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 | 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  |