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Review

Improvement of Thermochemical Processes of Laser-Matter Interaction and Optical Systems for Wavefront Shaping

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Abstract: Laser thermochemical processes of metal surface oxidation are promising for creating new advanced technologies to meet the growing needs of opto- and micro-electronics, photonics, catalysis, sensorics and other high-tech industries. The features of thermochemical processes of laser-matter interaction occurring in matter under exposure to intense light flows and optical systems for controlling the irradiance and wavefront spatial distribution were reviewed. The laser beam offers the possibility of good focusing, which allows us to conduct chemical reactions, including the heterogeneous oxidation of metals, locally, with high spatial resolution. In this case, the absorption mechanisms of the laser beam vary for metals and for oxides, resulting from a thermochemical reaction and represent semiconductors. For semiconductors, the intrinsic, intraband, impurity, or lattice absorption takes place. The morphology of a metal surface also influences its optical absorption capacity. The improvement of beam shaping systems with elements of computer optics, namely diffractive freeform optics, provides an opportunity for an efficient control of chemical processes by achieving the desired redistribution of the laser beam power density. Laser thermochemical processes of the formation of quasi-one-dimensional nanostructured metal oxides are of great interest for advanced research and for a wide range of applications. A special feature of these processes is that, in the case of a frequency-modulated laser beam the synergy between the heat associated effects of the laser pulses and the laser-induced vibrations allows for a significant increase in the diffusion coefficient, which is stimulated by the non-stationary stress-strain state of the material. Ensuring the means of control over the thermochemical reaction in local sections of the laser exposure zone is an issue that can be solved by adapting the shape of the laser beam by the diffractive freeform optics. The gained knowledge contributes as a foundation for new photonic technologies oriented on the formation of nanostructured metal oxides, involving control over the morphology of the synthesized structures.

Keywords: laser-matter interaction; thermochemistry; metal oxidation; wavefront shaping; computer optics; diffractive freeform optics; synergy; laser-induced vibrations; non-stationary stress-strain state; surface morphology optimization

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1. Introduction

With the development of opto- and micro-electronics, microsystems engineering and sensorics, the demands on performance and power consumption of the devices being developed are increasing, while reducing the weight and size of components is also a rising demand [1–3]. Laser thermochemical processes of oxidation of the metal surface are promising for creating new advanced technologies for the production of components for such devices [4]. Laser-induced thermochemical recording of nano- and micro-dimensional structures is attracting increased interest due to various important applications in photonics, e.g., for optical transducer fabrication [5–7]. The application of metal-metal oxide nanomaterials provides an opportunity to develop efficient heterogeneous catalysts with high selectivity, stability and fast performance [8]. A progressive application of oxide materials is the heterostructured solar energy [9].

The laser is a source of light. Compared to other light sources, the laser has several unique properties due to the coherent and highly directional nature of its emission [10–12]. "Non-laser" light does not have such features. The low divergence of laser beams allows optical systems to concentrate light energy in negligible volumes, creating enormous densities of energy [13]. Moreover, the radiation from such sources tends to fill a wide wavelength range, i.e., it is non-monochromatic. The monochromatization of such radiation is achieved at the cost of significant energy losses, which become greater the narrower the spectral line becomes. The above-mentioned features make lasers a unique source of light and open up entirely new possibilities for the use of light beams [14].

Laser-matter interaction is one of the most important scientific fields of modern laser physics [15]. This direction has significantly enhanced the understanding of the fundamental photophysical processes occurring in matter, primarily in condensed media [16], under the influence of intense light streams of different durations and wavelengths. It has also enabled the development of the physical foundations of numerous promising applied areas related to lasers and their applications [17,18]. Laser radiation, as a coherent light by itself, is also a form of electromagnetic radiation [19]. Therefore, the interaction of laser radiation with matter is the interaction with matter of an electromagnetic wave with its specific properties and characteristics (coherence, monochromaticity, etc.). The processes of laser exposure on materials are primarily related to local heating, i.e., the transfer of energy from the electromagnetic wave into the substance [20].

Among the effects caused by laser-matter interaction, a special place is held by the effects associated with chemical transformations of matter in the field of a powerful light wave [21]. As is known, the laser beam has the potential to initiate chemical reactions via its thermal effect on chemically active media [22], as well as via light excitation of the internal degrees of freedom of atoms and molecules [23]. The reactions in chemically active laser plasma are also intensively investigated in [24]. Each of these areas of laser chemistry, which focuses on the study of thermochemical, photochemical and plasma chemical processes, solves its own special problems, has its own original research methods, and makes use of the unique properties of laser radiation in its own way [25–27]. All of these areas are connected not only by the fact that their main tool is the laser, but also by the fact that they are united by the common scientific theory of the resonance interaction of electromagnetic irradiation with quantum systems [28].

The issues of interaction of laser radiation with metals, especially in an oxidizing environment, are of great importance. In [29], a significant change in the absorption capacity of metallic materials depending on the thickness of the oxide film was reported, as well as the need to consider thermochemical processes on metal surfaces in an oxygen-containing environment. These processes significantly change the heating dynamics since the absorption capacity significantly changes as a consequence of oxide film formation, which in turn changes the temperature time dependences, introducing a significant nonlinearity in them [30].

The high spatial coherence of the laser beam makes it possible to achieve good focusing, which in turn facilitates the occurrence of chemical reactions, including the heterogeneous oxidation of metals, locally, with high spatial resolution [31]. Chemical processes in the kinetics of which the thermal effect of the laser beam plays an essential role and which occur in conditions close to thermodynamic equilibrium are the subjects of the study of laser thermochemistry [32]. Under these conditions, the problems to be solved may be related to the particularities of the formation of a spatial and temporal distribution of the temperature field in a chemically active medium under the influence of a laser beam [33]. Nonisothermicity provides the opportunity to effectively control the chemical processes, and laser irradiation can be a unique tool to achieve this goal.

Successful implementation of such control is only possible if a certain spatial intensity profile is formed in the target area of the material. In this case, the desired result of laser-matter interaction can only be achieved by using appropriate beam parameter conversion systems that can produce the required temperature exposure [34]. The effect of such laser irradiation on opaque materials is largely determined by the capability of the optical system to redistribute the energy over their surface with a predetermined intensity [35]. The variety of optical systems in use and under development demonstrates the widespread interest of researchers on this important topic [36].

If an optical element is specified, then by solving the wavefront shaping problem, we can obtain the field distribution in the area of interest. This is the forward problem of focusing [37]. In order to create an optical element that focuses the beam into a desired region of space, it is necessary to solve the inverse problem of focusing. In this case, it is necessary not only to focus the beam into a predetermined area of the focal plane, but also to form in it a predefined intensity distribution [38,39]. From a mathematical point of view, the problem of creating an optical element that produces the desired wavefield is incorrect: firstly, the solution may not exist at all; secondly, it may be unstable. However, a solution to such problems is possible [40,41]. A typical inverse problem of focusing is the problem of an optical element that focuses the beam into a predetermined region of space, e.g., the focusing of a beam into a certain figure or a line in the focal plane. Since only the intensity distribution is specified in the focusing region and the phase is arbitrary, the specified degree of freedom makes it possible to design such an optical element [42].

