

# Glasses for lithography

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## Abstract

The highest resolution in a lithographic process is often determined by the properties of the resist material. With the currently used polymeric resists, a resolution of better than 100 nm has been achieved under manufacturing conditions, but the future nanoscale devices will require a 10 times superior resolution. In this paper we present an overview of the resist materials, especially with regard to limiting resolution. In principle, inorganic resists should have higher limiting resolution than polymer resists due to smaller fundamental structural units and stronger bonds in the former. However, compositional and/or structural inhomogeneities may limit their ultimate resolution. New results are presented that indicate chalcogenide glasses as promising photo and electron beam resists, which also have the advantages of greater hardness, resistance to acids, easy fabrication in thin film form, and the unique phenomena like radiation enhanced diffusion.

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## 1. Introduction

Lithography, in the context of modern technology, refers to the process which replicates a micropattern or an electronic circuit rapidly. The manufacture of numerous electronic, optoelectronic and photonic devices uses lithography as a necessary technique for fabricating micrometer size features in a substrate. For example, in microelectronics technology, transistors and other electrical circuit elements are produced in silicon. The desired modification of the electrical properties is accomplished by doping a pre-determined region with appropriate dopants. By comparison, the fabrication of microelectromechanical system (MEMS) requires removal of pre-selected regions of the substrate. Optoelectronic and photonic devices may require

compositional as well as topological modification of the substrate.

Overall, lithography involves an exposure (irradiation) source, a mask, a resist, and a series of fabrication steps that would accomplish pattern transfer from the mask to resist and then to substrate in which device is fabricated. The creation of mask or the use of resist is based on materials that are sensitive to specific radiation. Complex patterns are transferred into a resist by irradiating its selected regions. Due to its radiation-sensitivity the chemical reactivity of exposed regions is modified with respect to unexposed regions. A developer then preferentially etches out either the exposed (positive resist) or unexposed (negative resist) region, thus faithfully transferring the original pattern into the resist. Over the years the feature size of the elements to be fabricated by lithography has been decreasing continuously. With the push for nanofabrication of nano-electromechanical systems (NEMS) [1], single electron devices [2], electrical connection of individual molecules [3], etc., there is an emerging need for sub-10 nm structures. The ability to alter the structure and properties of a resist at this length

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scale will naturally introduce new technology for ultra-high density storage. In the next section, we examine the current status of resists, in particular the resolution limitations of present materials and the prospects of new materials.

## 2. Resist materials for lithography

Most lithography processes employ light-sensitive resists. The resolution of these photoresists is limited by the diffraction limit of the laser beam. Therefore, attempt is made to develop the lithography process using shorter and shorter wavelength light, with a corresponding optimization of polymer resists [4]. Considering the difficulty with the focusing of X-rays, there is practical limit of resolution using short wavelength light. As a result, the most promising nano-resolution lithography process of the future is likely to be based on electron beam lithography (EBL). Commercial EBL equipment is available, which allows patterning with  $\sim 1$  nm diameter electron beam. Note that EBL is a relatively slow process, which is more suitable for making master masks than large scale manufacturing. However, there has been parallel progress of the stamping technology, which in combination with such masks may indeed produce features of the size of several nm [5].

There is a variety of organic polymers and inorganic materials that are sensitive to radiation and hence suitable as resist. At present, polymethyl methacrylate (PMMA) based polymer resists containing small amounts of light-sensitive active agents are used in nearly all microelectronic processing. Structures  $< 20$  nm in width have been prepared using polymer EB resists [6], but 100 nm is a more typical dimension of the features manufactured by current technology [7].

The polymer resists are generally applied on the substrate by a convenient, inexpensive spin coating process. The process has a limit of 3:1 for the aspect ratio (image height to width ratio), which can be obtained in a feature [8]. Thus for fabricating 25 nm features the thickness of the resist should be  $< 100$  nm, which is too small for plasma etch transfer of the pattern into underlying layers. As a result, it appears that the finest features that can be fabricated using polymer resists is  $\sim 25$  nm, unless new polymers are developed with a much higher selectivity to etching than possible at present.

