Fatigue Damage Evaluation of Casting Austenitic Stainless Steel Based on EBSD Method

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Abstract. To give a reference for the fatigue damage evaluation of casting austenitic stainless steel (CASS) in nuclear piping system, the early-stage fatigue damage of Z3CN20-09 was investigated and the misorientation information was acquired and analyzed based on electron back-scattered diffraction (EBSD) technique. Two cyclic loading states were selected and compared with the as-received specimen. The main EBSD testing parameter, step size, was optimized and two local misorientation indexes were extracted as a criterion. Results show that with the decrement of step size, the misorientation information was more meticulous but the testing time increased quite a lot. 10 μm step size was selected with the consideration of balance between the testing quality and the efficiency for the coarse austenite. With the increasing loading cycles, the surface became obviously uneven and tremendous persistent slip bands (PSBs) were observed in austenite. The mean and peak value of local misorientation presented a monotonous growth with the development of fatigue damage. It increased from 1.85° to 2.05° for the mean value and from 1.65° to 1.95° for the peak value. The results indicate that the EBSD analysis in grain scale is necessary and effective for the heterogeneous mechanical damage evaluation of coarse-grained steel. It might provide a support for the deep understanding of the fatigue damage mechanism and the development of new nondestructive evaluation technique.

1 Introduction

Z3CN20-09M is one type of casting austenitic stainless steels, the average grain size of which might be up to several mm. It has been widely used in the piping of nuclear power plant because of its excellent mechanical properties and corrosion resistance[1]. However, residual stress caused in the process of casting or mechanical processing may lead to stress corrosion cracking. The thermal mechanical stress would also accelerate the reduction of service life. Many studies have demonstrated that the austenite grains show evident elastic anisotropy[2]. Moreover, due to the centrifugally casting technology used for Z3CN20-09M, the plastic deformation is quite uneven and the corresponding damage evaluation becomes difficult. Ultrasonic testing is effective and nondestructive in the defect and damage evaluation. While for coarse-grained austenite steel, it would make a strong scattering and attenuation on the wave propagation path. Consequently, developing corresponding damage evaluation method is of great importance for CASS. It is knows that grain orientation occupies a very important role in fatigue damage. Electron backscattered diffraction (EBSD)[3,4] is an effective method, for not only the analysis of grain size, texture, phase identification, but also the measurement of the crystal orientation point to
point. With the improvement of testing accuracy, the analysis of local deformation has attracted more and more attention. The damage could be quantitatively evaluated in virtue of some indexes related to misorientation. Some studies have been reported on austenitic stainless steel. Kamaya et al.\(^5\) used the local misorientation \(M_L\) and its average value to evaluate the plastic damage of 316 stainless steel, which indicates that the average of \(M_L\), had a good correlation with the macro plastic strain. Since the EBSD acquisition parameters and post-processing parameters are complicated, any inappropriate parameter selection would bring a deviation or error to the damage evaluation data\(^6\). Chen et al.\(^7\) studied the influence of testing parameters, such as step size and probe current, and found that there existed a maximum indexed point peak as a function of step size when the scans include grain boundaries. However, previous studies are mainly focused in fine grains. When it turns into coarse-grained metallic materials, the results are quite limited.

In this study, the main testing parameter of EBSD, i.e. step size, was optimized for Z3CN20-09M steel and two \(M_L\) indexes were extracted. The fatigue damage under different loading stages were characterized in the grain scale and the damage indexes were compared, which would provide a basis for the future study of fatigue damage mechanism and development of new damage evaluation technique.

### 2 Experiment

The material was Z3CN20-09M steel, cast using centrifugal process according to French specification RCC-M 2000. The composition was determined by X-ray fluorescence and the alloying elements are shown in Table 1. Along the axial direction of pipeline, the specimen was cut by electrospark machining from the outer surface of the 70-mm-thick pipe, of which the microstructure was columnar grains about 10mm in length in the radial direction. The size and shape of the specimen are shown in Fig. 1. The thickness was 1.0 mm and the width of the parallel was 10.0 mm. So the microstructure of the parallel surface was the top view of the columnar grains in an equiaxed shape.

Standard electrolytic etching and vibration polishing were used before fatigue testing. The surface morphology of testing specimen under different stages was acquired by Olympus OLS-4000 confocal laser scanning microscope (CLSM). EBSD data was collected using Oxford HKL system on Zeiss Supra 55 scanning electron microscope (SEM). The testing range was shown in red region in Fig. 1. The post processing was performed on the HKL channel 5 software. The same area was determined after each fatigue loading by EBSD at a step size of 10.0 \(\mu\)m.

