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Features of the Recovery Process of Austenitic Stainless Steel Obtained by Selective Laser Melting

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Abstract—The microstructure and microhardness of 316L-type austenitic stainless steel obtained by selective laser melting and then annealed at 900–1200°C have been studied. This paper is aimed at clarifying the features of the recovery process in the steel. The experimental samples were developed by selective laser melting using a ProX200 3D system in a nitrogen atmosphere without platform preheating. The laser power of 240 W, the beam speed of 1070 mm/s, the distance between tracks of 80 μm, and the layer thickness of 30 μm were applied. Annealing was carried out at temperatures of 900–1200°C for 1 h and at temperatures of 1000 and 1100°C for 1 to 10 h. It was found that the recovery processes at temperatures of 900–1100°C developed with a very sluggish kinetics. After 1 h annealing at 900–1100°C, the steel microstructure steel was characterized by a grain size of 25 ± 1 μm, and a dislocation density of 8.7×10^{13} m^{-2}. An increase in the annealing duration to 10 h did not lead to significant changes in the microstructure of the steel samples annealed at 1000°C, whereas the samples annealed at 1100°C experienced the developmen of primary recrystallization resulting in twofold decrease in the dislocation density. The development of recovery in the present steel during annealing at temperatures of 900–1100°C could be expressed by Arrhenius-type relationship with a quite low activation energy of about 10 kJ/mol.

Keywords: recovery, austenitic stainless steel, annealing, selective laser melting, additive manufacturing

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INTRODUCTION

At present, additive technologies are promising areas of modern materials science, since they allow the production of products of a very complex or even unique shape. Among the wide variety of types of additive technologies, a special place is occupied by the technology of selective laser melting (Selective Laser Melting—SLM or 3D metal printing), in which the formation of a three-dimensional product occurs by sequential layer-by-layer deposition of metal powder under the influence of focused laser beam. Among various metals and alloys, 316L-type austenitic stainless steel is one of the most frequently used materials for applications in medicine [1]. The method of selective laser melting makes it possible to produce stainless steel with both high strength and ductility, which is difficult to achieve by traditional metallurgical casting methods [2]. Considering the fast heating and cooling cycle of successive layers in the selective laser melting process, the influence of energy absorption and storage on the quality of products and the productivity of the process is almost inevitable. The properties of austenitic stainless steel obtained by additive manufacturing can be controlled by post-processing heat treatment, when the selecting the desired microstructure should be formed under appropriate conditions, i.e., temperatures and annealing times. This work is devoted to study the features of microstructural changes occurring in a 3D printed steel upon annealing.

EXPERIMENTAL

The chemical composition of the developed 316L steel samples consists of 0.022 C, 0.20 N, 0.35 Si, 1.11 Mn, 17.1 Cr, 12.6 Ni, 2.56 Mo, 0.01 S, 0.02 P, and Fe-balance (all in wt % as measured by a Foundry-Master OE750 optical emission spectrometer (Hitachi Ltd., Tokyo, Japan)). The experimental samples were developed by selective laser melting using a ProX200 3D system in a nitrogen atmosphere without platform preheating. The printing was carried out in a stripe style. The width of the strip was 5 mm, the direction of the strip changed by 90° from layer to layer. The layer thickness for 3D printing was 30 μm, the laser power was 240 W, the beam speed was 1070 mm/s, and the distance between the tracks was 80 μm. Annealing of the obtained samples was carried out in the tempera-