In view of the above mentioned limitations of the polymer resists, inorganic resists are being considered, especially for high resolution nano-lithography. Indeed, there are two good reasons that, in principle, inorganic resists should give higher resolution compared to organic polymers: (a) Organic polymers are made of much larger, long chain molecules than inorganic solids. Typical polymer resists have molecular weight of  $5\text{--}20 \times 10^3$  units, giving molecular pixel size of 4–9 nm. By comparison, homogeneous inorganic glasses have building block of size  $\leq 1$  nm, which will ultimately limit their resolution. Obviously, to achieve such high resolution, it will be necessary to produce films of homogeneity on the 1 nm scale. (b) In EBL using finely focused beam, the resolution is primarily

limited by the structural changes produced by the secondary electrons. Because the polymer resists are made of light elements and weak van der Waal bonding, the undesirable region affected by secondary electron scattering is expected to be significantly larger than that in the inorganic resists that are made of stronger covalent–ionic bond and often heavier elements. The operation of polymer resists is based on the fact that their degree of polymerization and hence subsequent reactivity to an etchant is altered by irradiation. Modification of atomic structure is also the basis of inorganic resists. However, some inorganic resists undergo compositional changes under irradiation, in addition to structural changes. Then the selectivity of such resists is greatly enhanced.

On structural grounds, inorganic resists may be divided into two categories: (a) Glassy resists, and (b) Polycrystalline resists. Silica glass [3], chalcogenide glasses [9], and amorphous peroxopolytungstic acid [10] are examples of the former whereas polycrystalline fluorides [11] and Al–Al<sub>2</sub>O<sub>3</sub> cermets [12] are examples of the latter. In addition, there are resists that include both inorganic and polymer components, e.g. spin-on-glass (SOG) that is a –Si–O polymer with attached methyl groups [13], and the composite of polymer resist with silica nano-particles [8]. Jeyakumar and Henderson [14] have evaluated the advantages of tethering heavy inorganic elements in organic resists. Due to space limitation, in the remainder of this paper we will focus only on inorganic glass resists.

## 3. Glass resists

The glass resists can be superior to currently used polymer resists in a number of applications. Like polymers, inorganic glasses can be fabricated as uniform film over large surfaces, both planar and non-planar. They have been prepared by vacuum evaporation, chemical vapor deposition (CVD), plasma enhanced CVD, radio frequency (RF) sputtering methods, etc. For over four decades chalcogenide glasses are known to be photosensitive. The effects of exposure to bandgap light on the physical and chemical properties of these materials have been investigated extensively. Therefore, much of the information about glass resists concerns chalcogenide glasses. By comparison, common oxide glasses are considered inert materials and, therefore, have been presumed to be unsuitable for lithography. Notwithstanding, limited observations are reported which indicate that even silica glass can function as an electron beam resist. In the following sections, we review these two types of glass resists.

### 3.1. Silica glass resist

Amorphous silica has major advantage that it is compatible with silicon technology. Because of the strong Si–O bond, amorphous silica film is commonly used as a protective layer rather than as a resist. Then it is particularly interesting that the foundation of silica as a resist

was laid by O’Keefe and Handy in 1968 when they reported that silica film thermally grown on silicon would dissolve several times faster in the so called ‘p-etch’ (HF:HNO<sub>3</sub>:H<sub>2</sub>O in 15:10:300) when it was irradiated with 1–15 kV electrons [15]. Unfortunately, the charging of silica film by electron beam easily attracts carbon contamination, which affects the etching process and results have poor reproducibility. Therefore, the usefulness of such films as an EB resist could not be established, until Allee and Broers proposed the use of a 100 nm thick PMMA sacrificial layer [16]. The sacrificial film was sufficiently thin for the electrons to transmit through and modify the structure of underlying silica film. At the same time, all the contamination was retained on the polymer film which was then uniformly dissolved away in acetone. Thus underlying silica film was modified by the electron beam but without contamination and could be used as an EB resist. By using high accelerating voltage of 300 kV, the forward scattering of electron was minimized and array of lines with separation as small as 21 nm could be fabricated within the silica film.

