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The BL01B1 infrared beamline at Shanghai Synchrotron Radiation Facility

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ABSTRACT

The NCPSS's (National Center for Protein Science Shanghai) BL01B1 beamline is the first infrared beamline at SSRF (Shanghai Synchrotron Radiation Facility). The optical layout and beam performance of the beamline are overviewed in this paper. There are two operational experimental stations at BL01B1 including an infrared microspectroscopy and imaging station, and a time-resolved infrared spectroscopy station. The equipment and characteristics of these two experimental stations are introduced. An experimental platform aiming at high pressure and low temperature IR/Raman investigations is under construction. The applications of the beamline stations and results obtained under the collaboration of BL01B1 and user groups are summarized.

1. Introduction

Fourier transform infrared spectroscopy (FTIR) is an effective method to study samples' constituent species and molecule structures. It has been widely used in chemistry, life science, pharmaceutical science, material science, etc. Comparing to synchrotron radiation FTIR (SR-FTIR) methods, traditional FTIR's functions are limited because of the brilliance of the light source [1]. Based on the excellent properties of synchrotron infrared radiation, many infrared beamlines have been built in the synchrotron radiation facilities all around the world during the last two decades [1–8]. By using the high brilliance of synchrotron infrared light, SR infrared microspectroscopy (IRMS) can reach higher spatial resolution with higher signal to noise ratio than conventional FTIR, especially when the aperture size below about 20 µm. Synchrotron radiation infrared light has a wide spectral coverage from near-IR to even THz region. SR-FTIR is also a non-destructive analytical method even for live biology systems. Time-resolved experiments can be proceeded due to synchrotron infrared radiation's specific time structures and time-resolved functions of FTIR spectrometer.

The BL01B1 infrared beamline is the first synchrotron radiation infrared beamline based on a third generation synchrotron radiation facility in China. The BL01B1 infrared beamline was built under the collaboration between NCPSS and SSRF, and then managed by NCPSS. BL01B1 started formal run at July 28th, 2015. Two experimental stations are now under operation. They are the infrared microspectroscopy and imaging station, and the time-resolved infrared spectroscopy station. A high pressure/low temperature platform is under construction. The optical system of the BL01B1 beamline, and the configuration of experiment stations are presented in this paper. Applications of BL01B1 and results of user groups are also reviewed.

2. Optical system

Synchrotron radiation infrared light of BL01B1 was extracted from the storage ring of SSRF which is a third generation synchrotron radiation facility. SSRF contains a 150 MeV linear accelerator, a 3.5 GeV booster, a 3.5 GeV storage ring, beamlines and experimental stations. The main specifications of the storage ring are listed in Table 1.

The optical layout of BL01B1 is shown in Fig. 1. The detailed design of the optical system has been reported elsewhere [9]. Bending magnet radiation (BMR) and edge radiation (ER) are extracted from the storage ring by a plane extraction mirror M1 and used as infrared synchrotron radiation light sources of BL01B1. To assure maximized cone of extracted radiation and photon flux, the vacuum chamber of BL01B1 was modified. The final obtained extraction solid angle at the horizontal and vertical directions are 40 mrad (−15 to 25 mrad) and 20 mrad (−10 to 10 mrad), respectively.

The plane mirror M1 is located 1.815 m away from the light source and reflect the incident light by 90° horizontally to a flat mirror M2. The M1 mirror is slotted in the center to let the intense X-ray and UV radiation pass through. Otherwise, the overheating of M1 will lead to serious distortion. M1 is made of Glidcop substrate coated with a ca. 200 nm layer of aluminum. The substrate material possesses good thermal conductance and elevated temperature strength. A photon

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Table 1
Main specifications of SSRF storage ring.

