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Surface modification of stainless steels by TEA CO_2 laser

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Abstract

The interaction of a transversely excited atmospheric (TEA) CO_2 laser with austenitic stainless AISI 316 and high-speed tool AISI M2 steel is considered. The results have shown that both types of steel were surface modified by the laser beam, for the laser energy densities used. Morphological features on the AISI M2 were more prominent in comparison to a AISI 316 steel. On the AISI M2 steel crater like forms, solidified boundary and hydrodynamical modifications were observed, while on the AISI 316 one, corrugation, cracking and resolidified areas were registered. The morphology changes of steel surfaces in dependence of laser pulse temporal shape were analyzed especially. The pulse with a tail for difference of a tail-free pulse, for the same incident peak power density, as a rule caused higher level of damage. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Surface modification studies of steel by various types of energetic beams, including a laser beam, is of a great fundamental and technological interest. In this context it must be emphasized that investigations of the laser beam interaction with various types of steel are highly interesting. The Nd:YAG, excimer and CO_2 (continuous-wave regime operation) are frequently used laser beams for this purpose [1–3].

The interaction of a pulsed transversely excited atmospheric (TEA) CO_2 laser beam with various steels including austenitic stainless and high-speed tool steel, are not so often considered in [4–6]. Among other applications, due to its good metallurgical, magnetic, vacuum and thermal properties, austenitic stainless steel, used in this investigation, is also an attractive material for nuclear technology. It is well known that quality of fusion plasma in the thermonuclear device depends not only on the reactor design, but also on the proper selection of the reactor vessel wall material, especially from reactor plasma facing materials [7]. The austenitic stainless steel is a serious candidate just for this application. Within the context of nuclear technology application of this type of laser it must be pointed out that it might be successfully employed for the surface radioactive decontamination of various metals, including stainless steel contaminated with different isotopes, especially the fission products [8].

The high-speed tool steel posses desirable physico-chemical characteristics: thermo-mechanical stability

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and high hardness, so that it finds application in mechanical industry primarily for tools. The austenitic stainless as well as high-speed tool steel can be additionally protected by coatings. Nitrides are considered as good materials for this purpose [7,9]. Detailed investigations of laser induced changes of coating deposited on austenitic stainless or high-speed tool steel, in the form of refractory transition metal nitrides (e.g. the titanium nitride), are presented in [10] and [11], respectively.

The purpose of this work was to study the effect of a TEA CO_2 laser, which emits infrared radiation at wavelength about 10 microns, on the austenitic stainless AISI 316 and high-speed tool AISI M2 steel. Special attention was devoted to monitoring of the surface morphology modification of the AISI 316 and AISI M2 steel as a function of the laser pulse shape. For the studied steels the American Iron Steel Institute (AISI) classification has been used.

2. Experiment

Laser induced surface modification was carried out by a pulsed, UV preionized TEA carbon dioxide laser. The laser operated with non-typical CO_2/X, X = H_2; H_2/N_2, for a difference of typical CO_2/Y, Y = He; He/N_2 gas mixtures. The presence of hydrogen in the laser increased the efficiency of the system [12]. It is well known [13] that the laser pulse shape can be controlled by adjusting gas mixture content. The nitrogen-rich mixture gave a longer laser pulse and a pulse with a tail (A type pulse) while its absence resulted in a tail-free pulse (C type pulse). Presence of a small quantity of nitrogen leads to reduction of tail (B type pulse) and output pulse laser energy is mainly consisted in the initial spike (about 70%). The laser pulse shapes applied for irradiation of the steel target are presented in Fig. 1.

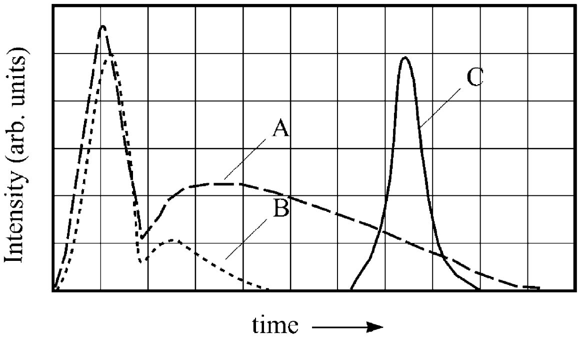

Fig. 1. Temporal shapes of the TEA CO_2 laser pulse employed in the experimental work: (– – –), horizontal scale 0.2 μs/div. (A type pulse); (- - - -), horizontal scale 0.2 μs/div. (B type pulse); and (—), horizontal scale 0.1 μs/div. (C type pulse).

