解决方案|ICP-OES 法测定半导体芯片清洗液中重金属

半导体作为现代电子工业发展的基础和支撑，在电子工业的应用和所选用的材料也越来越广泛。半导体芯片制成环节所用清洗液有蚀刻后清洗液，用于对晶圆表面清除残留物以及对 TiN 腐蚀进行清洗，POST CMP 清洗液，用于解决化学机械研磨后晶圆表面缺陷问题，以保证良率。随着通信技术的快速发展，半导体芯片清洗液的关注度及可靠性引起了越来越多的关注。本文采用 Quantima 电感耦合等离子发射光谱仪建立了清洗液中的相关金属元素检测解决方案，供相关人员参考。

实验部分

仪器设备

Quantima 电感耦合等离子发射光谱仪

实验条件

Element	λ mm	Power W	Neb L/min	Height mm	Plasma L/min	Aux L/min	Pump RPM	PMT V	Int s
Ca	393.366	1000	0.8	8.0	14.0	0.5	11	550	0.5
K	766.490	900	0.8	8.0	14.0	0.5	11	650	0.5
Zn	213.856	1000	0.8	8.0	14.0	0.5	11	600	0.5
Fe	259.940	1000	0.8	8.0	14.0	0.5	11	600	0.5
Mg	279.553	1000	0.8	8.0	14.0	0.5	11	600	0.5

Pb	220.353	1000	0.8	8.0	14.0	0.5	11	720	0.5
Na	589.592	1000	0.8	8.0	14.0	0.5	11	650	0.5
Sn	283.999	1000	0.8	8.0	14.0	0.5	11	550	0.5
Co	228.616	1000	0.8	8.0	14.0	0.5	11	550	0.5
Cu	324.754	1000	0.8	8.0	14.0	0.5	11	550	0.5
Mn	257.610	1000	0.8	8.0	14.0	0.5	11	500	0.5
Cr	283.563	1000	0.8	8.0	14.0	0.5	11	650	0.5
Ni	221.647	1000	0.8	8.0	14.0	0.5	11	600	0.5

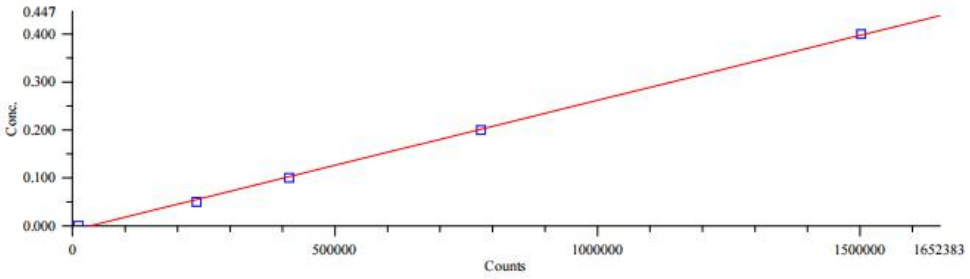
样品处理

准确量取 6.25mL 样品于 50mL 瓷坩埚中，电热板 120℃将水分蒸干，蒸干后转至可调温电炉上碳化，碳化后冷却至室温，加盖，转移至马弗炉中 500℃ 4h，冷却至室温后取出，开盖，加入 1.0mL 硝酸溶解残渣，电热板 120℃将酸赶至 0.2mL 取下冷却至室温，用去离子水转移至 25mL 容量瓶中，用去离子水冲坩埚至少 3 次，定容至刻度后摇匀备用。

实验结果

标准曲线

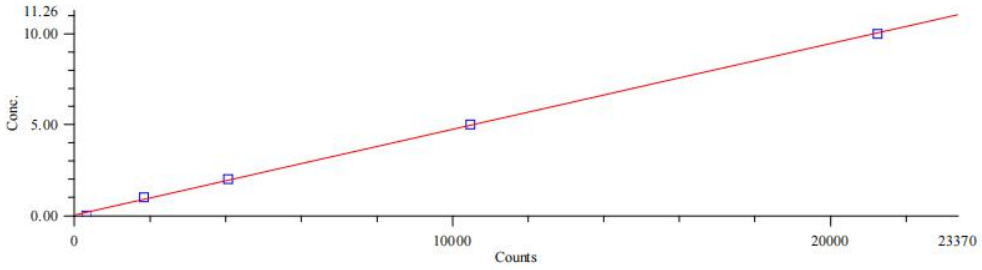
Ca II 393.366 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	1502170	0.4000	0.3979	-0.533
Standard 2	778155	0.2000	0.2016	0.781
Standard 3	412231	0.1000	0.1023	2.35
Standard 4	236176	0.05000	0.05461	9.23
Blank	11173.9	0	-0.006393	***

