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

包头市中鑫安泰磁业有限公司

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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 |