Tuning of sculptured-thin-film spectral-hole filters by postdeposition etching

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Abstract. Postdeposition chemical etching of spectral-hole filters, which were fabricated as chiral sculptured thin films with central 90-deg-twist defects, decreases the cross-sectional dimensions of the helical columns that such films comprise and blueshifts the spectral holes, thereby establishing the efficacy of postdeposition chemical etching as a means to tune the optical response characteristics of sculptured thin films.
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Subject terms: circular Bragg phenomenon; etching; sculptured thin film; spectral-hole filter.

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1 Introduction

Sculptured thin films (STFs) are engineered porous nanomaterials containing shaped parallel columns.[1–3] These films are grown by directing the vapor flux from either one source or many sources of bulk matter in an evacuated chamber toward a substrate whose position and orientation are dynamically controlled to grow columns of different shapes. The concurrent control of porosity makes STFs very attractive as optical filters and sensors[4–6] and even as laboratories for testing technoscientific concepts at the nanoscale.[7]

A prominent class of STFs contain helical columns and are called *chiral STFs* because of their structural handedness.[3,4] Being unidirectionally periodic also, chiral STFs exhibit the circular Bragg phenomenon (CBP). CBP is best described as the high reflectance, within a narrow spectral regime, of circularly polarized light of the same handedness as the chiral STF of sufficient thickness, while circularly polarized light of the opposite handedness is reflected very little. CBP is exploited for circular polarization filters and sensors.[4,8]

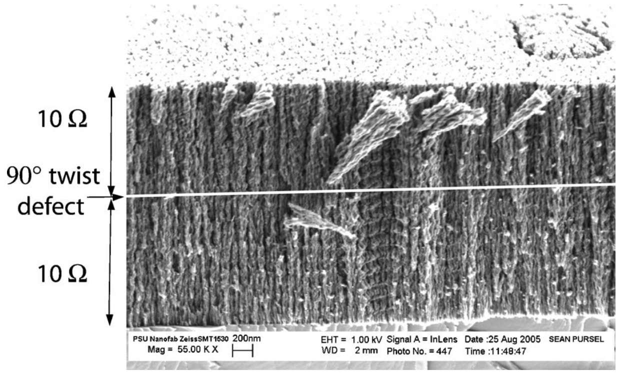

Fig. 1 Cross-sectional image on a scanning electron microscope of the 10-period, left-handed, chiral STF with a central 90-deg-twist defect. The film is made of titanium oxide, and the location of the twist defect is highlighted by a white line. Each period $2\Omega = 278$ nm.

As such optical devices have been fabricated with STF technology already for a few years, postdeposition processes to tune the optical response characteristics, directly or indirectly, are now being paid attention to. For incorporation into optical systems, STF devices must be environmentally stable and possess the correct morphological properties—such as porosity and thickness—that influence the optical response characteristcs. Porous films can be made environmentally more stable by capping[9] and/or annealing,[10] but both processes would affect the optical response characteristics.[11,12] Those effects must be taken into account when designing optical devices. At the same time, those effects provide an opportunity to tune the optical response characteristics after deposition.

Significantly in this context, postdeposition annealing of chiral STFs made of titanium oxide was recently shown to blueshift the spectral regime of the CBP. This blueshift may be attributed to many factors including columnar thinning. Indeed, theoretical research had earlier indicated that columnar thinning would cause the blueshift.[13] Thus, both theory and experiment suggested that controlled columnar thinning can be used to tailor the optical response characteristics of STFs, and we decided to develop postdeposition chemical etching as a way to tune those characteristics.

For this purpose, we chose a chiral STF with a central 90-deg-twist defect because it has a narrow low-reflectance regime—a spectral hole—right in the center of the high-reflectance Bragg regime for cohanded circularly polarized light.[5,14] The spectral hole, being 5 to 15 nm in the full-width-at-half-maximum bandwidth, is conveniently sharp to experimentally assess any changes in the optical re-

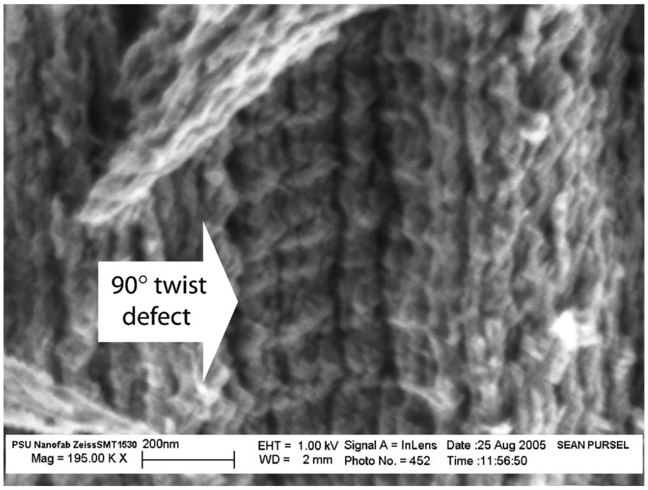

Fig. 2 A high-magnification cross-sectional image of the fabricated chiral STF to show the twist defect.

