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ABSTRACT

An attachment has been developed for x-ray diffractometer systems equipped with a domed stage when using a 2D or 1D detector. It consists of a single screen in front of the detector positioned such that it blocks diffraction from the dome. This results in measured data free of disturbing spurious peaks and background, thereby greatly facilitating further data analysis. Its working principle is universally applicable and allows for all specimen orientation movements needed for x-ray diffraction measurements, including texture, stress, and mapping.

I. INTRODUCTION

For x-ray diffraction *in situ* experiments at non-ambient temperatures, under vacuum, and in gas or humid atmospheres, a domed stage is a versatile tool.

Hemisphere dome-shaped housings generally consist of graphite, PEEK, or beryllium. They combine a gastight enclosure with x-ray transparency over the complete half space above the specimen, thereby giving access to the diffracted beam in all directions. By introducing the dome in the x-ray beam path, it is unavoidable that it produces its own diffraction and background (dome diffraction) at the position where the incident primary beam penetrates the dome.

Figure 1 shows the measured dome diffraction patterns of PEEK and graphite domes. In theory, the contribution of the dome, i.e., acquired by measuring the dome diffraction pattern without a specimen, can be subtracted from the measured diffraction pattern of a specimen with the dome.[1] In practice, however, this procedure is not straightforward because the dome diffraction pattern is likely to be dependent on diffraction geometry and experimental process parameters. The absence of dome diffraction makes corrections superfluous, which is to be preferred.

For classical (focusing) geometries with 0D (point) detectors, air scatter from the incident beam and dome diffraction is effectively reduced by inserting an anti-scatter receiving slit in the diffracted beam path: only the diffraction from the irradiated area on the specimen is seen by using the detector.

However, for geometries using 2D (area) or 1D (linear) detectors, it is not possible to insert such a slit because the whole relatively large detector entrance must be kept free to receive diffraction.

In those cases, diffraction from the dome can reach the detector unhindered and is superimposed on the diffraction pattern of the specimen. The resulting peak overlap and increased background level are highly unwanted.

A solution could be a screen positioned perpendicular above the diffractometer center, known as a knife-edge.[2,3] In case a dome is present, two options are possible:

Option 1: a knife-edge in the form of a screen placed on top of the dome is not effective in the 2θ region where dome diffraction is most prominent, as can be derived using Fig. 2.

Option 2: a knife-edge in the form of a screen placed inside the dome above the specimen stops dome diffraction, as can be derived using Fig. 2. An example of an application is given in Ref. 4. For measurements in the very low 2θ range, it could be combined with our presented attachment.

However, a screen inside a dome has some disadvantages as follows: (i) its position in height depends on specimen thickness; (ii) in a hot stage, it has to withstand high temperatures; (iii) mounting and alignment are difficult; and (iv) being fixed to the stage, rotation and translations are limited.

In this paper, a simple and universally applicable attachment is presented, further called *Dome Diffraction Screen* or DDS, which

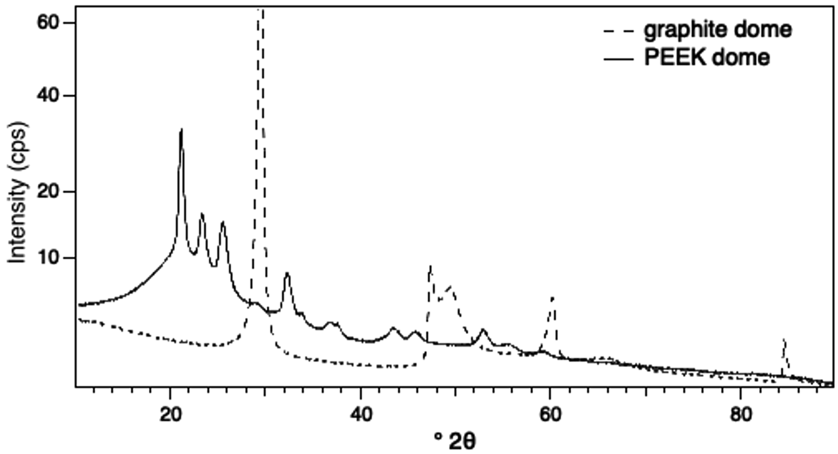

FIG. 1. Diffraction patterns of a PEEK and a graphite dome at room temperature for the CuKα wavelength. Measured with the system described in Sec. III using a Si⟨510⟩ wafer as a specimen, producing no reflections and background. Intensities on square root scale.

