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A device for the application of uniaxial strain to single crystal samples for use in synchrotron radiation experiments

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We present the design, construction, and testing of a straining device compatible with many different synchrotron radiation techniques, in a wide range of experimental environments (including low temperature, high field and ultra-high vacuum). The device has been tested by X-ray diffraction on single crystal samples of quasi-one-dimensional $\text{Cs}_2\text{Mo}_6\text{Se}_6$ and $\text{K}_2\text{Mo}_6\text{Se}_6$, in which microscopic strains up to a $\Delta c/c = 0.12\% \pm 0.01\%$ change in the c lattice parameters have been achieved. We have also used the device in an inelastic X-ray scattering experiment, to probe the strain-dependent speed of sound v along the c axis. A reduction $\Delta v/v$ of up to -3.8% was obtained at a strain of $\Delta c/c = 0.25\%$ in $\text{K}_2\text{Mo}_6\text{Se}_6$. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4933383>]

I. INTRODUCTION

Modulating the lattice parameters of crystalline materials allows us to continuously change their physical properties. This often permits access to novel emergent phases which are absent from the unstrained bulk.^{1,2} Perhaps the simplest and most intuitive method of tuning lattice parameters is by the application of strain. Currently, only a limited number of techniques exist for the application of strain to crystalline materials; the most commonly used are hydrostatic pressurisation, epitaxial growth on a mismatched substrate and mechanical straining, i.e., bending, stretching, or shearing a sample. Whilst hydrostatic conditions can be applied using a variety of devices and are often desirable due to the isotropic nature of the applied strain, they present a significant drawback: a pressure transmitting medium is required. Many of the most powerful structural or electronic probes using synchrotron light require a vacuum interface to the sample under measurement, including angle resolved photoemission (ARPES), resonant inelastic x-ray scattering (RIXS), and soft X-ray diffraction. This precludes the use of a transmitting medium, rendering hydrostatic conditions incompatible with these techniques.

An alternative method of applying strain, epitaxial growth, does allow unimpeded optical access to the sample surface,³ but can usually only provide a single discrete strain in any given sample.⁴ It is possible to achieve dynamic strain via epitaxial synthesis, by growing directly onto a piezoelectric material. This however requires a lattice match between the grown film and piezoelectric substrate^{5,6} and is hence only

possible in the small subset of materials that are isostructural to piezoelectric substrates (generally perovskites).

Mechanical methods are able to provide dynamic strain without the requirement of a transmitting medium.^{7–9} Dynamic rather than static strain is desirable as it offers the ability to continuously tune a material through distinct quantum ground states.^{8,10} Modern synchrotron techniques provide an ideal probe to observe and measure the continuous perturbation to the electronic and structural properties that tunable strain provides. When both optical access and dynamic strain are required, hydrostatic pressure and epitaxial growth are generally unsuitable due to the reasons discussed above and hence a mechanical device becomes essential. Numerous examples of such devices exist in the literature,^{11–14} but none of these were designed with the stringent requirements of a synchrotron beamline environment in mind. Here, we present the first mechanical straining device based on piezoelectric actuators specifically designed for use in synchrotron light-source experiments and demonstrate its performance using two separate X-ray techniques. Our device will enable the exploration of entire regions of phase space in complex materials which were previously inaccessible to synchrotron radiation.

II. DESIGN

Designing a straining device for use in synchrotron radiation experiments requires a number of factors to be taken into account. Most importantly, there must be unimpeded access to and from the strained sample, for both the incident photon beam and the scattered photons or photoemitted electrons. There is also limited space available in the sample-mounting environment — especially within ultra-high vacuum (UHV) chambers — and the inaccessibility of the straining

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device during measurements must be taken into consideration. A straining device based around two shear piezoelectric actuators addresses these points. The basic principle is that the application of a bias voltage to the piezoelectric actuators causes them to experience a shear displacement. This shear displacement can then be used to strain a sample mounted between the two actuators. This method enables the sample to be freely suspended, providing access to and from the sample for a range of X-ray scattering geometries. The piezoelectric actuators are comfortably small enough to fit within the space available at most synchrotron beamlines and the strain can be controlled remotely by varying the voltage bias across the actuators.

