Early history of X-ray lithography at IBM

by E. Spiller

We present a reconstruction of the early work on X-ray lithography at the IBM East Fishkill facility in 1969 and 1970 and a summary of the efforts at the Thomas J. Watson Research Center in Yorktown Heights, New York, between 1973 and 1976.

Introduction

In the late 1960s, integrated circuits had a linewidth of 5 μm and were fabricated by proximity printing with blue light and a separation of 25 μm between mask and wafer. Fresnel diffraction was a serious problem for this large separation, and it appeared difficult to produce dramatically higher circuit densities with this technique. At the IBM Thomas J. Watson Research Center in Yorktown Heights, New York, Thornley and Hatzakis [1] had demonstrated that devices with linewidths under 1 μm could be produced by electron beam writing. This work led to a major expansion of the electron beam efforts at the Yorktown Heights Research Center. Electron beam lithography was seen as the future lithographic technology for the fabrication of computer chips, whereas optical lithography was considered an old technology with little promise for the future and not worth a major effort in a research center. There was, however, at the Research Center a small group under J. Wilczynski, funded mainly from sources outside IBM Research, which designed and built optical projection systems; the message from this group, that optics would be able to produce linewidths as small as 1 μm, did not find much support.

IBM's efforts in X-ray lithography began at the East Fishkill plant, where L. Spector had organized a small group to study lithographic tools. R. Feder joined this group in the fall of 1968, on leave from IBM Research, "to make some useful contribution for the company." D. Havas of Spector's group discussed the problems with Fresnel diffraction in proximity printing at a seminar in August 1969. Feder suggested after the talk that the use of X-rays instead of light would eliminate all of these problems; he began some experiments before he returned to Yorktown Heights in the fall of 1969. A more official project to study X-ray lithography (involving D. Havas, R. Horwath, and F. Laming) took place in Poughkeepsie from October 1969 to January 1971.

This paper describes the work done in East Fishkill and Poughkeepsie from 1969 to 1970 and subsequent work at IBM Research in Yorktown Heights from 1973 to 1976. I was a member of the team in Yorktown Heights from 1973 to 1976, and my description of this period is based on personal recollection, notebook entries, and outside publications. Finding records for the work done from 1969 to 1970 was more difficult. These results have not been published in the open literature, and most of the internal records have disappeared or cannot be found. R. Feder and R. Horwath have retired from IBM, and their notebooks have been lost. D. Havas provided copies of his relevant notebook entries; the main information sources are two internal reports [2, 3].

The difficulties I encountered in locating documents concerning work done twenty years ago within the

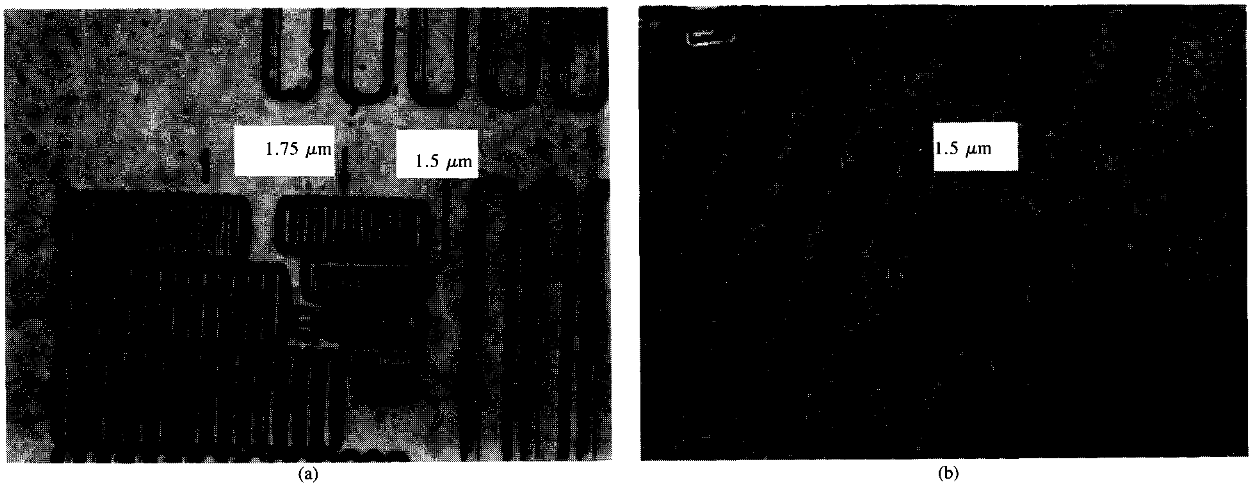

Figure 1

Mask pattern (Au on Si) of a high-resolution pattern (a) and X-ray ($\lambda = 2.3$ Å) proximity copy of the pattern on a photographic plate (b) (from Feder, 1970).

