Characterization of dislocation evolution in cyclically loaded stainless steels via X-ray diffraction line-profile analysis

by

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Abstract

Dislocations in stainless steels under cyclic loading were quantitatively evaluated via X-ray diffraction line-profile analysis to determine the relationship between the dislocation characteristics and low-cycle fatigue (LCF) life in different stainless steels. The dislocation density of AISI 316L and AISI 430 varied linearly with respect to the LCF life in a double-logarithmic graph, with different slopes of the line. The dislocation density normalized by the maximum work hardening for both stainless steels exhibited a log–log linear relationship with the LCF life. The fraction of screw dislocations in AISI 430 decreased with decreasing LCF life owing to the easy cross-slip of dislocations. Because of the difficulty of the cross-slip of dislocations in AISI 316L, the fraction of screw dislocations remained almost constant throughout the LCF life. Analysis of the crystallite size and the dislocation arrangement with respect to the dislocation density under tensile and cyclic loading revealed that the dislocation arrangement for cyclic loading was smaller than that for tensile loading. Thus, the dislocation arrangement was related to the cyclic loading. In the plot of the dislocation evolution versus the number of cycles, two stages were observed in the variation of the dislocation characteristics for both stainless steels. In the first stage, the dislocation density increased, and the crystallite size decreased. The dislocation arrangement parameter of AISI 316L and AISI 430 decreased and remained the same, respectively, in the first stage. In the second stage, the dislocation density, dislocation arrangement parameter, and crystallite size remained constant.

In a cyclically deformed metastable stainless steel (AISI 304), in addition to X-ray diffraction line-profile analysis, quantitative and qualitative data on the formation, fraction and distribution of martensite in the cyclically loaded AISI 304 with different stress amplitudes are studied by electron backscattered diffraction (EBSD). In spite of almost same tensile elongations in AISI 304 and AISI 316L, the LCF lives of AISI 304 were higher than those of AISI 316L. The dislocation density of austenite phase increased with decreasing the LCF life. In lower LCF lives, differences between dislocation density of AISI 304 and AISI 316L were increased, and contribution of $\alpha'$-martensite phase to work hardening of fatigued 304 increased with decreasing LCF life. However, contribution of austenite slightly changed. Higher work hardening via strain-induced martensite transformation improves LCF behavior of 304 compared with 316L in lower LCF life by increasing fatigue stress amplitude in a same LCF life.
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Chapter 1 Fatigue of metallic materials and characterization methods
1.1 Historical overview

Fatigue is the localized and progressive structural damage that occurs when a material is subjected to cyclic loading. The maximum stress values are less than the ultimate tensile strength and may be below the yield stress of the material. Fatigue has been a problem for engineers since the earliest days of industrial revolution. The first study of metal fatigue was conducted by a German mining engineer, W.A.J. Albert, who performed repeated proof load tests on iron mine-hoist chains around 1829. Since then, significant progress has been made in the study of fatigue [1] [2] [3].

Over the years, fatigue has been a widely accepted term in engineering field to describe damage and failure of materials under cyclic loading. In general, there are several common types of fatigue: mechanical, creep, thermo mechanical, sliding, rolling, fretting and corrosion fatigue [1]. Mechanical fatigue is due to fluctuations in applied stresses or strains. Cyclic loading in association with high temperatures, results in creep fatigue while a combination of cyclic loading and thermal high-low temperature fluctuation causes thermomechanical fatigue. Repeated applications of load in conjunction with sliding and rolling contact between material surfaces produce sliding and rolling contact fatigue respectively, while fretting fatigue occurs as a result of pulsating stresses along with oscillatory relative motion and frictional sliding between surfaces. Corrosion fatigue is caused by fluctuating load in the presence of corrosive environments.

