First testing of the fast kappa diffractometers at National Synchrotron Light Source and European Synchrotron Radiation Facility

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We present the design and first results of the performance of a new four-circle x-ray diffractometer featuring the less-common "kappa" geometry originally used on the CAD-4 instrument. This geometry permits access to all reciprocal-space settings while the mechanical supports remain entirely on one side of the beam; this is very useful for split ports on bending-magnet beamlines. Our design is able to carry heavy loads, such as a Displex-type cryostat, and operates at relatively high speeds because it uses direct drive servomotors. Considerable attention has been given to the distribution of loads which is optimized for horizontal-axis operation. A sphere of confusion of 50 μm was achieved fairly easily for the mutual alignment of the axes, and this can probably be improved. Our initial tests show that accurate alignment of crystals can be achieved, and reliable measurements have been made on a number of experimental systems. Rocking curves of silicon have been measured, but these are at the limit of the setting accuracy. Arbitrary settings are achieved in about 1 s, but up to an additional 1 s settling time is needed for high accuracy work. © *1995 American Institute of Physics.*

Precision geared goniometric drives have been developed since the earliest days of x-ray diffraction, and have been widely used over the years. They are the mechanical component of choice in almost all diffractometer designs. Only when the utmost in angular setting accuracy is required, say better than 0.001°, are they found to be inadequate and lever-arm/pivot mechanisms become preferred. One of the biggest advantages of gear-based goniometers is their high absolute angular precision, around 0.01° over the full 360° range.

With the arrival of the electronic revolution, mechanical components everywhere are being replaced one by one with their electronic equivalents, often with considerable improvement of performance and saving of cost. We believe that time has now arrived with goniometers: electronic servodrives are now competitive in cost and performance with their geared equivalents. The trend is expected to continue, favoring the servo choice even more strongly in the future.

We based our design on the "Megatorque" series of direct drive servomotors manufactured by Nippon Seiko Corp.[1] They have an incremental angular accuracy of 0.0006° [0.00014° for European Synchrotron Radiation Facility (ESRF) units] and absolute precision of 0.01° over 360°. The available torque (15 kg m for the largest size) is significantly *greater* than the recommended loading of the equivalent-sized geared goniometer (Huber, Microcontrole, or Kohzu), which is limited by excessive wearing of the worm gear. Of course the accuracy of an electronic system should not deteriorate over time. The angular stiffness of the chosen unit (10^{-5} rad/kg m for the large size) is limited by the quality of the bearing and rigidity of the housing; this is better than the figure for the equivalent sized goniometer from Microcontrole, and is probably a little worse than that of Huber, which employs a double bearing (actual figure not available; estimate based on loading recommendations).

A second innovation was to avoid the use of a χ circle, the device commonly used to obtain a wide range of reciprocal-space settings. The χ circle is the weakest component of a conventional four-circle diffractometer, as it is very prone to mechanical distortion, particularly when used on a horizontal axis instrument. The χ circle also puts considerable constraints on the regions of reciprocal space that can be reached, particularly at large 2θ. Another problem is one of space constraints due to nearby beamlines, especially when a bending magnet port is multiplexed by splitting the radiation fan into sectors, which is the situation of X16C at National Synchrotron Light Source (NSLS). Both these problems are solved by use of the so-called *kappa geometry*, originally used on the commercial CAD-4 instrument, which employs an oblique axis (called κ) set at 50° to ω on an arm. A second 50° arm then carries the ϕ axis that holds the sample. The sample can then be held at any angle within a volume slightly exceeding a hemisphere, and thus reach any diffraction condition required. Since all the goniometers and supports are on one side of the center line, there is minimal interference on the opposite side, leaving space for another beamline to pass through, or for sample conditioning hardware to approach. This arrangement of axes is clearly shown in the elevation view of the final design drawn in Fig. 1.