Specification	Value
Storage Ring Energy Eb	3.5 GeV
Circumference L0	432 m
Natural Emittance ε0	3.90 nm·rad
Multi-bunch Beam current I0	200–300 mA
Single-bunch Beam current Ib	5 mA
Number of Straight Sections N	20
Natural Energy Divergence σE	9.7×10^{-4}
Radio Frequency frf	499.654 MHz
Couple Efficiency κ	0.01
Radius of Curvature ρ	9.1673 m
Magnetic Field B0	1.2726 T

mask is placed in front of M1 to protect the end of the slot near the electron beam. A beam absorber is positioned behind M1 to dump the transmitted power. Additionally, the M1 mirror is indirectly water-cooled, and six temperature sensors are placed slightly inside the slot. These sensors are linked to a safety interlock system to protect M1. Once the temperatures of these sensors exceed the set range, the safety interlock system will work. Moreover, the M1 mirror can be moved at the vertical direction for maintenance.

After reaching the flat mirror M2, the photon beam is reflected vertically to a toroidal focusing mirror T1, and then directed through a hole in the tunnel wall to a wedged CVD diamond window. The CVD window separates the ultra-high vacuum section of the beamline from the downstream high vacuum section. Then, the photon beam goes through a variable slit that blocks the stray lights, and is reflected vertically downward by another toroidal focusing mirror T2. After T2, a set of active feedback systems developed at ALS is installed [10], but they are not in operation since the infrared beam is stable enough and there is currently no unwanted noise that need to be corrected. Two dichroic beamsplitters BS1 and BS3 are used to reflect the infrared radiation and transmit the visible light. Two position-sensitive detectors are installed after the two beamsplitters, respectively. The BS2 beamsplitter are used to control which experimental station the synchrotron infrared radiation is directed into. Two plane mirror M8 and M9 are used to reflect the synchrotron infrared radiation into the two experimental stations respectively.

3. Experimental stations

Two experimental stations are under operation at BL01B1 now. They are the infrared microspectroscopy and imaging station located after M9 mirror, and the time-resolved infrared spectroscopy station located after M8 mirror. A high pressure platform is also under construction at BL01B1 between these two experimental stations. The infrared microspectroscopy and imaging station is mainly used to non-destructively study sample's composition and spatial distributions of its' components at a higher resolution level with high signal to noise ratio. The time-resolved infrared spectroscopy station is used to study dynamic processes in the mid-infrared region and the far-infrared region. The high pressure platform is built to provide *in situ* infrared and Raman measurements under high pressure and low temperature experimental conditions.

Base on the optical system above, the photon flux, focusing performance, signal-to-noise ratio (SNR) and spatial resolution were tested, results and related measurement methods were reported in detail [9,11]. Briefly, the photon flux at the entrance of spectrometer are 1.9×10^{13} phs/s/0.1%bw@300 mA@1 μm for the infrared microspectroscopy and imaging station, and 1.3×10^{13} phs/s/0.1%bw @ 300 mA@1 μm for the time-resolved infrared spectroscopy station. Furthermore, the photon flux at the entrance of the spectrometer is predicted to range from 2.4×10^{13} phs/s/0.1%bw@300 mA@1 μm to 1.4×10^{11} phs/s/0.1%bw@300 mA@1000 μm.

The synchrotron infrared radiation shows highly collimated nature, considerable brightness and close to theoretical diffraction-limited spatial resolution. Curve of signal to noise ratio (SNR) obtained with the globar source and the synchrotron source (at 230 mA) with different aperture sizes have been reported [11]. The SNR of the synchrotron radiation is two orders of magnitude better than that of the globar source when infrared microscope aperture size is smaller than $15 \times 15\,\mu m^2$. According to the reported SNR, when the aperture size is $5 \times 5\,\mu m^2$, the synchrotron IR source is 350 times better than the globar source [11]. The intensity in arbitrary units of the peak-to-peak interferogram values of the synchrotron and globar with different aperture sizes was shown in Fig. 2. The Nicolet 6700 FTIR spectrometer, Nicolet Continuum Microscope, MCT-A detector and KBr beam splitter were used in the measurement. The peak to peak values were measured in transmission mode.

