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Optimization of parameters for depth resolution of galvanized steel by LIBS technique

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ABSTRACT

In this study laser induced breakdown spectroscopy (LIBS) is applied for depth profile analysis of galvanized steel sheet widely used in automobile industry. The effects of laser processing parameters, such as laser energy, laser spot size, and coating thickness were investigated to find out depth resolution by applying Taguchi optimization method. According to confirmation experiments of using optimum parameters, it is found out that experimental results are compatible with Taguchi Method with a 83% rate. Numerical simulation of the heat transfer equation is also performed for single laser pulse irradiation since the physical phenomena involved in the laser induced process can be more accurately studied by investigating the effect of single pulse irradiation on the target surface.

1. Introduction

Importance of surface analysis and characterization was increasing with improvement of modified materials. New technological developments such as modification of materials physical and chemical properties by changing their surface structure require suitable methods for depth profile analysis. Several different techniques such as secondary ion mass spectrometry (SIMS) (with a focused primary ion beam and collecting and analyzing ejected secondary ions) [1,2], Auger electron spectroscopy (AES) [3], X-ray photoelectron spectroscopy (XPS) [4], glow discharge optical emission spectrometry (GD–OES) and mass spectrometry (GD–MS) [5,6] are used to investigate compositional depth profiling of multilayered metal materials. Most of these techniques are limited due to several reasons such as restriction of sample shape or nature, long time requirement for analysis or poor resolution [7]. However, LIBS is a powerful tool for depth profiling of layered materials and sensitive to most of the elements in the periodic table. Moreover, it can perform a complete fast analysis in air at atmospheric pressure without limitations in sample size and nature [2,7].

The LIBS technique has been used in a wide range of divåerse application such as military, home security, ecology, and environmental applications. Oztoprak et al. [8] used LIBS to analyze the amount of diffusion depth of stellite powder into copper plate. They investigated the effect of laser peak power and pulse duration on the quality of laser welding processes. Peng et al. [9] investigated the applicability of the LIBS technique for rapid and simple detection of heavy metals in used batteries, which are hard and time consuming to analyse them by the conventional methods. As further improvement, Elsayed et al. [10] designed and built a passive, Q-switched pulsed, Nd:YAG laser system providing a potential compact robust laser source for portable laser induced breakdown spectroscopy systems.

The depth resolution is defined as the depth range over which a signal decreases (or increases) by a specified amount when profiling an ideally sharp interface between two media. The depth resolution corresponds to the depth range over which 84 to 16% (or 16 to 84%) change in the full signal is measured [11,12]. Novotny et al. [2] reported that the emission signal increased with the higher laser energy. This fact can be attributed to the increase in the amount of ablated material. Tokarev et al. [13] describes the higher AAR (Average Ablation Rate) values associated with the thinner coatings by the three-dimensional plasma expansion that takes place on the surface and in the shallow craters, which implies a more significant material removal. For thicker coatings, the plasma expansion inside the deep crater becomes one-dimensional, and the laser beam attenuation provokes a little lowering of the AAR. Coedo et al. [14] concluded that spot radius is directly related with crater formation. Different research groups studied

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depth profiling of coated samples using the LIBS technique in order to obtain AAR and high depth resolution. Some of these groups dealt with the effect of process parameters on depth resolution. Crater shape affects the efficiency of material removal, can affects particles as they escape from the crater and can even contribute to the ablation process itself. Coedo et al. [14] investigated the influence of laser energy on depth resolution for copper coated steel and attained the best depth resolution in between 2.5 and 5 GW/cm^2 of irradiance level. They also showed the AAR values increase slightly when the laser energy increases. åThey found out minimum AAR as 0.77 μm/pulse for 0.4 mJ/pulse. Vadillo et al. [15] measured the depth resolution for zinc coated steel between 400 and 800 nm per pulse by variation of the AAR and depth resolution vs fluence and thickness. Tereszchuk et al. [16] concluded that the higher resolution values are attainable as laser energies are reduced to a value near the ablation thresholds of the material. Vadillo and Laserna [17] have shown that high laser repetition rate is required to obtain the analytical signal. Coedo et al. [14] and Tereszchuk et al. [16] have studied in order to obtain the best depth resolution, the variation of Δz with the pulse energy and the crater diameter. Novotny et al. [2] concluded that the delay time appears to be another critical factor in-depth profiling by LIBS. They pointed out that delay time affects the tailing of the zinc emission signal. Novotny et al. [2] and Vadillo et al. [15] investigated the effect of both focal condition and properties of the media atmosphere on depth profile. åThey reported that the focus position must be below the sample surface to obtain significant maximum intensity in air. St-Onge and Sabsabi [18] indicated that a non-uniform radial energy distribution such as with a Gaussian laser beam can lead to a loss of depth resolution due to formation of a bell-shaped crater. Coedo et al. [14] have shown linear dependence of depth resolution with coating thickness. Afkhami and Tavassoli [7] also investigated the effect of copper and zinc thickness on AAR and Δz. They found that AAR values decrease when the coating thickness increases. Gragossian et al. [19] made a theoretical and experimental study on the ablation rate for different intensities. Presence of different material in sample such as aluminium, iron, manganese and phosphorous affects the ablation rate. Novotny et al. [2] concluded from the study on influence of surrounding atmosphere on ablation rate that the lowest ablation rate was observed in an air atmosphere.

