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改进"yo-yo"Cs/O交替激活方法对GaAs光阴极稳定性影响

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摘 要:为提高激活后GaAs光阴极的稳定性,延长微光夜视器件的工作寿命,围绕Cs/O激活方法和衰减特性进行实验研究。通过对比传统"yo-yo"激活法和改进"yo-yo"激活法在Cs/O激活光电流、光谱响应以及光照衰减方面的差异,发现采用改进"yo-yo"激活法的GaAs光阴极光谱灵敏度更高且稳定性更好。利用四极质谱仪监测真空腔内残气成分和分压强变化,基于衰减模型拟合光电流实验曲线,求得不同残气成分对GaAs光阴极性能衰减影响的权重因子。结果表明水蒸气和二氧化碳的影响最大,甲烷和一氧化碳次之,氢气几乎不产生影响,而其它碳氢有机分子也会产生负面影响。总体看来,改进的"yo-yo"激活法对GaAs光阴极表面吸附含氧气体分子造成的性能衰减具有明显的改善效果,这将有助于提高微光夜视器件中GaAs光阴极的稳定性。

关键词:GaAs光阴极;Cs/O激活;残余气体;稳定性;光谱响应
中图分类号:TN223 **文献标识码**:A **doi**:10.3788/gzxb20225102.0212001

0 引言

在微光夜视器件中,作为感光核心部件的负电子亲和势(Negative Electron Affinity, NEA)GaAs光阴极主要是通过在GaAs材料表面进行Cs/O交替激活制备而成[1-2]。与制备过程相反的是光阴极的中毒,其宏观表现是光阴极发射性能的衰减,微观表现则是与表面Cs-O激活层的脱附,或者是H_2O、CO_2和CO等其它杂质气体吸附导致的Cs-O激活层结构的破坏。对于超高真空系统中激活后的GaAs光阴极,真空环境直接影响了阴极的稳定性,真空度越好,残气越少,阴极寿命则越长。研究者们纷纷对真空系统中常规残气对GaAs光阴极寿命的影响展开了研究,结果表明H_2、N_2和CH_4对阴极激活层几乎没有影响,含氧化学活性气体O_2、H_2O、和CO_2会造成阴极量子效率急剧衰减,而CO是否会影响阴极稳定性还存在争议[3-6]。关于残余气体在NEA GaAs光阴极表面的吸附和脱附机制,KURIKI M等在2011年的研究表明NEA GaAs光阴极的衰减主要与温度热脱附和残余气体与激活层的化学反应两方面因素有关,而在实际使用中光照也会导致阴极性能的衰减[7]。此外,KURIKI M等又在2013年对NEA GaAs光阴极进行了不同残气分压强下的进气衰减试验,通过最小二乘法参数寻优的方法对阴极衰减曲线进行拟合,得到了不同单一气体的衰减系数,表明O_2的影响最大,CO_2其次[8]。

由于GaAs光阴极是在超高真空中通过严格的激活工艺而制备得到,因此激活工艺的好坏会直接影响阴极的稳定性。激活工艺的效果取决于表面净化程度、激活源质量和激活步骤等方面因素,其中Cs/O交替激活方式对GaAs光阴极发射性能具有重要影响。本文利用自研的超高真空光阴极制备与多信息在线测控系统,探索能够提高GaAs光阴极量子效率并增强阴极稳定性的Cs/O激活方法,并对真空腔内部的残余气

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体进行实时监测，通过最小二乘法对阴极光电流衰减曲线进行拟合，得到不同成分和分压强下的残气对Cs/O激活GaAs光阴极稳定性的影响。

