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

共收集到起草单位的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 | 193.2 |
| Fe | 239.5 | 242.9 |
| Mo | 281.6 | 289.2 |
| Al | 396.1 | 395.3 |
| Si | 288.1 | 289.2 |
| Pr | 405.6 | 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 | Si | Pr | Al |
| Avg | 0.0090 | 0.0790 | 0.0030 | 0.0190 | 17.026 | 0.0160 |
| ASD | 0.0000 | 0.0010 | 0.0010 | 0.0010 | 0.0360 | 0.0010 |
| RSD | 3.38 | 1.25 | 24.09 | 2.61 | 0.21 | 3.38 |
| 1 | 0.0090 | 0.0790 | 0.0030 | 0.0190 | 17.057 | 0.0150 |
| 2 | 0.0090 | 0.0780 | 0.0030 | 0.0200 | 17.052 | 0.0160 |
| 3 | 0.0090 | 0.0780 | 0.0040 | 0.0190 | 17.036 | 0.0150 |
| 4 | 0.0090 | 0.0780 | 0.0020 | 0.0190 | 17.003 | 0.0150 |
| 5 | 0.0090 | 0.0780 | 0.0030 | 0.0190 | 17.028 | 0.0150 |
| 6 | 0.0090 | 0.0810 | 0.0020 | 0.0200 | 17.069 | 0.0160 |
| 7 | 0.0090 | 0.0780 | 0.0030 | 0.0190 | 17.067 | 0.0160 |
| 8 | 0.0080 | 0.0800 | 0.0040 | 0.0200 | 16.968 | 0.0150 |
| 9 | 0.0090 | 0.0790 | 0.0030 | 0.0190 | 16.965 | 0.0150 |
| 10 | 0.0090 | 0.0790 | 0.0020 | 0.0200 | 17.014 | 0.0160 |
| 11 | 0.0090 | 0.0790 | 0.0030 | 0.0190 | 17.025 | 0.0160 |

表6 539 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0928 | 0.2302 | 0.0264 | 0.0625 | 18.908 | 0.0496 |
| ASD | 0.0007 | 0.0042 | 0.0007 | 0.0005 | 0.0404 | 0.0005 |
| RSD | 0.81 | 1.81 | 2.55 | 0.85 | 0.21 | 0.99 |
| 1 | 0.0913 | 0.2247 | 0.0258 | 0.0619 | 18.878 | 0.0486 |
| 2 | 0.0923 | 0.2285 | 0.0254 | 0.0620 | 18.913 | 0.0494 |
| 3 | 0.0931 | 0.2308 | 0.0268 | 0.0629 | 18.957 | 0.0498 |
| 4 | 0.0922 | 0.2251 | 0.0272 | 0.0622 | 18.969 | 0.0503 |
| 5 | 0.0930 | 0.2298 | 0.0258 | 0.0619 | 18.877 | 0.0492 |
| 6 | 0.0935 | 0.2343 | 0.0263 | 0.0631 | 18.969 | 0.0503 |
| 7 | 0.0932 | 0.2263 | 0.0254 | 0.0624 | 18.901 | 0.0494 |
| 8 | 0.0919 | 0.2322 | 0.0269 | 0.0634 | 18.855 | 0.0497 |
| 9 | 0.0933 | 0.2372 | 0.0267 | 0.0628 | 18.870 | 0.0498 |
| 10 | 0.0936 | 0.2283 | 0.0271 | 0.0619 | 18.908 | 0.0497 |
| 11 | 0.0933 | 0.2353 | 0.0268 | 0.0624 | 18.895 | 0.0493 |

表7 2-1 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.1615 | 1.0222 | 0.0024 | 0.0239 | 20.253 | 0.0049 |
| ASD | 0.0014 | 0.0137 | 0.0006 | 0.0003 | 0.0460 | 0.0001 |
| RSD | 0.84 | 1.34 | 26.19 | 1.38 | 0.23 | 2.91 |
| 1 | 0.1615 | 1.0535 | 0.0017 | 0.0243 | 20.256 | 0.0050 |
| 2 | 0.1598 | 1.0188 | 0.0022 | 0.0240 | 20.269 | 0.0051 |
| 3 | 0.1593 | 1.0442 | 0.0025 | 0.0236 | 20.184 | 0.0047 |
| 4 | 0.1604 | 1.0179 | 0.0016 | 0.0239 | 20.214 | 0.0049 |
| 5 | 0.1630 | 1.0223 | 0.0033 | 0.0235 | 20.245 | 0.0049 |
| 6 | 0.1615 | 1.0185 | 0.0022 | 0.0239 | 20.196 | 0.0047 |
| 7 | 0.1613 | 1.0108 | 0.0024 | 0.0236 | 20.341 | 0.0051 |
| 8 | 0.1611 | 1.0168 | 0.0032 | 0.0242 | 20.231 | 0.0049 |
| 9 | 0.1627 | 1.0157 | 0.0016 | 0.0244 | 20.280 | 0.0050 |
| 10 | 0.1638 | 1.0123 | 0.0031 | 0.0242 | 20.276 | 0.0051 |
| 11 | 0.1619 | 1.0137 | 0.0029 | 0.0235 | 20.295 | 0.0050 |

