

Standard Operating Procedure Raman Microscope - Renishaw inVia

The Renishaw inVia Raman Microscope can efficiently provide Raman spectra and photoluminescence measurements for chemical composition and structure analysis. It is supplied with the powerful WiRE™ software for intuitive operation and easy data manipulation, as well as the high sensitivity ultra-low noise RenCam CCD detector.

1.0 Operation Procedure

Laser Safety

- The user is reminded that when used in accordance with the operating instructions the user should not be exposed to a laser hazard greater than Class 1. However, the system uses Class 3B lasers and as such, ALL necessary safety conditions for such instruments must be adhered to.
- Class 3B lasers are potentially hazardous if a direct beam or specular reflection is viewed by the unprotected eye. Precautions should be taken to avoid direct beam viewing, and to control specular reflections.
- It is strongly recommended to use two sets of safety glasses and/or goggles suitable for each laser wavelength used with the system.
- The system is a fully interlocked system. It ensures laser safety by automatically shuttering lasers during excitation wavelength changes. The control software tests the integrity of the interlocks during routine operation.

1.1 Startup the System

- a. Turn on the main power switch
- b. Turn on the laser
 - ✓ If you use 514 nm or 488 nm laser, the laser switch is separate from the generator. Please see the left picture below. Turn the key to “Closed” position, then turn the laser power to the marked level.
 - ✓ If you use 785 nm laser, the laser switch key is on the generator. Please see the right picture below. Just turn the key to “Closed” position.
- c. Preheat the laser for 30 minutes



514 nm and 488 nm laser

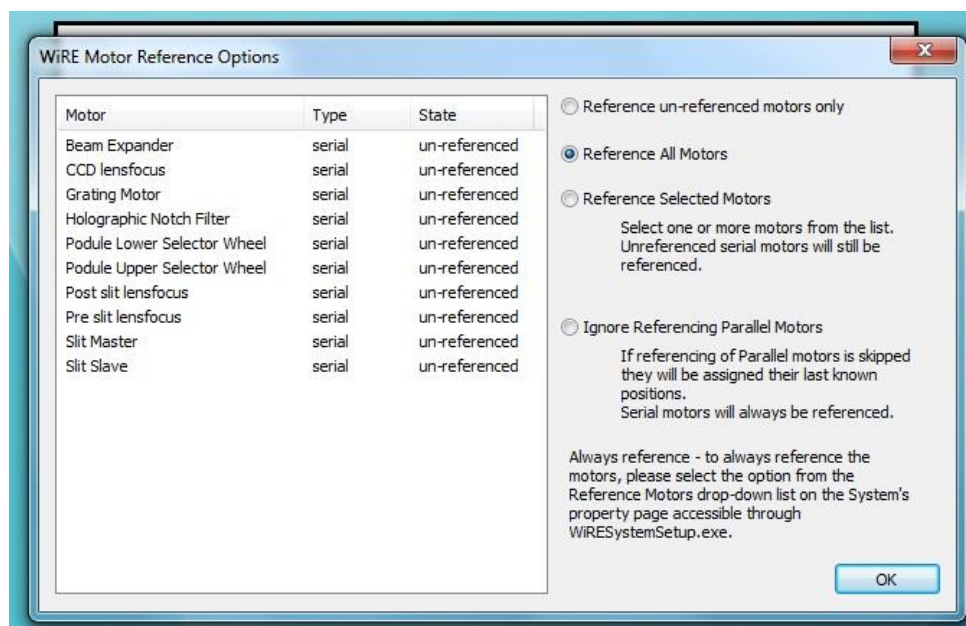


785 nm laser

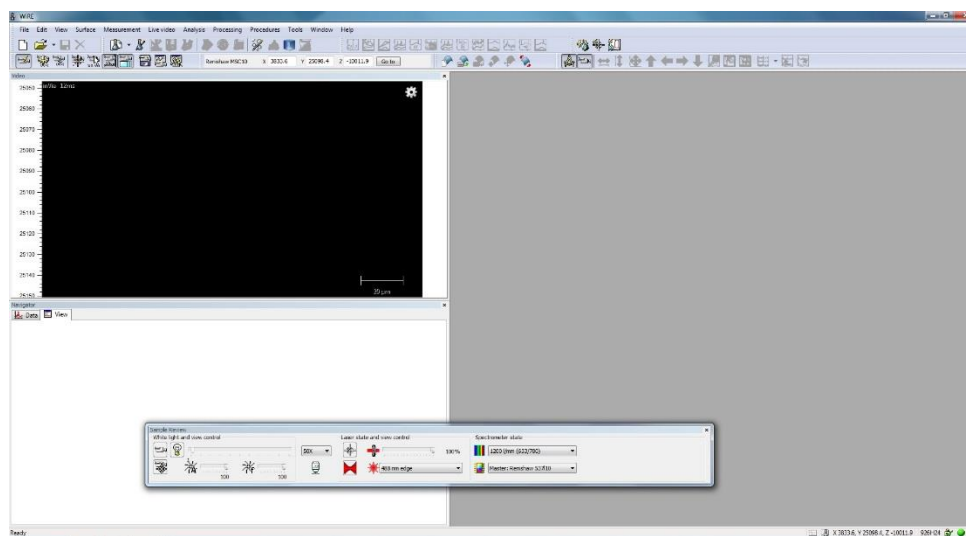
- d. Launch the control software WiRE4.2 from the desktop
- e. When there is an error message about the XYZ stage X Motor, click OK to acknowledge it