1 实验

激活实验中采用的GaAs阴极样品均从采用垂直梯度凝固法生长的直径2英寸（1英寸=2.54 cm）的同一p型GaAs单晶片解理得到，尺寸为11 mm×11 mm，厚度为350 μm，Zn掺杂浓度为1×10^{19} cm^{-3}。对解理的GaAs样品首先进行化学清洗，分别在四氯化碳、丙酮、无水乙醇、去离子水中各超声波清洗5 min，随后用浓度为40%的氢氟酸溶液腐蚀10 min，然后再用去离子水反复冲洗干净，最后用氮气吹干。将清洗吹干的样品送入真空度不低于1×10^{-7} Pa的超高真空系统进行高温加热净化，最高净化温度为600 ℃，保持时间为30 min，以去除表面残余的氧化物和碳污染杂质，获得原子级清洁表面[9]。待停止样品加热并冷却至室温后，开始进行Cs/O激活，激活装置示意如图1。激活光源采用12 V/100 W的卤素灯白光光源，Cs源和O源分别为镍管封装的铬酸铯和过氧化钡固态源，利用多信息在线测控系统调整施加的直流电流大小控制Cs和O放气量，并通过200 V高压收集光照射阴极产生的光电子，由计算机在线记录激活光电流的变化[10]。激活后采用633 nm单色红光照射Cs/O激活后的GaAs阴极样品[11]，由四极质谱议在线记录光电流衰减过程中真空腔内的残气变化，另外利用在线光谱响应测试系统对阴极样品衰减前后400～1 000 nm波段范围的光谱响应曲线进行测试。

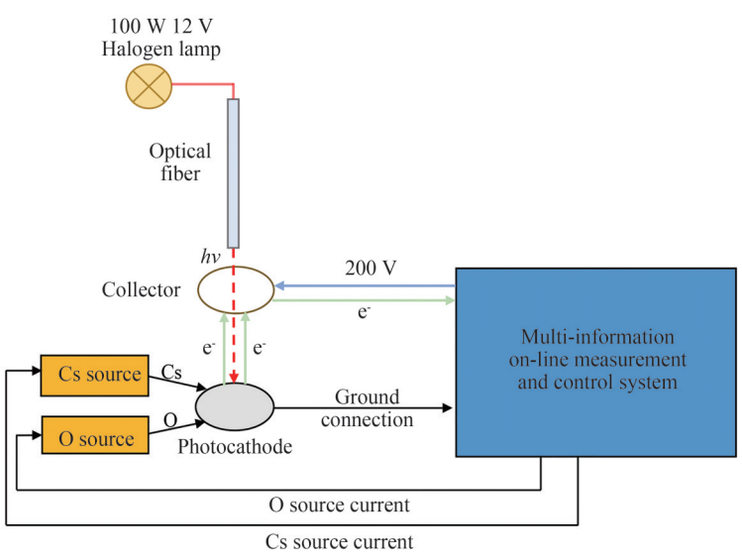

图1 GaAs光阴极在线Cs/O激活装置的示意

Fig. 1 Schematic of Cs/O on-line activation setup of GaAs photocathodes

在激活实验中，对GaAs阴极样品采用两种不同的Cs/O激活方法，分别记为激活实验1和激活实验2，具体激活方法如下：

激活实验1采用传统的“yo-yo”激活法[12]。保持Cs源为持续开启，待首次进Cs后光电流达到峰值，等待Cs稍微过量，光电流下降至峰值的85%，开启O源，等光电流出现新的峰值时关闭O源，待光电流上升至新的峰值，再下降至新峰值的85%时开启O源，如此反复直到光电流峰值不再增加。

激活实验2采用改进的“yo-yo”激活法。与激活实验1一样，保持Cs源为持续开启，待首次进Cs后光电流达到峰值后，等待Cs完全过量，光电流下降至光电流降速变缓时，开启O源，等光电流达到新的峰值时，关闭O源，待光电流再次下降至光电流降速变缓时，开启O源，如此反复直到光电流峰值不再增加。

2 结果与分析

2.1 光阴极激活过程

采用两种不同Cs/O激活方法制备的三组GaAs光阴极样品（即样品1-1和1-2，样品2-1和2-2，样

品3-1和3-2)的光电流如图2所示。表1给出了图2中样品1-1和1-2这两个同一时期、同一单晶片样品激活中的过程参量。可以发现，首次进Cs后光电流均是在18 min左右开始增长，且光电流到达第一次Cs峰的时间和第一个Cs峰的光电流都相差不大，这说明一开始Cs的沉积量大致相同。从交替次数和交替周期上看，激活中采用Cs完全过量的改进“yo-yo”激活法可以减少Cs/O交替次数，且Cs/O平均交替周期时间得到延长。从最终光电流上看，改进的“yo-yo”激活法相比传统的“yo-yo”激活法可获得更高的光电流，提高了18%。然而，由于改进“yo-yo”激活法需要等待Cs完全过量，因此总的激活时间要大于传统的“yo-yo”激活法。

