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自支撑Al滤光片的制备

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摘　要:分别以AZ50XT和聚乙烯醇为脱膜剂,在本底真空度为5.0×10^{-4} Pa的磁控溅射镀膜机中沉积Al膜,制备出厚度为80 nm的自支撑Al滤光片,并对滤光片进行表面缺陷分析。通过扫描电镜和CMOS相机观察得到,制备的Al滤光片表面均匀性较好,有少量针孔。对滤光片的光学性能进行了表征。用紫外可见分光光度计测得滤光片在可见光及红外光波段的透过率低于0.02%,基本满足使用要求。用软X射线透过率测试系统测得滤光片在1.6～10 keV能段的透过率高于90%,透过率曲线与理论结果基本一致,满足应用要求。用同步辐射装置测得两种滤光片在50～250 eV能段的最高透过率分别为53%和35%,受Al膜表面氧化和脱膜剂残留的影响,实际测得的透过率比理论计算值偏低。

关键词:薄膜;自支撑;脱膜剂;软X射线;极紫外;透过率
中图分类号:P171.3　　**文献标识码**:A　　**doi**:10.3788/gzxb20225106.0631001

0 引言

真空紫外及软X射线滤光薄膜在天体物理、材料科学、生物医学工程、微电子及国防军工等方面有非常重要的应用[1]。太阳活动是地球空间环境改变的根源所在,也是空间天气预报的首要监测目标。因此,对太阳活动的观测有十分重要的意义。对太阳活动监测和预警的最佳方法之一,就是对X射线和极紫外波段的成像和能谱观测[2]。利用X射线和极紫外望远镜对相应波段进行观测时,探测器通常会受到可见光以及红外光等杂散光的干扰,影响其探测精度和性能,因此必须在探测器前增加滤光片[3-4],这些滤光片通常为几百纳米的金属薄膜。例如,美国宇航局发射的太阳与日光层观测卫星(Solar and Heliosphere Observatory, SOHO)上的极紫外望远镜(Extreme-ultraviolet Imaging Telescope, EIT)[5]和日地关系观测台(Solar Terrestrial Relations Observatory, STEREO)上的极紫外成像仪(Extreme UltraViolet Imager, EUVI)[6-8]中都使用了150 nm的自支撑Al膜作为滤光片,以阻止杂散光对探测器的影响。国外的Luxel公司是主要的滤光片制造商,制备工艺成熟,制造的滤光片广泛应用于天文观测卫星,目前其制造的用于X射线和极紫外探测的滤光片多为自支撑的聚酰亚胺镀铝膜,但是在极紫外观测中,聚酰亚胺对紫外光有一定吸收,因而不利于极紫外波段的观测;2016年,NIKOLAY C等[9]研究了自支撑的Al/Si、Zr/Si、Be/Si和Cr/Si多层薄膜,2019年,该团队又对自支撑的铍基极紫外滤片进行了研究[10];2020年,JIMENEZ K等[11]对自支撑的Nb/Zr和Zr/Nb滤片的透射性能进行了研究。在国内,对X射线、极紫外自支撑滤片的研究以同济大学最具代表性,2009年,吴永刚团队[1]制备了自支撑的Cr/Al/Cr薄膜,2011年,其团队又对自支撑的Zr膜进行了研究[12];近年来,同济大学对极紫外薄膜元件的研究以多层膜反射镜为主。

自支撑薄膜的制备通常是在一些特殊的衬底上先合成薄膜,然后将衬底去除,得到所需的自支撑薄膜。目前获得自支撑薄膜的方法主要有衬底腐蚀和脱膜剂法[13]两种。脱膜剂的方法操作简单,易于实现。以往

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所用的脱膜剂(如 NaCl 和 CsI)多极易潮解,对存储环境有严格的要求,易于给镀在脱膜剂上的薄膜带来缺陷[14]。本文采用脱膜剂的方法,分别以 AZ50XT 光刻胶和聚乙烯醇作为脱膜层,制备出了 80 nm 的自支撑 Al滤光片,利用扫描电镜和CMOS相机对薄膜的表面形貌和针孔进行表征,同时对制备的自支撑 Al膜在紫外、可见和红外光波段,软X射线波段及极紫外波段的光学性能进行了表征。