The spatial resolution of the infrared microscope was measured using the step-edge method, and it was in good agreement with the theoretical diffraction-limited resolution. The spatial resolution in the horizontal and vertical directions are $(0.74 \pm 0.03)\lambda$ and $(0.72 \pm 0.03)\lambda$, respectively.

As shown in Fig. 3, the infrared microspectroscopy and imaging station is equipped with a Nicolet 6700 Fourier transform infrared spectrometer and a Nicolet Continuμm infrared microscope. The spectral range of this station is 600 cm^{-1}–10,000 cm^{-1}, and the spectral resolution is 0.2 cm^{-1}. The infrared microscope is equipped with a 10× visible objective, a 15× and a 32× IR/visible objective. CaF_2 and KBr beamsplitters are equipped in the spectrometer. MCT/A with liquid N_2 cooled and TEC InGaAs detectors are equipped in the Continuμm infrared microscope. MCT/A with liquid N_2 cooled and DTGS detectors are also equipped in the spectrometer. Various accessories are available for different experimental requirements. The spectrometer is purged with dry air to reduce the water and CO_2 absorption from the atmosphere inside the spectrometer and microscope.

The time-resolved infrared spectroscopy station is equipped with a Nicolet 8700 Fourier transform infrared spectrometer with step-scan mode for high-resolution time-resolved investigations. CaF_2, KBr and PE beamsplitters are equipped in the spectrometer for near-, mid- and far-infrared investigations. Multiple detectors are available for the spectrometer. They are the DTGS w/KBr, DTGS w/PE, MCT/A with liquid N_2 cooled, MCT/B with liquid N_2 cooled, Si-bolometer with liquid helium cooled and photodiode MCT detectors. The spectrometer is also purged with dry air to reduce the water and CO_2 absorption from the atmosphere inside the spectrometer and microscope. Various accessories are available for different experimental requirements.

Research in high pressure characterization from synchrotron light source has seen rapid growth over the latest years [12–16]. Specially, synchrotron-based high pressure infrared technique provides unique ability to explore the unknown properties in known matter. Under the collaboration between the Center for High Pressure Science and Technology Advanced Research and BL01B1 beamline, an experimental platform aiming at high pressure and low temperature IR/Raman investigations is being developed. The customized system is located between the two experimental stations. The system contains a Fourier transform infrared spectrometer, a MCT detector, sample stages, two objectives with long working distance, a cryostat, and a Raman system with laser wavelength of 561/660 nm. These inserted devices are compatible with the synchrotron infrared light to provide *in situ* infrared and Raman measurements under high pressure and low temperature experimental conditions. By using this system, *in situ* high pressure synchrotron infrared spectra measurements on organic-inorganic hybrid perovskites were performed and pressure dependence of various mid-IR modes have been investigated [16].