- f. When the “WiRE Motor Reference Options” window pops up, select “Reference All Motors”, then click OK.

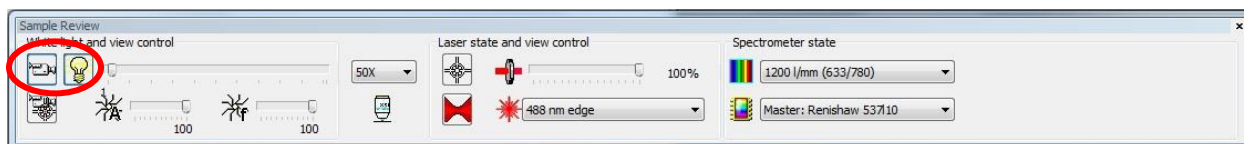


g. Wait 2-3 minutes for the system to finish initialization and display the main screen

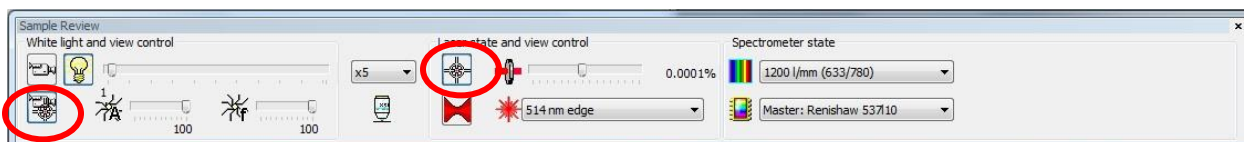


1.2 Align the Laser

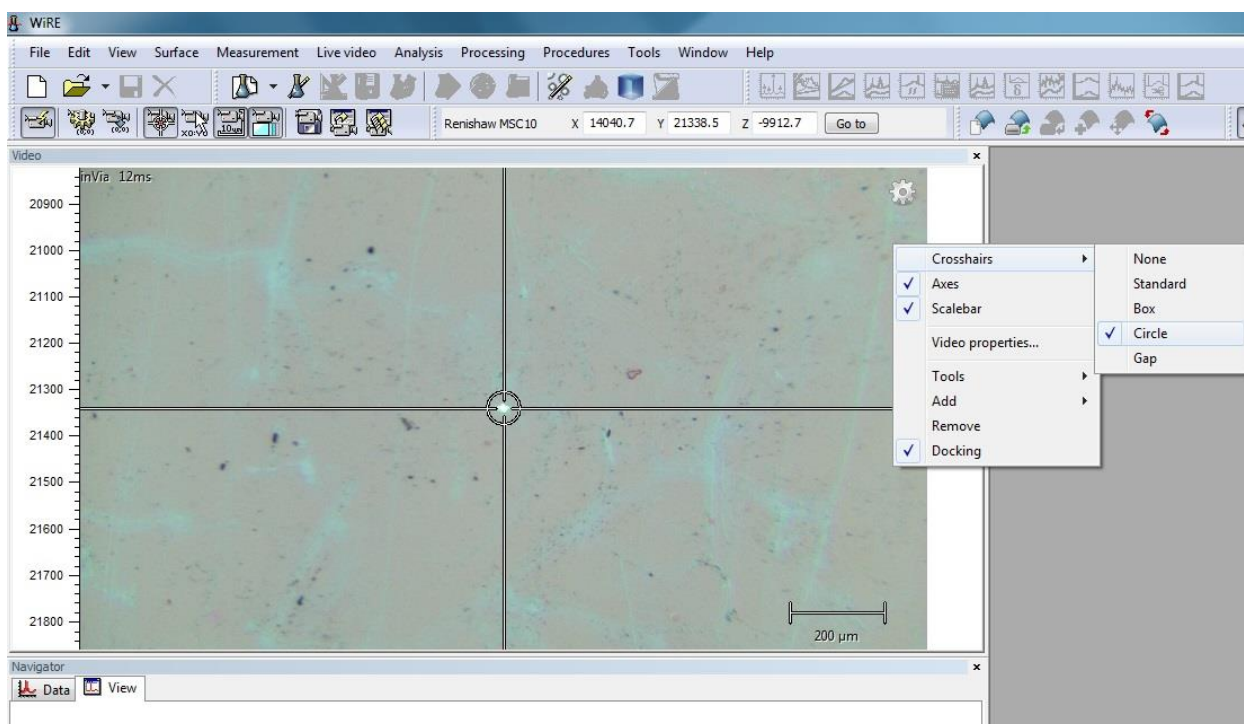
- In "Sample Review" bar, click "Sample Illumination On" and "View the Sample under Video And Eyepieces" buttons
- Adjust the white light intensity by dragging the "Lamp" slider
- Adjust the "AStop" and "FStop" sliders
- Select the correct objective lens (5x, 20x, 50x, L 50x, 100x)



