

# Section 1

## PROGRESS IN LASER FUSION

### 1.A X-Ray Microscopy of Inertial Fusion Targets Using a Laser-Produced Plasma as an X-Ray Source

#### Introduction

Currently, targets for direct-drive inertial fusion (IF) consist of glass microballoons that are filled with an equimolar ratio of the hydrogen isotopes deuterium and tritium (DT), and are overcoated with metallic x-ray signature layers and/or a plastic ablation layer. High spherical symmetry and an extremely smooth ( $<1\text{-}\mu\text{m}$ ) surface finish are required for successful implosion of the laser-driven IF target. With the use of submicron laser light to drive the implosion, these target parameters become especially stringent, i.e., the nonuniformities approach the laser wavelength itself.

In the past, a major perturbation in the implosion uniformity has been the drawn-glass capillary on which the IF target is mounted. X-ray pinhole camera images have clearly demonstrated the need to eliminate the stalk mounting because it is a channel for energy to leave the imploding target. The advent of submicron stalks such as spiderweb filaments<sup>1</sup> reduced this perturbation in many instances. However, ablation layer coatings could still not be performed in many cases with web-mounted microballoons because, upon being coated, the diameter of the stalk would increase by twice the thickness of the plastic coating.

With the introduction of the bouncing-pan parylene coating process,<sup>2</sup> the stalk perturbation has been eliminated. In this process, the filled glass or plastic microballoons are bounced on an aluminum cylinder,

with a pan-shaped end surface resonating at one of its natural longitudinal frequencies. The microballoons are bathed in a weak plasma to eliminate static charge buildup, which causes the extremely low-mass targets to stick to the pan. Using this technique, the IF targets can be coated with parylene without being supported and web mounted.

Several major drawbacks exist with this technique. The precharacterized microballoons lose their individual identity through the mixing that occurs during bouncing. Also, any particulate present in the pan during the coating procedure is collected by the targets and incorporated into the plastic coating. Therefore, recharacterization must be performed to determine the original parameters of each target, along with the coating uniformity and surface quality.

Several characterization techniques have been used in the past for IF targets, but few are suited for this purpose. For example, optical interferometry is limited to the characterization of transparent materials, but metal- and/or parylene-coated targets are either opaque or translucent at best. Another technique, scanning electron microscopy, is a destructive technique when used to determine coating thicknesses. Perhaps the most suitable technique for the recharacterization of bounce-coated microballoons is soft x-ray contact microradiography.<sup>3</sup>

X-ray microradiography has been described and utilized to characterize targets for several years by laboratories conducting IF experiments.<sup>4</sup> The process is capable of characterizing multilayered opaque IF targets with ease. The x-ray energy used can be specifically tailored to increase the contrast between the materials under examination. Also, x-ray microradiography is a batch characterization technique, capable of examining tens of targets in a single exposure. A typical image produced using this technique is shown (enlarged) in Fig. 30.1(a). The glass wall of the microballoon is the distinct white ring inside the lighter gray outline of the plastic coating. Various defects are made quite observable using this technique. For example, an enlargement of a surface defect evident in the outer rim of another image is given in Fig. 30.1(b).

It is clear from the enlarged image that the resolution limit of the x-ray contact microradiography technique is the grain size of the photographic emulsion used to create the original image. In the case of the Kodak 649-F plate used here, the grain size of the emulsion is of the order of  $0.5 \mu\text{m}$ ; therefore, the minimum feature size that may be observed would have to be as large as several grains, i.e., 1 to  $2 \mu\text{m}$ . Although the grain size limitation can be alleviated to some extent through the use of digital image processing,<sup>5</sup> the microradiography technique is limited in that it cannot be used to measure submicron features.

