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  |