- e. Use the joystick to focus on your interested point
- f. In "Sample Review" bar, click "Laser Shutter Open" and "View the Sample under Video And Laser" buttons
- g. Adjust the ND% to display a tiny small laser spot (0.0001% for 514 nm and 488 nm laser, $5 \times 10^{-8}\%$ for 785 nm laser)
- h. Select the desired laser wavelength (514 nm, 488 nm, 785 nm)
- i. Select the desired grating (1200 l/mm, 2400 l/mm, 1800 l/mm, 600 l/mm)
- j. Select the desired detector (Master detector, Andor detector)



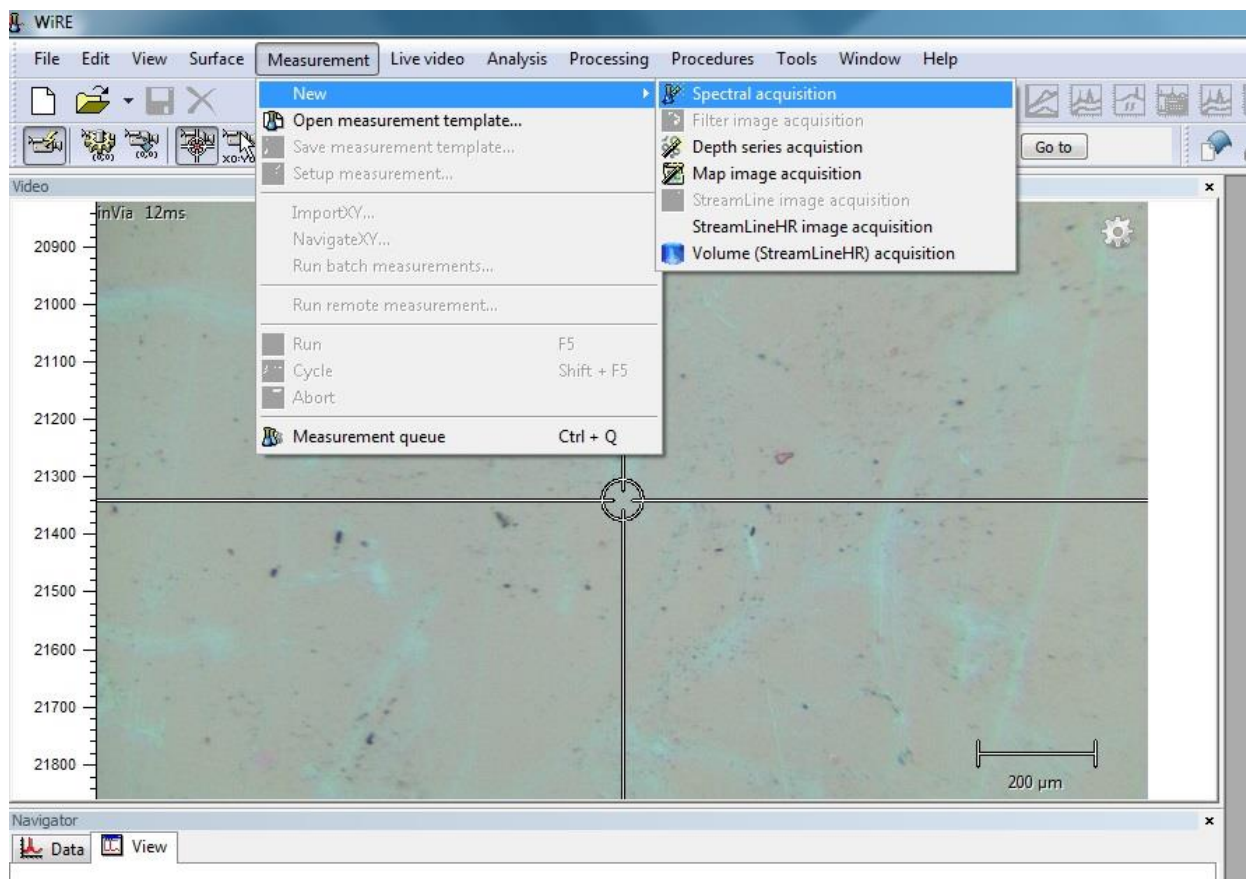
- k. In the "Video" window, right-click the mouse → Crosshairs → Circle. This will display the alignment marker.