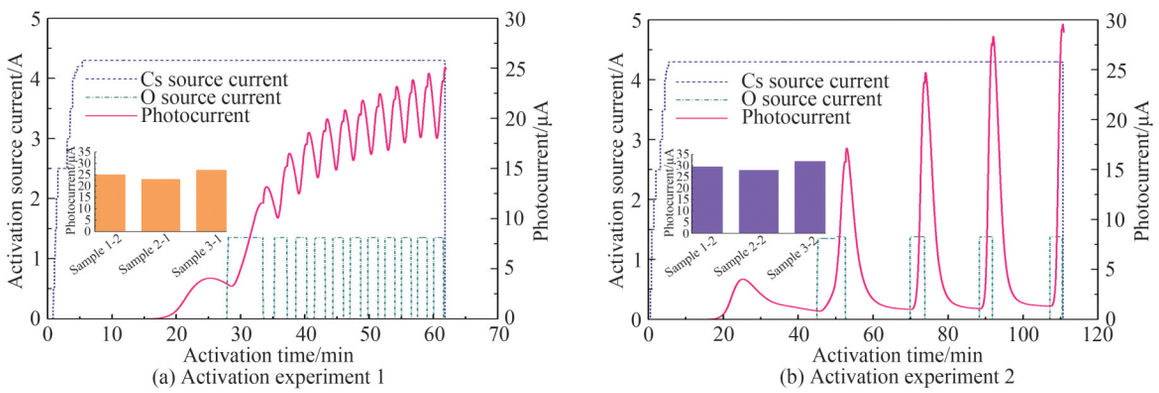

图2　采用不同Cs/O激活方法的GaAs光阴极光电流变化曲线

Fig. 2　Photocurrent change curves of GaAs photocathodes with different Cs/O activation methods

表1　采用不同Cs/O激活方法的GaAs光阴极激活过程参数

Table 1　Activation process parameters of GaAs photocathodes with different Cs/O activation methods

Experiment	First peak time/min	First peak photocurrent/μA	Alternation times	Average time per alternation/min	Final photocurrent/μA	Total activation time/min
1	24.8	3.95	11	2.5	25.1	62
2	25.2	4.02	4	16.2	29.5	111

2.2　光阴极激活后的衰减特性

激活后采用633 nm单色红光连续照射下的GaAs光阴极光电流的变化趋势如图3。可以看出，改进“yo-yo”激活法相比传统的“yo-yo”激活法具有更长的工作寿命。式(1)给出了在衰减过程中任意时刻某一确定入射光功率下衰减光电流的变化趋势模型，该模型与真空度、温度和衰减时间相关[7]。

$$i(P,t)=c_1\cdot\exp\left[-P\cdot t/c_2-v\cdot t\cdot\exp(-E/kT)\right] \tag{1}$$

式中，c_1为初始衰减系数，c_2为与真空度有关的衰减系数，t为衰减时间，P为衰减过程中的真空度，v为一比例常数，E为激活层在GaAs光阴极表面的结合能(单位为eV)，k为玻尔兹曼常数，T为热力学温度。这里假定

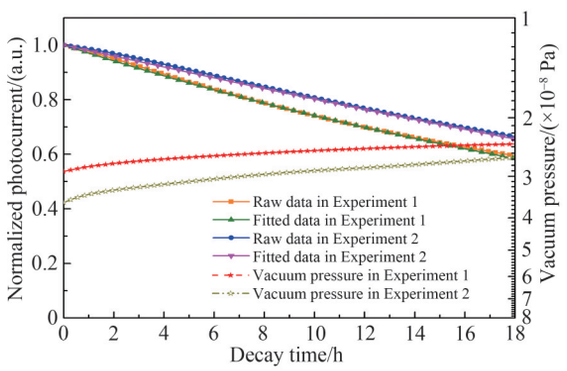

图3　采用不同Cs/O激活方法的GaAs光阴极的光电流衰减曲线

Fig. 3　Decay photocurrent curves of GaAs photocathodes with different Cs/O activation methods