As a rule, it is necessary to create such dimensions of the focusing area, which are comparable with the width of the diffraction-limited spot; therefore, the diffraction has a decisive role in the formation of the predetermined intensity profile. Some tasks require focusing the beam into a zone, the longitudinal dimensions of which are much larger than the transverse dimensions, usually coinciding with the width of the diffraction-limited spot. In such cases, the problem of beam focusing into a line (a segment, a set of segments) or a curve is solved [43].

In [44–46], new techniques of pulse-periodic laser irradiation were created and a number of nanomaterials ranging from nanoporous to layered ones based on oxide-metallic nanowires were synthesized. A significant increase in the diffusion coefficient in a metallic material was described. This has been explained by the synergy of heat associated effects and laser-induced vibrations, mainly in the sound frequency range, resulting from pulse-periodic laser irradiation with pulse durations in the micro- and milli-second range [47]. The application of this synergistic effect has enabled the implementation of a new approach to the formation of quasi-one-dimensional nanostructured metal oxides by laser-matter interaction. Expanding the field of application of laser irradiation as an advanced method for enhancing mass transfer in the solid phase of metallic materials requires a detailed and comprehensive study of new opportunities related to the formation of structures with improved physical and mechanical properties [48]. Ensuring the means for controlling the laser exposure in all local areas of the irradiated zone is one of the major problems. However, it can be solved by adapting the shape of the laser beam and redistributing the energy and power density using appropriate optical systems.

The main purpose of this article is to provide systematized information on the features of improving the processes of laser-matter interaction related to the thermochemical transformations of matter in the field of a powerful light wave. The main effort is devoted to the application of techniques substantially broadening the understanding of fundamental processes occurring in matter—first of all in condensed media—under the action of intense light flows, and the use of optical systems for controlling the irradiance and spatial distributions of the wavefront. In addition to nano- and micro-technologies for creating thin oxide layers on local areas of laser-heated surfaces, the synthesis of nanostructured metal oxides is of great interest both for promising research and for a wide range of applications. Such oxide nanomaterials show unique properties that may be significantly superior to those of their macroscale analogues. The development of this research promises to have a major impact on real industrial applications.

2. Absorption of Laser Beam Energy by Metals and Semiconductors

The energy of the laser beam is first absorbed in the affected zone and then transferred to the bulk material through heat conduction processes by means of electron-phonon coupling [49–51]. The mechanisms of laser beam absorption by metals and semiconductors are quite different. The interaction of light with metals is determined by the presence of a large number of electrons so weakly bound to the crystal lattice that these electrons can be considered virtually free [52]. The electrostatic positive charge of the metal lattice ions compensates for the negative charge of these electrons. The free electrons or conduction electrons are commonly referred to as electron gas. In the field of the incident electromagnetic wave, the free electrons oscillate and emit secondary waves, which when combined produce a strong reflected wave [53]. The absorption of light by the conduction electrons is only possible through their interaction with the metal lattice and is therefore partially converted into heat [54,55]. It should be noted that in a perfect conductor, where Joule heating [56] losses are absent, the absorption equals to zero and the incident light is completely reflected [57].

The absorption of light leads to an increase in the energy of the free electrons. Since the time for establishing equilibrium in an electron gas is much shorter than the time for establishing equilibrium between electrons and the lattice of atoms, two thermodynamic subsystems emerge in the metal with different temperatures: an electron subsystem and a phonon subsystem [58]. Part of the absorbed energy is transferred by the electrons to the lattice, but the transfer efficiency is low due to the large mass difference between electrons and ions. Therefore, at first the electron gas is significantly overheated compared to the lattice. However, the increase in temperature of the electron gas only occurs until the amount of energy transferred to the lattice is equal to the amount of energy received by the electrons from the electromagnetic wave. When processing in a continuous beam or in a pulsed mode with a duration up to the nanosecond range inclusive, the exposure time of the irradiation on the material is several orders of magnitude greater than the energy exchange time between the electrons and the lattice. In this case, their temperatures are considered equivalent [59]. However, when processing with ultrafast laser pulses, the characteristic energy exchange time between these subsystems exceeds the pulse time. This means that by the end of a femtosecond laser pulse, the energy absorbed by the matter is stored in the electronic subsystem, while the lattice remains practically at its original temperature. During the process of electron-lattice relaxation, which in metals occurs in times of the order of tens of picoseconds, lattice thermal conduction is virtually absent, which is a great advantage of femtosecond laser ablation that allows for precision processing of materials [60,61].

The electrical and optical properties of semiconductors are related to the fact that the electron-filled energy levels in the valence band are separated from the conduction band by the energy gap or band gap where electron states are forbidden. Accordingly, a quantum approach is used, referring to light as a flow of photons with the energy of a light quantum. Semiconductors have a low concentration of free electrons and if the energy of the light quantum is less than the band gap width, their optical properties are similar to those of dielectrics [62,63]. The surface of a semiconductor is usually a macroscopic discontinuity of the periodicity of the crystal lattice. The electronic processes on and near the surface are influenced not only by capture and recombination centres, but also by the state of the spatial charge region, which can be determined by the existence of local surface centres. Such centres can be related both directly to periodicity rupture and to adsorbed atoms and molecules. A distinction is made between intrinsic, intraband, impurity and lattice absorption of electromagnetic radiation by semiconductors [64].

Intrinsic or interband absorption of light [65,66] occurs when the energy of the quantum is greater than the band gap. In this case, due to the internal photoelectric effect, electrons move from the valence band to the conduction band. The reflectivity of the semiconductor increases. At the same time, the absorption of radiation by free carriers enables the acceleration of electrons in the conduction zone, which leads to an increase in their concentration as a result of the thermal ionisation of the valence zone, i.e., a self-accelerating process of warming up the substance [67].

Intraband absorption or absorption by free carriers, which are electrons and holes, is inherently similar to free electron absorption in metals [68,69]. The difference is the relatively low concentration of free carriers. Impurity absorption [70] is determined by the participation of carriers with energy states within the band gap. Lattice or residual absorption [71] takes place when the irradiation interacts directly with the ions of the semiconductor and the electron subsystem remains uninvolved. In this case, there is an interaction of photon with phonon, which has a quantum character with rather wide lines.