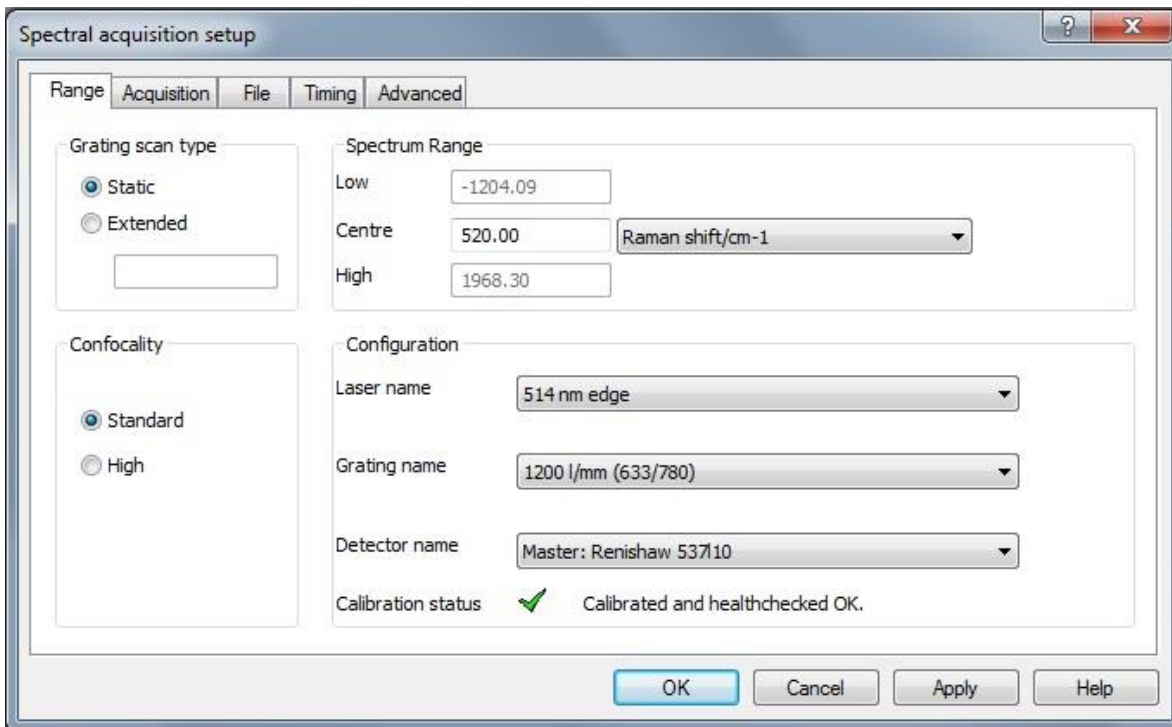
- l. Use the joystick to align the small laser spot on your interested point

1.3 Acquire the Spectrum

- a. Click Measurement → New → Spectral acquisition. This will open a “Spectral acquisition setup” dialog box. Usually, you only need to edit the first three tabs.



- b. In “Range” tab:
 - ✓ Select Static or Extended for the “Grating scan type”
 - ✓ Select “Raman shift/cm-1” for Raman tests; select “Wavelength/nm” for PL tests
 - ✓ Input the Center value of the Spectrum Range if Static is selected; input the Low and High edge values of the Spectrum Range if Extended is selected
 - ✓ Select Standard for the “Confocality”
 - ✓ Double-check the “Configuration”
- c. In “Acquisition” tab:
 - ✓ Input the “Exposure time” in second
 - ✓ Select the appropriate “Laser power %”
 - ✓ Input the number of “Accumulations”
 - ✓ Check “Close laser shutter on completion”



