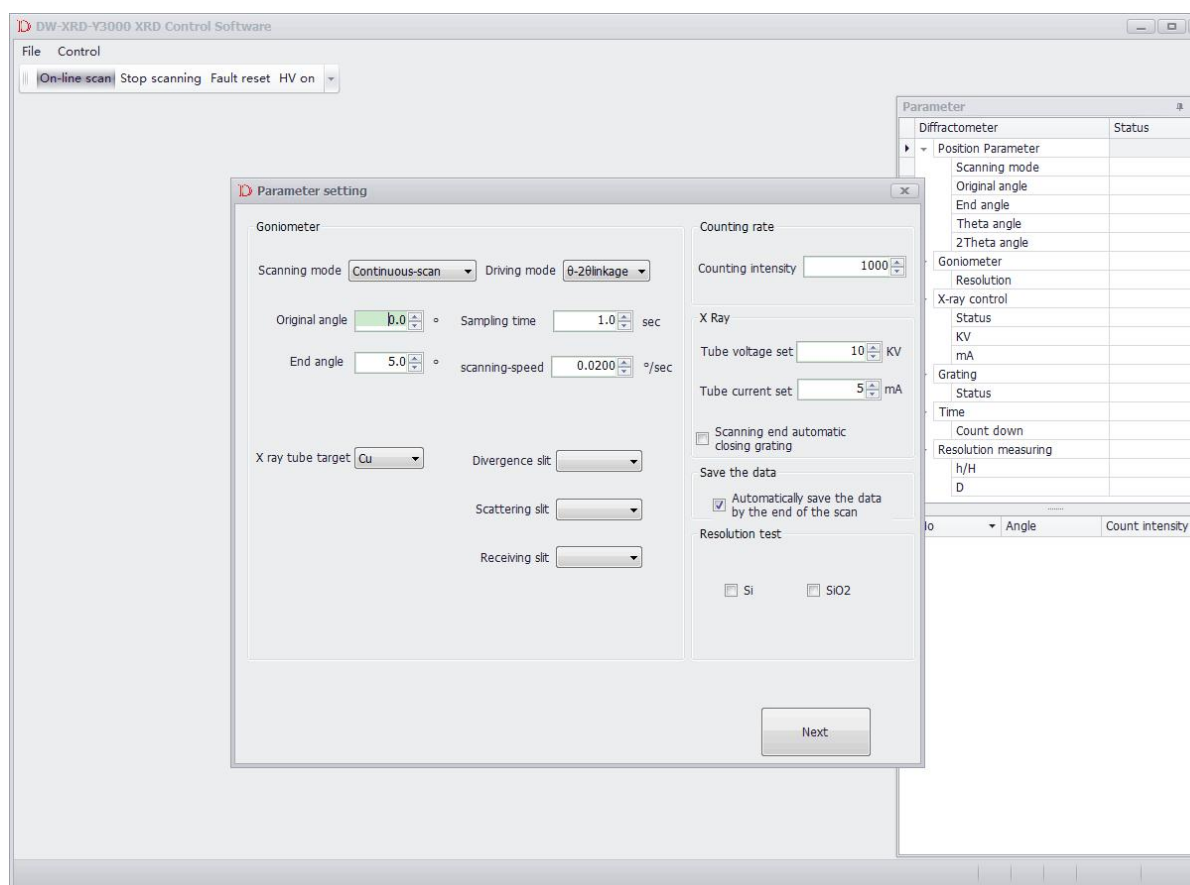
All kinds of control and parameter setting of the XRD are that the computer sends corresponding instructions to the XRD host, and the control unit of the host completes various corresponding action control and data measurement, and sends the measurement results back to the computer, forming a complete diffraction data file stored in the designated location of the computer hard disk. According to the analysis purpose, the measured data are processed by the diffraction data processing software.

The diffraction sample to be measured is prepared, inserted into the sample table of the Angle meter of the XRD, and closed the radiation protection door.

Objective: to carry out routine analysis of the materials, including qualitative and quantitative analysis, crystal cell parameters, grain size measurement, full-spectrum image fitting and fine repair crystal defects, etc.

