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# Topography of the Grain Boundary on Stainless Steel Surfaces Varied by Nanosecond Laser Irradiation<sup>†</sup>

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## Abstract

*We designed a laser local heating system for controlling microstructures including the grain boundary observation system. Two kinds of laser, the CW laser and a short pulse laser, were employed in this system. The CW laser and the short pulse laser were a fiber laser and a nanosecond laser, respectively. The sample was a stainless steel (Type 304) plate. Grain boundaries on the stainless steel plate surface were examined by optical image measurement as the topography of the grain boundaries was changed after the nanosecond laser irradiation. Then, the grain could be selected for local heating with the fiber laser.*

**KEY WORDS:** (Nanosecond laser), (Grain boundary), (Visualization), (Microstructure)

## 1. Introduction

Conventional heat treatments utilizing homogeneous temperature fields in electric furnaces have been commonly performed in industry to change parameters, such as type of phase, shape and size of grains, grain orientation and grain boundary structure in all material areas. Local distributions of microstructure parameters may disappear or become passive and disabled to contribute to the evolution of microstructure such as texture development. For example, during the course of cubic texture development, some cube-oriented grains cannot grow if they are surrounded by preferentially growing grains with other orientations.

In our previous study, a fiber-laser local heating system was developed in order to modify the microstructure of pure aluminum by controlling recrystallization, grain growth process and texture development<sup>1-3)</sup>. The local heating resulted in preferential recrystallization and growth in a very small region less than 100  $\mu\text{m}$  in diameter. Orientation of the recrystallized grain can be controlled by selecting the target area precisely as well as the heating condition. The local heating resulted in grain size development even during the post homogeneous heating of the large grains fabricated by the spot heating. With the help from computer simulation of grain growth process for this kind of heterogeneous microstructure, a fiber laser local heating method would offer a novel technique for

designing microstructures having quite different textures that had not been fabricated by conventional production processes. Some of the results have already been reported. However, this laser focusing system has no function for selecting grain.

In this study, we proposed a method to visualize the grain shape with a short pulse laser. The melting point of grains is usually higher than that of the grain boundaries. During short pulse laser irradiation, the grain boundaries visualization might be caused by slight change of the surface due to the difference of the melting points.

When a short pulse laser is available for surface treatment of the metal to observe the grain's shape, the short pulse laser irradiation units will be installed in the laser local heating system. The process concept of the laser local heating system with the grain boundaries visualization unit is shown in **Fig. 1**. Before the surface treatment shown in Figs. 1(b) and (c), microstructures such as grain shape and crystal orientation of the grains were observed with Electron Back-Scatter Diffraction pattern (EBSD)<sup>4)</sup> equipment as shown in Fig. 1(a). After EBSD analysis, the specimen was fixed on the stage and the shape of grains with crystal orientations we would like to grow was found by short pulsed laser irradiation. Chemical etching method for observation of grains is also good method. However, in the method with short pulse laser shown in Fig. 1(b), the specimen can be heated by the fiber laser irradiation immediately after the short

