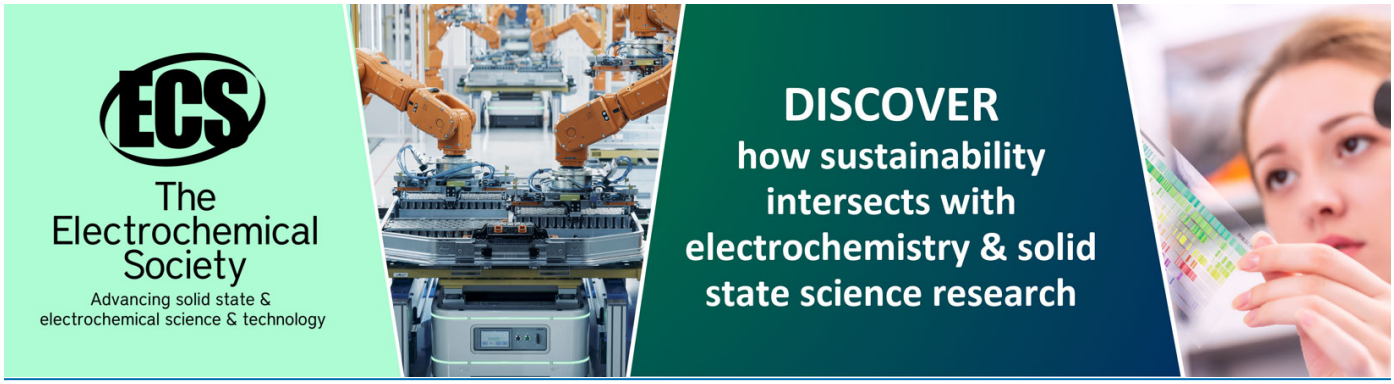
Micromanipulation and Pick-Up System for X-Ray Diffraction Characterization of Micrometer-Sized Single Particles



Journal of Physics: Conference Series **502** (2014) 012008 doi:10.1088/1742-6596/502/1/012008

Micromanipulation and Pick-Up System for X-Ray Diffraction Characterization of Micrometer-Sized Single Particles

Y Takeichi[1], N Inami[1], T Ueno[2], K Saito[1], H Otori[1], R Sagayama[1], R Kumai[1] and K Ono[1]

[1] Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan
[2] National Institute for Material Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

E-mail: yasuo.takeichi@kek.jp

Abstract. We describe a micromanipulation and pick-up system for preparing a micrometer-sized single particle for X-ray diffraction characterization. Combining a microgripper based on microelectromechanical systems, piezo-motor-driven linear stages, and a gamepad, the system provides precise and intuitive handling of the object. Single-crystal X-ray diffraction measurements of Sm-Fe-N permanent magnet were performed using this system. We also describe a method to distinguish crystallographically homogeneous particles found in powder-form samples.

1. Introduction

Powder X-ray diffraction measurement using synchrotron radiation provides fruitful information about the crystal structure and chemical composition of materials [1]. Rietveld analysis of the obtained diffraction pattern is the common method to determine lattice structures of the samples. In some cases, however, the Rietveld analysis does not converge well; for example, the Debye-Scherrer rings become discontinuous and spot-like if the number of particles included in the investigated volume is insufficient. It becomes more difficult to obtain a well converged structure when the sample material has a preferred orientation. Powder-form ferromagnetic samples with strong magnetic anisotropy are a case in point. To deduce the lattice structure of those materials, single-crystal X-ray diffraction measurement is necessary.

When the particle size is of 100 μm or more, one can separate and prepare a single sample specimen for measurement by hand using micro-handling tools [2] while viewing them with a stereoscopic microscope. For smaller particles, however, direct manipulation by hand is no longer applicable and micromanipulation requires the help of electronic devices.

Recently, the commercial use of micrometer-sized fabrication and motion technology has been widely extended; microelectromechanical systems (MEMS) are available for sample handling, as well as piezo-motor-driven stages for precise motion of the tool and/or the sample. Today, they are commercially available at reasonable prices. We have combined some devices to develop a micromanipulation and pick-up system. For use in routine X-ray diffraction measurements, easy and intuitive control of the system is desirable.