The laser has been running in a multimode regime. The beam cross-section is typically of quadratic form, so that spatial-uniform distribution of intensity can be assumed [14]. Detailed characteristics of the laser used in the experiment are presented in Table 1.

Table 1
The typical TEA CO_2 laser operational conditions during the irradiation experiment

Gas mixture	$CO_2/N_2/H_2$	$CO_2/N_2/H_2$	CO_2/H_2
Content	1/1.6/1.4	1/0.04/0.6	1/1.3
Output pulse energy (mJ)	220	41	30
FWHM[a] (ns)	120 (initial spike); ~2 μs (pulse tail)	80 (initial spike); ~0.8 μs (pulse tail)	80
Pulse type	A pulse	B pulse	C pulse
Peak power (MW)	0.5	0.3	0.375
Mode structure[b]	Multimode output		
Beam divergence[c] (mrad)	~10		
Laser cavity	Non-dispersive		
Spectral composition[d]	Simultaneous two-lines operation in P-branch $00^01 \rightarrow 10^00$ vibrational band		
Pulse repetition rate (Hz)	1		

[a] Full width at half maximum.
[b] The laser posses a high multimode output. The unfocused laser beam has a quadratic cross section with dimensions 1 cm × 1 cm.
[c] This value is measured in the relation to near field.
[d] The laser simultaneous operates at two wavelengths, i.e. 10.5709 and 10.5909 μm, P(18) and P(20) transitions. P(20) transition is more intensive.

The austenitic stainless AISI 316 as well as high-speed AISI M2 steel irradiation was performed with a focused laser beam. KBr lens with focal length of 6.0 cm ensured focusing onto the steel target. The incidence angle of the laser beam in respect to the surface was 90°. The interaction was conducted in air atmosphere, at a pressure of 1013 mbar and relative humidity of 60%.

Both, the austenitic stainless as well as high-speed tool steel employed in the experiment were of the plate form, rectangular shape, with dimensions 20 mm × 13 mm × 1.5 mm (length × width × thickness). For surface preparation, the standard metalographical procedure was applied (polishing, rinsing and drying). For characterization of the austenitic stainless AISI 316 and high-speed M2 steel, before and after laser irradiation, various analytical techniques were used. The surface morphology was monitored: by optical microscopy (OM), by scanning electron microscope (SEM) with secondary electron (SE) and back-scattered electron (BSE) detectors and, by atomic force microscope (AFM). Quantitative standardless compositional analysis has been done by an energy dispersive analyzer (EDAX). Phase composition and crystallographic structure were determined by the X-ray diffraction method. Reflectivity characterization for a spectral region of 2.5–25 μm was carried out by infrared spectrophotometer.

3. Results and discussion

3.1. Austenitic stainless AISI 316 steel

Structural and chemical composition analysis showed that steel material used in experiment is an austenitic stainless steel with the grain size above one hundred microns. The EDAX analysis verified chemical composition of steel AISI 316, i.e.: C, <0.5%; Cr, 16.8%; Mo, 1.6%; Si, 0.7%; Ni, 12.1%; Ti, <0.5% (all in the wt.%) and balance Fe. X-ray analysis has confirmed that the steel consisted of the γ-Fe phase with faced centered cubic crystal structure.

Reflectivity measurements in the spectral region of about 10 μm, have shown that polished austenitic stainless steel AISI 316 has initial reflectivity of 86%. It is well known that reflectivity of a target surface depends on its quality, its temperature, wavelength radiation used and number of previously accumulated pulses [15]. Reflectivity of metals is direrctly correlated to its absorptivity. Initially absorbed laser pulses seriously change the surface morphology and causes chemical transformations, like oxidation for example. These changes additionally increase the level of the target absorptivity. Consequently, the level of damage after the action of several hundred accumulated laser pulses is remarkable.