The transfer of energy absorbed by the free carriers and converted into heat from the surface layers of the semiconductor through its volume is carried out by thermal conduction [72]. At the initial stage of the process, when the concentration of free electrons in the semiconductor is negligible, lattice heat conduction predominates. As the concentration grows, increasingly more energy is transferred by the conduction electrons, and they contribute essentially to the total thermal conductivity [73]. Energy transfer in semiconductors can also be achieved by recombination. In this case, semiconductors produce electron-hole pairs due to the absorption of laser irradiation; during recombination, these pairs transfer the energy to the crystal lattice [74].

The morphology of a metal surface also influences its optical absorption capacity. Depending on the degree of roughness, which can be either smaller or larger than the laser wavelength, the enhancement of laser beam absorption can be due to the following mechanisms. When the roughness is smaller than the wavelength, the light absorption can be due to the anti-reflective effect of sub-wavelength surface textures [75]. In the case when the roughness is larger than the wavelength, the enhancement may be due a multiple reflection at the air-metal interface [76]. The optical properties of nanomaterials are also different from their bulk counterparts [77]. An analytical model for calculating optical absorption by a rough metal surface is formulated in [78]. Figure 1 illustrates the laser beam absorption and reflection scheme and the model for absorption calculation.

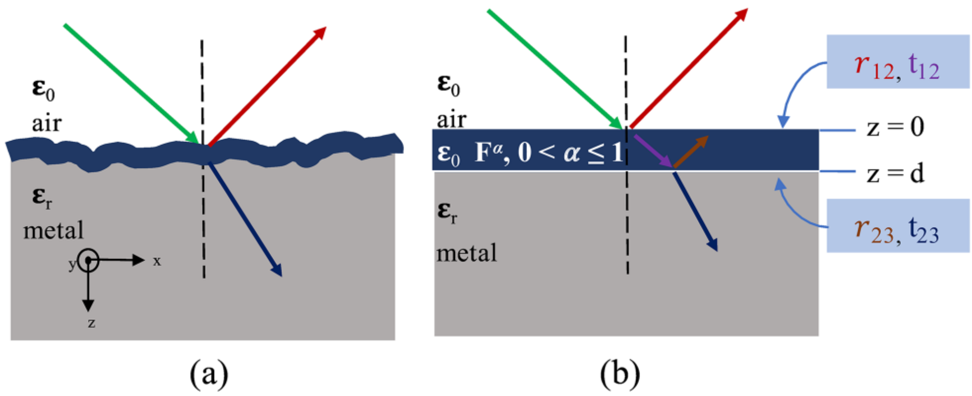

Figure 1. Scheme of laser beam absorption and reflection (**a**) and model for calculating absorption of a rough surface of a metallic material (**b**) [78].

In the model, the roughness at the air-metal interface is treated as a slab of thickness d in fractional dimensional space ($\mathbf{F}^{\alpha}$), and the parameter $0 < \alpha < 1$ is defined by the roughness value, where the value $\alpha = 1$ indicates its absence. This model [78] provides the possibility to pre-calculate the optical absorption capacity, up to high absorption values, in order to create a suitable surface roughness for metallic materials.

3. Computer Optics for Controlling the Irradiance, and Wavefront Spatial Distributions

It is known that the exposure to laser irradiation is accompanied by heat and mass transfer processes, the development of which depends on the energy as well as spatial and temporal characteristics of the laser beam [79]. The aim of the beam of light shaping process is the simultaneous control of both irradiance and wavefront spatial distributions when they can be shaped in an arbitrary manner [80,81]. For example, a beam shaper consisting of two plano-freeform lenses was developed in [80] (Figure 2): the first freeform lens produces an integrable ray mapping associated with the desired irradiance transformation, while the second lens is designed to adjust the wavefront.

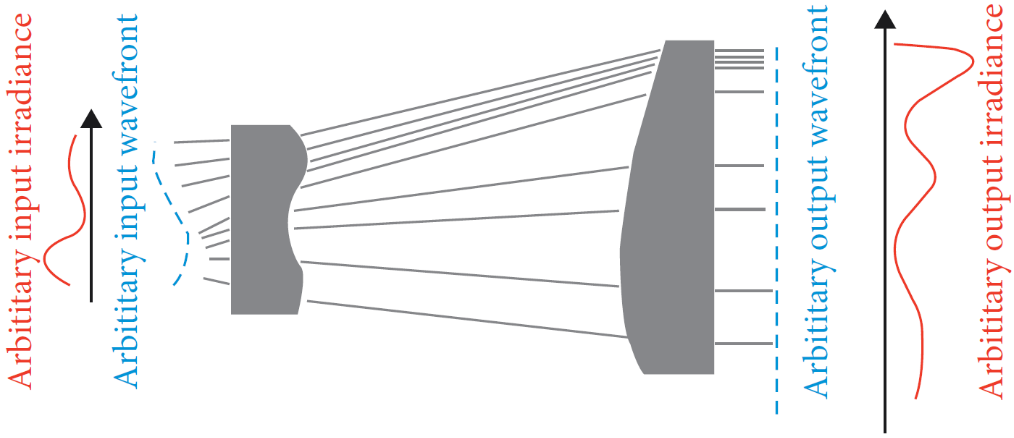

Figure 2. Beam shaper for arbitrary input/output beams. The freeform surfaces perform irradiation conversion—the first freeform lens that on the left; wavefront correction—the second lens that on the right [80].

In classical optics, the problem of creating wavefronts of the desired shape is solved by using compensating lenses [36], which allow wavefronts to be obtained as second-order rotational surfaces. However, the creation of a compensation lens is a unique task for each type of wavefront. Aspherical wavefronts of higher orders, as well as fronts without circular symmetry, appear to be virtually impossible to be shaped by compensating lenses with an acceptable complexity in general. Therefore, digital holography techniques are actively used to solve the problems of forming complex wavefronts [82,83]. In [84,85], it was shown that it is possible to reduce the quantization errors when creating aspherical fronts using binary computer-generated holograms. However, in this case only a small part of the illuminating beam energy corresponding to the first order of diffraction is diffracted into the required wavefront; a large part of the hologram resolution elements goes to the transmission of the carrier spatial frequency, while the relative opening of the wavefront is limited by the overlap of higher diffraction orders.

Several Refs. including [86–89] have shown that special circular or ring diffraction gratings, also called diffractive axicons, which have high energy efficiency not only for polyhedral, but also for binary or two-level versions, have advantages for creating axisymmetric aspherical wavefronts. Diffractive axicons provide an opportunity to diminish such disadvantages as the dependence of the generated beam parameters on the particular optical element, the dependence of the intensity distribution of the output beam on the manufacturing quality of the element and the non-uniform axial distribution of the intensity [90]. Computer optics techniques are used to create such wavefronts of complex shape [91]. One of the main goals of computer optics is to extend the range of design elements of optical systems. In addition to conventional lenses, prisms and mirrors, computational and control systems can be used to create optical elements with broader functionality [92]. One of the representatives of computer optics is a thin phase hologram, which carries explicit information about the phase component of the target wave and reconstructs it when illuminated by the reference wave. By combining such correctors with conventional lenses, optical systems with low spherical aberrations and new optical properties can be developed [93,94].