4. Applications

There are various applications of BL01B1 in different research areas

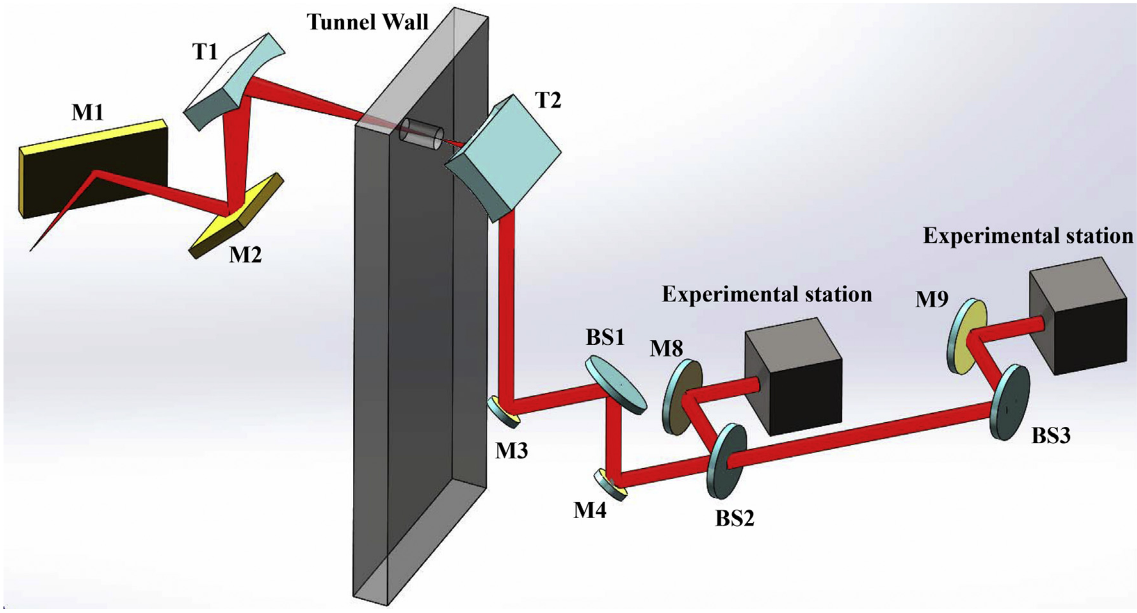

Fig. 1. Optical layout of BL01B1 beamline. M1: extraction plane mirror; M2, M3, M4, M8 and M9: plane mirror; T1 and T2: Toroidal mirror; BS1, BS2, BS3: Beamsplitters.

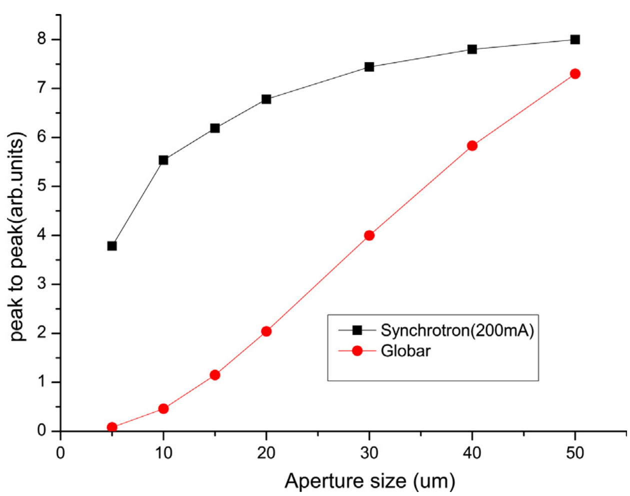

Fig. 2. Intensity of the peak-to-peak interferogram values (Transmission mode) of the synchrotron IR source (at 200 mA) and the internal globar source with different aperture sizes.

including protein science [17–20], cell biology [21,22], environmental science [23,24], pharmaceutical science [25,26], and high pressure science [16,27]. The applications and results obtained under the collaboration of BL01B1 and user groups will be briefly summarized in this section.

Animal silks are known for their excellent comprehensive mechanical properties and have drawn great attention in recent years [17]. Spider and silkworm silks have been model fibers to study the structure-property relationship, silk spinning procedures and the development of regenerated silk fibroin (RSF) fibers. Synchrotron radiation FTIR microspectroscopy has been proved to be an effective way to study the silk protein conformation in a range of single natural silk fiber. Xin Chen et al. studied microscopic structures of animal silks at BL01B1, and built precise correlation of mechanical properties and microscopic structures of animal silk [18]. By controlling the spinning procedure, animal silks with different mechanical properties were obtained. The related β-sheet content, orientation of α-helix, β-sheet and random coil structures were characterized with synchrotron FTIR microspectroscopy. Base on the structure-property relationship in this research, animal silks with anticipated properties can be obtained by controlling the spinning conditions. Several other related works have also been down by Xin Chen et al. [17–20]. Those works give scientists new insights into the animal silks and the development of RSF fibers.

