I. INTRODUCTION

Microlithography techniques used in the fabrication of photonic devices traditionally use rigid substrates such as Si wafers. However, there exist specific applications for devices such as infrared (IR) detector arrays and IR frequency selective surfaces (FSS) which require flexible substrates, so that the final structure is able to be conformally contacted to a nonplanar surface. The conformal substrate considered in this study was the HD Microsystems liquid polyimide.

FSS elements appropriate for the IR (Ref. 1) typically have critical dimensions between 0.2 and 2 μm, with excellent spatial uniformity of the final periodic structure required. The FSS shown in Fig. 1 consists of three layers on top of the underlying structural substrate, a continuous metallic ground plane, a continuous dielectric standoff layer, and the periodic microstructural FSS elements. The thickness of the standoff layer is typically λ/4n, where λ is the center of the design wavelength band of operation and n is the refractive index of the standoff layer, which yields thicknesses in the range of a few micrometers. The ground plane and FSS elements are fabricated from metallic films of typical thickness of 0.1–0.2 μm. The Jerusalem cross designs were used in this study as the unit cell geometry for the FSS (compared to the square loop designs of Ref. 1) because of the presence of a second harmonic resonance that was useful in applications such as frequency selective surface filters.2

II. MATERIAL SELECTION AND PROPERTIES

When fabricating on a flexible substrate, it is desirable that the tensile moduli of the substrate and standoff layer be similar, which indicates that polymers are considered as candidate materials. For example, HD Microsystems liquid polyimide and benzocyclobutene (a candidate polymer standoff layer) have moduli of 2.7 and 2.9 GPa, respectively. Standoff materials used previously1 had tensile moduli ranging from 80 GPa for a-Si to 223 GPa for zirconia. However, choice of the standoff layer material is constrained by two considerations that tend to exclude most polymers. To maintain proper functionality of the FSS, it is desirable to minimize absorption losses in the standoff layer. It is also difficult to fabricate many common polymers with the quarter-wave thickness required. Thus, the polymer chosen for the standoff layer must have high IR transparency as being capable of being spun or rolled out to the desired thickness with good uniformity.

The high resolution required in the fabrication of the FSS elements places constraints on the flatness and surface roughness of the combined underlying structure of the substrate, ground plane, and standoff layer. Typical flexible substrates such as polyimide sheets are both locally rough and difficult to secure in a sufficiently flat configuration. Previous research3,4 has addressed this issue by using liquid polyimide, which gives the user control over both the surface roughness and flatness of the polyimide substrate. However, the lithographic-resolution limit seen in these references was on the order of tens of micrometers, and relatively small areas of the substrate were used for the fabrication.

In this study, a full 10 cm (4 in.) diameter substrate was populated with FSS elements. The combination of large write area and small element size required that the substrate had less than 10 nm rms surface roughness, with a high degree of planarization.

To investigate the optical properties of the candidate polymers, a J.A. Woollam IR ellipsometer was used. The ellipsometer uses polarization data such as the amplitudes of the electrical field components and their relative phase shifts to determine the complex index of refraction by fitting to an appropriate model. To facilitate fabrication, three resists and spin-on dielectrics [ZEP, polydimethylglutarmide (PMGI), and benzocyclobutene (BCB)] were investigated to find standoff layer materials that met the optical and mechanical requirements. Zeon Chemicals ZEP 520A7 is a positive tone electron-beam resist that has favorable optical properties in
the mid-IR, as shown in Fig. 2. ZEP can be difficult to fabricate on because of its poor adhesion to most metals. An adhesion promoter such as hexamethyldisilazane (HMDS) can improve adhesion to some oxides, but fabricating small metallic elements onto ZEP remains a challenge.

A complete fabrication process was attempted with polydimethylglutarimide (PMGI). MicroChem PMGI SF7 was used. PMGI is from the same family of polymers as polymethylmethacrylate (PMMA) and shares many of the same optical and chemical properties. As seen in Fig. 2, the loss associated with PMGI in the IR is significant. In two passes of 10.6 μm radiation through a quarter-wave standoff layer, 44% of the radiation is absorbed. This leads to undesirable increases in emissivity beyond 10 μm in PMGI.

