第16卷第11期
2026年11月

中国无机分析化学
Chinese Journal of Inorganic Analytical Chemistry

Vol. 16, No. 11
2109～2117

doi: 10. 20236/j. CJIAC. 2026. 11. 014

陈光辉，田怡丰，邹景，等. 激光诱导击穿光谱(LIBS)结合光束整形装置的钢铁成分定量分析研究[J]. 中国无机分析化学，2026，16(11)：2109-2117.

Chen Guanghui, Tian Yifeng, Zou Jing, et al. Quantitative Analysis of Steel Components by Laser-induced Breakdown Spectroscopy Combined with a Beam Shaping Device[J]. Chinese Journal of Inorganic Analytical Chemistry, 2026, 16(11): 2109-2117.

激光诱导击穿光谱(LIBS)结合光束整形装置的钢铁成分定量分析研究

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摘　要　激光诱导击穿光谱(Laser-induced breakdown spectroscopy，LIBS)技术是一种实时在线的物质成分分析方法。但传统LIBS采用能量分布不均的高斯光束，易诱发等离子体屏蔽效应，降低激光-物质耦合效率，严重影响光谱信号稳定性与定量分析精度。为此，本文设计了一种由平凹柱面透镜与平凸柱面透镜组成的新型光束整形器，将高斯激光束整形为能量分布更均匀的近似平顶光束(Approximately flat-top beam，AFTB)，从而改善LIBS分析精度。以钢铁样品中的微量元素为分析对象，分别在传统LIBS和AFTB-LIBS条件下，系统优化了激光能量和离焦量等实验参数，并基于特征谱线强度对比评估了两种方法的光谱信号稳定性及定量分析性能。结果表明，在最优参数条件下，AFTB-LIBS能够显著增强各元素特征谱线强度，且谱线强度的相对标准偏差普遍低于传统LIBS，尤其对Cr和Si的稳定性改善作用明显。此外，AFTB-LIBS可有效提升对微量元素的定量分析精度，例如可将Si定标曲线的线性拟合决定系数R^2由传统LIBS的0. 895 8提升至0. 974 1。上述结果表明，本研究所提出的光束整形方法能够有效提升光谱信号的稳定性与元素的定量分析精度，为工业冶金领域实现钢铁成分的快速、高精度、低成本检测提供了技术参考。

关键词　钢铁；激光诱导击穿光谱；元素定量分析；光束整形

要点

1)　利用柱面镜组成激光光束整形系统，改善激光能量的分布均匀性。

收稿日期：2026-08-03　**修回日期**：2026-08-07　**录用日期**：2026-08-08　**在线出版日期**：2026-08-14

Received：2026-08-03　**Revised**：2026-08-07　**Accepted**：2026-08-08　**Online published**：2026-08-14

基金项目：国家自然科学基金资助项目(61705064)；新型电力系统运行与控制全国重点实验室资助项目(SKLD25KM39)；湖北省教育厅创新团队项目(T2025020)；湖北省教育厅科学研究计划项目(B2023162)；孝感市自然科学计划项目(XGKJ2025010094，XGKJ2024020016)

Fund：Supported by the National Natural Science Foundation of China (61705064)；State Key Laboratory of Power System Operation and Control (SKLD25KM39)；The Project of the Hubei Provincial Department of Education (T2025020)；Scientific Research Program of Hubei Provincial Department of Education (B2023162)；Xiaogan City Natural Science Plan Project (XGKJ2025010094, XGKJ2024020016)

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2）研究了光束整形方法对激光等离子体光谱的影响。

3）LIBS结合光束整形的方法能为钢铁的元素定量分析提供技术支持。

中图分类号：O657.38 **文献标志码：**A **文章编号：**2095-1035(2026)11-2109-09

钢铁合金的构成元素种类及其含量直接决定其物理化学特性和用途，使其在工业和建筑等领域获得广泛应用[1]。例如，合金中的Cr与氧气接触时，会形成一层有助于防止合金腐蚀的致密氧化铬层[2]；Cu和Mo同样可形成保护性的氧化层，从而在海水或含氯化物介质等特定腐蚀环境中有效抑制不锈钢的氧化和腐蚀[3]；Mn、Ni的含量多少均会影响合金成品的硬度[4]。因此，实现钢铁中元素成分的实时、快速、准确定量分析，是钢铁行业产品分类和质量控制的关键环节。然而，火花直读光谱[5]和电感耦合等离子体发射光谱法[6]等传统元素分析方法均存在制样复杂、分析耗时等不足；而X射线荧光光谱法[7]存在辐射安全风险，难以满足工业应用对实时分析的需求。