A realistic demonstration of silica as a practical EB resist has been made recently by Tsutsumi et al. [3] They have fabricated a Si nanowire memory transistor on silicon-on-insulator (SOI) wafer by using positive silica resist and EB lithography. This demo device is based on 15 nm wide and 20 nm thick nanowire that is fabricated with dimensional accuracy of 4.2% at the nm scale. The steps of the fabrication process are summarized in Fig. 1. Note that no sacrificial layer was needed. After a treatment with

oxygen plasma, the resist was developed for 110 s in a buffered HF solution (0.2 mol/dm<sup>3</sup> HF + 0.2 mol/dm<sup>3</sup> NH<sub>4</sub>F). The resulting silica layer served well as a mask for the subsequent removal of Si by reactive ion etching.

It is important to recognize that the etching rate of silica film is altered by exposure to electron beam even with 1–15 kV energy, which is much less than the radiation damage threshold for atomic displacements. Therefore, the EB lithographic resolution need not be limited by the damage from secondary electrons. Although the exact mechanism of EB lithographic process is not established, O’Keefe and Handy [15] suggest that the selectivity of EB irradiated silica arises primarily from the beam-induced loss of oxygen on the surface and/or the production of defects in the film [17].

### 3.2. Chalcogenide glass resists

Chalcogenide glasses have been shown as potential high resolution resists for photo [18–20], electron [21,22] and X-ray [23] lithography. For certain processing conditions these glasses may even be superior due to their greater hardness, resistance to acids, easy fabrication in thin film form, or the unique phenomena that they exhibit like photo-doping or photo-diffusion. Due to the high sensitivity of chalcogenide glass resists, exceptionally thin (10s nm) films are sufficient which are expected to yield better control of geometry than possible with polymer resists. Chalcogenide glass resists are suitable for selective etching of SiO<sub>2</sub>, Si, Cr, etc., which is used in microelectronic

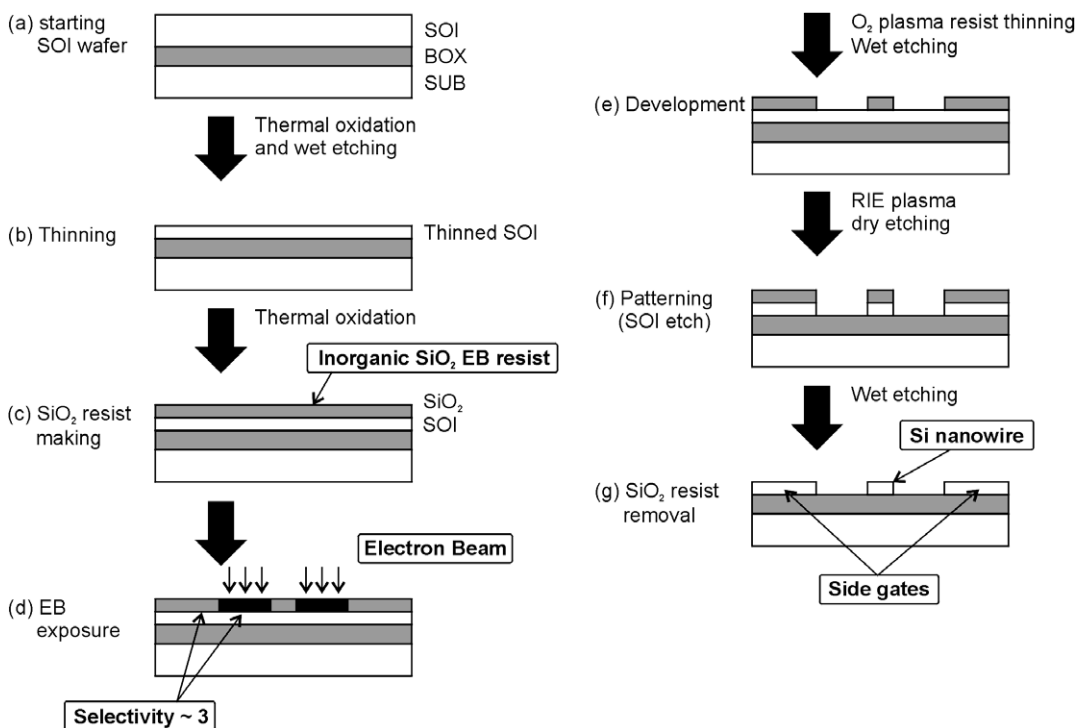


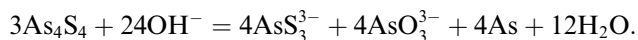
Fig. 1. Steps for the fabrication of a silicon nanowire transistor by electron beam (EB) lithography based on silica glass film resist [3], where SOI is silicon-on-insulator and RIE is reactive ion etching.

processing. The pre-baking and post-baking steps of polymer based lithography are completely eliminated. Finally, by simply changing the composition of the developer, certain chalcogenide glass can be used as a positive or a negative photoresist [24].