PMGI and PMMA are both cured at 180 °C, which is a lower cure temperature than the spin-on dielectrics require. PMGI is preferable over PMMA because it is not removed by solvents commonly used in postexposure lithographic processing such as acetone or xylene. PMGI also has this advantage over ZEP which is attacked by methylene chloride and acetone over time.5

III. FABRICATION TESTS FOR FEASIBILITY

Using the PMGI standoff layer, problems were encountered in the lift-off of the excess metal between structures, after the FSS pattern had been exposed in photoresist and metallized with Ti. Figure 3 shows the boundary between successfully lifted off structures, nonlifted off structures, and the region where the metal elements lost adhesion to the PMGI. The entire wafer was exposed, developed, and metallized. The lift-off was stopped before completion because the individual FSS elements were disassociating from the wafer in the solvent. The elements did not appear to lift-off immediately. Indeed, the small region of normally lifted off elements shows that there was some time between proper lift-off and disassociation of the elements, which unfortunately was not spatially uniform. The disassociated elements left behind ghost images that were measured via profilometry to be 20 nm deep.

The disassociation of the antenna elements and the appearance of ghost images appeared consistently over six separate trials without significant deviation. We suspect that this phenomenon was caused by the oxidation reaction between the polymer and the Ti elements. Normally, an oxidation reaction between a metal and a polymer is favorable and provides an adhesion mechanism. For polymers from the PMMA family, the new oxide species that forms after the reaction is not chemically bonded to the remainder of the polymer. During the reaction, thermal energy is transferred from the metal elements to the PMGI beneath causing an amorphization of the polymer carbon structure.6 This re-

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**Fig. 1.** FSS structural schematic with (a) cross section and (b) top view.

**Fig. 2.** Mid-IR optical properties of polymers.

**Fig. 3.** PMGI lift-off results: bottom, ghost images left behind after disassociation of elements; middle, properly lifted off elements; top middle, elements before lift-off.
Au is preferred for this reason, Ti may be used with appro-
but it is also desirable in the FSS design to have the highest
150 nm thick metal film is sufficient to prevent transmission,
50 nm and the skin depth of Au is about 12 nm. Either
layer between the Au and the BCB, but this was unsuccessful
in an inert environment. The cure temperature was not an
issue for liquid polyimide films, which were not observed to
deplanarize under these conditions.

In initial IR FSS fabrications, an optically thick (150 nm)
Au film was used as the ground plane, which created a prob-
lem with the use of a polymer standoff layer because of the
lack of adhesion between Au and BCB. During fabrication
trials, it appeared that the metal elements were dissociating
from the BCB, but ellipsometric measurements proved that
the BCB layer beneath the antenna elements was no longer
present following the lift-off procedure. It was concluded
that the BCB had insufficient adhesion with the Au ground
plane. A solution was attempted by evaporating a Ti seed
layer between the Au and the BCB, but this was unsuccessful
because the Ti did not adhere well to the Au.

Au adheres well to Ti because the surface of the Ti film is
rough and provides a means for the polycrystalline Au to
form a diffusive bond with the Ti. The Au film is smooth and
does not provide an opportunity for the Ti film to form a
strong bond. BCB forms a strong bond with Ti via oxidation.
For this reason, subsequent FSS designs used an all-Ti
ground plane. Figure 4 shows the spectral conductivity for
Au and Ti as well as the spectral skin depth for both metals.
At a wavelength of 10 μm, the skin depth of Ti is about
50 nm and the skin depth of Au is about 12 nm. Either
150 nm thick metal film is sufficient to prevent transmission,
but it is also desirable in the FSS design to have the highest
possible spectral conductivity in the mid-IR range. Although
Au is preferred for this reason, Ti may be used with appro-

![Spectral conductivity and skin depth derived from optical constants measured with IR-VASE ellipsometer.](image)

**IV. FABRICATION RESULTS**

Three separate masks were designed consisting of a
10 cm (4 in.) square fully populated with FSS elements. Two
different types of elements were used depending on the de-
sign. Two designs used Jerusalem cross elements as shown in
Fig. 1, one with a 12.7 μm periodicity and a 1.7 μm critical
dimension and the other with a 7 μm periodicity and a
1.3 μm critical dimension. The third type of element fabri-
cated was a square-loop design with a 10 μm periodicity
and a 1.2 μm critical dimension.

Fabrication of the FSS periodic structures used HD Mi-
crosystems liquid polyimide, which served as a structural
substrate beneath the ground plane shown in Fig. 1. Liquid
polyimide could be spun on and cured by the user. This
allowed for control over surface roughness. HD Microsys-
tems liquid polyimide was spun onto a prime-grade Si wafer
at 2000 rpm. Each layer was 13 μm thick and three layers
were spun for a total substrate thickness of about 40 μm.
The polyimide film was soft cured at 150 °C between each
layer, and the final three-layer film was hard cured at 300 °C
in an inert environment. The surface roughness was mea-
sured to be 4 nm rms with Dektak profilometry.