激光诱导击穿光谱(Laser-induced Breakdown Spectroscopy，LIBS)是一种原子光谱分析技术，其原理是将激光聚焦于样品表面激发生成等离子体，并通过光谱仪分析其辐射的光谱，从而实现样品中元素的定性与定量分析[8-10]。相比其他分析技术，LIBS具备样品处理简单、检测速度快、可多元素同时探测、可远程探测等独特优势，因此，近年来LIBS被广泛应用于煤炭检测[11]、农产品检测[12-13]、文化遗产[14]、土质检测[15-16]等多个领域。然而，由于LIBS的分析对象是一种高度瞬态且空间分布不均匀的激光诱导等离子体，其光谱信号稳定性偏低，导致其大规模商业化仍是一项艰巨的挑战[17-19]。此外，钢铁中包含的丰富元素极易导致复杂的基体效应和自吸收效应，而上述因素的共同作用通常导致LIBS检测复杂基质样品时分类识别率偏低[20-22]。

近年来，研究者提出了多种方法来提高LIBS分析精度，信号增强是改善LIBS信号的主要方法，其中光束整形法因操作简单、设备成本较低而备受关注[23]。由于传统激光器输出的高斯激光束能量分布呈中心高、边缘低的非均匀特征[24]，易导致激光烧蚀坑形态不稳定，进而影响等离子体的稳定性与均匀性。为此，研究者们提出了通过特定光学元件将激光束能量分布转换为其他模式的“光束整形辅助LIBS(Beam Shaping-assisted LIBS，BS-LIBS)”方法。例如，Gao等[25]发现，将高斯激光束转变为涡旋激光束可有效改善LIBS对半导体和金属样品检出限。Yan等[26]利用具有长焦深特性的贝塞尔光束，将LIBS对曲面钢铁样品的分类准确率提高至93.17%。其中，平顶光束的改善效果尤为显著，Ji等[27]成功将平顶光束应用于矿石样品中铀含量的分析，其谱线强度较传统LIBS提高了5倍以上，且相对标准偏差(Relative standard deviation，RSD)降低了约50%。Jia等[28]发现采用平顶光束时，分析元素的谱线强度RSD和检出限均约为高斯光束LIBS的50%，且平顶光束产生的激光烧蚀坑比高斯光束更加平滑。上述改善主要归因于平顶光束更均匀的能量分布，有助于减弱等离子体屏蔽效应并提高烧蚀效率[29]。然而，商用平顶光束整形器(DOE)价格昂贵(超过1 000美元)，且仅适用于具有特定参数的激光束，限制了其推广应用。

综上所述，探索一种经济、高效的光束整形方法，对提高LIBS分析精度具有重要意义。Zehra等[30]详细研究了线状等离子体相对于光谱仪光轴位置的影响，发现利用柱面透镜产生的线聚焦LIBS等离子体对钢中碳含量的检出限改善效果显著优于传统点状等离子体。符小清等[31]采用球面透镜与柱面透镜相结合的方式聚焦，获得了更稳定的等离子体辐射光谱。此外，柱面透镜在激光束参数适用范围和成本效益方面均较DOE具有明显优势。基于上述思路，本文提出一种由柱面透镜与平凸透镜组合的新型光束整形方法，该方法首先通过平凹柱面透镜与平凸柱面透镜组合，将高斯激光束沿单轴方向扩展为线状光束，使激光能量由中心高密度区向外缘扩散，从而获得能量分布更为均匀的光斑；随后，通过平凸透镜将该线状光束聚焦以激发等离子体。经上述两级整形后获得的光束称为近似平顶光束(Approximately Flat-top Beam，AFTB)。本研究分别在高斯光束(Gaussian Beam，GB)与AFTB条件下对比评估了LIBS对钢铁中微量元素的定量分析效果。实验结果表明，所提方法能有效抑制信号波动，提高元素校准曲线的拟合精度，为钢铁材料的高精度元素定量分析提供了技术支撑。

