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Three-dimensional observation and morphological analysis of inclusions in a Ni-Co-based superalloy using the serial sectioning method

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Abstract

In this study, we observed the microstructural defects of inclusions and pores in a relatively large volume of a Ni-Co-based superalloy, TMW-4M3, including more than one hundred thousand inclusions using the serial sectioning method with precision cutting and optical microscopy, and analyzed their statistical morphology. First, we applied ultrasonic elliptical vibration cutting (UEVC) to the Ni-Co-based superalloy, a difficult-to-cut material, to obtain a mirror cross-section suitable for observation. Cutting conditions of a depth of cut of 1 µm, a cutting speed of 120 mm/min, and an elliptical vibration amplitude of 1.5 $\mu m_{p\text{-}p}$ resulted in the best surface roughness (Ra 10.3 nm). Next, we performed serial sectioning of the superalloy with 105 and 1000 cross-sections under these conditions. Here, image tiling was also applied to obtain large volumes of 12.2 × 11.7 × 0.105 mm and 12.2 × 11.7 × 1 mm. Finally, the inclusion regions were extracted and their number, volume, and equivalent diameter were calculated. Histograms of the 3D features of nearly 140,000 inclusions were obtained. This statistical morphological information will contribute to the safe design of high-temperature components with stringent safety requirements.

Keywords: Ni-Co-based superalloy, inclusion, Elliptical vibration cutting, Precision cutting

1. Introduction

Ni-Co-based superalloys designed based on a combination of a Ni-based superalloy and a Co-based alloy, both with γ-γ′ two-phase structure, are attractive materials for turbine disks in jet engines operating in high-temperature and high-pressure environments [1]. The evaluation of the material properties and reliabilities requires an assessment of the shape, distribution, and number of micron-sized microstructural defects in the inclusions and pores that act as crack initiation sites of fatigue failure. However, this is based on previous 2D observations, which is not always sufficient for statistical evaluation [2]. Therefore, it is necessary to perform highly efficient 3D observations over a wide area of millimeters with submicron-high resolution to obtain information on the spatial distribution, number of such objects, and morphology. One such method is serial sectioning, which removes thin layers and acquires a series of cross-sectional images [3]. Polishing methods are commonly used to remove thin layers. However, a highly efficient method using precision cutting has been proposed [3], which has the advantage of less processing time and controllability of the amount of removal. In this study, a large amount of inclusion information of Ni-Co-based superalloy was obtained using the serial sectioning method with precision mirror cutting and subsequent morphological analysis. Using the cutting condition from several cutting experiments, a volume from 105 slices of images were obtained. The inclusions were segmented from the images and histograms of the volume and equivalent diameter of the inclusions were created for statistical evaluation. Since further accumulation of data is required for material fatigue statistics, we demonstrated 1000 slices imaging, and also assessed the life of the cutting tools, which often limits the imaging volume.

2. Research methods

First, suitable cutting conditions were searched using ultrasonic elliptical vibration cutting (UEVC) [4] to create a mirror surface via precision cutting, considering those for Inconel 718 [5], which is likely to have similar machinability to that of a Ni-Co-based superalloy. Thin layer removal and continuous cross-sectional image acquisition via image tiling were performed, and 3D images containing numerous inclusions were reconstructed. We used MATLAB (R2022b, R2012a) for image tiling and statistical evaluation of inclusion morphology. The inclusion regions were segmented by binarization of the image and labeled according to their 3D connections, and their number, volume, and equivalent diameters were calculated. The filtered results were also calculated, excluding those with fewer than three voxels of connection in the Z-direction, such as chips, dust, or too small.

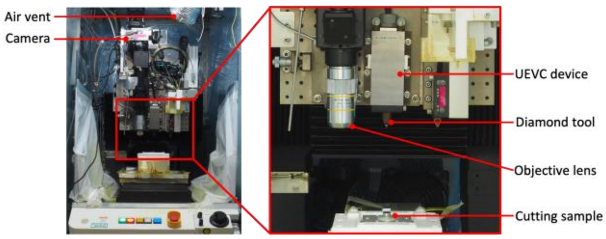

Figure 1. 3D internal structure microscope

3. Experimental device and Cutting sample

We used the UEVC device (EL-50Σ, Taga Electric Co., Ltd., Japan) to precisely cut the Ni-Co-based superalloy. The device was mounted on a 3D internal structure microscope (Riken Micro Slicer System: RMSS-005 [6], Japan) equipped with an epi-illumination imager as shown in Figure 1. An NC milling machine (MULTIPRO MP6 series, Takashima Sangyo Co., Ltd., Japan), a single-crystal diamond tool (UPC, A.L.M.T. Corp., Japan), and objective lens (M Plan Apo HR 10×, Mitutoyo Corp., Japan) were installed to enable fully automated cutting and observation. It uses planar cutting with a tool with a flat cutting edge of 1 mm, as shown in Figure 2. A single stroke can generate a mirror-like cross-section of the tool width. Shifting the cutting paths generates a wide cross-section. The sample material was TMW-4M3 [2], which was created at NIMS using the same casting and forging process as the turbine disk. This part was prepared as a cylindrical sample with a diameter and height of 10 mm.