Investigation of the AISI 316 steel morphological changes, induced by the laser radiation, showed their dependence on beam characteristics: pulse energy, laser pulse duration, peak power density, number of pulses, etc. The energy absorbed from the beam is converted into thermal energy, which generate a series of effects such as melting, vaporization of molten material, dissociation or ionization of the vaporized material and shock waves in the vapor and in the solids.

Damage threshold, measured in this context, depended on beam characteristics and material properties, too. The threshold defined as the minimum fluence that creates detectable damage of the steel surface, has been reached after 20 pulses. For austenitic stainless steel AISI 316 it was 23.0 and 7.0 J/cm^2, depending whether the laser operates in the tail or tail-free pulse regime.

The damage yield [11], also determined in this work, is a function of the number of accumulated laser pulses, Fig. 2. For tail-free pulses (C type pulse) this damage level (in the 20–500 pulses interval) is constantly smaller in relation to the pulses with a tail (A type pulse), Fig. 2. Saturation has been reached after ~200 pulses, C type pulse. The peak power density was the same for both pulses (170 MW/cm^2). Calculation of the temperature–time evolution on the target surface [16] for used laser pulse temporal shapes, i.e. for A and C pulse types, was carried out using the Eq. (2) from [13]. The equation includes absorptivity, specific heat, thermal diffusivity, target density and the laser beam intensity. The equation was solved numerically by a computer programme. For the A pulse type, i.e. for the pulse with tail, calculation shows that the temperature (given in arbitrary units) increases with time. It reached maximal value after about 1.2 μs. A local temperature maximum at about 0.25 μs was registered, but its value was lower in

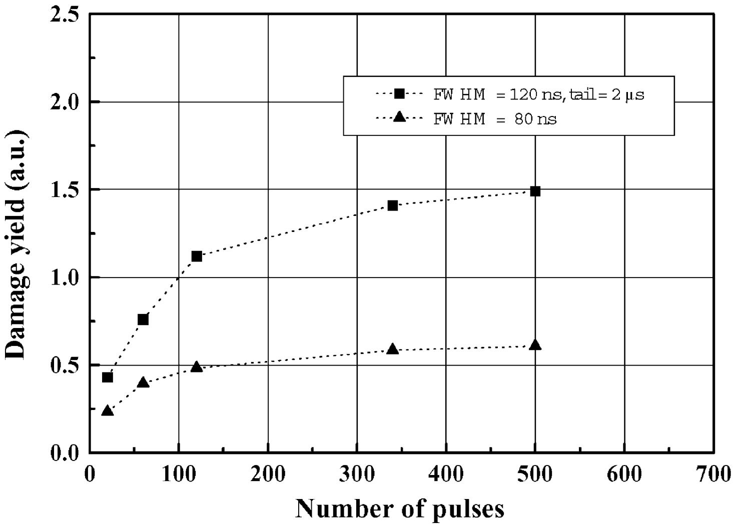

Fig. 2. Damage yield of austenitic stainless steel AISI 316 vs. number of accumulated laser pulses: (■), laser pulse with tail (A type pulse); (▲), tail-free laser pulses (C type pulse). Peak power density = 170 MW/cm^2.

respect to that at 1.2 μs. Maximal temperature on the target has been achieved in time which corresponded to about 2/3 of the tail duration. Thus, it can be assumed that the energy of laser sustained in the tail essentially affects surface modification of the target. For the C pulse type, i.e. tail-free pulse, the maximal temperature on the stainless steel target was reached after about 150 ns which was the higher value in relation to FWHM of the pulse, Table 1. The presence of the tail, Fig. 2, obviously has a significant effect on the damage level of the austenitic stainless steel target.

Surface changes in the irradiation zone, registered after 500 pulses, were more prominent than in the case of 20 pulses. The morphology of the austenitic stainless steel AISI 316 as a function of the laser pulse shape, is presented in Figs. 3 and 4. The results show that the action of 500 laser pulses, with deposited energy in the interval from 8.7 to 41.7 J/cm^2, leads to the important changes of the AISI 316 steel surface. Fig. 3b–d illustrates the effects of the laser irradiation, at the center and the periphery of the damage area, for three types of laser pulses.