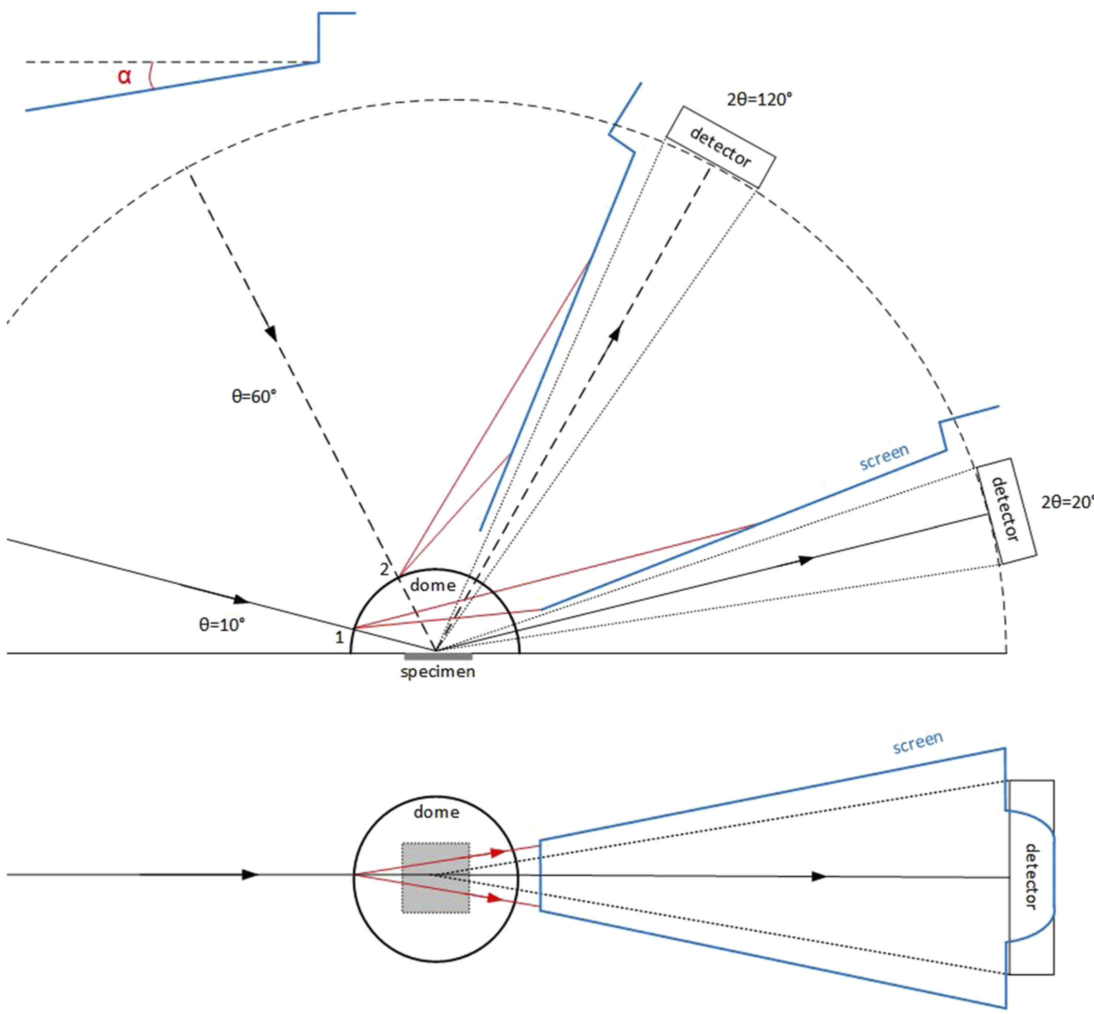

FIG. 2. Top: schematic symmetric diffraction geometry in the equatorial plane for $2\theta = 20°$ and 120°. The drawing with detector opening 38 mm serves as a tool to determine the dimensions and orientation of the DDS in this plane. Upper left inset: cross section of DDS showing deflection angle α (not to scale). Bottom: schematic diffraction geometry in the axial plane for $\theta = 0°$. The drawing with detector opening 77 mm serves as a tool to determine the dimensions of the DDS in this plane. For both drawings, dome diffraction is shown in red and DDS or screen is shown in blue.

prevents dome diffraction and incident beam air scatter reaching the detector. The DDS lacks the above-mentioned disadvantages of a screen inside the dome, which makes it efficient for all general purpose x-ray diffraction measurements.

