镨钕金属化学分析方法

碳、铁、钼、铝、硅和镨含量的测定

火花放电原子发射光谱法

精密度实验报告

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标样准备

共收到起草单位的20块实验用样品，其中19块用于曲线绘制。

如表1所示；

表1 用于绘制工作曲线的样品

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 名称 | C | Fe | Mo | Al  | Si  | Pr |
| 9# | 0.011  | 0.090  | / | 0.021  | 0.018  | 16.890  |
| 27 | 0.064  | 0.323 | 0.016  | 0.022  | 0.049  | 18.721  |
| 538 | 0.038  | 0.467  | 0.010  | 0.037  | 0.043  | 19.082  |
| 3-55 | 0.107  | 0.180  | / | 0.007  | 0.020  | 19.430  |
| 2-1 | 0.159  | 1.083  | / | 0.008  | 0.029  | 19.712  |
| 4-187 | 0.010  | 0.017  | / | 0.004  | / | 20.715  |
| 11-201 | 0.037  | 0.214  | / | 0.041  | 0.105  | 22.882  |
| 20-193 | 0.012  | 0.034  | / | 0.008  | 0.011  | 23.127  |
| 503 | 0.016  | 0.204  | 0.006  | 0.067  | 0.075  | 23.485  |
| 22-114 | 0.023  | 0.049  | / | 0.006  | 0.009  | 23.690  |
| PNMo-8 | 0.021  | 0.146  | 0.061  | 0.015  | 0.018  | 23.725  |
| PNMo-7 | 0.038  | 0.414  | 0.054  | 0.013  | 0.033  | 23.917  |
| PNMo-9 | 0.023  | 0.288  | 0.064  | 0.030  | 0.032  | 24.114  |
| 9-201 | 0.080  | 0.168  | / | 0.006  | 0.011  | 24.682  |
| 19# | 0.034  | 0.325  | / | 0.177  | 0.138  | 25.725  |
| NCS203036 | 0.090 | 1.480 | 0.0078 | 0.0087 | 0.045 | 30.82 |
| 9-200 | 0.473  | 1.261  | 0.0009  | 0.026  | 0.036  | 33.203  |
| NCS203035 | 0.046 | 0.56 | 0.032 | 0.019 | 0.023 | 20.85 |
| 539 | 0.086  | 0.275  | 0.026  | 0.060  | （0.080） | 18.798  |
| **曲线范围** | **0.010-0.473** | **0.017-1.480** | **0.0009-0.064** | **0.004-0.177** | **0.009-0.138** | **16.890-33.203** |
| **产品标准** | **<=0.05** | **<=0.3** | **<=0.1** | **<=0.1** | **<=0.05** | **18.0-32.0** |

1. 仪器设备
2. 钢研纳克HSRE 1000型稀土金属快速分析仪
3. 仪器主要工作条件：实验采用一次积分，充气、预燃、积分各阶段具体参数见表2。静止流量0.07L/min，分析流量9L/min。实验过程中选用的分析线对见表3. 分析线对选择主要考虑谱线干扰、曲线线性和测试结果精密度几个因素，选择干扰较少，拟合曲线相关系数接近于1且测试结果相对标准偏差较小的分析线对。。

表2实验条件选择

|  |  |  |  |
| --- | --- | --- | --- |
| 实验阶段 | 时间/s | 频率/Hz | 电压/V |
| 吹扫 | 10 | / | / |
| 预燃 | 10 | 500 | 380 |
| 燃烧 | 6 | 500 | 190 |

表3 实验选用的分析线对

|  |  |  |
| --- | --- | --- |
| 元素 | 分析谱线/nm | Nd参比谱线/nm |
| C | 193.09 | 193.4 |
| Fe | 239.5 | 242.9 |
| Mo | 281.6 | 275.8 |
| Al | 396.1 | 289.2 |
| Si | 288.1 | 289.2 |
| Pr | 405.4 | 406.0 |

