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Abstract. Recently published reports in the literature for bilayer lift-off processes have described recipes for the patterning of metals that have recommended metal-ion-free developers, which do etch aluminum. We report the first measurement of the dissolution rate of a commercial lift-off resist (LOR) in a sodium-based buffered commercial developer that does not etch aluminum. We describe a reliable lift-off recipe that is safe for multiple process steps in patterning thin (<100 nm) and thick aluminum devices with micron-feature sizes. Our patterning recipe consists of an acid cleaning of the substrate, the bilayer (positive photoresist/LOR) deposition and development, the sputtering of the aluminum film along with a palladium capping layer and finally, the lift-off of the metal film by immersion in the LOR solvent. The insertion into the recipe of postexposure and sequential develop-bake-develop process steps are necessary for an acceptable undercut. Our recipe also eliminates any need for accompanying sonication during lift-off that could lead to delamination of the metal pattern from the substrate. Fine patterns were achieved for both 100-nm-thick granular aluminum/palladium bilayer bolometers and 500-nm-thick aluminum gratings with 6-μm lines and 4-μm spaces. [DOI: 10.1117/1.JMM.14.1.014501]

Keywords: lift-off resist; granular aluminum bolometer; two-step development method; undercut.

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1 Introduction

One common method for producing metal patterns on semiconductors is bilayer lift-off.[1–10] One first coats the substrate with a nonphotosensitive material [the lift-off resist (LOR)] that is thicker than the desired metal thickness, followed by a standard i-line positive photoresist. No intermixing occurs. After exposure, the photoresist and LOR are developed separately. Since we are interested in patterning sputtered aluminum thin films, unlike recent publications describing a bilayer lift-off procedure[9,10] with metal-ion-free (MIF) developers, we have investigated the use of a sodium-based buffered developer that does not etch aluminum. The use of the latter developer is crucial for the successful patterning of thin (100 nm or less) granular aluminum (with a palladium capping layer) bolometers during the multiple-patterning process that subjects the patterned active region of the device to further immersions in the developer during the patterning of the accompanying contacts. The same may be said for any thin-film aluminum patterning process in which the aluminum film may be subjected to multiple immersions in the developer. Once the exposed photoresist has been developed, the remaining unexposed photoresist is subsequently soft baked a second time, but at a higher temperature than the first soft bake performed after spinning, in order to decrease its develop rate relative to the underlying LOR layer. A second and final develop step then dissolves away the LOR in the open areas. During this step, the development of the LOR is typically isotropic, and a desired undercut emerges beneath the overlying photoresist pattern. The resulting LOR aspect ratio, i.e., AR = lateral depth/vertical thickness, of the undercut can be chosen so that an opening always remains between the undercut and the sputtered metal, thereby allowing the LOR solvent to wash away the bilayer. We have measured the aspect ratios of the undercut for two different develop times and have demonstrated that either is sufficient for a clean lift-off with our sputtering approach (as compared with e-beam evaporation) that often uses large targets (e.g., 20 cm in diameter) with correspondingly larger solid angles subtended by the substrate. Additional benefits include the fact that the LOR is able to withstand significantly higher temperatures than the photoresist before hardening, so it is particularly suitable for e-beam evaporation.

2 Process Flow for Silicon Substrates

We describe a detailed recipe for bilayer lift-off of sputtered aluminum/palladium bilayer films on (100) silicon, although the recipe should also work for any substrate to which LOR adheres such as NiFe, GaAs, InP, and many other III–V materials. We use LOR-10B (MicroChem Corp., Upper Newton Falls, Massachusetts) and AZ5214E (MicroChemicals[11]) photoresists in a positive-tone mode with prediluted 1:1 AZ Developer (MicroChemicals). Both the Microposit Developer (Rohm and Haas Electronic Materials, Marlborough, Massachusetts) and the AZ Developer contain sodium silicates and phosphates and do not etch aluminum. On the other hand, tetramethyl ammonium hydroxide (TMAH)-developer (MIF) is usually recommended[9,10] for use with LOR, but the TMAH-MIF-developer etches aluminum at a rate of approximately 50 to 100 nm/min.[11] Our resulting process flow is listed in Table 1. All samples [12 mm × 16 mm metal 0.5 (100) silicon platelets] are initially acid-cleaned. We only do Steps 1 and 2 once, prior to the first patterning. For any additional patterning, Steps 1 and 2 would be skipped.