It should be noted that optics is also used directly in computing, although the focus is on connecting components of computers, communication or more fundamental systems that have some optical functions or elements that enable optical pattern recognition, etc.

The developed components such as optical logic gates, optical switches and spatial light modulators created a good starting basis for the eventual design of optical computers. Photonics is a new and promising tool for the next generation of computing hardware, and advances in digital computers are allowing us to design, simulate and build a new class of photonic devices and systems with unique tasks [95].

The approach to the creation of computer optics elements is described in detail in [96]. An optical element that reflects or transmits irradiation is characterised by an amplitude-phase reflection or transmission function. This function is determined by the conditions of the wavefield conversion problem to be solved. For the simplest cases, its analytical expression can be known, e.g., the phase function of a spherical, parabolic or cylindrical lens. However, in more complex cases, computer calculations are needed to determine this characteristic of the optical element. In this case, a computer can be used to perform numerical calculations in order to solve both the explicit problems of determining the amplitude-phase function of the optical element being created and the inverse problems. Figure 3 shows the scheme of the focusing of a laser beam where the final geometry is represented on a plane with the complex amplitude F_0 on the section $|x| \leq d$ with a focal distance $z = f$ [96]. The aperture of the optical element G is restricted by the curves $v = g_1(u)$ and $v = g_2(u)$ and by straight line segments $u = a$ and $u = b$. The function $\mu(x, \varepsilon)$ that characterises the distribution of energy in the ε-vicinity of the focusing section is represented on the right [96].

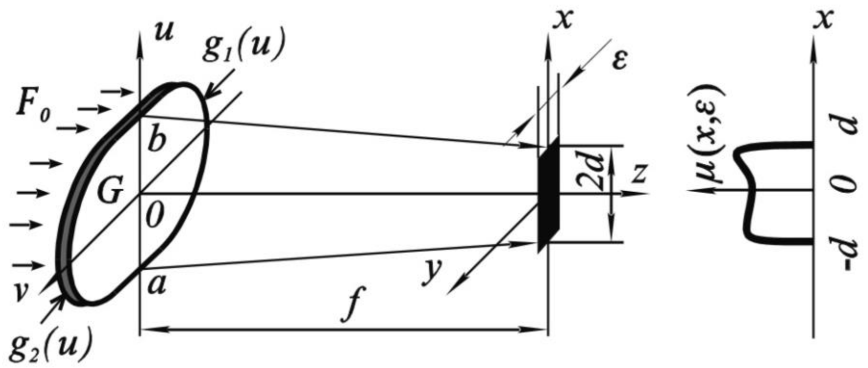

Figure 3. Focusing scheme of a laser beam with a geometry represented in two dimensions [96].

In addition to digital holography [97], the digital processing of images or fields in optical systems [98–100], the creation of optical elements for the analysis and shaping of the beam mode composition [101], the main areas of computer optics include the creation of wavefront correctors and spatial filters for optical systems, as well as solving inverse problems of diffraction theory and creating beam shaping devices based on the obtained results [102].

Using the related concepts of wavefront, beams and illuminance, it is possible to formulate the problem in the framework of geometric optics. The approximation of geometric optics implies that the characteristic dimensions of the optical surfaces and distributions of the generated light are much larger than the wavelength of light. Similarly, in [103], the problem of designing optical surfaces forming an output beam with a flat wavefront perpendicular to the z-axis and a given irradiance distribution in a certain plane $z = f_0$, situated after the optical element is solved. The beam is transformed by a refractive optical element that has two optical surfaces: f and g. The geometric formulation of the design problem of the optical element is shown in Figure 4, where the points in regions G and D correspond to the centres of the equal stream cells with which they are approximated. Refractive optical elements that convert the circular section of the beam into beams of different shapes (rectangular, triangular, cruciform) with flat wavefronts were developed.

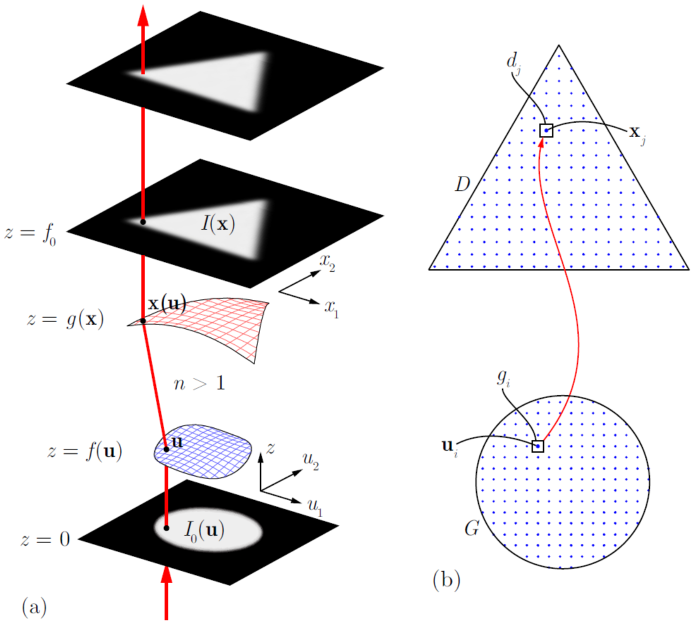

Figure 4. Geometry of the design problem of an optical element (**a**). Approximation of the circular area G and triangular area D by cells with equal streams (**b**) [103].

In [104], a solution to the three-dimensional problem of collimated beamforming design with a single freeform surface was obtained. Figure 5 shows the geometrical scheme of the freeform surface design. The unit vectors are represented by: **I**—incident beam, O—output beam and **N**—normal on plane **P**. The results can be generalised to achieve a lighting design with multiple freeform surfaces.

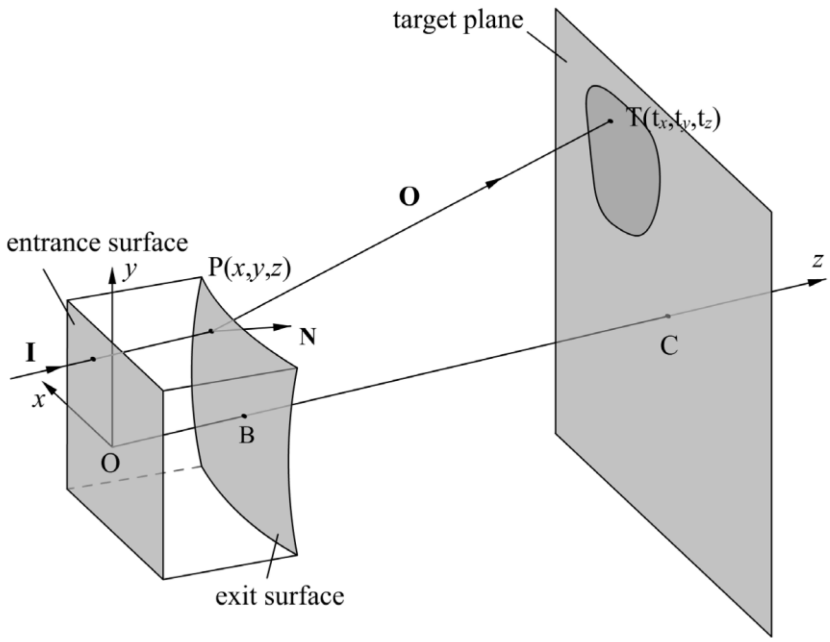

Figure 5. Geometric scheme of the freeform surface design [104].

