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Wafer-Scale Near-Perfect Ordered Porous Alumina on Substrates by Step and Flash Imprint Lithography

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ABSTRACT Nanoporous anodic aluminum oxide (AAO) has been widely used for the development of various functional nanostructures. So far, highly ordered AAO on substrates could only be prepared using a nanoindentation method *via* hard stamping and lithographic techniques that are not scalable to a wafer-scale. Here we report on a step and flash imprint lithography (SFIL)-based method to fabricate a near-perfect ordered AAO with square and hexagonal lattice configuration on silicon substrate over 4 in. wafer areas. SFIL was used to prepattern a polymer mask layer, and wet-etching process was employed to transfer the nanopatterns to aluminum (Al) films, thus creating ordered nanoindentation on the Al surface. The ordered nanoindentation guides the growth of nanochannels in the anodization step to create the ordered nanoporous structures. The proposed wafer-scale process is compatible with standard semiconductor fabrication and offers substantial advantages over conventional Al patterning methods in terms of patterning areas, throughput, process simplicity, and process robustness, allowing up to 10 000 imprints or pattern transfer to the Al films.

KEYWORDS: anodic aluminum oxide · step and flash imprint lithography · wafer scale

Anodic aluminum oxide (AAO), a self-ordered nanochannel material formed by the electrochemical oxidation of aluminum, has been extensively explored due to its versatile utilization as a template material for synthesis of multifunctional nanostructures. A variety of nanostructured metals, metal oxides, polymers, and semiconductors have been fabricated using anodized alumina, and their commercial values can be seen in the widespread use of various electronic, optoelectronic, magnetic, and energy storage devices.[1−3] These functional devices are generally constructed by integrating active components, such as nanowires or nanotubes, into device structures on rigid or flexible substrates in a well-controlled way. Silicon-based light-emitter,[4] high-performance multijunction solar cells and an advanced UV sensor system for detecting multiple gas species[5] have been developed by selective electrodeposition of the desired semiconductor material inside the AAO template pores from an appropriate chemical solution. Other structures and devices have also been fabricated on technology-relevant substrates,[6−11] yet all share one core problem that is still irresolvable to date: the lack of a simple fabrication process suited to economical volume production for producing perfectly ordered alumina nanopores on substrates over a large area.

The perfect geometry of AAO can be well-characterized as a honeycomb structure containing a closely packed high aspect ratio of nanochannels. However, the AAO pore arrays obtained are usually far from that of the idealized model, and the self-ordering phenomenon occurs only in narrow process windows, with a defect-free domain size limited to several micrometers.[12] Attempts to increase the size of self-ordered domains have been made by prepatterning the aluminum (Al) surface prior to the anodization process and using the patterns as the nucleation sites to guide the growth of the nanochannels. As summarized in Table 1, approaches to texture the Al surface include the use of a two-step anodization process,[13] hard stamping,[14−17] direct focused ion beam (FIB) lithography,[18] holographic lithography,[19] resist-assisted FIB lithography,[20] colloidal lithography,[21] block copolymer self-assembly,[22] soft imprinting,[23] and nanoimprint lithography.[24]

Most of the methods above, however, are generally developed to prepare highly ordered AAO on bulk Al foils. Up to now, the fabrication of highly ordered AAO on substrates (such as silicon or glass) is not very well developed and still remains a major challenge from the science and technology point of view. Owing to the small grain sizes and randomly distributed, densely spaced grain

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Received for review January 28, 2010 and accepted April 18, 2010.

Published online April 22, 2010.
10.1021/nn1001744

TABLE 1. Comparison of Various Methods To Create Nanoindentations on the Surface of Al[a]

