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Laboratory-modified powder diffraction specimen holder for filter paper

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A simple modification to an existing powder diffraction specimen holder is given to allow for the presentation of filter papers for analysis. A mild steel annulus holds the filter paper in place using magnets added to the rear of the specimen holder.

1. Introduction

The measurement of powders deposited on filter papers is often a key analysis in health science for the determination of respirable silica and other airborne dust (National Institute for Occupational Safety and Health, 2003). The dust can be extracted from the filter papers by dissolving or decomposing the filter paper, and then measured by mounting the resultant powder on a low-background holder; but this runs the risk of loss of sample and, in the case of quantification, difficulty in calibration. When using the filter paper directly, it must be held in place in some manner to allow for specimen holder loading and spinning during data collection.

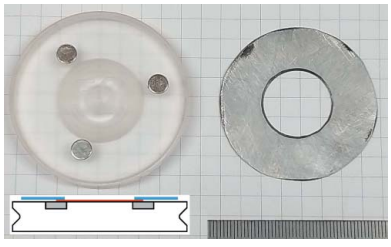
Purpose-built specimen holders are available from instrument vendors, but these can be costly. Outlined here is a simple modification to existing specimen holders to allow the presentation of filter papers in an inexpensive fashion.

2. Specimen holder modification

Specimen holders for a Bruker D8 diffractometer were used. These holders are 8.5 mm high, have an outer diameter of 51.5 mm and are made of polymethyl methacrylate. The front has a 1 mm-deep, 25 mm-diameter hole for the loading of powders, with the back of the holder being completely flat. The holders are otherwise symmetric front-to-back.

Fig 1 shows the modification and annulus. Three flat-bottomed, 2.5 mm-deep, 6 mm-diameter holes were drilled evenly around the back of the specimen holder, approximately 16 mm from the centre of the holder. Three 6 × 2 mm rare-earth magnets (Baledonia Tools; Timbecon) were press-fit into these holes. An annulus was made from squares of 0.8 mm-thick mild steel by drilling an inner hole of 20 mm, and then grinding the outer diameter to approximately 45 mm. The filter paper is then mounted by trapping it between the annulus and the surface of the specimen holder. The difference in outer diameter between the annulus and sample holder is required in order to not interfere with the sample holder loading and data collection processes.

As the filter paper is mounted on the surface of the specimen holder, some specimen displacement is inevitable, but it is believed that this small effect is offset by the simple



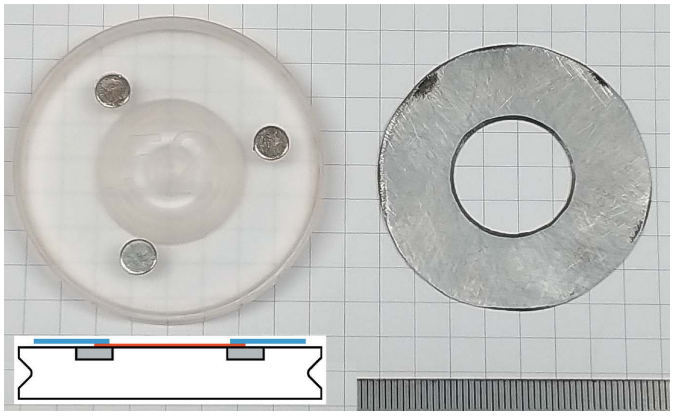


Figure 1
Back of the modified specimen holder, showing the press-fit rare-earth magnets and mild-steel annulus. The filter paper is placed in the centre of the holder and the annulus is placed on top. The inset shows the positions of the annulus, filter paper and specimen holder, the vertical axis is not to scale. The focussing circle is tangent to the top of the specimen holder, not the filter paper. The divisions in the ruler are 1 mm.

nature of the specimen holder construction. If it is necessary to remove all displacement errors, then a shallow (∼0.1 mm) depression could be machined in the rear of the sample holder to accommodate the filter paper thickness. The modification of the specimen holders does not affect their use as standard holders, which allows many to be modified without impacting on the availability of holders for other users.

3. Example application

These holders were used to collect calibration data for the determination of respirable silica (National Institute for Occupational Safety and Health, 2003; National Bureau of Standards, 1983) using a Bruker D8 Discover diffractometer with an Fe-filtered Co tube. The main analyte peak was found to be at 31.11° 2θ, which corresponds to a specimen displacement of approximately 0.1 mm and is consistent with the thickness of the filter paper.

Acknowledgements

The diffraction data were collected using the XRD instrumentation (ARC LE170100199) at the John de Laeter Centre, Curtin University.

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