SYNCHROTRON RADIATION RESEARCH AT SINGAPORE SYNCHROTRON LIGHT SOURCE*

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Abstract

SSLS is in routine operation with four beamlines since November 2003. Recent results are related to micro/nano-fabrication, bio and medical physics, materials science, and the development of future synchrotron radiation sources. The set up of an infrared spectro/microscopy beamline is in progress, planning for four more beamlines is under way.

INTRODUCTION

Synchrotron radiation research at SSLS is focused on the analytical characterization of materials and processes, on micro/nanofabrication, and on key concepts of 4th generation sources based on superconductive miniundulators (supraminis). The R&D programme to set up the relevant beamline equipment and test facilities was started at the end of 2000. User pilot operation began in October 2001, routine operation with four beamlines in November 2003. Recent results will be highlighted and further planning outlined.

FACILITY

SSLS is operating a compact 700 MeV electron storage ring that produces synchrotron radiation from two superconducting dipoles featuring a magnetic flux density of 4.5 T. The characteristic photon energy/wavelength is about 1.5 keV/0.85 nm, respectively. Hence, the useful spectral range extends from about 10 keV to the far infrared. Routinely, initial currents stored are well in excess of 400 mA with a lifetime of 11 h which improves to 17 h at smaller currents. Electron beam emittance is about 1.3 µm·rad due to the large bending angle of 180°.

A schematic layout of SSLS' facilities that are operational or under construction is depicted in Fig. 1.

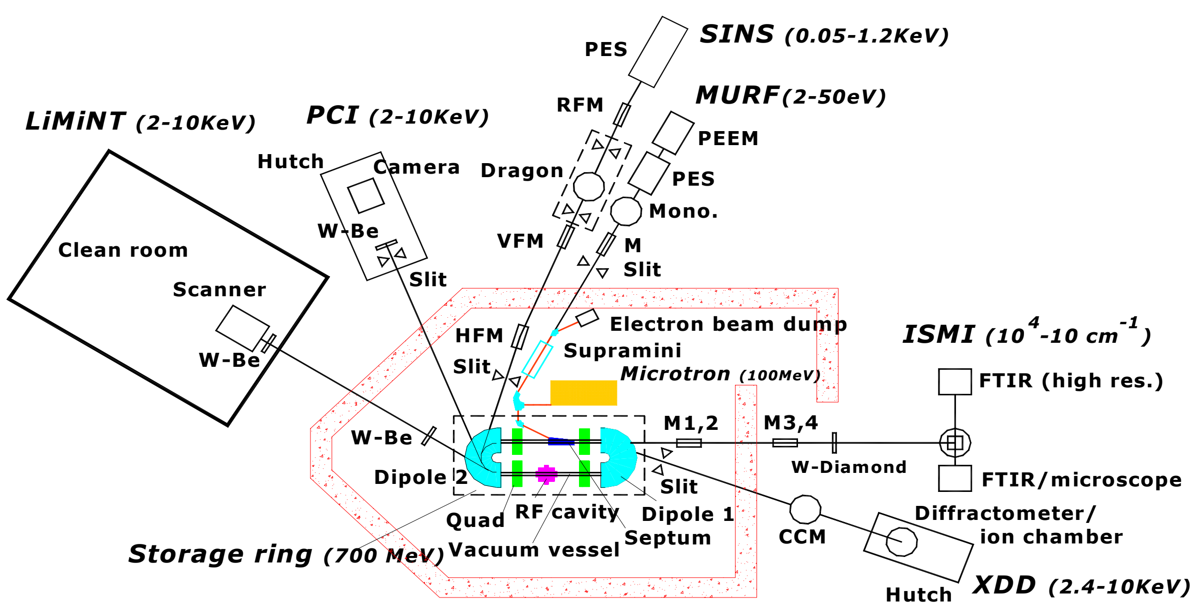

Fig. 1: Schematic layout of the accelerator system, the beamlines, and experimental facilities at SSLS.

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Four beamlines/experimental facilities are in operation, including the LiMiNT micro/nanotechnology lab, the PCI white light phase contrast imaging facility, the SINS soft X-ray beamline for surface, interface, and nanostructure science with a spectral range from 50 eV to 1.2 keV, and the XDD X-ray diffraction and absorption spectroscopy beamline. The ISMI facility for Infrared Spectro/Microscopy from the visible to the far infrared (10 cm^{-1}) is under construction. It features a UHV RAIRS chamber (reflectance absorbance infrared spectroscopy) in which a controllable number of monolayers of atoms or molecules can be adsorbed on a catalyst surface to study their chemical interaction.