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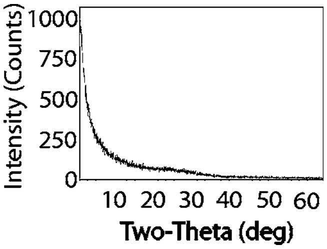

Fig. 3 XRD pattern of an as-deposited chiral STF made of titanium oxide.

sponse characteristics.[8] Spectral-hole filters are commonly used for optical distance measurement, interferometers, and surveillance systems.[15]

2 Experimental Procedures

2.1 *Fabrication*

The chosen spectral-hole filter was fabricated of titanium oxide. A physical vapor deposition method called *serial bideposition*[4,12] was used to grow a left-handed chiral STF containing 10 full periods, with the top 5 periods being rotated 90 deg about the thickness axis relative to the bottom 5 periods. With 7 cm^3 of 99.99%-pure titanium-oxide pellets (International Advanced Materials) in a boat kept 29 cm from an unheated 7059 Corning glass substrate, the STF was deposited at a base pressure of 8×10^{-7} Torr. The serial bideposition technique of Hodgkinson et al.[4] was slightly modified by using a constant substrate rotation (except when engineering the twist defect) and constant deposition rate.

Figures 1 and 2 contain cross-sectional images of the fabricated chiral STF with the central twist defect. Results from previous studies on STFs prepared in the same manner indicate the as-deposited STF lacks crystallinity,[12] which is also confirmed by the absence of any peaks in the x-ray diffraction (XRD) pattern shown in Fig. 3.

2.2 *Chemical Etching and Optical Characterization*

The centrally defected chiral STF was chemically etched and optically characterized 50 days after deposition. It was etched five times by immersion in 10% HCl, the first four times for 10 min and the last time for 40 min. After each immersion, the STF was thoroughly rinsed with deionized water and then placed on a warm hot plate for 60 to 120 s to drive off excess moisture; immediately thereafter, the film's circularly polarized transmission spectrum was recorded using two combinations of a Glan-Thompson linearly polarizing prism and a Fresnel rhomb. One of these combinations located in the optical path before the chiral STF set the handedness of the incident light, and one combination after the film set the handedness of the transmitted light whose intensity was measured with an Ocean Optics spectrometer. The entire sequence of postdeposition chemical etching and optical characterization was completed within 2 h.

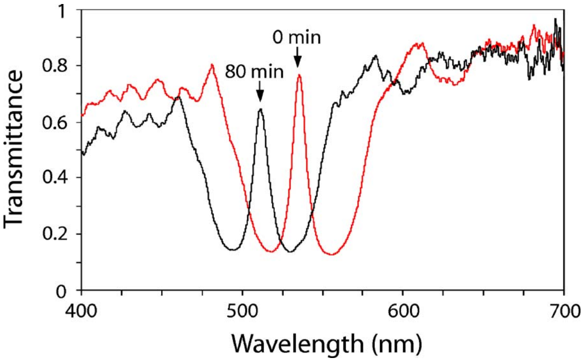

Fig. 4 Measured transmittance spectrums of an incident left circularly polarized plane wave as a left circularly polarized wave, before and after immersion of the centrally defected, left-handed chiral STF for 80 min in 10% HCl. The spectral hole in either transmittance spectrum is evident as a spike.

3 Results and Discussion

Immersion of the centrally defected chiral STF in 10% HCl for a total of 80 min caused the spectral hole to blueshift a total of 25 nm, as is evident from the two spikes in the transmittance spectrums presented in Fig. 4. Even more significantly, this figure shows that the entire Bragg regime blueshifts as a result of etching, thereby validating a theoretical prediction made three years ago.[13]

Evidence of the blueshift at intermediate steps of the etching-and-measurement sequence is available in Fig. 5, and the spectral location of the spectral hole as a function of the duration of immersion is presented in Table 1.

Each of the first four 10-min immersions caused the spectral hole to blueshift by 4 to 6 nm. The last 40-min immersion also caused a 6-nm blueshift, suggesting the occurrence of a rate-limiting transport phenomenon. Such a rate-limiting transport phenomenon has been seen in photolithographically defined narrow trenches with very high aspect ratios.[16] This limiting step allows controllable columnar thinning that does not directly depend on postetch rinsing. However, this limiting step could cause nonuniform etching, which is an undesirable possibility that requires further study and eventual elimination, possibly by initial agitation.

Thus, we have experimentally demonstrated that chemical etching by simple immersion in an acid for a specific duration causes the spectral hole in the reflectance spectrums of centrally defected chiral sculptured thin film to blueshift by a specific amount, which is in accord with

Table 1 Location of the spectral hole as a function of the duration of immersion for postdeposition etching.

Duration of immersion (min)	0	10	20	30	40	80
Location of spectral hole (nm)	536	530	525	520	517	511

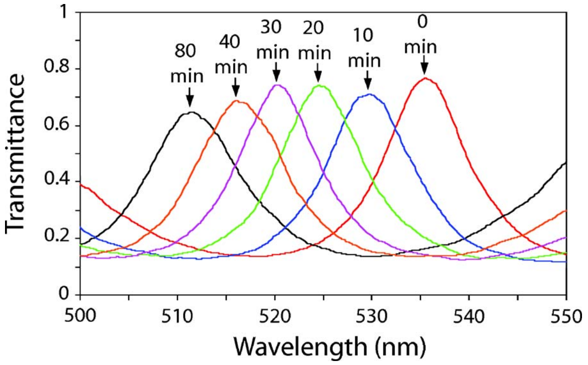

Fig. 5 Same as Fig. 4, except after 0, 10, 20, 30, 40, and 80 min of postdeposition chemical etching.

theoretical prediction of the consequences of columnar thinning.[13] Our experimental results also correlate well with the blueshift due to postdeposition annealing of chiral STFs.[12]

We are developing postdeposition chemical etching of STFs as a general technique to tune their optical response characteristics. The correlation of the duration of etching with porosity is currently being investigated. Future work will characterize postdeposition chemical etching of STFs using different bulk materials, acids, and crystalline phases to determine the role of the rate-limiting transport phenomenon and the effectiveness of etching for different types of STF devices.

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