Instrument conditions: divergence slit 1 °, 1 ° scattering slit, receiving slit 0.3 mm or 0.2 mm; Before starting to measure the sample, reset various control parameters in the data display area according to the sample test requirements:

Select the "online scan" button and enter the parameter setting interface



Driving mode: θ s- θ d、 θ s、 θ d; ----- Usually select θ s- θ d mode;

Original angle: xx.xx ----- Optional range 0°~160°;

End angle: xx.xx ----- Optional range 0°~160°

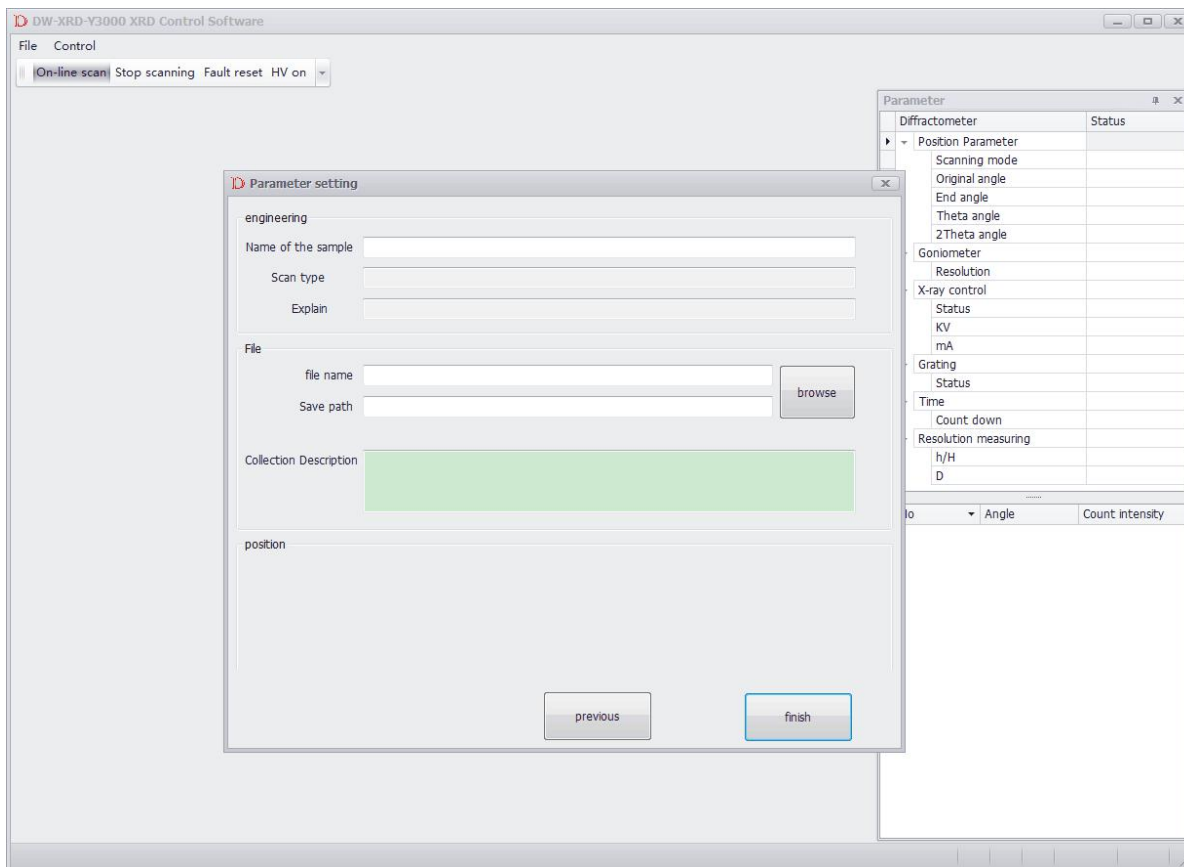
Step width angle: 0.xx ----- Usually select 0.01~0.05, recommended value 0.02