The fatigue test was carried out on a MTS hydraulic servo system under stress control at room temperature. The peak stress was 230 MPa and 250 MPa, respectively. The stress ratio \(R\) was 0.1 and the frequency was 2 Hz. The test was interrupted for the examination of the morphology and orientation when the surface of specimen generated certain damage.

| Table 1. Composition analysis of Z3CN20-09M (all in wt.%) |
|-----------------|-----|------|------|------|-----|-----|-----|-----|-----|-----|
| Element         | C   | S    | Si   | Mn   | P   | Cr  | Ni  | Mo  | Cu  | Co  | N   |
| Material        | 0.027| 0.014| 1.27 | 1.13 | 0.023| 20.19| 8.92| 0.21| 0.094| 0.04| 0.03|
| RCC-M           | \(\leq 0.04\) | \(\leq 0.015\) | \(\leq 1.50\) | \(\leq 1.5\) | \(\leq 0.03\) | 19–21 | 8–11 | --  | \(\leq 1.0\) | \(\leq 0.1\) | --  |
3 Results and Discussion

3.1 Optimization of Step Size

Step size, which refers to the interval between points in the processing of EBSD data acquisition, is a major testing parameter. Especially, the microstructure of Z3CN20-09M is a little complicated. According to the RCC-M 2000 specification, it commonly consists of about 12~20 vol.% ferrite phase and the other is mainly austenite. The two phases are nearly randomly distributed and the grain size is in the scale of mm for austenite while μm for ferrite (in the smallest dimension). So the description of this cross-scale microstructure under different fatigue damage states with the consideration of the balance between orientation details, scanning area and testing efficiency become the predominant issue of this study.

A typical area was selected and the step size optimization was conducted. The microstructure is shown in Fig. 2a. The long striped ferrite distributed on the austenite matrix, and the smallest dimension, i.e. in width, was only several μm, which was quite smaller compared with that of austenite grain. Four kinds of step size, 20 μm, 10 μm, 5 μm and 2 μm, were selected and the orientation information was acquired respectively. The results are also shown in Fig. 2. Different colors represent different crystal orientation and the adjacent pixels of the same or similar color forms grain structure. When the step size was 20μm, a relatively small ferrite phase area could not be resolved and correctly indexed, shown in Fig. 2b. The morphology of austenite grain can neither be identified. When the step size decreased to 10 μm, the shape of austenite phase can be distinguished and the ferrite phase (as indicated by arrows in Fig. 2) also became clear gradually, although the rim became jagged. In the condition of 2 μm, the quality of acquired images was significantly improved. The ferrite grain could be clearly seen, as well as the boundaries of austenite grains.

![Fig. 1. Geometry of fatigue test specimen (in mm).](image)

![Fig. 2. Orientation distribution maps in different step sizes: (a) SEM microstructure; (b) 20 μm; (c) 10 μm; (d) 5 μm; (e) 2 μm.](image)
To give a comparison on the above results, $M_L$ index was incorporated. The relative frequency distribution and peak and mean value were extracted from the $M_L$ maps under different step size and shown in Fig. 3. In the frequency distribution diagram of Fig. 3b, it is shown that the peak value as well as the mean value increased as the step size increased. When $20 \mu m$ was chosen, the relative frequency distribution presented a great dispersion, and the $M_L$ corresponding to the peak relative frequency ranged from $1.5^\circ$ to $3^\circ$. When it turned to smaller step size, the relative frequency distribution tended to form a normal distribution and the fluctuation was obviously weakened. For the $2 \mu m$ condition, the $M_L$ was mainly in the range from $0.5^\circ$ to $1.5^\circ$, and the relative frequency distribution showed a logarithmic distribution. Some studies have also demonstrated\cite{8} that the basic configuration of $M_L$ distribution inclined to form a logarithmic distribution, even after fatigue loading. When the sampling interval was small, the $M_L$ only reflected the damage information in a smaller area than that under a large step size. So it is expected that the calculated value was small. Besides, the sampling quantity was good enough to ensure the frequency distribution curve smoothing. While for the larger step size, the sampling quantity would worsen.

Because the microstructure of Z3CN20-09M is cross-scale, to ensure the finest acquisition of the orientation information, the dimension of the ferrite grains should be firstly considered. Generally, the ratio of step size to grain size could be set as $1/5$ to $1/10$. So from Fig. 2a, a step size of $2 \mu m$ or even $1 \mu m$ is preferred. For the red region with an area about $10 \text{ mm}^2$ in Fig. 1, the scanning time would be higher than 200 h. It is unacceptable from the viewpoint of engineering testing. One good hint is that the size of ferrite compared with the ultrasonic wave length used in field nondestructive testing was small, so the scattering effect was commonly ignored and only the austenite was considered\cite{9,10}. Consequently, the step size of $10 \mu m$ was selected here and the scanning time for one damaged state was about $2.5 h$.