1 滤光片的制备

1.1 镍框的制备

通过掩膜光刻和微电铸的工艺制备镍框,用来支撑 Al膜,其目的是使制备的自支撑 Al膜有规则的形状,并且在测试或实际应用中能够与设备进行匹配安装。掩膜光刻技术是指在光照作用下,借助光刻胶将掩膜版上的图形转移到基片上的技术。微电铸工艺是将光刻胶图形复制转移到金属材料上的一种技术方法,其主要利用电化学原理在光刻胶膜微结构中沉积金属,得到高精度的金属微结构[15]。

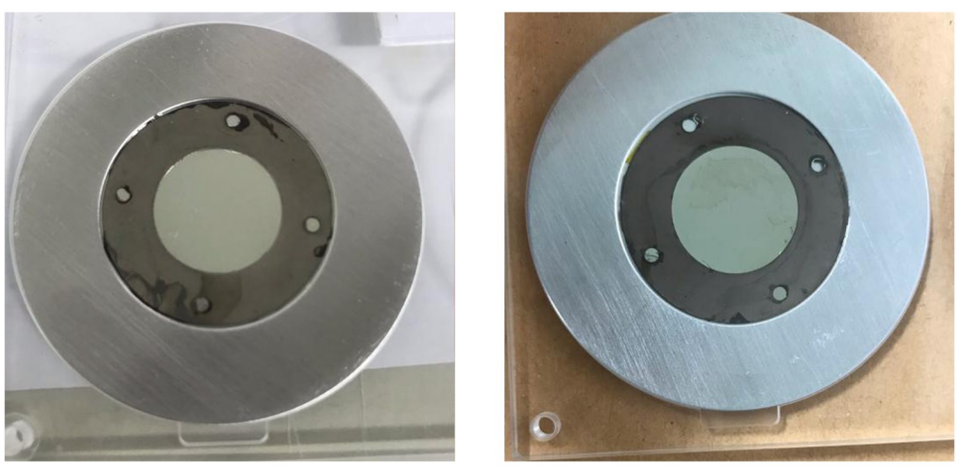
实验中,通过两次掩膜光刻在干净的Si片上复制镍框图形,然后利用微电铸工艺制备镍支撑框,制备完成后,将镍框取下以备使用。镍框的厚度约为 60～70 μm,内径为 16 mm,外径为 28 mm,与样品接触面光滑平整,易于粘接。

1.2 自支撑 Al膜的制备

实验中,分别使用 AZ50XT 光刻胶和聚乙烯醇来制备脱膜层。AZ50XT 是一种正性光刻胶,性能稳定,是应用于电镀工艺的超厚光刻胶。通过匀胶旋涂仪旋涂 AZ50XT 光刻胶制备脱膜层,胶层厚度约为 2～3 μm,制备的脱膜层表面均匀,无明显缺陷。

聚乙烯醇(PVA)是一种高分子聚合物,具有良好的水溶性、成膜性且无毒无味[16-19]。实验中使用浓度为 8% 的聚乙烯醇溶液,通过将其旋涂于干净的Si片上,然后在加热台上烘干来制备脱膜层。由于聚乙烯醇的水溶性会随着温度的升高和加热时间的增加而降低[20],为了防止脱膜时溶解速率过低,实验中将聚乙烯醇脱膜层放在 60℃加热台上烘 3 min,制得的脱膜层厚度约为 900 nm,表面有少量缺陷。

将涂有脱膜剂的Si片放入磁控溅射镀膜机中,沉积 80 nm 厚的金属 Al,Al的厚度通过晶振膜厚仪来监测。磁控溅射镀膜机中的 Al靶纯度为 99.99%,镀膜室本底真空度为 5.0×10^{-4} Pa。沉积完成后,用环氧树脂胶将制备好的镍框粘到样片表面,待其固化后,放入丙酮或去离子水中脱膜。当 Al膜浮起后,将其取出,并对其表面进行清洗。实验中制备的样品如图 1 所示,样品表面均匀平整,无明显缺陷。



(a) AZ50XT (b) Polyvinyl alcohol

图 1 两种脱膜剂制备的自支撑 Al滤光片样品

Fig. 1 Self-supporting Al filter prepared by two release agents

为了验证晶振膜厚仪对厚度监测的准确性,在相同条件下在干净的Si基片上沉积厚度为 80 nm 的 Al,然后用台阶仪对膜厚进行标定。实验中选取了四个位置进行标定,标定样品如图 2 所示。