company surprised me. I was not able to retrieve a single document from the stored records. The memory of a large company is the memory of its people, as contained in their personal files. Technical knowledge is lost when a person leaves, especially information that was originally too sensitive to be published. While the open literature provides an established retrieval system that works well, internal classified documents are much harder to locate, and in practice results that are not published are not only lost to the technical community but also to the people within a company.

X-ray lithography at East Fishkill

• *Technical work*

Feder used the last two months of his visit in East Fishkill for the first tests of his idea that proximity printing with X-rays instead of light would eliminate all diffraction problems. He obtained access to a Philips X-ray diffractometer which could be operated with either a copper or a chromium anode and produced copies of various masks on photographic plates. Mask substrates were 0.2-mm-thick wafers of beryllium or silicon, and the pattern was made of 1.5-μm-thick gold. One set of mask patterns was fabricated by evaporating gold through a metal shadow mask with 0.1-mm holes which was in contact with the mask substrate. For other sets, a U.S. Air Force resolution target was contact-printed with visible light onto a photoresist layer (AZ1350). The developed photoresist served as a mask to sputter-etch a 1.5-μm-thick gold layer.

For the X-ray exposures the photographic plate remained in a film cassette with the mask separated from the film by about 1 mm. There was a 10-cm-long, helium-filled "collimating" tube between the source and the film, and exposure times ranged from 5 s to 18 min. **Figure 1(a)** shows an optical micrograph of such a mask [2] with the smallest details measuring about 1.5 μm. The best image obtained from the Air Force target is shown in **Figure 1(b)**. Certainly all details visible in the mask have been successfully transferred to the film.

The X-ray lithography project in Spector's group, which had moved to the Poughkeepsie plant, officially began in October 1969, just after Feder returned to Yorktown Heights. The group defined three areas as important: mask materials, X-ray sources, and X-ray resist materials.

In addition to the masks used by Feder, the group fabricated masks on boron nitride substrates and used actual electronic circuit patterns. Some of their first exposures were produced in the medical department, using a Picker medical X-ray unit with tungsten anode operated at 40 kV and 50 mA. The mask was placed on top of a cassette with photographic film at a distance of 1 mm from the source and exposed between 1/10 and 1/20 s. In his notebook, Havas describes the setup but not the result, probably because no pattern was recognized on the film, since the mask pattern (with 750 Å of bismuth absorber) provided insufficient contrast under the hard X-rays emitted from the source.

The group, recognizing that better contrast required the use of longer wavelength, in the spring of 1970 acquired a Philips X-ray diffraction unit with targets of chromium (λ = 2.3 Å) and copper (λ = 1.54 Å). These were the longest wavelengths that could be obtained from sealed X-ray tubes and could still propagate for a short distance through air. The best images were obtained with $\lambda = 2.3$ Å on Kodak high-resolution photographic plates.

There were some efforts to use resist instead of photographic film. Initial attempts with polymethyl methacrylate (PMMA) resist [4] were not successful. There were some efforts with Laming to produce more sensitive resist [5]; some patterns were successfully recorded in polyvinyl chloride, but seemed to be of low quality.

Radiation damage to electronic circuits by X-rays was of some concern. Feder had observed a small shift in the threshold voltage of field-effect transistors caused by X-rays. The effect could be reduced by annealing and was considered unimportant for X-ray lithography.

• *Patent and publication efforts*

A notebook entry from August 18, 1969 indicates that R. Feder, D. Havas, and R. Horwath were submitting an invention disclosure to the patent department of the East Fishkill plant. Later there are short notes in Havas's progress reports about problems with the patent department (November 1969 and March 1970). The attitude of the patent department was that the project should be classified as secret, no patent should be filed, and no outside publication would be permitted. One argument, quoted by Feder, was that X-rays would be too dangerous! The experimental work was terminated by December 1970.

A report by Feder from August 28, 1970, was classified as IBM confidential; very few people outside the original group knew about it. Feder returned to Yorktown Heights in the fall of 1969 full of enthusiasm for the potential of X-ray lithography, but somewhat disappointed about his efforts to contribute to IBM's manufacturing capabilities. He proposed to continue the work at Yorktown Heights, with plans to use Al K_α ($\lambda = 8.3$ Å) and to expose resist materials; however, he could not convince management that the program was worthwhile, or that he should be allowed to publish his results.