Calibration	C0	C1	R
Coefficients	-0.00942	2.711e-7	0.9993

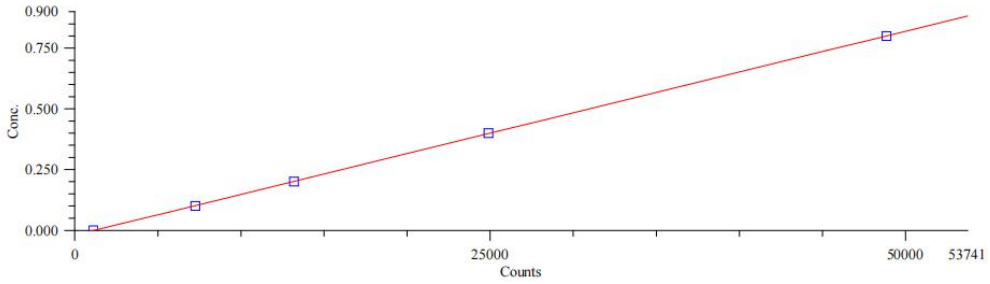
K I 766.490 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	21245.1	10.00	10.04	0.384
Standard 2	10467.2	5.000	4.960	-0.802
Standard 3	4053.83	2.000	1.938	-3.10
Standard 4	1836.06	1.000	0.8929	-10.7
Blank	303.515	0	0.1708	***

Calibration	C0	C1	R
Coefficients	0.027788	4.712e-4	0.9993

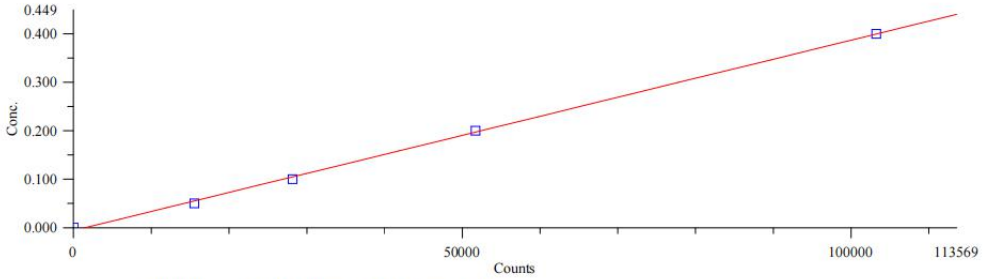
Zn I 213.856 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	48855.3	0.8000	0.8005	0.0592
Standard 2	24886.8	0.4000	0.3979	-0.529
Standard 3	13191.5	0.2000	0.2014	0.721
Standard 4	7251.49	0.1000	0.1017	1.67
Blank	1110.81	0	-0.001472	***

Calibration	C0	C1	R
Coefficients	-0.02013	1.680e-5	1.000

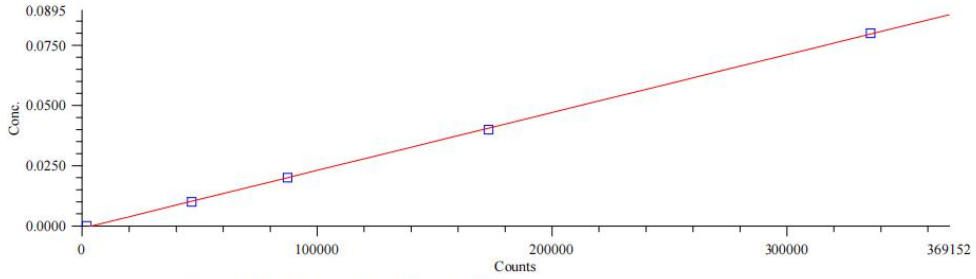
Fe II 259.940 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	103244	0.4000	0.3995	-0.123
Standard 2	51692.0	0.2000	0.1970	-1.49
Standard 3	28159.9	0.1000	0.1046	4.58
Standard 4	15517.1	0.05000	0.05492	9.84
Blank	3.12939	0	-0.006019	***

