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

包头市三隆稀有金属材料有限责任公司

二零二四年一月二十三日

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 | 12 | / | / |
| 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 | 0.0727 | 0.0029 | 0.0137 | 0.0197 | 16.842 |
| ASD | 0.0013 | 0.0050 | 0.0013 | 0.0025 | 0.0003 | 0.0428 |
| RSD | 11.69 | 6.85 | 42.42 | 18.13 | 1.60 | 0.25 |
| 1 | 0.0119 | 0.0730 | 0.0038 | 0.0118 | 0.0198 | 16.785 |
| 2 | 0.0111 | 0.0712 | 0.0033 | 0.0137 | 0.0200 | 16.817 |
| 3 | 0.0124 | 0.0748 | 0.0041 | 0.0104 | 0.0196 | 16.824 |
| 4 | 0.0092 | 0.0829 | 0.0014 | 0.0133 | 0.0197 | 16.798 |
| 5 | 0.0103 | 0.0723 | 0.0035 | 0.0128 | 0.0194 | 16.829 |
| 6 | 0.0116 | 0.0727 | 0.0035 | 0.0138 | 0.0198 | 16.811 |
| 7 | 0.0125 | 0.0740 | 0.0042 | 0.0126 | 0.0199 | 16.841 |
| 8 | 0.0124 | 0.0611 | 0.0000 | 0.0121 | 0.0190 | 16.865 |
| 9 | 0.0123 | 0.0731 | 0.0033 | 0.0150 | 0.0200 | 16.882 |
| 10 | 0.0088 | 0.0719 | 0.0028 | 0.0199 | 0.0198 | 16.923 |
| 11 | 0.0120 | 0.0727 | 0.0026 | 0.0152 | 0.0193 | 16.892 |

表6 539 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0902 | 0.2449 | 0.0243 | 0.0513 | 0.0624 | 18.679 |
| ASD | 0.0016 | 0.0047 | 0.0011 | 0.0052 | 0.0007 | 0.0514 |
| RSD | 1.75 | 1.91 | 4.42 | 10.06 | 1.07 | 0.28 |
| 1 | 0.0924 | 0.2406 | 0.0247 | 0.0524 | 0.0618 | 18.619 |
| 2 | 0.0918 | 0.2401 | 0.0238 | 0.0500 | 0.0627 | 18.712 |
| 3 | 0.0899 | 0.2436 | 0.0247 | 0.0486 | 0.0613 | 18.600 |
| 4 | 0.0904 | 0.2471 | 0.0233 | 0.0505 | 0.0627 | 18.667 |
| 5 | 0.0882 | 0.2476 | 0.0221 | 0.0650 | 0.0635 | 18.761 |
| 6 | 0.0932 | 0.2453 | 0.0251 | 0.0477 | 0.0628 | 18.761 |
| 7 | 0.0894 | 0.2463 | 0.0249 | 0.0503 | 0.0620 | 18.666 |
| 8 | 0.0898 | 0.2434 | 0.0236 | 0.0482 | 0.0621 | 18.689 |
| 9 | 0.0891 | 0.2567 | 0.0258 | 0.0482 | 0.0627 | 18.649 |
| 10 | 0.0891 | 0.2410 | 0.0253 | 0.0479 | 0.0618 | 18.690 |
| 11 | 0.0891 | 0.2426 | 0.0237 | 0.0562 | 0.0632 | 18.652 |

表7 2-1 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.1589 | 1.0771 | 0.0037 | 0.0023 | 0.0239 | 20.144 |
| ASD | 0.0017 | 0.0512 | 0.0008 | 0.0007 | 0.0003 | 0.0552 |
| RSD | 1.07 | 4.75 | 21.79 | 28.84 | 1.38 | 0.27 |
| 1 | 0.1595 | 1.0873 | 0.0030 | 0.0036 | 0.0235 | 20.045 |
| 2 | 0.1588 | 0.9336 | 0.0043 | 0.0026 | 0.0239 | 20.123 |
| 3 | 0.1590 | 1.0636 | 0.0048 | 0.0016 | 0.0237 | 20.127 |
| 4 | 0.1607 | 1.1003 | 0.0041 | 0.0014 | 0.0233 | 20.128 |
| 5 | 0.1611 | 1.0941 | 0.0044 | 0.0022 | 0.0237 | 20.096 |
| 6 | 0.1602 | 1.1142 | 0.0030 | 0.0026 | 0.0239 | 20.192 |
| 7 | 0.1557 | 1.0912 | 0.0037 | 0.0015 | 0.0240 | 20.258 |
| 8 | 0.1560 | 1.0893 | 0.0034 | 0.0018 | 0.0246 | 20.135 |
| 9 | 0.1592 | 1.1144 | 0.0036 | 0.0027 | 0.0239 | 20.131 |
| 10 | 0.1585 | 1.1064 | 0.0020 | 0.0024 | 0.0240 | 20.186 |
| 11 | 0.1589 | 1.0537 | 0.0042 | 0.0024 | 0.0239 | 20.158 |

