

permanent, but is reversibly enlarged or reduced with temperature.

The temperature dependence of the dynamic contribution to pore size may be estimated by means of mode coupling theory, which views a gel as consisting of N pores of diameter ξ over which the density fluctuations are correlated (6–8).

$$f = N6\pi\eta\xi \quad (2)$$

The theory further suggests

$$N = N_0 \left(\frac{\xi_0}{\xi} \right)^2 \text{ and } \xi = \xi_0 \left(\frac{T_{f=0} - T}{T} \right)^{-\nu} \quad (3)$$

where $T_{f=0}$ denotes the spinodal temperature (that is, the temperature at which the density fluctuations diverge). Thus

$$\frac{f}{\eta} = 6\pi N_0 \xi_0 \left(\frac{T_{f=0} - T}{T} \right)^{\nu} \quad (4)$$

The least-square analysis yields $\nu = 1/4$ and $T_{f=0} = 33.59^\circ\text{C}$ (Fig. 4).

The determination of various critical exponents indicated that the phase transition of NIPA gels belongs to the universality class of three-dimensional Ising models (4). The exponent $1/4$ is much smaller than the theoretical critical exponent $5/8$ of the three-dimensional Ising model (9, 10). This discrepancy between the exponents may be because the isochore is not critical. Or it may be that the pore size diverged upon phase separation in the metastable region before the gel reached the spinodal line. In this case $T_{f=0}$ should be considered as the temperature at which the domain size grows to infinity rather than the spinodal temperature.

By choosing an optimal combination of solvent and temperature the phenomenon should be universally observed in any gel. A drastic and reversible change in the friction may have applications in separation technology and may be relevant to some biological transport phenomena.

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Synchrotron X-ray Diffraction from a Microscopic Single Crystal Under Pressure

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Metallic filaments with submicrometer diameters have been fabricated. Standard diffraction techniques with conventional x-ray sources were unsuccessful in identifying the structure of these materials. However, with the use of synchrotron radiation produced on a wiggler beam line, diffraction data were obtained in measurement periods as short as 10 milliseconds. Two cylindrical single crystals of bismuth were studied, each with a diameter of 0.22 ± 0.02 micrometer. The volume of sample illuminated for these measurements was 0.38 cubic micrometer, less than 0.5 femtoliter. The crystals are grown in glass capillaries, and, because bismuth expands on solidification, they are under a residual hoop stress. The crystallographic data indicate the presence of a linear compressive strain of about 2 percent, which is assumed to be the result of a residual stress of about 2 gigapascals.

A MATERIALS FABRICATION TECHNIQUE has been developed that provides the ability to draw ultrafine metallic filaments. Previously, very fine wires of metals could be drawn at a temperature at which the metal is molten and confined within a softened glass capillary; the capillary is drawn into a fiber containing an axial metal filament. By cascading this process, we have been able to

produce metallic filaments with submicrometer diameters. Because of the minute quantity of material, efforts to determine the crystallographic properties of single filaments with the use of conventional x-ray sources have been unsuccessful. However, by using polychromatic synchrotron radiation produced with a wiggler insertion device (1), we have obtained structural data in measurement periods as short as 10 ms. The volume of sample illuminated for these measurements was only $0.38 \mu\text{m}^3$, smaller than that reported in earlier x-ray crystallographic studies. We believe that this investigation opens a new vista of opportunities for crystallographic research on a broad range of materials that can only be made in minute (submicrometer) sizes. The data were of sufficient quality to allow identification of a linear compressive strain of about 2%, which is be-

lieved to be associated with a residual stress of about 2 GPa.

The fabrication process is an extension of a method described by Taylor (2). The Taylor process, as it is sometimes called, involves melting a small amount of metal within a glass capillary and then drawing a fiber from the softened glass. By using small-diameter capillaries and pulling them quickly, so as to reduce them to a small diameter before the glass cools below its working temperature, Taylor produced fibers as fine as $1 \mu\text{m}$ in diameter containing even finer filaments. Apparently there have been few subsequent efforts to produce submicrometer-diameter filaments by this process, although considerable research has been devoted to developing related methods for producing these materials; much of this is reviewed by Donald (3).