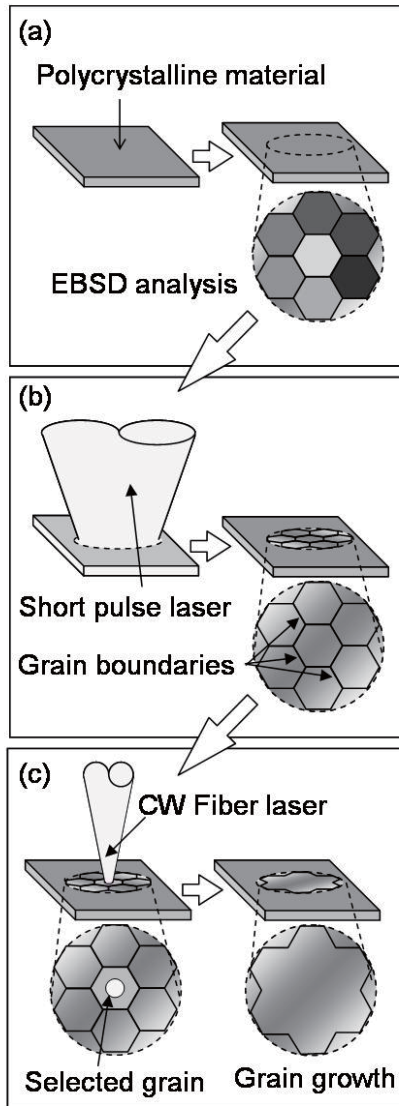
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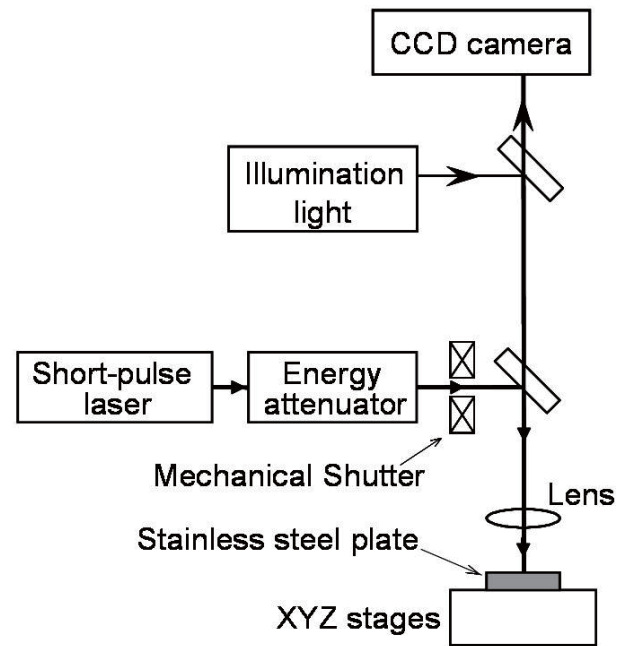


**Fig. 1** Specimen after EBSD analysis (a) and process concept of the grain boundaries visualization with short pulse laser (b) and CW fiber laser local heating system (c).

pulse laser irradiation as shown in Fig. 1(c). In the chemical etching method, it takes much more time than that for the short pulsed laser irradiation. We compared the area irradiated by the femtosecond laser irradiation with that by nanosecond laser irradiation in the experiments of the laser irradiation on the surface of stainless steel.

## 2. Experimental procedure

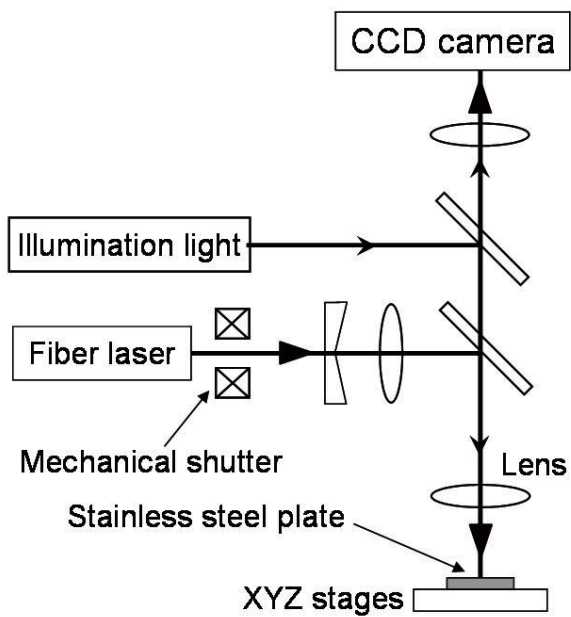
Schematic diagram of the experimental setup for visualizing grain boundaries with the short pulse laser is shown in Fig. 2. Specimens, commercially stainless steel plates of 1 mm in thickness, were prepared for the short pulse laser irradiation experiments as shown in Fig. 2. They were machined to square shape of 19 x 19 mm, with a final surface treatment by chemical polishing carried out prior to the laser irradiation. Two types of short pulse laser were used in these experiments, which were



**Fig. 2** Schematic diagram of experimental setup for visualizing grain boundaries with short pulse laser.

femtosecond and nanosecond lasers. The wavelength, pulse width and repetition rates of the femtosecond laser were 775 nm, 150 fs and 1 kHz, respectively. For nanosecond laser, they were 1064 nm, 6 ns and 10 Hz, respectively. The specimens were irradiated with the femtosecond laser at 100 to 500 mJ/cm<sup>2</sup> and with the nanosecond laser in the 200 to 800 mJ/cm<sup>2</sup> ranges. The laser power was controlled with an energy attenuator shown in Fig. 2. The number of pulses for the femtosecond laser and the nanosecond laser was in the range of 10 to 50. Each laser was focused on the specimen by a lens with focal length of 150 mm. Microstructure of the stainless steel plate was evaluated with a optical microscope, a scanning electron microscope (SEM), an atomic force microscope (AFM) and EBSD methods before and after the laser irradiations.