1. 绘制校准曲线

用BD4800砂带机和40目砂带制样，将样品一个面打磨至表面平整且有方向一致的清晰纹路。磨好的表面不要用手触摸或用其他物品擦拭，直接放在激发台上激发。在选定的实验条件下激发表2所列样品，每个样品激发4个点，以各元素分析线强度与参比线强度比值（相对强度）为横坐标，以各元素含量与Nd基体含量比值（相对含量）为纵坐标，绘制各元素校准曲线。由于火花发射光谱法为持久曲线法，为便于后期使用绘制的曲线分析样品，为每个元素选取接近于曲线上限和曲线下限附近的样品作为强度校正样品。本方法选取9-200、PNMo-8、503、9#等4块样品作为各元素的标准化样品（高低标），其参考含量见表4。

表4 各元素标准化样品（高低标参考含量）

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 名称 | C | Fe | Mo | Al  | Si  | Pr |
| 9# | 0.011  | 0.090  | / | 0.021  | 0.018  | 16.890  |
| 503 | 0.016  | 0.204  | 0.006  | 0.067  | 0.075  | 23.485  |
| PNMo-8 | 0.021  | 0.146  | 0.061  | 0.015  | 0.018  | 23.725  |
| 9-200 | 0.473  | 1.261  | 0.0009  | 0.026  | 0.036  | 33.203  |

1. 精密度实验

选取9#，503，PNMo-8，PNMo-9，19#，502，539，2-1，9-200九块样品作为精密度实验样品，依次编号为样品1~样品9。用表4中选取的标准化样品对绘制好的曲线进行全局校正后，用绘制的校准曲线分别对9个样品进行11次独立测定，结果如表5~13所示。测试结果中C、Fe、Mo、Al、Si保留至小数点后4位，Pr保留至小数点后3位，相对标准偏差RSD保留至小数点后2位，数值修约按照GB/T 8170的规定执行。

表5 9# 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0113  | 0.0804  | 0.0048  | 0.0198  | 0.0187  | 16.975  |
| ASD | 0.0008  | 0.0010  | 0.0010  | 0.0009  | 0.0002  | 0.0429  |
| RSD | 6.87  | 1.26  | 20.35  | 4.77  | 1.25  | 0.25  |
| 1 | 0.0127  | 0.0803  | 0.0053  | 0.0190  | 0.0187  | 17.036  |
| 2 | 0.0118  | 0.0819  | 0.0043  | 0.0190  | 0.0191  | 16.901  |
| 3 | 0.0098  | 0.0815  | 0.0022  | 0.0179  | 0.0188  | 16.988  |
| 4 | 0.0105  | 0.0800  | 0.0050  | 0.0199  | 0.0187  | 16.974  |
| 5 | 0.0116  | 0.0800  | 0.0045  | 0.0203  | 0.0188  | 16.953  |
| 6 | 0.0115  | 0.0797  | 0.0049  | 0.0204  | 0.0186  | 17.052  |
| 7 | 0.0121  | 0.0794  | 0.0048  | 0.0189  | 0.0184  | 16.968  |
| 8 | 0.0109  | 0.0811  | 0.0059  | 0.0206  | 0.0186  | 16.987  |
| 9 | 0.0114  | 0.0820  | 0.0056  | 0.0210  | 0.0187  | 16.988  |
| 10 | 0.0111  | 0.0795  | 0.0050  | 0.0202  | 0.0188  | 16.939  |
| 11 | 0.0113  | 0.0793  | 0.0053  | 0.0204  | 0.0182  | 16.944  |