在室温下的衰减过程表面激活层不会发生脱附现象，而只会发生残气的吸附[13]，那么，在温度设定为常温300 K情况下，可以将第二项整体看为一个只与时间相关的衰减项，衰减系数为c_3，也就是说式(1)可转换为

$$i(P,t)=c_1\cdot\exp(-P\cdot t/c_2-t/c_3) \tag{2}$$

利用式(2)对图3中采用不同Cs/O激活方法的GaAs光阴极样品的光电流衰减实验数据进行拟合，获得的拟合参数如表2。可以看出，即使激活实验1中的真空度要好于激活实验2中的真空度，然而采用改进“yo-yo”激活法的GaAs阴极样品的衰减系数c_2更大，大约是采用传统“yo-yo”激活法的3倍，而两者的衰减系数c_2大致相当，这就意味着采用改进“yo-yo”激活法的GaAs阴极样品的稳定性更好，对残气吸附的免疫性更强。衰减前后这两种采用不同Cs/O激活方法得到的GaAs光阴极的光谱响应曲线如图4。可见，激活后改进“yo-yo”激活法得到的光谱响应灵敏度相比传统的“yo-yo”激活法更高，且衰减后的灵敏度下降幅度更小，尤其是近红外波段两者的差距更为明显。在长波800 nm处，采用改进“yo-yo”激活法的阴极样品灵敏度在连续光照18 h衰减后下降了37%，而采用传统“yo-yo”激活法的阴极样品灵敏度则下降了63%。

表2 采用不同Cs/O激活方法的GaAs光阴极光电流衰减曲线拟合参数

Table 2 Fitted parameters of decay curves of GaAs photocathodes with different Cs/O activation methods

Experiment	c_1/μA	c_2/(Pa•s)	c_3/s
1	0.1692	$1.733\,8\times10^7$	$1.204\,9\times10^5$
2	0.2464	$5.144\,8\times10^7$	$1.546\,1\times10^5$

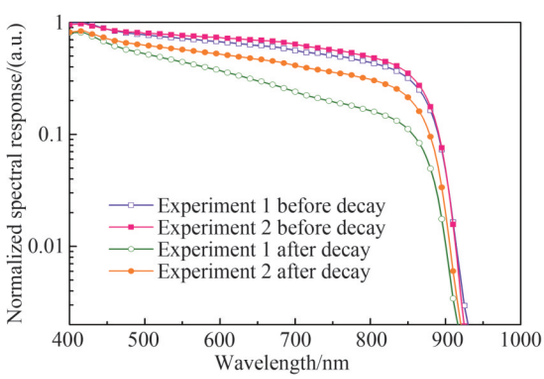

图4 采用不同Cs/O激活方法的GaAs光阴极衰减前后的光谱响应曲线

Fig. 4 Spectral response curves of GaAs photocathodes with different Cs/O activation methods before and after decay

2.3 残余气体对光阴极衰减的影响

真空度的变化来源于真空腔中残气的变化，以及残气和表面激活层相互作用的变化，因此在衰减过程中各种残气成分和分压强的变化会对Cs/O激活的GaAs光阴极性能产生影响。图5给出了传统的“yo-yo”激活法和改进“yo-yo”激活法得到的GaAs光阴极在衰减初始时刻的真空腔内残余气体的成分和分压强。可见，在衰减过程的初始时刻，对于传统的“yo-yo”激活法和改进的“yo-yo”激活法，两者真空腔内的残气成分和分压强非常相近，存在的主要质量数均为2、15、16、18、26、27、28、29、39、41、43和44。图6给出了采用这两种激活方法得到的GaAs光阴极在衰减过程中真空腔内主要残气质量数的分压强变化曲线，可见随着时间的变化，各种残气的分压强都在降低。

考虑衰减过程中真空度的变化是由各种吸附气体分子的引入而导致的，则式(2)可以写为

$$i(P,t)=c_1\cdot\exp\left[-\left(\sum_{i=1}^{N}a_i\cdot P_i\cdot t\right)/c_2-t/c_3\right] \tag{3}$$