图 3 为四个位置的标定结果,从图中可以看出,Al的厚度在误差允许范围内,平均值为 80.75 nm,因此晶振膜厚仪对厚度的监测基本准确,可以确定所制备的自支撑 Al滤光片厚度约为 80 nm。

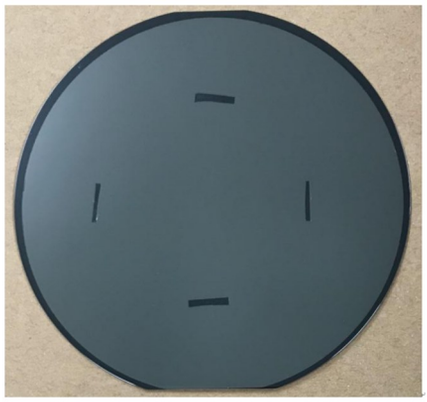


图2　标定样品

Fig. 2　Calibration sample

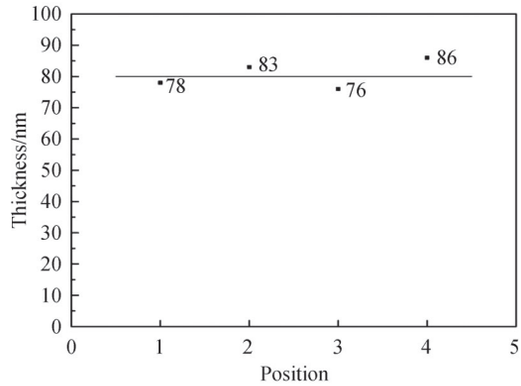
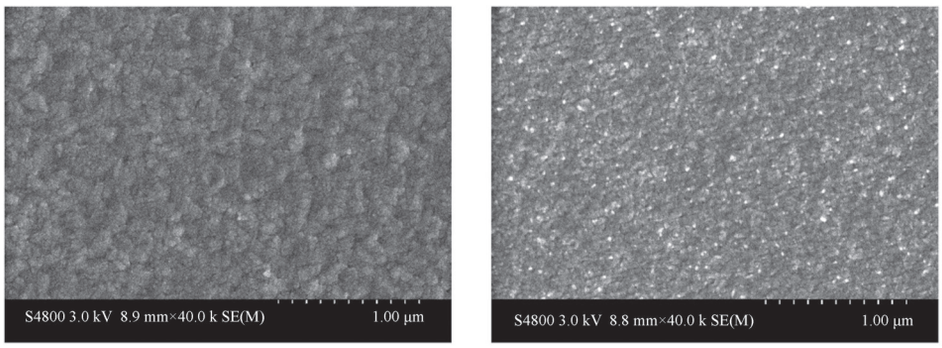


图3　Al膜厚度标定结果

Fig. 3　Al film thickness calibration result

薄膜表面的均匀性、平整性是表征薄膜性能的重要参数,为此使用扫描电子显微镜对薄膜表面进行观察,如图4所示。受光刻胶材料本身性质的影响,2~3 μm厚的胶层,其表面也不光滑,所以,用它制备的Al滤光片表面颗粒较大;而聚乙烯醇制备的脱膜层厚度约为900 nm,其表面比光刻胶脱膜层要光滑,因而用它制备的Al滤光片表面颗粒较小。两种脱膜剂制备的Al滤光片表面颗粒分布均匀,没有明显的微缺陷。