It is well known that the problem of determining the phase characteristics of light fields occurs in various studies devoted to optics [105]. Difficulties of directly measuring the phase transformation in the optical range cause an advance in solving the problems of reconstruction of the phase characteristics of light fields using the intensity characteristics. Additional data related to the field, e.g., information on the intensity distribution that refers to another cross-section of the field, are being collected [106]. The phase problem also includes inverse problems of diffraction theory aimed at calculating the phase function of optical elements that ensure the formation of certain light structures. If the phase function of the optical element is specified, then essentially it is always possible to solve the direct problem of wave diffraction on the optical element and obtain the field distribution in the region of interest. The inverse problem is more difficult to solve but once solved, in principle any arbitrary surface of the optical element can be calculated [107]. However, such a surface cannot be made using current state-of-the-art means [108]. The freeform optics for beam shaping were presented in [109] and used with the purpose of performing the specified laser exposure in [110]. The calculation of the irradiance distribution generated by a freeform surface is carried out using the pinhole approach, the diagram of which is shown in Figure 6. The freeform surface is represented by a set of holes, each of which generates an image of the source on the target plane. The resulting irradiance distribution is defined as a superposition of all source images.

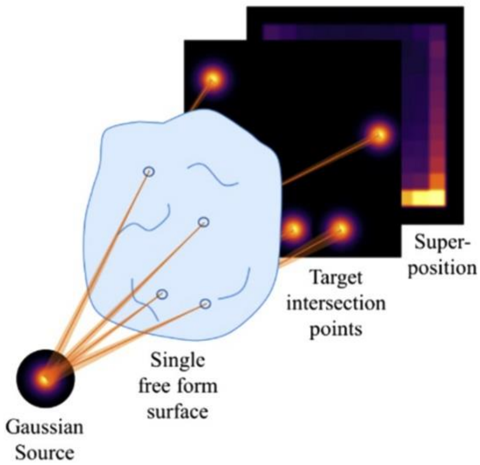

Figure 6. Diagram of the pinhole approach used to calculate the irradiance distribution generated by a freeform surface [110].

As shown in [111,112], both the calculation and production of optical elements that form a complex narrow-angle light distribution using surfaces of total internal reflection are quite challenging tasks. Currently, there are no generally accepted methods for constructing a ray-tracing function for an arbitrary region. In addition, to create complex beam distributions, the inverse problem of surface definition often cannot be solved with the required accuracy due to violation of the integrability condition. Moreover, the reflecting surface is additionally smoothed by rational B-splines during the process of mirror fabrication [113]. According to [114,115], after special corrective processing for adjusting the intensity, the "peak-to-valley" value (PV value) of freeform mirrors can reach from 500 nm to 1.5 µm, which can reduce the quality of the shaped irradiance distribution. It should be noted that the PV value only compares two local points on the surface—the highest and lowest relative to the reference surface. Local artefacts on the surface can cause the 'peak-to-valley' value to be quite large and the optics may perform quite well. Nevertheless, the PV value is an established unit of measurement and is quite widely used to measure the surface accuracy of freeform optics [116,117]. It is only in the case of significant local artefacts that an optical system with a higher PV-value may actually perform better than one with a lower PV value.

The high monochromaticity and large coherence length of the laser beam allow for the use of diffractive freeform optics for its shaping [118–120]. The diffractive freeform optics ensure the rotation of the laser beam, its spatial phase modulation, and the redistribution of its power over the treatment zone, which has a predefined shape. Each of these computer optical elements are designed as a reflecting or transmitting plate. The micro-relief of its surface is determined by the desired shape of the treatment zone, the power density distribution, and the wavelength λ of the laser irradiation. The height of the micro-relief peaks varies from the root of the peak to its top, from 0 to $\lambda/(2\cos\theta)$ for the reflective plate and from 0 to $\lambda/(n - 1)$ for the transmitting plate, where θ is the angle of incidence of the beam on the optical element and n is the refractive index of the transmitting plate material [121]. The reflective plate is either an entirely metal plate or a metal plate with a reflective coating applied over the micro-relief. The transmission plate is made from a material that is nearly transparent to the laser beam.

Diffractive optical elements synthesised by computer optics techniques allow us to perform the focusing of a laser beam into a predefined focal curve. Considering the complexity of solving the inverse focusing problem, the phase function of a diffractive optical element is more efficiently defined for focusing into simple focal curves, such as a straight-line segment, a ring, a half ring, etc. The most practical value lies in focusing the beam into a straight-line segment [122]. In most cases, a focal line of an arbitrary shape can be approximated by a set of linear segments with sufficient accuracy for practical purposes. Dividing the aperture of an optical element into several regions (by the number of line segments), each focussing the beam into a line segment of a desired length and direction, makes it possible to focus the beam into a complex focal curve. Thus, the problem of focusing into a linear segment can be regarded as a basic task for the formation of a focal line, which is a necessary step in the synthesis of diffractive optical elements for complex focal areas [123].

The maximum aperture size of a diffractive optical element is limited by three factors: physical, mathematical, and technological. The physical limitation is when the geometric optics and Kirchhoff approximations cannot be applied for large optical element apertures or large optical powers and the phase function calculation has to be carried out using the electromagnetic approach. A mathematical limitation is the lack or high complexity of methods for solving forward and backward wave diffraction problems in the general formulation [124]. By applying an iterative approach to the solution of the inverse diffraction problem under sharp focusing conditions, it is possible to obtain a complex distribution at the entry of the focusing system, creating the desired intensity distribution in the focal area. The complex distribution of the transverse components of the initial field, similar to the Laguerre-Gaussian mode with radial polarisation and focused into a light spot that is half the diffraction limit, was obtained in [125].

However, the most severe limiting factor of the maximum aperture size of diffractive optical elements is the technology of their production. The problems of developing technologies that allow for the transfer of the calculated relief function to the physical medium are also solved by a computer-controlled, software-driven technological automatic machines [126,127]. The characteristic dimensions of diffractive optical elements correspond to the order of the irradiation wavelength. Distinct from refractive optics, such optical elements for a specified and complex beam focusing may well already be created [128–130]. With the flexibility of computer-aided design and the micro-relief capabilities of modern precision technology, a unique optical tool for the desired wavefront control or for the defined shaping of laser beams has been obtained [131].