Samples Types	Al Pre-Patterning Methods	Ordered Pattern Area	Remarks
Bulk Al	Two-steps anodization[13]	Not available	This method requires the use of a thick Al film (>40 μm)
	Hard stamping using SiC stamp[14]	3 mm x 3 mm	SiC stamp was fabricated using electron beam lithography. Stamping pressure: 28 kN/cm^2.
	Hard stamping using Ni stamp[15]	Wafer scale	Ni stamp can be mass-replicated from master pattern, fabricated using laser interference lithography. Stamping pressure: 25 kN/cm^2.
	Hard stamping using optical diffraction grating[16]	5 mm x 5 mm	Close packed arrays of rhomb-shaped ridges were formed on Al surface by using a two-step press-in procedure of a right angle triangular master grating.
	Hard stamping using Si_3N_4 stamp[17]	4-inch wafer	Silicon nitride stamp was replicated from a silicon master, fabricated using deep-UV lithography and KOH wet etching. Stamping pressure: 5 kN/cm^2.
	Focused-ion-beam[18,20]	Not available	Resist-assisted or direct patterning onto Al surface.
	Colloidal lithography[21]	> cm^2 areas	Colloidal crystals were deposited onto mica surface using an accelerated evaporation induced self-assembly. Stamping pressure: 100 kN/cm^2.
	Block-copolymer self assembly[22]	Not available	Pore size of 12 nm with interval of 45 nm was fabricated using this method.
Al on substrates	Holographic lithography[19]	1 cm x 1 cm	Nanoscale surface corrugations were created by evaporating Al onto the photoresist-grating-patterns on a substrate, developed by holographic lithography.
	Soft imprinting[23]	>1.5 cm^2	This method utilizes argon milling technique to transfer the pattern from a master template, fabricated using a two-steps anodization process, onto the Al surface.
	Nanoimprint lithography[24]	2.5 inch wafer	A 2.5" nickel mold was replicated from a master pattern, fabricated using electron beam lithography, and imprinted into a thermoplastic resist. The resist patterns were then transferred to the Al surface *via* argon ion beam milling.

[a]The current work demonstrates the exceptional capability of prepatterning Al surface on substrates in a large area (4 in. wafer). The method is reliable and compatible with standard silicon fabrication techniques, giving a clear advantage for future development of nanoelectronic devices.

boundaries of the evaporated Al films, the pores of the resulting anodic films are found to be less ordered than those produced from Al foils.[8,19] Two-step anodization method cannot be adopted in the preparation of highly ordered AAO on substrates as the method requires the use of a thick Al film (≳40 μm) for long-time anodization. Depositing such a thick Al film on substrates is certainly not viable from the economical and practical stand points because the quality of the evaporated Al is compromised and the mechanical stress due to the evaporated Al on one side of the substrates could also result in the breakage of the substrates. While hard imprinting-based methods can be utilized to fabricate highly ordered AAO on substrates, the methods are also limited by the pattern transfer protocol, in which the high pressure used (few tones/cm^2 areas) tends to crack substrates with low mechanical strength and damage the imprint stamp after several uses. Other lithographic techniques were proposed to resolve some of these problems, but the methods are favorable for low volume, laboratory-scale production and they have intrinsic drawbacks of low throughput, which is not practical for large-area (wafer-scale) patterning. Their scalability to an industrial-scale mass fabrication also seems infeasible due to their tedious fabrication process and incompatibility to the wafer-scale batch microfabrication widely used by the semiconductor industry. These restrictions greatly impact the practical application of techno-