For the straining device presented here, two piezoelectric actuators (Figure 1) were mounted onto a machined stainless steel baseplate using an insulating epoxy glue (EPO-TEK H70E). Each actuator (Physik Instrumente P-141.10) is composed of 18 identical shear elements, comprising a piezoelectric ceramic (lead zirconate titanate, PZT) sandwiched between two thin metal electrodes. Applying a voltage bias to the electrodes creates a uniform electric field across the PZT and induces a shear displacement due to the piezoelectric effect. The piezoelectric ceramics are stacked and orientated such that alternate layers share electrodes and displace in the same direction; a stack therefore produces a shear displacement equal to the sum of the shear displacement of each of its elements. The shear piezoelectric actuator stacks have a specified maximum displacement of $\pm 5 \mu\text{m}$ at an applied voltage of $\pm 250 \text{ V}$: the combination of two stacks in our device geometry therefore provides a maximum travel distance of $20 \mu\text{m}$ at 300 K. The decision to use commercial actuators was taken purely for convenience: similar results could be obtained using homebuilt PZT shear stacks with CuBe foil electrodes.

The actuators were oriented to displace outwardly with the application of a common positive voltage bias, thus increasing the size of the gap G between them and exerting a uniaxial tensile strain on the mounted sample. Compressive strain may also be achieved using negative applied voltages. Two upper plates (one ceramic and one stainless steel) were attached to each actuator (Figure 1). The stainless steel top plate was glued to the insulating layer on top of the shear piezoelectric actuator using EPO-TEK H70E: this plate was designed to be a permanent threaded mount for the Macor (machinable ceramic) top plate, which is held in place using M1.6 stainless steel screws. Macor was selected to enable simultaneous 4-probe electrical transport experiments on strained crystals without shorting out the voltage and current contacts. A wedge-shaped design was chosen for both plates, to accommodate the large range of scattering or emission geometries required in synchrotron experiments. Additional M1.6 holes in the ceramic top plate allow a transfer piece to be attached to the straining device (Figure 2), which can be used to facilitate sample exchange during experiments.

In addition to acting as a mount for the piezoelectric actuators, the upper baseplate (Figure 1) provides a second (coarser) method of applying a static strain to the sample. This strain is generated by four M2 screws, located at the corners of the upper baseplate. Tightening these screws causes the ends of the baseplate to flex about the central fulcrum, displacing the shear piezoelectric actuators with respect to each other and increasing the gap size between them. The four M2 control screws also anchor the upper baseplate to the lower baseplate. For mounting at beamline sample spaces, the straining device clamps to a goniometer via an adapter piece, which is attached to the lower baseplate using an M3 bolt.

Although we have fabricated both baseplates from stainless steel, any other stiff metal which deforms elastically

FIG. 1. (Left) Cross-sectional schematic of the straining device, highlighting its dimensions. (Right) The component breakdown of the straining device.

FIG. 2. (Left) The straining device with transfer piece attached. (Right) A mounted $\text{Cs}_2\text{Mo}_6\text{Se}_6$ crystal, with both ends of the needle-shaped sample embedded firmly into silver epoxy.

could be used instead. For example, if we wish to fix the strain device to a cryogenic cold finger within a vacuum chamber, a higher thermal conductivity is desirable and hence CuBe would be a preferable material. In this case, the metallic upper plate should also be made in CuBe to limit the mismatch in thermal expansion coefficients across the device.

We note that our device is conceptually similar to an earlier design for performing transport measurements under strain using piezoelectric bimorphs.¹⁴ However, (1) the increased stiffness and lack of hysteresis in our shear piezo stacks and (2) the adjustable coarse strain provided by our twin baseplates represent significant improvements over this early work.