Beyond micro/nanofabrication and the analytical characterization of materials and processes SSLS is working at key issues of future 4^{th} generation light sources. Two main topics are pursued, namely, the conceptual study of the Linac Undulator Light Installation (LIULI) and the development of supraminis.

RESULTS

Lithography for micro/nanotechnology

With the LiMiNT micro/nanotechnology lab SSLS is capable of doing a complete prototyping as a one-stop shop using the integral cycle of the LIGA process for producing micro/nanostructures from mask writing via either laser direct writing or e beam lithography over X-ray irradiation, development, to electroplating in Ni, Cu, or Au, and, finally, hot embossing in a wide variety of plastics as one of the capabilities to go into higher volume production. The process chain also includes plasma cleaning and sputtering as well as substrate preparation processes including metal buffer layers, plating bases, and spin coating, polishing, and dicing. Furthermore, metrology using scanning electron microscopy (SEM), optical profilometry, and optical microscopy is available. The super-resolution process is being developed for nanolithography [1]. Fig. 2 shows the X-ray lithography scanner and Fig. 3 a view of some of the process equipment in the class 1000 cleanroom. Fig. 4 shows an X-ray mask fabricated at LiMiNT with various Au absorber micro and nanopattern on a graphite membrane. Fig. 5 depicts 1000 µm tall test structures in SU-8 resist. As the vanes are 5 µm thick the aspect ratio goes up to 200.

Fig. 2: X-ray lithography scanner. The beamline enters the class 1000 clean room from the rear wall.

Fig. 3: View of the clean room. Laser direct writer and plasma cleaner in the foreground, behind the scanning electron microscope with electron beam lithography add on. In the background from left the electroplating wet bench, chemical hoods, and the optical metrology.

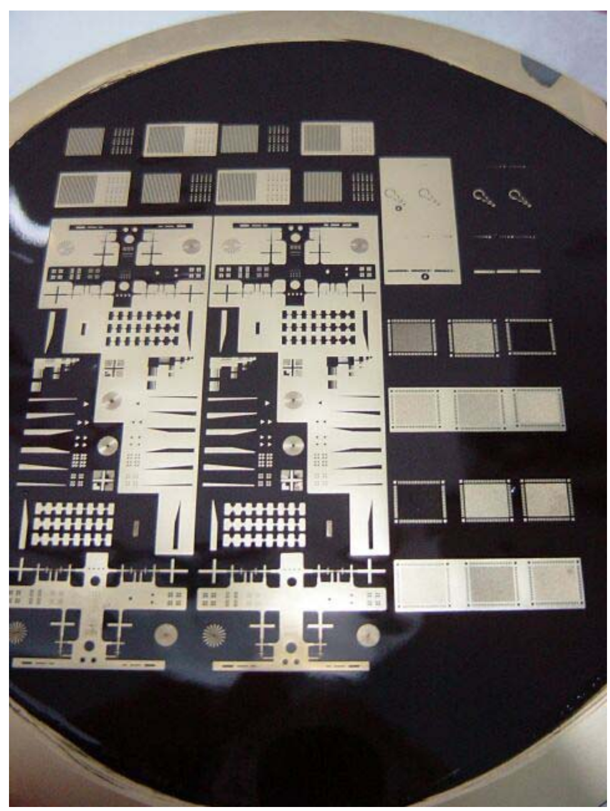

Fig. 4: 4 inch X-ray mask with Au absorber pattern on a graphite membrane.

Fig. 6 displays Ni nanorods 200 nm in diameter and 230 nm high on a Cu plating base. They were produced using electron beam lithography. Finally, Fig. 7 shows a study of structures for composite electromagnetic materials electroplated in Ni.

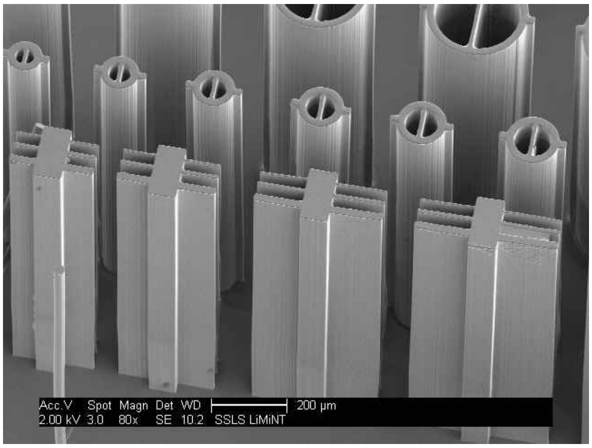

Fig. 5: Test structures in SU-8 resist 1000 µm tall. The vanes are 5 µm thick leading to an aspect ratio of up to 200.