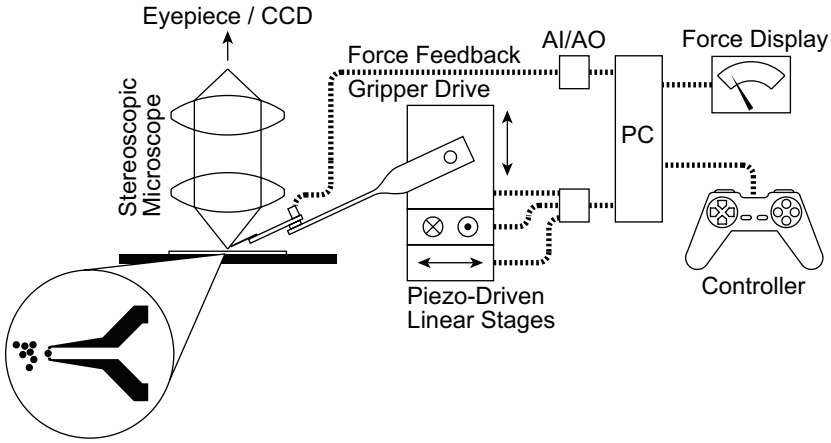


Figure 1. Schematic of the micromanipulation and pick-up system.

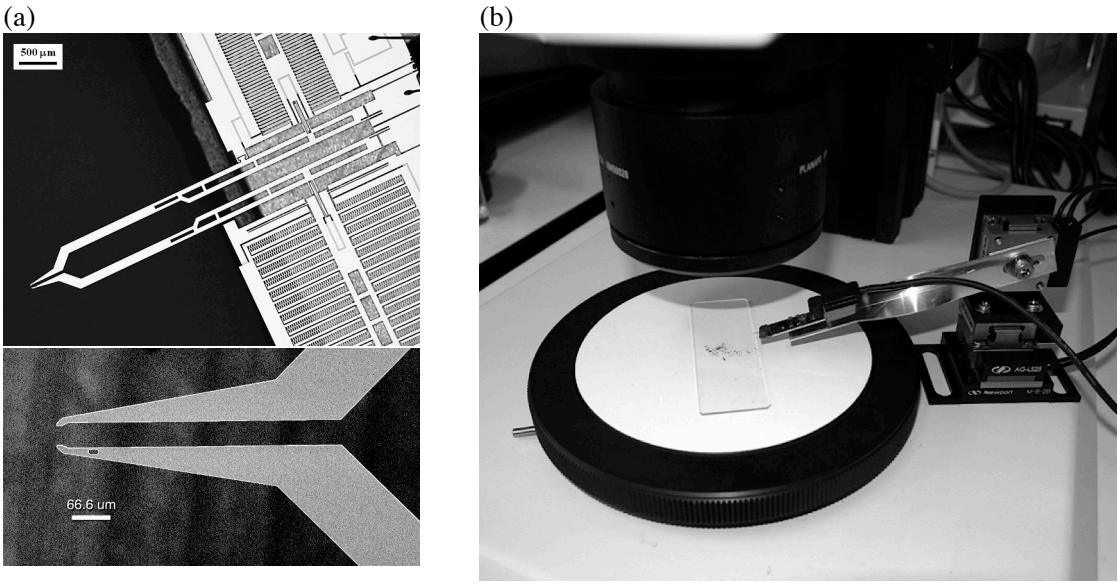


Figure 2. (a) The upper panel shows a microscopic image of the microgripper FT-G32; The scale bar is 500 μm. The lower panel shows a SEM image of the gripper tip; the scale bar is 66.6 μm. (b) Photograph of the micromanipulation and pick-up system. The sample powder is dispersed on the microscope slide.

2. Micromanipulation and pick-up system

Figure 1 shows a schematic of our micromanipulation and pick-up system. MEMS-based microgrippers (FemtoTools FT-G series [3]) are used for sample handling. The microgripper can grasp a particle up to 30 μm (FT-G32, see figure 2(a)) or up to 100 μm (FT-G102). Both the models of the gripper are equipped with gripping force sensors. The gripper is mounted on an XYZ stage composed of Newport Agilis piezo-motor-driven linear stages. They are driven at a wide range of speeds, up to 0.5 mm/s and down to incremental sub-micrometer motions. The operating program, based on National Instruments LabVIEW, controls the analog in/out for the gripper drive and force sensor, and the XYZ stage via a USB connection. All motions of the gripper and the stages are intuitively controlled using a commercial gamepad. The motion of the gripper is monitored with a stereoscopic

microscope (Leica M205 C) with magnification of up to 320 times. The microscope image is also monitored with a CCD camera connected to a PC via a Gigabit Ethernet.