Overall, sulfides and selenides of arsenic and germanium have been evaluated (a few studies have also investigated the effect of doping of such chalcogenides with Ga, In or Tl), with  $\text{As}_2\text{S}_3$  being the prototypic model composition. Typically, thin films of chalcogenide glass such as  $\text{As}_2\text{S}_3$  are deposited by vacuum deposition on a silicon or glass substrate. In principle, stoichiometric bulk  $\text{As}_2\text{S}_3$  glass in metastable equilibrium should consist of As atoms that are bonded to three S atoms, and the S atoms that are bonded to two As atoms; all atoms being present in relatively stable heteropolar As–S bonds. The fast quench rate during film formation introduces structural units with bonding defects. In the case of  $\text{As}_2\text{S}_3$  thin films, energetically less stable, wrong bonds are formed within structural units, e.g. S–S bonds within  $\text{S}_n$  chains,  $\text{S}_8$  rings,  $\text{S}_2$  bridges, etc., or As–As bonds within  $\text{As}_4\text{S}_4$  and  $\text{As}_4\text{S}_3$  cages (bulk samples of off-stoichiometric compositions will always have less stable homopolar bonds, but in their thin films the concentration of wrong bonds is even higher). These bonds are transformed into As–S stable bonds by exposure to light [25]



Different concentration of these bonding defects in as-evaporated and exposed films is the fundamental basis of selective etching of chalcogenide glasses. Presence of  $\text{As}_4\text{S}_4$  units in the structure of as-prepared films lowers their dissolution in alkaline medium as insoluble As thin film is formed on the surface of the layer during etching according to [25]:



The presence of units containing hydrophobic S–S bonds further reduces dissolution rate of as-evaporated films. By comparison, the exposed samples are chemically closer to the ideal, stoichiometric structure and hence dissolve easily according to [25]:



Thus positive type selective etching takes place when previously exposed  $\text{As}_2\text{S}_3$  film is etched in aqueous alkaline solvents. Similar behavior can be found even for non-stoichiometric films of As–S system where structural units with homopolar bonds are expected in both, as-prepared and exposed films, but in different concentration.

When the films are etched in amine based solvents, the consequence of photoinduced structural changes is just the opposite. In this case a negative resist is observed with the exposed part having lower solubility. The difference in etching rates of the exposed and unexposed films is seen in Fig. 2, where film thickness is plotted as a function of time of etching in a trimethyl amine solvent. Note that the slope

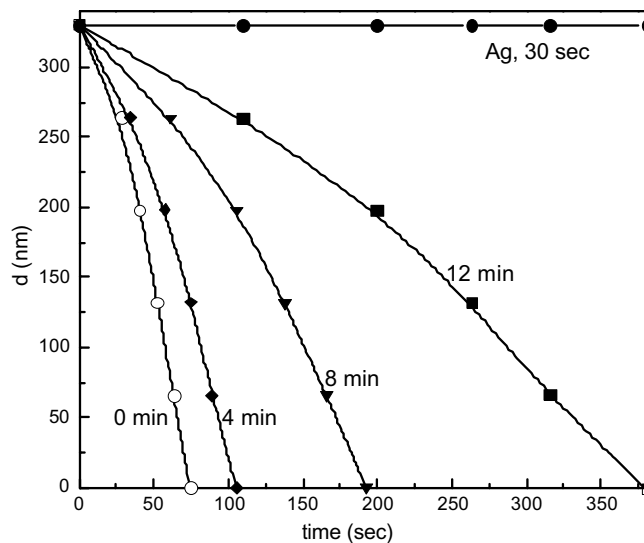


Fig. 2. The variation of thickness of an arsenic sulfide glass film as a function of time of etching in trimethyl amine solvent. The time on plots indicates exposure to halogen lamp white light. The flat line represents data for a sample with silver film on top; the sample was exposed to light as the other samples, and remaining metallic silver was removed by dissolving in  $\text{HNO}_3$  before conducting the etching experiment.