The first detailed research into fatigue was initiated in 1842 following the Versailles railway accident in France [1]. An early explanation for fatigue was the ‘crystallization theory’, which postulated that the cause of fatigue failure in materials resulted from microstructural crystallization due to the very fine and smooth appearance of fatigue crack. This theory remained unchallenged for several decades until the work of Ewing and Humphrey in 1903 showed the development of slip bands and subsequent fatigue cracks in polycrystalline materials [4]. They tested specimens of Swedish iron in rotating bend at stress levels above their fatigue limit, the specimen surface being polished and etched. They observed few slip lines initially and new slip lines formed close to existing ones, producing slip bands. Although these bands grew wider and denser, there were areas between the bands where no slip was observed. Successive workers using other ductile polycrystals and more sophisticated metallographic techniques have added more detail which, in general, has confirmed the sequence of events described by Ewing and Humphrey. For example, some decades later,
Thompson [5] tested annealed electropolished, polycrystalline high-purity copper specimen in reversed direct stress (zero mean load), the specimen being removed from the fatigue machine and examined metallurgically. Slip bands appeared early in the test and became more numerous as the test progressed. Electropolishing removed the roughness associated with slip bands, and most of them became invisible. A few, however, became accentuated and were termed persistent slip bands; fatigue cracks grew eventually from these bands. If the electropolishing was continued until the persistent bands were removed, it was found that the slip bands reformed and again became persistent. In many cases the pattern of the new bands reproduced in some detail that which had been removed, implying that slip was still active on the same planes. Because no new markings were ever uncovered in internal structure of material during the electropolishing, this shows that the origin of cracking was associated with the free surface.

One of the earliest systematic investigations of fatigue failure, worked by A.Wöhler from 1852-1869, was full scale fatigue testing in torsion, bend and axial loading of railway axles [1]. His work contributed to the characterization of fatigue behavior in terms of stress amplitude-life (S-N) curves and also to the introduction of the concept of fatigue endurance limit. Some engineers in the second half of the nineteenth century, such as Gerber (1874) and Goodman (1899) began developing methods for fatigue design and formulated ways to account for mean stresses. Bauschinger (1886) first observed the differences in the elastic limit for materials subjected to reverse loading, and later in 1910, the changes in stress-strain response during cycling were investigated by Bairstow, and he subsequently identified cyclic softening and hardening behavior in metals [1] [2] [3].

Basquin in 1910 first proposed equations for the characterization of S-N curves and it was later refined by Coffin and Manson for strain-based characterization of fatigue [6]. Investigations into damage accumulation models for fatigue were performed by Palmgren and Miner [7]. Neuber, in 1946, studied the effects of notch on monotonic and cyclic deformation, while Langer (1937) pioneered the work in variable amplitude fatigue.

In 1961, a power law was proposed by Paris, Gomez and Anderson which characterized the crack propagation rate in terms of stress intensity factor amplitude, $\Delta K$. This later became known as Paris law. The next important contribution came in 1970, when Elber showed that fatigue cracks could remain closed even when subjected to cyclic tensile loads. From this, the
concept of crack closure was born. Since then, Ritchie, and Suresh, amongst many others, have intensified research in crack closure and its effects on fatigue crack propagation.

More recently, significant interest in fatigue research has centered on short or small fatigue crack propagation. This problem was first identified by Pearson in 1975, who observed that crack propagation rates for short cracks are higher than those observed for long cracks at the same stress intensity range. Crack propagation, even at stress intensity ranges below the threshold for long cracks has also been detected. Such anomalous behavior contradicts conventional LEFM theory and thus far, significant progress has been made in characterizing the types of short cracks and explaining this unique behavior [8].

The past decade has witnessed a confluence of high-performance computing, advanced experimental methods for in situ and ex situ measurement of evolving microstructure at various length scales under cyclic loading, and substantial advances in understanding deformation mechanisms, material degradation, and relevant modeling concepts in fatigue. Together, these various elements have introduced new possibilities to consider the role of the microstructure on the initiation and propagation of small cracks in fatigue. Microstructure-sensitive computational methods for fatigue crack formation in polycrystalline microstructures over the past few decades were developed rapidly [9] [10] [11] [12] [13] [14] [15]. Such approaches can be pursued to explore effects of microstructure attributes that give rise to extreme value fatigue responses which are associated with the tails of probability distributions of potential surface and subsurface fatigue crack formation sites, including transitions between modes of crack formation, surface to subsurface transitions, and so on.

1.2 High cycle fatigue and low cycle fatigue

1.2.1 High cycle fatigue

High-cycle fatigue is considered when the cyclic loadings induce stresses close to but below the engineering yield stress so that the number of cycles to initiate a microcrack is high. The plastic strain is usually not measurable on a mesoscale, but dissipation exists on a microscale to induce the phenomenon of damage [16].
1.2.2 Low cycle fatigue

Low cycle fatigue failures may occur when structures are subjected to heavy cyclic loadings which induce irreversible strains on small or large scale, giving rise to damage up to crack initiation and propagation [16].