Samples suitable for straining in our device should ideally be needle-shaped single crystals approximately 1-3 mm in length and a few hundred micrometers thick, thus ensuring that they can be easily mounted between the ceramic top plates. Larger single crystals could be cleaved to the required dimensions, or cut using a wire saw. Samples should also be less stiff than the straining device. The stiffness of the straining device, with the assumption of a perfect epoxy glue, is estimated in Section V to be $\approx 16 \pm 6 \text{ N } \mu\text{m}^{-1}$. To mount a sample, the ends of the sample were glued to the ceramic top plates using a silver epoxy (EPO-TEK E-4110), securing it between the two piezoelectric shear actuators (Figure 2). We chose silver-loaded epoxy for our tests since in future, we envisage performing resistivity measurements concurrently with X-ray diffraction studies. However if resistance measurements are not required, other harder epoxies may be more efficient in transmitting the force from the piezoelectric actuator to the sample.¹⁵

The overall stiffness of our device is determined by the lower baseplate and the piezoelectric stacks. It is hence conceivable that experiments on particularly wide or stiff crystals could result in deformation of the baseplate and/or the piezoelectric actuators rather than crystal strain. However, as we shall demonstrate in Section IV, our device remains rigid and undeformed with respect to the crystals under test.

Instead, the major limiting factors in the application of strain are believed to be the epoxy glue used for sample mounting, and the formation of inhomogeneous strain fields within crystals under measurement. These will be discussed in detail in Section V.

Prior to X-ray synchrotron measurements, the device was tested using a four probe resistance measurement on strain gauge wire (constantan) and capacitive sensors to calibrate the movements of the piezoelectric actuators. Strain was observed during the resistance measurements; however, calibration of the microscopic straining capabilities are best performed by measuring the expansion of the strained lattice parameter of a single crystal using X-ray diffraction. We will now describe the material selection and experimental procedure for this calibration.

III. TEST MATERIAL

The primary effect of strain on a single crystal is to compress or expand the lattice along the strain direction. Due to the associated expansion or compression of the material orthogonal to this direction (which is determined by the Poisson ratio), any crystalline anisotropy may also be modified. Crystals with anisotropic structures and/or electronic properties are therefore interesting candidate materials to examine under strain, thus motivating our choice of an exceptionally one-dimensional family of crystals to test our device.

The series of molybdenum selenides ($M_2\text{Mo}_6\text{Se}_6$, where M is a group 1 A alkali metal or group 3 A metal In, Tl) are ideal materials to investigate the effects of uniaxial strain, due to their quasi-one-dimensional crystal structures (Figure 3). The molybdenum selenides with group 3 A (Tl or In) guest ions become superconducting at temperatures of 3-6.5 K and 2.9 K respectively, while those with group 1 A guest ions exhibit a temperature-dependent metal-to-insulator crossover.¹⁶ $\text{Tl}_2\text{Mo}_6\text{Se}_6$ displays metallic behaviour down to low temperature, but has been driven into a high resistance state by the application of uniaxial strain,⁸ which may result

FIG. 3. $M_2\text{Mo}_6\text{Se}_6$ crystal structure. (Left) A projection along the a -axis of the material. (Right) A projection along the c -axis.

from charge density wave formation. All $M_2\text{Mo}_6\text{Se}_6$ form long needle-like single crystals, with dimensions typically 3 mm by 200 μm . Samples with approximately these dimensions were used for the straining experiments presented in Sections IV and VI. The needle-like geometry reflects the underlying crystal structure, consisting of chains of stacked MoSe triangles extending along the c -axis direction. These chains form a hexagonal lattice (of space-group $P6_3/m$) and are weakly coupled by the guest ions M , which occupy the channels between the chains.¹⁷ Mo_6Se_6 nanowires have been predicted to exhibit high breaking strains of greater than 8.2%.^{18,19} In our experience, single crystal $M_2\text{Mo}_6\text{Se}_6$ breaks well before this limit along pre-existing cracks or defects.