Adipogenesis processes are related to several metabolic diseases and have been investigated by using human mesenchymal stem cells (hMSCs) as an *in vitro* model. Synchrotron radiation FTIR microspectroscopy at BL01B1 was used to study the early stage differentiation of hMSCs at single cell level by Junhong Lü et al. [21]. As a non-destructive and high resolution method to study cell biology, synchrotron radiation FTIR method was used to study the hMSCs characteristics and the spatial distributions of lipid, protein and nucleic acid. The dynamic change of these biology macromolecules in the hMSCs determination stage and the function of hMSCs into adiposities differentiation were identified. This work gave more insight into the understanding of the stem cell fate determination and early lipogenesis events. Another related work about food borne pathogenic bacteria was also reported [22], proving that synchrotron infrared method is an effective way to study bacteria at high resolution level.

Heavy metals in soil have been harmful to the environment and food that come from contaminated land [23]. Studying the interaction of heavy metal with soil dissolved organic matter and their distribution in the soil system are essential for understanding the distribution, reaction and fates of pollutants in the environment. Synchrotron radiation FTIR microspectroscopy measurements of Cd, minerals and organic components in soils were conducted at BL01B1 by Guanghui Yü et al. [23]. Combining with two-dimensional correlation spectroscopy, micro X-ray fluorescence spectromicroscopy and electron probe micro-analyzer mapping, the distribution of these matters and their interaction mechanisms in soil colloids were illustrated. This investigation provided an effective strategy of studying pollutants, minerals and organic components in the environment. Another study of copper and soil dissolved organic matter in soil by using related strategy was also reported [24].

Synchrotron radiation FTIR microspectroscopy has also been used to study pharmaceutical science at BL01B1 by Jiwen Zhang et al. [25]. Osmotic pump system is widely used for controlled release drug delivery system. This system was investigated through recording and mapping the chemical information of the tablet membranes [25]. The

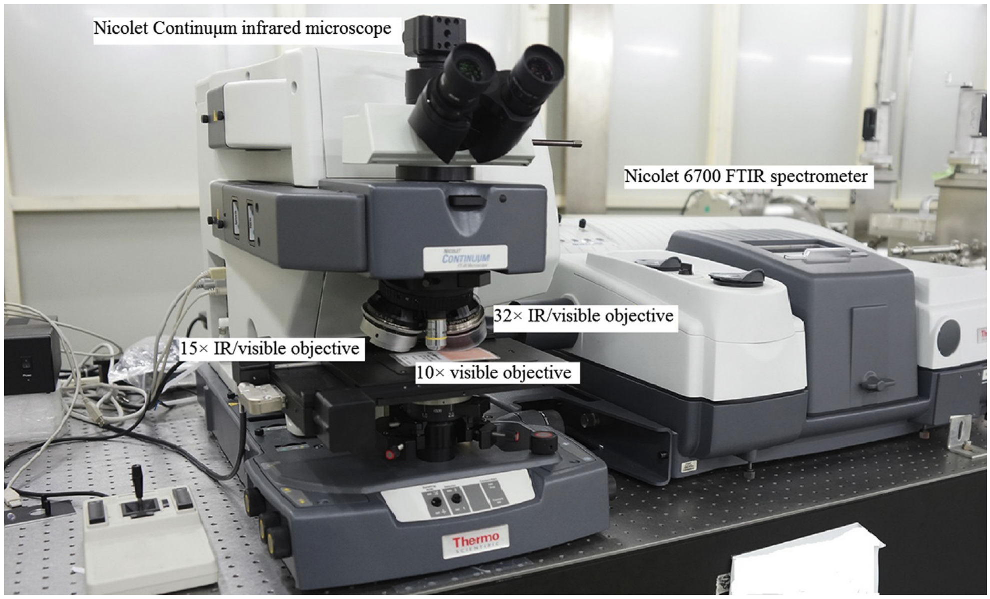

Fig. 3. The infrared microspectroscopy and imaging station.

hydration induced material transfer in the membranes and the dissolution mechanism of important components of the osmotic pump tablets were revealed. This research provided more information about using synchrotron based infrared method to investigate the composition, distribution and material dynamic transfer processes of pharmaceutical science. Distribution of protein/peptide (Exenatide) and excipient (PLGA) in a single microsphere was also studied by Jiwen Zhang et al. using SR-FTIR method [26]. Drug distribution in microspheres has important influence on the release kinetics of proteins from the microspheres. Quantitative distribution of Exenatide and PLGA was revealed, and a strategy of studying distribution of constituents in microspheres was developed.