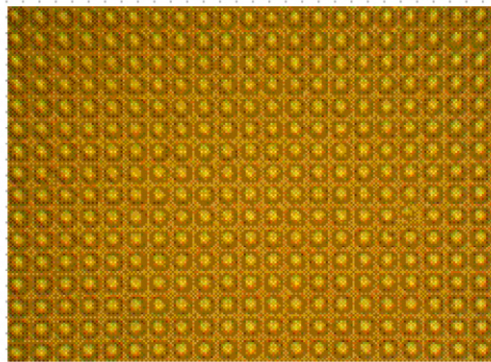


Fig. 3. A 12- $\mu\text{m}$  microlens array fabricated in a thin  $\text{As}_{35}\text{S}_{65}$  film. The focusing action of light by the lenses is clearly seen.

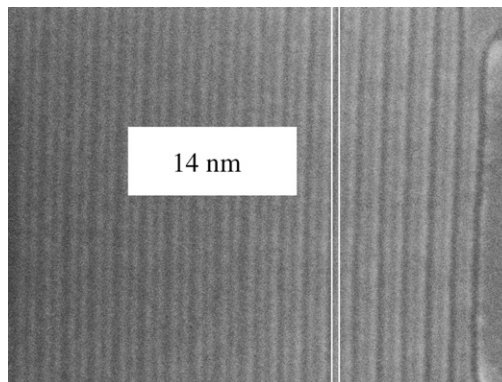


Fig. 4. Parallel lines fabricated in arsenic sulfide film by exposure to electron beam (30 keV, 40 pA, line dose: 1–10 nC/cm) followed by etching in an amine based solution.



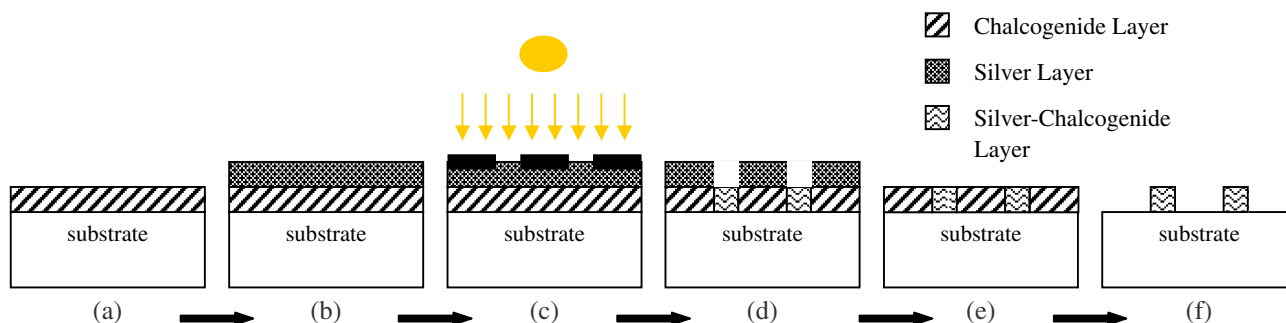


Fig. 5. Steps of lithographic process based on radiation induced silver diffusion in chalcogenide glass: (a) Deposition of chalcogenide layer, (b) Deposition of silver layer, (c) Exposure through mask, (d) Radiation induced silver diffusion completed, (e) Removal of remaining silver by sputtering or dissolving in acid, (f) Removal of ChG regions by etching to leave selected regions of the photoresist.

of the curves decreases as the exposure of the films to band-gap light increases. As an illustration of the application of the photoresist behavior of chalcogenide glass, Fig. 3 shows an array of microlenses that are fabricated in As<sub>35</sub>S<sub>65</sub> film based on this principle of photolithography.