To pick up a single particle from the powder-form sample, the powder should be first dispersed on a microscopic slide, as seen in figure 2(b). An organic solvent is often useful in sparsely dispersing dense powder. Next the gripper is slid carefully up to the object particle by moving the XYZ stage. Steric observation through the eyepiece is essential to control the gripper precisely. After gripping the particle, a constant-force gripping mode is available using force feedback control. This helps in holding soft or fragile materials such as biological cells. Finally, the particle can be mounted on polymer MicroMounts [2] or onto the tip of a glass needle for X-ray diffraction measurements.

3. X-ray diffraction measurement

A single-particle X-ray diffraction measurement of powdered samples was performed using the system described above. The sample was a Sm-Fe-N rare-earth permanent magnet [4] which has strong uniaxial magnetic anisotropy. First, a powder X-ray diffraction measurement of this sample was performed. However, the Rietveld analysis of the obtained two-theta diffraction patterns (not shown here) did not converge well, possibly owing to the preferred orientation caused by magnetostatic interactions between the particles.

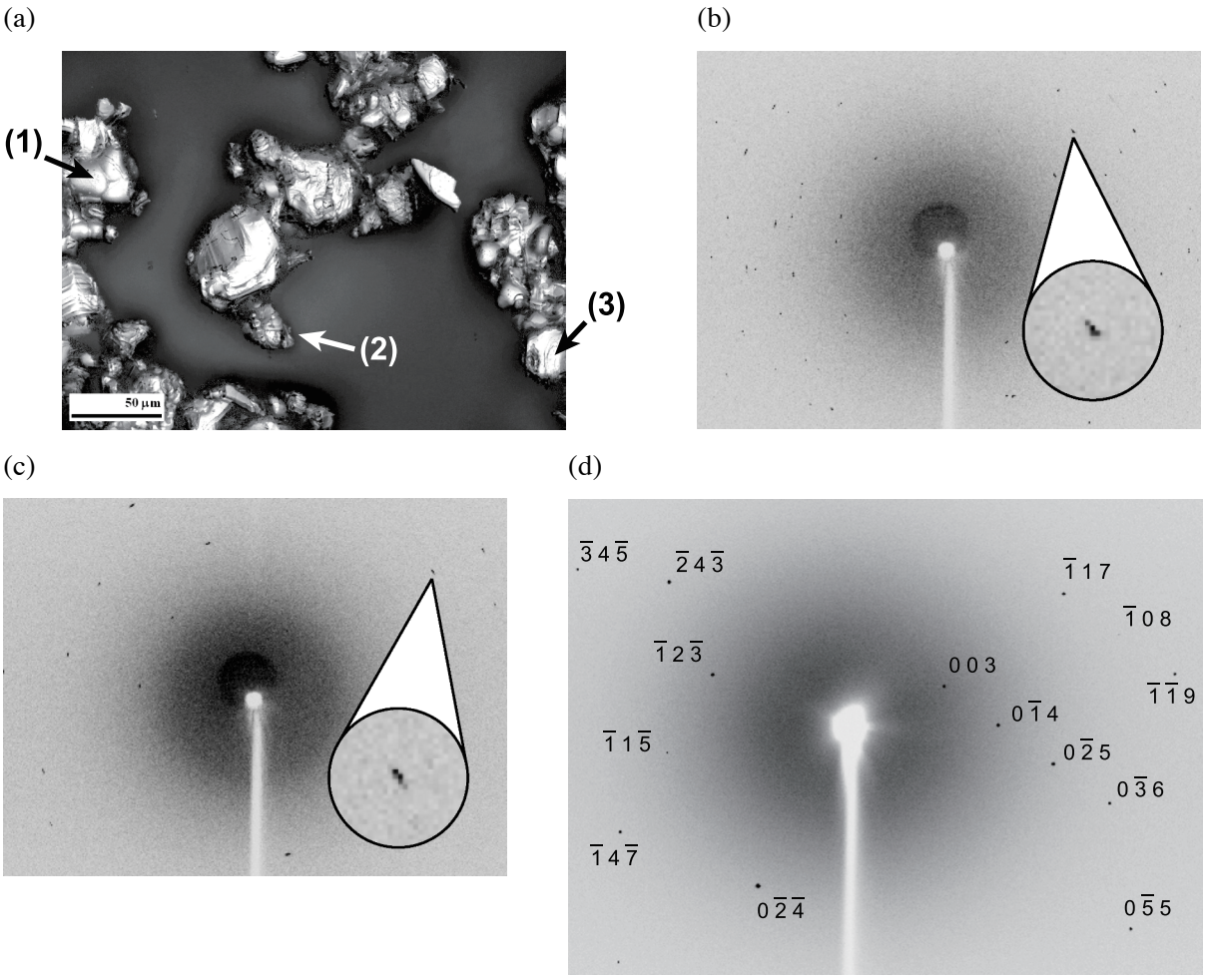


Figure 3. (a) Morphology of Sm-Fe-N powder observed with optical microscope. The scale bar is 50 μm. (b–d) are the X-ray diffraction patterns of (b) a polycrystalline Sm-Fe-N particle including subspots, (c) a particle consisting of a twin of single crystal grains, and (d) a single crystal particle.

Figure 3(a) shows a microscopic image of the sample. The particle size mainly ranges from 20 to 30 μm, but most of the main particles are accompanied with smaller ones of size ~10 μm. To sparsely disperse the particles sticking to each other, vacuum grease was used as solvent. After successful pick-up the sample using the FT-G102 gripper, it was stuck onto a glass needle.