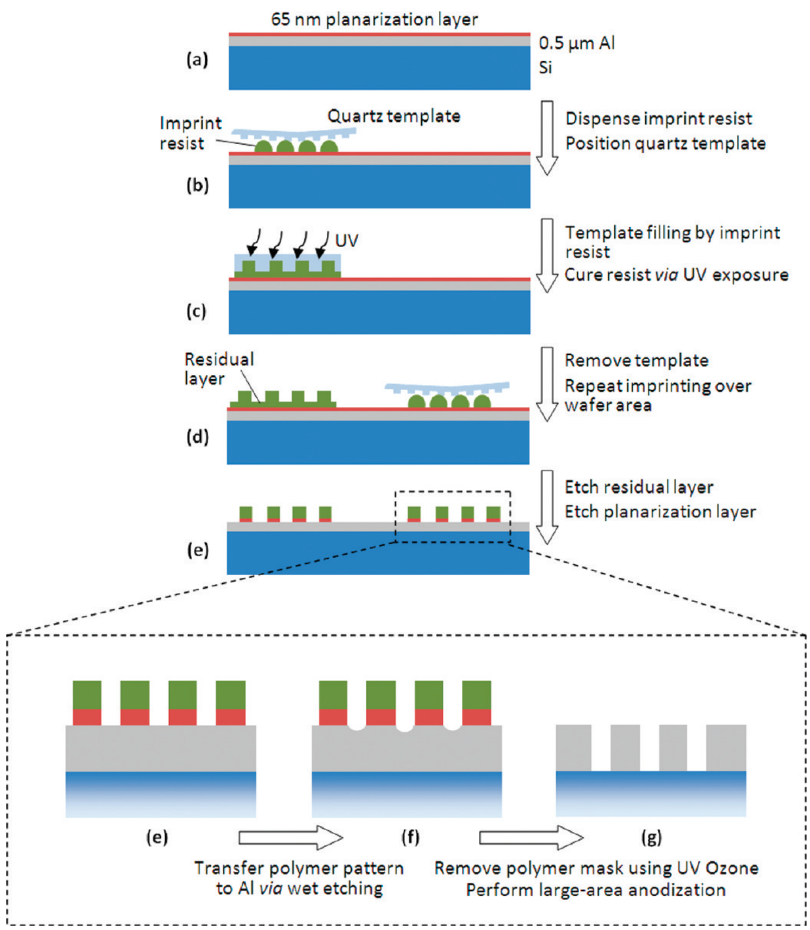

Figure 1. Schematic diagrams showing fabrication procedures to obtain a highly ordered array of high aspect ratio AAO pores on a substrate.

logically important one-dimensional nanostructured materials in the fields of sensing, storage, and optics, as their fabrication process involves the use of AAO as sacrificial templates. For these reasons, it is highly desirable to develop a technique that enables mass fabrication of long-range ordered nanoporous AAO in a single domain over large areas.

Herein, we describe a versatile step and flash imprint lithography (SFIL) procedure that enables the rapid formation of near-perfect ordered AAO on a 4 in. silicon substrate. Wafer-scale samples can be routinely fabricated in several steps, as schematically illustrated in Figure 1. Aluminum films with a thickness of ~500 nm were deposited on a 4 in. silicon substrate using electron beam evaporator with 5 N purity aluminum source (step a). An organic planarization layer material was then spun onto the Al-coated silicon wafer (step a), followed by dispensing nanoliter droplets of UV-curable, low-viscosity materials on the wafer, forming an etch barrier in the imprint area (step b). A patterned quartz template, featuring pillar structures, was subsequently leveled and brought into liquid contact with the substrate, displacing the solution, filling the imprint field *via* capillary action, and trapping the UV-polymerizable imprint field in the template relief (step b). A brief exposure of the solution to UV light initiates cross-linking of monomers in the fluid (step c), after which the template is separated from the substrate, leaving behind an exact inverse replica of the template pattern (step d). This imprint process was repeated throughout the wafer areas until a full-wafer imprint was obtained (step d). Following the imprint process, the sample was exposed to brief oxygen plasma to remove the residual and planarization layers, leaving an ordered array of polymeric holes on the substrate (step e). The pattern was subsequently transferred through the Al layer *via* wet etching to produce nanoindentation on the Al surface, after which the polymer mask was removed using a UV−ozone treatment (step f). After nanoindentation, anodization was conducted under a constant voltage in phosphoric acid to finally obtain a highly ordered array of high aspect ratio AAO pores on a substrate (step f).

Figure 2 shows the scanning electron microscopy (SEM) images of the sample during four key stages of the process in obtaining nanoindentation on the Al surface. As a representative example, we have used a quartz template, featuring 140 nm pillar structures hexagonally ordered across the 10 mm × 10 mm pat-

