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Effect of the laser heat treatment on the formation of the gradient structures in alloys based on Fe – Cr – Ni system

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Abstract. The possibility of producing gradient materials, i.e. materials with pre-set distribution of areas having fundamentally different physical and mechanical characteristics, with the help of laser heat treatment was investigated. Using as an example austenitic-martensitic alloys of iron-chromium-nickel, subjected to cold plastic deformation led to formation of martensite, we show that using laser at the temperature higher than the temperature of reverse martensite transformation leads to the formation of areas of high-strength austenite having predetermined form inside the martensite matrix. Influence of austenite areas geometry on mechanical properties of gradient material was studied.

1. Introduction
Martensite transformation and reverse martensite transformation in iron-based alloys with considerable temperature hysteresis between them [1,2] provide opportunities for producing alloys with different content and combination of martensite and austenite, and therefore materials with various physical and mechanical properties. Using martensite transformation and reverse martensite transformation allows obtaining materials structure and properties of which are pre-set along the article – so-called gradient materials. For instance, there are materials with pre-set distribution of macroscopic areas with considerably different magnetic and mechanical properties. In [3,4] the possibility of producing such materials on the base of austenitic-martensitic alloys of iron-chromium-nickel was considered. In order to get ferromagnetic part of such materials cold plastic deformation was used, as a result of which martensite transformation took place and led to forming of deformation-induced martensite. Austenite areas formation was obtained by heating at the temperature higher than the temperature of reverse martensite transformation. In [3,4] laser irradiation was used as the heat source. Using lasers for heat treatment leading to austenite formation is necessary due to high heating rate which provides, according to [5], austenite stability in the wide range of temperatures including room temperature. Paper [6] investigates connection between mechanical properties and structure of austenite, formed by laser heating. Microhardness measurements of austenite reached 3000 MPa, which is lower than that of martensite (5500 MPa), but considerably higher than that of “ordinary” austenite, formed in alloy at hot deformation or long-term holding at temperatures higher than the
temperature of reverse martensite transformation. The observed hardening of austenite is connected with the process of martensite-austenite transformation at ultra-fast heating, which occurs by a shearing mechanism. Electron-microscopic research [6] shows that transformations occurring by the same mechanism lead to keeping in austenite structure defects typical for deformation-induced martensite that provides its high strength. Using cyclic laser treatment (cycle of heating and cooling) leads to additional hardening of austenite that, according to [7], is connected to splitting its particles to nanocrystalline state.

This paper continues research of laser heat treatment influence on austenite formation in iron-chromium-nickel austenitic-martensitic alloys; in particular, direct mechanical tests were conducted by acquiring stress-deformation curves (stress-strain curves) for samples of gradient materials with various geometry of austenite areas.

2. Materials and methods of study
For our study we chose iron-chromium-nickel austenitic-martensitic alloys, their composition is given in table 1.

| Table 1. The chemical composition of studied alloys. |
|-----------------------------------------------|
| The content of alloying elements, masses (%)  |
| Cr    | Ni | V  | Mn      | Si  | C    | Fe          |
| 16.5  | 7.5| 0.04| 1.0     | 0.47| 0.07| Balance     |

Alloys of given composition were smelted in open induction furnace with using pure burden materials and applied on the billet 25 mm thick. The billet was firstly exposed to hot deformation in the temperature range of 1000 – 1200 °C on the sheet 4 mm thick and then to the cold deformation with reduction of 75% on the sheet 1 mm thick. After hot deformation the alloy had the structure of paramagnetic austenite with relatively low microhardness of 1500 MPa. During the cold deformation there was intensive formation of ferromagnetic austenite, and its microhardness increased to 5500 MPa, and there appeared saturation magnetic moment of 1.4 T. For obtaining austenite areas we used laser heating in the range of reverse martensite transformation temperatures, which were 850 – 900 °C according to magnetometrical measurements.

For the experiment on laser heat treatment we used YLS–1500–SM single-mode Ytterbium-doped continuous fiber laser (manufactured by IPG Photonics), its basic technical characteristics are given in table 2. For making local areas having pre-set geometry mask-plate 3 mm thick made of D16T alloy was put on the top of the sample. Focused laser radiation reached stack of the sample and mask-plate from above and in scanning mode it covered all the surface of the sample. Mask-plate protected the areas of the sample, which were not exposed to the radiation and therefore were not heated.

| Table 2. Main technical characteristics of the laser. |
|-----------------------------------------------|
| №       | Characteristics                  | Type / Value     |
| 1       | Mode of operation                | continuous-wave  |
| 2       | Maximum average power (W)       | 1500             |
| 3       | Power tenability (%)             | 10 – 100         |
| 4       | Polarization                     | random           |
| 5       | Central wavelength range (nm)    | 1070 ± 10        |
| 6       | Laser linewidth (nm)             | 4.55             |
| 7       | Maximum power consumption (kW)   | 4.2              |

In order to form austenite areas in martensite matrix of alloy the sample was placed under a mask-plate with a notch, geometrically corresponding to a processed zone. There were three types of notches: round-shaped area (G0), cross stripe (G1) and inclined stripe (G2). The notches in the mask-plates were made from the condition of equality of the zones areas of samples, which were laser heat treated. In the course of experiment studying the influence of laser heat treatment modes on
mechanical properties of the gradient material samples the radiation treatment of the local zones was done one time or five times. Every cycle “heating-cooling” included laser treatment of samples local zones from two sides. That was necessary for phase transformation of martensite to austenite throughout the thickness of the sample with minimal tapering of the formed austenite area. The value of the austenite remained constant for all the samples regardless the geometry of the treated zone and it was 0.3 cm$^3$.