The organic-inorganic hybrid perovskite materials were studied by users from Center for High Pressure Science and Technology Advanced Research (HPSTAR) [16,27]. A synergistically enhancement in both band gap narrowing and carrier-lifetime prolongation of the organic-inorganic hybrid lead triiodide perovskite materials was reported. Part of the *in situ* high pressure mid-infrared measurements on pressure-driven structural evolution of $MAPbI_3$ were carried out at the BL01B1 beamline.

5. Conclusions

In summary, the BL01B1 beamline of NCPSS at SSRF and its applications are overviewed. The optical layout of the beamline and performance of the photo beam are briefly introduced. The photon flux at the entrance of the spectrometer was measured. The photon beam shows highly collimated nature, considerable brightness and close to theoretical diffraction-limited spatial resolution. The SNR of the synchrotron radiation infrared light is much higher than that of a globar source. Two experimental stations are under operation at BL01B1. They are the infrared microspectroscopy and imaging station and the time-resolved infrared spectroscopy station. The equipment and characteristics of these two experimental stations are illustrated, and they are adequate to conduct high resolution SR-FTIR experiment. A high pressure platform under construction will be used to conduct *in situ* infrared and Raman measurements under high pressure and low temperature experimental conditions. The applications and results obtained under the collaboration of BL01B1 and user groups are reviewed. These applications prove that SR-FTIR is an effective method to study various subjects in protein science, cell biology, environmental science, high pressure science, pharmaceutical science, etc.

Conflict of interest

There are no conflicts of interest.

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References

[1] M.R. Stem, Understanding why researchers should use synchrotron-enhanced FTIR instead of traditional FTIR, J. Chem. Educ. 85 (7) (2008) 983–989.
[2] S.D. Bernardina, F. Alabarse, A. Kalinko, et al., New experimental set-ups for studying nanoconfined water on the AILES beamline at SOLEIL, Vib. Spectrosc. 75 (2014) 154–161.
[3] G. Cinque, M. Frogley, K. Wehbe, et al., Multimode InfraRed Imaging and Microspectroscopy (MIRIAM) beamline at diamond, Synchrotron Radiat. News 24 (5) (2011) 24–33.
[4] P. Roy, M. Rouzières, Z. Qi, et al., The AILES Infrared Beamline on the third generation Synchrotron Radiation Facility SOLEIL, Infrared Phys. Technol. 49 (1–2) (2006) 139–146.
[5] A. Nucara, S. Lupi, P. Calvani, The synchrotron infrared beamline SISSI at ELETTRA, Infrared Phys. Technol. 45 (5–6) (2004) 375–381.
[6] I. Yousef, S. Lefrançois, T. Moreno, et al., Simulation and design of an infrared beamline for SESAME (Synchrotron-Light for Experimental Science and Applications in the Middle East), Nucl. Inst. Methods Phys. Res. A 673 (4) (2012) 73–81.
[7] T.E. May, Infrared facility at the Canadian light source, Infrared Phys. Technol. 45 (5–6) (2004) 383–387.
[8] D. Creagh, J. McKinlay, P. Dumas, The design of the infrared beamline at the Australian synchrotron, Vib. Spectrosc. 41 (2) (2006) 213–220.
[9] T. Ji, Y.J. Tong, H.C. Zhu, et al., The status of the first infrared beamline at Shanghai Synchrotron Radiation Facility, Nucl. Inst. Methods Phys. Res. A 788 (2015) 116–121.
[10] T. Scarvie, N. Andresen, K. Baptiste, et al., Noise reduction efforts for the ALS infrared beamlines, Infrared Phys. Technol. 45 (705) (2004) 403–408.
[11] Z. Zhang, M. Chen, Y. Tong, et al., Performance of the infrared microspectroscopy station at SSRF, Infrared Phys. Technol. 67 (2014) 521–525.
[12] G. Shen, H.K. Mao, High-pressure studies with X-rays using diamond anvil cells, Rep. Progr. Phys. Phys. Soc. 80 (1) (2017) 016101.
[13] A. Voute, M. Deutsch, A. Kalinko, et al., New high-pressure/low-temperature set-up available at the AILES beamline, Vib. Spectrosc. 86 (2016) 17–23.
[14] L. Ehm, M. Vaughan, T. Duffy, et al., High-pressure research at the national synchrotron light source, Synchrotron Radiat. News 23 (3) (2010) 24–30.
[15] G. Shen, Y. Wang, V. Prakapenka, et al., High-pressure research at the advanced photon source, Synchrotron Radiat. News 23 (3) (2010) 32–38.