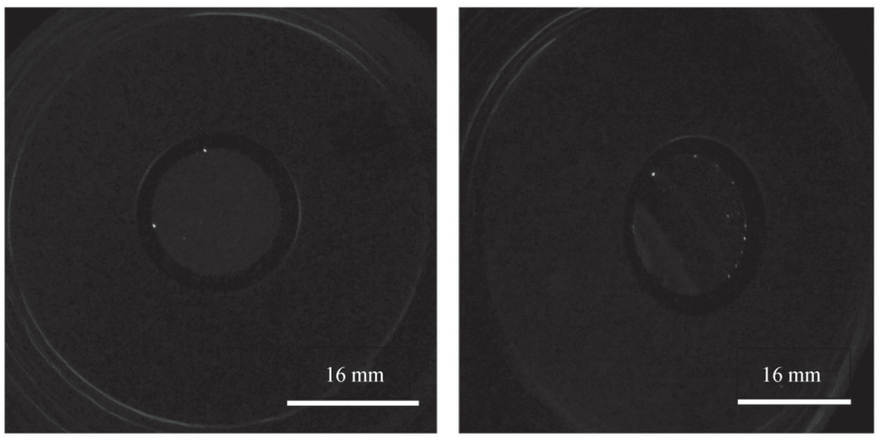


(a) AZ50XT　　(b) Polyvinyl alcohol

图4　Al滤光片扫描电镜图

Fig. 4　SEM images of Al filter

针孔是薄膜滤光片中普遍存在的问题,其不仅允许测试光谱区域以外的辐射到达探测器,而且是滤光片的机械弱点。为了表征滤光片表面的针孔,在封闭的暗室中,将制备的样品置于光源前,然后使用CMOS相机捕捉薄膜的漏光点,如图5所示。可以看出,AZ50XT脱膜剂制备的滤光片针孔较少,而聚乙烯醇脱膜



(a) AZ50XT　　(b) Polyvinyl alcohol

图5　Al滤光片针孔

Fig. 5　Pinholes of Al filter

剂制备的滤光片针孔要稍多一些。滤光片中心区域几乎没有针孔，镍框边缘处针孔的产生可能由粘附镍框时环氧树脂胶的溢出所引起。

2 自支撑Al滤光片的光学性能测试

2.1 紫外、可见以及红外光透过率测试

利用岛津UV3600Plus型紫外分光光度计测试了自支撑Al滤光片在紫外、可见及红外光波段的透过率，测试的波长范围为200～1 200 nm。图6展示了两种不同脱膜剂制备的80 nm自支撑Al滤光片的透过率测试结果，可以看出，两种脱膜剂制备的Al滤光片在紫外、可见以及红外光波段的抑制水平都能达到10^{-4}量级，透过率在紫外波段内略高于0.02%，在可见及红外光波段内都低于0.02%。受Al滤光片表面针孔数量的影响，AZ50XT光刻胶制备的Al滤光片对紫外、可见及红外光的抑制能力比用聚乙烯醇制备的Al滤光片更强。

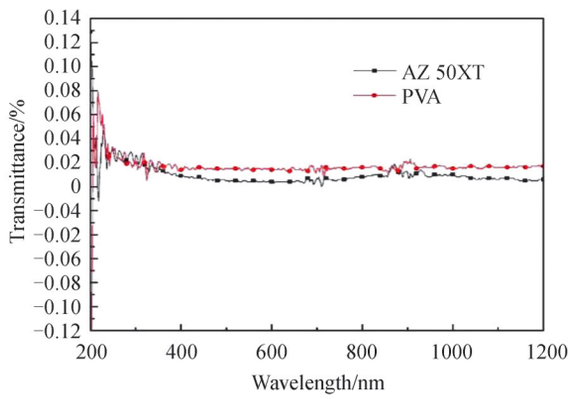


图6 不同脱膜剂制备的Al滤光片在紫外、可见以及红外光波段的透过率

Fig. 6 The transmittance of Al filters prepared by different release agents in ultraviolet, visible and infrared bands

将AZ50XT脱膜剂制备的80 nm Al滤光片与空间X射线探测常用的聚酰亚胺镀铝膜进行了比较，聚酰亚胺镀铝膜厚度为400 nm PI＋80 nm Al，测试结果如图7所示。可以看出，在400～1 200 nm波段，自支撑的单层Al滤光片对可见及红外光的抑制能力要优于聚酰亚胺镀铝膜。在200～400 nm波段，由于聚酰亚胺对紫外光有吸收，无聚酰亚胺支撑的Al滤光片对紫外光的透过率更高，因此，单层的Al滤光片更有利于紫外波段的探测。

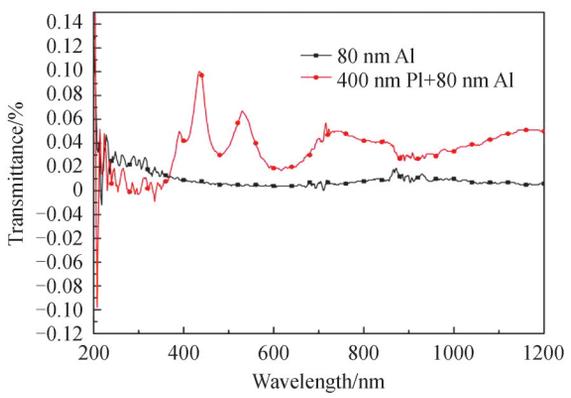


图7 80 nm自支撑Al滤光片与400 nm PI＋80 nm Al滤光片透过率比较

Fig. 7 Comparison of transmittance between 80 nm self-supporting Al filter and 400 nm PI ＋ 80 nm Al filter