表6 539 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0892  | 0.2362  | 0.0250  | 0.0572  | 0.0629  | 18.903  |
| ASD | 0.0011  | 0.0031  | 0.0010  | 0.0017  | 0.0006  | 0.0404  |
| RSD | 1.29  | 1.29  | 4.16  | 2.94  | 0.92  | 0.21  |
| 1 | 0.0904  | 0.2324  | 0.0231  | 0.0547  | 0.0625  | 18.836  |
| 2 | 0.0877  | 0.2390  | 0.0251  | 0.0554  | 0.0620  | 18.947  |
| 3 | 0.0893  | 0.2387  | 0.0246  | 0.0593  | 0.0638  | 18.937  |
| 4 | 0.0897  | 0.2404  | 0.0269  | 0.0560  | 0.0636  | 18.935  |
| 5 | 0.0874  | 0.2395  | 0.0255  | 0.0577  | 0.0633  | 18.872  |
| 6 | 0.0878  | 0.2334  | 0.0244  | 0.0578  | 0.0622  | 18.874  |
| 7 | 0.0886  | 0.2390  | 0.0238  | 0.0590  | 0.0629  | 18.847  |
| 8 | 0.0909  | 0.2340  | 0.0253  | 0.0596  | 0.0634  | 18.904  |
| 9 | 0.0896  | 0.2342  | 0.0247  | 0.0564  | 0.0630  | 18.899  |
| 10 | 0.0900  | 0.2330  | 0.0260  | 0.0579  | 0.0630  | 18.932  |
| 11 | 0.0893  | 0.2350  | 0.0254  | 0.0557  | 0.0625  | 18.946  |

表7 2-1 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.1530  | 1.0529  | 0.0041  | 0.0060  | 0.0231  | 20.193  |
| ASD | 0.0008  | 0.0225  | 0.0008  | 0.0008  | 0.0004  | 0.0372  |
| RSD | 0.55  | 2.13  | 18.48  | 13.82  | 1.93  | 0.18  |
| 1 | 0.1546  | 1.0218  | 0.0022  | 0.0059  | 0.0224  | 20.255  |
| 2 | 0.1529  | 1.0487  | 0.0042  | 0.0060  | 0.0234  | 20.145  |
| 3 | 0.1523  | 1.0610  | 0.0046  | 0.0051  | 0.0234  | 20.177  |
| 4 | 0.1534  | 1.0782  | 0.0033  | 0.0068  | 0.0238  | 20.182  |
| 5 | 0.1532  | 1.0214  | 0.0041  | 0.0060  | 0.0224  | 20.231  |
| 6 | 0.1536  | 1.0319  | 0.0047  | 0.0068  | 0.0228  | 20.216  |
| 7 | 0.1533  | 1.0771  | 0.0043  | 0.0060  | 0.0233  | 20.227  |
| 8 | 0.1538  | 1.0759  | 0.0038  | 0.0059  | 0.0233  | 20.152  |
| 9 | 0.1525  | 1.0773  | 0.0048  | 0.0042  | 0.0234  | 20.166  |
| 10 | 0.1519  | 1.0384  | 0.0044  | 0.0072  | 0.0230  | 20.156  |
| 11 | 0.1519  | 1.0500  | 0.0044  | 0.0064  | 0.0234  | 20.215  |

表8 502 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0333  | 0.3009  | 0.0160  | 0.0370  | 0.0446  | 23.804  |
| ASD | 0.0012  | 0.0047  | 0.0006  | 0.0009  | 0.0008  | 0.0726  |
| RSD | 3.55  | 1.56  | 3.86  | 2.56  | 1.71  | 0.30  |
| 1 | 0.0333  | 0.2958  | 0.0153  | 0.0351  | 0.0444  | 23.841  |
| 2 | 0.0328  | 0.3072  | 0.0149  | 0.0387  | 0.0448  | 23.821  |
| 3 | 0.0328  | 0.2978  | 0.0167  | 0.0374  | 0.0454  | 23.880  |
| 4 | 0.0320  | 0.3011  | 0.0162  | 0.0362  | 0.0447  | 23.759  |
| 5 | 0.0311  | 0.3016  | 0.0158  | 0.0365  | 0.0457  | 23.628  |
| 6 | 0.0346  | 0.3061  | 0.0156  | 0.0379  | 0.0450  | 23.900  |
| 7 | 0.0339  | 0.3067  | 0.0161  | 0.0370  | 0.0449  | 23.828  |
| 8 | 0.0328  | 0.3002  | 0.0160  | 0.0371  | 0.0442  | 23.771  |
| 9 | 0.0342  | 0.2953  | 0.0161  | 0.0365  | 0.0441  | 23.816  |
| 10 | 0.0352  | 0.3037  | 0.0171  | 0.0376  | 0.0446  | 23.824  |
| 11 | 0.0338  | 0.2943  | 0.0164  | 0.0369  | 0.0428  | 23.773  |

