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Formation of nanoporous structures in metallic materials by pulse-periodic laser treatment

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A method of the formation of nanoporous structures in metallic materials by pulse-periodic laser treatment was developed. In this study, the multicomponent aluminum–iron brass was considered and the nanoporous structure across the entire cross section of the material with a thickness of 50 μm was formed. The method was implemented using a CO_2 laser processing unit. The pulse-periodic laser treatment of the Cu–Zn–Al–Fe alloy with pulse frequency of 5 Hz has led to the formation of nanosized cavities due to accumulation of internal stresses during cyclic heating and cooling at high speeds. It was determined that the pores of a channel type with average widths of 80–100 nm are formed in the central region of the heat-affected zone during laser action with thermocycling. When implementing the chosen conditions of the pulse-periodic laser processing, the localness in depth and area of the physical processes occurring in the heat-affected zone is ensured, while maintaining the original properties of the material and the absence of significant deformations in the rest of the volume. This patented process is perspective for the production not only catalysts for chemical reactions, but for ultrafiltration and microfiltration membranes as well.

1. Introduction

Nanoporous materials with a high efficiency are used in such advanced industries as biotechnology, hydrogen and hydrocarbon power engineering, chemical, petrochemical, food and pharmaceutical industries, as well as for solving a number of critical issues related to preparation and purification of drinking water [1–3]. Improved physical, mechanical and technological properties of metallic nanoporous materials determine their advantages over currently used polymeric and ceramic materials. Generally, for the manufacturing of metallic nanoporous materials, chemical or electrochemical methods are used, such as electrochemical dealloying [4,5], selective anodic etching [6] and preferential dissolution [7,8]. In the latter case, the use of laser energy in the presence of an electrolyte, allows to increase the effect of preferential dissolution of the certain phase of the metallic material to create a permeable zone in its structure [9]. The commonly used techniques of fabrication of the metallic nanoporous materials have considerable technological constraints on the stability of pore sizes, and created products have reduced mechanical properties and the relatively high cost. In this regard the issues of development of new methods of the formation of nanoporous structures in metallic materials have great scientific and practical interest.

The application of laser action with high pulse repetition frequency is a progressive trend of nanoporous layer creation on the surface of metallic materials. The conditions of the formation of nanoporous structures by laser treatment in the metallic material, a two-component Cu–Zn alloy, were determined in Refs. [10,11]. Such structure is formed due to the creation of vacancies and their coagulation as a result of zinc sublimation from the surface of the material that leads to creation of concentration gradient, and diffusion to the surface of the component with relatively high vapor pressure. A condition for intensification of mass transfer in the solid phase of metallic materials is non-stationary local deformation caused by high-energy external effects [12–14]. At that, the exposure to pulse-periodic laser radiation with a pulse frequency up to 5000 Hz allows to form a sustained stress state on the surface of the samples. It is established that as a result of laser treatment at the surface layer of the material, the formation of nanopores takes place, which rather evenly distributed over the area; branched pores with distinctive dendrite structure are also formed. Diffractive optical elements–focusators of laser radiation were used to create a required power density distribution in cross-section of the laser beam [15–19].

A significant amount of researches were devoted to the questions of laser treatment; however, the processes of laser thermocycling were applied up to now only for testing materials and products from

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them [20–23]. In Refs. [24,25] a new possibility of nanoporous structures formation on the surface of metallic materials by the cyclic elasto-plastic deformation during the laser treatment with a small frequency of pulse-periodic radiation was implemented. Such an approach allows realizing the processes of nanoporous structures formation with the use of a much wider range of lasers. In this case the action of internal stresses resulting from zonal high-energy impact is the cause for the formation of nanosize cavities in alloys. The structures of metal materials with a high specific surface area have been obtained, which can be used for the manufacturing of catalysts for chemical reactions.

Among the main baromembrane processes are ultrafiltration and microfiltration. Metallic membranes have higher mechanical strength, thermal conductivity, thermal and chemical resistance; that allow cleaning by the reverse current of fluid and by the calcinations. In their manufacturing, an important aim is the formation of porous structures throughout the thickness of the metal material. The purpose of this research was to determine the possibility of nanoporous structures formation in metallic foils across the entire cross section by pulse-periodic laser treatment; it is needed to the increase of the potential for the practical use of this process. This will provide an opportunity to create nanoporous metal materials not only for use in the catalytic synthesis of chemical compounds, but also for filtration of gases and liquids in the ecology, nuclear energy, medicine, microbiology, food, pharmaceutical, electronic and other industries.

2. Experimental details

A multicomponent Cu–Zn–Al–Fe alloy (aluminum–iron brass with a copper content of 58–61%, aluminum and iron of 0.75–1.5%, manganese of 0.1–0.6%, and the rest is zinc) was selected as studied material. This complex-alloyed brass, having a relatively high mechanical properties and satisfactory corrosion resistance, is used for high strength products. Pre-grinded samples of size 25×15 mm^2 of the brass with thickness of 50 μm were placed on a metallic substrate.