Morphological characteristics have been analyzed in all investigated areas. Generally, the laser induced changes can be explained by thermally induced features in the bombarded zone as melting, thermal stress cracking, etc. Surface modifications, in details, can be presented in follows: (i) for all three types of laser pulse the corrugation, cracking of central zone area with resolidified regions were registered. In this zone the needle-shaped and lens-shaped forms have been created, too. Pulses with tail and with reduced tail (A and B types pulse) caused more evident morphology modification, Fig. 3b.1., b.2., c.1. and c.2. and Fig. 4b and c, in comparison with tail-free pulse, Fig. 3d.1. and d.2. and Fig. 4d. The presence of the tail, as mentioned above, resulted in a longer stabilization of the surface temperature in time [13], and (ii) toward a periphery there were appearances of radial cracking for all three types of laser pulses, Fig. 3b.3., c.3. and d.3. Hydrodynamical sputtering has not been recorded.

Changes in the central zone can probably be attributed to the appearance of martensite form. It is well known that in the course of steel thermal treatment at higher temperatures, dependent on its chemical composition (especially carbon content), the phase transformation will occur [17]. The steel with a low carbon content, like AISI 316, will be phase transformed in the temperature interval from 930 to 960 K. The action of a pulsed laser on a steel target caused rapid temperature changes within it, e.g. cooling was extremely fast [18], and carbon which is present cannot be