The schematic diagram of laser local heating system is shown in Fig. 3. A commercial Yb (ytterbium) fiber laser system (YLR-100-SM) was employed in our system. This system provides CW laser beam at a wavelength of 1076 nm and a maximum laser power of 100 W. Diameter of the laser beam was about 7 mm after the collimator lens. Laser power was measured by a power meter during laser irradiation. Laser power was varied by controlling the electric current in diode laser. The laser beam was focused on the stainless steel plate surface by a lens with a focal length of 30 mm. The stainless steel plate's position was controlled with an XYZ stage connected to a computer. A mechanical shutter was installed on the laser beam axis. Laser beam profile on the stainless steel plate surface was measured with a monitoring system shown in Fig. 3.

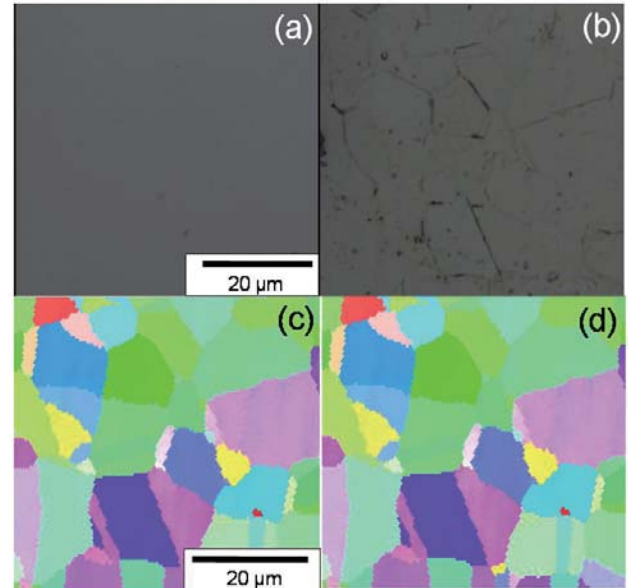


**Fig. 3** The schematic diagram of laser local heating system.

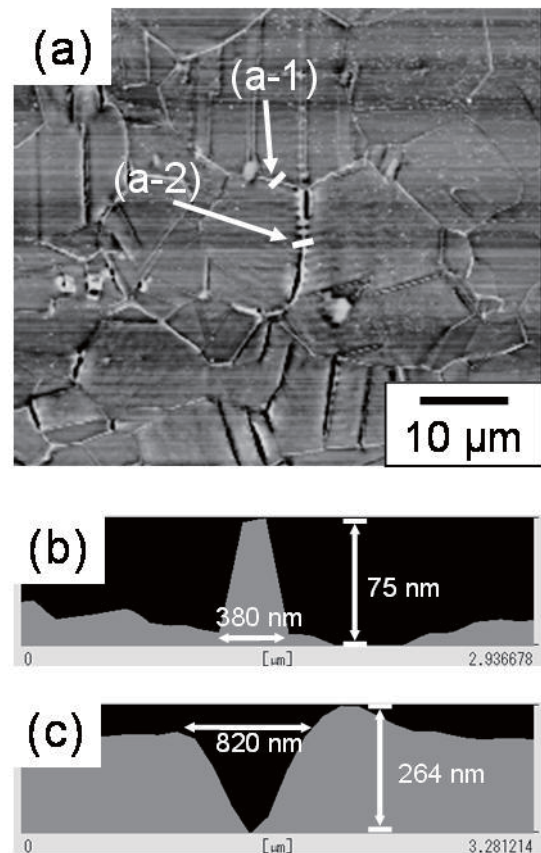
### 3. Results and discussion

For nanosecond laser irradiation, at 400 and 600  $\text{mJ}/\text{cm}^2$ , the surface morphology of the stainless steel was changed for 10 pulses in optical image measurements. These changes suggest that grain boundaries were visualized with variation of topography of the grain boundaries. At 200  $\text{mJ}/\text{cm}^2$ , the surface morphologies were changed for 50 pulses. The surface morphology change at 400 and 600  $\text{mJ}/\text{cm}^2$  for 10 pulses was clearer than that at 200  $\text{mJ}/\text{cm}^2$  for 50 pulses. For femtosecond laser irradiation in the 100 to 500  $\text{mJ}/\text{cm}^2$  ranges, grain boundaries were not observed in optical image measurements. As the SEM images show, topography of the grain boundaries were not varied on the stainless plate although periodic nanostructures with periods of about 600 nm were produced.