Sampling time: xx.x ----- Usually select 0.1~10, recommended value 0.5、1、2、3

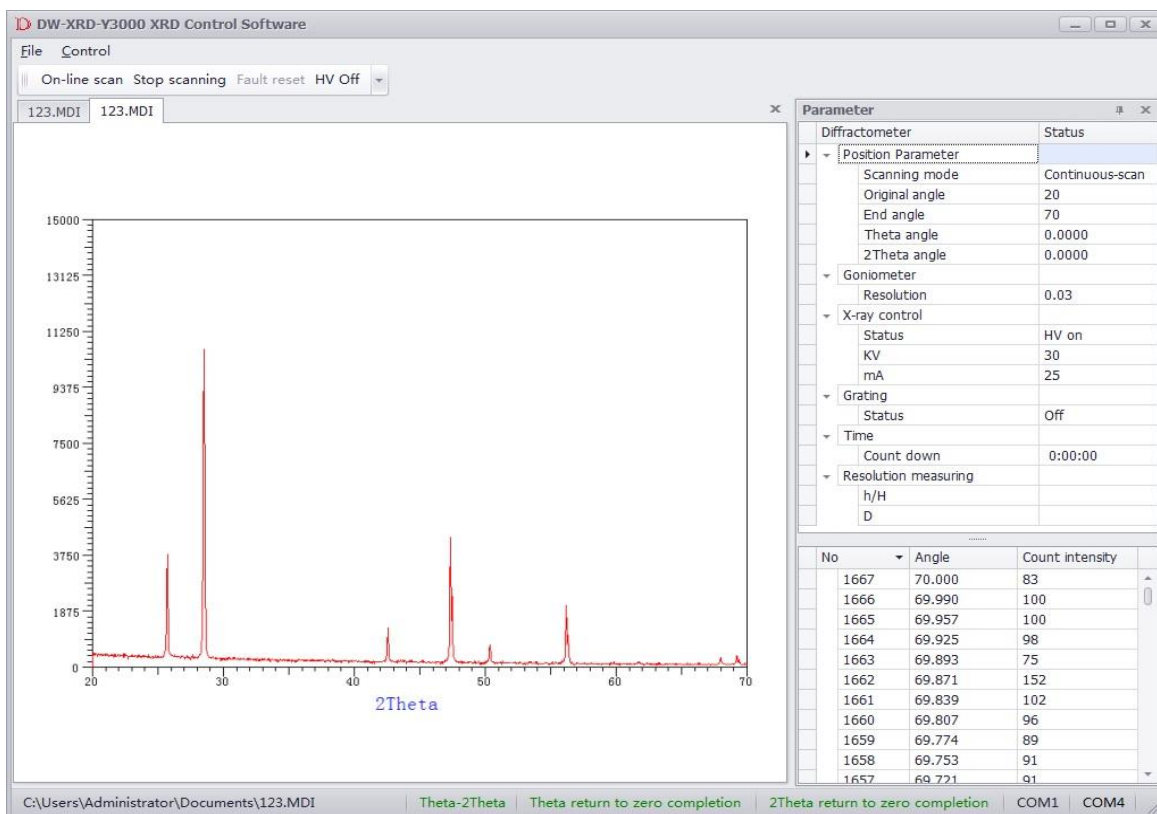
Tube voltage: xx ----- Range 10kV~60kV, recommended value 40(depends on the target setting)

Tube Current: xx ----- Range 5mA~80mA , recommended value 40;

When all parameters are set, click "finish".



In the file name option, name the sample file, click "browse" button again, select the location where the file is saved, click "finish", and the XRD begins to measure the sample.



Sample measurement interface is as above, including: measurement data drawing area, tube voltage and current indicating area, parameter setting data display area, control command area.

Used to display the measured data plot: sample measurement result, abscissa value (unit °) for the diffraction Angle, y coordinate for the diffraction intensity value units (CPS);

Tube voltage and current indicator area: used for real-time display of the tube voltage and current value of the XRD;

Note: when determining the measurement range of the Angle of the sample, 0.02 can be selected for the step width Angle, 0.1 can be selected for the measurement time, and the starting and ending angles can be determined according to the diffraction spectrum.

For qualitative analysis of general samples, 0.02 is selected for step width Angle and 1 is selected for measurement time. For the quantitative analysis of general samples, 0.02 is selected for the step width Angle, and 2-5 is suitable for the measurement time.

Rule for naming sample filename: file name + sequence number of sample. For example, the sample file name is SSAA, and the 5 samples are measured. The file names of the stored diffraction data are ssaa-1, ssaa-2, ssaa-3, ssaa-4 and ssaa-5.

6. Operation and fault diagnosis

6.1 Operation

For XRD –7000 X ray diffractometer, its x-ray generator, shutter and goniometer are all controlled by computer, so the operation is very simple, the operator should to input command in the keyboard or select the corresponding parameters in the parameter list to complete sample measurement, generally operate according to the following steps:

6.1.1 The sample preparation is the most basic operation of the sample measurement. The quality of the sample determines the accuracy of the measurement results. The sample preparation is generally as follows:

- A. When measuring the powder sample, the particle of the sample should not be less than 400 meshes;
- B. The surface of the sample must be flat with the sample plate, and the surface of the sample should be smooth and the level and density should be same.

- C. When measuring the block sample, the surface of the sample should be polished, and the plane of the sample should be in the same plane as the plane of the orifice sample plate.

6.1.2 When installing the sample, gently insert it and gently take it, this can avoid to vibrating and dropping the sample on the test bench.

6.1.3 Always close the hood and outer cover of the protective cover to prevent the radiation from scattering.

6.1.4 Connect the power of host and microcomputer system and guide the DW-XRD-Y3000 system control software.

6.1.5 Select operation 2, add the parameter table, press F1 key to open water pump, high pressure, and then start to measure. After the measurement is completed, the high pressure will be automatically closed. The operator needs to turn off the water pump after 20 minutes of cooling.