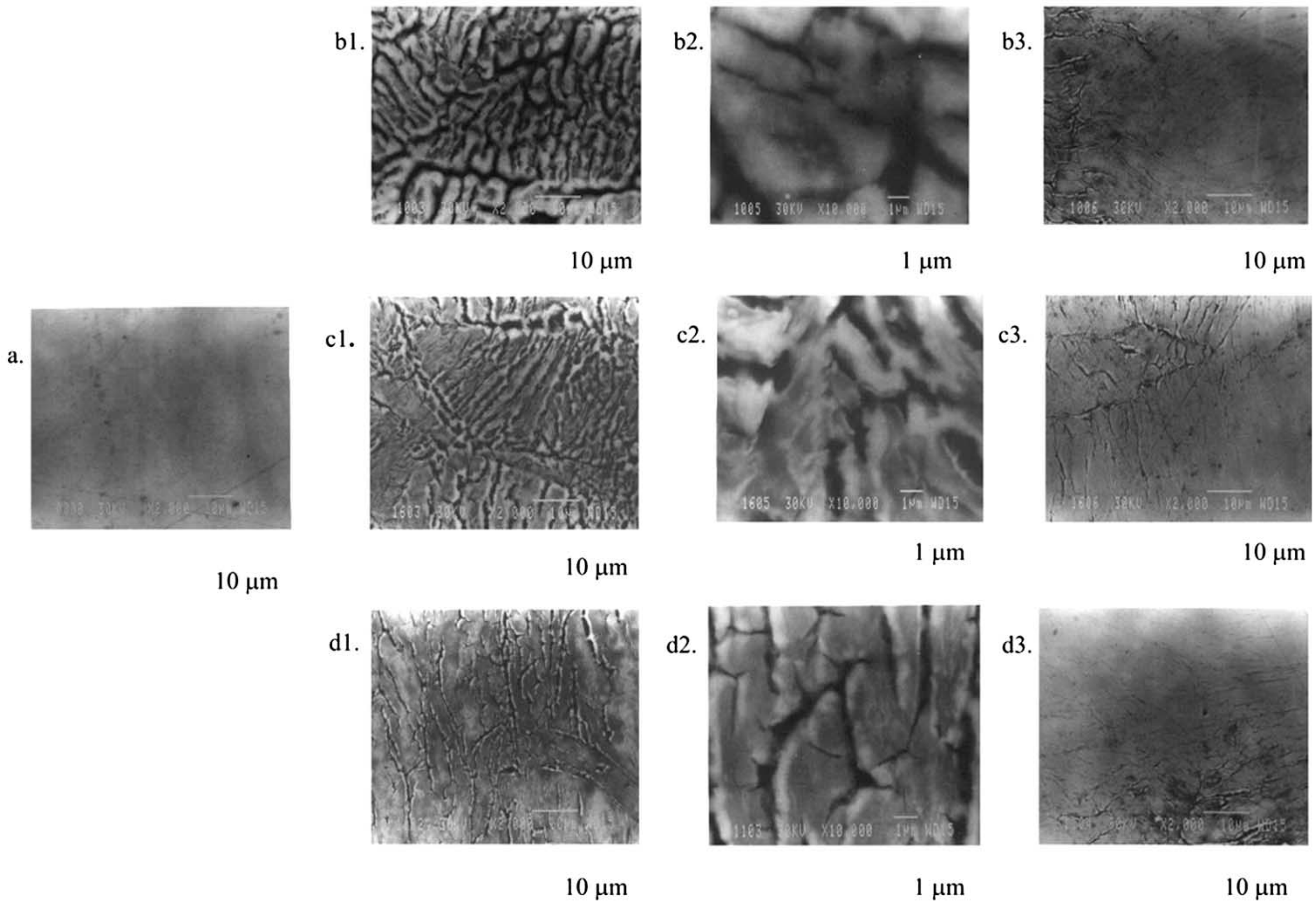

Fig. 3. The morphology of the austenic stainless steel AISI 316 changes induced by TEA CO_2 laser. The analysis has been carried out by scanning electron microscope. (a) The steel surface before laser treatment. Changes induced with 500 pulses. (b) Action of A pulse type, deposited energy = 41.7 J/cm^2. In central zone (b.1. and b.2.) and at the periphery of the interaction (b.3.). (c) Action of B pulse type, deposited energy = 13.5 J/cm^2. In central zone (c.1. and c.2.) and at the periphery of the interaction (c.3.). (d) Action of C pulse type, deposited energy = 8.7 J/cm^2. In central zone (d.1. and d.2.) and at the periphery of the interaction (d.3.).