Spectral acquisition setup

Range | Acquisition | File | Timing | Advanced

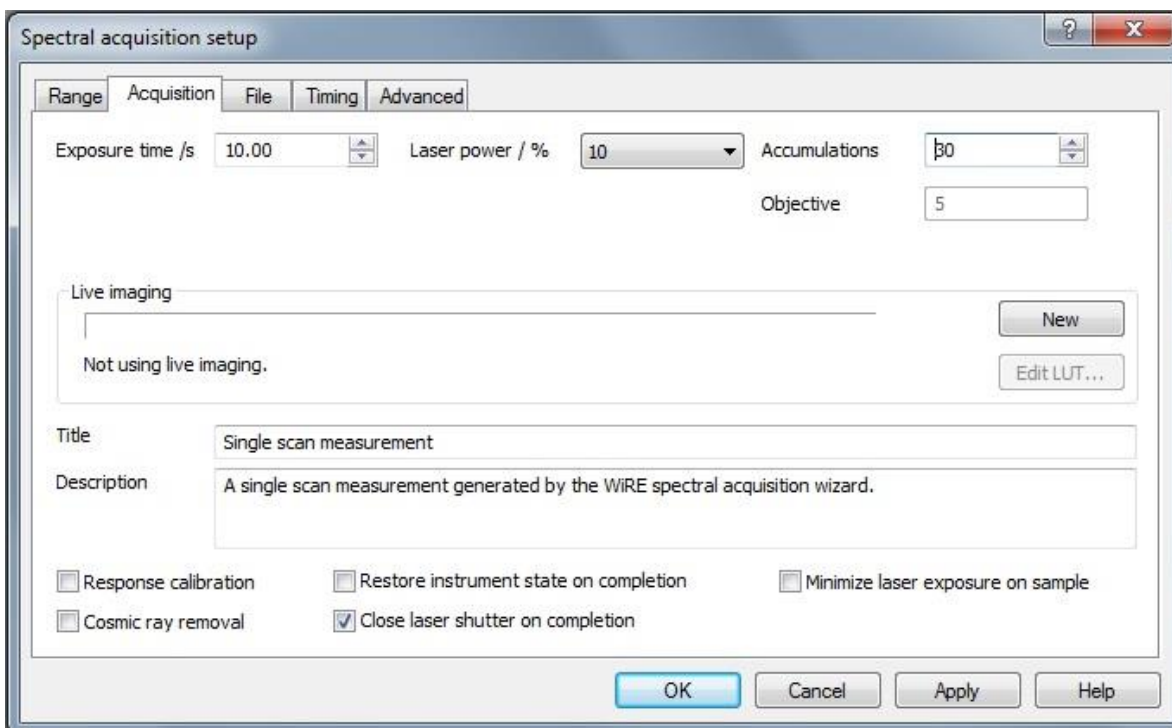
Grating scan type
☒ Static
☐ Extended

Confocality
☒ Standard
☐ High

Spectrum Range
 Low: -1204.09
 Centre: 520.00
 High: 1968.30
 Raman shift/cm-1

Configuration
 Laser name: 514 nm edge
 Grating name: 1200 l/mm (633/780)
 Detector name: Master: Renishaw 53710
 Calibration status: Calibrated and healthchecked OK.

OK Cancel Apply Help



Spectral acquisition setup

Range | Acquisition | File | Timing | Advanced

Exposure time /s: 10.00
 Laser power / %: 10
 Accumulations: 30
 Objective: 5

Live imaging
 Not using live imaging.

Title: Single scan measurement
 Description: A single scan measurement generated by the WIRE spectral acquisition wizard.

☐ Response calibration
☐ Cosmic ray removal
☐ Restore instrument state on completion
☒ Close laser shutter on completion
☐ Minimize laser exposure on sample