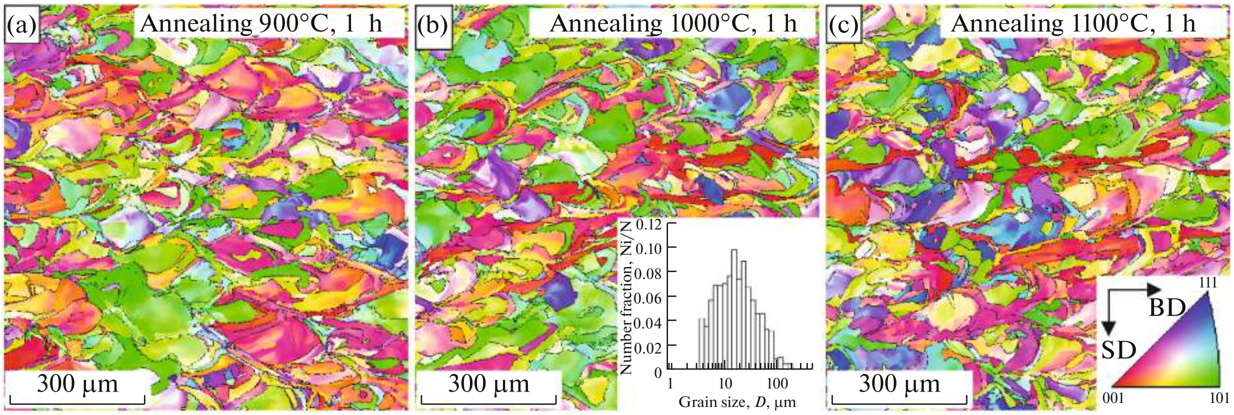

Fig. 1. Microstructures of 316L steel samples annealed at (a) 900°C, (b) 1000°C, and (c) 1100°C for 1 h.

ture range 900–1100°C for 1 to 10 h using conventional muffle furnace. Extent of the recovery was studied using the Vickers hardness test with a load of 3 N. The microstructure investigations were carried out using an FEI Quanta 600 scanning electron microscope (SEM). The samples for the SEM observations were electro-polished at a voltage of 21 V at room temperature using an electrolyte containing 10% perchloric acid and 90% acetic acid. The OIM images were collected with a step size of 1 μm. The OIM images were subjected to a clean-up procedure using the Grain Dilation method, setting the minimal grain size of 10 points.

RESULTS AND DISCUSSION

The OIM images of the microstructure of 316L steel samples subjected to annealing at temperatures of 900–1100°C for 1 h are shown in Fig. 1. The microstructure is represented by irregularly shaped grains resembling overlapping melt pools during 3D printing. Note the building direction (BD) is from left to right in Fig. 1. The grain size distribution in Fig. 1b is characterized by a peak in the region of 25 μm, which corresponds to the average grain size. The average grain size does not change with annealing temperature. The dislocation density is calculated as $\rho = 2KAM/(bl)$, where (b) and (l) are the Burgers vector and the scanning step size, respectively [3]. The dislocation density does not change remarkably with an increase in temperature from 900 to 1100°C during annealing for 1 h and remains at a high level of 8.7×10^{13} m^{-2}. This suggests that the recovery process in the steel is suppressed. Increasing the annealing time to 10 h at a temperature of 1000°C does not lead to significant microstructural changes (Fig. 2a). As a result of such treatment the high dislocation density is remained at a level of 8.7×10^{13} m^{-2}, and the average grain size is 25 ± 1 μm. It is worth noting that 316L steel obtained by conventional casting and then subjected to cold rolling with a reduction of 5% with subsequent annealing at temperatures of 700–1100°C [4] exhibited the rapid development primary recrystallization upon heating to 1000–1100°C. An increase in the annealing time usually leads to an increase in the size of the grains formed as a result of primary recrystallization [5]. However, the present 316L steel obtained by additive manufacturing exhibits signs of primary recrystallization after annealing for 10 h at temperature of 1100°C (Fig. 2b). The grain orientation spread (GOS) maps are also shown in Figs. 2a, 2b. Despite the rather long annealing time of 10 h and a high temperature of 1000°C, the large GOS values are clearly seen in Fig. 2a. The grain orientation spread is more than 2°–4°, which indicates a very low rate of static recovery, namely, subgrain coalescence. At the same time, annealing at a temperature of 1100°C (Fig. 2b) leads to almost complete softening. Almost all grains in the microstructure are characterized by GOS below 1°.