The energy action on the samples of the Cu–Zn–Al–Fe alloy was effected using the gas CO_2 laser ROFIN DC 010 with diffusion cooling and high-frequency pumping. The output power of the laser in the pulse-periodic mode of generation is adjustable in the range of 100–1000 W, the pulse frequency is from 2 to 5000 Hz, the pulse duration from 0.026 to 125 ms. Laser treatment of the surface of the material was performed using an optical device consisting of two telescopic systems, between which a stencil with a round hole was placed. The first telescopic system was used for the expansion of the laser beam, and the second one projected the image on the surface of the material. A diffractive optical elements–focusators of laser radiation had been used as a focusing element of the second telescopic system [26,27].

3. Results and discussion

A possibility of nanoporous structures formation in metallic material across the entire cross section of the samples with a thickness of 50 μm by pulse-periodic laser treatment with thermocycling has been studied. The required temperature rate conditions of laser processing to implement the necessary exposure for a limited in area and depth of the material volume in order to form the nanoporous structures were chosen by changing the laser power in the range of 150–300 W, pulse frequency from 3 to 6 Hz, and the diameter of the laser spot on the sample surface from 5 to 15 mm. The conditions of the pulse-periodic laser processing have been chosen, by which in the center of the heat-affected zone in the material under study was formed a plot with the corrugation-shaped relief and with a clear border. This plot that has a form of a circle with a diameter more than 4 mm is shown in Fig. 1. The corrugation-shaped relief was formed under following conditions: the laser power of 250 W, the pulse frequency of 5 Hz and the diameter of the laser spot on the sample surface of 7 mm. The reverse side of samples has a relatively more smooth surface relief (Fig. 2). The peripheral area of the heat-affected zone is shown in Fig. 3; it is characterized by the presence of the corrugations that oriented towards radial direction. Formation of the corrugations is a sign of the increasing volume of local plots of multicomponent aluminum–iron brass in the zones of the high temperature differences. Elemental analysis of the material before and after laser treatment was performed. The analytical scanning electron microscope VEGA\\SB, Tescan was used, equipped with the system

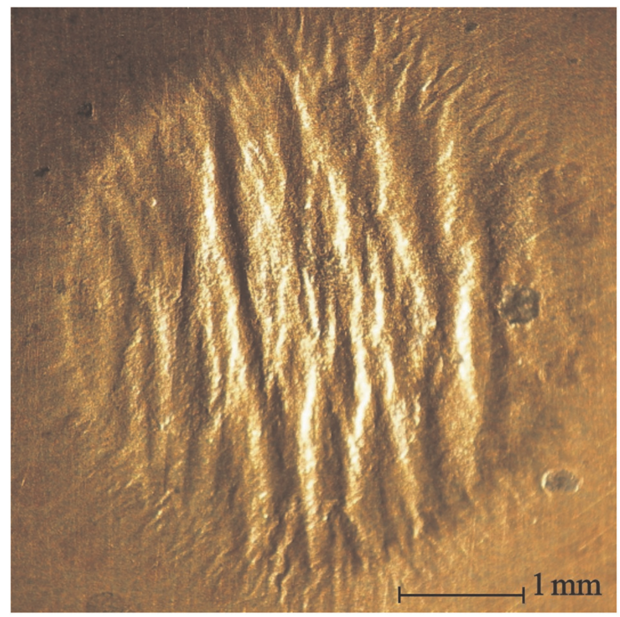

Fig. 1. The sample surface of the Cu–Zn–Al–Fe alloy with a thickness of 50 μm after laser treatment with thermocycling.

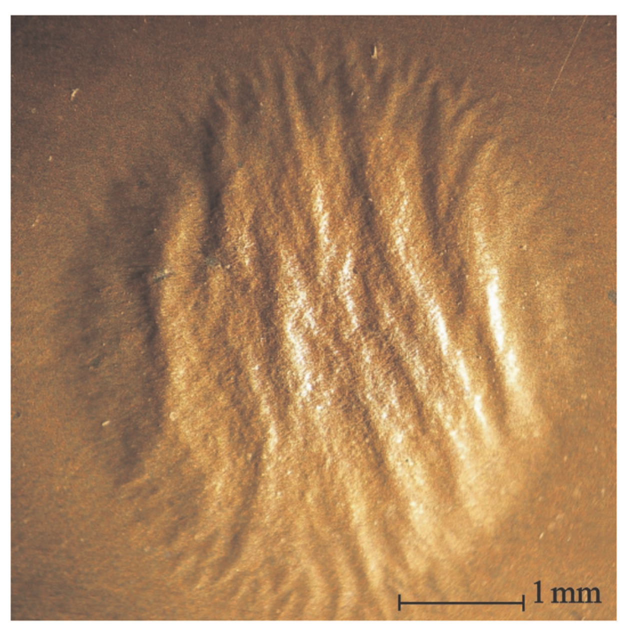

Fig. 2. The reverse side of the sample with a relatively more smooth surface relief.