IV. SYNCHROTRON RADIATION X-RAY DIFFRACTION

The performance of our straining device was assessed by measuring the expansion of the strained lattice parameter (in this case the c -axis) as a function of the displacement of the piezoelectric actuators. To achieve this, X-ray diffraction experiments were performed at the Diamond Light Source on the I16 beamline. The straining device was mounted on a six circle kappa diffractometer via a Huber 1005 goniometer. An X-ray beam energy of 12 keV with an energy resolution of 1.7 eV was used for all measurements. A ± 5 V dc voltage source interfaced with the beamline control software and amplified using a PI E-141 voltage amplifier was used to remotely control the piezoelectric actuators.

$M_2\text{Mo}_6\text{Se}_6$ single crystals were mounted onto the straining device, with their crystal c -axis perpendicular to the incident X-ray beam and parallel to the strain axis. After aligning to the (004) Bragg spot, uniaxial strain was applied to the sample in small increments: 10 V steps, corresponding to translations of 400 nm by the shear piezoelectric actuators. At each step, both the diffracted beam profile of the (004) Bragg spot and the gap size G were measured. By fitting the diffracted beam profile with a Gaussian distribution, the expansion in the c -axis parameter (i.e., the microscopic uniaxial strain) could be determined from the change in the 2θ peak position. At the maximum total displacement of the shear piezoelectric actuators (10 μm at 250 V), the applied voltage was gradually reduced back to zero. A small amount of strain was then manually introduced by tightening the screws on

the upper baseplate, effectively altering the strain at 0 V bias (the zero-point strain). The procedure for the application and measurement of uniaxial strain was then repeated at this new zero-point strain. This method extended the magnitude of the uniaxial strain beyond the limit imposed by the piezoelectric actuators, whilst maintaining the advantages of remote and precise strain control which they provide.

Two different $M_2\text{Mo}_6\text{Se}_6$ crystals featuring K and Cs guest ions were measured using this method. Maximum strains of $0.046\% \pm 0.01\%$ and $0.12\% \pm 0.01\%$ were achieved in $\text{K}_2\text{Mo}_6\text{Se}_6$ and $\text{Cs}_2\text{Mo}_6\text{Se}_6$, respectively (Figure 4).

To relate these crystal strains to the absolute displacement of the piezoelectric actuators, we determined the change in gap G between the two Macor top plates as a function of uniaxial strain. This was achieved using the X-ray beam in conjunction with a diode detector to measure the relative positions of the top plate edges, i.e., the two mounting points for the sample. Scanning the straining device across the beam yields an X-ray intensity profile as a function of device position, due to the shadow cast by the top plates on the diode detector. This allowed the relative positions of the two mounting points and therefore the gap size to be determined.

For both $\text{K}_2\text{Mo}_6\text{Se}_6$ and $\text{Cs}_2\text{Mo}_6\text{Se}_6$ (Figure 4), the increase in the gap size as a function of uniaxial strain was measured and a linear response observed. The strain produced in the samples was elastic, with no hysteresis visible within our experimental error of 0.01%. However, the gradient of the linear response differed between the two samples, and a large discrepancy between the sample strain and the change in gap size was observed in both crystals.

V. MECHANICAL PERFORMANCE

We first address the observed variation in strains between the two crystals. The longitudinal stiffness of $M_2\text{Mo}_6\text{Se}_6$ is

FIG. 4. Strain measurements from $\text{K}_2\text{Mo}_6\text{Se}_6$ (squares) and $\text{Cs}_2\text{Mo}_6\text{Se}_6$ (triangles). Different colours represent different experimental runs, where between runs the screws on the upper baseplate were tightened to introduce a larger zero-point strain.

primarily controlled by the Mo_6Se_6 chains and hence we do not anticipate any significant stiffness variation upon changing M . Instead, the disparity between samples is due to geometric effects, since the stiffness of an object depends on its shape as well as its composition. Given that the shape of the crystal and the thin layer of epoxy glue used for mounting inevitably change from one sample to the next, small variations in linear response between samples are normal.

The discrepancy between gap size G and sample strain implies that the transfer of strain from the piezoelectric actuators to the crystal is limited, with an efficiency of less than 10%. Factors which could explain this inefficiency include the epoxy glue used for sample mounting, or the creation of inhomogeneous strain fields within loaded samples due to pre-existent crystal defects. Alternatively, the finite stiffness of the device might result in G changing by a distance less than the total piezo stack travel. Our data indicate that the epoxy constitutes the weak point in our strain device, while also suggesting the presence of inhomogeneity in our crystalline samples. We outline the evidence supporting these statements in detail below.