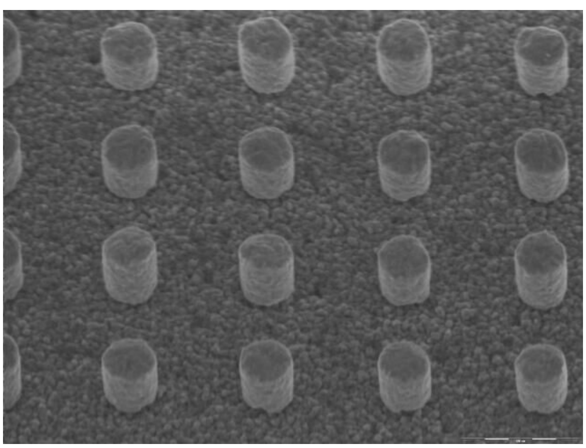

Fig. 6: Ni nanorods on a Cu plating base. Height and diameter are 230 nm and 200 nm, respectively.

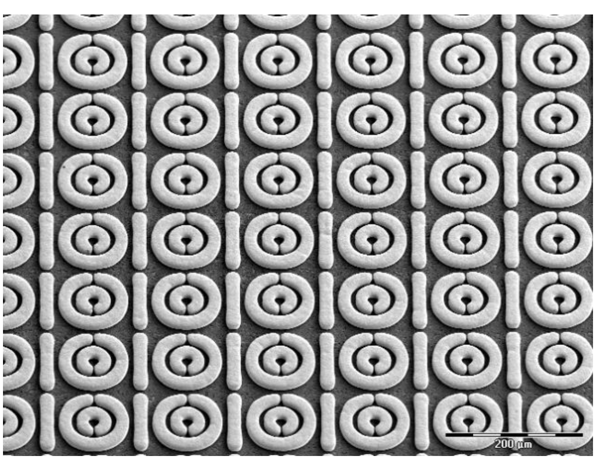

Fig. 7: Rod-split ring structures of Ni. Outer ring is 100 µm in diameter, spectral transmission is around 2.4 THz.

Phase contrast imaging

White light phase contrast imaging is in routine use at SSLS to study internal structures of samples with a sufficiently small thickness and low atomic number to be transparent enough for about 10 keV photons. As an example, a 500 µm thick Si wafer can be "looked through". Temporal variations of the internal structures under any processes can be monitored with a resolution of up to 100 ms.

The PCI beamline has been extended to 16.76 m from source to sample in order to achieve about 1 µm spatial resolution. Between the source point and the sample the photon beam has only to pass through a 500 µm thick Be window. It then traverses the sample whereby the angular distribution of the rays becomes modulated. About 2-3 cm downstream the sample the X-ray photons hit a $CdWO_4$ scintillation foil and are converted to visible light which is then recorded by a CCD camera equipped with a magnifying objective. The contrast formation is mostly due to refraction.

Most of the samples so far have been biological, medical, or plastic objects [2]. Hippeastrum leaves were studied revealing the cellular structure down to a single cell [3]. Fig. 8 illustrates an application to hydraulics showing water flowing in a polypropylene tube that is opaque in the visible. A few bubbles are sharply imaged. The width of the image is about 1 mm.

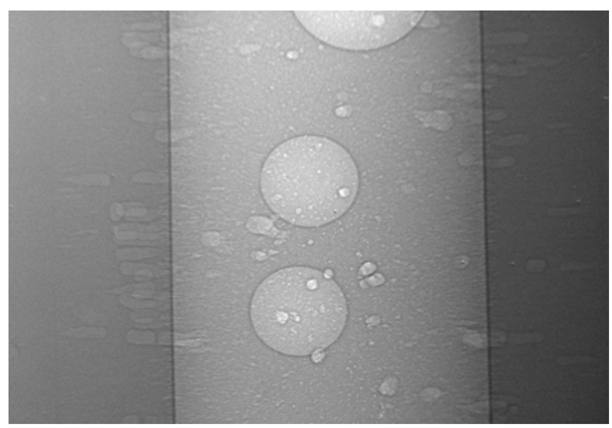

Fig. 8: Water with bubbles enclosed in a polypropylene tube. Image width about 1 mm. The two large bubbles have a diameter of about 160 µm.