The number of cycles to rupture can be on the order of 10 to 100 for aerospace rockets. It can be on the order of 100 to 1000 for nuclear or thermal power plants, chemical plants, and many domestic apparatuses as the butt hinges of polymeric boxes. The state of stress is somewhat higher than the yield stress. For aircraft engines or car engines where, on some parts, the number of cycles to rupture can be on the order of 1000 to 10000, the state of stress induces plastic strain on the order of the elastic strain. For $10^4$ or $10^5$ range, low- or high-cycle fatigue may be considered depend on the case and, moreover, on the degree of accuracy needed [16]. Low cycle fatigue is mainly governed by the local strain at meso-level and it is essentially encountered in metals.

1.3 Fatigue analysis

Generally, there are three main approaches (stress, strain and “damage” approach or fracture mechanics) to fatigue analysis [2] [3] [17] [18].

Under stress-based approach, the stress-life (S-N) curves (magnitude of a cyclic stress ($S$) against the number of cycles to failure ($N$)) are used primarily for fatigue life prediction. A main feature commonly found in S-N curves is the fatigue or endurance limit, $\sigma_D$, which defines the maximum stress amplitude possible for a defect-free specimen to have infinite life when mean stress is zero.

Early fatigue investigations were carried out using predominantly stress-based approaches. However, since the introduction of servo-hydraulic testing machines, strain-based approaches have become increasingly popular in their use to analyze fatigue problems. Both approaches represent extremes of completely unconstrained or stress-cycling conditions and completely constrained or strain-cycling conditions. However, fatigue under conditions of controlled strain is more favorable since most high-performance engineering components are subjected to a certain degree of structural constraint, reminiscent of strain-control. Furthermore, fracture mechanics has proved efficient in facilitating the understanding of crack propagation.
mechanisms and characterizing crack propagation rates. Stress or strain-based approaches may provide total life data for smooth samples subjected to complex loading histories [18].

The 304L or 316L steel structures, for example, used in the nuclear industry are particularly subjected to low-cycle fatigue (LCF). LCF analysis needs to handle local plasticity effects and takes into account both elastic and inelastic deformation under constrained conditions. However, according to fatigue tests of 304L, it was found that even when 304L steel is subjected to a small level strain amplitude loading, it shows cyclic plastic behavior as well [19]. In particular, the stress-based, strain-based, energy-based and mixed approach can all commonly be used for the fatigue analysis of structures in stainless steels [20].

1.4 Cyclic deformation

1.4.1 Cyclic stress – strain response

When a metal is subjected to cyclic loading, characteristic stress-strain response curves are observed depend on the material type, initial condition (annealed or work hardened) and loading conditions (stress or strain controlled, loading amplitude and so on). Under strain-controlled fatigue, cyclic hardening means stress amplitudes increases with increasing number of cycles. The opposite occurs for cyclic softening, where the stress amplitudes of hysteresis loops decreases with increasing number of cycling. In general, the rate of hardening progressively diminishes and reaches the maximum stress amplitude then cyclic softening occurs. And with continued cyclic loading, a quasi-steady state of stress-stress cycle (cyclic saturation) will be reached. It is common for cyclic saturation to be reached within half-life in most cases of fatigue loading. As a result, the calculation of fatigue parameters is usually based on mid-life hysteresis loop data [3] [18] [21].

For 304L steel, generally the fatigue behavior consists of three phases during fatigue under cyclic constant strain amplitude loading. There is first a consolidation phase with a primary hardening during the first cycles. This phase is followed by a cyclic softening phase, and then it reaches a stabilization phase. However, the stabilization is not observed for all tests, it depends on the imposed strain amplitude. Fig. 1.1a gives the change of stress for different strain amplitudes during the total strain-controlled fatigue test for 304L with the different strain amplitudes from 0.15% to 1% [19]. It can be found that cyclic hardening and softening behavior is significant. However, in the lower domain of plastic strain amplitude (less than 0.01%), the pronounced initial growth of the stress amplitude is followed by saturation (see
The saturated stress amplitude (in terms of hysteresis loop) is constant up to $3 \times 10^6$ - $5 \times 10^6$ cycles [22]. Furthermore, in the higher plastic amplitude domain (greater than 1%) due to the early fracture, the softening is only transient or absent and the stress amplitude increases during most of the fatigue life.