II. EXPERIMENTAL

The construction and dimensions of the DDS are based on the geometry of our 2D diffractometer system, consisting of a Bruker D8 Discover θ/θ diffractometer with radius 225 mm, an Eiger-2500k 2D detector in gamma orientation, and an UMC 1516 Eulerian cradle with an XYZ sample stage. The x-ray source is an Incoatec IμS microfocus tube with a Montel mirror producing a highly parallel beam with a maximum diameter of about 1 mm and CuKα wavelength.

An Anton Paar DHS 1100 Domed Hot Stage[5] with the PEEK or graphite dome with an inner diameter of 56 mm and a wall thickness of 0.25 mm is mounted on the platform of the Eulerian cradle.

A. Design principle

The top drawing in Fig. 2 shows the beam geometry with the dome and detector in the equatorial plane for $2\theta = 20°$ and 120° in symmetric θ/θ geometry. The DDS is indicated as a screen. Figure 2 illustrates that diffraction from the specimen can reach the detector unhindered, while dome diffraction from positions 1 and 2 is stopped by the DDS. With dimensions on scale, this drawing serves as a tool to determine the dimensions and orientation (defined by the deflecting angle α) of the DDS in this plane.

The bottom drawing in Fig. 2 shows the geometry in the axial plane for the case $2\theta = 0°$.

This drawing serves as a tool to determine the dimensions of the DDS in this plane.

A valuable by catch of the DDS is the shielding of air scatter produced by the incident beam as it travels through the free air path between the end of the collimator and the specimen.

Due to the fact that the DDS is always aimed at the irradiated area on the specimen, it will fulfill its purpose not only for the depicted symmetric θ/θ geometry but also for measurements with fixed incident beam, e.g., grazing incidence geometry.

By following a similar procedure, the above principle can be used for a 1D detector and instruments with other dimensions and/or manufacturers.

B. Construction, mounting, and alignment

Using the dimensions derived from Fig. 2, the DDS is made by laser cutting a template out of a steel plate with a thickness of 0.25 mm. Subsequent folding produces the desired shape. As the steel plate is rather thin, it tended to vibrate a little in the scanning mode. To counter this, plastic strips are placed on both sides; see Fig. 3.

Reproducible mounting and easy demounting of the DDS are achieved as follows. The housing of the Eiger-2500k detector contains two magnets to fix a filter and/or protection plate. Two magnets on the DDS were placed such that they coincide with the detector magnets, making a detachable but firm connection. An advantage of this mounting is that in the case of collision, no severe damage to the equipment is likely to occur as the DDS will just detach.

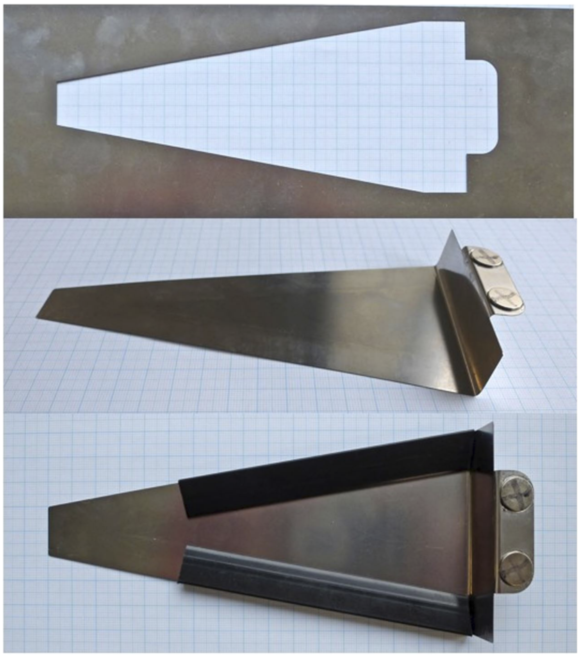

FIG. 3. From top to bottom: laser cut template, shaped device with two magnets, and final DDS with anti-vibration strips.