表9 503 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0163  | 0.1766  | 0.0080  | 0.0697  | 0.0792  | 23.627  |
| ASD | 0.0005  | 0.0025  | 0.0006  | 0.0010  | 0.0010  | 0.0318  |
| RSD | 2.78  | 1.40  | 7.76  | 1.50  | 1.31  | 0.13  |
| 1 | 0.0163  | 0.1752  | 0.0068  | 0.0684  | 0.0796  | 23.575  |
| 2 | 0.0165  | 0.1824  | 0.0080  | 0.0700  | 0.0800  | 23.660  |
| 3 | 0.0169  | 0.1765  | 0.0078  | 0.0691  | 0.0785  | 23.647  |
| 4 | 0.0162  | 0.1776  | 0.0088  | 0.0687  | 0.0791  | 23.662  |
| 5 | 0.0155  | 0.1747  | 0.0084  | 0.0697  | 0.0801  | 23.572  |
| 6 | 0.0166  | 0.1739  | 0.0077  | 0.0694  | 0.0789  | 23.604  |
| 7 | 0.0168  | 0.1744  | 0.0079  | 0.0702  | 0.0781  | 23.640  |
| 8 | 0.0159  | 0.1792  | 0.0074  | 0.0719  | 0.0788  | 23.650  |
| 9 | 0.0166  | 0.1758  | 0.0079  | 0.0686  | 0.0785  | 23.628  |
| 10 | 0.0158  | 0.1756  | 0.0087  | 0.0707  | 0.0780  | 23.611  |
| 11 | 0.0159  | 0.1769  | 0.0088  | 0.0703  | 0.0815  | 23.644  |

表10 PNMo-8 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0220  | 0.1212  | 0.0613  | 0.0152  | 0.0198  | 23.748  |
| ASD | 0.0007  | 0.0032  | 0.0030  | 0.0009  | 0.0003  | 0.1226  |
| RSD | 3.01  | 2.67  | 4.92  | 5.89  | 1.45  | 0.52  |
| 1 | 0.0228  | 0.1206  | 0.0565  | 0.0151  | 0.0197  | 23.706  |
| 2 | 0.0220  | 0.1271  | 0.0625  | 0.0149  | 0.0198  | 23.779  |
| 3 | 0.0221  | 0.1212  | 0.0635  | 0.0159  | 0.0200  | 23.697  |
| 4 | 0.0217  | 0.1188  | 0.0612  | 0.0168  | 0.0198  | 23.613  |
| 5 | 0.0225  | 0.1196  | 0.0606  | 0.0162  | 0.0200  | 23.636  |
| 6 | 0.0227  | 0.1153  | 0.0662  | 0.0153  | 0.0200  | 23.526  |
| 7 | 0.0217  | 0.1184  | 0.0617  | 0.0148  | 0.0197  | 23.893  |
| 8 | 0.0212  | 0.1248  | 0.0642  | 0.0140  | 0.0197  | 23.851  |
| 9 | 0.0210  | 0.1233  | 0.0562  | 0.0153  | 0.0196  | 23.909  |
| 10 | 0.0229  | 0.1215  | 0.0618  | 0.0141  | 0.0205  | 23.794  |
| 11 | 0.0214  | 0.1223  | 0.0598  | 0.0142  | 0.0194  | 23.823  |