In the method reported here we used a Taylor wire as a starting material and two different glasses, one of which has a substantially higher working temperature than the other. First a Taylor wire of Bi was drawn with GE 180 aluminosilicate glass in an apparatus that radiatively heated the feed material with an inductively heated silicon carbide ring. The glass capillary was advanced into the hot zone at a constant rate as the fiber was drawn from the bottom. Because Bi expands on solidification, it was necessary to draw the initial fiber slowly to prevent this expansion from splitting the fiber. Pulling at a speed of about 1 cm s^{-1} resulted in unidirectional solidification along the fiber axis and permitted the volume expansion to be accommodated by a flow of excess liquid up into the melt pool maintained within

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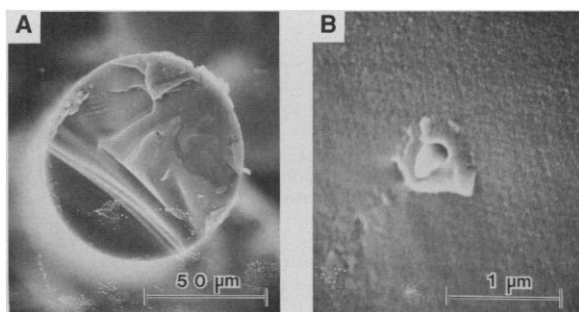


Fig. 1. (A) SEM view of the fractured end of a fiber segment. (B) Enlarged view of the fiber center showing the Bi core with a diameter of $0.22 \pm 0.02 \mu\text{m}$.

the unreduced portion of the capillary. The fiber so drawn had a diameter of about 0.34 mm and contained a Bi filament about 0.14 mm in diameter. This fiber was then inserted into a Pyrex capillary with outer and inner diameters of 7 and 1 mm, respectively. This combination was then drawn manually to further reduce the diameter of the Bi. This and a subsequent drawing were done at about 1000°C , which is below the temperature at which the 180 glass normally could be drawn. At this temperature, the highly viscous 180 glass core prevents surface energy-driven breakdown of the filament. After the manual drawing, the dimensions of the initial glass and the Bi core had been reduced to about 0.4 mm and $15 \mu\text{m}$, respectively. We produced the final filament by loading this into another Pyrex capillary, similar to the one described above, and mechanically pulling with spool

surface velocities ranging up to 1.2 m s^{-1} .

A scanning electron microscope (SEM) view of the fractured end of a fiber segment is shown in Fig. 1A; the fiber diameter is $87 \mu\text{m}$. An enlarged view of the center of this fiber is shown in Fig. 1B. The greatly deformed end of the Bi filament can be seen protruding from the circular hole that defined its perimeter before the fiber was fractured. The circular region of different contrast surrounding the filament defines the approximate extent of the aluminosilicate glass core. No evidence could be seen in the SEM of the interfaces between the different layers of Pyrex.

This SEM image (Fig. 1B), obtained from the same fiber segment as that used in the synchrotron experiments, was used to establish the diameter of the filament as $0.22 \pm 0.02 \mu\text{m}$. This measured diameter was within 10% of that predicted from a calculation based on an assumption that the reduction in diameter of the filament during the final drawing operation was proportional to the reduction in diameter of the glass capillary. Input to the calculation was the optically measured filament diameter after the second drawing.

We examined the filament within an intact length of fiber by optical microscopy, using reflected light at a magnification of $\times 1300$; no variations in apparent filament diameter or breaks could be seen. The filament, which was substantially smaller in diameter than the wavelength of light, could not be directly viewed by this technique, but it did produce refraction effects that allowed its presence to be confirmed.

Efforts to obtain structural information from a single filament with the use of conventional x-ray sources were unsuccessful. Therefore, the samples were brought to the National Synchrotron Light Source at Brookhaven National Laboratory. Single fibers were initially presumed to be polycrystalline and were examined for Debye-Scherrer cones of diffraction. They were illuminated with polychromatic radiation on a wiggler beam line (X17C). Several different samples were mounted with their fiber axes vertical in separate exposures to an x-ray beam with an approximate height of 5 mm and a width of $100 \mu\text{m}$. The samples were rotated about their fiber axes as the scattered radiation

was monitored with an energy-sensitive Ge detector at a fixed 2θ setting (θ is the dispersion angle). No diffraction peaks were observed. We then decided to treat the samples as if they were single crystals and scan areas of reciprocal space systematically in a search for diffracted beams. We used a four-circle goniometer and a computer-driven search routine to step-scan through reciprocal space (4). After an overnight search, we discovered three independent reflections. These peaks could be indexed to the normal Bi-I lattice; their positional coordinates allowed calculation of the orientation matrix. Subsequent centering and measurement of 25 independent Bi-I reflections followed.

