DETECTION OF VERY SMALL AMOUNT OF ASBESTOS BY SYNCHROTRON X-RAY POWDER DIFFRACTION

Asbestos is well recognized as a health hazard and is highly regulated. An estimated 1.3 million employees in construction and general industry have faced significant asbestos exposure on the job. Heaviest exposures occur in the construction industry, particularly during the removal of asbestos in renovation or demolition. Employees are also likely to be exposed during the manufacture of asbestos products (such as textiles, friction products, insulation, and other building materials) and while carrying out repair work on automotive brakes and clutches.

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In Japan, asbestos products are defined as products that include asbestos at more than 1.0 weight %. In view of the present situation, laws regarding asbestos regulation have been revised recently. In particular, the standard concentration of asbestos was reduced from 1.0 to 0.1 weight %. Thus far, phase contrast microscopy and laboratory X-ray diffraction have been used together for qualitative and guantitative analyses, respectively. However, the detection limit of asbestos by laboratory X-ray diffraction is considered to be in the range of 0.5-1.0 weight %. That is to say, laboratory X-ray diffraction is insufficient as a quantitative analysis method for the revised standards.

Here, we improved the detection limit for the amount of asbestos by synchrotron X-ray powder diffraction. Samples spray painted with asbestos that was filtered through filter paper after being concentrated were used for the analyses. The samples for analytical use took the form of films (Fig. 1). Accordingly, surface diffractometry using a 2dimensional detector was optimized as illustrated in



Fig. 1. Asbestos sample on filter paper.

Fig. 2. The experiment was carried out at the powder diffraction beamline **BL02B2**. We selected chrysotile (chemical formula: $Mg_3(Si_2O_5)(OH)_4$) from three types of asbestos as the sample for analytical use, because this is most difficult to detect by diffraction owing its poor crystallinity and because it is the only asbestos licensed to be manufactured.

First, we measured samples with three different concentrations of asbestos (0.1, 0.3 and 0.7 weight %) to evaluate the potential of synchrotron X-ray surface diffractometry for quantitative analysis. The arrows in Fig. 3 shows 002 peaks diffracted from each sample. By using a 2-dimensional detector, namely an imaging plate (IP), a whole powder pattern can be collected in 1 minute. The integrated intensity of the 002 reflection is proportional to the concentration of asbestos. The result shows that the synchrotron X-ray has a sufficient ability to detect 0.1 weight % asbestos. To determine the detection limit for the amount of



Incident X-ray

Fig. 2. Surface diffractometry using a 2-dimensional detector for analytical use.



asbestos, we measured 0.02 mg of chrysotile, which corresponds to 0.02 weight %. As a result, an X-ray exposure time of only 5 seconds enabled us to detect asbestos as shown in the left-hand side of Fig. 4. The 1 minute data in the right-hand side of Fig. 4 showed adequate counting statistics for quantitative analysis. Thus, we have demonstrated the potential of synchrotron X-ray powder diffraction in the fields of not only advanced materials science but also routine analytical research [1-5].

On the other hand, additional time was required for sample exchange, sample position alignment and IP exchange/readout, which takes more than ten times as long as the measurement. This point prevented us from promoting the analytical research using synchrotron radiation. To overcome this bottleneck, we are now developing an auto sample exchanger, an image recognition system and an X-ray detector to achieve a high-throughput of synchrotron X-ray powder diffraction [6]. I believe that this system should contribute to materials science research as well as analytical research.



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