Calibration	C0	C1	R
Coefficients	-0.00603	3.928e-6	0.9991

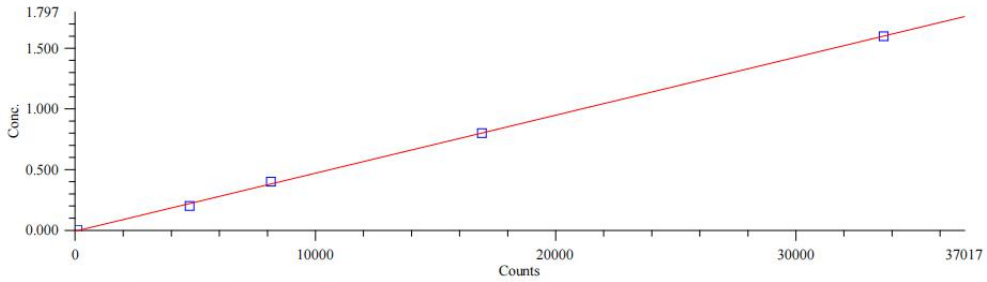
Mg II 279.553 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	335593	0.08000	0.07966	-0.425
Standard 2	172960	0.04000	0.04059	1.46
Standard 3	87421.9	0.02000	0.02003	0.172
Standard 4	46605.6	0.01000	0.01023	2.28
Blank	1920.27	0	-0.0005084	***

Calibration	C0	C1	R
Coefficients	-9.70e-4	2.403e-7	0.9998

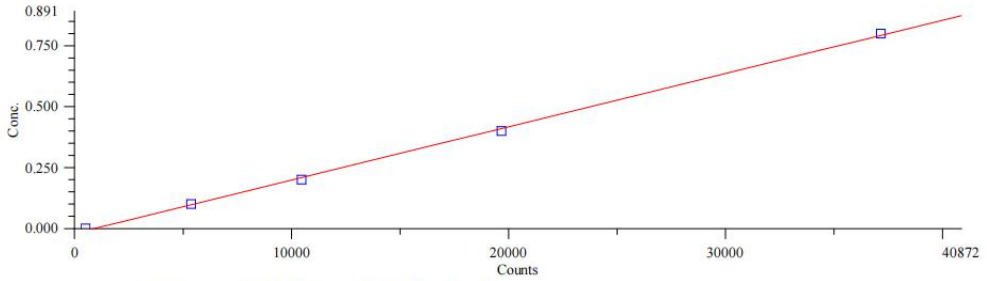
Pb II 220.353 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	33651.9	1.600	1.601	0.0665
Standard 2	16926.3	0.8000	0.8013	0.164
Standard 3	8147.81	0.4000	0.3816	-4.61
Standard 4	4757.91	0.2000	0.2195	9.73
Blank	97.1668	0	-0.003395	***

Calibration	C0	C1	R
Coefficients	-0.00804	4.782e-5	0.9995

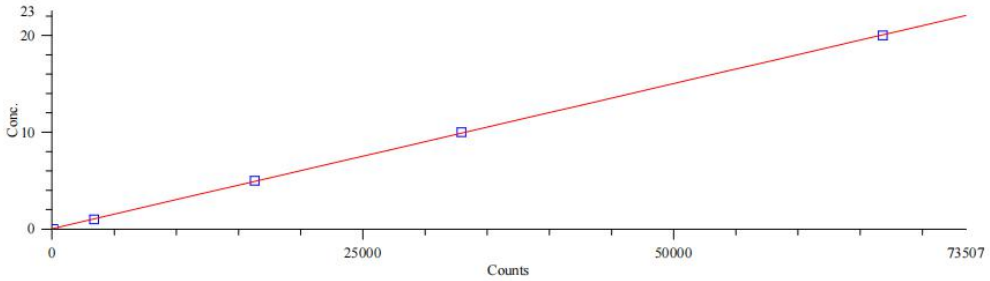
Na I 589.592 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	37156.4	0.8000	0.7926	-0.919
Standard 2	19673.2	0.4000	0.4102	2.56
Standard 3	10458.1	0.2000	0.2087	4.34
Standard 4	5367.32	0.1000	0.09733	-2.67
Blank	511.289	0	-0.008888	***

