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Development of Three-dimensional Observation Method with Electrolytic Etching for Microstructure of Stainless Steel Manufactured by Additive Manufacturing

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Controlling the metallographic structure has been proposed as a new possibility of metal additive manufacturing technology[1)]. This technology is considered to lead to the development of functional materials such as materials having different Young's moduli depending on the directions and regions. However, in reality, modeling by additive manufacturing can be regarded as a process involving complicated repetitive heat treatment, and it is not easy to predict the structure to be formed and design an appropriate manufacturing process. Therefore, in order to search for an appropriate process, efficient structure observation of a plurality of materials manufactured having different manufacturing conditions is necessary. Further, it is difficult to grasp the three-dimensional structure of the structure and to quantify information such as the shape and the number of the structure by the conventional two-dimensional observation method. Therefore, efficient three-dimensional structure measurement technology is desired.

Stainless steel is one of the promising materials in metal additive manufacturing. Automated serial sectioning has been developed as a method for observing the three-dimensional structure of steel materials[2)]. In particular, the three-dimensional internal structure microscope[3)] that uses precision Numerical Control Shaper cutting and elliptical vibration cutting technology can control the removal amount more accurately than the polishing method, and has high quantitativeness of observation. However, this method uses chemical etching and cannot perform the electrolytic etching necessary for observing the structure of stainless steel. Therefore, in this research, in order to observe the three-dimensional structure of stainless steel formed by selective laser melting (SLM), we have developed a fully automatic observation method that incorporates an electrolytic etching mechanism in a three-dimensional internal structure microscope using precision cutting(figure1). Furthermore, using the developed technique, the metallographic structure of AISI 420 martensitic stainless steel (SS) produced with SLM was observed.

The details of observation by the developed method will be described. The sample was mirror-finished by shaper cutting using elliptical vibration cutting with a single crystal diamond tool. The tool edge width was 1 mm, the cutting depth was 1.5μm, the feed rate was 2.5 mm/s, the frequency of the elliptical vibration was 39.1 kHz, and the amplitude was 2μmp-p. Thereafter, electrolytic etching using a 10% oxalic acid aqueous solution was performed on the observation device. The current density was 1.5 mA / mm^2, and the etching time was 90s. Next, the sample was washed and dried. Thereafter, the cross section was photographed with a microscope. These series of operations were automatically performed by numerical control, and 200 continuous cross-sectional images were acquired.

A part of the image obtained by the observation was cut out and used, and a three-dimensional image of the metal structure of AISI 420 SS produced by SLM was constructed using image processing software (Fig.2. (b)). The actual field of view is $312 \times 312 \times 300\ \mu m^3$. The voxel size is $0.165 \times 0.165 \times 1.5\mu m$. As an example of quantification of microstructure information by three-dimensional observation, the radius of a fan-shaped melt pool by laser irradiation was calculated. In the SLM, since the scanning direction of the laser differs depending on the part (Fig.2. (c)), the resultant cross sections have different appearances depending on directions (Fig.2. (a), (d)). Therefore, two-dimensional observation from a single cross section is insufficient. From a cross section perpendicular to the laser scanning (Fig.2. (d)), the melt pool radius was measured from the number of pixels. The average of five melt pools was 88.2μm. Since the lamination pitch is 50μm, the uppermost powder layer and the below layer would be melted together by laser irradiation. Our three-dimensional observation method and subsequent quantification of the melt pool radius is useful for relating the degree of metal melting and the lamination conditions.

In conclusion, in this study, we have developed a new three-dimensional metallographic observation method that can observe the three-dimensional structure of stainless steel material fully automatically using elliptical vibration cutting and electrolytic etching. It performed three-dimensional observation of microstructure of SS manufactured by SLM. Melt pool radius was measured using our 3D data as an example. It showed that our method enabled to quantify 3D structure information, which was difficult in two-dimensional observation.

