Construction of an Imaging Measurement System for Material Property Change under Controlled Mechanical Stress

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Material strength measurements have been performed by combining mechanical material characterization and lattice diffraction experiments using hard X-rays.[1)] For example, if X-ray diffraction experiments are performed while tensile tests are performed, plastic deformation due to stress can be analyzed at the lattice level. While this is a very powerful material characterization technique for materials such as metals, it does not provide information on molecular state changes and stress-induced electronic state transitions in polymer materials, for example, polymers and pressure phase transition barrow plastics. Combined with a measurement system in the energy band involved in chemical bonding, it would advance our understanding of adhesion states such as interfaces in polymer materials and composites.

Therefore, in this study, we developed a system aimed at implementing a Raman spectroscopy imaging system and a stress control system on a soft X-ray absorption spectroscopy and photoelectron spectroscopy microscope. Figure 1 shows a conceptual diagram of the system. In this example, a MEMS with a cantilevered beam structure is implemented in a Raman spectroscopic imaging and synchrotron radiation (SR) analysis system. In the following, two examples are shown.

The first is an example in which Raman spectroscopy is performed while tensile stress is applied to PTFE using the tensile test system in Fig. 2(a). In this case, although MEMS is not used, it is possible to apply a larger stress compared to MEMS. 785 nm excitation light Raman spectroscopy results are shown in Fig. 2(b) just before the PTFE ruptures. Under the applied stress, the peak at 1352 cm^{-1} increases and a new peak structure appears around 730 cm^{-1}. This new peak is attributed to CF_2. This means that the bonding state of the side chain becomes visible when it is pulled.

Second, MEMS with a cantilever structure was introduced to SR photoemission electron microscopy. Figure 2(c) shows a conceptual diagram of the PEEM holder

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[1] J. Shin, T. W. Cornelius, S. Labat, F. Lauraux, M. –I Richard, G. Richer, N. P. Blanchard, D. S. Gianola, and O. Thomas, J. Appl. Cryst. **51**, 781 (2018).

and MEMS. Figure 2(d) presents a plot of the voltage-induced curvature of the introduced MEMS as the focal point of the PEEM of BL17SU at SPring-8. As shown in Figs. 2(e) and 2(f), applying voltage to the MEMS does indeed change the focal distance due to the curvature of the cantilever beam. It was shown that the MEMS can be driven in this voltage range with little hysteresis.

We have succeeded in constructing a system for measuring the change in physical properties imaging under mechanical stress control. This mechanism makes it possible to quantitatively reveal chemical bonding states and electronic state displacements under stress application.

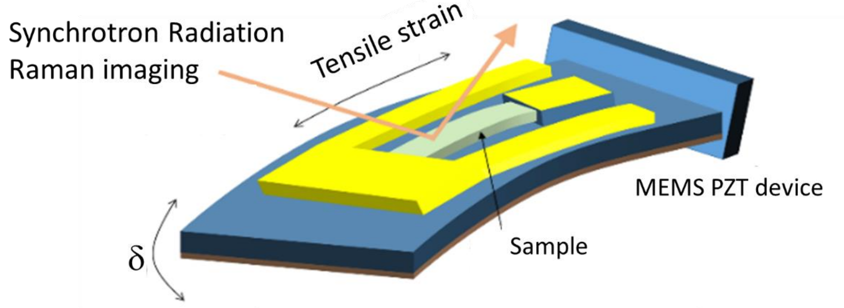

Figure 1: Schematic of operando system combined with synchrotron radiation analysis or Raman imaging and MEMS PZT device that can control tensile strain.

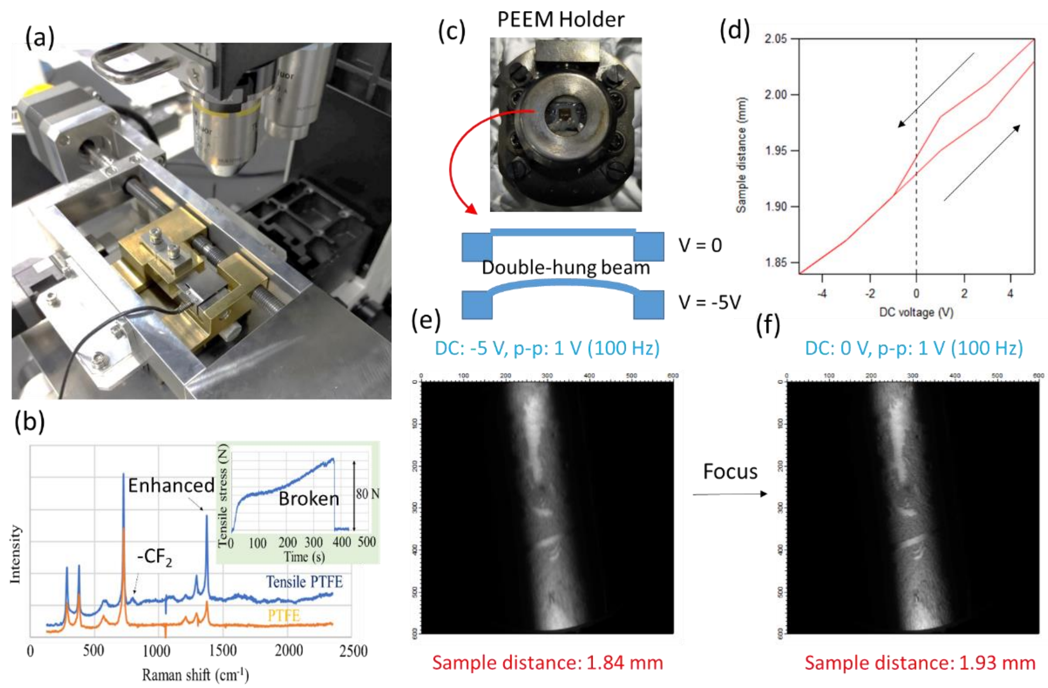

Figure 2: (a) Optical photograph of micro Raman imaging system combined with tensile strain measurement. (b) Raman spectra of PTFE with/without tensile strain. (c) PEEM-Holder of BL17SU at SPring-8. MEMS device was mounted on it. (d) DC voltage dependence of Sample focus point of PEEM. PEEM observations of surface of double-hung beams on MEMS PZT device under the application of DC voltages of (e) -5 and (f) 0 V, respectively.