表11 PNMo-9 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0233  | 0.2632  | 0.0664  | 0.0297  | 0.0357  | 24.104  |
| ASD | 0.0007  | 0.0049  | 0.0028  | 0.0012  | 0.0006  | 0.0511  |
| RSD | 3.04  | 1.86  | 4.17  | 4.01  | 1.57  | 0.21  |
| 1 | 0.0244  | 0.2631  | 0.0664  | 0.0294  | 0.0358  | 24.133  |
| 2 | 0.0228  | 0.2582  | 0.0657  | 0.0281  | 0.0354  | 24.116  |
| 3 | 0.0233  | 0.2663  | 0.0630  | 0.0295  | 0.0367  | 24.170  |
| 4 | 0.0233  | 0.2559  | 0.0655  | 0.0283  | 0.0359  | 24.095  |
| 5 | 0.0246  | 0.2740  | 0.0644  | 0.0299  | 0.0365  | 24.128  |
| 6 | 0.0228  | 0.2610  | 0.0653  | 0.0310  | 0.0358  | 24.114  |
| 7 | 0.0227  | 0.2597  | 0.0662  | 0.0311  | 0.0353  | 24.067  |
| 8 | 0.0233  | 0.2665  | 0.0661  | 0.0291  | 0.0350  | 24.147  |
| 9 | 0.0240  | 0.2629  | 0.0671  | 0.0281  | 0.0351  | 24.118  |
| 10 | 0.0224  | 0.2620  | 0.0662  | 0.0310  | 0.0357  | 24.079  |
| 11 | 0.0232  | 0.2656  | 0.0740  | 0.0308  | 0.0351  | 23.978  |

表12 19# 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0415  | 0.2925  | 0.0048  | 0.1854  | 0.1387  | 25.604  |
| ASD | 0.0013  | 0.0091  | 0.0004  | 0.0043  | 0.0019  | 0.0906  |
| RSD | 3.08  | 3.11  | 8.18  | 2.29  | 1.35  | 0.35  |
| 1 | 0.0414  | 0.2839  | 0.0051  | 0.1798  | 0.1389  | 25.692  |
| 2 | 0.0432  | 0.2969  | 0.0046  | 0.1878  | 0.1402  | 25.597  |
| 3 | 0.0434  | 0.2826  | 0.0050  | 0.1782  | 0.1359  | 25.826  |
| 4 | 0.0404  | 0.2970  | 0.0047  | 0.1887  | 0.1397  | 25.577  |
| 5 | 0.0396  | 0.2995  | 0.0052  | 0.1860  | 0.1395  | 25.541  |
| 6 | 0.0431  | 0.2989  | 0.0043  | 0.1866  | 0.1405  | 25.500  |
| 7 | 0.0419  | 0.3077  | 0.0050  | 0.1898  | 0.1411  | 25.653  |
| 8 | 0.0404  | 0.2959  | 0.0054  | 0.1865  | 0.1399  | 25.570  |
| 9 | 0.0410  | 0.2929  | 0.0050  | 0.1907  | 0.1375  | 25.574  |
| 10 | 0.0408  | 0.2781  | 0.0042  | 0.1799  | 0.1371  | 25.546  |
| 11 | 0.0409  | 0.2838  | 0.0044  | 0.1857  | 0.1357  | 25.567  |

表13 9-200 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.4620  | 1.2501  | 0.0036  | 0.0266  | 0.0342  | 33.201  |
| ASD | 0.0050  | 0.0261  | 0.0006  | 0.0007  | 0.0004  | 0.0665  |
| RSD | 1.08  | 2.09  | 16.25  | 2.76  | 1.24  | 0.20  |
| 1 | 0.4543  | 1.2464  | 0.0034  | 0.0255  | 0.0342  | 33.214  |
| 2 | 0.4594  | 1.2168  | 0.0034  | 0.0264  | 0.0337  | 33.134  |
| 3 | 0.4598  | 1.2573  | 0.0039  | 0.0258  | 0.0337  | 33.139  |
| 4 | 0.4635  | 1.2250  | 0.0038  | 0.0273  | 0.0336  | 33.117  |
| 5 | 0.4647  | 1.2728  | 0.0031  | 0.0276  | 0.0341  | 33.213  |
| 6 | 0.4611  | 1.2061  | 0.0032  | 0.0265  | 0.0342  | 33.135  |
| 7 | 0.4604  | 1.2468  | 0.0032  | 0.0258  | 0.0346  | 33.209  |
| 8 | 0.4650  | 1.2846  | 0.0052  | 0.0277  | 0.0345  | 33.285  |
| 9 | 0.4729  | 1.2856  | 0.0037  | 0.0269  | 0.0343  | 33.303  |
| 10 | 0.4649  | 1.2591  | 0.0033  | 0.0264  | 0.0344  | 33.281  |
| 11 | 0.4565  | 1.2505  | 0.0039  | 0.0268  | 0.0350  | 33.185  |