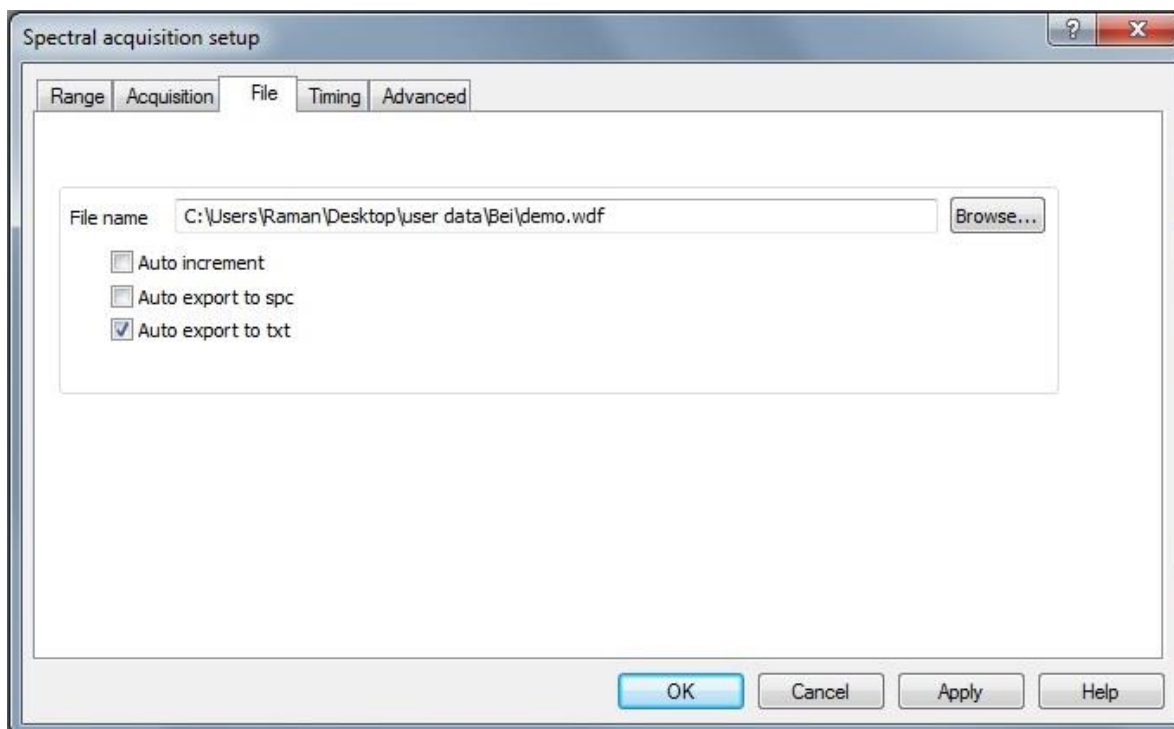
New Edit LUT...

OK Cancel Apply Help

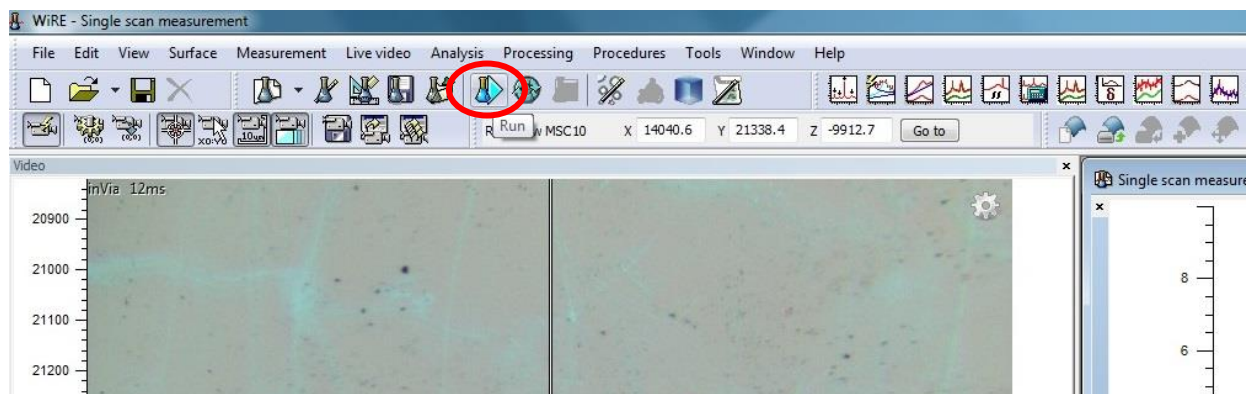
d. In "File" tab:

- ✓ Click the Browse button to specify the directory for saving the results. The results will be saved as .wdf file.

- ✓ Check “Auto export to txt”