2.2 软X射线透过率测试

自支撑Al滤光片的软X射线透过率测试是用实验室自己搭建的系统进行的。该系统由X射线光管（靶材为Ag，窗口为Be）、真空腔室，平移台和硅漂移探测器（Silicon Drift Detector，SDD）组成，在给定的管电

压、管电流下,通过测定透射光强(经过滤光片)与入射光强(不经过滤光片)的比值,来表征薄膜在软X射线波段的透过率[21-22]。因为计数与X射线的强度成正比,因此可以用计数来表示不同能量段的软X射线强度。测试时X射线光管的管电压为10 kV,管电流为5 μA,因为Be窗的存在,低能光子被屏蔽,没有低能光子从X射线光管中射出,所以,1.6 keV以下的能量段可以忽略。

两种脱膜剂制备的80 nm自支撑Al膜的软X射线透过率如图8所示,可以看出,两种样品在1.6~10 keV能量段的X射线透过率基本一致,且都高于90%。为了验证测试结果的可靠性,将实测结果与文献[23]中理论计算结果进行比较,如图8所示,实际测得的透过率曲线趋势与理论值基本一致,证明测试结果可靠。

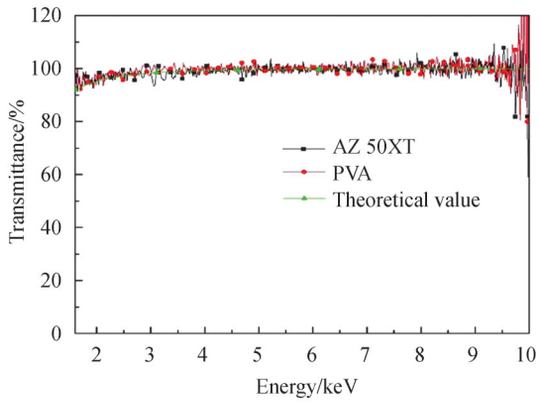


图8 两种脱膜剂制备的Al滤光片在软X射线波段的透过率以及与理论值的比较

Fig. 8 The transmittance of Al filter prepared with two kinds of release agents in soft X-ray band and the comparison with the theoretical value

1.3 极紫外波段透过率测试

为了表征制备的自支撑Al滤光片在极紫外波段的光学性能,测试了50~250 eV能段内滤光片的透过率,结果如图9所示。可以看出,AZ50XT脱膜剂制备的Al滤光片在其通带内的透过率高于聚乙烯醇脱膜剂制备的滤光片,前者的透过率能达到53%,而后者只能达到35%,并且实际测得的透过率与理论值相比有较大的差异。

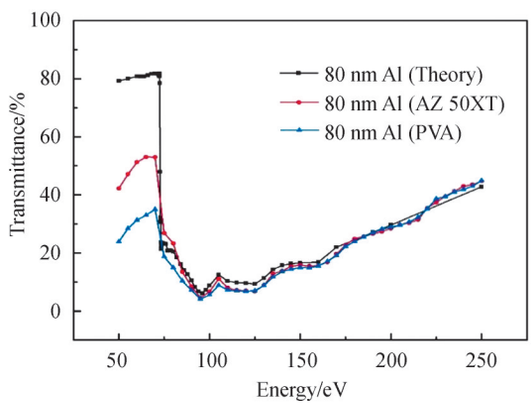


图9 滤光片在50~250 eV能段的测量值与理论值比较

Fig. 9 Comparison between the measured and theoretical values of the filter in the energy range of 50~250 eV

Al滤光片表面氧化、脱膜剂残留都会导致滤光片的实际透过率低于理论值,为此,通过X射线光电子能谱对两种脱膜剂制备的Al滤光片表面进行元素分析,测试表面为与脱膜剂接触的一面,X射线光电子能谱(X-ray Photoelectron Spectroscopy,XPS)测试结果如图10所示。可以看出滤光片表面有较强的C、O元素的峰,没有明显的Al元素的峰,说明Al滤光片表面有脱膜剂残留,脱膜剂中的C、O等元素对极紫外光的吸收导致实际测得的透过率比理论值要低。表1为曲线拟合后得到的各谱峰面积,可见两种脱膜剂制备的Al滤光片表面O元素含量差异较大,这与残留脱膜剂的化学组成不同有关。与AZ50XT光刻胶在丙酮中的溶