1　实验部分

1.1　实验装置

实验装置示意图见图1，采用调Q的Nd：YAG脉冲激光器(Quantel，Ultra 100)，其工作波长为1 064 nm，脉宽为10 ns，工作频率设为1 Hz。输出的激光脉冲通过旋转半波片(HWP)改变激光的偏振方向后，再通过格兰棱镜(GP)实现对激光能量大小的调节。激光经凹透镜L1(焦距－50 mm)与凸透镜L2(焦距100 mm)组成的扩束系统进行扩束，经反射镜反射后，再通过平凹柱面透镜L3(焦距－50 mm)与平凸柱面透镜L4(焦距75 mm)组成的光束整形器，将高斯光束转换为近似平顶光束(AFTB)，最后通过平凸透镜L5(焦距50 mm)聚焦于样品表面以激发等离子体。等离子体辐射光谱由旁轴光学收集系统聚焦采集，并通过光纤传输至光谱仪(AvaSpec-Mini4096CL，光谱采集范围为186～797 nm，光谱分辨率约为0.05 nm，最小门宽为9 μs)。为对比不同激光束条件下激发的等离子体光谱信号强度及稳定性，本研究对激光能量及离焦量等实验参数进行了优化，其中光谱仪的探测延时和门宽分别设为1.5和9 μs。

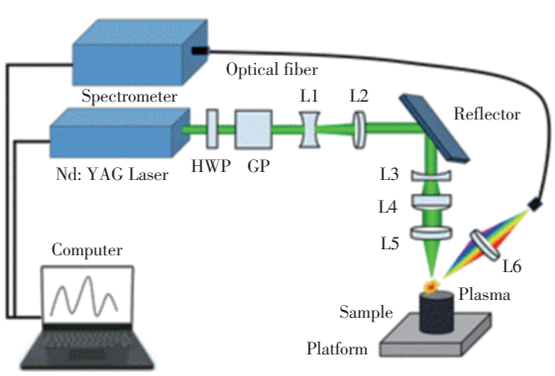


图1　光束整形辅助LIBS实验装置示意图

Figure 1　Schematic of the beam-shaping-assisted LIBS setup.

1.2　实验样品

本实验采用由钢研纳克检测技术股份有限公司和山东省冶金科学研究院股份有限公司提供的9个标准钢铁合金样品。样品中微量元素的含量由ICP-AES法分析所得，如表1所示，其中基体元素Fe的含量均超过90%。本研究选取Mn Ⅰ 403.076 nm、Cu Ⅰ 324.75 nm、Si Ⅰ 288.15 nm、Cr Ⅰ 425.43 nm等钢铁中所含微量元素的特征谱线为研究对象，分析了光束整形方法对光谱强度以及LIBS定量分析精度的影响。为排除样品表面污染物对分析结果的影响，以及确保光谱数据采集条件的一致性，在每个样品表面选取了4个采样点，每个采样点首先经过20次激光脉冲预烧蚀后，再采集后续30次等离子体光谱。因此，每种光束条件下采集的光谱数据共计1 080组(30×4×9)。

表1　钢铁合金标准样品的参考成分

Table 1　Certified reference concentrations of the steel alloy samples　　/%

No.	Si	Mn	Cr	Cu	Fe
1	0.054 0	0.018 0	0.023 0	0.003 6	99.778 4
2	0.077 0	0.104 0	0.042 0	0.049 0	99.169 8
3	1.550 0	0.303 0	0.236 0	0.403 0	95.898 9
4	1.090 0	0.669 0	0.102 0	0.316 0	95.951 7
5	0.769 0	1.040 0	0.106 0	0.248 0	95.651 6
6	0.436 0	1.470 0	0.409 0	0.167 0	95.840 4
7	0.176 0	2.100 0	0.612 0	0.075 0	95.322 2
8	0.174 0	1.040 0	0.194 0	0.017 0	98.267 0
9	0.272 0	0.604 0	0.044 0	0.141 0	98.400 4