**Fig. 1.1.** (a) Cyclic hardening/softening curves for 304L steel [19] (b) Evolution of stress-strain loops under controlled strain cycling amplitude ± 0.005, a progressive decrease in stress amplitude is observed with increasing cycles, final instability is due to macrocrack propagation.

**Fig. 1.2.** Cyclic hardening/softening curves for 304L steel (a) 316L steel (b)316L steel [22].

Other research work showed that, for 304L or 316L steel hardening/softening behavior, the stress/strain curve at mid-life and the crack initiation life are both strongly influenced by temperature [17] [23] [24] (see Fig. 1.3). The evolution of maximum and minimum stress at
the same total strain amplitude shows that the material has a higher tensile stress and a higher fatigue life at 90°C than at other higher temperature levels.

Fig. 1.3. Temperature effect on the fatigue behavior of 304L (a) Evolution of minimum and maximum with increase of the number of cycles (b) Stress/strain curves at mid-life under 90, 165 and 320 °C, total strain amplitude equal to ± 0.2%, f = 1 Hz [25].

1.4.2 Evolution of dislocation microstructure during cyclic loading

It is important to explain the stress-strain response of fatigue loading on the basis of microstructure because microstructural processes underlie and influence the examination, interpretation, and understanding of macroscopic behavior. The basic mechanism for cyclic deformation involves irreversible dislocation movement within the microstructure.

Experiments of fully reversed fatigue under constant amplitudes of resolved plastic shear strain point to the existence of a saturation stress after initial cyclic hardening. A plot of peak resolved shear stress at saturation as a function of plastic shear strain amplitude provides the cyclic stress-strain curve for a copper single crystal, which was first experimentally measured by Mughrabi [26]. Fig. 1.4 shows schematically a diagram for FCC single crystals oriented for single slip.
The microstructural processes that take place in polycrystals are basically the same as those studied for single crystals: at low amplitudes, loose veins are formed; in an intermediate range, veins with PSBs are formed and at high amplitudes, cells are formed. Even when cyclic saturation is reached, minor changes to dislocation structures have been found. It is important to realize that dislocation arrangement and cell size are determined primarily by the plastic strain amplitude [1] [3] [22] [27] [28]. For 316L or 304L steel, there are also three similar regimes considered to describe the microstructure evolution as shown in Fig. 1.5 [22] [27]. The dislocation arrangement for slip systems during cyclic loading is highly dependent on the loading amplitude and number of cycles during fatigue [22] [27] [28].

Fig. 1.4. Schematic diagram showing different regimes of the saturation stress-strain curve [1].
Cyclic loading in small plastic amplitudes yields arrangements of edge dislocation dipoles caused by single slip. Dislocations often agglomerate into bundles or veins which are separated by regions of low dislocation density. At low plastic amplitudes in region A (in Fig. 1.4), cyclic hardening is due almost entirely to the accumulation of primary edge dislocations. During the initial phases of cycling, dislocations are produced which accumulate on the primary glide plane [1]. Fully reversed cyclic strain produces approximately equal amounts of positive and negative edge dislocations which are attracted over small distances to form dislocation dipoles. This process of positive and negative dislocation attraction can also be termed trapping. Only edge dislocations are likely to form dipoles since screw dislocations have a tendency of cross slipping, which is promoted in materials with high stacking fault energies, mutually annihilating each other in the process. Fig. 1.6 shows the dislocation structures of a 316L specimen cycled with rather low plastic strain amplitude of $5 \times 10^{-4}$ at middle life and at failure [27].
Fig. 1.6. Dislocation structure of a 316L specimen cycled with a plastic strain amplitude of $5 \times 10^{-4}$ (a) $N=2.2 \times 10^6$ cycles (at mid-life) (b) $N = 5.3 \times 10^6$ cycles (at failure) [27].