Figure 3a shows the dependence of microhardness on temperature for an annealing time of 1 h. It can be seen from the graph that there is a slight decrease in the values of microhardness, Hv, with an increase in the annealing temperature to 1100°C, despite the sluggish recovery as evidenced by microstructural studies. An increase in temperature to 1200°C leads to significant decrease in microhardness to 180 Hv. Based on the data obtained, two regions can be distinguished by dividing them by temperatures below 1100°C and above 1100°C. Annealing for 1 h at temperatures below 1100°C is accompanied with a sluggish static recovery. Correspondingly, the high dislocation density of 8.7×10^{13} m^{-2} remains in the obtained microstructures. On the contrary, at temperatures above 1100°C accelerated recovery occurs with subsequent recrystallization decreasing the hardness and the dislocation density to the level of 3.8×10^{13} m^{-2}. It should be noted that in a similar steel obtained by conventional metallurgy, accelerated recovery and subsequent recrystallization were observed at lower temperatures below 1000°C [4].

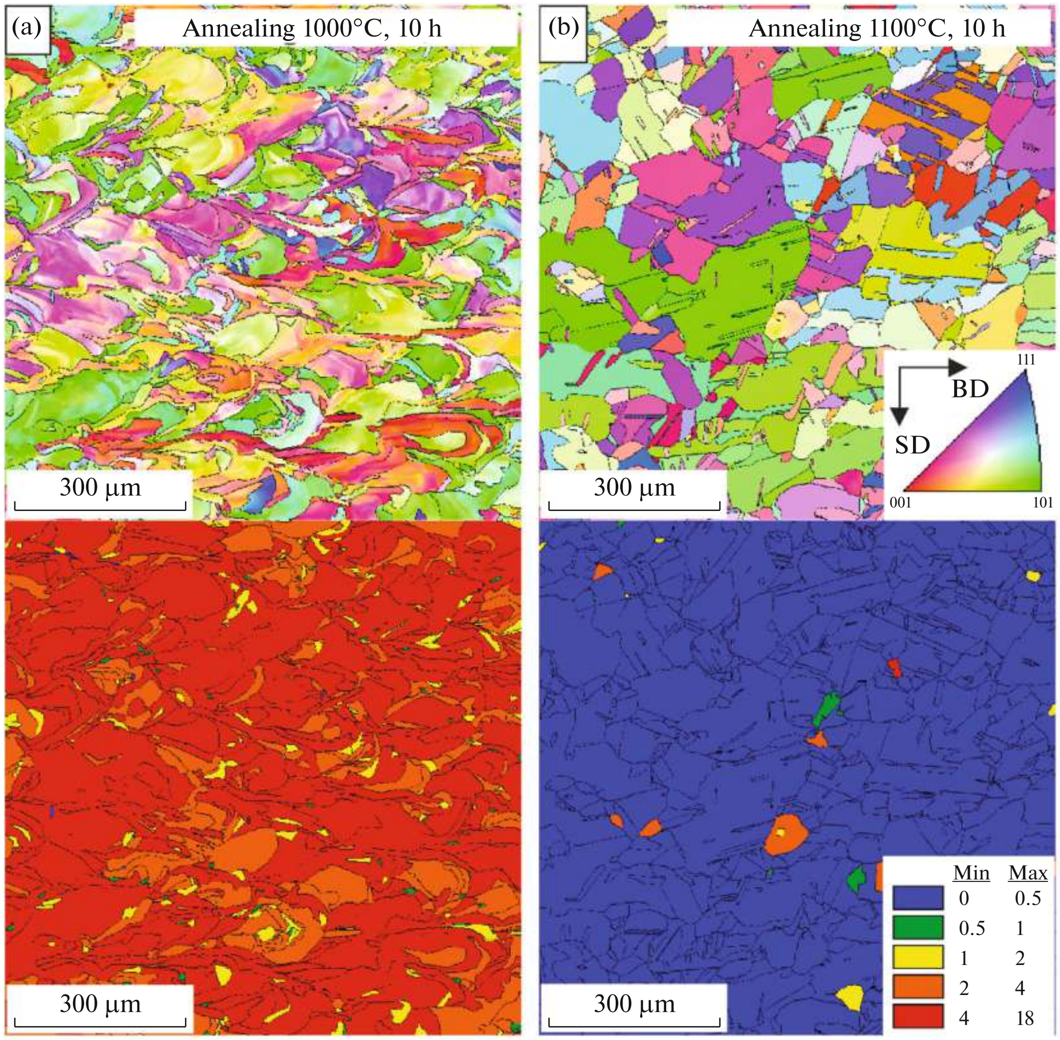

Fig. 2. Microstructures and maps of grain orientation spread (GOS) obtained as a result of annealing for 10 h at temperatures of (a) 1000°C and (b) 1100°C.