6.1.6 After data processing of the measurement results according to the DW-XRD-Y3000 system, the measurement results are output by printer or plotter.

6.2 Fault diagnosis

Once the failure occurs, the following methods can be excluded according to the phenomenon: XRD in use needs maintenance. Generally, inspection and cleaning must be done during 1 ~ 3 months.

6.2.1 After you power on the main power switch, the power light on the main control panel is not illuminated: the cooling water pipe and water tank of the water filter must be checked and cleaned every 1 year, prevent the incrustation scale to reduce the cooling effect. The input power is faulty or the power fuse F1 is damaged.

6.2.2 High voltage can't be open and high voltage indicator light can't be illuminated, there are several reasons;

- A .The lock is locked;

- B. The water relay is not closed, the water pump does not turn or the x-ray tube is clogged with impurities, and the water flow is insufficient;
- C. The control board has problems with the communication interface of 1810

6.2.3 The shutter can't be open;

The indicator light in the shutter box or the K1 and K2 relay on the control board are damaged;

6.2.4 When the high voltage is open, the kV meter was indicated, but the mA meter did not indicate: the fuse F2 in the control box damaged or bad contact between the high voltage cable in the control brake and the x-ray tube;

6.2.5 kV meter and mA meter are not stable when the high voltage is opening: there is large capacity of power equipment working in the same power supply;

6.2.6 After guiding the DW-XRD-Y3000 system, the microcomputer and the high voltage control unit (LG PLC) and the goniometer PLC can't be connected: computer serial port COM1 or LG PLC interface COM4 has problems, or communication cable is not connected;

6.2.7 The sample measurement angle is inaccurate and the diffraction intensity is low.

The goniometer 's beam path adjustment is not accurate, the detector is not set right and the upper and lower limit parameters are not correct.

6.2.8 The sample measurement is not counted:

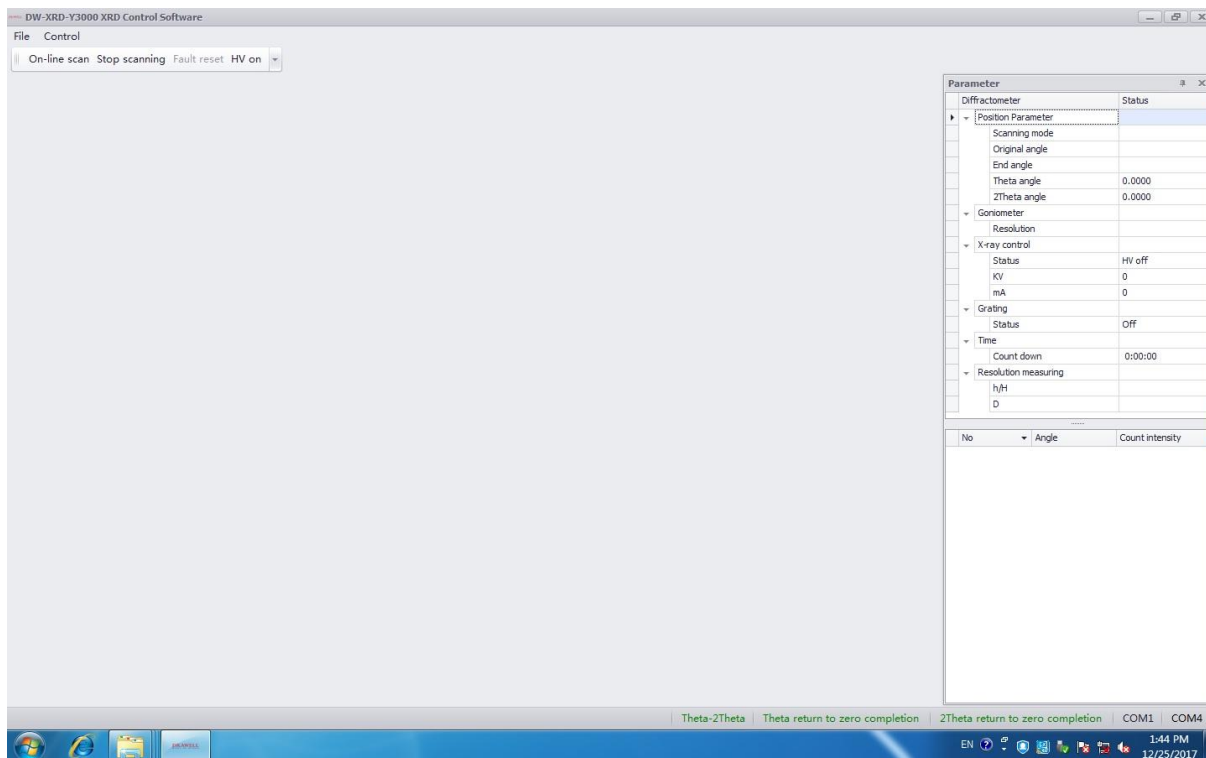
- A. The power output of $\pm 12V$ and $-15V$ has a problem, please check whether the fuse of the power unit is fused;
- B. The voltage output of the 2000V high voltage plate in the control unit has problems;