Calibration	C0	C1	R
Coefficients	-0.02007	2.187e-5	0.9992

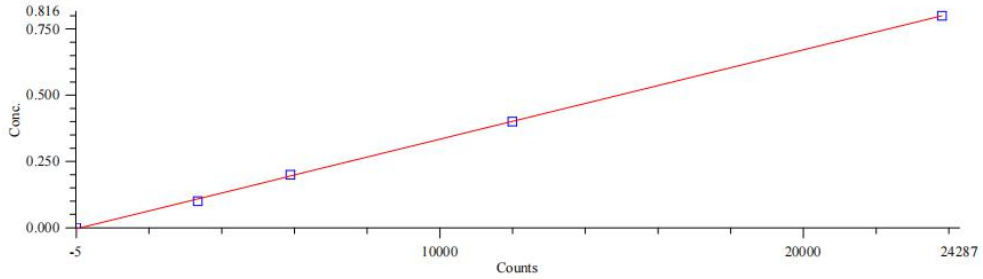
Sn I 283.999 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	66824.4	20.00	20.07	0.325
Standard 2	32916.0	10.00	9.901	-0.990
Standard 3	16306.4	5.000	4.922	-1.55
Standard 4	3388.32	1.000	1.050	5.01
Blank	90.3448	0	0.06151	***

Calibration	C0	C1	R
Coefficients	0.034430	2.998e-4	0.9999

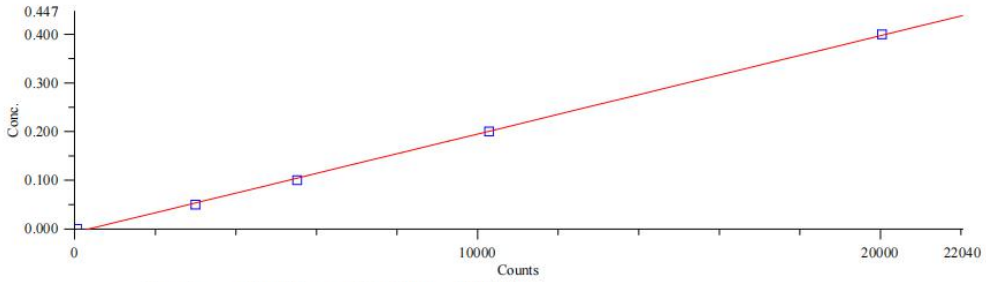
Co II 228.616 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	23810.7	0.8000	0.7996	-0.0462
Standard 2	11993.3	0.4000	0.4008	0.202
Standard 3	5888.80	0.2000	0.1948	-2.60
Standard 4	3343.71	0.1000	0.1089	8.90
Blank	-5.41907	0	-0.004129	***

Calibration	C0	C1	R
Coefficients	-0.00395	3.375e-5	0.9997

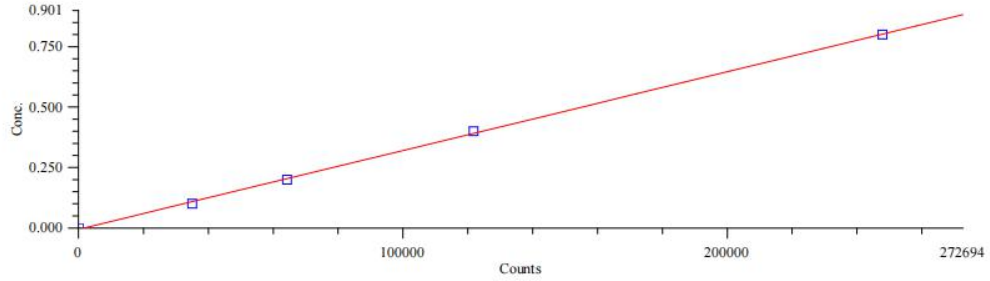
Cu I 324.754 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	20036.3	0.4000	0.3979	-0.514
Standard 2	10287.7	0.2000	0.2008	0.377
Standard 3	5520.32	0.1000	0.1043	4.32
Standard 4	2997.72	0.05000	0.05330	6.59
Blank	50.5923	0	-0.006317	***