表8 502 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0356 | 0.3040 | 0.0167 | 0.0376 | 0.0445 | 23.581 |
| ASD | 0.0012 | 0.0202 | 0.0009 | 0.0023 | 0.0006 | 0.0589 |
| RSD | 3.36 | 6.63 | 5.18 | 6.14 | 1.35 | 0.25 |
| 1 | 0.0342 | 0.3233 | 0.0175 | 0.0408 | 0.0445 | 23.557 |
| 2 | 0.0353 | 0.3069 | 0.0162 | 0.0393 | 0.0442 | 23.558 |
| 3 | 0.0365 | 0.3153 | 0.0167 | 0.0382 | 0.0456 | 23.638 |
| 4 | 0.0339 | 0.3237 | 0.0178 | 0.0362 | 0.0444 | 23.614 |
| 5 | 0.0336 | 0.3112 | 0.0160 | 0.0419 | 0.0456 | 23.569 |
| 6 | 0.0366 | 0.3088 | 0.0184 | 0.0369 | 0.0442 | 23.504 |
| 7 | 0.0358 | 0.2647 | 0.0166 | 0.0378 | 0.0448 | 23.551 |
| 8 | 0.0370 | 0.3044 | 0.0163 | 0.0366 | 0.0436 | 23.512 |
| 9 | 0.0366 | 0.2666 | 0.0155 | 0.0368 | 0.0444 | 23.711 |
| 10 | 0.0363 | 0.3031 | 0.0161 | 0.0340 | 0.0442 | 23.607 |
| 11 | 0.0358 | 0.3162 | 0.0170 | 0.0355 | 0.0444 | 23.565 |

表9 503 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0171 | 0.1772 | 0.0097 | 0.0658 | 0.0790 | 23.565 |
| ASD | 0.0013 | 0.0111 | 0.0007 | 0.0019 | 0.0013 | 0.0829 |
| RSD | 7.59 | 6.25 | 7.62 | 2.91 | 1.58 | 0.35 |
| 1 | 0.0165 | 0.1800 | 0.0100 | 0.0672 | 0.0804 | 23.579 |
| 2 | 0.0161 | 0.1847 | 0.0096 | 0.0635 | 0.0787 | 23.588 |
| 3 | 0.0169 | 0.1597 | 0.0085 | 0.0683 | 0.0796 | 23.546 |
| 4 | 0.0192 | 0.1840 | 0.0106 | 0.0644 | 0.0798 | 23.555 |
| 5 | 0.0187 | 0.1810 | 0.0102 | 0.0635 | 0.0770 | 23.531 |
| 6 | 0.0169 | 0.1827 | 0.0091 | 0.0655 | 0.0795 | 23.544 |
| 7 | 0.0178 | 0.1847 | 0.0099 | 0.0643 | 0.0796 | 23.800 |
| 8 | 0.0146 | 0.1829 | 0.0100 | 0.0692 | 0.0807 | 23.520 |
| 9 | 0.0179 | 0.1805 | 0.0098 | 0.0651 | 0.0777 | 23.541 |
| 10 | 0.0161 | 0.1781 | 0.0108 | 0.0663 | 0.0772 | 23.485 |
| 11 | 0.0172 | 0.1514 | 0.0086 | 0.0670 | 0.0786 | 23.522 |

表10 PNMo-8 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0239 | 0.1205 | 0.0551 | 0.0156 | 0.0206 | 23.699 |
| ASD | 0.0011 | 0.0063 | 0.0025 | 0.0011 | 0.0002 | 0.0567 |
| RSD | 4.71 | 5.25 | 4.45 | 7.33 | 1.15 | 0.24 |
| 1 | 0.0241 | 0.1229 | 0.0589 | 0.0164 | 0.0203 | 23.632 |
| 2 | 0.0224 | 0.1211 | 0.0585 | 0.0159 | 0.0206 | 23.610 |
| 3 | 0.0232 | 0.1220 | 0.0560 | 0.0137 | 0.0205 | 23.662 |
| 4 | 0.0239 | 0.1205 | 0.0578 | 0.0146 | 0.0207 | 23.690 |
| 5 | 0.0235 | 0.1185 | 0.0543 | 0.0154 | 0.0207 | 23.738 |
| 6 | 0.0247 | 0.1197 | 0.0546 | 0.0170 | 0.0207 | 23.734 |
| 7 | 0.0227 | 0.1212 | 0.0543 | 0.0160 | 0.0203 | 23.748 |
| 8 | 0.0266 | 0.1253 | 0.0532 | 0.0154 | 0.0209 | 23.719 |
| 9 | 0.0237 | 0.1037 | 0.0550 | 0.0173 | 0.0206 | 23.694 |
| 10 | 0.0235 | 0.1210 | 0.0527 | 0.0140 | 0.0205 | 23.654 |
| 11 | 0.0243 | 0.1295 | 0.0512 | 0.0159 | 0.0211 | 23.802 |