Soft X-ray beamline

The SINS beamline provides 50 – 1200 eV photons for surface, interface, and nanostructure science. The experimental station is equipped with photoemission spectroscopy (PES) and absorption spectroscopy (XAS). The polarization of the incoming photon beam can be varied from left-hand circular over linear to right-hand circular polarization by adjusting the vertical position and the pitch of the vertically focusing mirror in the beamline. Hence, X-ray magnetic circular dichroism (XMCD) studies can also be performed. The beamline is based on the Dragon design [4] with a plane-elliptical mirror for condensing and horizontal focusing, a spherical mirror focusing the photon beam on the entrance slit of the monochromator, a grating monochromator featuring 4 spherical gratings with 1200, 600, 300, and 130 lines per mm which focus the beam on the exit slit, and a toroidal mirror to refocus the beam onto the sample. The sample is located in a fast-entry UHV surface science station featuring a hemispherical electron energy analyzer for photoemission spectroscopy (PES), a standard twin anode X-ray tube, a low energy electron diffraction (LEED) system, sputter and evaporation sources, and a magnetic coil. It will soon be upgraded with *in situ* STM/AFM.

Fig. 9 shows the SINS beamline looked at from the end station upstream. The UHV chamber and the hemi-spherical energy analyzer are clearly to be seen, behind them the UHV vessel of the toroidal refocusing mirror.

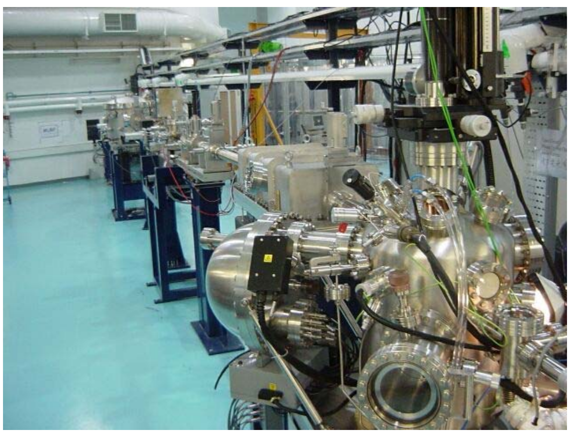

Fig. 9: SINS beamline as seen from the endstation.

Some preliminary result is shown in Fig. 10. When measuring XPS spectra of nanoparticles consisting of Fe and Co the initial curve did not show any iron peak at 300 eV kinetic energy while cobalt was clearly visible. Only after sputtering, the iron peak showed up suggesting that in the original nanoparticles cobalt enclosed the iron completely and needed to be removed by sputtering to reveal it.

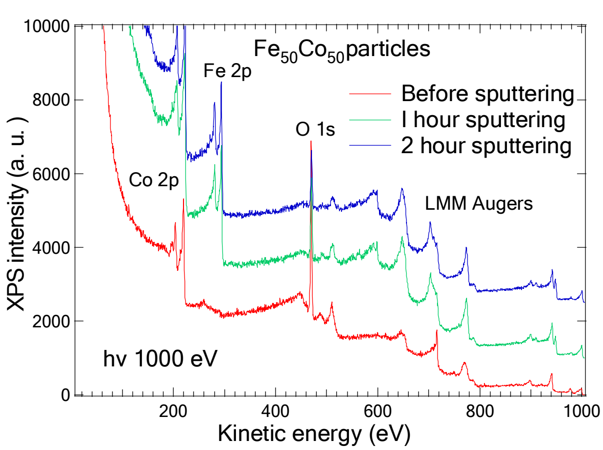

Fig. 10: XPS spectra of Fe/Co nanoparticles. Bottom curve was measured prior to sputtering, middle and top curve after 1 h and 2 h of sputtering, respectively.

Fig. 11 illustrates the use of XMCD for studying magnetization in sputtered Ni layers in comparison to standard polycrystalline Ni foil.