[16] L.P. Kong, G. Liu, J. Gong, et al., Simultaneous band-gap narrowing and carrier-lifetime prolongation of organic-inorganic trihalide perovskites, PNAS 113 (32) (2016) 8910–8915.
[17] G.Q. Fang, Y.F. Huang, Y.Z. Tang, et al., Insights into silk formation process: correlation of mechanical properties and structural evolution during artificial spinning of silk fibers, Biomater. Sci. Eng. 2 (11) (2016) 1992–2000.
[18] G. Fang, Y. Tang, Z. Qi, et al., Precise correlation of macroscopic mechanical properties and microscopic structures of animal silks – using *Antheraea pernyi* silkworm silk as an example, J. Mater. Chem. B 5 (30) (2017) 6042–6048.
[19] G. Fang, Z. Zheng, J. Yao, et al., Tough protein–carbon nanotube hybrid fibers comparable to natural spider silks, J. Mater. Chem. B 3 (19) (2015) 3940–3947.
[20] G. Fang, S. Sapru, S. Behera, et al., Exploration of the tight structural–mechanical relationship in mulberry and non-mulberry silkworm silks, J. Mater. Chem. B 4 (24) (2016) 4337–4347.
[21] Z.X. Liu, Y.Z. Tang, F. Chen, et al., Synchrotron FTIR microspectroscopy reveals early adipogenic differentiation of human mesenchymal stem cells at single-cell level, Biochem. Biophys. Res. Commun. 478 (3) (2016) 1286–1291.
[22] Y.D. Wang, X.L. Li, Z.X. Liu, et al., Discrimination of foodborne pathogenic bacteria using synchrotron FTIR microspectroscopy, Nucl. Sci. Tech. 28 (4) (2017) 38–43.
[23] F. Sun, M.L. Polizzotto, D. Guan, et al., Exploring the interactions and binding sites between Cd and functional groups in soil using two-dimensional correlation spectroscopy and synchrotron radiation based spectromicroscopies, J. Hazard. Mater. 326 (2017) 18–25.
[24] F.S. Sun, Y.Q. Li, X. Wang, et al., Using new hetero-spectral two-dimensional correlation analyses and synchrotron-radiation-based spectromicroscopy to characterize binding of Cu to soil dissolved organic matter, Environ. Pollut. 223 (2017) 457–465.
[25] L. Wu, X.Z. Yin, Z. Guo, et al., Hydration induced material transfer in membranes of osmotic pump tablets measured by synchrotron radiation based FTIR, Eur. J. Pharm. Sci. 84 (2016) 132–138.
[26] M. Wang, X. Lu, X. Yin, et al., Synchrotron radiation-based Fourier-transform infrared spectromicroscopy for characterization of the protein/peptide distribution in single microspheres, Acta Pharm. Sinica B 5 (3) (2015) 270–276.
[27] G. Liu, J. Gong, L. Kong, et al., Isothermal pressure-derived metastable states in 2D hybrid perovskites showing enduring bandgap narrowing, PNAS 115 (32) (2018) 8076–8081.