X-ray diffraction measurements were performed at BL-8A/B of the Photon Factory, Japan. The X-ray energy was set to 18 keV. A careful examination of the diffraction pattern on the imaging plate reveals crystallographic homogeneity of the measured particle. Figure 3(b) shows the diffraction spots accompanied with weaker subspots obtained from the large particle (> 30 μm) marked as region (1) in figure 3(a). The particle marked as region (2) exhibited arrays of two or more distinct spots, as shown in figure 3(c). Those particles were deduced to be polycrystalline.

Figure 3(d) shows the diffraction pattern of a particle of size ~20 μm (region (3) in figure 3(a)) after repeated pick-up and measurement trials. All the observed spots were indexed in an analysis using results of successive measurements from rotating the sample. We therefore concluded that the single-crystal X-ray diffraction measurement of this material was performed successfully. The method described above enables us to analyze crystallographic structure more precisely than that reported in previous literature using the powder diffraction method [5, 6]. The crystal structure obtained from the present results will be described elsewhere [7].

4. Conclusion

We have developed a micromanipulation and pick-up system for X-ray diffraction measurements. The system enables us to pick up a single particle from powder-form samples of varying sizes from several to 100 μm. We have succeeded in distinguishing the crystallographic homogeneity of the ferromagnetic particles. The first results of single-crystal X-ray diffraction measurements of Sm-Fe-N permanent magnet was presented.

5. Acknowledgement

The part of this work is supported by the Elements Strategy Initiative Center for Magnetic Materials under the outsourcing project of MEXT. The authors thank Sumitomo Metal Mining Co. Ltd. for providing the samples.

References

[1] Will G 2006 *Powder Diffraction: The Rietveld Method and the Two-Stage Method* (Berlin: Springer)
[2] MiTeGen LLC, http://www.mitegen.com
[3] Vijayasai A P, Sivakumar G, Mulsow M, Lacouture S, Holness A and Dallas T E 2010 *Rev. Sci. Instrum.* **81** 105114
[4] Coey J M D and Sun H 1990 *J. Mag. Mag. Mat.* **87** L251
[5] Sun H, Coey J M D, Otani Y, Hurley D P F 1990 *J. Phys.: Condens. Matter* **2** 6465
[6] Katter M, Wecker J, Kuhrt C, Schultz L, Grössinger R 1992 *J. Mag. Mag. Mat.* **117** 419
[7] Inami N, Takeichi Y, Ueno T, Saito K, Sagayama R, Kumai R, Ono K, accepted to *J. Appl. Phys.*