Havas and Horwath gave a final presentation to East Fishkill management on January 22, 1971; their report, dated December 10, 1971, was classified as IBM confidential [3]. However, they were allowed to present some of their results outside the company at the Fall Meeting of the Electrochemical Society in October 1972 [6]. They were less optimistic than Feder about the potential of X-ray lithography. They were worried that backscattering of electrons from the substrate would reduce resolution and contrast, and that the mask substrate would transmit only the undesirable harder X-rays, which are very inefficient for the exposure of resist. They were aware that soft X-rays could give much better replication, but considered the use of a vacuum system for X-ray lithography to be unacceptable.

There are some indications that there were many additional discussions about X-ray lithography within IBM in the 1960s. I. Haller found among his old files a memorandum to J. Schiller from 1965, in which he estimated that about 100 hours' exposure would be required for polymer resist using a dental X-ray machine as source.[1] He also found an invention disclosure of L. Kaplan from March 1969, which proposed X-ray lithography.[2] Kaplan suggested a wavelength of 7 Å with 0.1-mm-thick glass as mask substrate. The transmission of such a mask would have been around 10^{-80}! All efforts at IBM before 1972 seemed to have one common problem: The mask substrate was about 100 times thicker than the X-ray resist. X-rays which were able to penetrate such a thick mask substrate without substantial absorption could not be absorbed by the thin resist, making the system very inefficient. The "horror vacui" was another severe handicap for early efforts within IBM.

The Spears–Smith papers

The two papers on X-ray lithography by Spears and Smith [7, 8] appeared in 1972. Their work went beyond the East Fishkill effort in making the step to longer wavelength ($\lambda = 8.3$ Å). This was made possible through the use of thinner mask substrates (3-μm-thick Si membranes) and a modified electron beam evaporator with an Al target as an X-ray source. The source and the mask–wafer assembly were mounted in a vacuum system, thus avoiding any air absorption. This system had sufficient intensity to expose PMMA resist and to fabricate an acoustic surface wave transducer with 1.3-μm electrode spacing as the first device. The papers were noticed at IBM Research, and a task force (W. Crowder, J. Cocke, E. Spiller, and J. Wilczynski) was formed in the spring of 1973 to evaluate X-ray lithography and to recommend a course of action. The result was the formation of a small group (Feder, Spiller, and J. Topalian) to explore X-ray lithography and, if feasible, use it for the fabrication of magnetic bubble memories. Bubble memories appeared to be an ideal vehicle for demonstrating the technology, because they required only one high-resolution lithographic exposure, thus eliminating the need to design and build a high-resolution alignment system. Furthermore, it appeared that a capability to fabricate submicron lines would immediately be useful for the fabrication of high-density devices [9].

There was some concern as to how to manage the program. It was obvious that electron beam lithography would be needed to write the mask, and that the work should be performed within the electron beam lithography group. However, there was a possible conflict of interest,

[1] I. Haller, memorandum to J. Schiller, November 30, 1965.
[2] L. H. Kaplan, "High Resolution Masked X-Ray Exposure of Photoresists," IBM Invention Disclosure FI8-69-0345, March 24, 1969.

because X-ray lithography could be seen as competing with electron beam lithography. The final agreement was that the new group would be part of the electron beam lithography department, but that all technical decisions would be made within the group and no one outside the group would have authority to interfere with the technical work. We decided to report the group's progress in such a way that no individual would receive special credit.

The following were defined as first goals:

1. Confirmation of the work of Spears and Smith.
2. Exploring whether throughput was sufficient for production.
3. Exploring limits of resolution.
4. Fabricating magnetic bubble memories.

We were confident that these goals could be reached within a short time.

X-ray lithography at IBM Research from 1973 to 1976

Confirmation of the Spears–Smith result took only a few days' time. Since I had worked on a project to fabricate multilayer mirrors for the soft X-ray region, with the long-term goal of producing an imaging system for lithographic applications [10], I had an electron beam evaporation system. The evaporator could easily be transformed into an X-ray source for $\lambda = 8.3$ Å by loading the crucible with aluminum. PMMA spun on a silicon wafer was exposed to the radiation from this source with a fine copper mesh. Exposure doses and development times similar to those by Spears and Smith showed good replication of the copper mask in the resist films.