6.2.9 There is a sharp spike in the diffraction pattern:

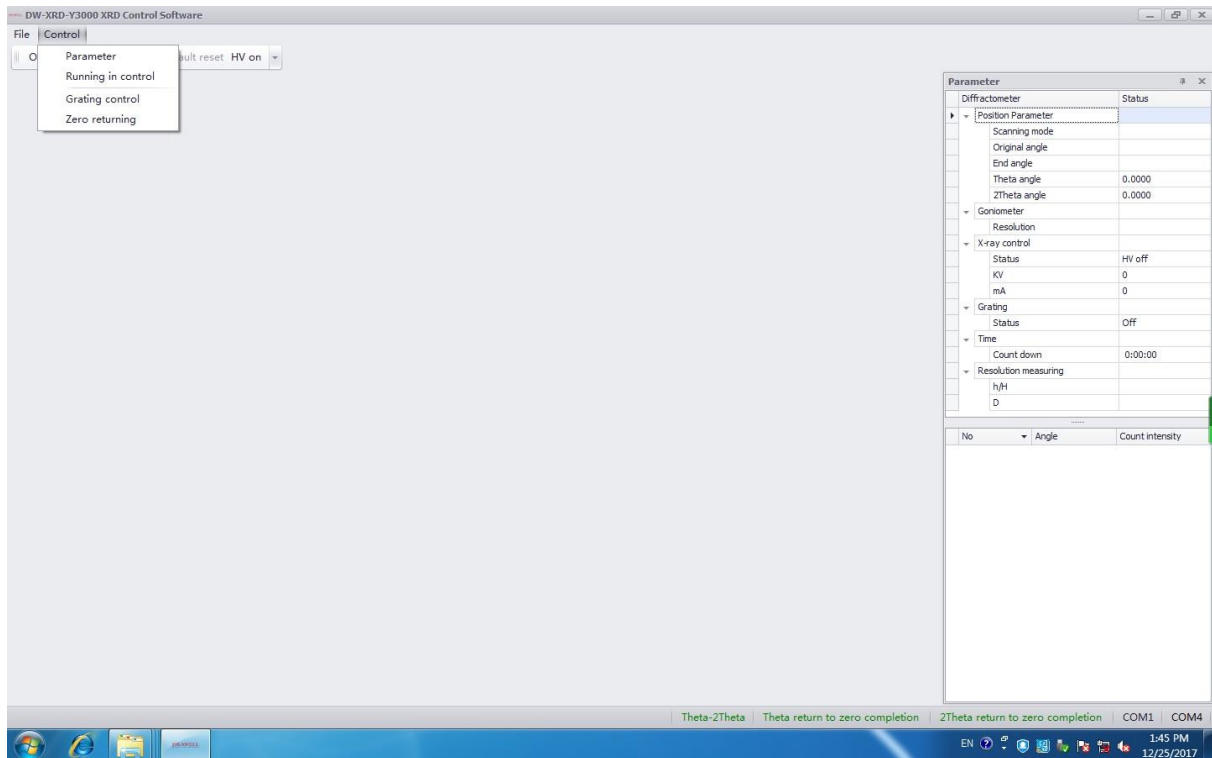
There are strong electric field interferences around the instrument, such as electric drill, high frequency furnace, etc.

7. DW-XRD-Y3000 the diffraction instrument software debugging manual

Start the DW-XRD-Y3000 diffractometer software to enter the operation interface. The software needs to use the Windows7 operating system to achieve the best operating conditions.



Menu bar includes two menus: "Document" and "Control". If you click "Control", the control & operation interface will come out.



