Investigation of the effect of cyclic laser heating for creating dispersed structures in the austenitic-martensitic alloys based on Fe-Cr-Ni system

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Abstract. The effect of cyclic laser heating on the formation of the austenite structure in the austenitic-martensitic alloys based on Fe-Cr-Ni system is investigated. It is shown that under the influence of ultra-fast laser heating on the martensite, which was formed during plastic deformation, the reverse martensitic transformation occurs, and austenite with high strength characteristics is formed. Repeated and multiple laser heating effectively grinds areas of austenite to a size close to the large nanoparticles. There is an additional increase in the strength characteristics of austenite as a result of this fragmentation.

1. Introduction

Production of metallic materials that combine high strength and plastic properties is one of the main tasks of modern metallurgy. Typically, increasing the strength of the alloys is accompanied by decrease of its ductility. One known exception to this rule is the grinding of the crystal structure down to the nano-crystalline state. Currently, such materials are prepared by methods of crystallization from the amorphous state, mechanical alloying and severe plastic deformation. Neither of these methods provides a nano-crystalline structure in the large items. One of the perspective methods of preparing dispersed structures in the large items is a cyclic thermal processing comprising multiple heating-cooling cycles. In [1] it was shown that the use heating-cooling cycles in the iron-nickel system leads to a sequence of transformations $\alpha \rightarrow \gamma \rightarrow \alpha$ and grinding of α-phase particles.

In the present study we investigated the possibility of the γ-phase dispersed particles formation by cyclic laser heat treatment. The austenitic-martensitic alloys based on Fe-Cr-Ni system were selected for studying. The need to obtain the γ-phase with high strength properties in these alloys is associated with the fact that they are the basis for the creation of "gradient" materials, in which areas of high strength α- and γ-phases are distributed in a specific manner [2-4].

2. Materials and research methods

The alloy based on Fe-Cr-Ni system with the following chemical composition: Cr - 16.5%, Ni - 7.0%, C - 0.2%, Fe - base, was selected for the researches [5]. At first, the conditions of obtaining α- and γ-phases were defined for this alloy. Methods of X-ray diffraction analysis, dilatometric and magnetometric studies were used for this purpose. Mainly austenite was present in the alloy after its melting in the open furnace and hot deformation at temperature 1000-1150 ºC. The cold plastic

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deformation with at least 75% reduction was performed to obtain martensite. Such deformation led to γ→α transformation with up to 70% α-phase formation. Magnetometric studies showed that complete conversion of martensite formed during cold deformation to austenite was achieved after heating up to temperatures 1025-1075 ºC. During subsequent cooling, two scenarios can be implemented. In the first case, isothermal γ→α-transformation was observed upon cooling at room temperature after slow heating. In the second case, after rapid heating (not slower than 300ºC/min) and cooling at room temperature, only γ-phase was fixed. It was found that the formation of α- and γ-phases during such cooling did not depend on the heating temperature, provided the cooling was carried out starting from the temperature range of γ-phase existence. These results are consistent with the data presented earlier in [6].

The cyclic ultrafast laser heating of deformed α-phase with temperatures up to 1050 ºC was used for fixing the γ-phase at a room temperature and its subsequent grinding. Ten cycles of heating-cooling were applied. The samples for X-ray examinations and the foils for electron microscopy studies were prepared after each cycle of heating-cooling.

An ytterbium fiber laser (wavelength λ = 1068 nm) with a maximum power of 5 kW, operating in continuous mode, was used for ultra-fast heating of samples. The laser was connected to the optical scanning head. In the optical head construction two mirrors were provided, which allowed to deflect the laser beam in mutually orthogonal directions. Thus, the maximum laser processing surface was 30×30 cm^2.

Mask-plate (3 mm thick duralumin) was superimposed on the top side of the sample to heat up the sample by laser irradiation in the required zones and thus to create the necessary geometry of thermal influence zones. A stack of the mask-plate and the sample was irradiated by the laser beam from the top side. The laser beam operated in the scanning mode travelled the entire surface of the stack. Certain areas of the sample were protected (shielded) by the mask-plate, hence they were not subjected to laser irradiation, and, consequently, were not heated. Thus, only the required area of the sample was treated by laser radiation and was heated to a predetermined temperature. Different samples were heated from 1 to 10 times. Each sample was cooled in air to a room temperature after the laser heat treatment.

Linear speed of surface scanning by laser beam was constant (30 m/min). The diameter of the laser spot on the sample surface was 0.5 mm, and the distance between the scan paths was 0.25 mm, resulting in 50% degree of overlap between the scan paths. Samples had to be heated up to a temperature of 1050 ºC. The laser power used for the first heating of the sample and for the subsequent heatings was different. All processing parameters are listed in table 1, including the temperature to which the surface of the sample was heated.

We controlled the heating temperature of the sample surface with a thermal imager (model T650sc by FLIR Systems). The device allows to register the temperature with an accuracy of 1ºC in a temperature range from 0 to 2000 ºC.