- (i) Figure 4 shows that the change in gap size reaches $\approx 1\%$ when the maximum voltage is applied to the piezoactuators. Given that the theoretical maximum displacement of these actuators is $10\text{ }\mu\text{m}$ and the initial gap size $\approx 1.17\text{ mm}$, it is clear that the Macor top plates are moving apart as intended. A deformation of the strain device due to an excessively stiff sample would instead lead to a smaller increase in the gap size, well below the 1% which we observe.
- (ii) Tightening the four M2 screws designed to apply coarse strain results in a large increase in the experimentally measured strain within our crystals. If our device were less stiff than the samples under measurement, tightening these screws would merely flex the baseplate and piezo stacks, leading to little or no change in the sample strain. Therefore, (i) and (ii) both provide experimental confirmation that the device is considerably stiffer than the ensemble of glue and sample.
- (iii) Some evidence for several distinct homogeneous regions was visible during X-ray measurements. Specifically, during alignment, as the X-ray beam impinges on different parts of the sample, some variations in strain were apparent, indicating that strain fields that develop within the crystals are not fully homogeneous. Such inhomogeneity is likely to arise from the presence of dislocations and defects.
- (iv) To justify our conclusion that the limiting factor in the device performance is the epoxy glue, we have calculated the approximate stiffness k for the four main components of the device: the sample, epoxy, piezoelectric stacks, and twin baseplates. For the sample and the epoxy, we use Hooke's Law (Eq. (1)) which relates the stiffness of a rod under axial strain to its length l , cross-sectional area A and Young's modulus E ,

$$k = \frac{F}{\delta x} = \frac{AE}{l}. \quad (1)$$

Using $E = 400\text{ GPa}$ for Mo_6Se_6 nanowires,⁸ we estimate $k_{\text{sample}} = 13 \pm 8\text{ N }\mu\text{m}^{-1}$. A slightly higher value for the Mo_6Se_6 Young's modulus of 570 GPa has been calculated elsewhere in the literature,¹⁸ but the uncertainty in E is small compared to that in A and l . This sample stiffness is likely to be an overestimate, since we have assumed a perfectly homogeneous crystalline sample. In reality, any dislocations and defects in the crystal structure will create multiple homogeneous yet weakly mechanically coupled crystalline regions within a single sample. This structural inhomogeneity reduces the total elastic modulus of the crystal and hence lowers k_{sample} , as well as creating the inhomogeneous strain field which we observe.

The geometry and Young's modulus of the epoxy glue are more challenging to define, leading to a larger error in the calculated stiffness. For the epoxy cross-sectional area, we estimate a value twice that of the crystal, with the applied force being distributed along a length of 0.1 mm . Using the storage modulus²⁰ as an estimate for the Young's modulus, we obtain $k_{\text{epoxy}} \sim 4 \pm 3\text{ N }\mu\text{m}^{-1}$.

The piezoelectric shear stacks have a shear stiffness^{21,22} of $k_{\text{shearpiezo}} = 100\text{ N }\mu\text{m}^{-1}$, which exceeds that of the sample by an order of magnitude. However, the bending stiffness of the piezoelectric actuators must also be considered, since this limits the total stack stiffness. To calculate the bending stiffness, the piezoelectric actuator was approximated to act as a cantilever, whose stiffness k can be described using

$$k = \frac{Ewt^3}{4L^3}, \quad (2)$$

where L is the cantilever length, w the width, and t the thickness and E the Young's modulus.