1.3　光束整形方法

Liu等[32]利用ZEMAX软件对激光通过由柱面透镜构成的光束整形系统的光路变化进行仿真，结果表明，该系统可成功将圆形高斯光束整形为矩形平顶光束，验证了柱面透镜光束整形方法的可行性。因此，本研究采用平凹柱面透镜、平凸柱面透镜与平凸透镜组合实现光束整形与聚焦，其在X、Y两个相互垂直方向上的激光光路传播ZEMAX仿真结果如图2a所示。由于柱面透镜仅能对单一光轴方向进行扩束或缩放，当平凹柱面透镜与平凸柱面透镜共用同一聚焦轴时，该装置实质上是伽利略扩束器在单轴方向上的变形，其作用是将激光能量沿该轴方向重新分布。图2b为ZEMAX仿真的GB与AFTB光束能量分布示意图，由图2b可知，传统GB光束具有中心能量密度高、边缘能量密度低的特征，这不仅易激发出温度分布不均匀的等离子体，还易在激光-等离子体相互作用区域诱发等离子体屏蔽效应，从而降低激光与样品的耦合效率，而该效应正是影响LIBS信号强度和不确定性的主要因素之一[33-35]。采用本研究所提出的光束整形结合LIBS法(AFTB-LIBS)，GB光束中心的高能量密度可重新分布至边缘低能量密度区域，从而实现更均衡的能量分布，即整形后的AFTB光束有助于获得更稳定、更强的等离子体光谱信号。

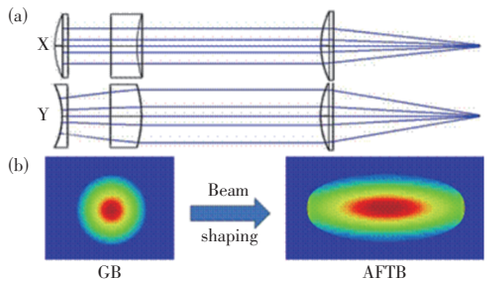


图2 仿真结果：(a)光束整形系统的光路图；(b)GB、AFTB的激光能量分布

Figure 2 Simulation results: (a) Optical path diagram of beam shaping system; (b) Laser energy distribution of GB and AFTB.

2 结果与分析

2.1 实验参数优化

本研究选取4号样品作为参考样品，用于分析激光能量与离焦量等参数对光谱数据质量的影响。本研究首先在离焦量为－3 mm时，对比了不同激光能量(范围：20～55 mJ；步长：5 mJ)对采集的谱线强度的影响。图3a、3b分别展示了LIBS与AFTB-LIBS采集的不同特征谱线强度的变化规律(图中数据点为多次光谱的平均值，误差棒为标准偏差)。

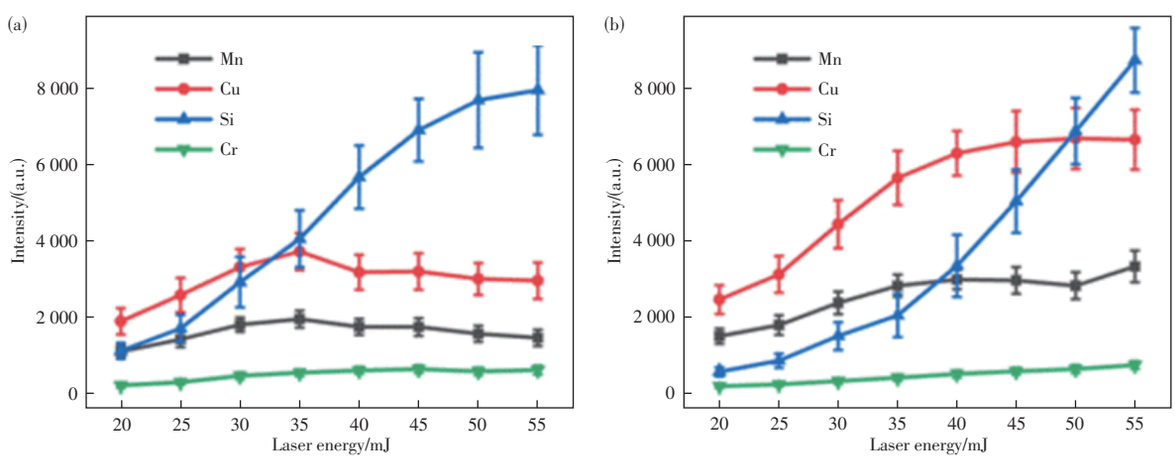


图3 不同激光能量对不同特征谱线强度的影响：(a)LIBS与(b)AFTB-LIBS

Figure 3 Influence of different laser energies on intensities of characteristic spectral lines: (a) LIBS and (b) AFTB-LIBS.