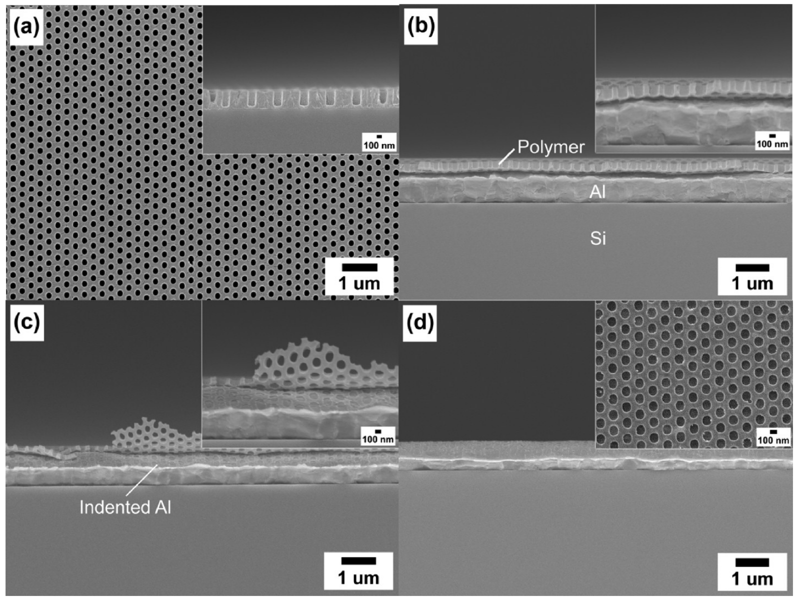

Figure 2. Scanning electron microscopy (SEM) images of the sample at four crucial stages in preparing nanoindentation on the Al surface: (a) step and flash imprint lithography process (inset shows the cross-sectional view of the inverse polymer replica of the template pattern); (b) removal of residual and planarization layers (inset shows the opened nanopores at the bottom of the polymer mask); (c) wet-etching process (inset shows the magnification image of the nanoindented Al surface); (d) removal of polymer mask layer (inset shows a clean prepatterned Al surface after UV−ozone exposure).

terned area for the SFIL process. In contrast to the conventional nanoimprint lithography (NIL), SFIL imprints features at a constant temperature (~22 °C) with low applied pressures (<100 mbar), allowing it to produce high fidelity nanometer-scale features with minimum distortions. In addition, the absence of heating and cooling cycles, as well as the low pressures during imprinting, reduces stress and wear on the nanoimprint template, giving rise to the lifetime of a single template to reach approximately 10 000 imprints.[25] Figure 2a shows the top and cross-sectional (inset) views of the polymer patterns on Al-coated silicon substrate replicated from the quartz template. The polymer patterns have a topography inverse of that of the master quartz pattern, containing 140 nm holes structures with a ~60 nm thick residual layer. An anisotropic RIE process was used to etch away the ~60 nm residual and ~65 nm planarization layers so that the pattern can be transferred to the Al surface during the wet-etching process. Complete removal of these two layers was confirmed by the presence of through holes or opened pores at the bottom of polymer layer, as clearly seen in Figure 2b. After a brief wet etching of the sample in a mixture of phosphoric, acetic, and nitric acids, there is ~50 nm indentation apparent on the Al surface corresponding to the patterns defined in the SFIL step (Figure 2c). In order to ensure a good electrical contact with the electrode during anodization, the Al surface was cleaned from the polymer mask layer by utilizing a combination of UV light, high concentration of ozone, and controlled heating at 100 °C for 20 min. Figure 2d shows that the Al surface was completely free of polymer mask layer, and this observation was further confirmed by analyzing the surface morphology of the indented and non-indented Al surface (see inset).

After nanoindentation, anodization was conducted under a constant applied voltage of 120 V using a direct current (dc) source in 0.3 M H_3PO_4 at 10 °C. The applied voltage V (in volts) was determined based on the interpore distance D (in nanometers) of the resulting 2D nanochannel array according to the empirical relationship $D = 2.5V$ first reported by Keller *et al.*[26] In this work, the pitch of the pillar structures of the quartz template dictates the interpore distance, and the anodization voltage should be selected to correspond to the pitch size of the pillars template. Theoretically, it is possible to prepattern Al for any interpore distance down to sub-100 nm feature resolution using SFIL, provided that the process and infrastructure associated with the fabrication of templates are addressed. Experimentally, we have successfully prepatterned and anodized Al on a 4 in. silicon wafer using pillar templates with a pitch size ranging from 200, 250, and 300 nm.

Figure 3 summarizes the obtained nanopore array architectures of the anodic alumina with different interpore distance and pore arrangement. As shown in Figure 3a, a highly ordered arrangement of nanopores can be produced using SFIL in large area, removing the uncertainty of having randomly oriented domains observed in purely self-ordered growth alumina. Each of the shallow indentations defined by SFIL and created by wet etching serves as nucleation sites for the development of pores and results in the eventual growth of the pore channels. Some defects were still observed on the sample, and we believe they could be due to the particle contamination on Al-coated silicon wafer prior to the texturing process and the quality of deposited Al films on the substrate. The effect of surface texturing is further elucidated by comparing the surface view of porous alumina obtained from prepatterned (Figure 3b, left side) and nonpatterned Al films (Figure 3b, right side) anodized using the same condition. These images were taken after the sample experienced a pore-widening treatment in 5 wt % H_3PO_4 for 90 min. It is obvious that the porous structures produced from nonpatterned Al films look amorphous without any ordering in terms of pore sizes, pore shapes, and pore arrangement. On the contrary, highly ordered porous structures can be obtained from prepatterned Al films. The diameter of the pores was measured to be ~140 nm, and their interpore distance was ~300 nm, corresponding to the pitch of the pillar structures used for the SFIL patterning.

