

Lithium metal for x-ray refractive optics

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Lithium metal is the best material for refractive lenses that must focus x-rays with energies below 15 keV, but to date no lens from Li has been reported. This letter demonstrates focusing of 10 keV x-rays with a one-dimensional sawtooth lens made from Li. The lens' theoretical gain is 4.5, with manufacturing imperfections likely responsible for the threefold gain that is observed. Despite the Li reactivity the lens is stable over months of operation if kept under vacuum. © 2001 American Institute of Physics. [DOI: 10.1063/1.1425068]

A recent reevaluation¹ of x-ray refraction for focusing synchrotron x-ray sources suggested using a single lens made from high density materials such as gold or tungsten. Such refractive lenses² became real through the compound refractive lens (CRL) concept, $N \sim 100$ lenses in series. Then the single lens' excessive focal length $f \sim 100$ m reduces to a more useful $f/N \sim 1$ m. The first CRL³ focused in one dimension by means of an array of small holes drilled in aluminum. Since then several low atomic number materials have been used to make CRLs of various types. To date the lenses that perform closest to theoretical expectations use aluminum⁴ or silicon,⁵ because their fabrication techniques are very well established. Good lenses from plastics are already commercially available.⁶ The theoretically excellent performance of Be lenses is not yet achieved in practice, e.g., due to intrinsic scattering or/and difficulties in manufacturing.⁷

This letter shows focusing of 10 keV photons⁸ with a refractive x-ray lens made from Li. Lithium is attractive because it gives x-rays the largest phase shift per attenuation length. This figure of merit⁹ for refractive lens materials is δ/β , or its various equivalents. Here δ is the refractive index decrement in the well-known¹⁰ index of refraction $n(k) = 1 - \delta - i\beta$ for x-rays with wave vector $k = 2\pi/\lambda$, while $\beta = 1/(2\ell_a|k|)$ is the attenuation (of the amplitude, per radian: ℓ_a is the usual attenuation length for the intensity).

Lithium is not yet common in x-ray research, in part because of lack of familiarity and because it needs special handling and safety measures. Although Li burns in water and corrodes rapidly in humid air, it is stable when the air is dry enough (tens of ppm), and in dry argon or another inert environment. In such Li-friendly circumstances Li is easy to shape as required for a lens.

Lithium lenses can even be handled for some time in normal, humid air if they are covered after manufacture with a submicron coating of parylene.¹¹ Parylene 0.3 μm thick protects Li for many minutes, long enough to transfer the lens from a dry transportation container to another protective environment wherein the Li will be exposed to the x-rays. The lens here operates inside a 125 mm long vacuum cross that has two 125 μm thick and 19 mm diameter beryllium windows for the x-ray beam. It is kept under ~ 100 μPa pressure with an ion pump. Under these conditions the lens is stable: after one month in operation the lens still focuses as before.

For manufacturing ease this Li lens uses Cederstrom's¹² sawtooth or alligator geometry: the lens looks like two jaws with many small teeth. Figure 1 shows 30 of the 80 Li teeth in one such jaw, photographed from the top. The teeth are 6.3 mm wide, 1.5 mm apart, and 0.75 mm high, with a tooth angle of 90° . The teeth are held rigidly in a brass mold whose edge is visible on the top left and bottom right. The teeth are nice and straight, but they are not perfectly sharp. The image shows one dark stripe betraying contamination of the Li surface in the center parallel to the jaw, and similar contamination close to the brass holders.

The focusing tests are performed with 10 keV x-rays from the double crystal monochromator on the 7ID undulator beamline of the Advanced Photon Source, operated by the University of Michigan, Howard University, Lucent Technologies-Bell Labs Collaborative Access Team (MHATT-CAT). For ease of alignment, the test uses only a

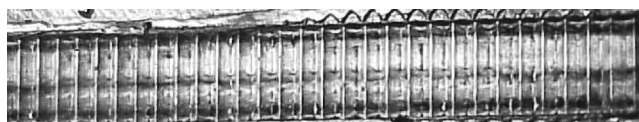


FIG. 1. Closeup of teeth in Li lens.

