Formation of fine granular area in a non-defect matrix of austenitic stainless steel during very high cycle fatigue

Guocai Chai1,2 | Jens Bergström3 | Christer Burman3

Abstract
A fine granular area, FGA, is a typical phenomenon observed at the very high cycle fatigue fracture crack origin with a subsurface defect in the material. The FGA has been widely investigated, and different mechanisms have been proposed. In this paper, the formation of FGA in a non-defect matrix of one austenitic steel during very high cycle fatigue was studied using a progressive stepwise load-increasing method and electron scanning microscopy/electron channeling contrast imaging (ECCI) technique. A nano rough surface area or FGA at the fatigue crack origin has been observed in the subsurface matrix without any defect. It is a new phenomenon. A mechanism was proposed using the dislocation plasticity theory. The formation of FGA in a non-defect matrix is a localized plasticity exhausting process by strain localization, grain fragmentation, stress concentration and nano crack initiation and propagation along low-angle grain boundaries.

KEYWORDS
austenitic stainless steel, dislocation, FGA, grain boundary, VHCF

Highlights
• Novel approaches were developed to study the formation of FGA in the matrix in the VHCF regime.
• FGA formed in a subsurface matrix without defect is a new observation—a material nature.
• Strain localization, dislocation sub-cell and grain fragmentation lead to nanostructure.
• Formation of FGA is due to crack initiation and propagation at low-angle grain boundary.
1 | INTRODUCTION

Very high cycle fatigue (VHCF) of metallic materials has been classified as a type of fatigue with a fatigue life beyond $10^7$ loading cycles. Since the earlier works, it has become an important topic since structural integrity design and analysis of many engineering structures and components are related to fatigue in the VHCF regime in recent decades. Great efforts have been paid to study the VHCF behavior of different metallic materials since the research of VHCF is a challenge to the traditional fatigue concept on both modes of fatigue crack initiation and fracture features. Fatigue crack initiation in metals can shift from surface crack origin to subsurface crack origin with decreasing applied stress or increasing fatigue life from HCF to VHCF regime. Subsurface fatigue crack initiation in the VHCF regime starts mainly at subsurface defects such as inclusions, pores, and microstructure inhomogeneities, but can also start in some phase or matrix that is not associated with pre-existing defects (subsurface non-defect fatigue crack origin (SNDFCO)).

Fatigue crack initiation at pre-existing defects has been widely investigated and reviewed. For high-strength steels, fatigue crack initiation is characterized by the formation of a subsurface “fish eye,” with a relatively rough surface area in the vicinity around the defect. High-resolution scanning electron microscope (SEM) or transmission electron microscope (TEM) investigations have shown that this rough area has a fine granular microstructure and is therefore called a fine granular area (FGA).

Several mechanisms have been proposed to explain this phenomenon. Sakai et al. have proposed that the formation of FGA includes the following three stages: formation of a fine granular layer, nucleation and coalescence of the micro-debonding region, and finally a completed formation of an FGA. The occurrence of the dark, rough area was described as a polygonization process by the diffusion of chemical particulates during cracking. However, the paper did not provide explanations of how these three stages occurred. Hong et al. have proposed numerous cyclic pressing, NCP, a model to explain the formation of FGA. The repeated cyclic loading will cause the cumulation of plastic deformation in the matrix near the inclusion, which leads to fatigue crack initiation. With further cyclic loading, the microstructure of the originated crack surfaces will be fragmented due to the crack closure and residual stress release. The original coarse grains will become nanograins due to localized intensive plastic deformation, and microstructure studies have been done to verify this model. Grad et al. have proposed a model based on local plasticity in a highly stressed volume at a defect or crack tip. During cyclic loading, stress concentration around inclusion can lead to dislocation initiation and motion and a local plastic zone, which causes the formation of dislocation cells and, consequently, new finer-grain boundaries similar to the polygonization process proposed by Sakai et al. In these models, it was proposed that small grains in metals can lead to a reduction of the threshold value of the stress intensity factor ($K_{th}$). The local grain refinement leads to a sufficient decrease in the local threshold value, and a crack can initiate and propagate in the fine-grained volume. Another similar model is the fragmentation of martensitic laths and the formation of dislocation cells. FGA phenomena observed and studied so far are mainly correlated to inclusion/defect initiations. For VHCF origin in the matrix, there may be some weak phase, grain boundary (GB), or others not associated with pre-existing defects. However, the mechanisms for the formation of these non-defect fatigue crack origins are still not clear.