解度相比，聚乙烯醇在水中的溶解度要低得多，因此，用聚乙烯醇脱膜剂制备的Al滤光片表面会残留更多的脱膜剂，导致其在通带内的透过率低于AZ50XT脱膜剂制备的Al滤光片。

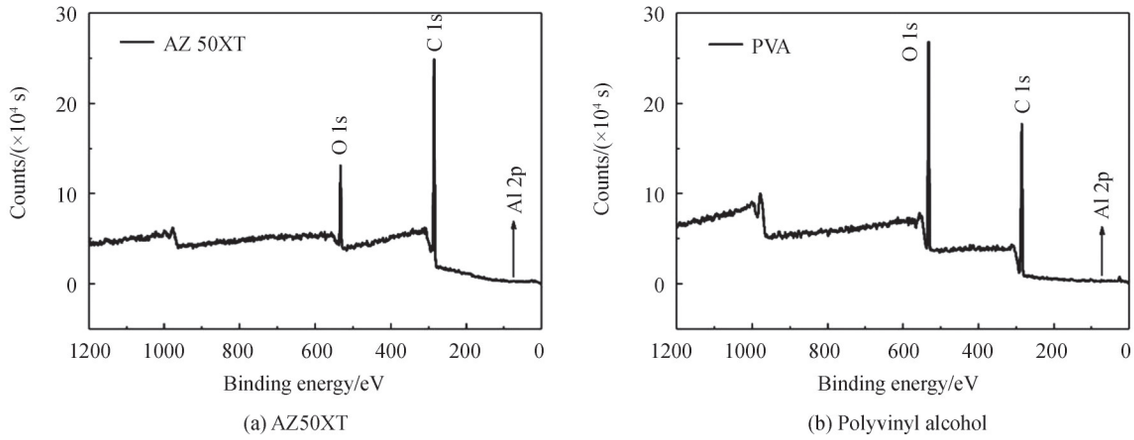


图10 两种脱膜剂制备的Al滤光片XPS测试结果

Fig. 10 XPS test results of Al filters prepared by two release agents

表1 两种样品中C、O、Al元素谱峰面积

Table 1 The peak areas of C, O and Al elements in the two samples

Sample	Name	Bending energy peak	Peak area/(count•s^{-1}•eV)
AZ50XT	C1s	285.03	651 118.28
	O1s	533.06	288 188.34
	Al2p	73.31	3 736.61
PVA	C1s	285.03	609 719.20
	O1s	533.06	739 981.45
	Al2p	74.27	3 072.78

3 结论

本文分别以AZ50XT光刻胶和聚乙烯醇作为脱膜剂，用磁控溅射沉积Al，制备了80 nm自支撑Al滤光片，通过扫描电镜和CMOS相机分析了滤光片表面缺陷及针孔，并在紫外、可见以及红外光波段，软X射线波段和极紫外波段对滤光片的光学性能进行了表征。研究表明，两种脱膜剂制备的Al滤光片表面均匀性较好，以AZ50XT为脱膜剂制备的滤光片表面针孔比聚乙烯醇脱膜剂制备的滤光片少，使得前者对紫外、可见及红外光的抑制能力更强。在1.6～10 keV的软X射线能段，两种脱膜剂制备的自支撑Al滤光片的透过率均高于90%，二者不存在较大差异，并与理论计算基本一致。在50～250 eV能段，AZ50XT脱膜剂制备的滤光片在其通带内的透过率达到53%，而聚乙烯醇脱膜剂制备的滤光片在其通带内的透过率为35%，由于Al滤光片表面氧化、脱膜剂残留等，实际测得的透过率比理论值要低。研究结果表明，AZ50XT光刻胶比聚乙烯醇更适合做脱膜剂来制备自支撑薄膜。

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Preparation of Self-supporting Al Filter