Having opted for the kappa geometry, we then sought to build an instrument that would work with bulky sample environments, such as a cryostat or a vacuum chamber. We designed for 10 kg of load and a clearance along the ϕ axis of 330 mm, sufficient to allow a Displex-style cryostat to be

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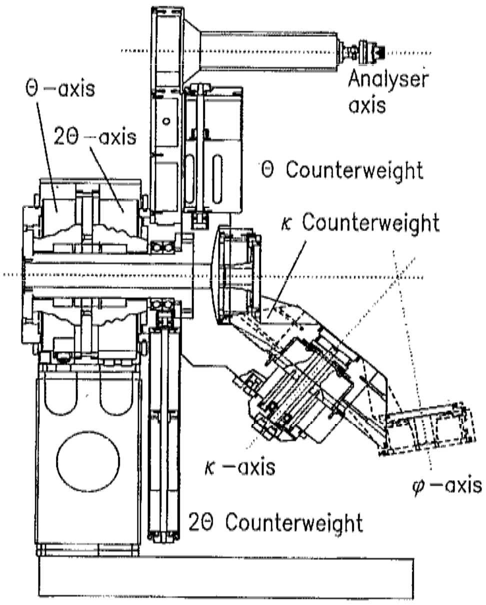

FIG. 1. Elevation sketch of the kappa diffractometer showing the layout of the axes. The scale is set by the clearance distance between the bolting surface of the ϕ circle and the center of all the axes, which is 330 mm. The setting shown is $2\theta=90$, $\theta=90$. Alternate positions of the κ arm are drawn, $\kappa=0$ as a full curve and $\kappa=180$ as a dashed curve.

held at its heavy end. The two arms are 120 and 100 mm in thickness and are made of a special Al-Mg-Mn alloy (5083-O) for stiffness. Finite element analysis of the design predicted a total deflection (theoretical) of less than 25 μm at the sample under full load of 10 kg. The arms have complex shapes in order to preserve their rigidity while maintaining strength; an exploded view of the assembly is shown in Fig. 2. The ω and 2θ axes are exactly counterbalanced by means of continuously adjustable weights. The twisting forces on all the arms are minimized by judicious positioning of the load carrying bearings (see Fig. 2). The ω-2θ layout is also optimized for horizontal axis operation to minimize the deflection of the 2θ arm.

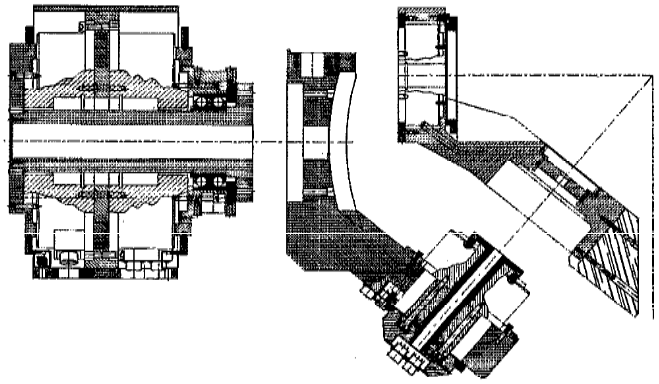

FIG. 2. Exploded view of cross-sectional drawings of the critical components, the θ-2θ assembly on the left, the θ arm (ω) in the middle, and the κ arm on the right. The θ-2θ assembly has a thrust bearing between the two axes in addition to the two motor bearings carried by the support; this aids the mutual concentricity of the two principle axes. An outrigger bearing on the κ axis helps to increase its angular stiffness.