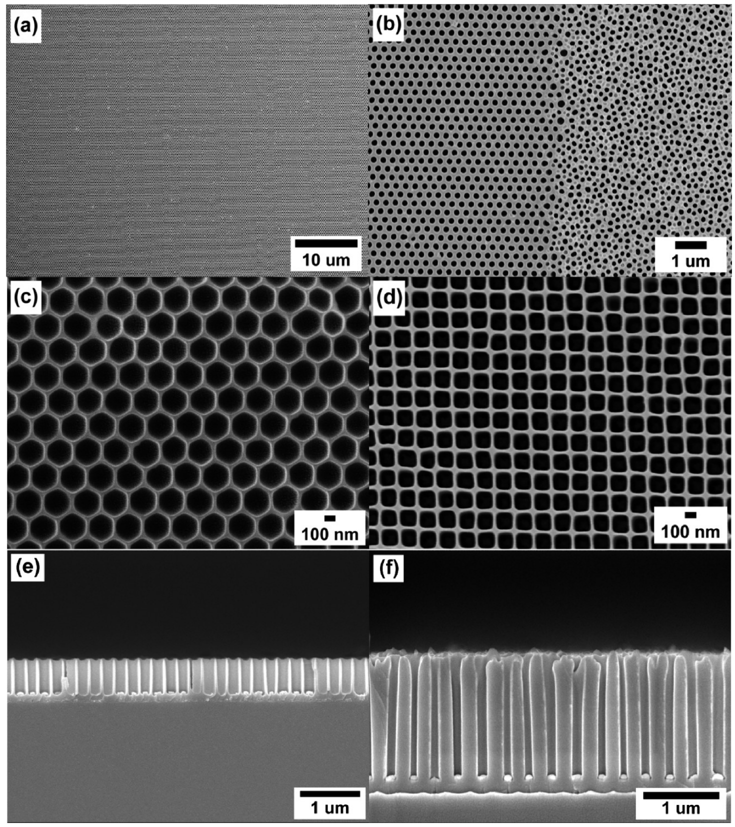

Figure 3. Typical top view (a–d) and cross-sectional view (e,f) SEM images of near-perfect ordered AAO nanopores: (a) sample made from prepatterned Al using 140 nm hexagonal-ordered pillar structures with 300 nm pitch; (b) higher magnification of sample (a) with clear distinction between prepatterned (left) and nonpatterned (right) AAO pore arrangement; (c) honeycomb-like structure made from prepatterned Al using 120 nm hexagonal-ordered pillar structures with 250 nm pitch; (d,e) samples made from prepatterned Al using 100 nm square-ordered pillar structures with 200 nm pitch; (f) sample made from thicker Al films (1 μm) prepatterned using 140 nm hexagonal-ordered pillar structures with 300 nm pitch.

Figure 3c shows another example of porous alumina that was prepatterned using 120 nm pillar structures with 250 nm pitch size and anodized under a voltage condition of 100 V. A honeycomb-like structure with a pore diameter of ~200 nm and interpore distance of ~250 nm was obtained after a pore-widening treatment of 4 h. Although the position of the pores was exactly controlled by the indentation sites on the Al surface (as shown in Figure 3a–c), the shape of the AAO pores is determined not by the shape of the indented Al surface but by the self-ordering pore development of anodic alumina. This is evident from Figure 3d, where we obtained pore arrays with square openings, arranged in a square lattice configuration. The sample was prepared by prepatterning the Al films with 100 nm square-ordered cylindrical pillar structures and performing the anodization at 80 V (corresponds to 200 nm pitch distance), followed by a pore-widening treatment for 2 h. The cross-sectional view of the sample is shown in Figure 3e. In order to confirm the vertical growth of the pore structures along the pore axis during anodization, thicker Al films of ~1 μm were deposited on the substrate and prepatterned using 140 nm pillar templates. The resultant porous structures were straight and uniform in diameter, as shown in Figure 3f.