式中，i代表气体分子质量数的序号，N是吸附气体分子的种数，a_i代表吸附气体分子的影响权重因子(i=1～12分别代表质量数为2、15、16、18、26、27、28、29、39、41、43和44的气体分子)，而P_i代表不同吸附气体分子质量数的分压强。由式(3)可知，权重因子值越小，表示残气分子对阴极表面的负面作用越小。

通过式(3)，利用最小二乘法参数寻优的方法获得不同残气成分对GaAs光阴极光电流衰减影响的权重因子，如表3。可以看出，质量数为18、27、39和44的气体分子对光电流衰减影响最大，紧接着是质量数为

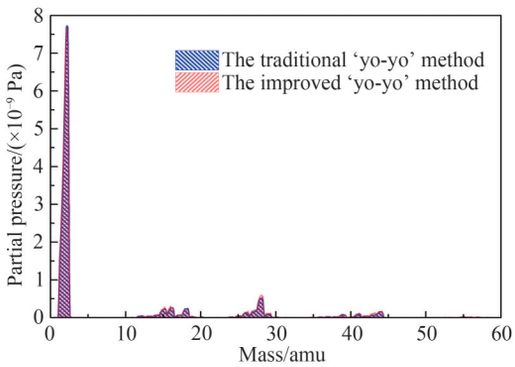

图5　采用不同Cs/O激活方法的GaAs光阴极在衰减初始时刻的残气成分和分压强

Fig. 5　Components and partial pressure of residual gases at the initial moment of decay for GaAs photocathodes with different Cs/O activation methods

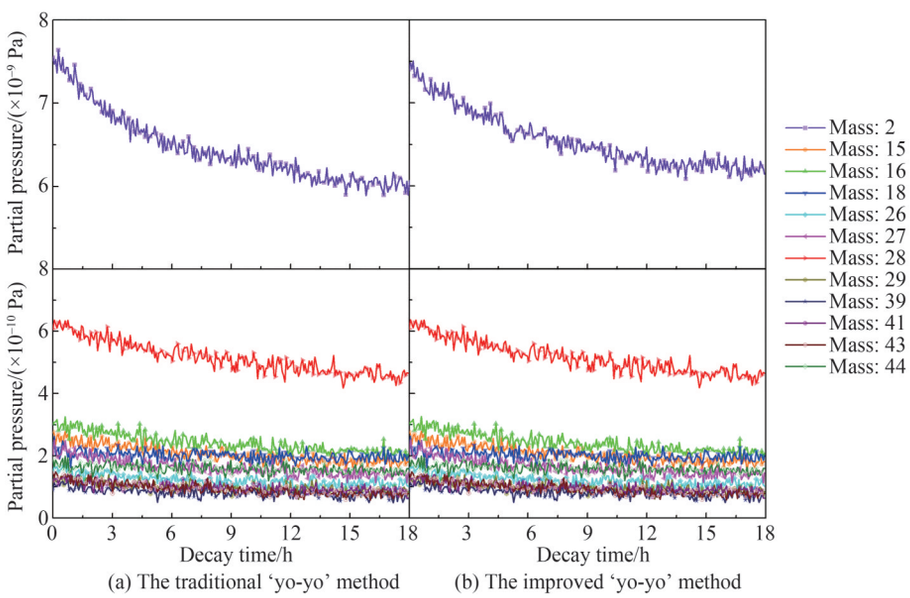

图6　采用不同Cs/O激活方法的GaAs光阴极在衰减过程中的残气分压强变化曲线

Fig. 6　Partial pressure variation curves of residual gases in the decay processes for GaAs photocathodes with different Cs/O activation methods

表3　不同残气成分对GaAs光阴极光电流衰减影响的权重因子

Table 3　Weight factors of influence of different residual gas components on photocurrent decay of GaAs photocathodes

Mass number	Experiment 1	Experiment 2
a_1(2)	0.003 4	0.001 8
a_2(15)	0.192 8	0.161 8
a_3(16)	0.190 2	0.180 0
a_4(18)	0.788 7	0.592 4
a_5(26)	0.252 1	0.208 8
a_6(27)	0.472 1	0.225 0
a_7(28)	0.145 0	0.117 5
a_8(29)	0.074 4	0.058 8
a_9(39)	0.324 6	0.161 2
a_{10}(41)	0.003 3	0.000 6
a_{11}(43)	0.194 3	0.184 4
a_{12}(44)	0.451 9	0.227 9

