THE IMPACT OF CONTROLLED EN 10083-2: C45 STEEL SURFACE OXIDATION ON NIR LIGHT ABSORPTION

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Abstract

An increase in surface absorptivity can be helpful with a number of laser heat treatment processes. Absorptivity can be increased by, for example, absorption coating or surface oxidation. The method of controlled laser oxidation was developed after a series of experiments. A scanning laser system was used to create an oxide layer on steel EN 10083-2: C45 surface. Subsequently, the absorbency and thickness of the formed oxide layer were measured, depending on the irradiation energy emitted into the surface. It has been found that higher the thickness of the oxide layer increases absorption, but the growth is not linear. For the wavelength of 808 nm, there is a local absorption maximum for irradiation energy around 2 J/mm^2. The wavelength of 808 nm was taken as a reference because we own a laser for heat treatment emitting at that wavelength. These results have shown that the use of surface oxidation increases the coupling of laser energy to material surface in the order of tens of percent. This advantage can be used to process complex workpieces where individual components need to be processed with different parameters.

Keywords: Oxide layer, NIR absorption, IR radiation, surface oxidation, controlled heating

1. INTRODUCTION

The formation of the oxide layer on the surface of the material results in a change of its properties as a whole. Of course, the oxide layer has different electrical, optical and mechanical properties. This paper will be focused especially on the change of optical properties. The incident, reflected, absorbed, and transmitted fluxes are connected together by energy conservation law [1]. From this fact can be deduced that the sum of optical properties reflectivity (ρ), absorptivity (α), and transmissivity (τ) equals one:

$$\rho + \alpha + \tau = 1 \tag{1}$$

For an opaque object without transmission (τ = 0) the relation (1) simplifies

$$\rho + \alpha = 1 \tag{2}$$

Kirchhoff's law provides a relation between the absorption and emission processes and thus between emissivity and absorptivity

$$\varepsilon = \alpha \tag{3}$$

These relations are valid for similar spectral or total spectral condition. In this case, there are three sets of optical properties (three different wavelengths) for that Kirchhoff's law, is valid. The three wavelengths are the wavelength of engraving laser 1064 nm, the wavelength of hardening laser 808 nm, and the measuring range of infrared camera 1 μm.

The effect of the coating applied to the surface of the material on laser absorption is described in [2], the measurement showed that the absorptivity of graphite absorber at the specimen surface changes with reference to the absorbing coating thickness. This means that an optimum coating thickness depending on the type of absorber and the laser hardening conditions exists. The optimum coating thickness of the graphite absorber was determined in the range of 32 to 35 μm. The influence of the thickness of the absorptive coating on the parameters of the hardened structure was investigated in [3]. Results showed that an optimum range of coating thickness which produces a large hardened layer dimension could be selected. This range becomes smaller as the hardening velocity increases.

A sequential method of surface absorption determination in the laser quenching process is described in [4]. This paper presents an efficient algorithm for determining the surface absorptivity in the process of laser hardening. A regression model for the reflectivity coefficient evaluation in laser surface hardening is presented in [5]. The model is numerically calculated by comparing the actual surface temperature to the theoretical prediction obtained by process simulation. Paper [6] describes the numerical-experimental analysis of the surface oxidation effect on the laser hardening. The model is able to offer the temporal course of the oxide thickness and the calculation of the increment of the absorption as a consequence of the development of the oxide layer. The relative error of this method for determining the maximum temperature is less than 5%.

Two different mechanisms of radiative absorption for the surface oxidation material were described in [7]. The thickness of oxide layer influences the absorptive mechanism. The oxide layer of low thickness (in the order of 100 nm) has high transmission and absorption on the surface of the underlying material dominates. The absorptivity of oxide layer increases with it's growing thickness. Absorption through the oxide layer is predominant for thicker layers (in the order of 500 nm or more) of oxide.

The phenomenon called total reflection [8] can occur for a certain thickness of the oxide layer and the given value of the wavelength of the incident light.

An increase in the surface absorption of the material can be used, for example, in the laser quenching process [9], especially for precise heat treatment [10].

The aim of the paper is the creation of different oxide layers with variant influence on the absorptivity of laser radiation with 808 nm wavelength.