Calibration	C0	C1	R
Coefficients	-0.00734	2.023e-5	0.9993

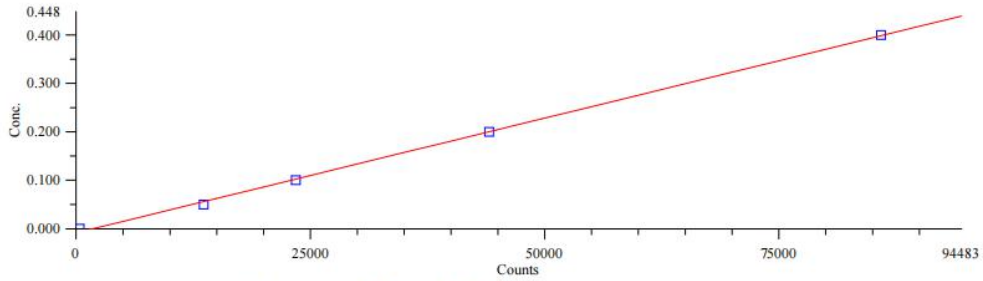
Mn II 257.610 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	247904	0.8000	0.8022	0.273
Standard 2	121758	0.4000	0.3912	-2.21
Standard 3	64232.5	0.2000	0.2037	1.86
Standard 4	34991.2	0.1000	0.1084	8.44
Blank	18.5699	0	-0.005510	***

Calibration	C0	C1	R
Coefficients	-0.00557	3.258e-6	0.9995

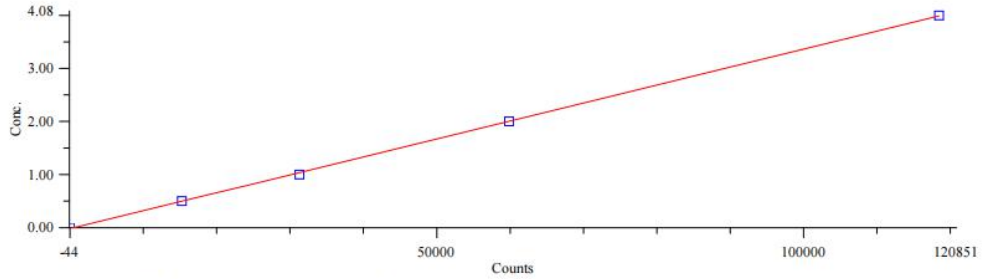
Cr II 283.563 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	85894.0	0.4000	0.3983	-0.417
Standard 2	44067.0	0.2000	0.2002	0.0920
Standard 3	23444.3	0.1000	0.1025	2.49
Standard 4	13596.5	0.05000	0.05584	11.7
Blank	366.635	0	-0.006839	***

Calibration Coefficients	C0	C1	R
	-0.00858	4.737e-6	0.9991

Ni II 221.647 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	118482	4.000	3.989	-0.277
Standard 2	59863.3	2.000	2.004	0.215
Standard 3	31281.9	1.000	1.037	3.66
Standard 4	15257.1	0.5000	0.4941	-1.18
Blank	-43.9482	0	-0.02394	***

Calibration Coefficients	C0	C1	R
	-0.02245	3.386e-5	0.9998

样品检测结果

单位：mg/kg

样品	Ni	Cr	Mn	Cu	Co	Sn	Na	Pb	Mg	Fe	Zn	K	Ca
	未检出	0.15	未检出	0.056	未检出	未检出	0.84	0.076	0.061	0.18	0.64	未检出	0.15

实验总结

电感耦合等离子体原子发射光谱法测试金属含量具有灵敏度高、检出限低、干扰少、线性范围宽等优点，且分析过程简单，可以满足快速高效的分析测定要求。本文通过选择合适的操作条件和分析谱线测得经过预处理的清洗液试样中的金属元素，获得客户的认可。该方法可供相关人员参考。