Using Laser-induced Breakdown Spectroscopy Technique to Identify the Low-Carbon Steel in the Industrial Alloy

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ABSTRACT

Laser-induced breakdown spectroscopy system (LIBS) had been designed for analysis of the industrial alloy via analysis induced plasma emission, laser spectroscopy to plasma produce one of the applications resulting from the interaction of the laser beam with material. The design consists of Nd:YAG laser passively Q-Switched with output energy (50–mJ @ 1064 nm) and (9 ns) pulse duration the laser beam was focused via converging lens with a focal length (100mm) that generates (7.07*10⁸ W/mm²) power intensity, optical analysis system was used that operated on the analysis of plasma light resulting from the interaction of lasers with the target, within spectrum range of 320–740 nm and (0.5nm) optical resolution. The analysis results obtained show a variety of metallic elements in the industrial alloy, the results obtained by using X-ray fluorescence system (XRF), and other spectral references have been compared. LIBS have shown detection of the main elements in the sample with other elements are manganese, aluminum, sulfur, phosphorus, silicon, and carbon, these elements not identified in the XRF.

Key words: Laser-induced breakdown spectroscopy LIBS; XRFs; Plasma; Low-carbon steel.

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INTRODUCTION

The low-carbon steel is a steel alloy consisting of iron and carbon. Several other elements are allowed in alloy, with low-maximum percentages. They are manganese, silicon, and copper, and rare-earth elements to improve the physicochemical properties, such as (Al, Co, Ti, S, P, Cr, W, etc.), and other elements can be found in (F. Cverna, P. Conti, A.S.M. Committee; 2006, T. Proulx; 2011). There are four types of carbon steel based on the amount of carbon in the alloy. Lower carbon steels are softer and more easily formed, it too important alloy that have widely used in industry. Laser-induced breakdown spectroscopy (LIBS) is a type of atomic emission spectroscopy which uses a highly energetic laser pulse as the excitation source (A.W. Miziolek; 2006, D.A. Cremers; 2013), that have been reported for the first time in 1962 (F.C. Brech; 1962), evolved since then to the technique of chemical analysis (F. Anabitarte, A. Cobo, J. Lopez-Higuera; 2012).

LIBS technique that uses a high-intensity laser pulse to generate the plasma, the target can be solid, or liquid, or gas. A laser pulse of high-peak power is focused on the sample surface, transporting a large amount of energy to the target, begin temperatures to rise, melts, evaporates, and ionization of materials on the surface, this leads to the formation of hot small plasma. Due to the high intensive energy on target surface the plasma begins to expand and formed by ionized gas, during the period excited atoms begins to relax back to ground level, and then light-emitting unique called plasma spectrum emissions. Because of all element of the periodic table has a spectral fingerprint especially that can be distinct from the other materials (M. Reference; 2007), and then subsequent material is determined by the optical detect devices.

This technique tremendous achievements in laser technology and reagents has achieved that later became the effective techniques for the analysis of metals (R. Barbini, F. Colao, R. Fantoni, A. Palucci, F. Capitelli; 1999), boosted it the great development in their ability to quantitative and qualitative analysis to a wide range of material (A. Stankova, N. Gilon, L. Dutruch, V. Kanicky; 2010, Z. Abdel-Salam, J. Al Sharnoubi, M.A. Harith; 2013), and these include metal alloys (M. Dzjubenko, S. Kolpakov, D. Kulishenko, A. Priyomko; 2007), soil (S.C. Jantzi, J.R. Almirall; 2011), explosives (J.L. Gottfried, F.C. Lucia, C.A. Munson, A.W. Miziolek; 2009), rocks and sediments (I. Rauschenbach, V. Lazic, S. Pavlov, H. Hubers, E.K. Jessberger; 2008, W. Tawfik; 2007), as well as many other applications, making this primarly technology in terms of the multiplicity of advantages, including ability to analyze materials various (B.C. Windom, P.K. Diwakar, D.W. Hahn; 2006), non-destructive (J.P. McDonald, D.K. Das, J.A. Nees, T.M. Pollock, S.M. Yalisove; 2008), because it removes a very tiny amount of up to tens or hundreds of Nano-grams and has a high sensitivity and rapid response in detection (A. Stankova, N. Gilon, L. Dutruch, V. Kanicky; 2010), no sample preparation is required to obtain useful results (P. Dewalle, J.B. Sirven, A. Roynette, F. Gensdarmes, amp, amp, ois, L. Golanski, S. Motellier; 2011), relatively inexpensive compared with other conventional techniques, analysis on-site (D. Wilsch, F. Weritz, H. Wiggenhauser; 2003), and laboratory.

