The influence of oxide layer pattern size created on the surface of C45 steel on laser light absorption during laser quenching procedure

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Abstract. An increase in surface absorptivity can be helpful with a number of laser heat treatment processes. This study examines the relationship between the surface condition and its influence on interaction with a laser beam. The aim of this paper is to examine the effect of the oxide layer pattern size on the absorption of IR radiation and the influence of the ambient atmosphere. The process was carried with inert gas shielding. The oxide layers are created by the controlled heating method. The controlled heating method uses a scanning laser system to create oxide layers of different thickness on steel EN 10083-2: C45. Subsequently, the absorbency of the formed oxide layer was measured, depending on the irradiation energy emitted into the surface. According to recent experiments has been found that the higher the thickness of the oxide layer increases absorption. 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. The thickness and hardness of the laser hardened layer were considered. That paper describes possibilities of precise laser quenching of the oxidized C45 material surface.

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. There are three sets of optical properties (three different wavelengths) for the Kirchhoff’s law \[1\]. The three wavelengths are the wavelength of engraving laser 1064 nm, the wavelength of quenching laser 808 nm, and the measuring range of infrared camera 1 μm.

A sequential method of surface absorption determination in the laser quenching process is described in \[2\]. This paper presents an efficient algorithm for determining the surface absorptivity in the process of laser quenching. A regression model for the reflectivity coefficient evaluation in laser surface quenching is presented in \[3\]. The model is numerically calculated by comparing the actual surface temperature to the theoretical prediction obtained by process simulation. Paper \[4\] describes the numerical-experimental analysis of the surface oxidation effect on the laser quenching. 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 \[5\]. 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 the 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 [6] 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, especially for precise heat treatment [7].

The absorptivity can be obtained by the method of measurement of spectral normal hemispherical reflectivity at room temperature (SNHRRT) [8] or for high-temperature assessment the method of measurement of the spectral normal emissivity at high temperature (SNEHT) [9] can be used.

The aim of the paper is the description of the behavior of different oxide layer pattern sizes created by the method of controlled heating. The influence of the width of the oxide layer and the gap between oxide layers on the absorption of laser radiation during the heat treatment process using argon as shielding gas is investigated. The aim is to enable very precise choice of the area which should be hardened. It will be done using the selective surface oxidation procedure. The method of processing can change optical properties of the material surface (absorptivity).

2 Experimental procedure

Tests were performed on a rectangular block from rolled steel EN 10083-2: C45 of 200x100x20 mm³. The oxide layer was created by marking laser SPI-G3-SP-20P with MOPA arrangement by the method of controlled heating [10]. Heating parameters of the creation of the oxide layer were chosen based on the previous experiment the oxide layer with the highest thickness (fluence - 50 J/mm²) was selected.

Oxide layer patterns were formed on the sample surface according to Figure 1. After that the surface layer of the sample was modified by controlled oxidation procedure, the modified sample surface was subjected to a laser quenching. The sample was processed by the circular laser beam with a diameter of 8 mm. Four separated hardened tracks were created on the sample. The tracks were put perpendicularly to oxide pattern direction.

![Figure 1. Sample with different oxide layer patterns, created with different width and gap distances between them, laser tracks are marked in red, the dimensions are in mm](image-url)
Laser quenching procedure was implemented using argon as a shielding gas. A nozzle was used which feeds the shielding gas argon directly to the interaction area of the laser beam and the surface of the treated material. The argon as shielding gas prevents the surface oxidation of the processed material. The thickness of the oxide layer was measured by scanning electron microscope Analytical SEM Hitachi SU-70 [10].

The surface temperature was measured during the quenching process. The measurement was made by the thermal camera. The thermal camera measures on 1 μm wavelength, so that it is not very sensitive to selected emissivity value. Because metals have an emissivity around 0.9 for temperatures around 1000°C and the measured temperature changes by 50°C for emissivity in range 0.7-1.

The chemical composition of an oxide layer on the surface of the sample was inspected by grazing-incidence x-ray diffraction. The composition was measured for basic material and for oxidated surface (fluence – 50 J/mm²).

The absorptivity value was obtained by the method of measurement of spectral normal hemispherical reflectivity at room temperature (SNHRRT).

3 Results and discussion

The absorptivity was measured in ultraviolet-visible – near-infrared light spectral region (Figure 2.). It was measured on surface modified by controlled oxidation in comparison to the surface of the basic unmodified material. The oxide layer was created by laser fluence of 50 J/mm². The absorptivity at 808 nm (wavelength of quenching laser) measured for the modified surface was around 20 percent higher in comparison to absorptivity of the unmodified material surface.