Excluding the stainless steel and Macor top-plates (which we do not expect to deform), Eq. (2) provides an estimate of $k_{\text{piezobend}} = 22\text{ N }\mu\text{m}^{-1}$ for the bending stiffness of a single piezoelectric actuator within our device geometry. Given that two actuators are used simultaneously in our straining device, the total bending stiffness of the piezoelectric actuators is half this value, i.e., $k_{\text{piezototal}} = 11\text{ N }\mu\text{m}^{-1}$. In the eventual case of $k_{\text{piezobend}}$ limiting the sample strain (which would be highlighted by a change in G smaller than the piezo travel), one could increase the bending stiffness by using actuators with a larger cross section, or alternatively adjust the sample dimensions to reduce k_{sample} .

The complex geometry of the twin baseplate design is difficult to model. We therefore disregard the upper baseplate (which is weak due to its central fulcrum) and any rigidifying effects of the adapter piece, focusing solely on the lower baseplate (which may be modeled as a single beam) to obtain a lower limit for the overall baseplate stiffness.

In our straining device, the piezoelectric actuators are positioned symmetrically at a distance of 7 mm either sides of the centre point of the baseplate. Under load, the torque applied by the piezoelectric actuators to the baseplate will cause it to deform. Given the geometry of the situation, this deformation will result in a change in the curvature R of the baseplate.

Changes in the curvature R of a beam (in our case the baseplate) may be modelled using,

$$R = \frac{IE}{M}, \quad (3)$$

where R is calculated using the bending moment M (i.e., the torque applied to the baseplate by the piezo stacks), together with the Young's modulus E of stainless steel and the baseplate moment of inertia I .

As a consequence of the change in curvature of the baseplate, the piezoelectric actuators will be tilted bringing the ceramic top plates closer together and resulting in a decrease in G . Using simple trigonometry this decrease in G can be calculated by relating the deflection at the ends of the baseplate, due to the change in R , to a shift in the position, δx , at the top of the piezoelectric actuators. Using Eq. (3) and the calculated value for δx , this yields a stiffness of $k_{\text{baseplate}} > 16 \pm 6 \text{ N } \mu\text{m}^{-1}$. We stress that this is a severe underestimate of the true baseplate stiffness, due to the simplifications detailed above.

These stiffness estimates for individual components suggest that our straining device may not be much stiffer than our samples. However, experimentally we have proven in a number of different samples that our straining device is capable of inducing significant levels of strain in single crystals (Figure 4), even breaking the crystals in several cases. Given (a) the experimental evidence for strain in our crystals and (b) the increase in G whose magnitude corresponds to the piezo travel, we conclude that the sample stiffness is indeed less than that of the straining device. Successful transmission of the strain from device to sample is likely to be limited by a combination of crystal inhomogeneity and the low elastic stiffness of the epoxy glue, rather than any intrinsic weakness in the straining device.

Despite the inefficiency in the strain transfer, it is important to note that the induced strain in our crystals was stable within measurement uncertainties of 0.01% over time-periods of up to 10 h. This is a prerequisite for time-consuming measurements such as inelastic X-ray scattering (IXS) (see Sec. VI) and also ARPES or RIXS. Cyclically removing and re-applying, the piezo voltage resulted in reproducible strains. This implies that once the epoxy glue was cured and the sample bonded, no irreversible slippage in the glue occurred.

VI. INELASTIC X-RAY SCATTERING

IXS²³ was used to measure the change in the lattice dynamics, specifically the change in the longitudinal speed of sound, in $\text{K}_2\text{Mo}_6\text{Se}_6$ as a function of strain. The straining device was mounted on the ID28 beamline at the European Synchrotron Radiation Facility, using a Huber 1005 goniometer with the crystal c -axis in the scattering plane. The beamline was operated at an energy resolution of 3.2 meV at an incident energy of 17.794 keV. The sample was aligned on the (002) Bragg spot and strain was applied along the c -axis. We measured the acoustic phonon dispersion and thus the speed of sound as a function of strain along the 00 l direction as follows: IXS spectra were acquired in a sequence of momentum points $\mathbf{Q} = (002) + (00l)$ with $l = 0.05, 0.075$ and 0.1 . The phonon energies $\epsilon(l)$ extracted from these spectra by peak-fitting form a straight line passing through the origin (insert of Figure 5).