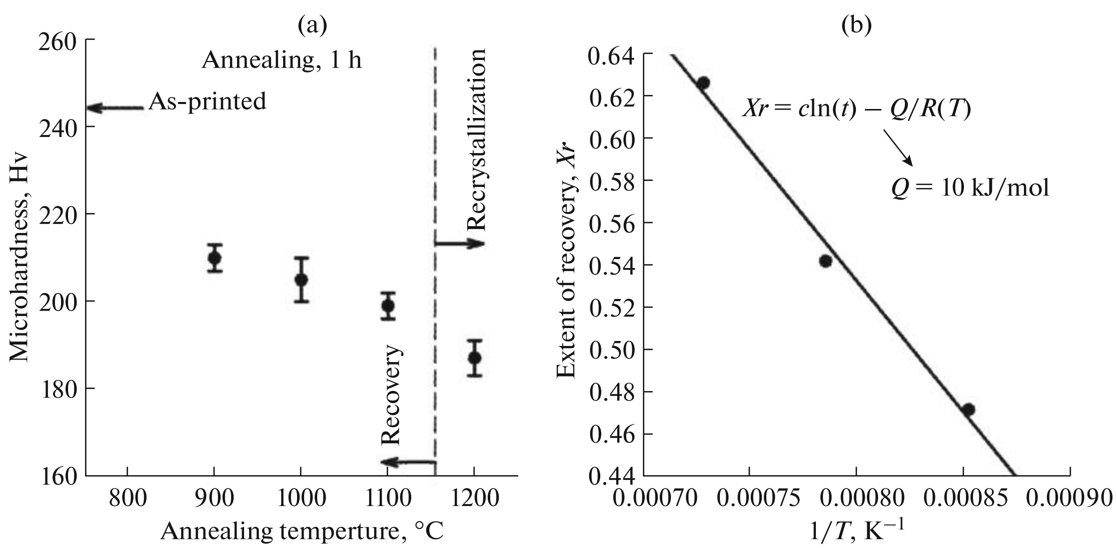

Fig. 3. Dependence of microhardness on temperature after 1 h annealing (a), and dependence of the recovery value on the reciprocal temperature (b).

The recovery rate Xr was calculated by the following formula [6] using the microhardness values of the annealed samples (Hv), the initial as-printed sample (Hv_d), and the fully recrystallized sample (Hv_0):

$$Xr = \frac{(Hv_d - Hv)}{(Hv_d - Hv_0)}. \quad (1)$$

From the graph (Fig. 3b) of the recovery dependence on the reciprocal temperature ($Xr = c\ln(t) - Q/(RT)$), the activation energy (Q) for recovery was determined as 10 kJ/mol. This very low value of activation energy suggests the suppression of recovery in austenitic stainless steel obtained by additive manufacturing.

CONCLUSIONS

The features of recovery during annealing of austenitic stainless steel 316L obtained as a result of additive manufacturing were studied. The main results can be summarized as follows. Two temperature regions with different governing softening mechanisms can be distinguished for 1 h annealing. Recovery was the main softening mechanisms operating during annealing at temperatures below 1100°C. Recovery annealing at temperatures of 900–1000°C and exposure for 1 h did not lead to significant microstructural changes. The dislocation density remained at a level of 8.7×10^{13} m^{-2}, and an average grain size was 25 ± 1 μm. Even increasing the exposure time to 10 h at 1000°C did not lead to significant recovery of the steel samples. The recovery process could be characterized by a very low activation energy of 10 kJ/mol. After annealing at temperature above 1100°C, primary recrystallization occurred leading to significant softening with a decrease in the dislocation density to 3.8×10^{13} m^{-2}. The development of primary recrystallization was also observed at temperature of 1100°C after annealing for 10 h.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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