结果表明，非金属元素Si的谱线强度随能量增大呈单调递增趋势，且在55 mJ时AFTB-LIBS激发的Si谱线强度仍未表现出饱和趋势，说明该方法在较高激光能量下具备激发更强光谱信号的潜力；当能量超过35 mJ时，AFTB-LIBS激发的金属元素Mn与Cu的谱线强度也显著优于同等条件下的LIBS谱线强度。相比之下，Cr的谱线强度随能量变化极不明显，这可能是由于4号样品中Cr含量过低(仅0.102%)，致使两种激发方式下的有效激发信号均较弱，侧面表明AFTB-LIBS对高含量或高灵敏度元素的增强效果更为显著。

此外，光谱数据的稳定性也是影响LIBS分析精度的重要指标，图4a为不同激光能量和光束类型条件下金属元素Mn谱线强度的RSD结果：在20～55 mJ，LIBS的信号RSD在40 mJ时达到相对较低值11.97%；而AFTB-LIBS对应的信号RSD在40 mJ时可达最低值8.35%。此外，图4b展示了非金属元素Si的RSD结果，当激光能量超过30 mJ时，LIBS信号RSD显著下降，并在40～45 mJ处维持在较低水平(11.82%～14.60%)；而AFTB-LIBS的信号RSD在20～40 mJ内均超过20%，但在55 mJ时RSD可降至9.67%，展现出优于LIBS的信号稳定性。

Mn与Si谱线强度RSD在高激光能量条件下存在较大差异可能归因于多个因素，如微量元素Si相对较高的浓度(超过1%)以及Si的第一电离能相对较高，导致其难以被充分激发。由于本研究所提的

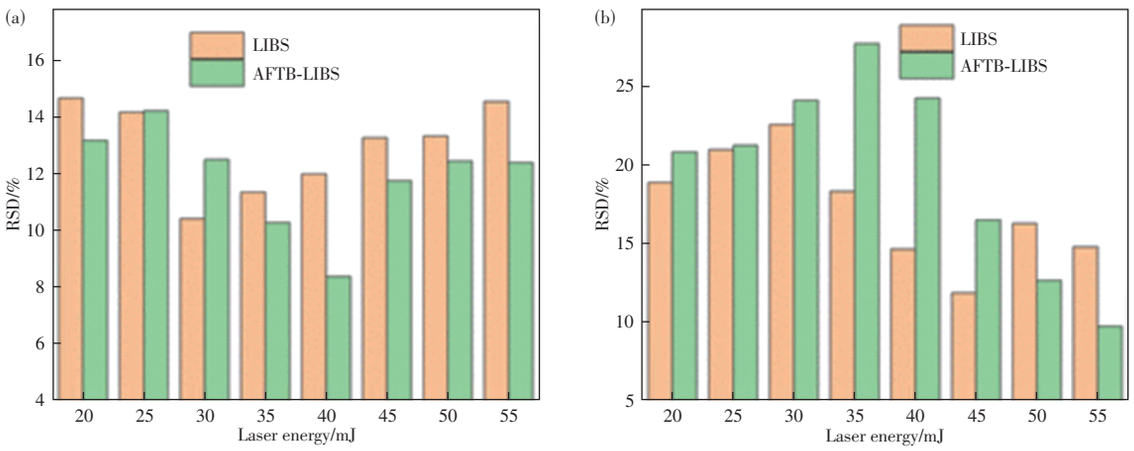


图4　不同条件下信号RSD的变化：(a) Mn I 403.076 nm与(b) Si I 288.15 nm

Figure 4　Variation of signal RSD under different conditions: (a) Mn I 403.076 nm and (b) Si I 288.15 nm.