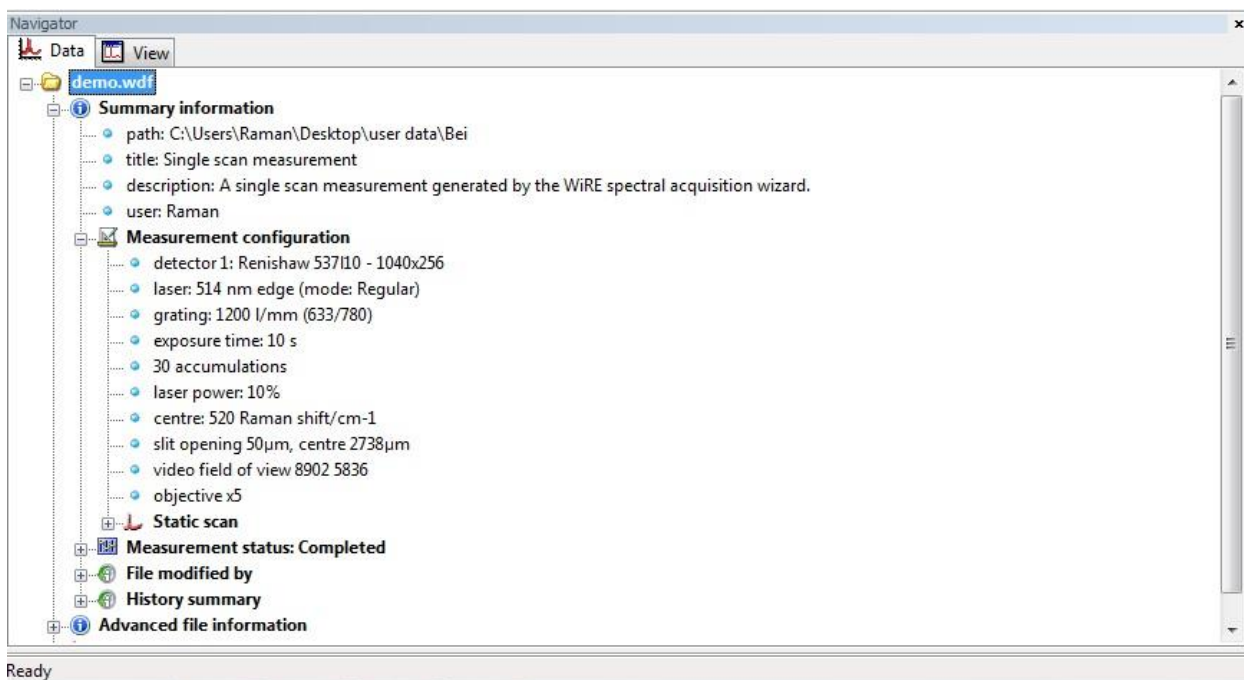


- e. Click Apply, then click OK.
- f. Click “Run” to start the spectral acquisition; if you wish to abort the acquisition, click “Abort”

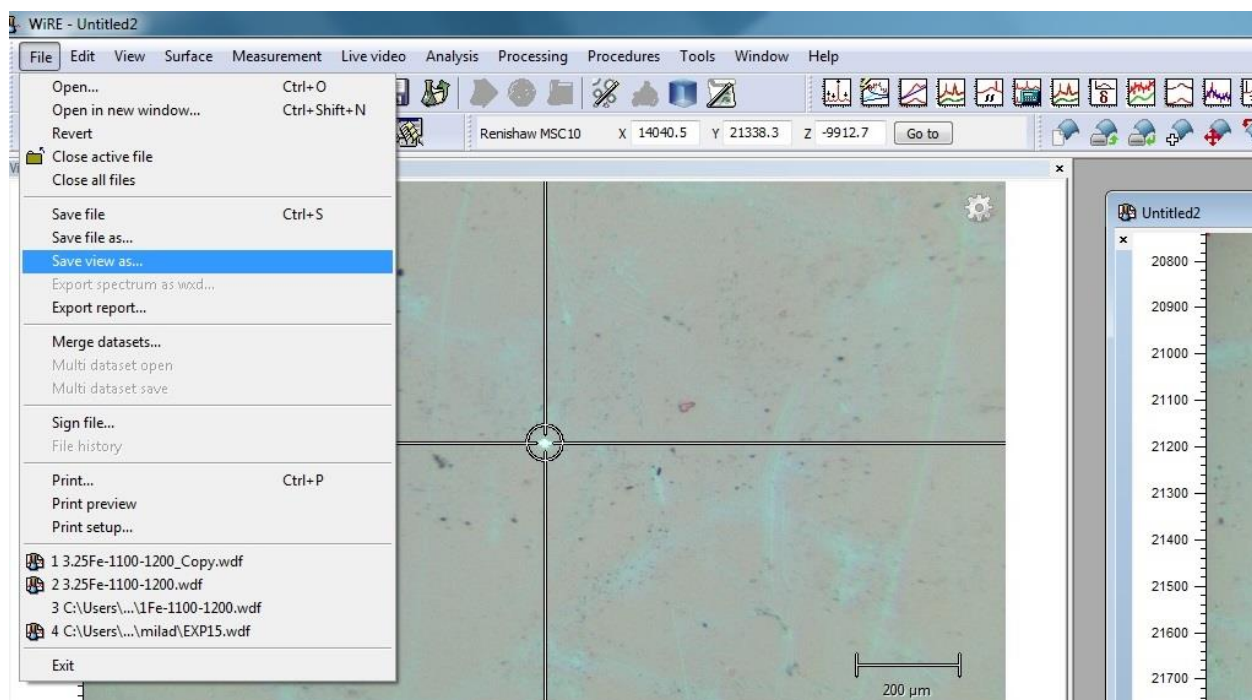
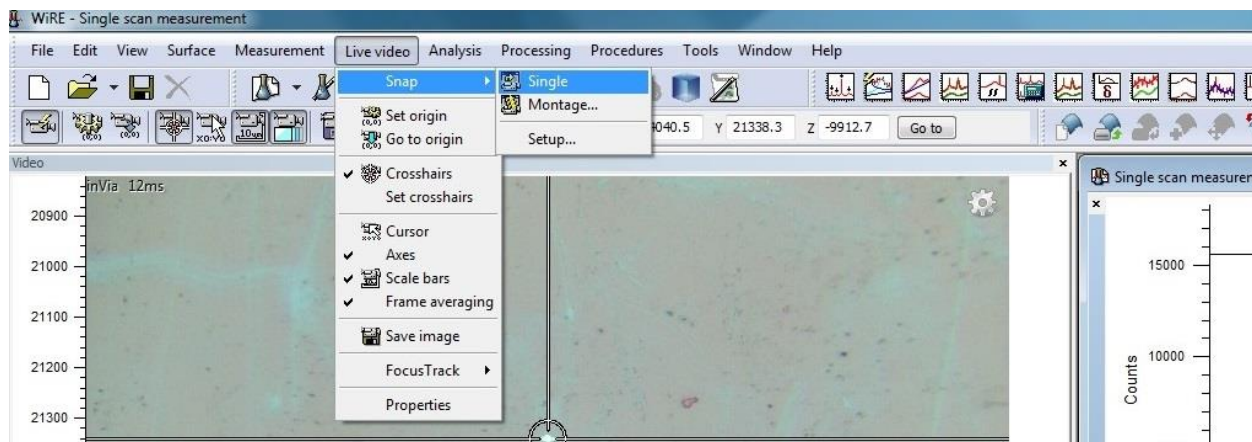