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Table 1 Process flow.

Process steps	Comments
1. Piranha clean: Heat 200 mL 96% sulfuric acid to 80°C, pour 50 ml 30% hydrogen peroxide into the acid. Mix thoroughly—temperature increases to 130°C	Immerse samples for 10 min. Rinse in deionized water for 5 min
2. HF dip: Add 20 mL 49% HF to 200 mL deionized water	Immerse samples for 3 min. Rinse in deionized water for 5 min. Blow dry with N_2
3. Solvent clean: spin for 30 s at 3000 rpm (for subsequent patterning only)	Spray 10 s with acetone, followed by 10 s isopropanol
4. Solvent hot-plate bake	200°C, 5 min
5. Spin LOR-10B: 15 s at 0 rpm 45 s at 4000 rpm	Dispense LOR-10B Distribute LOR-10B (thickness 1.0 μm)
6. LOR-10B hot-plate bake	180°C, 3 min
7. Spin AZ5214: 15 s at 0 rpm 50 s at 5000 rpm	Dispense AZ5214 photoresist Distribute AZ5214 (thickness 1.25 μm)
8. AZ hot-plate bake	100°C, 2 min
9. Expose, soft-contact	i-line, 365 nm, 75 mJ
10. First develop	1:1 AZ Developer, 60 s, 64°C
11. Hot-plate bake	120°C, 5 min (harden PR)
12. Second develop, 3½ to 4½ min, 390 nm/min LOR dissolution rate	1:1 AZ Developer, AR = 1.3 to 1.5 for 210 to 270 s, respectively
13. Sputter aluminum/palladium bilayer, or other metal	Aluminum thickness < LOR-10*B*, with 5 nm palladium capping layer
14. Lift-off in PG Remover	80°C, 5 to 10 min followed by IPA spray Rinse deionized water, blow dry (do not use acetone)

3 Results

Following the recommendation described by Liang et al.,[7] we also found it necessary to incorporate additional Steps 10 and 11 (see Table 1) into the process flow in order to result in a bilayer with a sufficient undercut. The additional steps include a preliminary develop to remove the exposed photoresist, followed by a 5 min, 120°C hot-plate bake to further harden the remaining photoresist. Initial tests without incorporating Steps 10 and 11 from Table 1 failed to result in any noticeable undercut, even with a final develop time of 5 min, and resulting lift-off attempts were problematic. The Step 11 120°C bake reduces the etch rate of the photoresist sufficiently for the undercut to develop in Step 12. The lateral (approximately isotropic) dissolution rate of the LOR-10B in AZ Developer, for the LOR baked at 180°C for 3 min on a hot plate, was measured to be 390 nm/min. To our knowledge, this is the first reported data in the literature for the dissolution rate of LOR-10B in AZ Developer.