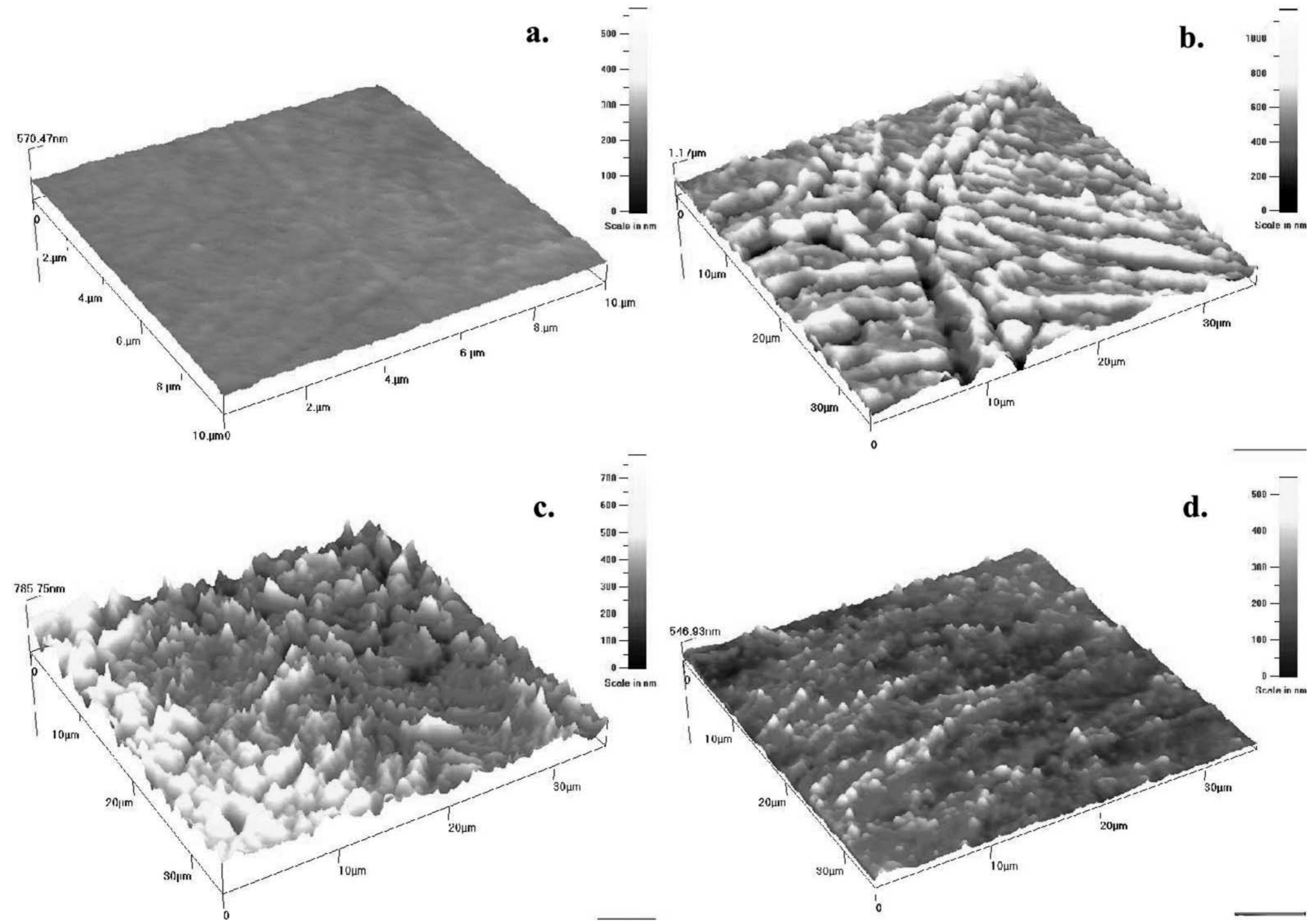

Fig. 4. Three-dimensional surface changes of AISI 316 steel induced by TEA CO_2 laser. The analyzes has been performed by atomic force microscope. (a) The steel surface before laser treatment. Changes induced with 500 pulses in central damage zone. (b) Action of A pulse type. (c) Action of B pulse type. (d) Action of C pulse type. The conditions of irradiation were the same as on Fig. 3. The length of scale on (a) and (b), (c), (d) corresponds to 10 and 35 μm, respectively.

separated from α-ferrite as a new created phase. The new phase becomes over-saturated with carbon and this structure is known in literature as martensite [17]. This structure is qualitatively characterized by existing of needle-shaped and lens-shaped forms on steel surface.

3.2. High-speed tool AISI M2 steel

Structural and chemical composition analysis showed that material used in our experiment was the high-speed tool steel with the grain size of several tens of microns. The EDAX analysis verified the chemical composition of the high-speed tool steel AISI M2, i.e.: C, 0.9%; Cr, 4.0%; Mo, 5.0%; W, 6.5%; V, 1.9% (all in the wt.%) and balance Fe. X-ray analysis has confirmed that the steel consisted α-Fe phase with the body centered cubic crystal structure. The sample of AISI M2 steel was thermally treated before laser irradiation. This treatment comprised the quenching and annealing process. First one has been carried out at a temperature of 1473 and, the second at 803 K. After this treatment the sample was cooled down at room temperature.

The reflectivity measurements of polished high-speed tool steel AISI M2 samples have indicated that the initial reflectivity in the spectral region of about 10 μm was 83%.

Investigations of the high-speed tool steel AISI M2 morphological changes, induced by TEA CO_2 laser radiation, have confirmed their dependence on the laser beam characteristics. For this type of steel all modifications were examined at laser temporal operation in two modes, A and C pulse mode, Fig. 1.

The damage threshold for AISI M2 steel is determined to be 9.1 and 4.0 J/cm^2 for A and C type pulse, respectively. The damage yield as a function of number of accumulated laser pulses is presented in Fig. 5. Tail (A type pulse) and tail-free pulses (C type pulse) posses the similar trend of damage changes (in the 20–500 pulse interval) but with different curve slopes. The peak power density was the same, i.e. 170 MW/cm^2 for both laser pulses in the course of this measurement.

The surface variations in the irradiation zone, registered after 500 pulses, were more evident than in case of 20 pulses. The morphology of the high-speed tool steel AISI M2 as a function of the laser pulse shape, is

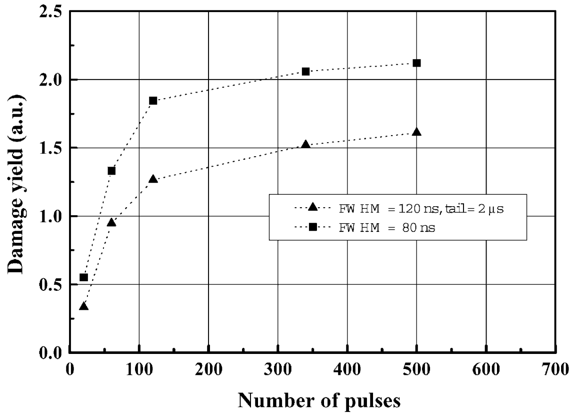

Fig. 5. Damage yield of high-speed tool steel AISI M2 vs. number of accumulated laser pulses: (■), A type pulse and (▲), C type pulse. Peak power density = 170 MW/cm^2.