Nishihara et al. [26] showed that similar to phodarkening the optical properties of chalcogenide films are altered by exposure to electron beam. Although there is much less understanding of the electron beam-induced changes in the structure of chalcogenide glasses, these parallel observations led to the exploration of chalcogenide glass also as an EB resist [27]. Schröter et al. [22] have extended this process for fabricating arrays of micro-optic elements 100 nm in diameter, 410 nm in height and 300 nm periodicity. In our laboratory we have fabricated structures with a higher resolution, see Fig. 4. The parallel lines in the figure are separated by 14 nm indicating one of the finest structures fabricated in a glass.<sup>2</sup> The measurement of etched depth by atomic force microscopy shows a linear relationship with the electron dose. Thus by controlling the electron dose with a well programmed beam, one can readily make gray masks or nano-architectures with predetermined variation of surface topography. The mechanism of EB lithography is probably similar to that described above for photolithography, but we have observed significant differences in the X-ray photoelectron spectra of the light and electron beam irradiated samples.

### 3.2.1. Resists based on radiation induced metal diffusion in chalcogenide glass

Several chalcogenide glasses exhibit the unique property of photo-diffusion, such that on exposure to bandgap light certain metals (e.g. silver, zinc, copper and indium) diffuse rapidly from the surface into the bulk [28]. The basic premise of the radiation induced diffusion, which is highest for silver, is that the radiation creates electrons and holes. In chalcogenides the holes have higher mobility than the electrons, and so the latter diffuse away preferentially from

the irradiated region. A negatively charged space is created in the illuminated region, which attracts metal ions from the surface and strongly enhances their diffusion. Thus the chemical composition as well as the structure of irradiated region is dramatically modified compared to the unirradiated region. As a result, the selectivity or contrast of the resist is greatly enhanced [29]. One expects that any type of irradiation that produces electron–hole pair can be used for activating the chalcogenide resist. Indeed bandgap light, X-rays, electron beam, ion beam, etc. [23,39] have been shown to be viable options for lithography with such resists. Fig. 5 shows major steps of lithography that exploits radiation induced silver diffusion. Although in this method of lithography, additional steps of silver deposition and removal of undiffused metallic silver are needed, the significantly enhanced selectivity and sharpness of the etched patterns have stimulated extensive research on this process [30].

The diffusion of silver in chalcogenide glass films can occur spontaneously and thus reducing the accuracy of lithography. This difficulty, if encountered, can be circumvented by replacing the film of metallic silver with a film of silver salt such as Ag<sub>2</sub>S or Ag<sub>2</sub>Se [31]. Annealing of the chalcogenide film before depositing silver layer also appears to keep sharp silver concentration profile [32]. Another likely problem with silver diffused chalcogenide resists is lateral diffusion of silver between the exposed and unexposed regions [33,34]. Its adverse impact can be reduced to some extent by the proper choice of chalcogenide composition, deposition conditions that control the nanoscale structure of the films, and writing fine lines prior to large patterns. Wagner et al. [35] have pointed out that in the Ag–As–S ternary system, glass formation is centered around A<sub>35</sub>S<sub>65</sub> and AgAsS<sub>2</sub> compositions. Therefore, inasmuch as glassy state remains stable, silver diffusion in A<sub>35</sub>S<sub>65</sub> promotes the formation of glass close to the AgAsS<sub>2</sub> composition, rather than a range of composition between them. Therefore, a sharp boundary is found between the silver diffused and undiffused regions.

Finally, it is useful to note that as lithography is shifting away from wet etching toward a completely dry operation, the chalcogenide glass resists appear to respond well to dry plasma etching [36–38]. For Ge–Se films, dry etching by

<sup>2</sup> Since the submission of this manuscript, we have fabricated gratings separated by 7 nm.

SF<sub>6</sub> plasma gives the best contrast and sensitivity. The selectivity is good enough that 100 nm thick film would be sufficient for lithography [39].

#### 4. Summary

The widely used polymer resists are approaching the limit of their resolution for fabricating nanoscale features in the emerging nanotechnology. Inorganic resists are expected to fill the need of resists for high resolution nano-lithography. In particular, silica and chalcogenide inorganic glasses, which can be fabricated in thin film form by common preparation methods, may offer additional processing advantages over the currently used polymer resists. Usefulness of thermally grown silica as a resist has been established for fabricating nanodevices. By comparison, chalcogenide glasses have much higher selectivity and sensitivity but their compatibility with the existing device manufacturing processes remains to be validated.

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