表8 502 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0347 | 0.2950 | 0.0165 | 0.0449 | 23.866 | 0.0383 |
| ASD | 0.0006 | 0.0066 | 0.0008 | 0.0007 | 0.0374 | 0.0006 |
| RSD | 1.70 | 2.24 | 4.76 | 1.56 | 0.16 | 1.52 |
| 1 | 0.0355 | 0.2952 | 0.0168 | 0.0441 | 23.870 | 0.0377 |
| 2 | 0.0351 | 0.2825 | 0.0151 | 0.0438 | 23.827 | 0.0372 |
| 3 | 0.0345 | 0.2906 | 0.0164 | 0.0450 | 23.857 | 0.0382 |
| 4 | 0.0347 | 0.2940 | 0.0157 | 0.0443 | 23.875 | 0.0382 |
| 5 | 0.0357 | 0.3004 | 0.0169 | 0.0448 | 23.878 | 0.0380 |
| 6 | 0.0345 | 0.3063 | 0.0158 | 0.0458 | 23.806 | 0.0384 |
| 7 | 0.0342 | 0.2888 | 0.0164 | 0.0453 | 23.821 | 0.0384 |
| 8 | 0.0345 | 0.2988 | 0.0167 | 0.0455 | 23.860 | 0.0388 |
| 9 | 0.0348 | 0.2913 | 0.0180 | 0.0442 | 23.906 | 0.0387 |
| 10 | 0.0344 | 0.2956 | 0.0164 | 0.0455 | 23.930 | 0.0390 |
| 11 | 0.0336 | 0.3009 | 0.0172 | 0.0456 | 23.895 | 0.0392 |

表9 503 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0152 | 0.1723 | 0.0074 | 0.0783 | 23.736 | 0.0656 |
| ASD | 0.0006 | 0.0031 | 0.0003 | 0.0008 | 0.0422 | 0.0003 |
| RSD | 4.04 | 1.82 | 4.49 | 1.02 | 0.18 | 0.49 |
| 1 | 0.0164 | 0.1698 | 0.0074 | 0.0783 | 23.712 | 0.0655 |
| 2 | 0.0158 | 0.1746 | 0.0069 | 0.0785 | 23.757 | 0.0656 |
| 3 | 0.0151 | 0.1723 | 0.0072 | 0.0777 | 23.701 | 0.0653 |
| 4 | 0.0157 | 0.1725 | 0.0077 | 0.0776 | 23.785 | 0.0655 |
| 5 | 0.0150 | 0.1721 | 0.0077 | 0.0778 | 23.731 | 0.0655 |
| 6 | 0.0146 | 0.1723 | 0.0073 | 0.0781 | 23.714 | 0.0657 |
| 7 | 0.0153 | 0.1739 | 0.0068 | 0.0782 | 23.718 | 0.0656 |
| 8 | 0.0144 | 0.1673 | 0.0075 | 0.0779 | 23.722 | 0.0654 |
| 9 | 0.0151 | 0.1679 | 0.0077 | 0.0774 | 23.840 | 0.0652 |
| 10 | 0.0148 | 0.1759 | 0.0075 | 0.0791 | 23.714 | 0.0662 |
| 11 | 0.0145 | 0.1775 | 0.0078 | 0.0802 | 23.707 | 0.0662 |

表10 PNMo-8 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0219 | 0.1188 | 0.0648 | 0.0201 | 23.931 | 0.0166 |
| ASD | 0.0008 | 0.0023 | 0.0026 | 0.0004 | 0.0355 | 0.0002 |
| RSD | 3.81 | 1.97 | 3.98 | 1.94 | 0.15 | 1.40 |
| 1 | 0.0242 | 0.1184 | 0.0661 | 0.0203 | 23.868 | 0.0164 |
| 2 | 0.0216 | 0.1166 | 0.0647 | 0.0198 | 23.946 | 0.0161 |
| 3 | 0.0219 | 0.1189 | 0.0613 | 0.0200 | 23.919 | 0.0167 |
| 4 | 0.0220 | 0.1165 | 0.0645 | 0.0195 | 23.973 | 0.0168 |
| 5 | 0.0220 | 0.1175 | 0.0663 | 0.0199 | 23.959 | 0.0165 |
| 6 | 0.0216 | 0.1231 | 0.0694 | 0.0208 | 23.895 | 0.0169 |
| 7 | 0.0214 | 0.1171 | 0.0655 | 0.0201 | 23.965 | 0.0169 |
| 8 | 0.0216 | 0.1194 | 0.0644 | 0.0201 | 23.910 | 0.0167 |
| 9 | 0.0211 | 0.1206 | 0.0610 | 0.0202 | 23.948 | 0.0166 |
| 10 | 0.0218 | 0.1164 | 0.0621 | 0.0200 | 23.963 | 0.0167 |
| 11 | 0.0212 | 0.1223 | 0.0674 | 0.0208 | 23.893 | 0.0166 |