Two different samples were mounted in turn on the χ circle of the diffractometer. Each fiber contained a Bi filament with a diameter of $0.22 \pm 0.02 \mu\text{m}$, and each was mounted with its fiber axis approximately coincident with the ϕ axis of the goniometer. In this orientation, reflections from planes whose reciprocal lattice vectors were normal to the fiber axis would have χ angles close to 0° , whereas those from planes with reciprocal lattice vectors parallel to the fiber axis would have χ angles close to 90° . Because energy-dispersive diffraction was used, when the crystal was aligned for a specific Bragg peak all members of that class, within the detection limits of the system, were recorded simultaneously (5). The measured $(0,k,2k)$ spectrum is shown in Fig. 2A.

Several years ago, two groups independently reported successful x-ray diffraction measurements on extremely small single crystals, using synchrotron-produced radiation: Eisenberger *et al.*, working on SPEAR at the Stanford Synchrotron Radiation Laboratory, Stanford University (6), and Bachmann *et al.*, working on DORIS-II at HASYLAB, Deutsches Elektronen-Synchrotron, in Hamburg, Federal Republic of Germany (7). More recently, the latter effort has been extended to smaller crystal sizes by Rieck *et al.*, using CHESS at Cornell University (8). Each of these efforts was direct-

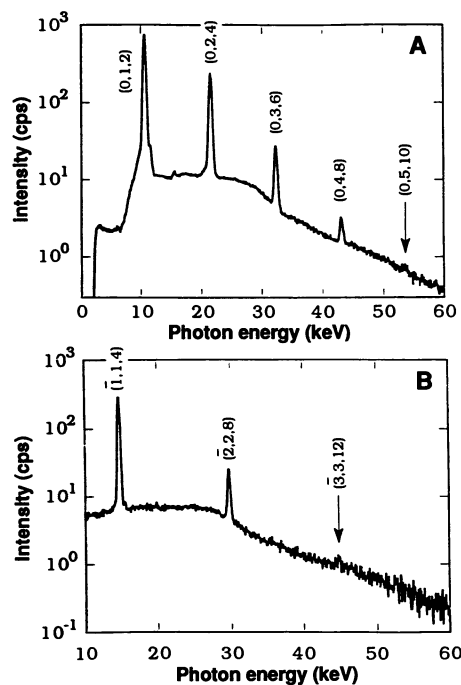


Fig. 2. (A) Energy-dispersive diffraction spectrum of the $(0,k,2k)$ class of reflections from the Bi filament recorded at $2\theta = 20.00^\circ$ for a period of 5 min. (B) Energy-dispersive diffraction spectrum of the $(h,h,4h)$ class of reflections recorded from a volume of $0.38 \mu\text{m}^3$ at $2\theta = 20.00^\circ$ for 1 min.

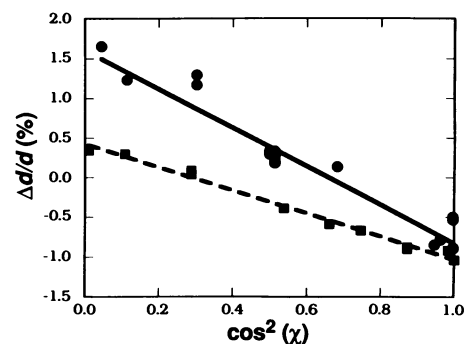


Fig. 3. Variation of linear strain with the angle, χ , between the reciprocal lattice vector and the filament axis for two different samples: sample 1 (circles and solid line) and sample 2 (squares and dashed line).

ed toward establishing the diffraction detection limits that are possible when synchrotron-produced radiation is used.

To compare different materials in terms of their relative ability to scatter x-rays, we use the scattering power, a dimensionless quantity:

$$\text{scattering power} = \left(\frac{F_{000}}{V_u} \right)^2 V_c \lambda^3 \quad (1)$$