Computer optics open up new possibilities for creating optical elements that allow one to adjust the amplitude and phase distribution of the field in light beams. Optical circuits using correctors allow for the formation of a wavefront of a predefined shape. The functional purpose of diffractive optical elements is either to create a reference aspherical wavefront of arbitrary order from a planar or spherical one, or to convert one wavefront into another, referred to as compensation [132]. In optical reference schemes the wavefront from the aspherical mirror or lens under study is analysed, while the compensator forms a reference wavefront for interferometric examination of the manufactured optical surface. This analysis is carried out by comparing the examined wavefront with the reference one according to the interferometric method. The shadowing method analyses the wavefront reflected or transmitted from the inspected element and transformed by the diffractive optical compensator. The Foucault knife-edge method is easier to implement but has a low signal-to-noise ratio as the zero spatial frequency, which is the most energetic frequency, is not being used for the analysis [133].

Ref. [134] aims to develop the methods of the laser-matter interaction with the application of freeform diffractive optics, capable of redistributing the laser beam intensity. Based on the use of methods for solving the inverse problem of heat conduction, an algorithm for the calculation of the power density distribution of the laser beam was developed in order to create a desired thermal effect on materials. An improvement in the parameters of the temperature field in the chrome–nickel–molybdenum steel during laser heat treatment was demonstrated. It was shown that the redistribution of the specific power in a moving heat source influences the temperature distribution in the processed area. Equality of maximum temperatures in the centre and at the periphery of the heat-affected zone is achieved [134]. In addition to achieving more uniform temperature conditions across the width of the heat-affected zone, the proposed approach allows us to increase the width of the targeted isotherms of the temperature field. Thus, with the same laser power, larger areas can be processed per unit of time, or the number of passes can be reduced. An example of the results of the calculation of the maximum temperature values before and after specific power adjustment is shown in Figure 7. A new approach for structures formation to improve the properties of materials by laser irradiation having a predetermined distribution of energy density has been applied. This is achieved by developing models of the process and modelling the effects of laser irradiation; designing special optical systems for appropriate beam shaping; investigating the structures and properties of materials subjected to laser irradiation [135,136]. In addition to achieving a more uniform temperature regime across the width of the heat-affected zone, the introduced approach allows one to increase the width of the target temperature field isotherms.

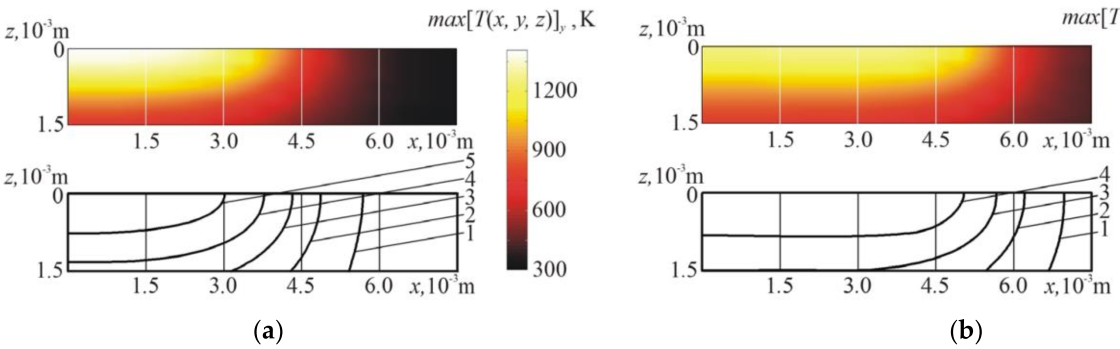

Figure 7. Maximum temperature values $max[T(x, y, z)]_y$ across the width of the heat-affected zone of a rectangle-shaped heat source, obtained by calculation considering the power 950 W, a constant linear velocity $\upsilon = 1.1 \times 10^{-2}$ m/s on the surface of chrome–nickel–molybdenum steel before (**a**) and after adjustment of power density (**b**). Temperature *T*: (isothermal: 1–600 K; 2–750 K; 3–900 K; 4–1050 K; 5–1200 K) [134].

The energy parameters of the laser beam, including the spatial distribution of the energy density in the focusing area, are essential for creating a material heating zone with the required physical properties. An improvement of the beam shaping systems with freeform optics ensures flexible control and desired redistribution of the laser beam density. The important distinguishing aspect is the application of diffractive freeform optics for beam shaping to increase the precision of energy transfer to material surfaces, including those of a complex shape [137].

4. Laser Thermochemical Processes of the Formation of Quasi-One-Dimensional Nanostructured Metal Oxides

Laser irradiation provides versatile opportunities for initiating chemical reactions through thermal exposure; through resonance excitation of energy levels of atoms and molecules;

through excitation or breaking of molecular bonds attenuated by resonant electromagnetic excitation. Investigations revealed characteristic features of the thermochemical and vibrational effects of laser irradiation when the process proceeded differently compared to conventional heating under isothermal conditions at an appropriate temperature [138].

The oxidation of metals is a process consisting of a series of sequential stages. These include the transportation of oxygen molecules from the gas volume to the boundary between the phases; the adsorption of oxygen on the surface; the transfer of reagents through the growing oxide layer; and the chemical oxidation reaction itself [139]. In this case, the total reaction rate is limited by the slowest stage. In the course of the chemical reaction under the influence of laser irradiation, the thermal and "chemical" degrees of freedom of the system become bound to each other. This connection is conditioned, on the one hand, by the so-called Arrhenius (exponential) dependence of the reaction rate constant on temperature, and, on the other hand, by the change in the absorption characteristics of the system during the chemical reaction [140].

The growth of the oxide film causes a change in the absorption capacity and hence a change in the rate of temperature increase [141,142]. The main task is to prevent the thermochemical instability, i.e., the emergence of a positive feedback in the system and an avalanche-like acceleration of the oxidation reaction after a certain characteristic time after the beginning of the irradiation, meaning the reaction activation time, which is defined as the moment when the reaction rate is at its maximum. In a commonly applied problem statement for a laser based thermochemical reaction, the temperature change in the irradiated area is described by a heat balance equation [143]. In order to calculate the heating of a metal in an oxidising atmosphere, a system of equations consisting of a thermal conductivity equation with a laser heat source; a heat transfer equation with the medium; an oxidation kinetics equation, and an equation for the change in absorptivity during oxidation is solved [144].