presented in Fig. 6. It can be seen that the action of 500 laser pulses with deposited energy in the interval of 14.0 to 78.0 J/cm^2 leads to significant changes in the AISI M2 steel surface. Wavy-like structures appeared in the central zone of damage, Fig. 6b1. and c1., and distances between two adjacent tops are 10 μm, which is approximately the same value as the wavelength of the laser light (10.6 μm). This structure is more dominant for pulse with tail. Thermal stress cracking, which was visible in this zone, is a consequence of non-uniform temperature distribution. In the periphery of interaction area, Fig. 6b3. and c3., the hydrodynamical structure in form of resolidified droplets of material has been registered.

The high-speed tool steel AISI M2 undergoes higher morphological changes than the austenitic stainless steel AISI 316 if irradiated by similar laser peak power density. This fact can be attributed primarily to different chemical compositions. The high-speed tool steel AISI M2 is reach in carbon. Structures created in the course of thermal treatment of the steel depend on the carbon contents, thermal process rate, etc. [16,17]. During laser heating the AISI M2 steel changes its crystal structure, i.e. an $\alpha \rightarrow \gamma$ phase transformation is possible [16,18]. In the cooling process carbon atoms complicate the $\gamma \rightarrow \alpha$ transformation.

For the case of fast cooling, as in our sample, crystallization occurs probably in the form of fine laminated pearlite over-saturating with carbon.