2. EXPERIMENTAL PROCEDURE

2.1. Surface modification process

Tests were performed on a cylindrical sample from rolled steel EN 10083-2: C45 and with a diameter of 25 mm and height of 5 mm. Oxide layers were created by marking laser SPI-G3-SP-20P with MOPA arrangement. Parameters, to heating but not melting the surface, were chosen. The scanning speed of the laser beam and the laser power were chosen common for all samples. The power was set to 17.25 W and the value of 800 mm/s was selected for scanning velocity. The depth of the oxide layer depends on fluence used for area treatment. The fluence characterizes the amount of radiant energy emitted into defined area unit. The quantum of the radiant energy was controlled by hatch density and allows operated growth of oxide layer. It means that it is controlled by the value of ls distance between laser crossings. A longer period of interaction is needed for higher fluence. The manner of heating the treated surface layer by the laser is featured in **Figure 1**. The laser beam is quickly deflecting in the scanning direction from one side to the other and the speed of advance motion specifies the amount of energy irradiated the sample surface, fluence value. 8 different oxide layers were made overall.

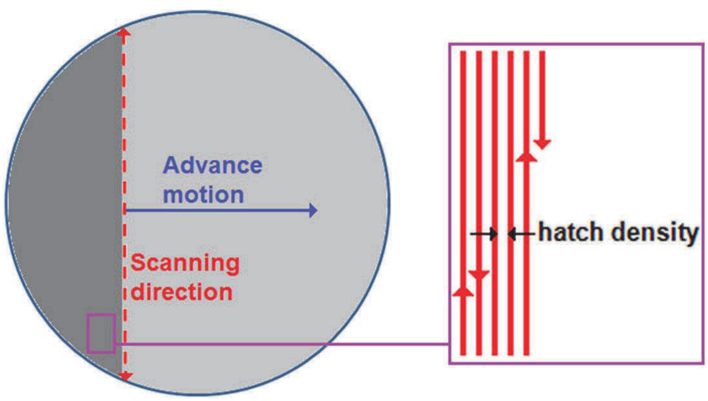

Figure 1 Principle of controlled oxidation method

The method of measurement of spectral normal hemispherical reflectivity at room temperature was applied. FTIR spectrometer Nicolet 6700 and UV/VIS spectrophotometer Specord 210 BU were used for absorptivity measurement.

The thickness of the oxide layer was measured by scanning electron microscope Analytical SEM Hitachi SU-70 (**Figure 4**). The groove was created on the sample surface to cut through the oxide layer. Then the thickness of layer's fragments was measured. The finding of the correctly positioned fragment was considerable with regard to measurement's precision. The measured sample was made of magnetic steel, so it was necessary to switch off the immersion optics so that the measurement was not influenced by the magnetic field of the sample. The thickness of the fragment was assessed in the graphic editor by pixel counting and this value was multiplied by pixel size obtained in additional ASEM measurement file.

The surface roughness of the samples was measured on KLA-Tencor P-6 profilometer. The roughness was measured on 5 mm long trajectory.

3. RESULTS AND DISCUSSION

Absorptivities of the 8 layers were measured in the ultraviolet (UV), visual (VIS), near-infrared (NIR) and mid-infrared (MIR) region. Values of absorptivity enrolled in **Table 1** were measured at wavelength 808 nm. Note the absorptivity was increased in the range of 20%. The increase is caused by the growing thickness of oxide layer.

Table 1 Oxide layer - parameters of the layer

Fluence [J/mm^2]	0.0	0.3	1.5	2.5	6.0	15.0	30.0	50.0
Absorptivity$_{808}$ [%]	70.2	75.3	88.0	87.1	85.4	86.7	87.2	88.3
Layer thickness [nm]	50	125	140	320	295	580	630	945
Parameter of roughness R_z [μm]	17.3	28.6	30.4	27.4	26.3	32.1	24.7	21.4

Note that no high difference between roughness values in relation to created oxide layer was measured. It could be caused by high surface roughness of substrate material.