The remaining process steps were carried out as they
would have been if the substrate was simply a Si wafer. The
150 nm thick Ti ground plane was deposited onto the liquid
polyimide surface using an electron-beam evaporation sys-
tem. Next, BCB was spun onto the Ti at a speed and viscos-
ity combination appropriate to the desired thickness. Undi-
luted BCB was spun at 1500 rpm to achieve a thickness of
1.8 μm. Charts for BCB showing the film thickness as a
function of spin speed for given viscosities may be obtained
from the Dow Corporation. BCB also uses an adhesion pro-
moter (Dow AP3000) that goes on before the polymer film.
The BCB film was soft baked at 120 °C and hard cured in an
inert environment at 250 °C.
Electron-beam lithography was used to write the elements. This was done to ease fabrication issues associated with the resolution of the available photolithography systems. ZEP 520A7 was used as the electron-beam resist and cured for 3 min at 180 °C. The liquid polyimide substrate led to nearly complete uniformity and well resolved elements across the populated region.

The final fabrication steps were to deposit a metal film onto the patterned resist. Depending on the FSS design, 100 nm of either Ti or Au was used for the antenna elements. A 10 nm thick Ti adhesion layer was used beneath the Au film. Both metals were deposited via electron-beam evaporation at 5.0 × 10⁻⁶ Torr and 50 °C. ZEP was removed by methylene chloride. Although lift-off was much faster with ZEP compared to standard photoresists, the wafer had to be quickly rinsed and reimmersed in solvent to prevent metal contaminants from settling on the surface.

Once the lithography processing was completed, the liquid polyimide could be peeled off the Si wafer. One way to peel the liquid polyimide off the substrate was to place a piece of weak adhesive tape across the surface and then pull the tape off. This did not remove the FSS structures and several repetitions would cause the polyimide to begin to separate from the substrate. Another method to remove the liquid polyimide from the Si wafer is to break the wafer and carefully peel the polyimide off as the two-halfs of the wafer are pulled apart. Figure 5 shows the FSS after removal from the Si wafer. The metallized FSS elements are shown to be intact after removal in part (b) of Fig. 5.

The substrate flatness of the sample was measured using a Newton interferometer while the samples were still fixed to Si wafers. A transparent optical flat was placed in contact with the sample and a fringe pattern was observed using a mercury lamp. The lamp was filtered so that only the green line with λ=546 nm was transmitted. Flatness was measured by counting the number of circular fringes per unit length. The liquid polyimide film on Si showed 0.06λ/mm in the center of the wafer and 0.08λ/mm toward the edges of the wafer. Deviations from flatness in the polyimide film were radially symmetric. This was likely a result of the spin-on application of the film. For comparison, a polyimide sheet was glued to a substrate and measured in the same manner. The polyimide sheet showed deviations in flatness ranging from 1.25 to 3.75λ/mm.

The liquid polyimide FSS substrates were easily able to conform around a cylindrical axis, and there did not appear to be any damage to the FSS elements as the substrate was plastically deformed. The substrates are reasonably easy to handle but can be torn by a shearing force. They can handle a moderate degree of stretching along the plane of the structures. The liquid polyimide membranes may be used at temperatures up to 300 °C. The fabrication techniques demonstrated in this article open the door for the fabrication of other structures such as IR detectors that may need to employ lithography on a similar element size.

V. SUMMARY

Three different periodic FSS structures were fabricated on liquid polyimide membranes. The periodic structures had periodicities from 1.2 to 1.7 μm and fully populated a 10 cm (4 in.) diameter wafer. The FSS showed excellent uniformity across the wafer. The success of the fabrication suggests that the degree of nonflatness and surface roughness in these films was acceptable for 1–2 μm critical dimension lithography processes. The liquid polyimide structures had deviations in flatness from 0.06 to 0.08λ/mm and 4 nm rms surface roughness. BCB was used as an intermediate layer beneath the periodic structures. It was found to have sufficiently low absorption in the mid-IR as well as favorable adhesion properties for FSS fabrication. Two other polymers were found to have unfavorable adhesion properties. Au and Ti were used for the periodic structures as well as for the ground plane beneath the intermediate layer. The conductivities of Au and Ti at IR frequencies were measured, and Ti was found to be less conductive (by nearly an order of magnitude) than Au. The fabrication techniques described here would be suitable for the fabrication of many different types of periodic microstructures on flexible substrates.

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