Table 1. Parameters of laser irradiation and temperature of heating

Quantity of heating cycles	Laser mode	Linear speed of scanning (m/min)	Diameter of the laser spot (μm)	Overlap of scanning paths (%)	Laser power (W)	Temperature of the sample surface heating (ºC)
1	Continuous, λ=1068 nm	30	500	50	1260	1050
2 .. 10					925	

X-ray diffraction analysis was performed on a DRON-3 unit with the Fe radiation. The quantity of γ-phase and α-phase and degree of its dispersion were determined for the following cases: in the initial state (after the plastic deformation), and after the first, seventh and tenth heating-cooling cycles. The data are given in table 2. The phase content was determined by the method of homologous pairs, and the degree of dispersion - by line broadening. Electron microscopic studies were performed on a microscope with an accelerating voltage of 120 kV.

3. Experimental results and discussion

As a result of X-ray diffraction analysis, it was found that in the initial state (after plastic deformation) the alloy consisted essentially of a single α-phase. The structure of alloy changed dramatically after the first heating-cooling cycle, when up to 70% of γ-phase was formed. The quantity of γ-phase increased slightly after seven and ten heating-cooling cycles, while there was γ-phase's line broadening, indicating an increase in its degree of dispersion (table 2).

Table 2. Parameters of structures in the austenitic-martensitic alloys based on Fe-Cr-Ni system after various treatments

Processing mode	γ-phase content (%)	Line broadening 220_{α} γ-phase (degree)	Average size of the γ-phase domains (μm)
Plastic deformation	-	-	-
Plastic deformation followed by one cycle of heating-cooling	75	0,75	2,0
Plastic deformation followed by seven cycles of heating-cooling	80	0,8	1,0
Plastic deformation followed by ten cycles of heating-cooling	90	0,83	0,25

Electron microscopic studies of the microstructure of the alloy showed that the structure in the initial deformed state was a mixture of highly deformed martensite and a minor amount of residual austenite fragments with sizes up to 0.5 μm (figure 1, a). After one cycle of heating-cooling the microstructure of sample was sufficiently uniform. It represented recrystallized structure of irregularly shaped austenite grains with sizes up to 2 μm having high density of dislocations, twins and a large amount of stacking faults (figure 1, b). Inhomogeneous microstructure was observed in the samples after seven cycles of heating-cooling. The structure was a mixture of highly fragmented austenite resulting from the reverse α→γ transformation, which inherited the defect structure of martensite, and the areas of the initial stage of recrystallization, which were highly fragmented austenite with some fine recrystallized grains with size not greater than 1 μm. Completely recrystallized areas with larger grains (up to 2 μm) with a high dislocation density and a large quantity of stacking faults were also observed. Generally a mixed microstructure of fragmented austenite with small recrystallized grains was dominant (figure 1, c). After ten cycles of heating-cooling (figure 1, d) of the samples we observed, firstly, areas of strongly fragmented austenite resulting from the reverse α→γ conversion, and secondly, the areas of an initial stage of recrystallization, representing the large quantity of fine recrystallized grains with size from about 0.1 μm to about 0.5 μm. These grains are quite evenly spaced in a fragmented austenitic matrix. Also, a small quantity of large recrystallized grains with a high degree of defects was found.

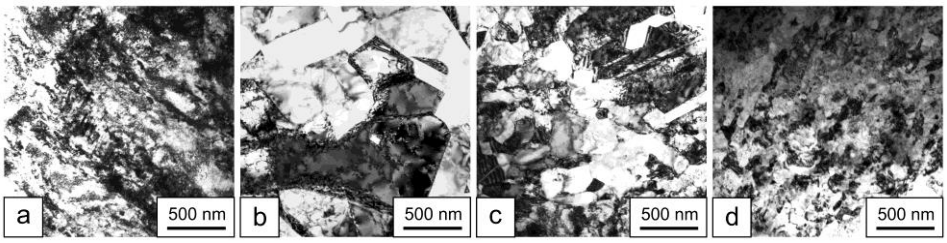


Figure 1. The microstructure of alloy after plastic deformation (a); plastic deformation and subsequent heating-cooling cycles: one cycle (b), seven cycles (c), ten cycles (d).

Thus, the results of electron microscopic examination of the microstructure were correlated with the data of X-ray analysis. These results showed that transformation of significant amount of α-phase to γ-phase occurred after one heating-cooling cycle. Grinding of the phase to the large nanoparticles occurred after repeated laser heating-cooling cycles.

Microhardness of γ-phase after one heating-cooling cycle reached 3000 MPa. This result is significantly higher than in the case of the austenite formation in the alloy after melting and hot deformation. This indicates that the strength of the γ-phase generated by laser heating substantially

exceeds its typical strength values. Apparently, this is due to inheritance of the defects of deformed martensite in the structure of austenite. Reducing the size of γ-phase with increasing quantity of heating-cooling cycles results in further strengthening while maintaining a high degree of plasticity of austenite.

4. Conclusions

Laser cyclic heat treatment of austenitic-martensitic alloys based on Fe-Cr-Ni system leads to fragmentation of particles of γ-phase to the formation of large nanoparticles. In this case, the strength characteristics of austenite improve significantly while maintaining its high ductility.

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