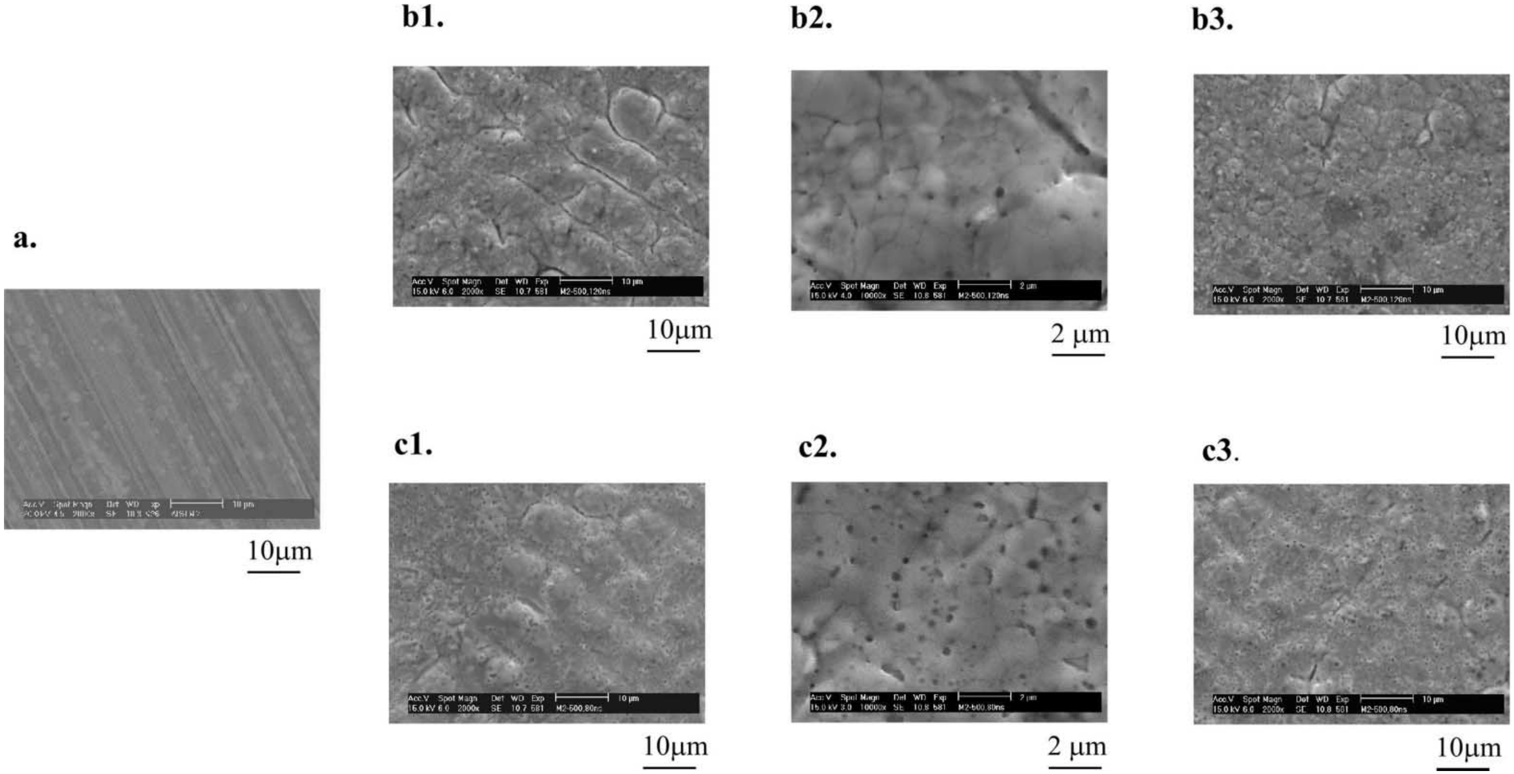

Fig. 6. The morphology of the high-speed tool steel AISI M2 changes induced by TEA CO_2 laser. The analysis has been conducted by SEM. (a) The steel surface before laser treatment. Changes induced with 500 pulses. (b) Action of A pulse type, deposited energy = 78.0 J/cm^2. In central zone (b1. and b2.) and at the periphery of the interaction (b3.). (c) Action of C pulse, deposited energy = 14.0 J/cm^2; c1. and c2. are central zones and c3. the periphery of interaction.

Table 2
Morphological changes on various types of steels as a function of laser pulse mode operation[a]

Steels type	Laser pulse type	
	A pulse type (pulse with tail)	C pulse type (tail-free pulse)
Austenitic stainless steel AISI 316	C; CR; Ps; martensite-like forms; threshold, 23.0 J/cm^2	C; CR; Pw; martensite-like forms; threshold, 7.0 J/cm^2
High-speed tool steel AISI M2	WS; CR; HS; Ps; pearlite-like forms; threshold, 9.1 J/cm^2	WS; CR; HS; Ps; pearlite-like forms; threshold, 4.0 J/cm^2

[a] C, corrugation; CR, cracking; Ps, strongly created plasma (in front of target); Pw, weekly creation of plasma; WS, wavy-like structure; HS, hydrodynamical structure.

Central parts, Fig. 6b1. and c1., looks like the open structure of laminated pearlite [16,17].

4. Conclusion

The interaction of a pulsed TEA CO_2 laser with austenitic stainless AISI 316 and high-speed tool AISI M2 steel have been studied. Both types of steel have been modified by TEA CO_2 laser radiation.

Special attention in this study was devoted to the morphological changes monitoring and their dependence on the laser temporal pulse shapes. For the same type of steel, pulses with tail, in respect to the tail-free pulses, induce more radical modifications on the surface, as a rule.

Calculation of the temperature–time evolution on the target surface has shown that the maximal temperature on the target was achieved in time which corresponded to about 2/3 of the tail duration. Energy of laser sustained in the tail of pulse essentially affects the surface modification of the target.

Morphological features on the AISI M2 were more drastic then on the AISI 316 steel, for almost the same value of peak power density and pulse temporal shape. Macro-features of changes on the AISI M2 were crater-like, while on the AISI 316 they were observed only on the surface. Microscopical feature, Table 2, shows wavy-like structure, thermal stress cracking as well as hydrodynamical form on the AISI M2 steel, while corrugation and cracking of the central and peripheral zone with resolidified regions were registered for AISI 316.

The interaction of laser radiation with steels, for used energy densities, was always followed with creation of plasma in front of steel-target. Within the context of this work the plasma was not studied spectroscopically. Detailed investigation of plasma would give data about constituents of air (via corresponding spectral bands) and probably about the main constituents of employed steels (via the spectral lines analyzes).

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