In this article, we describe a soft x-ray contact microscopy technique that is inherently capable of submicron resolution. The process, a hybrid of contact x-ray microradiography and x-ray lithography,

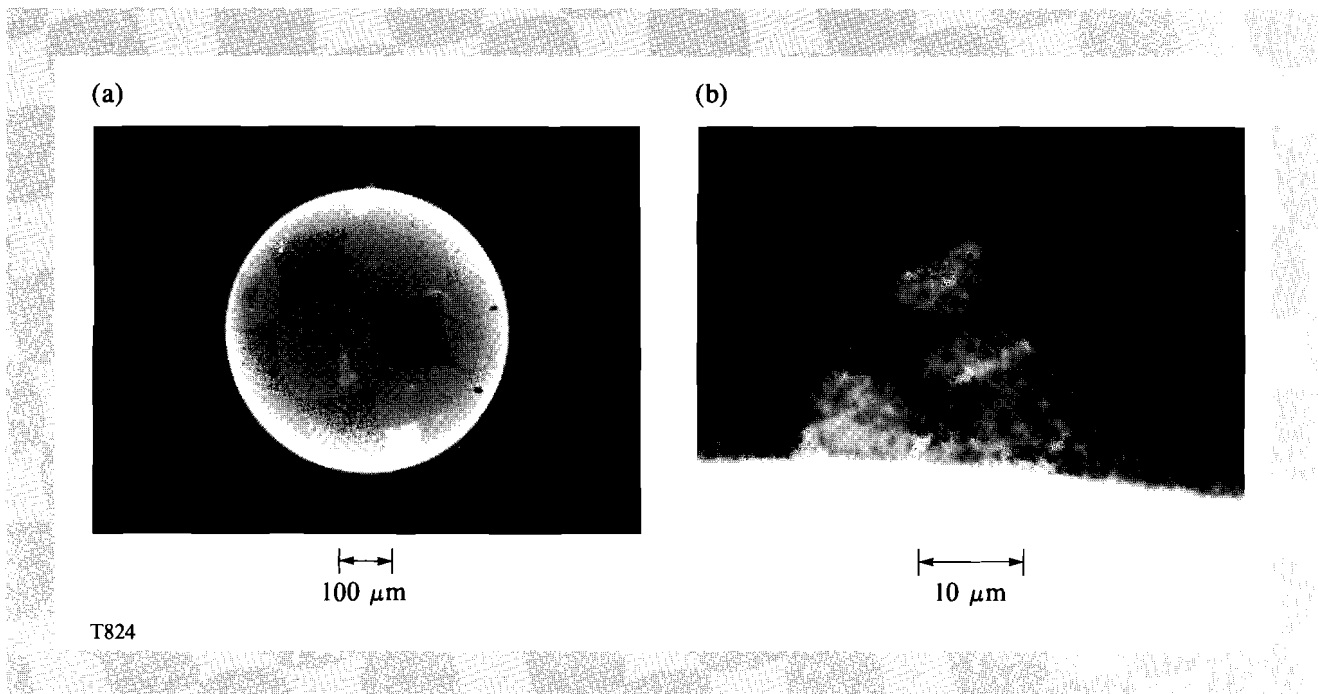


Fig. 30.1

- (a) X-ray microradiograph of a plastic-coated glass microballoon.  
 (b) X-ray microradiograph of a surface defect in the plastic coating on a glass microballoon.

employs an x-ray sensitive photoresist as the recording medium. First developed for biological cell morphological characterization,<sup>6</sup> the process has been adapted here for use as a target characterization scheme by using a laser-produced plasma as an x-ray source.

#### Background

Images of x-ray lithographic masks with a resolution of tens of nanometers have been recorded using the x-ray resist polymethyl methacrylate (PMMA).<sup>7</sup> The resolution of a photoresist is inversely proportional to its sensitivity. Hence, the more sensitive resist poly(butene-1-sulfone) (PBS) was used in this experiment because the intensity of our laser-produced plasma x-ray source is insufficient to expose PMMA properly.

Contact x-ray microscopy is an improvement of conventional soft x-ray contact microradiography. A specimen is held in intimate contact with a polymer photoresist and is irradiated at normal incidence with soft x rays. The resist suffers different degrees of local radiation damage in direct proportion to the x-ray transmittance of the specimen's structure. The molecular weight of the resist is therefore locally decreased, causing an increased solubility of that region in an appropriate solvent. Upon development, the surface of the resist represents a two-dimensional topographic map of the three-dimensional specimen's density distribution.

Soft x-ray contact microscopy has undergone much development in past years in biological cell morphology and cell chemical composition determination.<sup>8</sup> Due to the shorter wavelength of x rays, better resolution than light microscopy can be obtained. However, this technique cannot match the resolution of the scanning electron microscope.