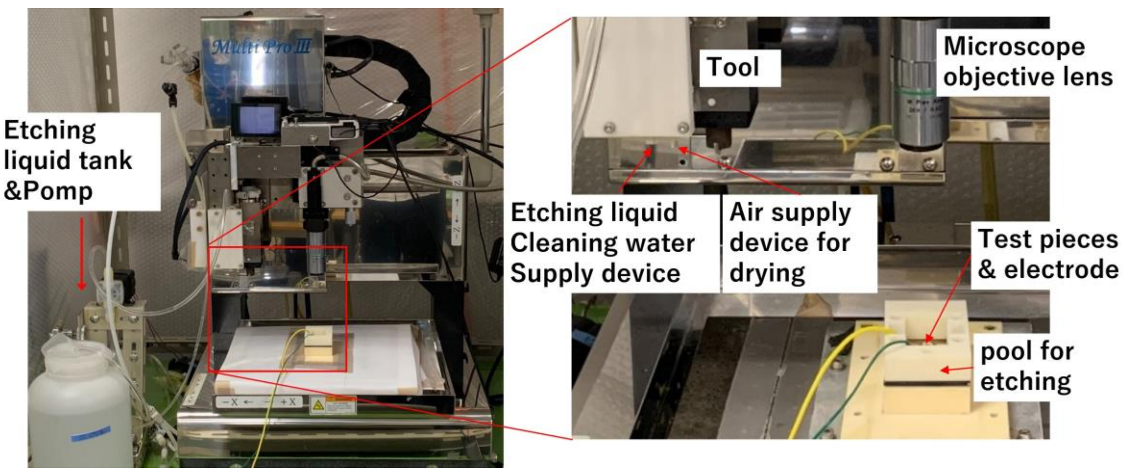

Figure 1. Three-dimensional internal structure microscope with electrolytic etching function

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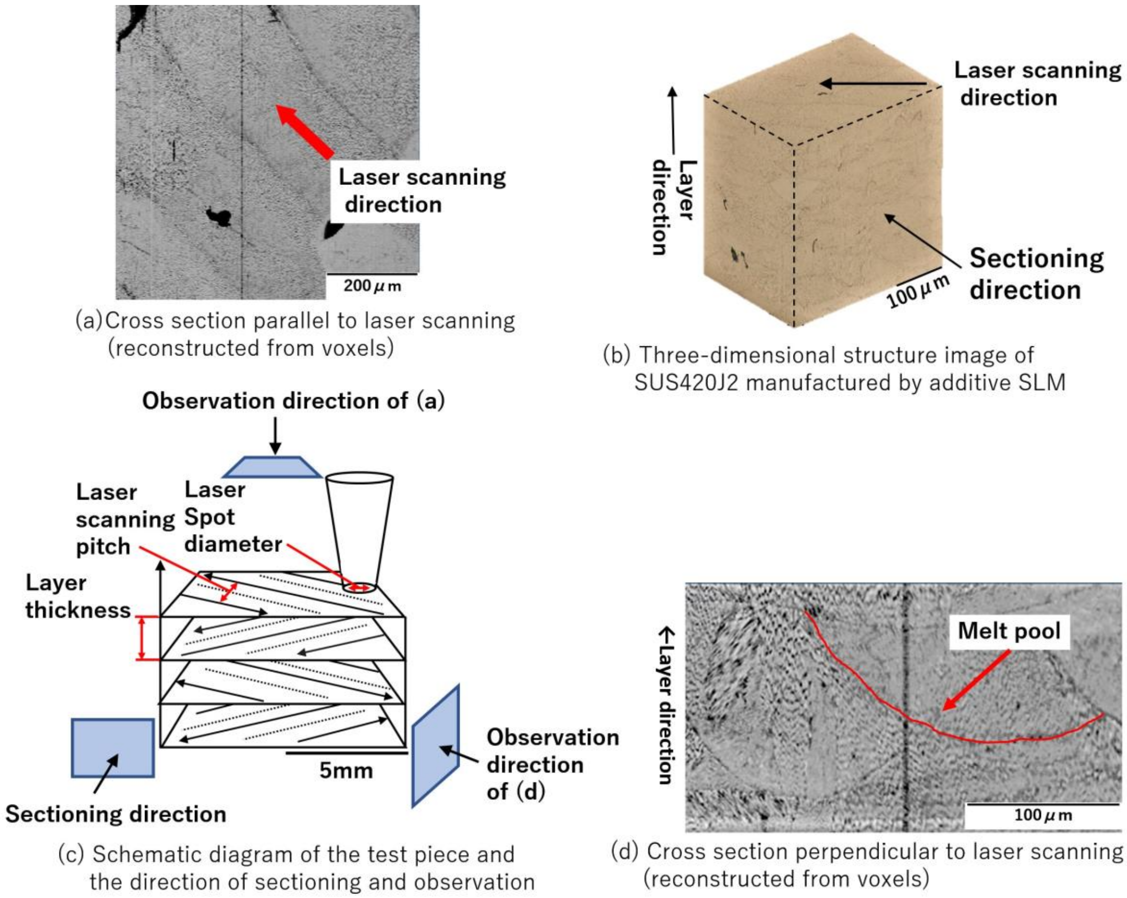

Figure 2. Three-dimensional observation of microstructure of AISI 420 martensitic stainless steel manufactured by selective laser melting

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