**Fig. 3.** Relative frequency distribution diagram and peak and mean value columnar map in different step size.

### 3.2 Damage Parameter Analysis

To compare and analyze the evolution of fatigue damage and the evaluation parameters, three damage stages was designed based on the preliminary observation of the specimen surface. The as-received state was regarded as the stage 1 and that after fatigue loading to 300 cycles at maximum stress of 230 MPa was stage 2. Upon this basis, another 4000 cycles was loaded under the peak stress of 250 MPa and the stage 3 was obtained.

**3.2.1 Observation of Surface Morphology**

The sample surface was observed by LSCM and the stage 1 and 3 were shown in Fig. 4. In Fig. 4a, the as-received specimen surface was flat and smooth. The ferrite grains presented a uniform distribution on the austenite matrix. It was mainly in the form of striped shape. The
local area indicated in Fig. 4a was magnified in Fig. 4b. The phase boundaries were very clear. After fatigue loading of stage 2 and 3, the damage turned to be obvious (Fig. 4c). Some regions were dark, which indicated that the surface were quite rough. Tremendous PSBs appeared mainly in austenite grains and some were near the boundary of two phases.

![Image](image_url)

**Fig. 4.** Surface morphology comparison: (a) as-received specimen; (b) local magnification of a; (c) after 4000 cycles at 250MPa; (d) local magnification of c.

### 3.2.2 $M_L$ analysis

$M_L$ is a mean value of misorientation between a reference point and its surrounding 8 points [8]. A line was drawn between two adjacent points when the misorientation was larger than 5°. If a series of these lines formed a closed region, the lines were defined as a grain boundary, and the misorientation between the points of different grains was not included in the calculation of $M_L$. Researches [11, 12] show that $M_L$ had a good correspondence with the density of geometrically necessary dislocations, and it reflected the degree of plastic strain quantitatively. So using $M_L$ to evaluate the damage behavior is effective.

Fig. 5 is the inverse pole figure of specimen surface obtained by post-processing. The austenite grain size could be clearly recognized and the average grain size approached to 1 mm. Besides, most grains occurred at more positive potentials for the <101> orientation except a few grains tended toward <001>. The corresponding $M_L$ maps at three different stages are shown in Fig. 6 and a color code was used to represent misorientation angle value. To give a quantitative comparison, the mean and peak value distribution were extracted in Fig. 7. It was found that the color of $M_L$ maps tended to develop from blue to red with the cyclic loading. It is more evident in Fig. 6c. No matter the mean or the peak value of $M_L$, they both presented a monotonous growth with the fatigue loading. It increased from 1.85° to 2.05° for the mean value and from 1.65° to 1.95° for the other. Moreover, the frequency distribution curve continuously followed a good normal distribution, which is different from that in Fig. 3a. The main reason is the increment of the amount of testing points. During the realistic fatigue damage procedure, the evolution of dislocations and the orientation information would be not quite perfect as indicated in Fig. 7, but the overall trend would be similar. More meticulous work is now continued and it is hopeful to provide a new insight for the quantitative assessment of the fatigue damage based on ultrasonic technique.

![Image](image_url)

**Fig. 5.** Inverse pole figure of EBSD acquisition area.
4 Conclusion

The early-stage fatigue damage of Z3CN20-09 was investigated and the misorientation information was acquired and analyzed based on EBSD technique. The testing parameter step size was optimized and the $M_L$ indexes were extracted. The results obtained are summarized as follows:

1. With the decrement of step size, the misorientation information was more meticulous but the testing time increased quite a lot. For the coarse austenite grain with a diameter about 1 mm, 10 μm step size was selected to achieve a balance between the testing quality and the efficiency.

2. The morphology analysis indicated that with the increasing loading cycles, the surface of the specimen became obviously uneven and tremendous PSBs were observed mainly in austenite grains and at the boundary between ferrite and austenite phase.

3. The mean and peak value of $M_L$ presented a monotonous growth with the development of fatigue damage. It increased from 1.85° to 2.05° for the mean value and from 1.65° to 1.95° for the other one, which are directly related to the density of geometrically necessary dislocations. The EBSD analysis in grain scale is effective for the heterogeneous mechanical damage of coarse-grained metallic materials and might provide a new insight for the development of new non-destructive evaluation technique.
Acknowledgment

This work has been supported by the National Natural Science Foundation of China (Grant No. 51405061) and the National Basic Research Program of China (Grant No. 2015CB057306).

References