The x-ray source used in previous soft x-ray microscopy work was either a stationary target or synchrotron radiation.<sup>9</sup> Both the stationary target source and the more intense and tunable synchrotron source have the disadvantage of emitting a continuous flux of x rays. Thus, any vibration in the imaging apparatus or change in the sample would blur the resulting image of the specimen.

A pulsed x-ray source is desirable for most soft x-ray microscopy work. One such source, the gas puff z-pinch,<sup>10</sup> has been proven to be less than an optimum x-ray source. A laser-produced plasma as an x-ray source has recently been demonstrated in several papers.<sup>11</sup> Source characteristics of laser-produced plasma x-rays, such as very small size, subnanosecond pulse duration, and tunability of wavelength, are the properties most desirable for x-ray lithography, and are not available from any other type of source. The x-ray flux has been shown to be sufficient to produce a relief image in many photoresist materials. The sensitivity of some x-ray resists was found to be one order of magnitude higher than the sensitivity published when a laser-produced plasma was used in place of conventional x-ray sources.<sup>12</sup>

### Experimental

A schematic of the experimental configuration is shown in Fig. 30.2. The photoresist, which was filtered to 0.2- $\mu\text{m}$  maximum particulate size upon shipment, was dispensed directly from the bottle and spin coated onto silicon wafer substrates to yield a film thickness of approximately 0.5  $\mu\text{m}$ . This thickness was determined using thickness versus spin-rate curves supplied by the PBS manufacturer.<sup>13</sup> (Thicker films may be desirable to maximize the x-ray dose absorbed in the resist; however, as in photographic emulsions, a loss in resolution may result from the use of excessively thick resist films.) The coated substrates were then prebaked at 120°C for one hour to eliminate any residual solvent present in the resist film and to promote adhesion to the silicon substrate. The photoresist-coated wafer was then held in intimate contact with the CH-coated IF targets, which were supported between two tensioned, 2- $\mu\text{m}$ -thick polyester films. A 2- $\mu\text{m}$ -thick polyester film coated with 1000 Å of aluminum was placed between this assembly and the plasma x-ray source to filter out radiation with wavelengths longer than those of the soft x-rays. Earlier experiments had shown an intense background overexposure that had produced only a shadow of the IF target, thus overwhelming the internal structure of the target made visible by soft x rays. Filtration eliminated the previously encountered long-wavelength background exposure, and the image produced was solely due to soft x-ray exposure.

To create the x-ray-emitting plasmas, the frequency-doubled GDL (Nd:glass laser) operating at a 1-ns pulse duration with a wavelength of 534 nm was focused to a 100- $\mu\text{m}$ -diameter spot onto a metal foil target. The target chamber, which contained the imaging assembly, was evacuated to a background pressure of the order of  $10^{-6}$  torr. The microscopy setup was placed at a distance of 10 cm from and directly facing the laser target, and was oriented at an angle of 30° with respect to the incoming laser beam, which irradiated the foil target at normal incidence. Using this geometry, the penumbral blurring due to the finite extent of the x-ray source is of the order of 0.2  $\mu\text{m}$ .