As known, the primary cause of fatigue damage is usually intimately related to some form of cyclic slip irreversibility. Under cyclic loading, it is now well understood that in materials having no inclusions, the crack initiates from the surface (stage I), followed by crack propagation (short and long crack, stage II). In the VHCF range, surface damage becomes more or less negligibly small with a low-stress amplitude applied. Then, cracks that initiate at internal heterogeneities by stress localization or concentration can grow slowly. At the current stage, both crack initiation and growth during VHCF are still not verified physically-metallurgically. However, it was reported that cyclic strain localization has occurred as a consequence of a very high number of loading cycles in spite of the fact that the loading amplitude was below the traditional persistent slip band (PSB) threshold. The high strain or stress localization can then cause fatigue damage and crack initiation.

There are different experimental techniques developed recently to study fatigue damage and crack initiation mechanisms during the VHCF process. A fracture surface topographic analysis (FRASTA) method has been used to study crack initiation and propagation behavior. Analytical approaches based on localized stress concentration and simulation have been used to study the large scatter of testing results. Three-point bending and stepwise load block have been used to study fatigue damage. In situ damage assessment or crack initiation study using either nonlinear ultrasonic measurement or synchrotron X-ray diffraction are very interesting methods to study VHCF behavior.

The fatigue life of a material is commonly characterized by an S-N curve or Wöhler curve using several samples at each load level and evaluated by statistical methods. As a rapid and alternative method, the progressive stepwise load increasing test (PSLIT) to evaluate...
fatigue endurance with a single sample has been proposed. The cyclic loading started from a low-stress level till a certain number of cycles. Then load/stress was linearly increased until the sample failed. This method has been utilized for different materials and has shown the ability to provide reasonable material endurance limits. Recently, this method was used to study fatigue and damage behavior in steels in the high cycle or VHCF regions. In this study, the number of cycles for each load step is higher than $10^7$ cycles, mostly higher than $10^8$ cycles or within the VHCF regime. The results show that this method can predict the fatigue damage process, especially the damage rate in individual samples.

In the present paper, the VHCF behavior in austenitic stainless steel has been studied with the purpose of making a fundamental study on the formation of FGA in a metal matrix without defects. PSLIT is used for fatigue testing. The main assumption is that material fatigue is a local material damage process. To avoid fatigue crack initiation at the sample surface or a subsurface inclusion, high strain or stress localization in the matrix is a critical condition that can lead to fatigue damage and crack initiation. Multiple crystalline materials is naturally heterogeneous due to different crystal orientations. Some crystals/grains with low Taylor factor can damage first during cyclic loading. If this damage or dislocation slipping is so small, then it may not lead to the formation of PSB, instead the formation of dislocation sub-cell or grain fragmentation, and finally the formation of crack due to plasticity exhaustion. This can be obtained by localized fatigue damage in “soft grains” in the matrix with PSLIT. The micro- or nanoscopic fatigue damage and crack initiation behavior are investigated using the electron channeling contrast imaging (ECCI) technique. A combination of dislocation plasticity theory and fracture mechanics has been applied, which may provide a better understanding of the formation of FGA.

2 | MATERIAL AND EXPERIMENTAL

In this investigation, one AISI 316-type austenitic stainless steel grade with high purity was used. The samples were taken in a bar material with a diameter of 15 mm, which was solution-annealed at 1050°C for 20 min and then quenched in water. Table 1 shows its chemical composition and mechanical properties. Figure 1A shows the microstructures of the material in the transversal section. The grain sizes are rather equiaxial, with a size of about 43 μm and with plenty of annealing twins.

VHCF testing was performed using an ultrasonic fatigue testing machine with a displacement-controlled mode and a frequency of 20 kHz. An hourglass geometry sample with a waist diameter of 3 mm was used (Figure 1B). A stress ratio of $R = 0.1$ was applied. In order to avoid excessive heating of the samples, a pressurized air-cooling was applied. To study the damage behavior, a progressive stepwise load increasing test (PSLIT) was used. Each load step was 10 MPa maintained about or more than $10^8$ cycles, and then stepwise increased until the sample failed. In order to study the step length effect, the number of cycles at each step was varied from $10^8$ to $10^9$ cycles. Eleven samples were used. Table 2 shows the testing details of all samples with a progressive stepwise load. The last number of cycles was the cycles to failure at that stress level.