After cycling under a little higher plastic strain amplitude from regime A to B, the accumulation of dislocations occurs predominantly in the form of mutually trapped edge dislocations, typically called veins, bundles or loop patches, making up approximately 50% by volume of the matrix shown in Fig. 1.7a. Dislocation veins contribute to the cyclic hardening during the initial stages of fatigue by partially impeding dislocation motion on the primary slip system. Matrix veins are known to accommodate only small plastic strains of the order of $10^{-4}$ and undergo microyielding [1]. One of the most visible features of cyclic saturation is the localization of slip along bands. This process is formed at strain amplitudes corresponding to the beginning of region B and is intensified as the applied plastic strain is increased as shown in Fig. 1.8. These slip lines were termed persistent slip bands firstly by the group of Thompson [5]. They found that in Ni and Cu, the bands persistently reappeared at the same sites during continued cycling even after a thin layer of the surface containing these bands was removed by electropolishing. Some studies of microhardness measurements on fatigue-induced slip bands revealed that the PSBs are much softer than the matrix [29]. These results imply that during saturation in the plateau region of the cyclic stress-strain curve, essentially the entire deformation is carried by PSBs. In fact, the formation of the PSBs appears to be closely related to the occurrence of the plateau. The increase in the volume of the crystal covered by the PSBs is due to the increase in plastic strain amplitude [1]. From Fig. 1.8, it is clearly shown matrix structure with PSB structure in the 316L specimens cycled at beginning of the plateau regime which is also in accordance with the descriptions of those in single crystal [27].
**Fig. 1.7.** Schematic representation of the dislocation arrangements in (a) a matrix structure and (b) a persistent slip band [1].

**Fig. 1.8.** Matrix structure with PSB structure in the 316L specimens cycled in the plateau regime (a) with a plastic strain amplitude of $2 \times 10^{-4}$, $N=1.3 \times 10^6$ cycles (at failure) (b) a plastic strain amplitude of $1 \times 10^{-3}$, $N=9 \times 10^4$ cycles [27].

The dislocation structures in PSBs are considerably different to that observed in a matrix. It consists of dislocation veins that only occupy 10% of volume and are arranged into wall-like configurations (Fig. 1.7b) [1]. PSBs can support high plastic shear strains of the order of 0.01 and undergo macroyielding. A dynamic equilibrium between dislocation multiplication and annihilation has been identified as the saturation mechanism for fatigue involving...
intermediate degrees of plastic straining and PSB formation. Dislocation walls (veins) and dislocation channels take part in plastic deformation by macroyielding of PSB. Edge dislocations bow out from walls, traverse the channels, and penetrate partially into the opposite wall leading to the existence of screw dislocation segments that glide along the channels. During cyclic deformation, edge dislocations will glide between walls with constant renewal of dislocations caused by a dynamic process of dislocation formation and annihilation. Annihilation occurs by climbing of edge dislocations of opposite signs in the wall structures of PSBs or by cross slip between screw dislocations. Therefore, bundles and walls consist primarily of edge dislocations [1].

At higher loading amplitudes, secondary slip occurs, and the multiple slip contributes to the formation of labyrinth and cell-like dislocation structures (region C in Fig. 1.4). Fig. 1.9 shows PSBs structures in specimens of 316L steel cycled with plastic strain amplitude of $5 \times 10^{-3}$ at failure.

![Fig. 1.9. PSB structures in specimens of 316L steel cycled with plastic strain amplitude of $5 \times 10^{-3}$, $N_f=2800$ (a) cell structure with a PSB (b) PSBs with wall structure.](image)

Cell size has been reported to decrease with increasing plastic strain amplitude and saturation. For material with high stacking fault energy, cell size is independent of the initial condition of the material (i.e. cell structures during cyclic saturation are the same for annealed or cold worked materials). It was also revealed that cell size increases as the test temperature increases [30] [31] [32] [33].
It was observed that microstructures in 304L under two levels of plastic strain amplitude 0.15% and 1% in high-temperature environment at 300°C as shown in Fig. 1.10. The results show that the temperature has no effect on the microstructures type changes [34].

![Microstructures observed in 304L at 300°C](image)

**Fig. 1.10.** Microstructures observed in 304L at 300°C [34] (a) Planar dislocation structure (b) channels and veins (c) PSB structure.