For optimum performance, it is necessary to align the DDS carefully. First, a quick scan of the dome diffraction in the low angle 2θ region without DDS is made using a Si⟨510⟩ wafer as a specimen without background and reflections. Second, the scan is repeated with the DDS mounted. The deflecting angle α (see Fig. 2) can then be adjusted by bending until the dome diffraction is just invisible.

Once this has been done, the DDS is ready to use for all kinds of experiments without the need for further adjustments. If the diffractometer radius is enlarged by shifting the detector on its track, the DDS can stay in place and only the deflecting angle α needs to be adjusted accordingly.

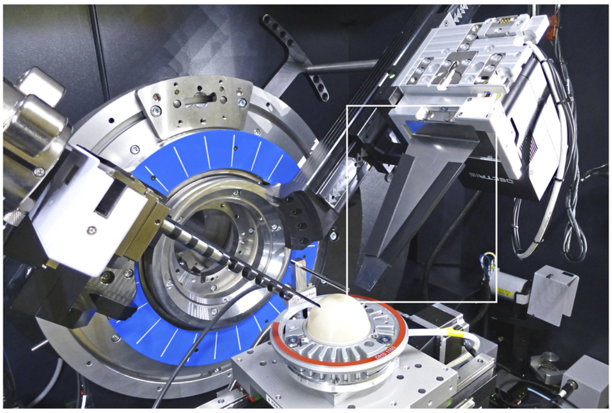

FIG. 4. 2D system with the Anton Paar stage, PEEK dome, and DDS (outlined).

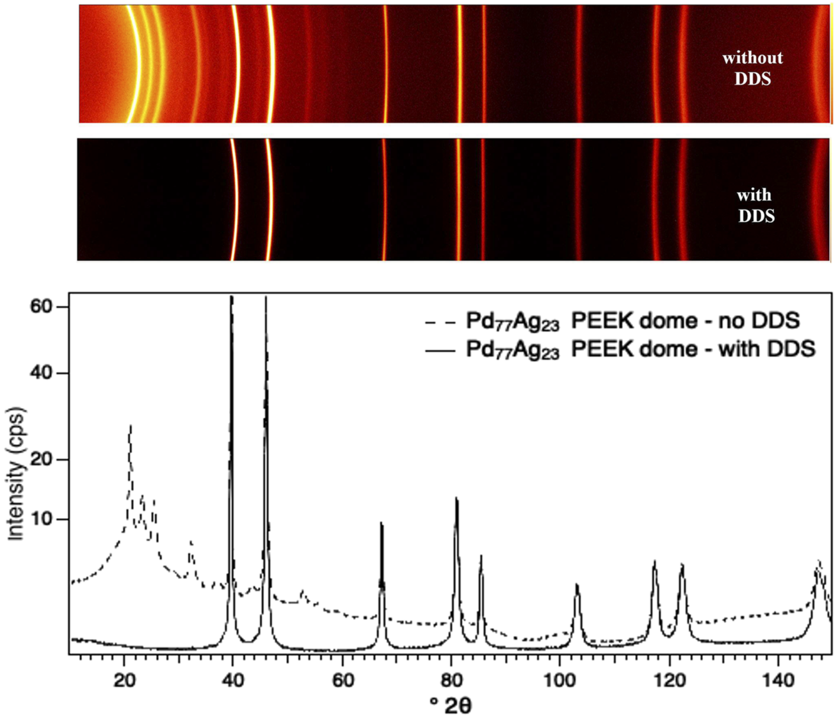

FIG. 5. $Pd_{77}Ag_{23}$ specimen with the PEEK dome measured without and with DDS. Top: 2D frames. Bottom: integrated intensity plots on square root scale. Wavelength CuKα.