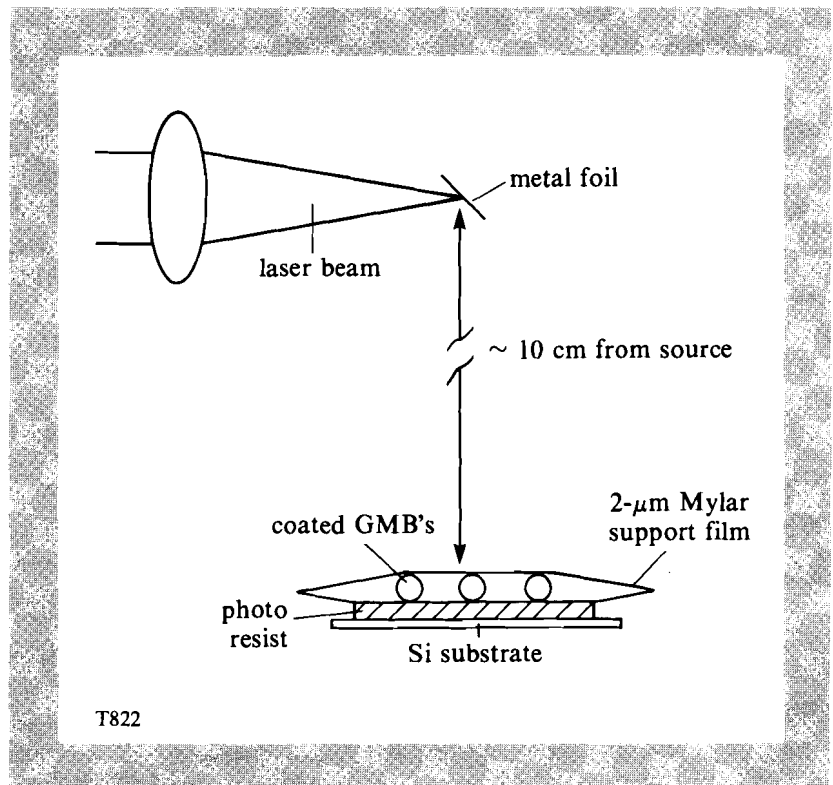


Fig. 30.2  
Experimental configuration of x-ray microscopy, using a laser-produced plasma as an x-ray source.

Various target materials were tested in order to obtain the x-ray spectral characteristics and intensity required to produce useful images. X-ray emissions from silver, gold, copper, iron, and titanium foils irradiated by a single, 534-nm laser shot with an energy of 120 J were not energetic enough to produce significant image contrast between the CH coating and the underlying glass microshell. On the other hand, single laser shots with the same energy characteristics on molybdenum foils did produce x rays with the necessary spectral properties to make the plastic-glass interface quite visible. Multiple laser shots on molybdenum foils with the same photoresist sample remaining in the imaging assembly enabled a greater degree of x-ray exposure and created a clearly visible image of the plastic-glass interface with good contrast.

Upon exposure, the photoresist samples were developed as follows. The PBS-coated silicon wafer was held in a rapidly flowing stream of developer consisting of a 70% methyl isoamyl ketone/30% 2-pentanone mixture for 5 seconds. The wafer was then dipped in a 60% methyl isoamyl ketone/40% isopropyl alcohol rinse for 20 seconds. Compressed dry nitrogen was blown on the sample until the liquids were completely removed. The image present on the surface of the resist was then examined under a differential interference contrast microscope. The above development process was repeated until the desired contrast at the plastic-glass interface and the overall background-to-image contrast were obtained. Typical total times that the resist spent in the developer ranged from 5 to 25 seconds. Finally, to greatly increase the contrast of the resultant image, the photoresist film was postbaked for 30 min at 120°C. As stated by the

manufacturer, the total linewidth variation resulting from the development procedure given above should be approximately  $0.1 \mu\text{m}$ .

### Results and Discussion

Several types of representative IF targets were used to evaluate this characterization technique. To examine the feasibility of the x-ray microscopy process in determining CH-coating parameters, glass microballoons and solid glass spheres with several-microns-thick plastic coatings were used. Also, in an effort to evaluate the ultimate resolution of this technique, batches of identical, precharacterized glass microballoons were coated with  $0.4$  to  $1.0 \mu\text{m}$  of parylene in thickness increments of  $0.2 \pm 0.05 \mu\text{m}$ . These IF targets were then simultaneously evaluated using the x-ray microscopy technique with a laser-produced plasma. For comparison purposes, x-ray microscopy of the same targets was also performed using a conventional x-ray source producing  $15\text{-kV}_{\text{peak}}$  bremsstrahlung at a source current of  $2.5 \text{ mA}$ . Using the conventional source under the same geometrical conditions as previously stated, exposures of two to four days were required to produce images of similar clarity.