The monochromaticity of the laser irradiation permits the introduction of thermal energy into the system for one reaction component only if the wavelength is chosen appropriately. This selectivity of the thermal feedback leads to entirely differing types of dynamic behaviour of the system when the wavelength of the beam is shifted from one reaction component to another. Thus, if the irradiation is absorbed by the end product of the reaction and the direct reaction is accelerated by the heat, a positive feedback is created in the system. As the concentration of the product grows, the absorption increases, the temperature of the system rises, and self-acceleration of the reaction, or thermochemical instability, takes place [145]. This is the case for the growth of oxide films on metallic materials during heating by laser irradiation. If in the opposite case, for example, the reaction product absorbs less irradiation than the initial matter, a negative feedback is created in the system; thus, the chemical process slows down and self-stabilises. Therefore, the absorbed energy has the meaning of the transmission coefficient through the feedback channel. This offers the possibility of controlling the system behaviour by changing the energy or power of laser beam, and its density distribution [146].

Using a frequency-modulated laser beam in the frequency range from 3 Hz to 5 kHz, in [147,148] it has been demonstrated that the synergy of the thermal related (heat associated) effects of laser pulses and of laser-induced vibrations mainly in the sound frequency range allows for the significant increase in the diffusion coefficient in materials. The condition for intensifying mass transfer in the solid phase of the heated in an oxidation-free environment or selectively oxidable metallic materials was identified as a non-stationary stress-strain state caused by laser-induced vibrations [48]. So far, only the general behaviour was identified, however, the knowledge of this synergetic effect—a newly identified physical effect—brings about significant advancements in the development of novel processes based on the laser-matter interaction. Figure 8 shows a photo of the setup used to investigate the vibration characteristics of objects during the formation of laser-induced nanopores. Figure 9 illustrates a common dependence diagram of vibration rate from the frequency of the sample during laser irradiation. Figure 10 shows images of the sample deformations using PSV Presentation software.

Figure 8. Setup for investigating the vibration characteristics: 1—diffusion-cooled and radio-frequency excited CO_2 laser; 2—three-axis scanning laser vibrometer; 3—optical system; 4—two-coordinate measuring instrument; 5—non-contact thermometer; 6—brass sample [147].

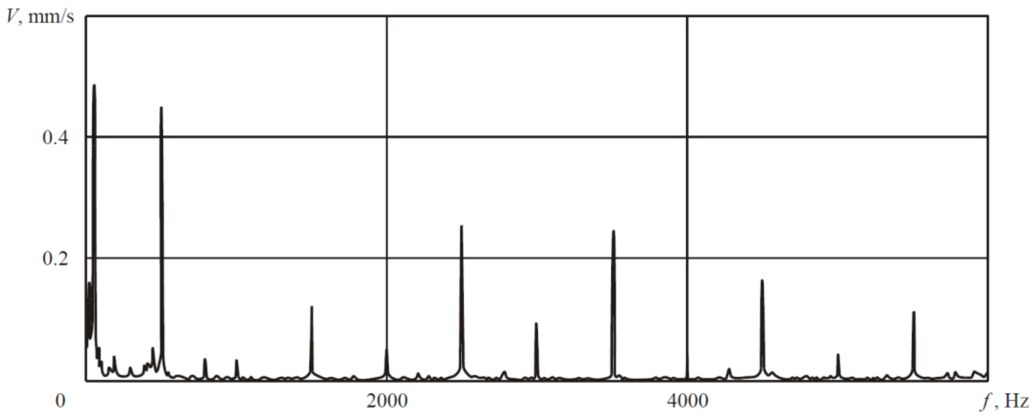

Figure 9. Vibration rate–frequency diagram, averaged over the sample surface during laser irradiation with pulse frequency 500 Hz [48].

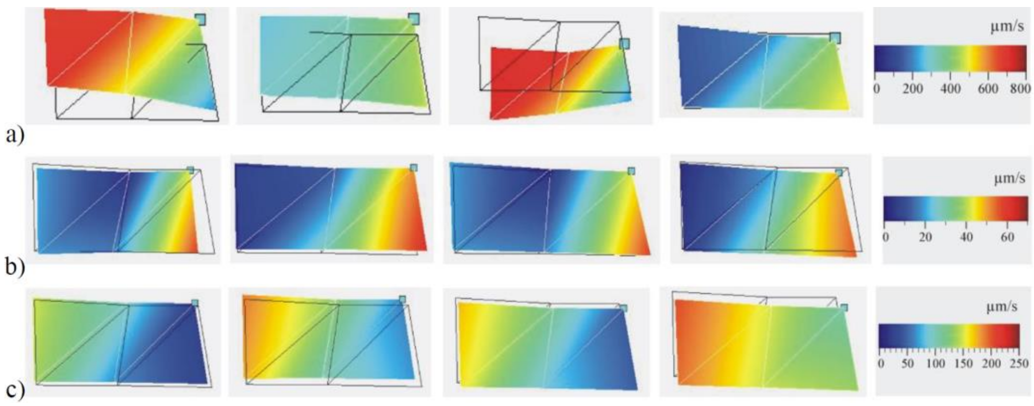

Figure 10. Images of the sample that were re-established with PSV Presentation software corresponding to the frequency of 500 Hz (**a**), 1000 Hz (**b**), 1500 Hz (**c**); with a time step of 0.05 ms [48].

By investigating the results of sample responses to laser vibrational excitation, it was found that the values of the vibration rate have local maxima in the case of frequencies which are multiples of the laser beam modulation frequency, and the values of the vibration rate in these maxima tend to decrease with increasing frequency. Moreover, increased values of the rate of vibration occur at frequencies that are near the natural oscillation frequency, for which the selected samples size was calculated according to [149,150] and was approximately 48.5 Hz.

Possibilities for the periodically pulsed laser irradiation have been evaluated and published in [151]. A significant increase (at least 200–300% in comparison to plain exposure to laser beam heating) in the diffusion coefficient in a metallic material was described. The use of this synergistic effect made it possible to synthesize a range of nanomaterials from nanoporous to layered based on quasi-one-dimensional nanostructured metal oxides on planar conductive substrates. ZnO nanowires, which had a predominantly vertical orientation, were formed on the oxide layer during laser processing with a beam power of 330 W and duration of 23 s in the region with a maximum temperature below 600 °C (Figure 11a). These had an average cross-sectional size of about 30–40 nm and a length of 500–600 nm. The nanowires had a maximum length of 1 µm, while their density was 10–20 μm^{-2}. In the region where the temperature exceeded 600 °C, nanowires alternated with nanosheets. The length of the synthesised nanowires varied between 0.5–3 µm and diameter varied between 40–60 nm. The longitudinal dimension of the nanosheets reached a value of 3 µm and the transverse size was 1 µm (Figure 11b) [151].

