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In Situ Synchrotron X-ray Diffraction Technique for Contact Surfaces

Kazuyuki Yagi*

*Kyushu University

1. Introduction

The contact area with relative motion involves many complex phenomena such as friction, wear, noise, vibration, heat generation. To understand these phenomena occurring in the contact area it is necessary to extract information from the contact area. However, direct access to the contact area is difficult because it is surrounded by solid surfaces, which is equivalent to an enclosed space. One of methods to overcome this problem is to replace one or both of surface materials is replaced with transparent material. My research group developed a new in situ analysis method to understand the behavior of grain structures of solid surfaces using synchrotron X-ray diffraction (XRD) analysis[1-4]. In this presentation, in situ observation system and results will be introduced.

2. Experimental

Figure 1 shows the schematic diagram of a special friction test rig for synchrotron X-ray diffraction technique. This test rig creates a contact area between a rotating sapphire ring and a stationary metal pin. An in situ observation system comprising of microscope, a visible camera and a near infrared camera is installed above the friction test rig to capture visible and near infrared images of the contact area. From the side of the test rig, X-ray beam is shone into the contact area. A two- dimensional detector is installed at the opposite side to the entrance of the X-ray beam to capture in situ diffraction patterns. The developed in situ analysis system could obtain time resolved XRD spectra of the contact area during tests.

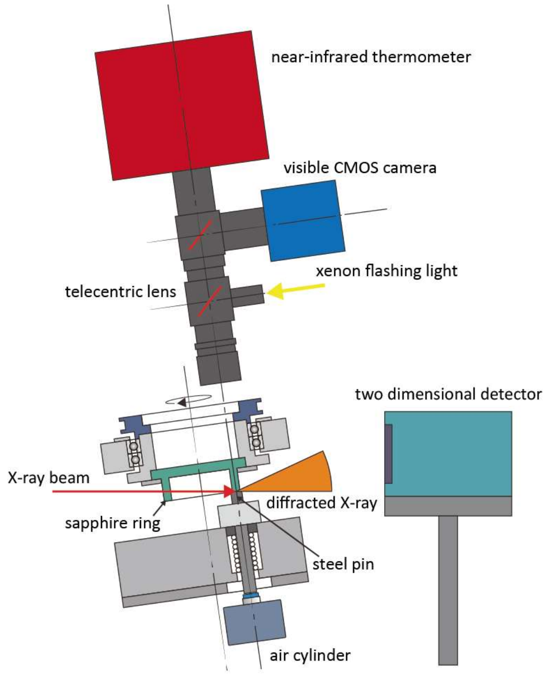

Fig. 1 Schematic diagram of in situ synchrotron X-Ray diffraction system[3].

3. Results

Figure 2 shows an example of sets of visible images, near infrared images, and XRD spectra. The sliding direction of the sapphire ring is left to right. At t = 1.1 s, wear debris accumulated at the contact area. The strongest peak of the XRD spectrum came from martensite of bulk grain structure. At t = 1.133 s, plastic flow appeared with a flat spot. In the near infrared image, a high temperature of 1168 °C was observed at the same location of the flat spot. The XRD spectrum indicated a slight increase in austenite peak. At t = 1.700 s and 1.800 s, austenite peaks (111) and (200) grew up while martensite peak (110) decreased. In the contact area high temperature rise occurred at plastic flow spots.

Figure 3 shows a transition in the Debye–Scherrer ring during a scuffing test. At t = 0.300 s, the ring of martensite (110) appeared with a uniform distribution in the circumferential direction. At t = 8.000 s and 20.000 s, the ring of austenite (111) was evolved because of plastic flow. In the ring the intensity was stronger at the left side than that in the right side. At t = 30.000 s, only the ring of martensite (110) remained because plastic flow disappeared, and austenite crystal grains were cooled to return to BCC structure. The non-uniform distribution, which was observed in the ring of austenite at t = 8.000 s and 20.000 s, remained. This implies that alignment of crystal grains occurred in the sliding direction because of plastic flow observed in Fig. 2.

Figure 4 shows variations in XRD spectrum during a friction test. In this friction test, pure polysulfide was used as lubricant to investigate the evolution of chemical reaction films arising from additive in friction. Many small peaks can be seen in the XRD spectra besides steel. The intensity of these peaks was much smaller than that of steel because these films are thin. The evolutions of oxide iron of Fe_2O_3 and Fe_3O_4 and iron sulfide of FeS and FeS_2 occurred during friction.