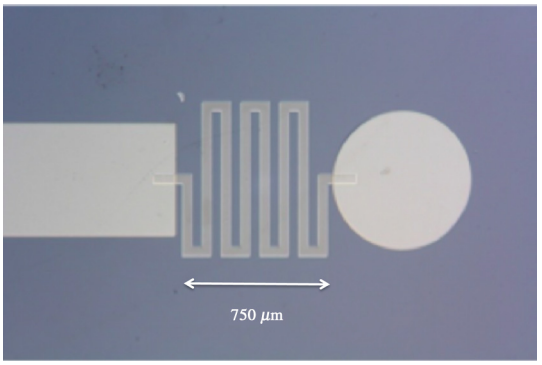
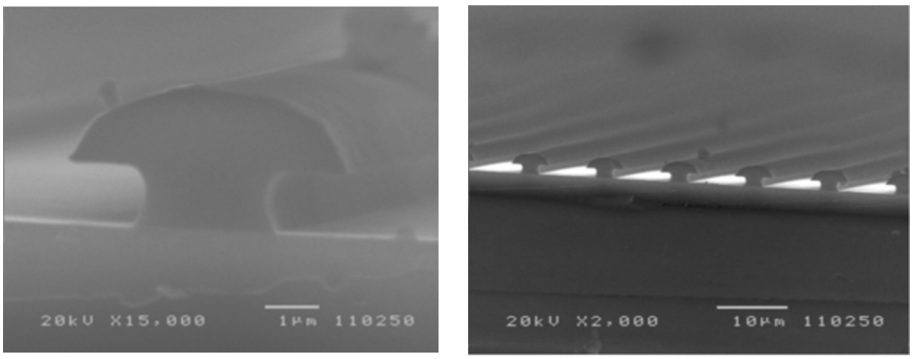


Fig. 1 Granular aluminum/palladium 100-nm-thick bilayer microbolometer (serpentine) with separately patterned 300-nm-thick aluminum contact pads on a (100) silicon substrate. The line width of the serpentine is 50 μm. The portion of the thin bolometer serpentine, seen extending beneath the contact pads, would be destroyed with a recipe using tetramethyl ammonium hydroxide (TMAH)-based developers.

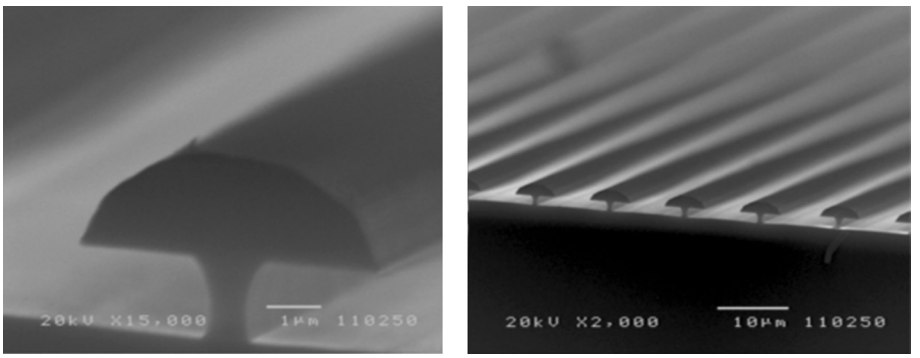
We use our process to reliably fabricate sputtered superconducting granular aluminum/palladium bilayer microbolometers.[12] Our prior approach,[12] which used single-layer image-reversal photolithography, was neither as robust nor reliable as the one described herein. Figure 1 is an optical micrograph of a sputtered patterned granular aluminum/palladium bilayer microbolometer using the current process. The active region of the bolometer, used as a fast and sensitive detector of acoustic phonons,[13] is a serpentine 100-nm-thick granular aluminum film (dc-magnetron sputtered aluminum, in partial pressures of 0.8 mT oxygen and 8.5 mT argon and flow rate of 30 sccm, to result in a superconducting transition temperature of 1.8 K), followed by an *in situ* sputtered 5-nm-thick palladium film (the palladium-capping layer prevents the subsequent formation of an oxide layer on the aluminum). Also seen are the separately patterned, larger electrical contact pads; they are 300-nm-thick sputtered aluminum (with argon only, for a resulting 1.2 K superconducting transition temperature) followed by the second *in situ* sputtering of a 5-nm-thick palladium film. It is important to note that the end portions of the thin bolometer serpentine, seen in Fig. 1 extending beneath the contact pads, would be destroyed with a recipe using a TMAH-based developer.

