

THE INFLUENCE OF CONTROLLED STEEL SURFACE OXIDATION ON NEAR-INFRARED LIGHT ABSORPTION IN INERT ATMOSPHERE

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Abstract

The aim of this paper is to examine the effect of the oxide layer thickness on the absorption of IR radiation in the inert gas atmosphere. The oxide layers are created by the controlled heating method, while the different thickness of layers is achieved by changing the heat input. The tests were performed on steel EN 10083-2: C45. The absorption was measured in UV - NIR spectral region. The samples were afterward processed by HPDD laser. Firstly, the hardening was carried out in the air but the second experiment was performed with using of shielding gas fed to the interaction area. The thickness and hardness of laser hardened layer were considered. The chemical composition of an oxide layer on the surface of the sample was inspected by grazing-incidence x-ray diffraction. The difference between hardening with use of shielding gas and hardening in the air was assessed.

Keywords: Laser hardening, an oxide layer, shielding gas, absorption, laser

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], 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 μ m.

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 μ m. 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, especially for precise heat treatment [9].

The aim of the paper is the description of the behavior of different oxide layers created by the method of controlled heating [10]. The influence of the thickness of the oxide layer on the absorption of laser radiation during the heat treatment process is investigated [10]. The effect of the use of the shielding gas during the laser surface hardening process on the rate of laser radiation absorption is assessed.

2. EXPERIMENTAL PROCEDURE

Tests were performed on a rectangular block from rolled steel EN 10083-2: C45 of 200x100x20 mm³. Oxide layers were created by marking laser SPI-G3-SP-20P with MOPA arrangement by the method of controlled heating. Parameters were chosen based on previous experiments. 7 oxide layers of different thickness were formed on the sample surface (Figure 1). After that the surface layer of the sample was modified by controlled oxidation procedure, the sample was subjected to a laser hardening. Samples, prepared according to Figure 1, were processed by the wide laser beam. Three separated hardened tracks were created on each sample. The tracks were put perpendicularly to oxide pattern direction.

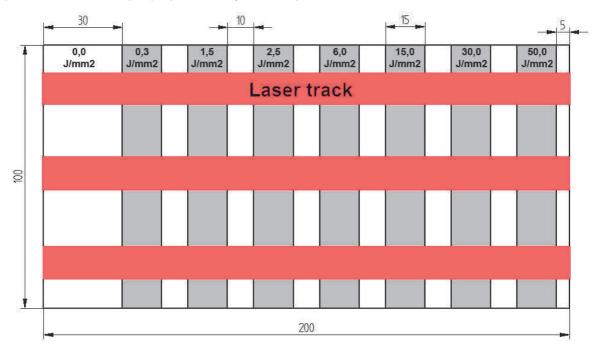


Figure 1 Sample with 7 different oxide layers, created by different fluence parameter, laser tracks are marked in red, the dimensions are in mm

Two laser quenching procedures were performed that differed in using shielding gas. In the first case, the laser quenching process took place in the air. During the second treatment, a nozzle was used which feeds the shielding gas argon directly to the interaction area of the laser beam with 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 both of these processes. The measurement was made by the thermal camera. The thermal camera measures on 1 um wavelength, so that it is not very sensitive to selected emissivity value.

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 one selected modified surface (fluence - 50 J/mm²).

3. RESULTS AND DISCUSSION

Temperature measurement was performed during the heat treatment process. The comparison of the temperatures measured during hardening process performed in air versus process using argon as shielding gas is visualized in **Figure 2**. The difference between the absorption of the modified surface and the basic material is higher for the process with shielding gas. The temperature difference between basic material and modified surface during hardening in the air is around 150°C but the difference during processing using the shielding gas is around 600°C. The temperature measured for basic material during processing with shielding gas depicted in **Figure 2** is around 800°C, but that is not measured temperature because the measuring range of the thermal camera was selected from 800°C to 1700°C. The temperature of basic material during the laser hardening process is significantly lower.

The difference in depth of hardened layer between processing in air and treatment with shielding gas is shown in **Table 1**. **Table 1** shows that the depth of hardened layer for modified areas differs very slightly depending on the amount of oxygen presented in the interaction zone during the process, but this effect can be very serious for basic material.

The changes in chemical composition of the surface oxide layer are demonstrated in **Figures 3** and **4**. The amount of oxygen-rich oxides rises during processing in the air, but it declines for processing with shielding gas, where the amount of FeO increases.

Table 1 Depth of hardened layer for processing in air (DoHL_{air}) and treatment with Argon as shielding gas (DoHL_{Ar})

Fluence [J/mm ²]	0.0	0.3	1.5	2.5	6.0	15.0	30.0	50.0
DoHL _{air} [µm]	300	300	385	400	385	400	420	450
DoHL _{Ar} [µm]	0	0	260	320	260	350	400	420

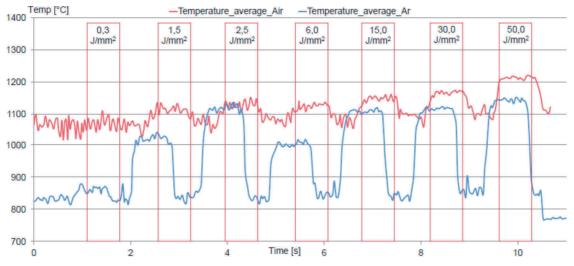


Figure 2 Graphical representation of temperature measured during laser hardening process in the air (red line) and with argon shielding (blue line)