Two types of surfaces were used to study the fracture and damage behavior using an SEM. The first is the

| TABLE 1 Chemical composition (wt%) and mechanical properties of the alloy used. |
|---|---|---|---|---|---|---|---|---|
| C | Si | Mn | Cr | Ni | N | Mo | Fe | $\sigma_{UT}$ [MPa] | $\sigma_{UT}$ [MPa] | A [%] |
| 0.018 | 0.48 | 1.66 | 17.4 | 14.2 | 0.08 | 2.74 | Bal. | 254 | 605 | 63.4 |

FIGURE 1 (A) Microstructure of the alloy used. (B) Sample geometry used for very high cycle fatigue (VHCF) testing.
The following is a brief description of the testing details with a progressive stepwise load. Figure 2A shows the correlations between the applied CHAI ET AL. 2367 TABLE 2

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3.1 RESULTS AND DISCUSSION

The applied stress and accumulated number of cycles to failure, according to Table 2, are shown in Figure 2. All samples started with the same stress level of 120 MPa and 108 cycles. In this paper, fatigue strength is defined as fatigue stress that has passed 108 cycles. Figure 2A shows that samples No 9 and No 10 have the longest total fatigue lives at the last step or failure stress with the total accumulated number of cycles. The applied stress and accumulated number of cycles to failure, according to Table 2, are shown in Figure 2. All samples started with the same stress level of 120 MPa and 108 cycles. In this paper, fatigue strength is defined as fatigue stress that has passed 108 cycles. Figure 2A shows that samples No 9 and No 10 have the longest total fatigue lives at the last step or failure stress with the total accumulated number of cycles. The applied stress and accumulated number of cycles to failure, according to Table 2, are shown in Figure 2. All samples started with the same stress level of 120 MPa and 108 cycles. In this paper, fatigue strength is defined as fatigue stress that has passed 108 cycles. Figure 2A shows that samples No 9 and No 10 have the longest total fatigue lives at the last step or failure stress with the total accumulated number of cycles. The applied stress and accumulated number of cycles to failure, according to Table 2, are shown in Figure 2. All samples started with the same stress level of 120 MPa and 108 cycles. In this paper, fatigue strength is defined as fatigue stress that has passed 108 cycles. Figure 2A shows that samples No 9 and No 10 have the longest total fatigue lives at the last step or failure stress with the total accumulated number of cycles.
above. However, there is no correlation between the last applied stress and the number of fatigue life.

The above results indicate that each sample with a different number of cycles at each step can have different fatigue behavior. This may be related to the fatigue damage process during cyclic deformation at each step. The fatigue damage, $D$, under variable applied stress amplitude can be evaluated by Palmgren–Miner’s rule with the following expression:\(^{(42)}\):

$$D = \frac{n_1}{N_1} + \frac{n_2}{N_2} + \ldots + \frac{n_k}{N_k} = \sum_{i=1}^{k} \frac{n_i}{N_i},$$

(1)

where $n_i$ is the number of cycles accumulated at stress $\sigma_i$, and $N_i$ is the number of cycles to failure at the $i_{th}$ stress. When the damage $D$ reaches a critical limit, for example, at $D = 1$, failure will occur.

In this paper, energy consumption, $\Delta U$, is used to describe the fatigue damage process since the total strain energy stored in a system will be consumed by each cyclic deformation. The fatigue damage should therefore be correlated to the strain energy consumption during cyclic loading as follows:\(^{(43)}\):

$$U = \frac{1}{2} \frac{V}{E} \sigma^2$$

and thus, for cyclic loading $\Delta U_i = \frac{1}{2} \frac{V}{E} \sigma_i^2$,

(2)

where $U$ is the elastic deformation energy, $\Delta U_i$ is the energy consumed at the $i_{th}$ load step with a maximum cyclic stress $\sigma_i$ and a number of cycles $N_i$, $E$ is Young’s modulus, and $V$ is the volume. The damage fraction or damage rate ($D_f$) of each load step is defined as follows:

$$D_f = \frac{\Delta U_i}{\sum \Delta U_i} = \frac{N_i \sigma_i^2}{\sum N_i \sigma_i^2}.$$  

(3)