Other published methods[14] of granular aluminum bolometer fabrication have used single-layer lift-off processing with the less-isotropic (as compared with sputtering) e-beam evaporation. In order to pattern the electrical contact pads, these methods required the use of a contact mask to be carefully rotated (with custom solenoid-actuators mounted within the chamber) into position upon the freshly evaporated granular aluminum, without breaking vacuum. An exposure of the aluminum to air prior to fabricating electrical contacts using a contact mask would result in an insulating and intervening oxide layer. Our aluminum (with the palladium capping layer) metallization using the bilayer lift-off recipe



(a) (b)

Fig. 2 (a) SEM micrograph of a portion of a linear grating, prior to sputtering, resulting from the process flow given Table 1 with a step 12 develop time of 3-1/2 min. Aspect ratio = 1.3. (b) SEM micrograph at lower resolutions showing multiple periods of the same 10-μm period grating pattern.



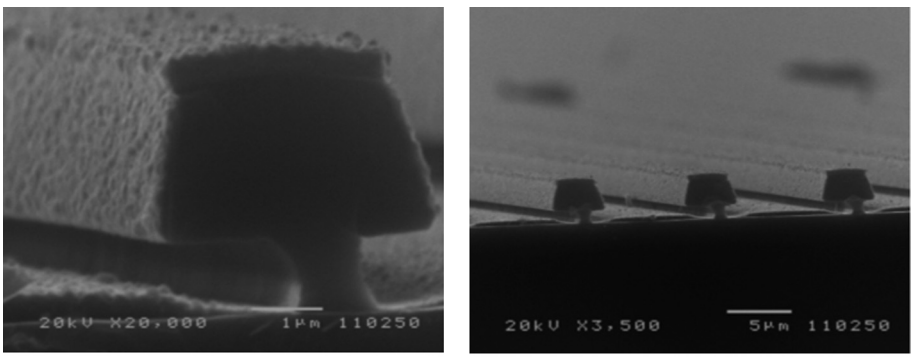
(a) (b)

Fig. 3 (a) SEM micrograph of a portion of a linear grating, prior to sputtering, resulting from the process flow given in Table 1 with a step 12 develop time of 4-1/2 min. Aspect ratio = 1.5. (b) SEM micrograph at lower resolution showing multiple periods of the same 10-μm period grating pattern.

should work for e-beam evaporation as well, with the added advantage that the higher temperatures (compared with sputtering) should not affect the LOR-10B significantly as its glass temperature is 190°C.

Figures 2 and 3 show scanning electron the scanning electron micrographs (SEM) micrographs of our results, using the process outlined in Table 1, in patterning a 10-μm-period linear grating (60% fill factor), just prior to sputtering (Step 13). The patterns shown in Figs. 2 and 3 have used development times of 3-1/2 and 4-1/2 min, respectively, resulting in undercuts with aspect ratios of 1.3 and 1.5, respectively.

Shown in Figs. 4 and 5, respectively, are SEM micrographs of a sputtered 500-nm-thick aluminum 10-μm-period linear grating after lift-off processing. The granularity of the sputtered aluminum can be clearly seen in both micrographs. We find that our process flow results in convenient and reproducible lift-off of sputtered aluminum (and other



(a) (b)

Fig. 4 (a) SEM micrographs of 500-nm-thick aluminum sputtered linear 10-μm period grating before lift-off processing. (b) SEM micrograph at lower resolution.