Table 3 shows main parameters of laser heat treatment mode, formed austenite areas of various geometry, and also the temperatures reached by the sample surface during the treatment.

Table 3. Parameters of laser heat treatment mode.

| Geometry of the processed area | G0  | G1  | G2  |
|-------------------------------|-----|-----|-----|
| Linear speed of sample surface scanning by laser beam (m/min) | 60  |     |     |
| Laser radiation power (W)     | 350 |     |     |
| Density of the lines during scanning (line/mm) | 20  |     |     |
| Number of passes              | 6   |     |     |
| Power density (W/cm$^2$)      | $7\times10^6$ |     |     |
| Surface temperature (°C)      | 875 | 850 | 900 |

To establish the fact of austenite formation during the laser heat treatment we conducted magnetooptic study, which allowed constructing spatial distribution of magnetization in the samples, exposed to laser treatment [8].

For uniaxial static tensile testing with using of the samples, exposed to plastic deformation and later laser heating, electromechanical tension testing machine Instron 5982 was used. In the course of experiment the values of loads applied to the samples and shifts of grip relatively to the initial position were recorded with high precision. These data allowed plotting the stress-strain curves for samples with various geometry of the treated areas.

Using method of measuring tangential displacements with the help of the Instron 5982 setup spatial distribution of longitudinal and transverse relative transformations on the surface of the samples containing various types of austenite areas was visualized.

Samples after 5 cycles of laser treatment were studied according to the method of transmission electron microscopy. Blanks for thin foils with thickness ~ 1 mm were cut so as to make the research zone correspond to the austenite zone. Samples preparation for transmission electron microscopy was carried out by electropolishing method with “window” technique at the temperature of 20 – 50 °C and voltage ~ 20 V. The study of fine structure was conducted at the accelerating voltage 120 kV. The analysis of microstructure was performed by comparing bright-field and dark-field electron microscope images and corresponding micro-diffraction images.

3. Experiments results
Figure 1 shows curves “Stress-Deformation” for samples of gradient materials with various geometry of austenite areas formed during laser heat treatment, and also stress-strain curve for cold deformation sample. The data shows that forming austenite areas leads to decreasing of ultimate strength compared to cold deformation sample. At the same time the maximum decrease is observed for round-shapes austenite samples, while the minimum decrease is observed for the transverse ones. Nevertheless, strength decrease that takes place during austenite formation is not so great compared to hot deformation induced austenite or austenite formed during heat treatment of cold-deformed samples at the temperature 850 °C during one hour.
Figure 1. Graphical dependencies “Stress – Deformation” in the samples of different kinds: “left” is for 1 cycle “heating-cooling”, “right” is for 5 cycles “heating-cooling”.

Table 4 shows measurement results of mechanical properties of gradient materials with different austenite area geometry formed at one cycle “heating-cooling”.

**Table 4. Mechanical properties of gradient materials with different austenite area geometry.**

| Sample structure | Austenite area geometry | Ultimate strength (MPa) | Microhardness (MPa) |
|------------------|-------------------------|-------------------------|---------------------|
| Deformation martensite | –                       | 1700.95                 | 5500                |
| Martensite with austenite areas | G0                      | 956.01                  |                     |
|                        | G1                      | 995.14                  |                     |
|                        | G2                      | 1372.63                 |                     |
| Ordinary austenite    | –                       | 520.25                  | 1500                |

Figure 2 shows electron microscope images of austenite microstructures in 5G0 and 5G1 samples after 5 cycles of laser treatment. Depending on geometry and location of irradiated zones in the samples, microstructure of austenite phase has different characteristics. 5G0 sample shows clearly expressed dislocation substructure with stacking faults, whereas 5G1 sample shows deformation twins.

Figure 2. Electron microscope image of 5G0 and 5G1 samples microstructure after 5 cycles of laser treatment: a – austenite microstructure in 5G0 sample with corresponding micro-diffraction; b – austenite microstructure in 5G1 sample with corresponding micro-diffraction.
Data given in table 4 show considerable hardening of austenite obtained by laser heat treatment compared to ordinary austenite formed by hot deformation or annealing cold-deformed austenite sample at the temperature of 850 °C during 1 hour.

Differences in ultimate strength values of samples with different austenite geometry areas are connected with different structures of internal stresses, appearing during alloy deformation.

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