One of the main problems in depth profile analysis to obtain higher depth resolution is to select the most appropriate parameters affecting it. Vaporization, atomization, ionization, formation of molecules and fragments, shock waves, plasma initiation and expansion, and a hybrid of these and other processes influence depth profile [17]. When the solid target is irradiated by laser beam, ablation process starts as a result of surface absorption of the radiation. The temperature at the target surface increases and this is followed by melting and evaporation of the target material. The heat conduction equation is used for modelling of the heating process. Although, the laser processes are often applied by multi-pulse irradiation, the physical phenomena involved in laser induced process can be more accurately studied by investigating the effect of single pulse irradiation on the target surface, since first laser pulse strongly determines the surface absorption and plasma formation threshold for the subsequent laser pulses [20–24]. Beside experimental investigation, numerical modelling can give better insight into the mechanism of laser induced structure formation on solid surfaces. On the other hand, laser energy, laser spot size and coating thickness are important parameters for these investigations. For instance, inadequate laser intensity [18], non-uniform energy distribution [18,25] reduce depth resolution. Effective parameters have to be controlled to obtain higher depth resolution (smaller Δz). Determination of the parameters on depth resolution by classical experimental design methods takes long time and high cost. Taguchi Method, which decreases number of trials, is used in many areas such as science, engineering, biotechnology, marketing and advertising by researchers to avoid from previously mentioned disadvantages [26–32].

The aim of this study is to obtain high depth resolution using high resolution spectrometers coupled to a nanosecond Nd:YAG laser. Determining optimum conditions to obtain higher depth resolution, Taguchi method was chosen to perform the experiments. For the analysis of experiments, calculated signal-to noise (S/N) ratios were used with the approach of "smaller the better". Many parameters such as energy, spot size, coating thickness, focal position, repetition rate, delay time, properties of media atmosphere, energy distribution and kind of material affect AAR and Δz in LIBS analysis. The Taguchi techniques are widely applied åin material processing and analysis [28,33,34]. In this work effects of laser parameters were investigated by the Taguchi method to reduce the time and experimental costs for LIBS applications. åIn this work, the process parameters affecting the performance of experiments were determined and the factors such as coating thickness, pulse energy and spot size affecting these parameters were investigated to define the best conditions. The effect of ablation process by single laser pulse irradiation was also studied by numerical calculation using heat conduction equation to get material melting and evaporating conditions.

2. Experimental and analysis method

2.1. Material and experimental setup

In depth profile analysis, zinc coated (galvanized) iron sheets were ablated by focusing a Nd:YAG laser beam with a 150 mm focal length lens as shown in Fig. 1. The laser operated at 1064 nm wavelength with a pulse width of 4.4 ns, beam diameter of 6 mm and repetition rate of 20 Hz. The laser pulse energy was measured by a power meter (841PE, Newport). As it is reported by Coedo et al. [14] focusing above the surface is not suitable for depth profile analysis because of the shielding by secondary plasma. Laser spot size was changed by moving the position of focusing lens. Therefore spot radius was changed in between 160 and 320 μm by adjusting the distance between sample and lens. åThe beam waist can be obtained from

$$w_0 = 1.22\frac{\lambda f}{d}$$

where f is the focal length of lens, and d is laser beam diameter at lens.

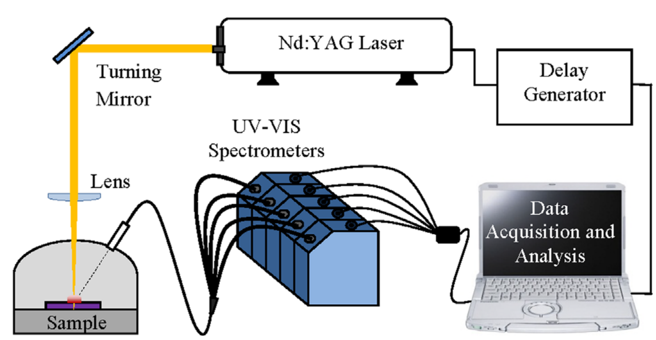

Fig. 1. The experimental LIBS for analysis of galvanized steel sheets set up. Plasma image at is transported to spectrometers via fiber bundle for 5-channel. Signals are collected in computer and spectrum is obtained.