In this paper will be devoted particularly in the design and construction of LIBS system for the analysis of the industrial alloy (low-carbon steel), during the emission spectrum of plasma in the air generated from the basic waveforms passive Q-Switched Laser Nd: YAG emitted from solid targets.
**Methodology**

The analysis of environmental conflict is the foundation for understanding the sensitivity of the conflict itself. It aims at understanding the interactions between the conflicting parties and those who assist them in resolving the dispute (Jantzi & Almirall; 2011), and ignoring such analysis could intensify the complexity of the current situation. Subsequently, for the sake of finding a solution, this study focuses on the transboundary wastewater pollution conflict between Israel and the oPt. There are many methods and procedures for addressing environmental conflict, including but not limited to, negotiation, mediation, diplomacy, which eventually would provide general solutions and guidelines on how the conflict could be resolved.

But since each case has its own specificity, these solutions will not be applicable on all cases. Accordingly, this study follows an approach in analysing the conflict; where the conflict history, causes, nature, and parties have been identified in the introduction and the case study sections. The conflict roots alongside the obstacles that prevented reaching a solution were analyzed by concentrating on the conflict level, its impact on human beings’ lives, and the technical, social, and political aspects; as well as the parties’ interests in this continuing conflict. Furthermore, a conflict map has been used as a tool for analysis; this tool is based on defining the parties by using circles in which the circle size indicates the party’s power. The circles are linked by lines where the shape of the line and its size indicate the nature of the link between the parties. In order to reach a win-win result, Stakeholder Analysis (STA) is used to determine the needs of conflicting parties who have ‘stake’ and interest in reforms. It is essential to have information about stakeholders’ interests and willingness to support the solution, thus ensuring the adaptation of realistic and sustainable policies (Jantzi & Almirall; 2011). In the STA part, stakeholders’ matrix, engagement towards conflict, and responses to the conflict were used as tools to understand their positions, their relationships with other groups, and their desire to find suitable solutions. In the last section, win-win solutions were developed based on the analysis of the conflict and of stakeholders, which helps in identifying possible negotiable strategies with the conflicting stakeholders.

**Experimental Setup**

The experimental setup Figure 1, is equipped with a high-power Nd:YAG laser passively Q-switched that yields 50-mJ of pulse energy at the fundamental IR wavelength (1064 nm) with a 9 ns pulse width, the fundamental diameter of the laser beam was (5mm) that is focused onto the sample by a plano-convex lens with a focal length of (10cm), the diameter of the focused on the sample was (1mm), peak power of the laser pulse (5.56MW) and power intensity (7.07*10^8 W/Cm^2). The sample is placed in the sample holder in ambient atmosphere, the emission from plasma is then collected in front of the plasma with observance to the laser beam direction, plasma emission was collected by 15mm diameter imaging lens, and focused onto optical fiber type (SMA, 50μm/0.22 NA), which deliver the plasma light to the entrance slit of spectrum analyzer model (CCS-100) with (1200 Line/mm) grating and 20-μm slit dimension, Which serves to deflect light according to wavelength and then reversed by mirrors to detect and convert optical signals to digital, and then moves the digital signal to the application, which shows us the spectral lines for the materials and then analyzed.
Before the sample test in LIBS system, were analyzed by XRFs, for the purpose of the determination type elements in the sample. The industrial alloy used in our experience of the type of low-carbon steel (galvanized), dimensions 3.5*4.5cm. So that the sample size is sufficient to cover an area of X-ray detector to be analyzed. As shown in figure 2, and table.1 shows elements that have been identified in the industrial alloy, by XRF.

Figure 1. Schematic diagram of LIBS experimental setup, and Figure 2. The sample used in our experiment, low-carbon steel.

**Analysis Sample**

The analysis sample is low-carbon steel by technique of LIBS. The composition of low-carbon steel as shown in Table 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>λ (nm)</th>
<th>Sample</th>
<th>λ (nm)</th>
<th>Sample</th>
<th>λ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>327.8548</td>
<td>Cr</td>
<td>518.459</td>
<td>W</td>
<td>636.235</td>
</tr>
<tr>
<td>Si</td>
<td>492.5283</td>
<td>520.6038</td>
<td>Cu</td>
<td>329.99</td>
<td>660.2328</td>
</tr>
<tr>
<td>Ti</td>
<td>334.1874</td>
<td>393.3222</td>
<td>C</td>
<td>566.894</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>399.5308</td>
<td>472.816</td>
<td>589.2021</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>463.172</td>
<td>481.8052</td>
<td>610.766</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>467.9028</td>
<td>500.1863</td>
<td>668.879</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>394.4006</td>
<td>369.152</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Results and Discussion**