表11 PNMo-9 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0252 | 0.2752 | 0.0619 | 0.0290 | 0.0353 | 24.041 |
| ASD | 0.0016 | 0.0055 | 0.0030 | 0.0025 | 0.0006 | 0.0652 |
| RSD | 6.46 | 1.99 | 4.90 | 8.52 | 1.62 | 0.27 |
| 1 | 0.0244 | 0.2696 | 0.0644 | 0.0268 | 0.0341 | 23.934 |
| 2 | 0.0251 | 0.2659 | 0.0590 | 0.0297 | 0.0348 | 23.952 |
| 3 | 0.0234 | 0.2673 | 0.0608 | 0.0302 | 0.0352 | 23.989 |
| 4 | 0.0263 | 0.2771 | 0.0615 | 0.0350 | 0.0356 | 24.017 |
| 5 | 0.0234 | 0.2789 | 0.0656 | 0.0302 | 0.0350 | 23.998 |
| 6 | 0.0287 | 0.2801 | 0.0598 | 0.0303 | 0.0359 | 24.104 |
| 7 | 0.0241 | 0.2733 | 0.0596 | 0.0276 | 0.0352 | 24.078 |
| 8 | 0.0241 | 0.2767 | 0.0604 | 0.0271 | 0.0357 | 24.095 |
| 9 | 0.0270 | 0.2756 | 0.0688 | 0.0277 | 0.0355 | 24.069 |
| 10 | 0.0251 | 0.2818 | 0.0608 | 0.0286 | 0.0362 | 24.110 |
| 11 | 0.0257 | 0.2805 | 0.0606 | 0.0262 | 0.0353 | 24.106 |

表12 19# 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.0435 | 0.3036 | 0.0049 | 0.1719 | 0.1381 | 25.460 |
| ASD | 0.0021 | 0.0169 | 0.0012 | 0.0054 | 0.0009 | 0.0409 |
| RSD | 4.77 | 5.56 | 25.38 | 3.16 | 0.63 | 0.16 |
| 1 | 0.0444 | 0.2998 | 0.0043 | 0.1728 | 0.1379 | 25.466 |
| 2 | 0.0418 | 0.3120 | 0.0042 | 0.1783 | 0.1378 | 25.444 |
| 3 | 0.0471 | 0.3178 | 0.0056 | 0.1742 | 0.1389 | 25.408 |
| 4 | 0.0450 | 0.3082 | 0.0051 | 0.1734 | 0.1380 | 25.474 |
| 5 | 0.0430 | 0.3175 | 0.0023 | 0.1708 | 0.1388 | 25.477 |
| 6 | 0.0432 | 0.3061 | 0.0054 | 0.1668 | 0.1384 | 25.564 |
| 7 | 0.0406 | 0.2978 | 0.0057 | 0.1743 | 0.1383 | 25.423 |
| 8 | 0.0445 | 0.3117 | 0.0050 | 0.1729 | 0.1390 | 25.451 |
| 9 | 0.0418 | 0.2564 | 0.0035 | 0.1797 | 0.1387 | 25.427 |
| 10 | 0.0459 | 0.3063 | 0.0069 | 0.1611 | 0.1376 | 25.462 |
| 11 | 0.0411 | 0.3059 | 0.0053 | 0.1660 | 0.1359 | 25.465 |

表13 9-200 精密度实验结果

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | C | Fe | Mo | Al | Si | Pr |
| Avg | 0.4770 | 1.2566 | 0.0043 | 0.0459 | 0.0352 | 33.200 |
| ASD | 0.0078 | 0.0219 | 0.0008 | 0.0045 | 0.0003 | 0.0687 |
| RSD | 1.63 | 1.74 | 17.45 | 9.86 | 0.90 | 0.21 |
| 1 | 0.4758 | 1.2974 | 0.0046 | 0.0494 | 0.0346 | 33.206 |
| 2 | 0.4935 | 1.2520 | 0.0033 | 0.0443 | 0.0354 | 33.085 |
| 3 | 0.4699 | 1.2371 | 0.0043 | 0.0437 | 0.0355 | 33.221 |
| 4 | 0.4813 | 1.2608 | 0.0031 | 0.0516 | 0.0346 | 33.222 |
| 5 | 0.4776 | 1.2554 | 0.0042 | 0.0516 | 0.0354 | 33.331 |
| 6 | 0.4714 | 1.2475 | 0.0039 | 0.0412 | 0.0353 | 33.260 |
| 7 | 0.4869 | 1.2557 | 0.0054 | 0.0506 | 0.0352 | 33.151 |
| 8 | 0.4755 | 1.2515 | 0.0045 | 0.0410 | 0.0352 | 33.126 |
| 9 | 0.4719 | 1.2140 | 0.0054 | 0.0488 | 0.0349 | 33.179 |
| 10 | 0.4760 | 1.2706 | 0.0039 | 0.0413 | 0.0354 | 33.255 |
| 11 | 0.4669 | 1.2811 | 0.0049 | 0.0412 | 0.0352 | 33.169 |