The preparation of a laboratory took several months; it was completed in November 1973. The laboratory was equipped with a clean room with hoods for the processing of resists, equipment for electroplating metal patterns, an evaporator for thin-film deposition, and microscopes for inspection and measurement of the patterns. A vacuum chamber with a small electron beam evaporator served as an X-ray source, and a dome at a distance of 18 cm could hold up to 19 mask–wafer combinations, which could be exposed simultaneously. We had access to a scanning electron microscope for inspection of resist patterns and devices. Masks were fabricated for us with electron beam lithography (H. Luhn, P. Chang), and we learned about electroplating from L. Romankiw and E. Castellani.

The substrates for the masks were initially 6- or 3-μm-thick Mylar foils stretched over a stainless steel ring. However, commercial Mylar film contained some particles with sufficient X-ray contrast to show up in the copies; therefore, we switched to polyimide membranes mounted on a ring of silicon, which we could fabricate ourselves with the required cleanliness. We also did some tests with boron-doped silicon membranes and with double layers of Si_3N_4 and SiO_2 [11]. The membranes were coated with about 200 Å of gold, which served as a conducting base for the electroplating of the pattern into the developed resist. We did not use the electron-beam-generated mask directly to make devices, but used copies, made with X-rays, which had thicker Au with better-defined pattern sidewalls and higher contrast.

The daily routine in the new laboratory soon developed into a standard pattern. The exposure system was loaded with masks and wafers every evening, and the night was used for exposure. This resulted in an exposure time of 16 hours, with a much larger distance between source and wafer than would have been necessary to obtain the required resolution. The day was then used for processing of the wafers and for preparation of the next batch of wafers. We also decided that every member of the group should be involved in every aspect of the work, such that work would not stop if one member of the group were absent.

• *Throughput*

Work on improving the throughput of the system had two obvious directions: more sensitive resists and brighter sources. The theoretical limit of the sensitivity of any photon detector is determined by the shot noise in the number of detected photons. Thus, it was very easy to estimate that one should be able to increase the sensitivity of resists over that of PMMA by more than a factor of 1000 for a resolution of 0.1 μm. Improving the resist therefore seemed to offer the largest payoff. One way to improve the sensitivity of a resist, obvious to us, was to increase its absorption of X-rays.

We contacted Haller and Romankiw in August 1973, asking them for methods of incorporating heavy elements into PMMA resist. Romankiw provided samples in which heavy elements (Au, U) were incorporated in colloidal form, while Haller synthesized copolymers of methyl methacrylate and of the Tl and Cs salts of methacrylic acid. Interestingly, the copolymer worked very well and showed improved sensitivity, even without the heavy-metal inclusion [12].

Another way to increase the absorption of the resist was to overcoat it with a thin layer of a strong absorber. The secondary electrons emitted from the absorber would travel into the resist layer beneath and provide an additional exposure. We used erbium as an overcoat because it had the required high absorption and because it could be removed very easily from the resist surface with a mild acid before the resist was developed.

There was also a small effort to build a more powerful X-ray source to replace our small e-beam evaporator [13]. However, the most powerful X-ray source available was an electron synchrotron, and we arranged a trip to the

DESY synchrotron in Hamburg, W. Germany, in June 1975. D. Eastman and W. Gudat joined us for this project. The hospitality of our hosts from the DESY synchrotron was remarkable; our visit was scheduled during the last three weeks before a major shutdown, and we received almost all of the beam time for our experiments during the last ten days before the shutdown! We could replicate patterns down to 700-Å linewidth and obtained good 1-μm line patterns for mask–wafer spacings up to 1 mm [14]. The paper describing these results had considerable impact; it was used as an important document in support of the construction of dedicated storage rings for synchrotron radiation. Every storage ring built since has a beamline for X-ray lithography.

There was no doubt after this work that X-ray lithography could provide any throughput one would ever need, if a dedicated storage ring was used as an X-ray source.

• *Resolution*

The range of the secondary electrons, which are generated by the absorption of an X-ray photon, represents one resolution limit for X-ray lithography. The thin film of erbium on top of the resist, which we had used to enhance resist sensitivity, gave us an elegant method to measure this range. An increase in the exposure dose near the erbium layer was caused by the absorption of the secondary electrons emitted from the erbium into the resist, and the resist showed an increased dissolution within the range of these secondary electrons. A measurement of the dissolution rate versus thickness for exposures with various photon energies therefore yielded the effective range of the exposing electrons. This range increased with photon energy and had a value of $\delta = 50$ Å for $\lambda = 50$ Å [15]. We concluded that 50 Å would be the ultimate resolution of X-ray lithography obtainable with $\lambda = 50$ Å for the case in which mask and wafer were in intimate contact, because at this wavelength the range of secondary electrons matched the resolution limit given by diffraction. Since we did not have masks with such fine features, we used thin biological specimens to explore the replication of very fine details.