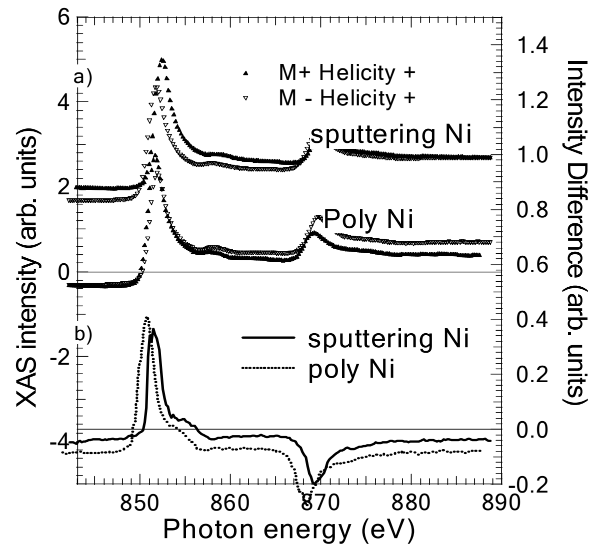

Fig. 11: XMCD spectra of sputtered Ni layers and standard polycrystalline Ni foils.

XDD beamline

The X-ray development and demonstration beamline is equipped with a 4-circle Euler diffractometer, a Si(111) channel-cut monochromator and ionization chambers. Thus, various methods involving diffraction and absorption spectroscopy can be used. Fig. 12 illustrates an application to the diagnostics of thin films using reflecto-metry. Fig. 13 displays high-resolution diffraction data from a GaAs/InGaAs multiple quantum well. The main GaAs Bragg peak as well as satellites and thickness fringes are observed. The simulation was done for a smooth interface structure. It models the measured data reasonably well.

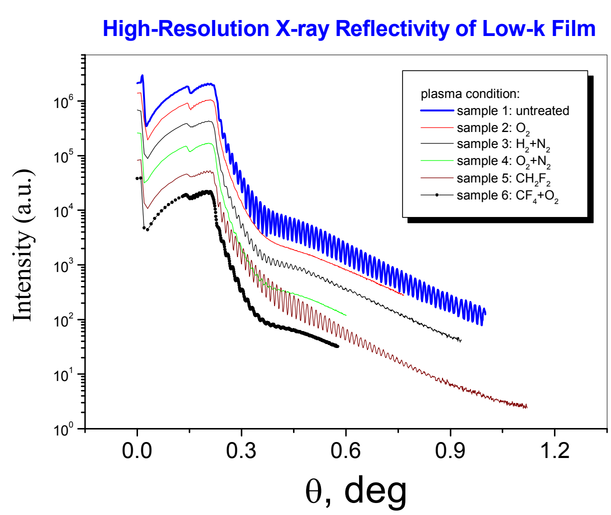

Fig. 12: X-ray reflectivity of thin layers of low-dielectric-constant Si subject to various plasma treatments. The layers are 260 nm thick and are deposited on a Si wafer.

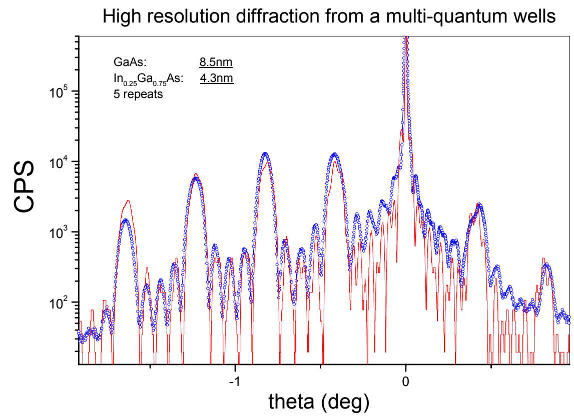

Fig. 13: High-resolution diffraction from a GaAs/InGaAs multiple quantum well. Measured data dotted, simulation solid line.

In its present implementation, the beamline is lacking mirrors, however, upgrading with a telecentric pair of condenser and refocusing mirrors is being pursued.

LIULI and supramini

LIULI is a conceptual study of a 4th generation light source based on supraminis and a superconducting linac. It foresees a photoinjector and a recirculation loop for energy recovery/doubling studies.

Hardware progress was made with a (50×14 mm) prototype supramini. It was built by ACCEL and delivered to SSLS recently. Fig. 14 shows the components during unpacking. After a qualification and training phase at SSLS the device is planned to be installed at the 30 MeV linac of the Shanghai Synchrotron Radiation Facility for beam experiments.

Fig. 14: Supramini vessel, girder, 3 cryocoolers, and power supplies and controls.

CONCLUSION

After 3 years into its development programme SSLS is in routine operation and has produced results in the fields of micro/nanofabrication, bio and medical physics, materials science, and source development. Strong activities aiming to set up new beamlines are in progress, in particular, an X-ray microimaging facility, a small angle scattering beamline, an EUV lithography test facility, and an advanced photoemission electron microscope.

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