7.1 Mechanical beam path adjustment

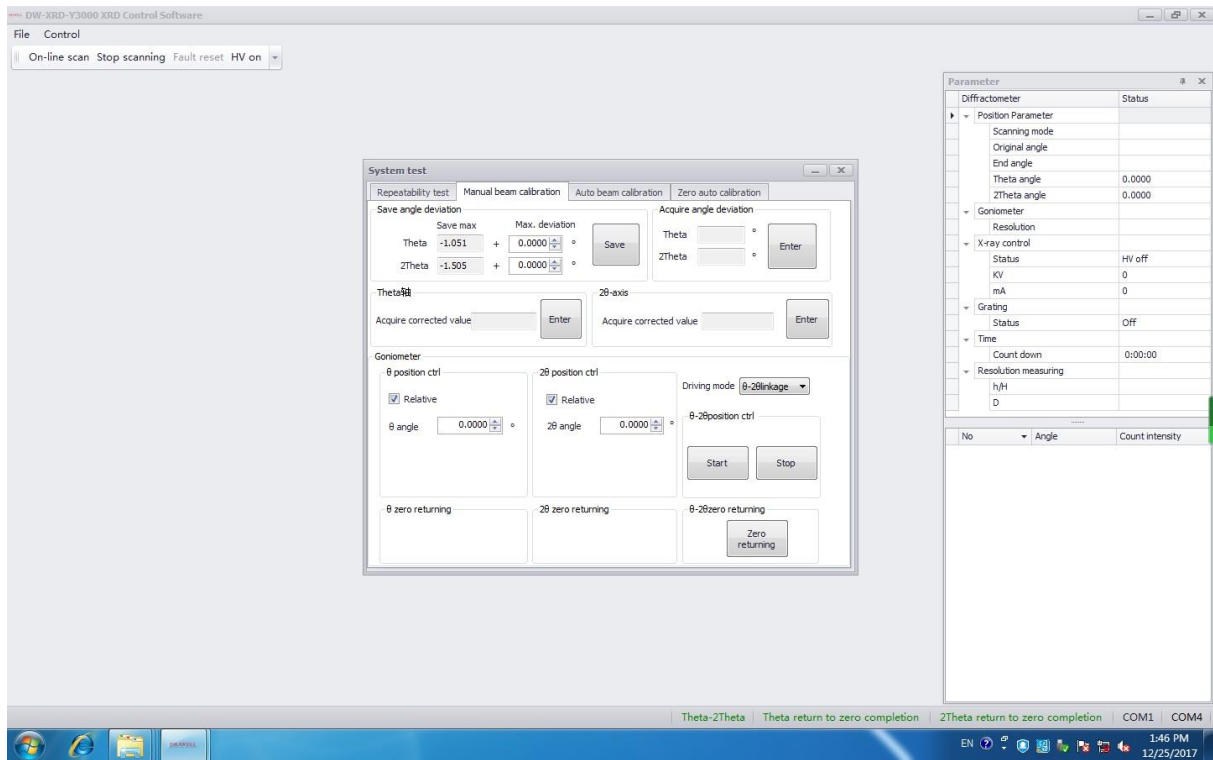
The adjustment of mechanical beam is mainly manual adjustment of the electrical zero point, and it also can control the one way movement or linkage of θ and 2θ axis respectively.

Driving mode: θ one way, 2θ one way, θ - 2θ linkage

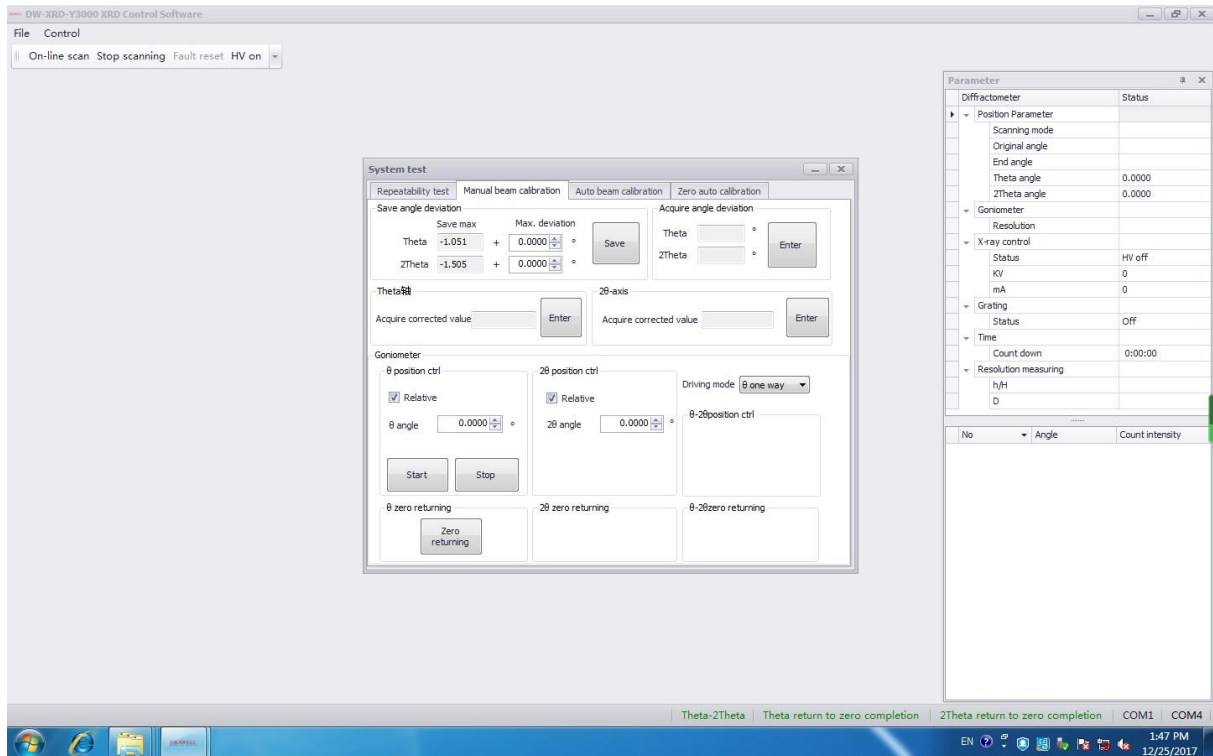
Relative: setting angle to calculate the moving step according to current Angle position. If the current angle is 5° , and setting angle is 10° , from 5° to 10° , moving step length is 5° , that means moving to 10° it will stop. If setting angle is 5° , the motor won't turn. If setting angle is -5° , it will move along the opposite direction and stop at -5° .