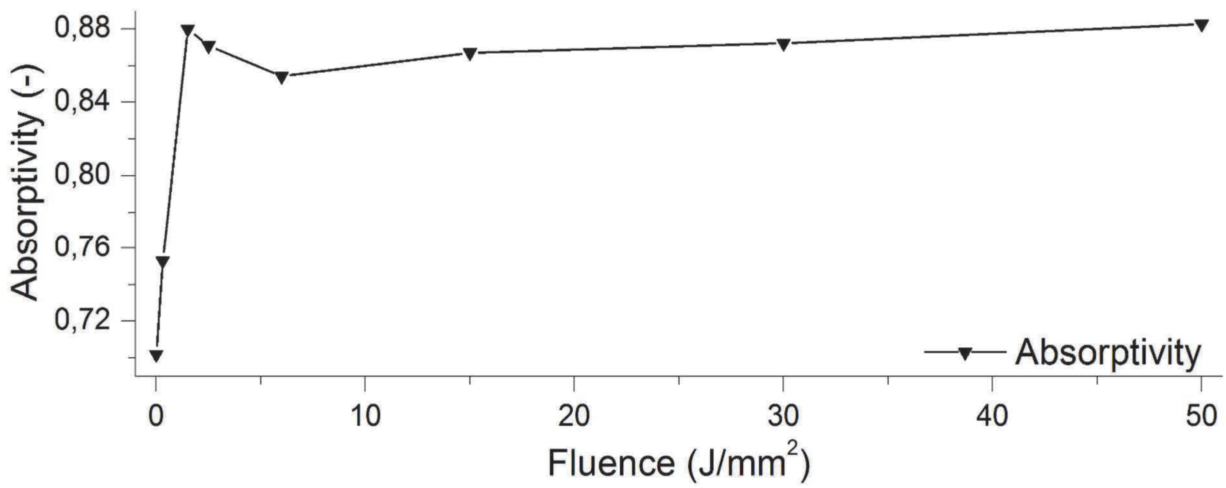

Figure 2 Graph of the absorptivity measurement

While the thickness of oxide layer grows linearly in relation to fluence (**Figure 3**), the absorptivity does not (**Figure 2**). Specific properties of given oxide layer can take effect. Total reflection phenomenon can occur and cause the oxide layer with specific parameters has higher absorption than the others. The phenomenon could fulfill for fluence around 2 J/mm^2 the increase for higher fluencies around 30 - 50 J/mm^2 is caused by growing absorptivity of the oxide layer.