4. Summary

The in situ synchrotron X-ray diffraction technique system developed in my research group found interesting phenomena occurring in contact areas. The in situ observation system could capture plastic flow with high heat generation, phase transformation and alignment of crystal grains of steel surface. It also succeeded in capturing in situ the evolution of chemical reaction films. The developed in situ observation system would contribute a better understanding of tribological phenomena.

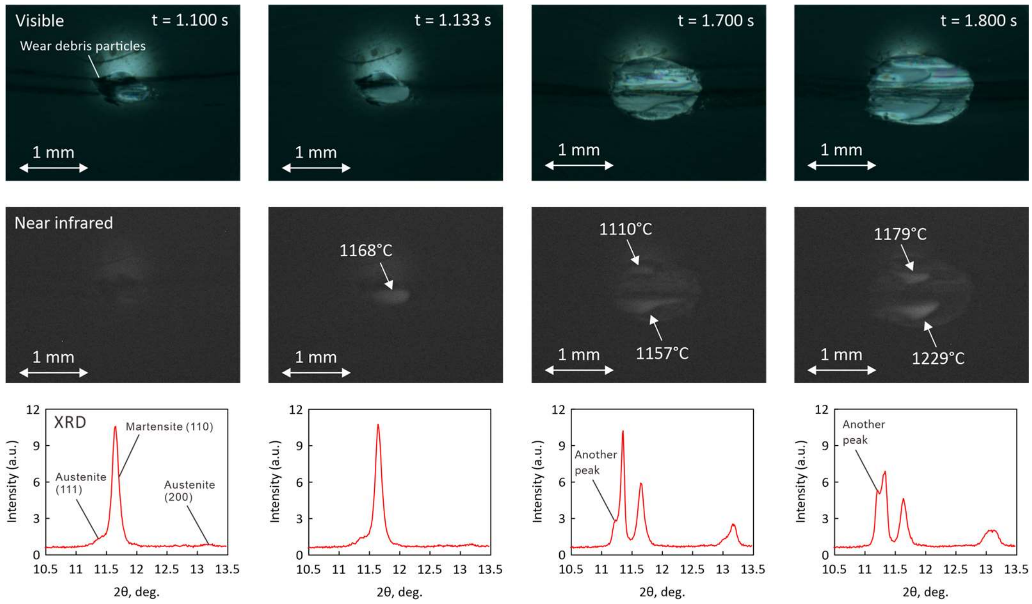

Fig. 2　Variations in contact area and XRD spectrum with exposure time of 30 ms in friction test[2)]

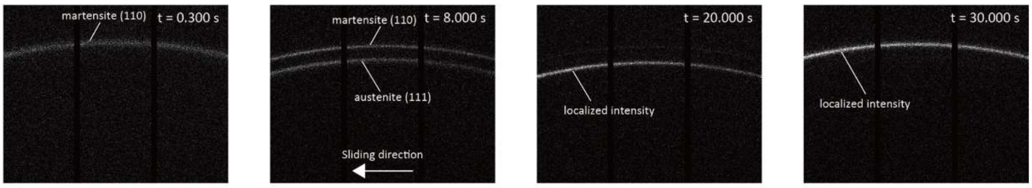

Fig. 3　Transition in the Debye–Scherrer ring with exposure time of 30 ms in friction test[3)]

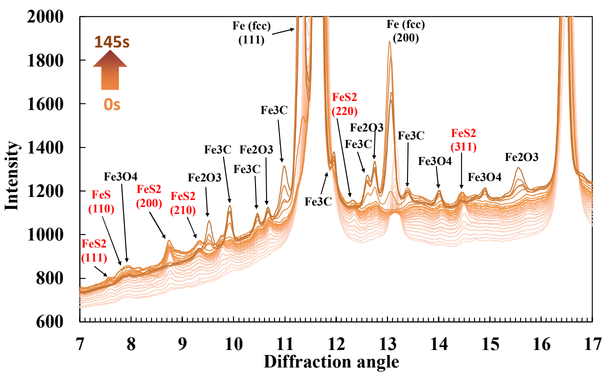

Fig. 4　In situ XRD spectra of evolved chemical reaction films with exposure time of 6 s in friction test[4)]

Acknowledgements

In situ synchrotron X-ray diffraction technique for contact surfaces was developed in collaborative research with TOYOTA Motor Corporation and TOYOTA CENTRAL R&D LABS., INC.. The friction tests for Figs. 2 and 3 were conducted at the Toyota beamline BL33XU of SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2013B7021 and 2014B7021). The friction tests for Fig. 4 were conducted at BL13XU supported by JSPS KAKENHI Grant number 22H00188 with the approval of JASRI (Proposal No. 2022B0574 and 2024A1592).

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