Absolute: Setting the single step length. If setting angle is 5° , move 5° every time relative to the current angle position.

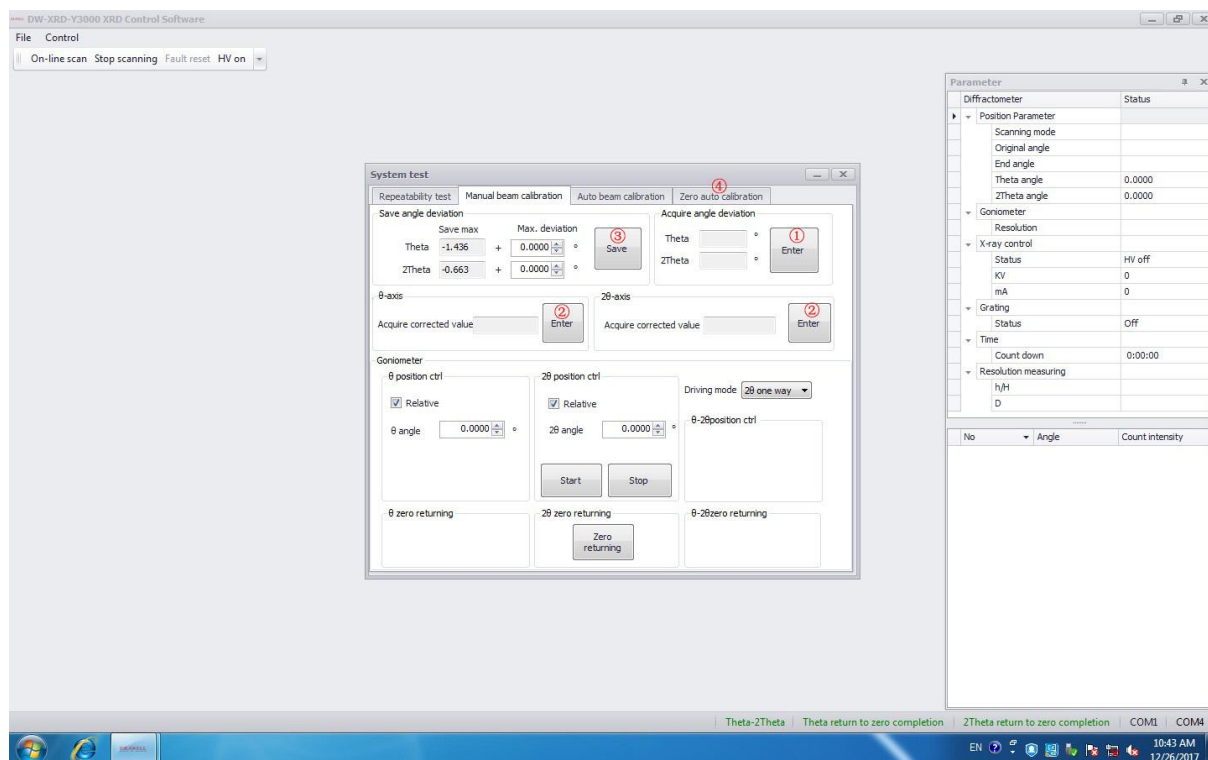
θ axis and 2θ axis linkage



θ axis one way



2θ axis one way



“Enter ①” means the system automatic logs the current angles of Theta and 2 Theta axis, at the same time it also logs the return travel of Theta and 2Theta and origin of limit switch in the direction of sheet metal back to zero until stop get the reverse process, then the origin limit switch after leaving the metal stop getting forward, reverse stroke and positive difference is the (revised).