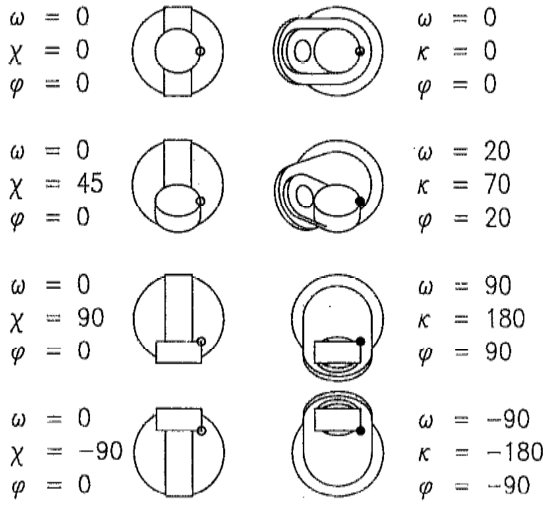

FIG. 3. Sketch showing the relationship between the Eulerian (left) and kappa (right) settings of the diffractometer. This is a sideview of the instrument, with its principal 2θ axis pointing out of the page. The incident x-ray beam is traveling horizontally from left to right. In each case the sample, attached to the ϕ circle, has the same orientation in the left- and right-hand panels, as designated by the dot. Motions of ω alone or ϕ alone are the same in both cases. The figure concentrates on the motion of (Eulerian) χ, which results in a compound setting of (kappa) $\{\omega_K,\kappa,\phi_K\}$. For simplicity, the case of $\alpha_0=45°$ has been considered.

A simple angle transformation relates the $\{\omega_K,\kappa,\phi_K\}$ setting onto the conventional Eulerian $\{\omega,\chi,\phi\}$ one,

$$\sin(\chi/2)=\sin(\kappa/2)\sin\alpha_0,$$

$$\omega=\omega_K-\cos(\kappa/2)/\cos(\chi/2),$$

$$\phi=\phi_K-\cos(\kappa/2)/\cos(\chi/2),$$

where $\alpha_0=50°$ is the inclination angle between the κ and ω and between the κ and ϕ axes. The definitions of the zeros of the angles must conform to the convention of Fig. 3, which also serves to illustrate the analogy between the Eulerian and kappa settings. Note the angles indicated are approximate and that $\alpha_0=45°$ has been used. It is therefore possible to use any existing diffractometer control instrumentation with trivial modification. In the NSLS X16C application, the interfacing of the new motor drivers to the SUPER diffractometer control program[2] is achieved by defining "pseudomotors" for the conventional angles. Then the kappa geometry can by invisible to the user, who is presented with all results in the familiar Eulerian coordinate system. In the ESRF application, an analogous interface has been made for SPEC.[3]

The performance of the motors was evaluated independently in the laboratory using a Megatorque motor attached to a heavy weight, 150 mm off axis. An angular slew of 90° was performed and the angular position was logged versus time in Fig. 4(a). The 5 kg weight arrived in 0.2 s and settled within a further 0.2 s; the linear ramp is limited by the programmed maximum velocity of the servo controller. The 50 kg weight follows a smooth parabolic trajectory of constant acceleration determined by the maximum available torque; it arrived in 0.4 s and settled within a further 0.4 s. Note that it takes just as long to slow down as it does to speed up. In both cases underdamped oscillations of the position take

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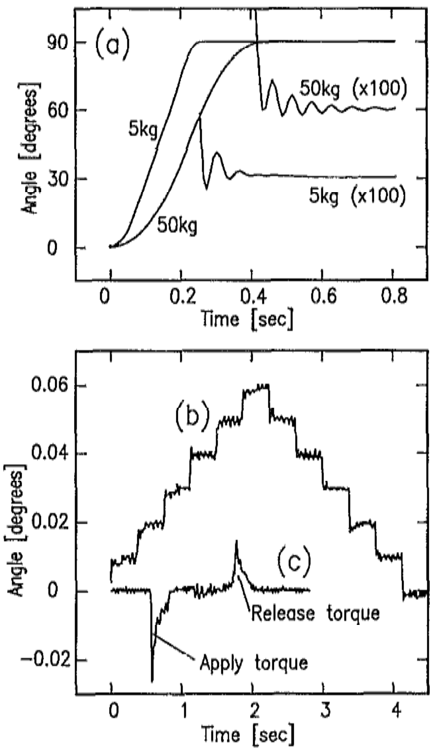