光束整形法降低了激光束的最高能量密度，因此相比于金属元素，只有当激光能量相对较高时AFTB-LIBS才能激发出Si较强且稳定的光谱强度。

综合图3和图4的分析可知，对于LIBS，在40 mJ时大部分元素的信号强度达到饱和值，并且RSD相对较低，因此选定40 mJ为最佳激光能量；对于AFTB-LIBS，金属元素谱线强度仍未表现出明显的饱和趋势，并且考虑到Si信号的强度及稳定性，故确定AFTB-LIBS的最佳能量为55 mJ。

基于此，进一步优化离焦量。如图5所示，随着离焦量由−5 mm变化至0 mm，不同光束模式下各特征谱线强度均呈现典型的先升后降规律，且在−3 mm离焦量处达到峰值。因此，最终选定−3 mm为最佳离焦量。

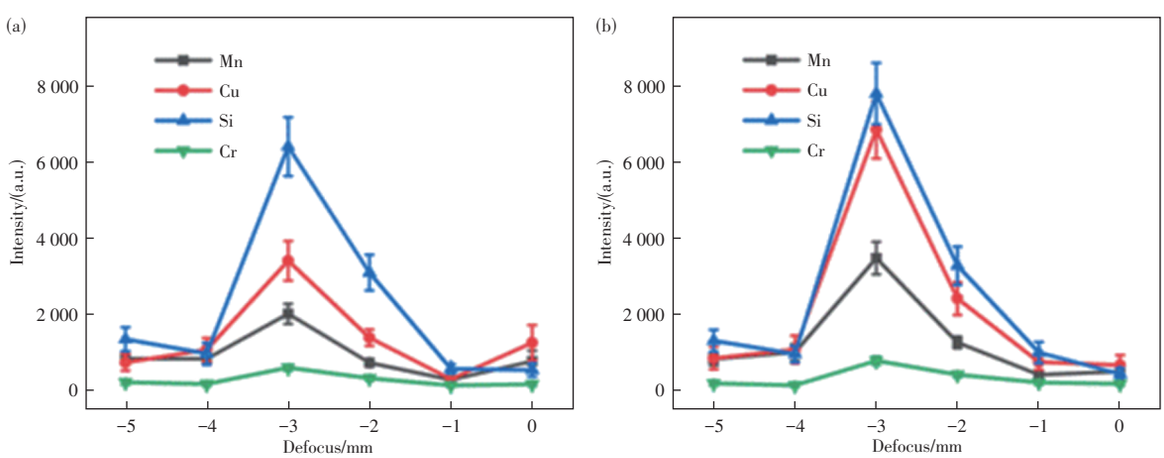


图5　不同离焦量对不同特征谱线强度的影响：(a) LIBS与(b) AFTB-LIBS

Figure 5　Influence of different defocus on intensities of characteristic spectral lines: (a) LIBS and (b) AFTB-LIBS.

2.2　定量分析

图6展示了LIBS和AFTB-LIBS在最优实验参数条件下采集的平均光谱，可以看出，AFTB-LIBS激发的几乎所有特征谱线强度均高于LIBS，说明所提方法对LIBS信号的增强效果。本研究采用了决定系数（Determination Coefficient，R^2）、均方根误差（Root Mean Square Error，RMSE）、平均相对误差（Average Relative Error，ARE）与谱线强度的平均RSD来评估两种方法的分析精度。

图7展示了根据特征谱线强度建立的Mn、Cr、Cu和Si的校准曲线，其中实线、虚线分别为线性、非线性拟合的结果，误差棒表示测量结果的标准偏差，具体的线性定量分析结果列于表2。图7b—7d中LIBS谱线强度随样品浓度增加并未明显增强，甚

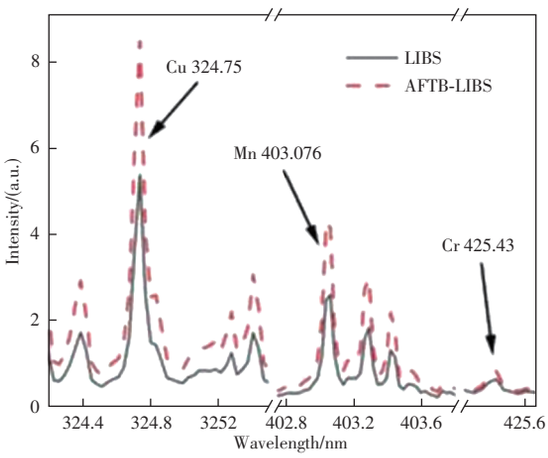


图6 LIBS与AFTB-LIBS采集的平均光谱对比结果

Figure 6 Comparison of average spectra collected by LIBS and AFTB-LIBS.