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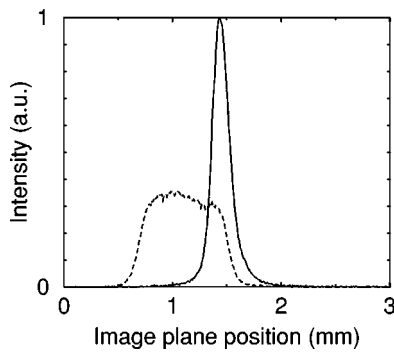


FIG. 2. X-ray intensity in original (dashed) and focused beam (solid) 8.5 m behind Li lens.

single jaw, with the focusing dimension horizontal. The measured gain would double by using a second jaw collecting the second half of the beam. The horizontal source size is 0.8 mm FWHM. A white beam slit 26.5 m from the source collimates the beam to 1 mm horizontally and 0.5 mm vertically.

The lens is 49.2 m from the source. A second slit 75 mm upstream of the lens limits the beam to 0.7 mm, less than the 0.75 mm high teeth in the single jaw under test. The aperturing avoids blurring of the focus from x-rays beyond the parabolic approximation valid¹² for the alligator lens. The focused x-rays create visible fluorescence in a 0.5 mm thick YAG:Ce-doped single crystal located 8.5 m behind the Li lens. The fluorescence is imaged 1:1 onto a *k*-Space Qmax650 CCD camera. The measurement is linear in x-ray intensity: its resolution is 19.5 μm or about 3 pixels FWHM.

Figure 2 is a horizontal cross section through the original beam (dashed) and the focused beam (solid). The original beam is a 0.82 mm wide projection of the 0.7 mm slit on the scintillator 8.5 m downstream. The focused beam's center is shifted 0.5 mm, corresponding to an average deflection by 30 teeth (each 90° top-angle tooth deflects over an angle 2δ , where $\delta=10^{-6}$).¹³ The FWHM is 0.18 mm, 40 μm more than the 0.14 mm FWHM expected from the $M=5.8$ demagnification of the 0.8 mm wide source 49.2 m away.

For a top-hat initial beam, the average x-ray transmission through the Li is $T=0.74$. The theoretical gain $G=MT$ is then 4.5. From Fig. 2 the actual gain is 3. The reduction in gain is roughly consistent with the 40% larger size of the focal spot. Both lower gain and larger focal spot probably reflect imperfections in the lens teeth. The teeth in Fig. 1 line up well and they are straight, but also slightly rounded at the top. However, rounding affects only a small portion of the beam, and can be ignored.

The dominant problem seems to be small angle scattering, most likely from surface roughness. The surface quality of the Li teeth is comparable to that of the die surface. The fabrication process for this die, conventional machining without subsequent lapping or polishing, cannot give surfaces better than 1 μm (40 $\mu\text{in.}$) rms. The roughness is mostly parallel to the teeth, and the resulting scattering is mostly in the (horizontal) focusing direction.

That roughness may be important is clear from a simple estimate. Each surface with $d=1$ μm rms roughness adds a random phase shift $k\delta d=0.05$ radians onto a 10 keV beam (the wave vector $k=5\times 10^{10}/\text{m}$). Randomly adding the scat-

tering from $2N=160$ surfaces gives a random phase variation of $\sqrt{2N}\times 0.05=0.6$ radians, which is appreciable.

As seen previously,¹⁴ scattering is obvious when a thin x-ray beam shines straight through the lens. In those tests an initially 29 μm wide and uniform beam scatters into a 0.1 to 0.15 mm wide swath at 7 m. A second half-length Li lens prototype transmits this same beam almost without widening. The latter lens is made with a die consisting of 1 mm thick microscope slides at a 45° angle. Glass surfaces are optically smooth, i.e., a rms roughness of 25 nm or better. Then, as observed, a lens made with a glass die should scatter little.