FIG. 5. Longitudinal speed of sound in $\text{K}_2\text{Mo}_6\text{Se}_6$ as a function of strain obtained from *inset* strain dependent low- q phonon measurements of $\text{K}_2\text{Mo}_6\text{Se}_6$.

The slope ν from a linear regression $\epsilon_{\text{fit}} = \nu \cdot l$ corresponds to the speed of sound (after conversion from lattice units). Since the phonons measured in this geometry propagate along the one-dimensional c axis, we refer to ν as the longitudinal speed of sound.

Using this method, we observed a large and linear decrease of $\frac{\Delta \nu}{\nu} = -3.8\% \pm 0.7\%$ (Figure 5) in the longitudinal speed of sound with a uniaxial strain of up to $\frac{\Delta c}{c} = 0.25\%$ applied to the $\text{K}_2\text{Mo}_6\text{Se}_6$ crystal. The technique of calibrating the strain, Δc , by X-ray diffraction was also applied in this IXS experiment, since the spectrometer acts as a diffractometer when tuned to zero energy shift. It should be noted that we did observe a broadening of the lattice parameter distribution as measured by the 2θ scattering profile upon increasing the strain from 0.06% to 0.17%, indicative of a deterioration of sample quality and a slightly inhomogeneous strain field. Nevertheless, due to the small diameter of the highly focused X-ray beam ($250 \times 80 \mu\text{m}$) it was possible to locate regions of well-defined strain in the sample.

For IXS experiments, the free passage of both the incident and scattered X-ray beams to and from the sample is very important, as any scattering from the supporting material or container walls would add a background to the spectra that often prohibits analysis or at least contributes to experimental errors. Our straining device avoids this by freely suspending the sample between the piezoelectric actuators.

VII. CONCLUSIONS AND OUTLOOK

The piezoelectric straining device was successfully tested on two different synchrotron beamlines using distinct X-ray techniques (diffraction and IXS). We were able to exert and measure substantial uniaxial strains of up to 0.25% in a range of crystalline materials, which in several cases proved sufficient to break the crystal. These levels of strain were

sufficient to produce an observable difference in the physical properties of the material, i.e., the longitudinal speed of sound.

The device was shown to be reliable over the course of several X-ray experiments, with the strain proving stable and reproducible throughout the measurement duration. The performance of the device was principally limited by the rigidity of the epoxy glue used to mount samples. In the event of a new, stiffer epoxy becoming available, it is plausible that the stiffness of particularly wide/thick samples with high Young's moduli and low defect densities could exceed that of the straining device. However, this situation could easily be remedied by increasing the thickness of the lower baseplate and using shorter piezo stacks with a larger cross section.

Whilst the experiments described here were performed at room temperature, the device is in principle capable of operation at low temperatures. However, a decrease in the performance of the piezoelectric actuators is expected as the temperature falls: our experience with similar piezos in scanning probe microscopes indicates that the actuator travel may drop by a factor ~ 4 between room temperature and 20 K. To counteract this, different mounting techniques (e.g., clamps) or stronger glues could be explored to achieve a greater conversion of the piezoelectric actuator motion into the microscopic strain experienced by the sample. Operation at high temperatures is also possible, provided that these do not exceed the piezo Curie temperature, which for the piezoelectric actuators used here was 400 K.

Further miniaturization of the device may also be envisaged, although this would likely necessitate a reduction in the linear actuation range. In this case, a stiffer coupling of the sample to the device would become increasingly important, since the reduced strain relative to the change in gap size is the principal limitation to the dynamic strain range of our device. Careful material choice has also ensured that our device is fully ultra-high vacuum compatible, thus enabling it to be used in a range of X-ray techniques such as ARPES experiments where crystal de-twinning by uniaxial strain has recently been reported.^{24,25}

In summary, our results reveal the exciting prospect of future experiments on crystals in which the application of dynamic strain has hitherto been unachievable. Our straining device allows dynamic strain to be used as a method of perturbing the delicate balance between competing physical phases whilst simultaneously observing their evolution using synchrotron radiation, hence opening a new experimental frontier.

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