In comparison with our previous approach using the soft-imprinting method,[23] the approach presented herein has a number of advantages. In the soft-imprinting method, a free-standing highly ordered AAO template was fabricated *via* a two-step anodization process and used as a mask to create nanoindentation on the Al surface *via* argon plasma etching. The SFIL patterning method resolves the issue of large-area fabrication and the problems of having inefficient pattern transfer from the highly ordered AAO mask to the Al surface due to insufficient pore openings of the mask and imperfect adhesion between the mask and the substrate. The SFIL patterning method also allows the fabrication of AAO template with a wide range of interpore distances and pore arrangement (triangular, square, or hexagonal lattice configuration).

An alternative patterning strategy that is viable to grow a highly ordered, single-domain AAO template on a large area of foreign substrates is *via* a holographic lithography (HL) technique.[19] Essentially, the HL process involves the formation of a stationary spatial variation of intensity created by the interference of two or more beams of light. The pattern that emerges out of the intensity distribution is transferred to a light-sensitive medium, such as a photoresist, to yield structures. In the case of growing ordered structures in the AAO template, one-dimensional (1D) or two-dimensional (2D) photoresist grating patterns were developed on substrates and a conformal deposition of Al films was performed on the grating patterned substrates to create surface-corrugated Al films. It was dem-

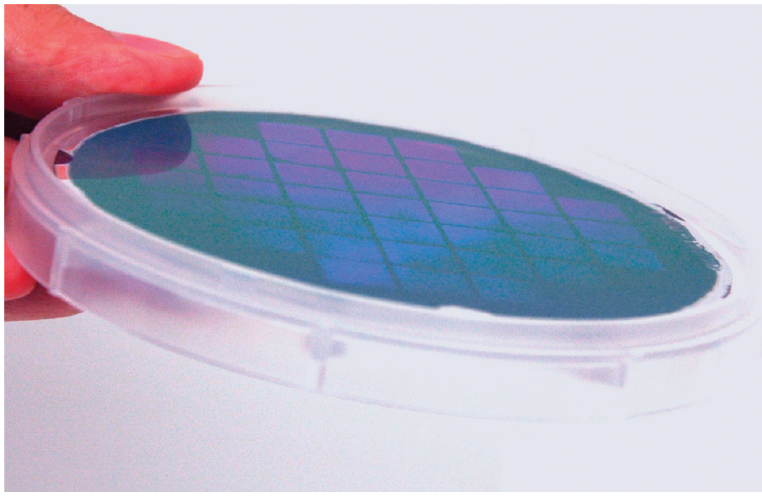

Figure 4. Photograph of a near-perfect AAO template on a 4 in. silicon substrate. The 10 mm × 10 mm square areas with bright light diffraction indicate the anodized sample that was prepatterned using SFIL.

onstrated that the predefined nanoscale periodic corrugation of the Al film surface compensates the randomizing effect of grain boundaries in Al films and assists in controlling the development of highly ordered pore structures across the entire patterned area. While the HL technique offers flexibility and controllability in generating a highly ordered AAO template in a large area, the method requires a complex optical setup and has some issues on the material platform related to the process. For some device applications, it may be desirable to remove the HL-patterned polymeric structures, which were used to construct the surface-corrugated Al films. However, removal of the highly cross-linked polymers (commonly epoxy-based materials) is difficult[27] and may require rather extreme processing such as resist burning, which may result in damage to the other structures or building blocks of the device. To avoid this problem, a UV-curable acrylic-based material was used in the SFIL process so that it can easily be removed through UV−ozone plasma etching. The material used in SFIL also has other advantages, such as low viscosity, which allows access to high aspect ratio structures on the template; rapid photopolymerization to maintain high-throughput during the step and repeat imprinting process; minimal shrinkage upon polymerization to ensure good pattern transfer from the template; and finally thermal stability to common temperatures associated with reactive ion etching process.