Figure 3A shows the correlation of damage fraction, $\Delta U_i/\sum \Delta U_i$, according to Equation (3) in each sample during each step versus the accumulated number of cycles. This diagram is actually related to the damage rate of each sample during the fatigue process. They show different damage evolutions. Sample No 9 shows the lowest damage levels, and samples No 4, 6, and 8 show the highest damages. These damage evolutions in Figure 3A can be strongly correlated to the total fatigue life, as shown in Figure 2B, and the fatigue strength in Table 2. They have almost the same ranks. As expected, lower damage in the sample can lead to a longer fatigue life. All curves show a similar slope, it is therefore believed that the damage mechanism in these samples during the fatigue process is similar, but the damage evolution is different, probably due to different numbers of cycles at each step that may cause different cyclic deformation responses either to damage or strengthening. The previous discussion is also confirmed by Figure 3B, which shows that the limiting failure strain energy is increasing with the number of cycles in a consistent manner. The results may also give some explanation why large scatters appear in the different samples. This method can be used not only to study the damage and damage rate but also to qualify the material behavior during cyclic loading.

3.2 Fatigue crack initiation and propagation

The fracture study shows that besides samples No 4, 6, and 8 with surface defect origins, all other samples have SNDCF with a diameter of about 15–40 $\mu$m near the surface. Figure 4 shows some examples of subsurface fatigue crack initiations in the non-defect matrix. Figure 4A,B shows the fatigue crack initiation in sample No 9 after a total fatigue life of $1.15 \times 10^{10}$ cycles. A fatigue crack initiation origin with an FGA near the sample surface has been observed. Different from earlier studies,\(^{7,8,15–21}\) the fatigue crack initiation origin with an
FGA observed here occurred in the matrix without any defect. This means that the fatigue damage and consequently the formation of fatigue crack initiation origin can mainly be related to the material behavior. Hence, it is of great interest to study the fundamental nature of fatigue damage in the material. This is also the first report on the formation of fatigue crack initiation origin with an FGA in this type of material. So far, we do not know where/when crack initiation or small crack propagation occurs, which will become a further investigation. The FGA in sample No 9 is similar to those observed in the subsurface inclusion crack origin. However, this type of FGA is usually near the sample surface, as shown in Figure 4C.

In Figure 5A, a facet fracture near the FGA boundary can be observed. After a close study, very fine striations can be observed just after the FGA boundary, Figure 5B. This indicates that the crack propagation after the formation of FGA is a stage II process with multi-dislocation slip and a very low crack propagation rate. The formation of FGA is a stage I process according to the classical theory. The crack propagation afterwards is similar to those of ductile materials by the formation of striations during further cyclic loading. Figure 5C shows striations formed at about 65 μm from the FGA boundary. The width of the striation is about 0.063 μm, and it still corresponds to a comparatively low crack propagation rate of 6.3 × 10^{-8} m/cycles.

3.3 | Formation of FGA in non-defect matrix

As known, fatigue damage in metallic material is always correlated to plastic deformation. The FGA formations previously studied are mainly related to the materials with either inclusion, multi-phase, or structure inhomogeneity. This will cause localized stress concentration at these defects and material damage during cyclic loading even with stress lower than the yield strength of the material and leads to the formation of FGA. For a single-phase material like the austenitic stainless steel with a homogeneous structure; however, it is difficult to use the earlier theory or models to give an explanation on the formation of FGA in the material matrix without a defect.

In order to study the cyclic damage and the formation of the fine-grained structure in this homogeneous single-
phase material during the VHCF process, the microstructures of the fatigue-tested samples No 5 and No 9 from the fractured part to about 6 mm inside the polished longitudinal section samples have been studied progressively. Since an hourglass geometry sample (Figure 1B) in resonance loading was used, where the applied stress is at maximum at the sample midsection and then declines towards the sample ends, damage variations with strain or stress from the fractured part to the inside matrix can be studied. Figure 6 shows damage variations in sample No 9. In the area with less applied stress, SCZ at grain boundaries (GBs) and at GB triple and quadruple junctions can be observed (Figure 6A). With further cyclic loading/straining, new small grains can be observed at triple junctions or GBs, Figure 6B, and finally, a fine-grained structure can be observed near the fracture, Figure 6C. This indicates that strain localization during cyclic loading will cause grain fragmentation even at a low-stress amplitude.