where F_{000} and V_u are the number of electrons in, and the volume of, the unit cell, respectively, V_c is the volume of the crystal, and λ is the x-ray wavelength. For routine structure determinations, scattering powers typically range between 10^{16} and 10^{17} . Eisenberger *et al.* performed their measurements on an $800\text{-}\mu\text{m}^3$ crystal of zeolite, using a wiggler synchrotron beam diffracted from a double-crystal Ge monochromator set for 1.74 \AA (6). These values correspond to a scattering power of about 3×10^{14} . Bachmann *et al.* made measurements on a crystal of CaF_2 with a volume of $200\text{ }\mu\text{m}^3$ and x-rays of 0.91 \AA , giving a scattering power of 1.3×10^{14} (7). Rieck *et al.* used a CaF_2 crystal with a volume of $2.2\text{ }\mu\text{m}^3$ and 1.56 \AA photons; these correspond to a scattering power of 7.1×10^{12} (8). [We are also aware of preliminary measurements from a $0.4\text{-}\mu\text{m}$ single-crystal sphere of Mo reported at the 1990 International Union of Crystallography Congress held in Bordeaux, France, but insufficient details are given in the abstract to evaluate the scattering power. (9).] The work reported here represents an x-ray investigation of an unknown crystal of submicrometer dimensions; in the other studies, known test crystals were used.

In order to assess the diffraction detection limit of our system, we selected a reflection with a χ angle of about 0° and then systematically reduced the vertical aperture defining our incident beam and monitored the transmitted beam with an ionization chamber. There was a linear response down to $10\text{ }\mu\text{m}$; below that the readings were erratic. At this $10\text{-}\mu\text{m}$ setting, the volume of the sample illuminated was $0.38\text{ }\mu\text{m}^3$. The ($h, h, 4h$) class of reflections, shown in Fig. 2B, were recorded from this volume in a period of 1 min. The scattering powers calculated from Eq. 1 for each of the three peaks in Fig. 2B are 1.2×10^{12} for the (1,1,4), 1.6×10^{11} for the (2,2,8), and 4.4×10^{10} for the (3,3,12). These scattering powers are all smaller than those in the earlier published reports (6–8). Despite this diminished scattering power and the strong background signal from the encapsulating glass, there was still a relatively strong diffraction signal; the (3,3,12) reflection is weak, but it can be seen above this background. This result also demonstrates that it is not necessary to first monochromatize the illuminating x-rays, as was done in each of the earlier studies.

Interatomic planar distances, d spacings,

were calculated for each reflection from the measured energy of the diffraction peak. The peak centroids were determined by a least-square fit to a Gaussian curve; the reliability factors for most reflections were better than 99.9%. These measured d spacings were compared with those calculated from the published lattice parameters for Bi (10); the difference between the two values was defined as Δd . There was a systematic variation in $\Delta d/d$ with the χ angle, amounting to a net compressive linear strain of about 2.5% for one sample and about 1.5% for the other sample (Fig. 3). If the materials were isotropic, then the equivalent volumetric strains would result from stresses on the order of about 2 GPa. It is presumed that these residual stress states are caused by Bi expansion upon solidification in the presence of the encapsulating glasses.

Although crystallographic studies have been carried out on smaller samples by electron diffraction techniques, such procedures cannot be used on embedded crystals. Thinning the fibers examined here would have eliminated the residual strains. Removing volume constraints by thinning can even induce phase transformations in metastable crystals.

Note added: Since submitting this manuscript, we have successfully obtained single-

crystal diffraction data from three smaller Bi filaments; their calculated diameters are 905, 670, and 420 \AA . The scattering power for the (0,1,4) reflection measured from a volume of 28 attoliters was 1.8×10^{10} .

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Possible Horizontal Transfer of *Drosophila* Genes by the Mite *Proctolaelaps regalis*

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There is strong inferential evidence for recent horizontal gene transfer of the P (mobile) element to *Drosophila melanogaster* from a species of the *Drosophila willistoni* group. One potential vector of this transfer is a semiparasitic mite, *Proctolaelaps regalis* DeLeon, whose morphology, behavior, and co-occurrence with *Drosophila* are consistent with the properties necessary for such a vector. Southern blot hybridization, polymerase chain reaction (PCR) amplification, and DNA sequencing showed that samples of *P. regalis* associated with a P strain of *D. melanogaster* carried P element sequences. Similarly, *Drosophila* ribosomal DNA sequences were identified in *P. regalis* samples that had been associated with *Drosophila* cultures. These results have potentially important evolutionary implications, not only for understanding the mechanisms by which genes may be transferred between reproductively isolated species, but also for improved detection of some host-parasite and predator-prey relationships.

PLEMENTS CONSTITUTE ONE OF THE most intensively studied families of transposable (mobile) elements in

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Drosophila melanogaster (1). P elements are present in multiple copies in so-called P strains of this species, but are completely absent in others, designated M strains (2). The historical dichotomy of P and M strain distributions in *D. melanogaster* strongly suggests that P elements spread through this species as recently as the last half century (3, 4). This conclusion is also supported by the uneven worldwide geographical distribution