Figure 30.3 shows an image typical of those produced, using the technique described in this report. This image was one of several made to determine if the necessary contrast could be obtained at the plastic glass interface and between the subject and background, and thus prove the feasibility of this technique. It was produced under the previously described laser and geometric conditions by two successive laser shots of nearly equal energy, which totaled  $265 \text{ J}$  on a molybdenum foil. Clearly visible is the  $6\text{-}\mu\text{m}$ -thick CH coating on the

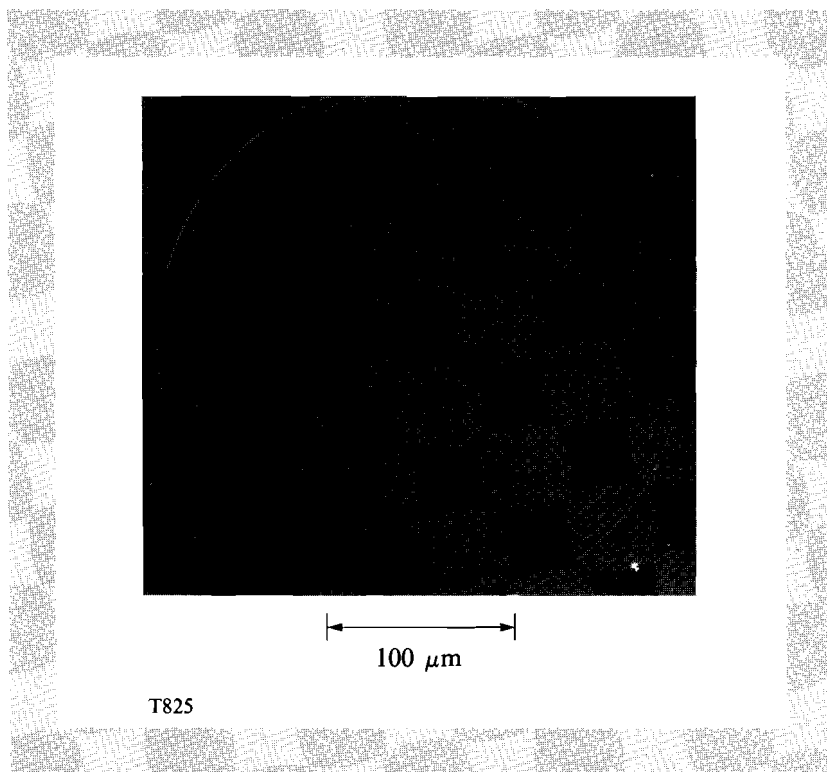


Fig. 30.3  
X-ray micrograph of a glass microballoon coated with a  $6\text{-}\mu\text{m}$ -thick parylene layer.

solid glass sphere. The variation in contrast about the perimeter of the image is a function of the optics used to view it and should be disregarded.

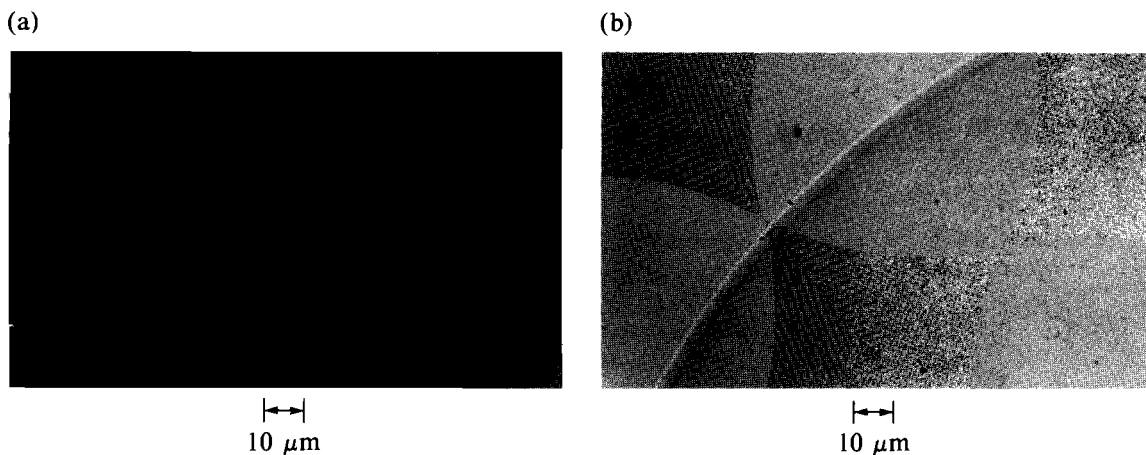
Figures 30.4(a) and 30.4(b) are included for image-quality comparison. In each, the outer rim of an image of the same IF target is shown. The image in Fig. 30.4(a) was made using the same laser-produced plasma as the image in Fig. 30.3. On the other hand, the one shown in Fig.30.4(b) was made using a conventional x-ray source under the conditions stated earlier. The laser-produced plasma emits x rays that do not have sufficient energy to appreciably pass through the glass shell; hence, only the 0.6- $\mu\text{m}$  CH coating on its surface is visible in Fig. 30.4(a). On the other hand, the image in Fig. 30.4(b), produced using 15-kV<sub>peak</sub> bremsstrahlung, clearly shows the 2- $\mu\text{m}$  wall of the glass shell and, to a lesser extent, the outer CH coating. X rays in this energy range are not only sufficiently energetic to penetrate the glass microballoon, but they are capable of producing reasonable contrast between the glass and plastic layers.