Optical images of the specimen surface before and after the nanosecond laser irradiation at 400  $\text{mJ}/\text{cm}^2$  for 10 pulses were shown in **Figs. 4(a)** and **(b)**, respectively. The EBSD patterns of the specimen surface shown in **Figs. 4(a)** and **(b)** are shown in **Figs. 4(c)** and **(d)**. As **Figs. 4(c)** and **(d)** show, the grains' size and shape were not altered by nanosecond laser irradiation. These results indicate that grain boundaries of the stainless steel could be visualized with nanosecond laser. **Figure 5(a)** shows AFM image of the stainless steel plate surface after the nanosecond laser irradiation at 600  $\text{mJ}/\text{cm}^2$  for 300 pulses. Cross sections at (a-1) and (a-2) in **Fig. 5(a)** are shown in **Figs 5(b)** and **(c)**, respectively. The results of this analysis indicate that heaving and caving at grain boundaries. The height of heaving and the depth of the caving at grain boundaries were 75 nm and 264 nm, respectively, as shown in **Figs. 5(b)** and **(c)**. Heaving and caving of the grain boundaries might be caused due to the difference of the melting point between the grain and the grain

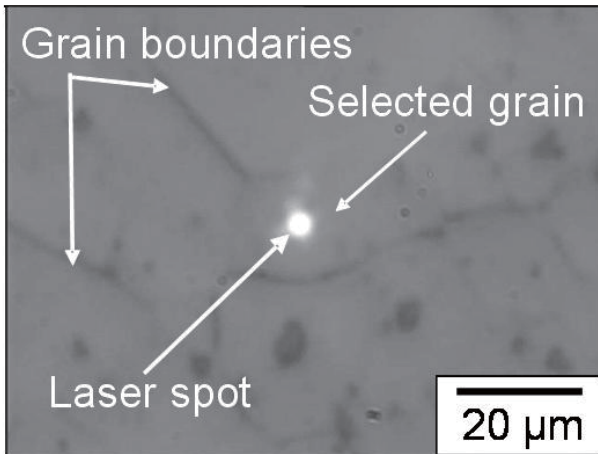


**Fig. 4** Optical image (a) and EBSD pattern (c) of the stainless steel plate surface before the nanosecond laser irradiation. Optical image (b) and EBSD pattern (d) of the stainless steel plate surface after the nanosecond laser irradiation at 400  $\text{mJ}/\text{cm}^2$  for 10 pulses.



**Fig. 5(a)** AFM image of the stainless steel plate surface after the nanosecond laser irradiation at 600  $\text{mJ}/\text{cm}^2$  for 300 pulses, (b) cross section at (a-1) in (a) and (c) cross section at (a-2) in (a).

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**Fig. 6** Optical image of the specimen surface monitored with CCD camera in Fig. 3. The selected grain could be illuminated with a CW fiber laser.

boundaries. The optical image of the specimen surface monitored with CCD camera in Fig. 3 was shown in **Fig. 6**. Figure 6 indicates that the selected grain could be illuminated with a CW fiber laser.

#### 4. Summary

We proposed the observation system of grain for laser focusing on selected grain with a short pulse laser.

The area created by femtosecond laser irradiation was compared with that by nanosecond laser irradiation in experiments of laser irradiation to the surface of stainless steel. After femtosecond laser irradiation, a grain boundary was not observed, however, after nanosecond laser irradiation, a grain boundary was observed. The EBSD pattern of the stainless steel after nanosecond laser irradiation was similar to that before the irradiation. This indicates the grain size and shape were not changed by nanosecond laser irradiation. Thus, a grain boundary visualization system could be obtained by the method with the nanosecond laser. AFM analysis indicates that heaving and caving were caused at the grain boundaries by nanosecond laser irradiation.

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