表11 PNMo-9 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0236 | 0.2567 | 0.0704 | 0.0359 | 24.261 | 0.0315 |
| ASD | 0.0004 | 0.0041 | 0.0016 | 0.0005 | 0.0394 | 0.0004 |
| RSD | 1.68 | 1.59 | 2.22 | 1.37 | 0.16 | 1.23 |
| 1 | 0.0237 | 0.2588 | 0.0732 | 0.0357 | 24.238 | 0.0310 |
| 2 | 0.0233 | 0.2591 | 0.0691 | 0.0359 | 24.316 | 0.0313 |
| 3 | 0.0242 | 0.2614 | 0.0701 | 0.0363 | 24.251 | 0.0314 |
| 4 | 0.0234 | 0.2510 | 0.0683 | 0.0349 | 24.300 | 0.0316 |
| 5 | 0.0243 | 0.2576 | 0.0688 | 0.0356 | 24.203 | 0.0308 |
| 6 | 0.0234 | 0.2598 | 0.0707 | 0.0364 | 24.214 | 0.0314 |
| 7 | 0.0233 | 0.2600 | 0.0694 | 0.0365 | 24.269 | 0.0316 |
| 8 | 0.0238 | 0.2549 | 0.0725 | 0.0356 | 24.277 | 0.0317 |
| 9 | 0.0231 | 0.2518 | 0.0700 | 0.0362 | 24.226 | 0.0323 |
| 10 | 0.0236 | 0.2501 | 0.0718 | 0.0353 | 24.262 | 0.0316 |
| 11 | 0.0232 | 0.2598 | 0.0709 | 0.0358 | 24.317 | 0.0315 |

表12 19# 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.0415 | 0.2865 | 0.0035 | 0.1376 | 25.730 | 0.1734 |
| ASD | 0.0011 | 0.0052 | 0.0004 | 0.0014 | 0.0346 | 0.0014 |
| RSD | 2.63 | 1.81 | 12.31 | 1.01 | 0.13 | 0.83 |
| 1 | 0.0412 | 0.2799 | 0.0035 | 0.1354 | 25.746 | 0.1733 |
| 2 | 0.0401 | 0.2802 | 0.0031 | 0.1356 | 25.730 | 0.1713 |
| 3 | 0.0408 | 0.2884 | 0.0039 | 0.1375 | 25.655 | 0.1761 |
| 4 | 0.0413 | 0.2885 | 0.0030 | 0.1385 | 25.677 | 0.1750 |
| 5 | 0.0414 | 0.2920 | 0.0039 | 0.1383 | 25.727 | 0.1736 |
| 6 | 0.0434 | 0.2852 | 0.0032 | 0.1379 | 25.747 | 0.1723 |
| 7 | 0.0409 | 0.2897 | 0.0042 | 0.1396 | 25.730 | 0.1724 |
| 8 | 0.0436 | 0.2891 | 0.0032 | 0.1392 | 25.752 | 0.1741 |
| 9 | 0.0422 | 0.2863 | 0.0030 | 0.1374 | 25.746 | 0.1732 |
| 10 | 0.0414 | 0.2941 | 0.0038 | 0.1382 | 25.741 | 0.1742 |
| 11 | 0.0408 | 0.2781 | 0.0032 | 0.1362 | 25.776 | 0.1716 |

表13 9-200 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Si | Pr | Al |
| Avg | 0.4743 | 1.2211 | 0.0017 | 0.0341 | 33.461 | 0.0357 |
| ASD | 0.0064 | 0.0255 | 0.0004 | 0.0003 | 0.0516 | 0.0004 |
| RSD | 1.35 | 2.09 | 26.17 | 0.99 | 0.15 | 1.10 |
| 1 | 0.4824 | 1.2585 | 0.0009 | 0.0345 | 33.541 | 0.0351 |
| 2 | 0.4683 | 1.2331 | 0.0025 | 0.0340 | 33.491 | 0.0351 |
| 3 | 0.4784 | 1.2598 | 0.0017 | 0.0345 | 33.488 | 0.0358 |
| 4 | 0.4708 | 1.2195 | 0.0019 | 0.0343 | 33.444 | 0.0361 |
| 5 | 0.4761 | 1.2259 | 0.0014 | 0.0341 | 33.502 | 0.0355 |
| 6 | 0.4790 | 1.1913 | 0.0022 | 0.0340 | 33.450 | 0.0359 |
| 7 | 0.4663 | 1.2379 | 0.0018 | 0.0334 | 33.397 | 0.0358 |
| 8 | 0.4636 | 1.2061 | 0.0014 | 0.0342 | 33.386 | 0.0361 |
| 9 | 0.4745 | 1.1891 | 0.0020 | 0.0336 | 33.445 | 0.0357 |
| 10 | 0.4749 | 1.1889 | 0.0014 | 0.0341 | 33.409 | 0.0361 |
| 11 | 0.4828 | 1.2223 | 0.0015 | 0.0342 | 33.523 | 0.0352 |