至出现了下降趋势；而AFTB-LIBS不仅可将Si定标曲线的线性拟合R^2从传统LIBS的0.895 8提升至0.974 1，还可采用非线性拟合将Cu、Si定标曲线的R^2均提升至0.99以上，该结果表明AFTB光束可能有助于抑制导致定标曲线非线性的自吸收效应。

表2的结果表明，与传统LIBS相比，尽管RMSE、ARE等评估指标无明显改善，但AFTB-LIBS激发的各元素谱线强度RSD普遍更低，尤其对原始R^2值较低的Cr和Si具有明显的改善作用。此外，AFTB-LIBS对Cu校准曲线的提升效果尚不显著，其原因可能是Cu在样品中的浓度跨度较小，自吸收效应尚未主导定标曲线，在高浓度梯度下其改善效果或可进一步显现。综上，本研究提出的光束整形方法在改善LIBS定量分析能力与信号稳定性方面展现出良好的应用前景。

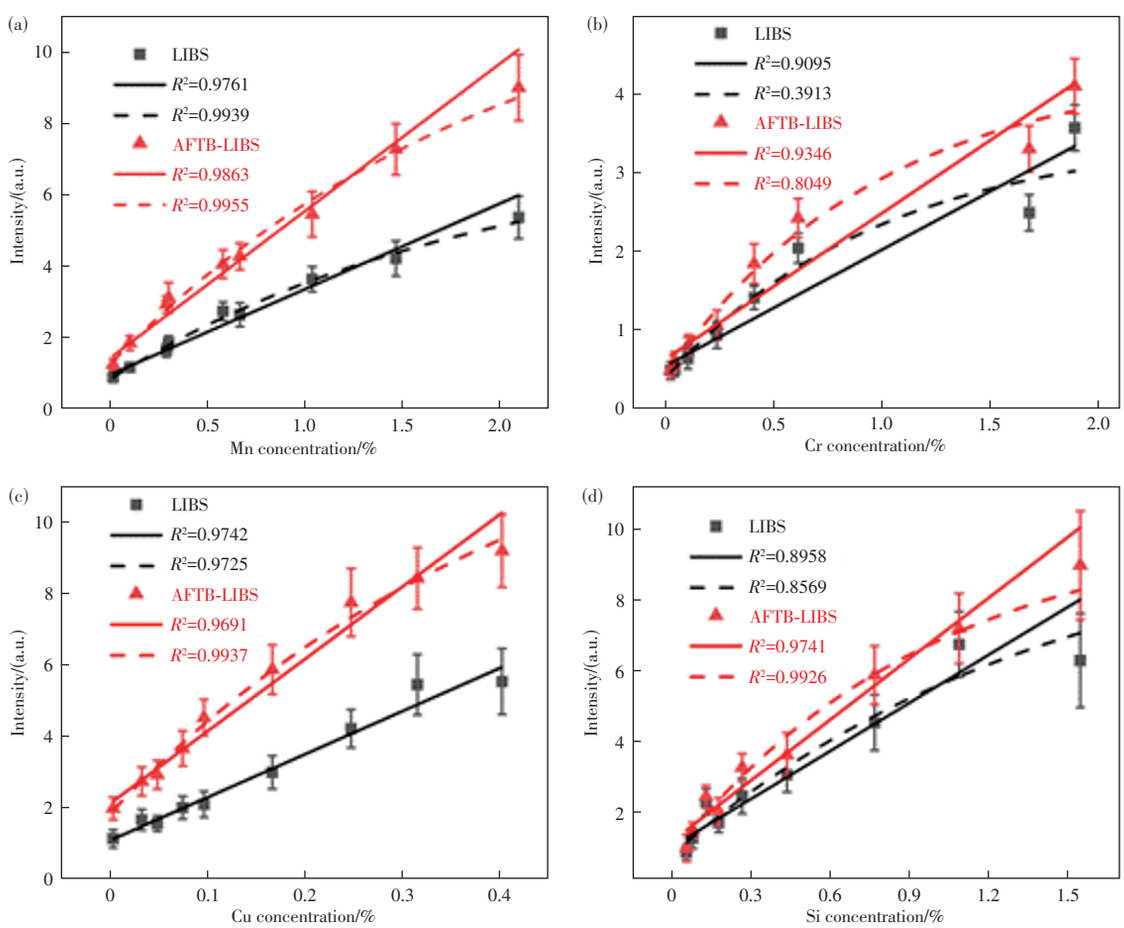


图7 各元素定标曲线结果：(a)Mn、(b)Cu、(c)Cr、(d)Si

Figure 7 Calibrationcurves of (a) Mn, (b) Cu, (c) Cr, and (d) Si for different lasertypes.