In Fig. 30.4(a), there are no distinct lines delineating the plastic-glass and image-background interfaces. Instead, these regions are bordered by a brighter ring of finite width. This is chiefly caused by penumbral blurring because of the finite extent of the source, and the ultimate resolution of the microscope optics used to view and enlarge it.

Penumbral blurring of the image can be reduced by increasing the distance from the source to the sample; however, penumbral blurring decreases linearly with increased distance, while the x-ray irradiance on the imaging medium falls off quadratically. Hence, exposure is sacrificed as the penumbral blurring is reduced. The spatial resolution

Fig. 30.4

- (a) X-ray micrograph of a glass microballoon overcoated with a 0.6- $\mu\text{m}$ -thick parylene layer.
- (b) X-ray micrograph of the same IF target but using a conventional bremsstrahlung source.



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can be improved by using scanning electron microscopy to view the resultant image; but, because PBS was designed to be an electron beam resist, damage to the image occurs immediately upon examination. Certain replication techniques and subsequent viewing with a transmission electron microscope have been developed to alleviate this problem.<sup>14</sup>

A comparison can be made between the images shown in Figs. 30.4(a) and 30.5 to determine experimentally the ultimate resolution of this technique under the present geometrical conditions. In Fig. 30.4(a), the  $0.6\text{-}\mu\text{m}$  CH coating is comprised of two  $0.2\text{-}\mu\text{m}$  blurred rings as described above, with a  $0.2\text{-}\mu\text{m}$  gap between them. The outer blurred ring corresponds to the outer edge of the target and the inner blurred ring to the CH-glass interface. However, this gap is not seen in the image of the  $0.4\text{-}\mu\text{m}$  coating shown in Fig. 30.5: the blurred interfaces between the glass and plastic and between the image and background have merged and, therefore, the limit of the resolution is of the order of half this coating thickness, i.e.,  $\sim 0.2\text{ }\mu\text{m}$ . This number is in agreement with the penumbral blurring as calculated from the previously stated geometric conditions; it is also of the order of the resolution limit of an optical microscope. Therefore, using this technique under the present conditions, material coating thicknesses under  $0.4\text{ }\mu\text{m}$  cannot be determined.