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Abstract: The self-supporting metal film is very important in the observation of X-ray and extreme ultraviolet bands. When the X-ray and extreme ultraviolet telescopes are used to observe the corresponding bands, the detector is usually disturbed by visible and infrared light and other stray light, they affect its detection accuracy and performance. Therefore, it is necessary to add filters in front of the detector, the filters are usually hundreds of nanometers of metal film. The preparation of self-supporting thin films is usually deposited on some special substrates, and then the substrate is removed to obtain the required self-supporting thin films. At present, there are two main methods for obtaining self-supporting films, one is substrate etching; the other is the release agent. The substrate etching process is complex, and the method of release agent is simple and easy to implement. Therefore, this study uses the method of release agent to prepare the self-supporting Al filter. The release agent used in the past is very easy to dissolve, such as NaCl, CsI, etc., and they are easy to bring defects to the film deposited on the release agent. In this paper, AZ50XT photoresist and polyvinyl alcohol are used as release agents. They have good film-forming properties, stable performance, and are good release agent materials. To make the self-supporting Al film have a regular shape and easy to test, it is necessary to prepare a nickel supporting frame. We copied the nickel frame pattern on clean Si wafer by two mask lithography, and then prepared the nickel frame by the micro-electroforming process. After preparation, the nickel frame was removed for use. The thickness of the nickel frame is about 60～70 μm, the inner diameter is 16 mm, the outer diameter is 28 mm. The interface between the nickel frame and the sample is smooth and easy to bond. In the experiment, AZ50XT photoresist and polyvinyl alcohol release layers were prepared on clean Si substrate by spin coater, and then dried on the heating table. After the sample was cooled to room temperature, placed it in the magnetron sputtering coating machine with background vacuum of 5.0×10^{-4} Pa to deposit Al film, the deposition thickness was 80 nm. The thickness of Al film was monitored by crystal oscillation film thickness meter, and the step measuring instrument was used to verify whether the thickness of Al film deposited was 80 nm. The test results show the average thickness of Al film is 80.75 nm, it was within the allowable error range. After the deposition of Al film was completed, the nickel frame has adhered to the surface of Al film by epoxy resin adhesive. After the epoxy resin adhesive was completely cured, the sample was put into acetone or deionized water for release, the self-supporting Al filter was obtained. The defects and pinholes on the surface of the filter were analyzed by scanning electron microscopy and a CMOS camera. The analysis results showed that the prepared Al filter surface was uniform and dense, with only a few pinholes. The optical properties of the prepared filter were characterized by Ultraviolet-visible spectrophotometer, soft X-ray transmittance test system and synchrotron radiation device. The transmittance of Al filters prepared by two kinds of release agents is higher than 0.02% in the ultraviolet band, and lower than 0.02% in the visible and infrared bands, they basically meet the requirements of use. The transmittance of Al filter prepared by AZ50XT photoresist is lower than that prepared by polyvinyl alcohol, because the preparation process of photoresist is advanced and the uniformity is better, the surface of the release layer prepared with it is flat and smooth, and there is almost no pinhole on the surface of Al film. The polyvinyl alcohol solution can produce gel during the preparation process, and there is a little gel residue even after repeated filtration, therefore, the prepared release layers have defects, resulting in a few pinholes on the surface of Al film. Compared with the polyimide-aluminum filters commonly used in space X-ray detection, the self-supporting single-layer Al filter has better ability to suppress visible and infrared light. In the 200～400 nm band, the Al filter without polyimide support has higher transmittance to ultraviolet light, so it is more conducive to extreme ultraviolet detection. The soft X-ray transmittance test system is built by our

laboratory, the system consists of an X-ray light tube (target is Ag, window is Be), a vacuum chamber, a translation stage and an SDD detector. Under the given tube voltage and tube current, the transmittance of the film in the soft X-ray band is characterized by measuring the ratio of the transmitted light intensity to the incident light intensity. Due to the existence of Be window in the X-ray tube, low-energy photons are shielded, and no low-energy photons are emitted from the X-ray tube. Therefore, the energy section below 1.6 keV can be ignored. The transmittance of the filter is higher than 90% in the energy range of 1.6 ~ 10 keV. In order to verify the reliability of the test results, we compare the test results with the theoretical calculation results. The transmittance curve is consistent with the theoretical results, which meets the application requirements. Finally, the transmittance of the filter in the energy range of 50~250 eV was measured by synchrotron radiation device. The test results show the highest transmittance of the prepared two filters in this energy range can reach 53% and 35%, respectively. Due to the oxidation of the Al filter surface and the residue of the release agent, the actually measured transmittance is much lower than the theoretical value.

Key words: Thin films; Self-supporting; Release agent; Soft X-ray; Extreme ultraviolet; Transmittance

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