表2　LIBS和AFTB-LIBS的定量分析结果

Table 2　Quantitative analysis results of LIBS and AFTB-LIBS

Elements (Spectral line)	Methods	R^2	RMSE/%	ARE/%	RSD/%
Mn I	LIBS	0.9761	0.1006	97.90	11.7
403.076 nm	AFTB-LIBS	0.9863	0.0761	103.53	10.6
Cu I	LIBS	0.9742	0.0210	14.24	16.2
324.75 nm	AFTB-LIBS	0.9691	0.0231	75.45	12.6
Cr I	LIBS	0.9095	0.2036	112.18	14.6
425.43 nm	AFTB-LIBS	0.9346	0.1731	130.86	14.4
Si I	LIBS	0.8958	0.1597	69.33	18.8
288.15 nm	AFTB-LIBS	0.9741	0.0786	50.24	17.7

3　结论

本研究针对传统LIBS采用高斯光束时易诱发等离子体屏蔽效应，从而导致采集的光谱信号稳定性差的问题，设计了一种由平凹柱面透镜与平凸柱面透镜组成的新型光束整形器，将高斯激光束转换为能量分布更为均匀的近似平顶光束AFTB，并评估了该方法对钢铁样品中微量元素的分析结果。结果表明，在优化实验条件下，相较于传统LIBS，AFTB-LIBS激发的各元素特征谱线强度均显著增强，且谱线强度RSD整体较低。AFTB-LIBS建立的各元素定标曲线线性拟合R^2基本有所提高，特别是Si的R^2从传统LIBS的0.895 8提升至0.974 1；因此，采用自主设计的光束整形装置结合AFTB-LIBS方法，能够有效克服钢铁合金基体的光谱干扰，显著提升LIBS技术在钢铁成分定量分析中的准确度与精密度，为工业冶金领域实现钢铁产品的快速、低成本、高精度元素检测提供了有效的技术方案。

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Quantitative Analysis of Steel Components by Laser-induced Breakdown Spectroscopy Combined with a Beam Shaping Device

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Abstract Laser-induced breakdown spectroscopy (LIBS) is a real-time, online technique for material composition analysis. However, conventional LIBS typically employs Gaussian beams with non-uniform energy distribution, which readily induces plasma shielding effects, reducing the laser-material coupling efficiency and severely affecting the stability of spectral signals and quantitative analysis accuracy. To address this issue, a novel beam shaper composed of a plano-concave cylindrical lens and a plano-convex cylindrical lens was designed in this work to reshape the Gaussian laser beam into an approximately flat-top beam (AFTB) with more uniform energy distribution, thereby improving the analytical performance of LIBS. Taking trace elements in steel samples as the research objects, the effects of laser energy and defocusing distance on the spectral characteristics were systematically optimized under both conventional LIBS and AFTB-LIBS conditions. The signal stability and quantitative analysis performance of the two methods were comparatively evaluated based on the intensities of characteristic spectral lines. The results demonstrated that under optimized parameters, AFTB-LIBS significantly enhanced the intensities of characteristic spectral lines for all elements, and the relative standard deviations (RSD) of the spectral intensities were generally lower than those of conventional LIBS, particularly showing remarkable improvements in stability for Cr and Si. Furthermore, AFTB-LIBS effectively improved the quantitative analysis accuracy of trace elements; for instance, the coefficient of determination (R^2) for the Si calibration curve was increased from 0.895 8 (conventional LIBS) to 0.974 1. These findings indicate that the proposed beam shaping method can effectively enhance the stability of spectral signals and the accuracy of quantitative analysis, providing a practical technical reference for rapid, high-precision, and low-cost detection of steel components in the industrial metallurgical field.

Keywords steel; laser-induced breakdown spectroscopy; quantitative elemental analysis; beam shaping

HIGHLIGHTS

1) A laser beam shaping system composed of cylindrical lenses is utilized to improve the uniformity of laser energy distribution.
2) The influence of the beam shaping method on laser-induced plasma spectra was investigated.
3) LIBS combined with beam shaping provides technical support for quantitative elemental analysis of steel.