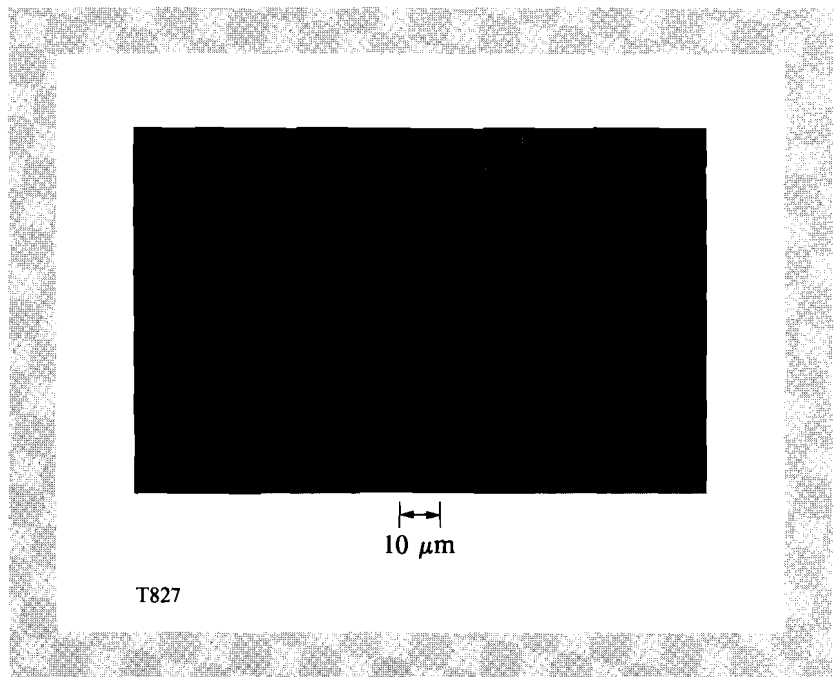


Fig. 30.5  
X-ray micrograph of a glass microballoon overcoated with a  $0.4\text{-}\mu\text{m}$ -thick parylene layer.

### Conclusions

We have developed a soft x-ray contact microscopy technique to characterize submicron CH coatings on IF targets with a resolution of  $\sim 0.2\text{ }\mu\text{m}$ . The process is a hybrid of soft x-ray contact microradiography and x-ray lithography, and uses a laser-produced plasma as an x-ray source. When time is not a constraint, the process can be performed in the laboratory with a conventional stationary target x-ray source to yield similar results. The most significant



advantage of this technique over microradiography is that it enables submicron resolution of CH coating layers without the need for costly and time-consuming digital image analysis.

#### ACKNOWLEDGMENT

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#### REFERENCES

1. B. A. Brinker, J. M. Cavese, J. R. Miller, S. G. Noyes, S. Sheble, and L. T. Whitaker, *J. Vac. Sci. Technol. A* **1**, 941 (1983).
2. R. Q. Gram, H. Kim, J. F. Mason, and M. Wittman, *J. Vac. Sci. Technol. A* **4**, 1145 (1981).
3. H. Kim and M. D. Wittman, *J. Vac. Sci. Technol. A* **3**, 1262 (1985).
4. T. M. Henderson, D. E. Cielaszyk, and R. J. Simms, *Rev. Sci. Instrum.* **48**, 835 (1977); R. L. Whitman and R. H. Day, *Appl. Opt.* **19**, 1718 (1980); R. M. Singleton and J. T. Weir, *J. Vac. Sci. Technol. A* **18**, 1264 (1981).
5. R. M. Day *et al.*, 8th International Congress on X-Ray Optics and Microanalysis (1977), pp. 260-267.
6. R. Feder and D. Sayre, *Ann. NY Acad. Sci.* **342**, 213 (1980).
7. E. Spiller, R. Feder, and J. Topalian, *Phys. Technol.* **22** (January 1977).
8. P. C. Cheng, J. Wm. McGowan, K. H. Tan, R. Feder, and D. M. Shinozaki, in *Examining the Submicron World*, edited by R. Feder *et al.* (Plenum, New York, 1986).
9. R. Feder *et al.*, *Science* **197**, 259 (1977); K. H. Tan, P. C. Cheng, G. M. Bancroft, and J. Wm. McGowan, *Can. J. Spectrosc.* **29**, 134 (1984).
10. J. Bailey, Y. Ettinger, A. Fisher, and R. Feder, *Appl. Phys. Lett.* **40**, 33 (1982); R. Feder, J. S. Pearlman, J. C. Riordan, and J. L. Costa, *J. Microsc.* **135**, 347 (1984).
11. M. L. Gimer, in *X-Ray Microscopy*, edited by G. Schmahl and D. Rudolph (Springer-Verlag, 1984); P. Gohil *et al.*, *Appl. Opt.* **24**, 2024 (1985).
12. B. Yaakobi, H. Kim, J. M. Soures, H. W. Deckman, and J. Dunsmuir, *Appl. Phys. Lett.* **43**, 686 (1983).
13. Mead Technologies, Inc., Rolla, MO.
14. P. C. Cheng *et al.*, *Nuclear Instruments and Methods in Physics Research A* **246**, 668-674 (1986).