In a polycrystalline austenitic steel, each grain has a different orientation and, consequently, different Schmid factor during loading. According to Schmid’s law, a grain with a larger Schmid factor will have a higher shear stress that may lead to plastic deformation in the crystal plane and cause strain localization. Earlier studies have shown that strain localization in some grains can be more than five times higher than the average strain in the matrix in the austenitic material during VHCF, especially at low-stress amplitudes. In certain grains, the interactions of the moving dislocations at crystal planes will form dislocation sub-cells or low-angle GBs that cause grain fragmentation. This is confirmed by this work. Figure 7 shows the dislocations in the matrix and formation of dislocation sub-cells (A) and the fine low-angle GB grains (B).

Once the localized stress concentration is higher than the critical shear stress for dislocation slip, dislocations can move freely in high Schmid factor cube grains or soft grains, which causes stress concentration at the GB and formation of dislocation sub-cells in the matrix during continuous deformation and crystal rotation. Grain fragmentation is a continuous process during the whole VHCF process, which causes the dislocation sub-cells to become smaller and smaller (new sub-cell in Figure 7A). On the other hand, high-strain localization can cause exhaustion of local plasticity and, consequently, the formation of localized SCZ at the low-angle GBs, and it leads to crack initiation. Figure 8C shows a short crack observed in fine grains with low-angle GBs. The crack started at a triple junction point and propagated either
along the GB or across the grain in transgranular cracking and then ended at another triple junction point. The crack propagation along the GB may cause a nano-rough surface that has been observed at the FGA. This may be the crack initiation for the formation of FGA. Once this crack reaches a critical length, the crack will propagate with a normal stage II process, as discussed in references,\(^{25,45}\) and an FGA has formed. It is interesting to mention that the average grain size of the material is about 43 μm, but the FGA size is between 14 to 58 μm. They have a similar size level. This may further confirm the localized damage in “soft grain” has caused the formation of FGA. Figure 8A shows the crack path from crack initiation to propagation. Fine-grained layers at the crack initiation area and a crack path near the crack initiation area were observed. This layer is rather thin, only a few micrometers, as shown in Figure 8B. This is consistent with earlier observations with focused ion beam (FIB)/TEM investigation.\(^{20,26}\) Actually, there is a grain size gradient from very fine to large from the FGA to the inside of the sample. Two nanograin layers appear. Very fine nanograins can be observed near the fracture surface, and then grain size becomes progressively larger towards the inside. This further confirms that a grain fragmentation process by the formation of dislocation sub-cell or low-angle GB is a continuous process during cyclic loading/straining. The size becomes smaller near the stress/strain localization area, as shown in Figure 8C. This may also lead to a lower SIF threshold for crack propagation.\(^{15,19}\)

The above results and discussion provide a proposal mechanism for the formation of FGA in a non-defect matrix of this homogeneous single-phase alloy:

1. Cyclic loading will cause stress or strain localization, especially at triple junction points and GBs.
2. Dislocation slip initiation in the localized stress concentration area causes grain fragmentation by the formation of dislocation sub-cells or low-angle GBs. The size becomes smaller with continuous cyclic loading.
3. Fatigue crack initiation starts at a low-angle GB (triple junction) due to local plasticity exhaustion.
4. Crack propagation at nanograin boundaries can cause a rough fracture surface
5. When a short crack reaches a critical size, a normal stage II crack propagation can occur, extending out of the generated FGA.

This mechanism provides a confirmation of the interesting models on the formation of FGA at a subsurface inclusion recently.\(^{7,15,19,21,45}\) However, the current study provides a fundamental understanding of how material damage and crack initiation occur in a material matrix during VHCF. The formation of FGA near a subsurface...
inclusion is only a special case due to the stress concentration formed near a subsurface inclusion, which may lead to a shorter VHCF life.

4 CONCLUSIONS

Damage and formation of the FGA in a non-defect matrix of one austenitic steel submitted to VHCF were investigated, which leads to the following conclusions.

During cyclic loading within the VHCF region, strain localization will occur in the soft austenitic grains. Dislocation slipping and crystal rotation leads to the formation of dislocation sub-cells with low-angle GBs. Cyclic hardening is a result of this process.

Fatigue crack initiation will then occur at triple junction points of low-angle GBs by strain localization or stress concentration that causes localized plasticity exhaustion.

An FGA is completed once the short crack FGA propagation reaches a stress intensity threshold for further crack propagation. Crack propagation at low-angle GBs may cause a rough surface in the FGA.

The progressive stepwise load increasing test (PSLIT) is a suitable method for fundamental studies on the damage and crack initiation in pure matrix materials.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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