FIG. 4. Mechanical performance of one of the series of NSK servomotors used to construct the kappa diffractometer. (a) Angle vs time curve for a 90° move carrying weights of 5 kg (boxes) and 50 kg (crosses). The inset curves are magnified ×100 and offset vertically for clarity, but both refer to the original time axis. (b) Angle vs time curve for a step scan of programmed small 0.01° increments showing the position fluctuations in the recorded position. (c) Angle vs time curve response to an external torque of 1.5 kg m applied at a time of 0.55 s and removed at 1.75 s.

place on final approach [×100 inset in Fig. 4(a)]. In the software, the motor is not considered to have arrived until both the position is correct and the speed is zero, within a specified tolerance. Figure 4(b) shows the position response to a programmed staircase of small 0.01° steps. Dithering of the servoposition at the 0.001° rms level is visible on each step, and varies from step to step; the settling time is of order 30 ms for such small moves. Finally, the response of the servo to an external transient is shown in Fig. 4(c). A spring was used to apply a torque of 1.5 kg m on the end of a long lever between the times of 0.55 and 1.75 s. There is a momentary rotation due to the transient, but the motor recovers its position entirely in between, showing that the effective static mechanical compliance of the system in the rotational direction is zero.

We fabricated two prototype instruments in the machine shop of the University of Illinois Material Research Laboratory. One is being tested on beamline X16C at NSLS, the other on ID11 at ESRF. Both units are functional and are seeing regular use. A 50 μm sphere of confusion was readily obtained by means of set screw adjustments on all the axis positions, and shims in the slots provided. Loading of the end of the kappa arm with a 6 kg weight produced a deflection of 25 μm, again roughly consistent with the specification.

At NSLS the kappa diffractometer has been used for studying CoSi thin films on Si substrates.[4] The narrow reflections of the substrate provided a critical test of the overall positioning accuracy. An orientation matrix using seven well-separated reflections proved to be very accurate, and each peak was reproduced exactly. The speed is particularly helpful in this endeavor, as it takes a mere second or two to pass from one reflection to the next. The entire alignment was carried out in less than 1 h. After alignment, diffraction peaks from the film were measured as a function of energy near the Co K-absorption edge. Diffraction EXAFS (DAFS) oscillations are clearly seen in the data, shown in Fig. 5, the relative magnitude of which was substantially enhanced when a bcc-forbidden reflection was used, with its structure factor proportional to f_{Co}-f_{Si}. This is because the crystal structure of the films was of the CsCl type, unusual for cobalt silicides.

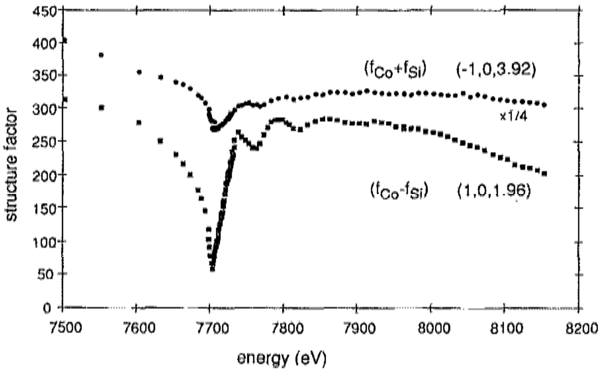

FIG. 5. DAFS data recorded with the kappa diffractometer on X16C at NSLS from a thin film sample of CoSi. Each point is a measurement of the crystallographic structure factor at a different photon energy, and required resetting of the diffractometer as well as the beamline monochromator.

At ESRF the kappa diffractometer has also been used in commissioning experiences at the ID 11 materials' science beamline. In crystallographic studies of $Mg(HCOO)_2 2H_2O$, good quality orientation matrices have been obtained with excellent reproducibility of both positions and peak shapes. The diffractometer has also been used in a Compton scattering experiment with a spectrometer mounted on the 2θ arm.

ACKNOWLEDGMENTS

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[1] Nippon Seiko Corp., Tokyo, Japan.

[2] R. M. Fleming, AT&T Bell Laboratories, personal communication.

[3] Certified Scientific Software, Cambridge, MA.

[4] R. Schuster, D. Adler, A. Ghosh, I. K. Robinson, and H. von Känel (to be published).

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