Furthermore, as for cyclic softening observed in Fig. 1.1 and Fig. 1.2, two ways of physical explanations to this macro behavior can be found. On one hand, cyclic softening can result from a decrease in dislocation density through dislocation annihilation and change in dislocation structure [35] [36]. On the other hand, cyclic softening can also be explained by using the concept of ‘dislocation starvation’ proposed by Li and Laird [37] [38]. According to Li and Laird, cyclic softening occurs for polycrystals due to ‘dislocation starvation’ - i.e. the inability or difficulty in generating dislocations to carry the applied strain in the first few cycles. The initial stress applied is high in order to generate enough dislocations to carry the imposed strain. Dislocations are required to carry strain – the more mobile the dislocations, the better they can carry the applied strain. As a result, cyclic softening can occur due to difficult dislocation generation in the early stage, or through dislocation annihilation where reduced dislocation density reduces the resistance to dislocation movement.
1.5 Fatigue crack initiation and propagation

1.5.1 Fatigue crack initiation

The development of a fatigue crack is traditionally classified into two main phases: 1) crack initiation and microcrack propagation 2) stable crack propagation and fracture. The definition of crack “initiation” has evolved with the advancement of crack detection methods. This limit of crack detection has improved over the years from several millimeters to the order of 1 μm or less. Since a fatigue crack must be of a certain length before it can be observed, some microcrack propagation will always occur before the measured cycles to crack “initiation” is detected [2] [3].

It should be noted that surface micro-notches, inclusions or inherent microscopic defects, all provide avenues for immediate crack propagation. Henceforth, the term fatigue crack “initiation” will be quoted in inverted commas to remind that such a phase though often quoted in textbooks and in literature, may not actually exist in reality. It should be reminded that the word “initiation” is a loosely coined termed used simply to refer to the onset of a detectable crack and not the very beginning of crack propagation.

In 1903, Ewing first demonstrated the microcracks occur as a result of cyclic deformation leading to strain localization. This strain localization is caused by irreversible dislocation movement (slip along crystallographic planes when stress is applied) and with time, the dislocations agglomerate into bundles at critical stress or strain levels causing strain localization to occur, thus forming thin lamellae of persistent slip bands also known as PSBs [3] [39] [40]. The PSBs at free surface is usually the preferred area for the “initiation” of fatigue cracks [17] [39] [40] [41]. The following early crack propagation in stage I on single crystallographic slip bands inclined by about 45° to the applied stress (Fig. 1.11) is determined by cyclic shear displacement at the crack tip [1] [3].
This initial crack propagation is identified as stage I crack propagation and usually penetrates only a few tenths of one millimeter (i.e. one or a few grains) into the specimen. Fatigue crack “initiation” and crack propagation during stage I, occurs by slip plane cracking. Stage I crack propagation is observed both in high cycle fatigue and in low cycle fatigue in 304L and is strongly affected by environments and microstructures that tend to concentrate slip and enhance slip reversibility [24].

The cyclic shear stress is required to form cyclic slips. On the microscale, the shear stress is not homogeneously distributed through the material. The shear stress on crystallographic slip planes differs from one grain to the others, depend on the size and shape of the grains, on the crystallographic orientation of the grains, and on the elastic anisotropy of the material. In some grains at the material surface, these conditions are more favorable for cyclic slip than in other surface grains. If slip occurs in a grain, a slip step will be created at the material surface. A slip step implies that a new surface of the material will be exposed to the environment. The fresh surface material will be immediately covered by an oxide layer in most environments, at least for most structural materials. Such monolayer strongly adheres to the material surface and is not easily removed. Another significant aspect is that slip during the increase of the load also implies some strain hardening in the slip band. As a consequence, upon unloading, a larger shear stress will be present on the same slip band, but now in the reversed direction. It is clear that the oxide monolayer cannot simply be removed from the slip step and that strain hardening
in the slip band is also not fully reversible. As a consequence, reversed slip, although occurring in the same slip band, will occur on adjacent parallel slip planes [1] [3]. Fig. 1.12 schematically describes in extrusions and intrusions produced by cyclic plastic strain on the surface.

Fig. 1.12. A rough surface consisting of extrusions and intrusions produced by cyclic plastic strain (redrawn from [1]).