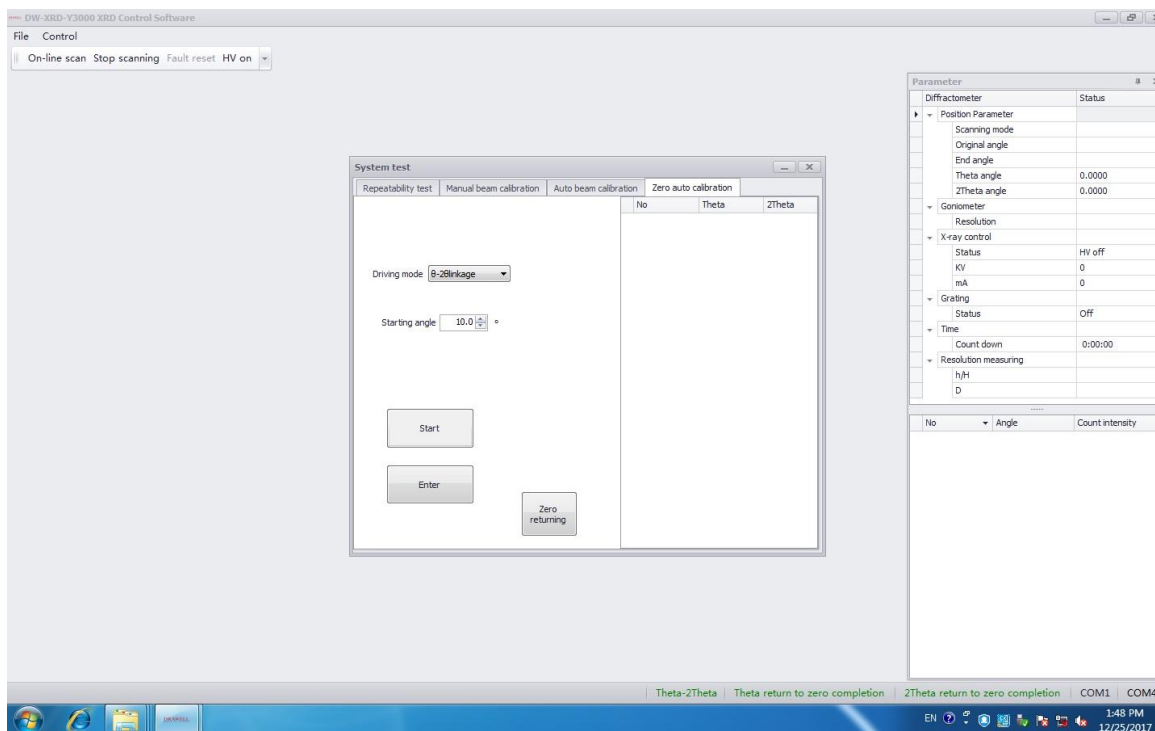
“Enter ②”:when you find the electrical zero, the current acquire corrected value will be logged in the PLC register. (after three point one is found through a straight edge line, the Theta and 2 Theta corrected value will be logged in the PLC first; when you through the figure to reach three point one line, Theta and 2 Theta corrected value will be logged to the PLC first, this is the final logging value.)

“Save ③”:By manually modifying the maximum deviation value of Theta and 2Theta, you can get to the stop position every time you return.

Theta and 2Theta are adjusted separately, one adjusted to the other.

7.2 Zero auto calibration

When choosing this function (Zero auto calibration ④), it is mainly used to debug the Theta and 2 Theta and low-speed movement after the origin switch to come into contact with sheet metal, until the detected Theta and 2 Theta is the motor shaft after the photoelectric signal to stop, to calculate the Theta and 2 is from the motor shaft stop to after leaving the sheet metal, due to the motor in the movement to stop, every stop have different deviation, so the system will according to set the starting point of value, the automatic cycle after 10 times back and forth movement automatically calculates the average Theta and Theta axis 2 out of 10 times metal and displayed in the control interface, data is written to the PLC via a button to check the address. In this way, the origin switch will automatically break away from the metal after the zero button is returned. (Before using this feature, make sure that the software Theta axis buttons and the goniometer are aligned, and the same is true for the 2Theta axis. Specific operation can be seen in mechanical light adjustment)



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