- g. After the spectral acquisition, you can review the measurement parameters in the “Navigator” window



- h. If you wish to capture and save an image of your sample:
- ✓ Close the measurement result window(s)
 - ✓ Click Live video → Snap → Single
 - ✓ Click File → Save view as..., the captured image will be saved as .emf file



1.4 Shutdown the System

- Close the WiRE4.2 control software
- Turn the laser power to the minimum level (Min.)
- Turn the laser switch key to "Open" position
- Turn off the main power switch

2.0 Specifications/Features

- Three laser wavelengths: 488 nm, 514 nm, 785 nm
- Raman shift range: up to 4000 cm^{-1}
- Photoluminescence (PL) range: up to 950 nm
- Objectives: x5, x20, x50, x100, L x50, SLW x20

	x5	x20	x50	x100	L x50	SLW x20
WD (mm)	14.0	1.15	0.5	0.22	8.2	25.0
Cryostat use	Yes	No	No	No	Yes	Yes

- Two sets of gratings: 1200 l/m + 2400 l/m, 600 l/m + 1800 l/m
- Measurement capabilities:
 - Spectral acquisition
 - Depth series acquisition
 - Map image acquisition
 - StreamlineHR image acquisition
 - Automatic image montaging
- Basic Raman spectra analysis capabilities (e.g. peak pick-up, substrate baseline, etc.)
- WIRE™ 4.2 control and analysis software

3.0 User Requirements

The Renishaw inVia Raman Microscope must be used by authorized personnel only. All authorized users are expected to read and understand this SOP and follow the operation instructions carefully. No unauthorized user may operate this Raman unless accompanied by an authorized user. All visitors must be briefed on proper safety protocol and must wear appropriate personal protective equipment. To become an authorized user, one must:

1. Complete Environment, Health & Safety (EH&S) training
2. Complete initial orientation and training for the Materials Characterization Laboratory
3. Receive training on this piece of equipment from lab personnel
4. Schedule equipment time using the calendar
5. Read and fully understand this SOP

4.0 General Safety

4.1 Required Personal Protective Equipment

Users must wear lab coats, safety glasses, and gloves. Shorts, open-toed shoes, high heels, and skirts, are forbidden.

4.2 Emergency Procedures and Contacts

For non-life threatening emergencies: notify the MCL facility manager and your PI immediately.

Facility manager: Zongmin (Shirley) Bei, Ph.D.

Office: 109B Furnas Hall, Tel: (716) 645-5165, Cell: (585) 354-5623

Email: zongminb@buffalo.edu

or for police / ambulance, call 645-2222

In case of fire or other life threatening emergency: Exit the laboratory through an emergency exit door. Pull one of the fire alarms located in the main hallway. Dial campus police / ambulance at 645-2222.

4.3 University after hours laboratory use policy

No working alone, use the buddy system!

#	Revised by	Date	Modification
1	Zongmin (Shirley) Bei	05/10/2016	Document initial release
2	Zongmin (Shirley) Bei	07/07/2016	2.0 Specifications
3			
4			
5			