4. Experimental results

4.1. Precision cutting condition search and surface roughness

The sample surfaces were polished beforehand. Precision cutting was performed under six conditions: cutting speeds of 30, 60, 90, 120, 150, and 180 mm/min at a tool width of 1 mm, depth of cut of 1 µm × 10 times, and elliptical vibration amplitude of 1.5 $\mu m_{p\text{-}p}$. A mirror surface with a roughness of approximately Ra of 10 nm was created under all conditions. The roughness values of the polished and cut surfaces at a cutting speed of 120 mm/min are shown in Figure 3. Their surface roughness values were Ra = 20.1 nm and 10.3 nm, respectively. The UEVC achieved sufficient surface roughness for observation, and we selected 120 mm/min for subsequent experiments.

4.2. Continuous 105 cross-section 3D observation

3D observation of 105 continuous cross-sections was performed using precision cutting. Each cross-section was obtained as 54 tiling images (9 in the x-direction and 6 in the y-direction) with a voxel pitch of 0.391 × 0.391 × 1 µm and an optical resolution of 0.6 µm at high resolution. The total observation time was approximately 10.5 h.

4.3. Inclusion morphology analysis using 3D data of 105 continuous cross-sections

The volume and equivalent diameter of each inclusion were calculated assuming that they were spherical. Figures 4 (a) and (b) show histograms of the volume and equivalent diameter of the inclusions in a double-logarithmic graph, respectively. The red bars are from all the inclusions, whereas the blue bars are from those filtered, which connect more than three continuous cross-sections. The filtering was to exclude the cutting chips and those that were too small. The number was approximately 140,000 prior to filtering. After filtering, those with more than 10 µm³ and the largest volumes were 8583 and 7027 µm³, respectively. Therefore, our method demonstrated a statistical evaluation of the inclusion size and percentage of their existence.

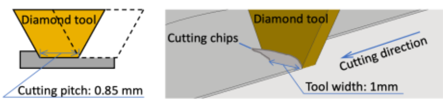

Figure 2. Creation of observation surface by precision cutting process

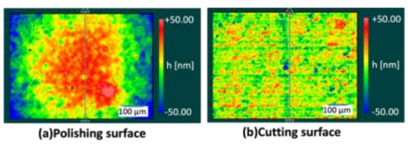

Figure 3. Surface roughness (120 mm/min)

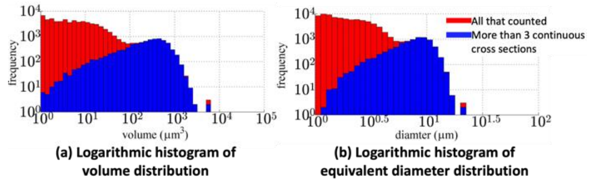

Figure 4. Histogram of volume and equivalent diameter of inclusions

4.4. Continuous 1000 cross-section 3D observation

A 3D observation of 1000 continuous cross-sections was performed using the same precision cutting and observation conditions. Approximately 100 h of automated observation was conducted to obtain a total of 31083 × 30025 × 1000 voxels. Figure 5 shows a 2D cross-sectional image (12.2 × 11.7 mm) and a 3D image of a part of the sample (2346 × 1564 × 1000 µm). The spatial distribution and various sizes of the numerous inclusions were confirmed in the volume. Future analyses of the data will provide a more precise statistical inspection of the inclusion morphology.

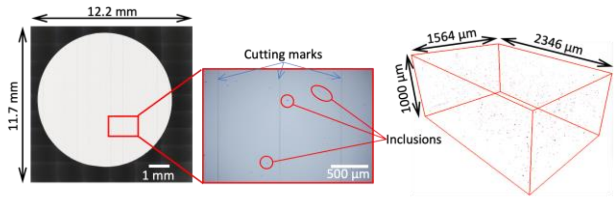

Figure 5. Cross-sectional image by precision cutting and 3D image

5. Conclusion

(1) We proposed a statistical evaluation method for 3D morphology of many inclusions in the Ni-Co-based superalloy by serial sectioning using UEVC and morphological analysis.
(2) We found a suitable UEVC speed for mirror surface generation of TMW-4M3 with a surface roughness Ra of 10.3 nm.
(3) 3D observation of TMW-4M3 with 105 and 1000 cross-sections were demonstrated to obtain large volumes, 12.2 × 11.7 × 0.105 and 12.2 × 11.7 × 1 mm, respectively, with a voxel pitch of 0.391 × 0.391 × 1 µm.
(4) Approximately 140,000 inclusions were evaluated from the 3D image with 105 sections, and those with more than 10 µm³ and the largest volumes were 8583 and 7027 µm³, respectively.

Our statistical inclusion evaluation method based on a large 3D imaging will contribute to advanced material development.

Acknowledgments

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