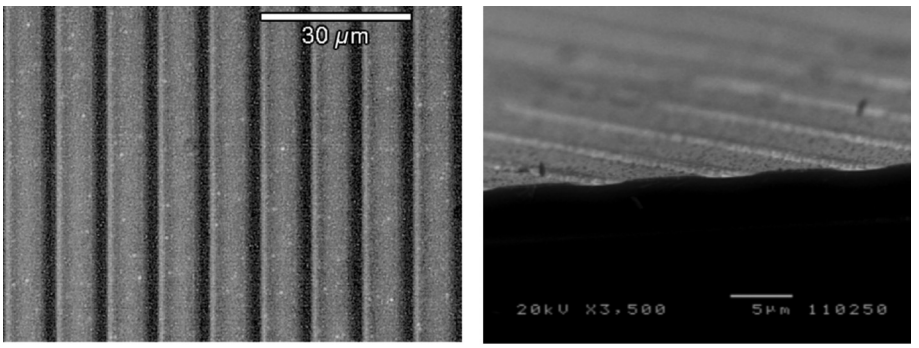


Fig. 5 (a) Optical micrograph and (b) SEM micrograph after lift-off of a 500-nm-thick aluminum linear grating with a 10-μm period.

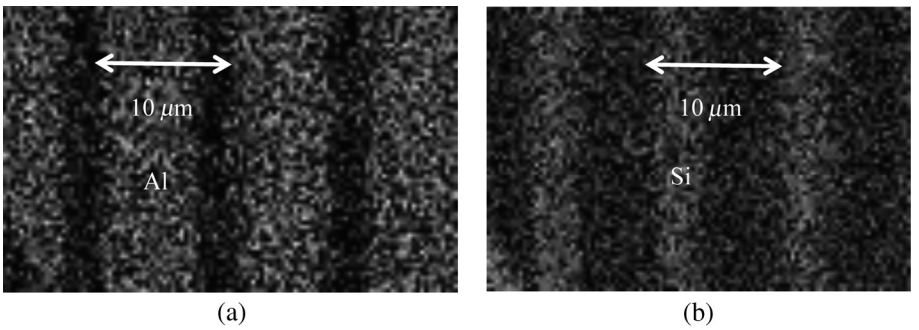


Fig. 6 (a) X-ray EDS image of aluminum 10-μm period linear grating pattern. (b) X-ray EDS image of silicon substrate (bright) for same 10-μm period grating pattern.

metals) for thicknesses less than the thickness of the LOR-10B. It should be noted that no sonication has been used for lift-off processing, which is particularly important for substrates with poor adhesion characteristics compared with silicon.

Energy-dispersive x-ray spectroscopy (EDX or EDS)[15,16] has been performed in order to correlate the elemental compositions of the samples with the topological/atomic mass contrast seen in the images shown in Fig. 5. Shown in Fig. 6 is the x-ray EDS micrograph images of the same part of the aluminum grating pattern shown in Fig. 5. The x-ray EDS image shown in Fig. 6(a) represents the map of x-rays with energies in the range 1410 to 1570 eV, i.e., for an aluminum map. The x-ray EDS image shown in Fig. 6(b) is the map of x-rays with energies of 1660 to 1820 eV range, i.e., for the silicon map.[15,16] The presence of the wider aluminum lines and smaller openings (silicon) for the 60% fill factor 10-μm period grating is clearly observed.

4 Conclusions

A straightforward bilayer lift-off recipe has been characterized, which uses a commercial sodium-based buffered developer that is safe for aluminum patterning in processes that may require multiple immersions in the developer. The incorporation of a sequence of the postexposure develop-bake-develop steps into the process was crucial for obtaining an undercut. Undercuts with aspect ratios of 1.3 and 1.5 have been demonstrated. The lateral dissolution rate of 390 nm/min for LOR-10B in AZ Developer has also been measured and, to the author's knowledge, has not been previously reported in the literature. The recipe is very useful for the reliable and convenient fabrication of thin (100 nm) granular aluminum/palladium bilayer bolometers, as well as thick (500 nm) sputtered aluminum gratings with feature sizes of the order of microns. The bilayer lift-off approach was found to be necessary for the patterning of sputtered metal films due to the large solid angle of our large aluminum target (20 cm in diameter) subtended by the sample. Finally, we note that the lift-off in the LOR solvent requires no sonication that otherwise might delaminate metallic patterns from the substrate.

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