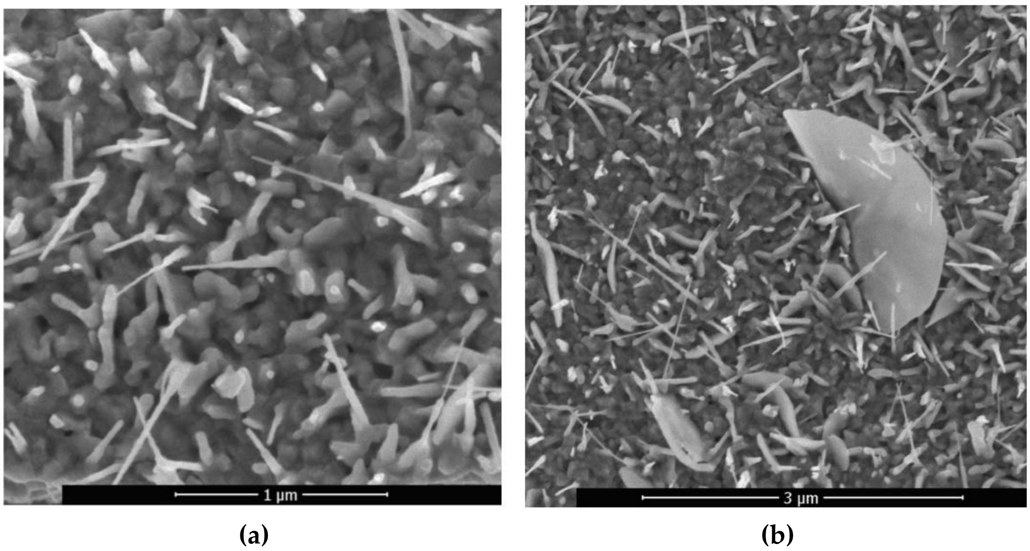

Figure 11. Scanning electron microscopy (SEM) image of ZnO nanowires (**a**), and ZnO structure consisting of nanowires and nanosheets (**b**) [151].

The main goal of the studies is to investigate the diffusion processes stimulated by the non-stationary stress-strain state in polycrystalline materials; determine conditions for the intensification of diffusion in solid state due to effects of laser pulses with tunable beam wavelength, pulse duration, and frequency. The acquired knowledge contributes to the creation of foundations, for novel photonic technologies for the formation of quasi-one-dimensional nanostructured metal oxides on planar conductive substrates, which involve the controlling of the morphology of structures under synthesis. The main effort is directed towards a better comprehension of the fundamental laser–matter interactions, as well as at the laser irradiation for enhancing mass transfer in the solid phase of materials and further study of the discovered synergy of heat exposure by laser irradiation and laser-induced vibrations [152]. Ensuring the means of control over the thermochemical reaction in local sections of the laser exposure zone is an issue that can be solved by adapting the shape of the laser beam and also by redistributing the energy and power density. In this regard, the diffractive freeform optics is an effective means to solve this issue.

5. Conclusions

The presented analysis proves the necessity of improving the processes of laser-matter interaction related to thermochemical transformations of matter in the field of powerful light wave, and optical systems for controlling the irradiance, and wavefront spatial distributions. The application of new techniques of periodical pulsed laser irradiation significantly supplements the concepts of fundamental processes occurring in matter—primarily in condensed media—under the action of intense light flows.

The high spatial coherence of the laser beam makes it possible to achieve good focusing which, in turn, allows for chemical reactions, including the heterogeneous oxidation of metals, to be carried out locally, with a high spatial resolution. The absorption mechanisms of the laser beam are distinguished between metals and semiconductors, i.e., oxides formed by a thermochemical reaction. In the case of semiconductors, the light absorption process is more complex than in metals. When analysing the thermal effects of laser irradiation on semiconductors, the following mechanisms of light absorption are distinguished: intrinsic or interband absorption, intraband absorption or absorption by free carriers, impurity absorption, and lattice or residual absorption.

Depending on the degree of roughness, the enhancement of laser beam absorption can be due to the various mechanisms. When the roughness is smaller than the wavelength, the light absorption can be due to the anti-reflective effect of sub-wavelength surface textures. In the cases where the roughness is larger than the wavelength, the enhancement may be due a multiple reflection at the air-metal interface.

During the processes of interaction of laser radiation with metals in an oxidising environment, leading to the formation of semiconductor layers on the metal surface, there is a significant change in the absorptivity of metallic materials, depending on the thickness of the oxide film. These processes significantly change the heating dynamics, making it necessary to take into consideration the change in the absorption capacity of the surface, which introduces a significant non-linearity in the time dependence of the temperature.

The enhancement of laser beam shaping systems using computer optics elements offers the possibility of efficient control of chemical processes by achieving the desired redistribution of power density. One of the main aims of computer optics is to extend the range of optical system components by means of computational tools and control systems; thus, optical elements with broader functionality can be created. The high monochromaticity and long coherence length of the laser beam make it possible to use diffractive freeform optics for beam shaping. By applying an iterative approach to solve the inverse diffraction problem under sharp focusing conditions, it is possible to obtain a complex distribution at the entrance of the focusing system, creating a desired intensity distribution in the focal region. Through the flexibility of computer design and the capabilities of modern precision technology, a unique optical tool for the necessary wavefront control and predetermined laser beam shaping is obtained.

The features of laser thermochemical processes of formation of quasi- one-dimensional nanostructured metal oxides are presented. The main task of controlling such processes is to prevent thermochemical instability, i.e., the emergence of positive feedback in the system and avalanche-like acceleration of the oxidation reaction after a certain time. The absorbed energy has the meaning of the transfer coefficient or the feedback coefficient. This makes it possible to control the behaviour of the system by changing the energy or power of the laser beam as well as their density distribution.

In cases where a metallic material exposed to a frequency-modulated laser beam in the sound or infrasound frequency range, it has been demonstrated that the synergy of the heat associated effects of laser pulses and laser-induced vibrations can significantly increase the diffusion coefficient in the materials. The main goal of research is to investigate the conditions for intensification of diffusion processes stimulated by non-stationary stress-strain state induced by laser-induced vibrations in polycrystalline materials.

Improvement of thermochemical processes of laser-matter interaction and optical systems for wavefront shaping is of considerable scientific and practical interest. The formation of oxide layers on surfaces allows for the modification of metallic materials to impart them unique strength, anti-corrosion and other performance characteristics, as well as to form structures with the required set of physical and mechanical properties. Apart from nano- and micro-technologies of creation of thin oxide layers on local surface spots heated by the laser beam and modification of the surface chemical-physical properties, the methods of nanostructured metal oxide synthesis are of great interest for both fundamental research and for a wide range of applications. Such oxide nanomaterials exhibit unique properties that can be markedly superior to those of their macroscale counterparts.

The development of this research promises to have a major impact on real-world applications in the industry. The acquired knowledge contributes to the creation of a basis for new photonic technologies aimed at the formation of quasi-one-dimensional nanostructured metal oxides on planar conductive substrates, which imply control over the morphology of the synthesised structures. This ensures that the means of control over the thermochemical reaction in local sections of the laser exposure zone can be performed by adapting the shape of the laser beam and also by redistributing the energy and power density using the diffractive freeform optics.

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