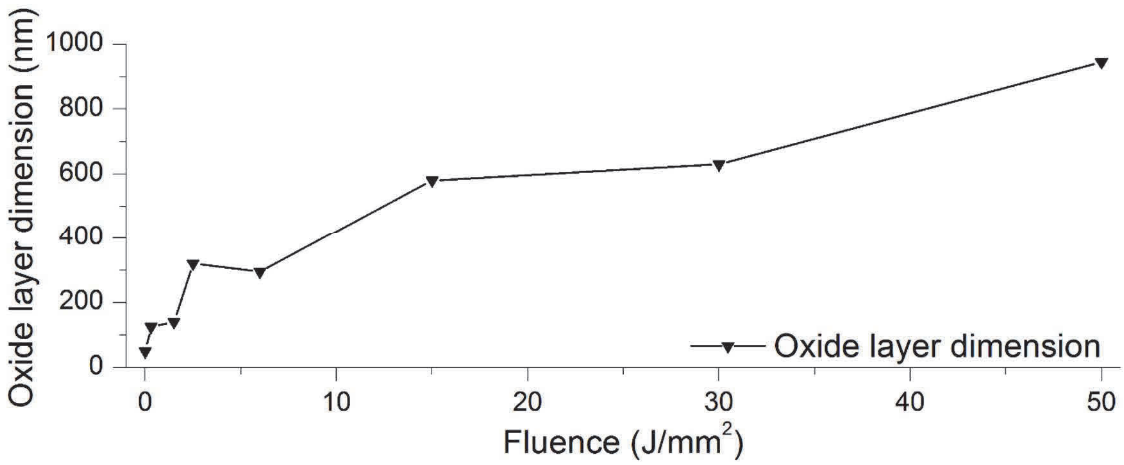

Figure 3 Graph of the thickness of oxide layer

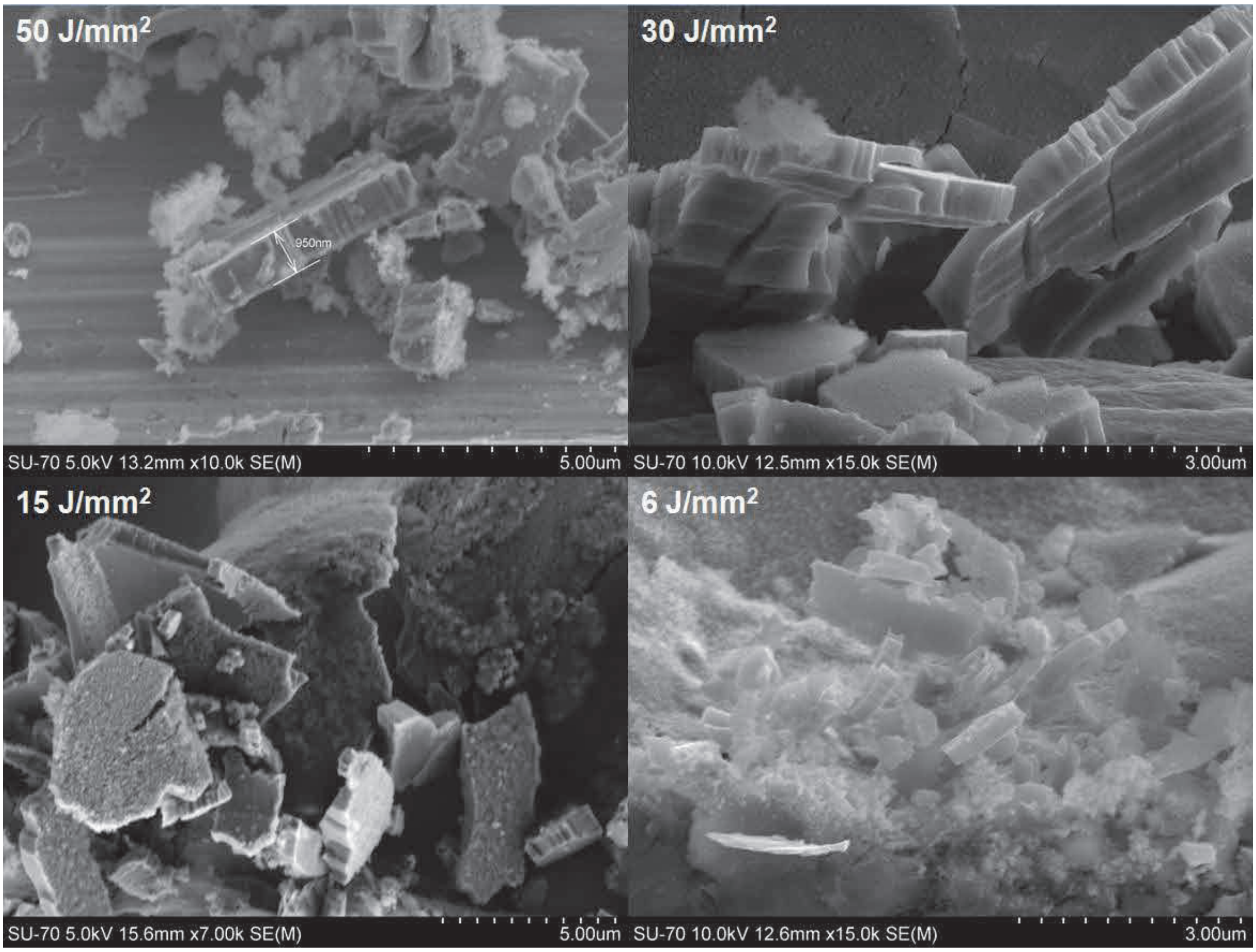

Figure 4 ASEM pictures of oxide layer for different fluence

4. CONCLUSION

A fiber laser beam is an efficient tool for controlled laser heating. The method of controlled heating of the sample by the laser, which was designed during the solution of the projects, was used to form an oxide layer on the surface of the sample. In the next step, the absorption of samples with the oxide layer on the surface was measured. The measurement revealed significant differences in absorptivity. Therefore further analyzes such as the measurement of oxide layers thickness have been performed. The dependence of absorption on the thickness of the oxide layer is not linear. The local maximum in the absorption graph could be caused by the phenomenon of total reflection. Additional tests are needed to clarify existing hypotheses.

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