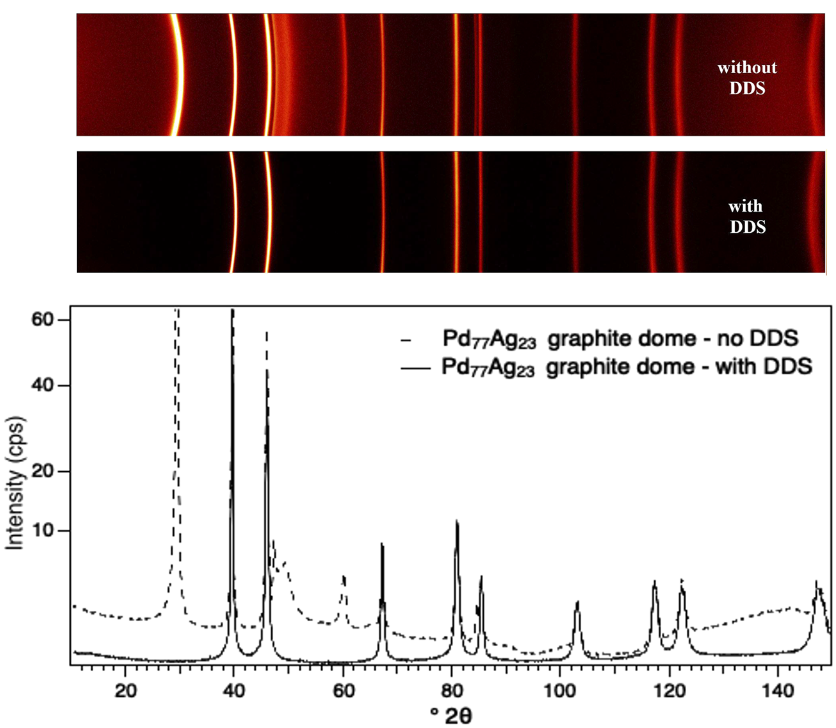

FIG. 6. $Pd_{77}Ag_{23}$ specimen with the graphite dome measured without and with DDS. Top: 2D frames. Bottom: integrated intensity plots on square root scale. Wavelength CuKα.

Figure 4 shows a photograph of the DDS mounted on our 2D system.

C. Performance

Performance of the DDS was investigated by using a $Pd_{77}Ag_{23}$ specimen mounted on the Anton Paar Domed Hot Stage with the PEEK and graphite dome. Two scans at room temperature are made: one without and one with the DDS in a fixed position. Figures 5 and 6 show that dome diffraction in terms of reflections and background is virtually absent in the scans made with the DDS. It also shows that the diffraction pattern of the specimen has not been affected.

The scans show that the DDS is effective from $10^\circ 2\theta$. For lower angles, the alignment of the DDS will be critical, but in that region, only background and no reflections from the dome are present. It has been tested that the DDS allows for measurements at room and elevated temperatures with full rotation and maximum tilt angles of the stage, even when the additional cooling nozzle for the PEEK dome is present. In addition, translations of the stage are possible and only limited in the direction of the detector. This freedom of specimen movements allows us to perform all types of XRD measurements, including texture and stress analysis.

III. CONCLUSIONS

The present *Dome Diffraction Screen* (DDS) effectively blocks the diffraction from the PEEK and graphite dome of a hot stage with a 2D detector. Consequently, application of the DDS dramatically improves measurements. Due to its simplicity and independency of software, it is applicable to a broad range of diffractometer systems, domes of various materials and diameters, and detectors. It can be stated that the DDS enables us to perform reliable measurements with domed stages that are otherwise hard to obtain.

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available within the article.

REFERENCES

[1] P. Hegele *et al.*, "In-situ XRD study of phase transformation kinetics in a Co-Cr-W-alloy manufactured by laser powder-bed fusion," Crystals **11**, 176 (2021).

[2] Dynamic Beam Optimization, Bruker Product Sheet XRD 52, 2020.

[3] B. B. He *et al.*, "Retractable knife-edge for XRD combinatorial screening," Powder Diffr. **18**, 171 (2016).

[4] Y. Shen, E. E. Pedersen, M. Christensen, and B. B. Iversen, "An electrochemical cell for in operando studies of lithium/sodium batteries using a conventional x-ray powder diffractometer," Rev. Sci. Instrum. **85**, 104103 (2014).

[5] Instruction Manual DHS 1100 Domed Hot Stage, Anton Paar GmbH, October 2016, Document number C58IB002EN-F.