The smallest features on our electron-beam-generated masks were 700 Å (a zone plate pattern provided by M. Hatzakis), and we had no problems replicating these features. Patterns having a 500-Å linewidth, produced by electroplating gold into fine cracks in resist, could be replicated and processed faithfully, while features smaller than 100 Å could be seen in shadowgraphs of biological objects [16, 17]. It turned out that the resolution of PMMA was very close to its theoretical limit as determined from the range of secondary electrons, and that the sensitivity was close to the limit given by the shot effect of the photoelectrons.

• *Bubble memory and other applications*

Starting in 1974, we delivered resist patterns to the bubble memory project. We were all very excited, because we believed that bubble memories could become an important product, and we were sure that we could supply the lithographic tool to manufacture these devices, to linewidths below 0.1 μm. However, the enthusiasm for bubble memories faded, and plans to develop a product were canceled in 1976.

Considering all the capabilities of X-ray lithography, such as good throughput and resolution, large depth of focus, the capability to write on top of a complicated topography, and large processing tolerances, we were sure that X-ray lithography would become the tool of choice for fabrication of all other integrated circuits in the future.

However, optical lithography had changed dramatically in the time between 1969 and 1976. Proximity printing had been replaced by projection printing, and there was no doubt that optics could produce patterns with linewidths below 1 μm. X-ray lithography could offer an easier process, because of its larger exposure and development latitude. However, the transition to X-ray lithography was conceived in the factories as a large change in technology with many problems still unknown. It became clear that managers in factories would always prefer small, more evolutionary improvements in optical lithography to any more drastic change, and that the switch to X-ray lithography would only occur when optics had reached its limit.

We estimated that the transition would occur when devices reached linewidths below 0.5 μm and that the need for such resolution would occur after 1990.[3] My personal judgment was that this was more time than was needed to develop the technique into a manufacturing tool, and that it would be more interesting to use some of the time to develop X-ray imaging systems using multilayer X-ray mirrors. I requested a leave of absence from the program and transferred to the Physical Sciences Department to pursue these efforts [10]. Feder also went to the Physical Sciences Department and continued to work in X-ray microscopy, while Topalian moved to Boca Raton.

• *Publication activity*

The problems which Feder had experienced in East Fishkill did not reappear at Yorktown Heights. We presented our first results at the Symposium on Electron, Ion and Photon Beam Technology in May 1975, and prepared two manuscripts [18, 19], one for the *Journal of Vacuum Science and Technology* and the other for the *IBM Journal of Research and Development*. Although the papers had been cleared by our local management, the manuscript for the IBM *Journal* was still troubled by the

[3] E. Spiller, memos to management, October 20, 1976 and June 25, 1977.

ghosts that had haunted Feder in East Fishkill. Because the editor needed divisional clearances which required that certain critical technical details be withheld, we eventually withdrew the manuscript and sent it to *Solid State Technology*, where it was published in April 1976.

A final paper, which reviewed our work and the state of the art in X-ray lithography, was published in 1977 [20].

Epilog

The project began with the goal of replacing optical lithography in manufacturing by providing a tool with higher resolution and higher yield which could be used for the fabrication of magnetic bubble devices. After the loss of the bubble memory project, silicon devices appeared to be the next obvious application for the technology. However, even today, fifteen years later, optical lithography is still the tool of choice, and no chips are produced by X-ray lithography. In hindsight, our work in X-ray lithography was done much too early. This seems to be a common scenario for work done at research and development laboratories. The most exciting problems for researchers are topics that are new and exciting to other researchers; being ahead of everybody is one of the main goals. It is, however, not a good investment decision to develop a technology before it is needed; a tool should be ready just in time and not earlier. Technical feasibility is only one necessary requirement for success; market forces and economic competition usually determine the final outcome. Any improvement in the decision process, in deciding which development program to pursue, would represent a huge savings in the development costs for a large company. A broadening of the process such that any possible dissent or alternative technology is better included might help, but it could also slow down the process. And there is the additional danger that a good project might not be pursued because the evaluation was too critical.

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