15、16、26、28和43，而质量数为2、29和41的气体分子几乎不存在影响。通过比较可以发现，改进“yo-yo”激活法相比传统的“yo-yo”激活法对真空腔内残气分子吸附导致的光阴极性能衰减具有更好的免疫能力。由四极质谱参考手册可知[14]，质量数为2的气体是氢气，质量数为15和16的气体是甲烷电离产生的次峰和主峰，质量数为18的气体主要是水蒸气，质量数为28的气体主要是一氧化碳，质量数为44的气体主要是二氧化碳，质量数包括26、27、28的气体为碳氢有机气体分子A(如乙烯C_2H_4)，质量数包括27、28和29的气体为碳氢有机气体分子B(如丙烷C_3H_8)，而质量数包括39、41、43的气体为另一种碳氢有机气体分子C(如丁烷C_4H_{10})。由拟合结果可知，对于GaAs光阴极激活真空腔内的无机物气体分子，水蒸气和二氧化碳的影响最大，甲烷和一氧化碳次之，而氢气几乎不产生影响。另外，一些碳氢有机气体分子也会对GaAs光阴极的性能衰减产生比较大的负面作用。

此外，为了验证改进“yo-yo”激活法对氧气分子吸附免疫性的改善效果，通过对激活用的固态氧源施加不同大小的电流，在激活腔内通入了不同分压强的氧气，测试了采用这两种激活方法的GaAs光阴极样品在633 nm单色红光下的光电流衰减情况，如图7所示。结果表明，随着氧气分压强的增大，光电流的衰减速率会增大。然而，对于采用改进“yo-yo”激活法的GaAs光阴极样品而言，其光电流衰减速率要明显低于采用传统“yo-yo”激活法的GaAs光阴极样品。当氧气分压强在6×10^{-10} Pa时，采用改进“yo-yo”激活法的光电流衰减速率相比传统“yo-yo”激活法减缓了75%，而当氧气分压强为1.2×10^{-9} Pa时，采用改进“yo-yo”激活法的光电流衰减速率相比传统“yo-yo”激活法减缓了54%。

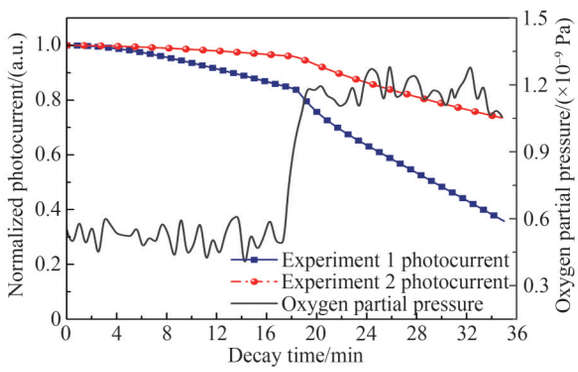

图7 采用不同Cs/O激活方法的GaAs阴极光电流随氧气分压强的变化

Fig. 7 Variation of photocurrent with oxygen partial pressure for GaAs photocathodes with different Cs/O activation methods

3 结论

本文利用超高真空光阴极制备与多信息在线测控系统，开展了不同Cs/O交替激活方法对GaAs光阴极稳定性的实验研究。结果表明，对于Cs/O激活的GaAs光阴极，改进“yo-yo”激活法能够获得更高的光电流和光谱响应，最重要的是，在衰减稳定性方面要明显优于传统的“yo-yo”激活法。此外，通过四极质谱仪监测真空腔内残气变化，利用光阴极衰减模型拟合光电流衰减曲线，发现水蒸气和二氧化碳的影响最大，甲烷和一氧化碳次之，氢气几乎不产生影响，而其它碳氢有机气体分子会有负面影响。相比传统“yo-yo”激活法，改进的“yo-yo”激活法对GaAs光阴极吸附含氧气体分子的免疫能力具有很好的改善效果。本文实验结果对于探索提高以GaAs光阴极为核心的微光夜视器件寿命的方法具有一定的参考价值。