It is encouraging that small-angle scattering decreases when the tooth surface is made smoother. Complete suppression of small-angle scatter by still smoother surfaces would show that scattering is not intrinsic to Li metal, as it is for some kinds of beryllium⁷ and for graphite.¹⁵ Here scattering seems to prevent the lens from achieving its limit, although figure errors may also contribute.

How does Li as material for an x-ray lens compare with other lens materials (Be, plastics, or Al)? A good measure for the Li refractive lens is the fraction of theoretical performance. For the intensity gain, this fraction is 0.66 at 10 keV for the Li refractive lens here. The scaling of gain with material is well known theoretically:⁹ in one dimension the gain scales as δ/β , in two dimensions as $(\delta/\beta)^2$. At 10 keV Li δ/β is more than twice that of beryllium and an order of magnitude more than plastics. Therefore, our 0.66-quality Li lens should outperform the same type of lens made from ideal Be by at least 25%, and an ideal lens from plastics at least fivefold. At higher photon energies, Compton scattering exceeds the Li photoelectric absorption. Then Li's δ/β converges to that of other materials. For these harder photons, the other low atomic number materials are just as good as Li, and may be preferred for their manufacturing or handling convenience.

Even though further work is needed to make fully satisfactory x-ray lenses from Li, the data from our prototype single-jaw lens already prove that Li is a viable material. Lithium's tabulated x-ray attenuation and refractive index decrement are consistent with recent optical measurements on our Li lenses.¹⁶ The prototype lens shows scattering that may be avoidable with better manufacturing, while intrinsic scattering in the Li has not yet been identified as a problem.

Deterioration of the Li is not a problem either. The Li lens has been in operation on the 7ID beamline for over a month, without any visible change in parameters. Its thrice-higher peak intensity has already proven to be useful in an ongoing experiment.

Future work will include verifying the predicted lens properties at lower photon energies, measuring the refractive constant and small angle scattering properties of Li with more precision, and combining two jaws into a single 1D lens.¹² A further step is to make Li lenses two dimensional. Parabolic lenses with almost optical quality surfaces made from aluminum achieve a diffraction-limited focal spot size:⁴ similar lenses from Li should realize the maximum possible⁹ gain achievable with x-ray refraction.

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¹S. Suehiro, H. Miyaji, and H. Hayashi, *Nature (London)* **352**, 385 (1991).

²Refractive lenses have been considered and judged impractical since the early days of x-ray research, viz., P. Kirkpatrick and H. H. Pattee, *X-Ray Microscopy in The Encyclopedia of Physics*, edited by S. Flugge, p. 323 (1957); A. G. Michette, *Optical Systems for X-Rays* (Plenum, New York, 1986); A. G. Michette, *Nature (London)* **353**, 510 (1991).

³A. Snigirev, V. Kohn, A. Snigireva, and B. Lengeler, *Nature (London)* **384**, 49 (1996).

⁴B. Lengeler, J. Tuemmler, A. Snigirev, I. Snigireva, and C. Raven, *J. Appl. Phys.* **84**, 5855 (1998); B. Lengeler, C. Schroer, J. Tuemmler, B. Benner, M. Richwin, A. Snigirev, I. Snigireva, and M. Drakopoulos, *J. Synchrotron Radiat.* **6**, 1153 (1999). For other papers see www.physik.rwth-aachen.de/group/physik2b/xray/.

⁵V. V. Aristov, V. V. Starkov, L. G. Shabel'nikov, S. M. Kuznetsov, A. P. Ushakova, M. V. Grigoriev, and V. M. Tseilin, *Opt. Commun.* **161**, 203 (1999); V. V. Aristov, M. V. Grigoriev, S. M. Kuznetsov, L. G. Shabel'nikov, V. A. Yunkin, M. Hoffman, and E. Voges, *ibid.* **177**, 33 (2000); B. Cederstrom, Ph.D. thesis, Royal Institute of Technology (Stockholm, Sweden), 2001; (available from his web site on www.particle.kth.se/).