Based on the spectral lines of the elements of pure metal has been analyzed in the LIBS, and compared with National Institute of Standards and Technology (NIST) atomic spectra database (A. Kramida, Ralchenko; 2013), the elements have been identified in the sample, which the matched with XRF, table. 2 shows elements that have been identified in the industrial alloy, by XRF. LIBS has been used to determine the elements in the sample, Figure. 3 shown typical LIBS spectrum in different wavelength range and table 3. elements that have been identified in our LIBS. All the elements in the sample may identified in LIBS. In addition, other elements were identified, is not specified in the XRF, the elements are (Al, Mn, S, P, Si, and C). Table 3. Illustrates the elements that have been identified in the XRF with the LIBS, compared with NIST. The rates of the difference between the results obtained are very few. The detected elements in the sample by the LIBS technique are the 12 elements. In the XRFs has been identified six of the elements. As in Table 2, XRF has a range of disadvantages; these
disadvantages make them unable to identify some elements. The main disadvantage of the XRF method is that it is not suitable for analysis of very small samples (B. Beckhoff, B. Kanngießer, N. Langhoff, R. Wedell, H. Wolff; 2007), but XRF is limited to the analysis of relatively large samples (typically >1 gram), the samples must be in powder form and effectively homogenized, XRF is used for the analysis of materials for which compositionally similar, well-characterized standards are available, and materials containing high-abundances of elements for which absorption and fluorescence effects are well understood (V. Rai; 2011). Portable XRF systems are available but suffer from an inability to detect elements with an atomic number below 12 and greater than 92 (J.W. Robinson, E.M.S. Frame; 2004), and show an interference effect that can mask the analytic elements. Light elements (below 19 K) have very limited sensitivity, because they have higher detection limits ranging from the hundreds to thousands of ppm (J. Girard; 2013, D. Bakken, S. Grids; 2014). Although X-rays do not penetrate the surface and hence XRF is a nondestructive technique the deep regions cannot be interrogated, but with LIBS is possible.

![Figure 3. LIBS spectra recorded from low-carbon steel](image)

**Table 2. Analysis of concentrations of elements in sample" low-carbon steel"**

<table>
<thead>
<tr>
<th>Element</th>
<th>Content %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium (Ti)</td>
<td>1.1245</td>
</tr>
<tr>
<td>Chromium (Cr)</td>
<td>0.6277</td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>50.9412</td>
</tr>
<tr>
<td>Cobalt (Co)</td>
<td>0.4973</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>4.225</td>
</tr>
<tr>
<td>Tungsten (W)</td>
<td>42.5844</td>
</tr>
</tbody>
</table>

**Table 3. Analytical lines of “low-carbon steel” elements between the measurement and reference.**

<table>
<thead>
<tr>
<th>No</th>
<th>Measured (\lambda) (nm)</th>
<th>NIST (\lambda) (Reference – measured)</th>
<th>(\Delta\lambda) (Reference – measured)</th>
<th>XRF elements</th>
<th>Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>327.8548</td>
<td>327.8548</td>
<td>0</td>
<td>-</td>
<td>Mn I</td>
</tr>
<tr>
<td>2</td>
<td>329.99</td>
<td>330.0881</td>
<td>0.0981</td>
<td>Cu</td>
<td>Cu II</td>
</tr>
<tr>
<td>3</td>
<td>334.1874</td>
<td>334.1874</td>
<td>0</td>
<td>Ti</td>
<td>Ti I</td>
</tr>
<tr>
<td>4</td>
<td>393.3222</td>
<td>393.3268</td>
<td>0.0046</td>
<td>Cu</td>
<td>Cu II</td>
</tr>
</tbody>
</table>
CONCLUSIONS

LIBS is low cost and high-efficiency system to analyze the industrial alloy compared to all other technologies of analysis. Via XRF we cannot analyze all component of (low-carbon steel) because of the limited elements detection of this system, light elements below 19 K have very limited sensitivity, although the X-rays do not penetrate the surface and hence XRF is a non-destructive technique deeper regions cannot be interrogated which is possible with LIBS. LIBS have shown high susceptibility to identify the following elements (manganese, copper, titanium, aluminum, cobalt, sulfur, phosphorus, iron, silicon, chromium, carbon, and tungsten). LIBS it's a quick way to analyze the material, non-destructive at the same time, and no sample preparation is required to obtain useful results.

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REFERENCES


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