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Effect of Improved ′yo-yo′ Cs/O Alternate Activation Method on Stability of GaAs Photocathode

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Abstract: In modern low-light-level night vision devices, the negative-electron-affinity GaAs photocathode, as the photosensitive core component, is usually prepared by alternating Cs/O activation on the cleaned GaAs surface. Meanwhile, the stability of GaAs photocathode is directly affected by the quality of Cs/O activation process and the residual gases in the vacuum environment. In order to improve the stability of GaAs photocathodes after activation and prolong the operating lifetime of the low-light-level night vision devices, experimental researches were executed from the perspectives of Cs/O activation method and decay characteristic with the aid of the self-developed ultra-high vacuum photocathode preparation and multi-information on-line measurement and control system. Two different Cs-O activation methods, namely the traditional ′yo-yo′ method and the improved ′yo-yo′ activation method were performed on the p-type GaAs (100) substrates grown by the vertical gradient freeze method. During the traditional ′yo-yo′ activation process, the O source was introduced when the photocurrent dropped to 85% of the previous peak and the Cs source was kept continuously with a slight overdose, and then the O source was closed when the photocurrent reached a new peak. While in the process of the improved ′yo-yo′ activation, the Cs source was maintained continuously and the O source was introduced when the photocurrent dropped to the minimum with the complete Cs overdose in the Cs/O alternate activation cycles, and the O source was also closed when the photocurrent reached a new peak. The Cs/O activation results show that the improved ′yo-yo′ activation method with less alternate activation cycles can obtain a higher photocurrent peak and a higher spectral sensitivity than the traditional one with more alternate activation cycles. In addition, the photocurrent decay results under the long-time illumination of 633 nm

demonstrate that the GaAs photocathode with the improved ′yo-yo′ activation method can achieve a longer operating lifetime and a better stability than the traditional one. After 18 hours of decay under continuous illumination, the GaAs cathode sample using the improved ′yo-yo′ activation method exhibited a smaller drop of spectral sensitivity, especially in the near-infrared waveband. obvious. At 800 nm, the sensitivity of the cathode sample using the improved ′yo-yo′ activation method is decreased by 37%, while that of the cathode sample using the traditional one is decreased by 63%. Furthermore, by measuring the changes of components and partial pressure of residual gases in the activation chamber with the quadrupole mass spectrometry, and through fitting the photocurrent decay data based on the decay model related to the vacuum pressure and the partial pressure of residual gases, the weight factors of influence of different residual gases on the decay of photocathode performance were obtained. The fitting results show that the water vapor and carbon dioxide have the greatest impact, followed by methane and carbon monoxide, while hydrogen has almost no impact, and other hydrocarbon organic molecules also have the negative impact. By comparison of weight factors of residual gas components, it is found that the improved ′yo-yo′ activation method has better immunity to the degradation of GaAs photocathode performance caused by the adsorption of residual gas molecules in the vacuum chamber than the traditional one. In order to verify the improvement effect of the improved ′yo-yo′ activation method on the immunity of oxygen molecules, the photocurrent decay cases of GaAs photocathode samples activated by the two different activation methods, were tested under the illumination of 633 nm red light by introducing oxygen with different partial pressures into the activation chamber. The results show that the decay rate of photocurrent increases with the increase of oxygen partial pressure. Whereas, the photocurrent decay rate of GaAs photocathode sample with the improved ′yo-yo′ activation method is significantly lower than that of GaAs photocathode sample with the traditional ′yo-yo′ activation method. When the partial pressure of oxygen is 6×10^{-10} Pa, the photocurrent decay rate of the improved ′yo-yo′ activation method is reduced by 75% compared with that of the traditional one, and when the oxygen partial pressure is 1.2×10^{-9} Pa, the photocurrent decay rate of the improved ′yo-yo′ activation method is reduced by 54% compared with that of the traditional one. In general, the improved ′yo-yo′ activation method can obviously delay the performance degradation rate of GaAs photocathode caused by the adsorption of oxygen-containing gas molecules on the surface, which will help to improve the stability of GaAs photocathode in low-light-level night vision devices.

Key words: GaAs photocathode; Cs/O activation; Residual gas; Stability; Spectral response

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