⁶H. R. Beguiristain, M. A. Piestrup, R. H. Pantell, C. K. Gary, J. T. Cremer, and R. Tatchyn, in *Synchrotron Radiation Instrumentation: Eleventh U.S. National Conference*, edited by P. Pianetta (AIP, Melville, NY, 2000), Vol. CP521; M. A. Piestrup, J. T. Cremer, H. R. Beguiristain, C. K. Gary, and R. H. Pantell, *Rev. Sci. Instrum.* **71**, 4375 (2000); plastic lenses continue to improve as the surface gets closer to optical quality [M. A. Piestrup, private communication (2001)].

⁷Snigirev-Lengeler type Be lenses are available from www.accel.com. Several CRL developers considered beryllium, e.g., R. K. Smither A. M.

Khounsary, and S. L. Xu, *Proc. SPIE* **3151**, 150 (1997); P. Elleaume, *Nucl. Instrum. Methods Phys. Res. A* **412**, 483 (1998); A. Q. R. Baron, Y. Komura, V. V. Krishnamurthy, Yu. V. Shvyd'ko, and T. Ishikawa, *J. Synchrotron Radiat.* **6**, 953 (1999); M. A. Piestrup (personal communication); B. Cederstrom, Ref. 5. Intrinsic small-angle scattering from many materials is documented by J. Tuemmler, Ph.D. thesis, RWTH Aachen, Germany, 2000; available from the website in Ref. 4.

⁸There are no published reports on successful lenses from Li, although some CRL developers tried, e.g., J. Tuemmler, Ph.D. thesis, Ref. 7; C. Schroer (private communication).

⁹B. X. Yang, *Nucl. Instrum. Methods Phys. Res. A* **328**, 578 (1993).

¹⁰See, e.g., A. H. Compton and S. K. Allison, *X-Rays in Theory and Experiment* (Van Nostrand, Amsterdam, 1935); A. G. Michette and C. J. Buckley, *X-Ray Science and Technology* (IOP, 1993); D. Attwood, *Soft X-Rays and Extreme Ultraviolet Radiation* (Cambridge University Press, Cambridge, UK, 2000).

¹¹Data for parylene-coated Li are available on Ecopulse's web site: www.ecopulse.com.

¹²B. Cederstrom, R. Cahn, M. Danielsson, M. Lundqvist, and D. Nygren, *Nature (London)* **404**, 951 (2000); also B. Cederstrom, Ref. 5.

¹³See, e.g., www-cxro.lbl.gov. Its data come from B. L. Henke, E. M. Gullickson, and J. C. Davis, *At. Data Nucl. Data Tables* **54**, 181 (1993). Also physics.nist.gov/Phys.Ref.Data. Its optical constants are based on C. T. Chantler, *J. Phys. Chem. Ref. Data* **24**, 71 (1995).

¹⁴N. R. Pereira, E. Dufresne, D. A. Arms, R. Clarke, S. B. Dierker, and D. Foster, *Proc. SPIE* **4502**, (2001). The paper is available on: www.ecopulse.com and mhatt.aps.anl.gov.

¹⁵Early measurements of δ used a single prism, e.g., A. E. Lindl in *Handbuch der Experimentalphysik*, edited by W. Wien and F. Harms (Akademische Verlagsgesellschaft, 1930), Vol. 24, p. 114; B. Davis and C. M. Slack, *Phys. Rev.* **27**, 18 (1926); C. M. Slack, *ibid.* **27**, 691 (1926).

¹⁶D. A. Arms, N. R. Pereira, E. M. Dufresne, R. Clarke, S. B. Dierker, and D. Foster, *Rev. Sci. Instrum.* (to be published). The actual deflection is consistent with the literature's $\delta=0.96\times 10^{-6}$. The 58 mm attenuation length in our Li is consistent with NIST's 55 mm.