While several methods have been reported to grow highly ordered AAO on substrates, each with its own set of advantages and disadvantages, the fundamental challenge in developing a simple fabrication process of cost-effective, high-throughput, and compatible to silicon fabrication technique has yet to be entirely resolved. An industrial-compatible nanofabrication technology, nanoimprint lithography, was recently proposed to manufacture ideally ordered alumina nanopores for patterned disk media application.[24] In this approach, a nickel mold, replicated from a 2.5 in. master pattern prepared *via* electron beam lithography, was used to imprint a thermoplastic resist (polymethylmethacrylate), and the patterns were then transferred to the Al surface to guide the development of the alumina nanopores at the initial stage of the anodization. Despite the success of large-area alumina patterning *via* nanoimprint lithography, the initial method of fabricating the master mold as large as 2.5 in. wafer areas using electron beam lithography is clearly neither practical nor cost-efficient. The corresponding process becomes challenging when there is a need to manufacture a much larger area of ideally ordered alumina nanopores. To circumvent this disadvantage, SFIL employs the step and repeat patterning process to replicate a small patterned area from the template onto a full-wafer area. This distinctive ability of SFIL enables a full-wafer patterning process while still offering an economical and straightforward solution to the costly large-area e-beam written templates.

Figure 4 shows a photograph of a near-perfect ordered AAO template fabricated on a 4 in. silicon substrate using the SFIL patterning method. For easy visualization of the nonpatterned and prepatterned anodized sample, white light was illuminated onto the sample because a well-ordered porous structure should show strong light diffraction and therefore the anodized pattern quality can be easily examined by the naked eye. Homogeneous pattern can be achieved over large areas, which was clearly indicated by a uniform single-color tone on the prepatterned 10 mm × 10 mm square areas.

In summary, we have demonstrated the fabrication of a high-quality, wafer-scale, and high aspect ratio AAO template with controllable interpore distance and pore lattice configuration by step and flash imprint lithography (SFIL). Samples as large as 4 in. diameter can be routinely fabricated, and highly ordered AAO with interpore distances of 100, 250, and 300 nm, arranged in square and hexagonal lattice configuration, was produced. Besides providing a versatile route to create a near-perfect ordered high aspect ratio of nanopore structures, the SFIL method has major merits in its compatibility with semiconductor device manufacturing and its long lifetime of the template for Al patterning, and hence may open up a new way of producing perfectly ordered AAO on substrates for various device applications.

METHODS

Nanofabrication: Step and flash imprinting process was performed using Imprio 100, Molecular Imprints, Inc. Aluminum (99.999% purity) films ~0.5 μm thick were deposited on a 4 in. silicon substrate by electron beam evaporation (Edwards Auto306). An organic material (Transpin HE-0600, Molecular Imprints, Inc.) was spun onto the Al-coated silicon wafer at two-step spin speeds, 1800 rpm for 3 s and 3000 rpm for 30 s, to obtain a 65 nm thick planarization layer. Liquid acrylate imprint resist was subsequently dispensed across the surface of the silicon wafer. A quartz template, with the size of 65 mm × 65 mm and 10 mm × 10 mm active patterned area, was lowered until contact was made with the imprint resist, and capillary action induced the liquid imprint resist to completely fill the region between the substrate and the topography of the imprint template. Fabrication of the quartz template was realized using conventional electron beam lithography and etching process. Several types of quartz templates were employed in these studies: (1) 140 nm hexagonally ordered pillar templates with a height of 300 nm and a pitch of 300 nm; (2) 120 nm hexagonally ordered pillar templates with a height of 300 nm and a pitch of 250 nm; (3) 100 nm squarely ordered pillar templates with a height of 300 nm and a pitch of 200 nm. Following the imprint process, the imprint resist was photopolymerized *via* ultraviolet illumination, after which the template is separated from the substrate, leaving behind an exact inverse replica of the template pattern. This imprint process was repeated throughout the wafer areas until a full-wafer imprint was obtained. After the imprinting process, the sample was exposed to oxygen plasma in a RIE Oxford Etcher, Plasmalab ICP180 with the following parameters: 80 sccm of O_2, 100 mT of chamber pressure, 100 W of RIE power, and 120 s of etching time. The polymer mask was removed by treatment with UV−ozone in a dry stripper (Samco UV-1) at 100 °C for 20 min. After nanoindentation, anodization was conducted under a constant voltage of 120 V (for 140 nm pillars), 100 V (for 120 nm pillars), and 80 V (for 200 nm pillars) in 0.3 M H_3PO_4 at 10 °C.

Characterization: High-resolution SEM imaging was carried out with a JEOL FESEM JSM-6700F.

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