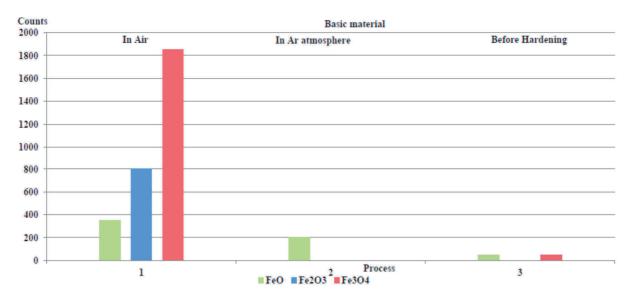


Figure 3 The chemical composition of oxide layer depending on the type of processing - for basic material

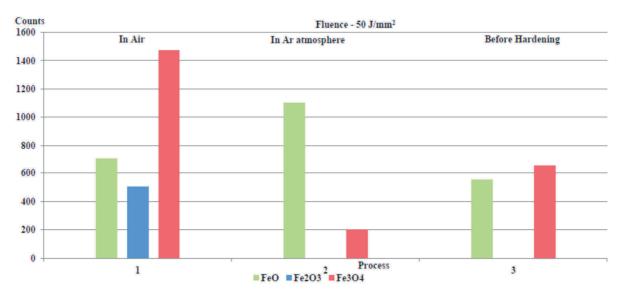


Figure 4 The chemical composition of oxide layer depending on the type of processing - for modified surface

4. CONCLUSION

The paper deals with the effect of oxide layer created on the surface of the material by the method of controlled heating on the surface absorptivity of the modified material. The influence of using the shielding gas during the process is discussed.

The result is that the effect of the oxide layer is significantly higher for laser hardening process using argon as a shielding gas. All analyzes show that the use of argon as a shielding gas increases the difference between the amount of energy absorbed by the base material and its modified surface. It is possible to precisely select the areas to be hardened against the regions where the heat-unaffected material should be preserved, even if the whole surface is processed by the laser beam with the same parameters.

The paper also concerns with the change in the composition of the oxide layer on the surface in dependence on the use of shielding gas.



ACKNOWLEDGEMENTS

The result was developed within the CENTEM project, reg. no. CZ.1.05/2.1.00/03.0088, cofunded by the ERDF as part of the Ministry of Education, Youth and Sports OP RDI programme and, in the follow-up sustainability stage, supported through CENTEM PLUS (LO1402) by financial means from the Ministry of Education, Youth and Sports under the "National Sustainability Programme I." and project no. SGS-2016-005.

REFERENCES

- [1] ROBERTO RINALDI, BORIS G. VAINER, PETR KUNC, IVANA KNIZKOVA, KLAUS GOTTSCHALK, GIOVANNI M. CARLOMAGNO, CHRISTOPHE ALLOUIS, ROCCO PAGLIARA, RALPH A. ROTOLANTE, ERMANNO GRINZATO, Carosena Meola. *Infrared Thermography (Recent Advances and Future Trends)*. Carosena Meola, Department of Aerospace Engineering University of Naples Federico II, Italy, 2012. ISBN 9781608051434.
- [2] GRUM, J. and KEK, T. The influence of different conditions of laser-beam interaction in laser surface hardening of steels. *Thin Solid Films*. 2004. Vol. 453-454, p. 94-99. DOI 10.1016/j.tsf.2003.11.177.
- [3] WOO, H G and CHO, H S. Ht ## OLOtt Estimation of hardened layer dimensions in laser surface hardening processes with variations of coating thickness. . 1998. Vol. 102, p. 205-217.
- [4] NGUYEN, Quan and YANG, Ching-yu. International Journal of Heat and Mass Transfer A sequential method to determine the surface absorptivity in the process of laser surface hardening. *International Journal of Heat and Mass Transfer* [online]. 2016. Vol. 95, p. 224-229. DOI 10.1016/j.ijheatmasstransfer.2015.11.087.
- [5] FORTUNATO, Alessandro, ASCARI, Alessandro, ORAZI, Leonardo, CAMPANA, Giampaolo and CUCCOLINI, Gabriele. Surface & Coatings Technology Numerical evaluation of the re fl ectivity coef fi cient in laser surface hardening simulation. . 2012. Vol. 206, p. 3179-3185. DOI 10.1016/j.surfcoat.2011.12.043.
- [6] CORDOVILLA, Francisco, GARCÍA-BELTRÁN, Ángel, DOMINGUEZ, Jesús and SANCHO, Paula. Applied Surface Science Numerical-experimental analysis of the effect of surface oxidation on the laser transformation hardening of Cr Mo steels. . 2015. Vol. 357, p. 1236-1243.
- [7] PANTSAR, Henrikki and KUJANP, Veli. Effect of oxide layer growth on diode laser beam transformation hardening of steels. . 2006. Vol. 200, p. 2627-2633. DOI 10.1016/j.surfcoat.2004.09.001.
- [8] GOOS, F. and HÄNCHEN, H. Über das Eindringen des totalreflektierten Lichtes in das dünnere. *Medium Ann. Physik.* 1947. Vol. 435, no. 5, p. 1947.
- [9] 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.
- [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.