![Figure 2. Measured absorptivities in the UV-VIS-NIR light spectral region for surface modified by controlled oxidation (blue color) in comparison to the basic unmodified material surface (red color)](image)

| Table 1. Surface hardness and depth of hardened layer – for surfaces modified by controlled oxidation |
|---------------------------------|----------------|----------------|----------------|----------------|----------------|
| The width of the oxide layer pattern | 1 mm pattern | 2 mm pattern | 3 mm pattern | 4 mm pattern | 5 mm pattern |
| Hardness [HV1] | 515 | 512 | 526 | 541 | 563 |
| Depth [μm] | 300 | 350 | 390 | 410 | 420 |
Figure 3. ASEM picture of oxide layer thickness for 50 J/mm$^2$ fluence

Surface hardness and depth of the hardened layer were measured in cross-section of the sample (Table 1.). The depth of the hardened layer was determined as the distance from the surface where 80% of the surface hardness was achieved. The hardness parameters were measured for modifications (oxide) widths from 1 mm to 5 mm and gaps of basic material between them from 1 mm to 5 mm (Figure 1.). The laser quenching process respects the lowest gap area for all widths of oxide patterns, 1 mm area of unhardened material has been preserved. The measured thickness of the oxide layer formed by 50 J/mm$^2$ laser fluence was around 1 μm (Figure 3.). A scratch was made into the oxide layer and the thickness of the fragments was measured.

The surface temperature was measured during the quenching process with shielding gas. The temperature measured on the modified surface was around 1100°C (austenitization occurs) and on the unmodified basic material was measured temperature around 600°C (no structural changes occur) [10].

The composition of the created oxide layer is around 45% FeO and 55% Fe$_3$O$_4$ before laser quenching process. The composition changes to 85% FeO and 15% Fe$_3$O$_4$ after laser quenching process.

4 Conclusion

The paper deals with the effect of oxide layer pattern size created on the surface of the material by the method of controlled heating on the surface absorptivity of the laser beam during the process with argon shielding.

The using of shielding gas allows us higher selectivity of laser quenching procedure. The work focuses on the determination of the modification size or size of the gap between them that will be respected by laser quenching process.

In the paper were analyzed thickness of the oxide layer and the composition of the layer created by the method of controlled heating. The surface hardness and depth of hardened layer were measured after laser quenching process. The influence of created oxide layer pattern size on these material properties was inspected.

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References

[1] RINALDI R, VAINER G B, KUNC P, KNIZKOVA I, GOTTSCHALK K, CARLOMAGNO M G, ALLOUIS CH, PAGLIARA R, ROTOLANTE R A, GRINZATO E, Infrared Thermography (Recent Advances and Future Trends). Infrared Thermography. 2012. Vol. 241. Carosena Meola.

[2] NGUYEN Q and Y, A sequential method to determine the surface absorptivity in the process of laser surface hardening. International Journal of Heat and Mass Transfer. 2016. Vol. 95, p. 224–229. Ching-yu. DOI 10.1016/j.ijheatmasstransfer.2015.11.087.

[3] FORTUNATO A, ASCARI A, ORAZI L, CAMPANA G, and CUCCOLINI G, Numerical evaluation of the reflectivity coefficient in laser surface hardening simulation. Surface & Coatings Technology. 2012. Vol. 206, p. 3179–3185. DOI 10.1016/j.surfcoat.2011.12.043.

[4] CORDOVILLA F, GARCÍA-BELTRÁN Á, DOMINGUEZ J, and SANCHO P, Numerical-experimental analysis of the effect of surface oxidation on the laser transformation hardening of Cr – Mo steels. Applied Surface Science. 2015. Vol. 357, p. 1236–1243.

[5] PANTSAR H and KUJANP V, Effect of oxide layer growth on diode laser beam transformation hardening of steels. Surface & Coatings Technology. 2006. Vol. 200, p. 2627–2633. DOI 10.1016/j.surfcoat.2004.09.001.

[6] GOOS F and HÄNCHEN H, Über das Eindringen des totalreflektierten Lichtes in das dünnere. Medium Ann. Physik. 1947. Vol. 435, no. 5, p. 1947.

[7] HRUŠKA M, VOSTŘÁK M, SMAZALOVÁ E and ŠVANTNER M, 3D scanning laser hardening. METAL 2014 - 23rd International Conference on Metallurgy and Materials, Conference Proceedings. 2014. P. 921–926.

[8] ZHANG K, YU K, LIU Y, and ZHAO Y. An improved algorithm for spectral emissivity measurements at low temperatures based on the multi-temperature calibration method. International Journal of Heat and Mass Transfer [online]. 2017. Vol. 114, p. 1037–1044. DOI 10.1016/j.ijheatmasstransfer.2017.06.133.

[9] HONNER M, HONNEROVÁ P, KUČERA M and MARTAN J. Laser scanning heating method for high-temperature spectral emissivity analyses. Applied Thermal Engineering. 2016. Vol. 94, p. 76–81. DOI 10.1016/j.applthermaleng.2015.10.121.

[10] HRUŠKA M and VOSTŘÁK M, The impact of controlled en 10083-2: C45 steel surface oxidation on NIR light absorption. METAL 2017 - 26th International Conference on Metallurgy and Materials, Conference Proceedings. 2017. P. 1153-1158.