Fig. 1.13 shows the formation and development of cyclic slip bands and a microcrack in a pure copper specimen [43] [44]. The lower restraint on cyclic slip at the material surface has been considered as a favorable condition for crack initiation at the free surface. However, more arguments for crack initiation at the material surface in polished specimens are present [1] [2] [3] [16] [18]. A very practical reason is the inhomogeneous stress distribution due to a notch effect of a hole, an inclusion, grain boundaries or some other geometric discontinuity. Because of an inhomogeneous stress distribution, a peak stress occurs on the surface (stress concentration). Furthermore, surface roughness also promotes crack initiation at the material surface. Other surface conditions with a similar effect are corrosion pits and fretting fatigue damage both occurring at the material surface.
Fig. 1.13. (a) Development of cyclic slip bands and a microcrack in a pure copper specimen, totally reversed stress amplitude, 77.5 MPa stress amplitude, $2 \times 10^6$ life (a1) Slip lines are clearly visible (a2) Same as in (a1) but plastically strained (5%) which opens a microcrack, see arrow [43] (b) Intrusion and extrusion [45].

As long as the size of the microcrack is still on the order of the grain size, the microcrack is obviously present in an anisotropic material with a crystalline structure and a number of definite slip systems. The microcrack contributes to a more inhomogeneous local stress distribution, with a stress concentration at the tip of the microcrack. As a result, more than one slip system may be activated. Moreover, if the crack is growing into some adjacent grains, the constraint on slip displacements will increase due to the presence of the neighboring grains. Similarly, it will become increasingly difficult to accommodate the slip displacements by slip on one slip system only. Several slip systems will thus be activated. The microcrack propagation direction will then deviate from the initial slip band orientation. Because microcrack propagation depends on crystallography, microstructural barriers to slip can imply an obstacle for crack propagation. As the illustrative results presented in [46] (see Fig. 1.14), the crack propagation rate measured as the crack length increment per cycle decreased when the crack tip approached the first grain boundary. After penetrating through the grain boundary,
the crack propagation rate increased during growth into the next grain, but it decreased again when approaching the second grain boundary. After passing that grain boundary, the microcrack continued to grow with a steadily increasing rate.

**Fig. 1.14.** Grain boundary effect on crack propagation rate in an Al-alloy [52]. The crack length was measured along the material surface.

During these phases, microcrack propagation is strongly affected by the microstructure of the material and crack advance is dominated by local plasticity. This aspect is especially important for the materials for which a substantial part of the total life is spent during short fatigue crack propagation. For example, cyclically loaded components in structural applications often undergo stress amplitude that is close to the fatigue limit of the material used. Under such conditions, the stages of crack initiation and short crack propagation are considered to play an important role for the lifetime [40] [47] [48]. **Fig. 1.15** shows the surface observation of the short cracks with the size about 100 to 200 µm. This point was also confirmed by the work of [49], crack distribution during fatigue test also showed that the sizes of cracks were mainly found in the range from 20 to 120 µm.
Fig. 1.15. Typical cracks and slip lines on the specimen surface in 304L at the N = 15000 cycles under total strain-controlled amplitude of ±0.3% (N/Nf=50%)

Fig. 1.16. Crack distribution during fatigue test in 304L under total strain-controlled amplitude of ±0.3%, Nf=25000 cycles [49].

Furthermore, concerning numerical simulation some studies about the initiation and propagation of the stage I cracks can be found in literature. Some models of short crack fatigue growth have been proposed [42] [50]. Brinckman and Van der Giessen used computational framework that ties together dislocation dynamics, the fields due to crystallographic surface steps and cohesive surfaces to model near-atomic separation leading to fracture [51]. Crack tip shielding by dislocations is an essential feature of the modeling of initiation and propagation of the cracks. Some researchers considered that crack tip shielding by dislocation can cause a
reduction in fatigue growth rate and several multiscale simulation of crack tip shielding by dislocation has been worked out for confirming these conclusions [52].

When the crack propagates long enough and out of the stage I, the influence of the microstructure can become negligible. It is well established that the part of life of a component spent in the Paris regime [53], can readily be estimated.

1.5.2 Fatigue crack propagation

When the crack is long then the microstructure no longer affects the crack propagation. At this moment, the rate at which a long crack grows has considerable importance in determining the life of a material. The crack growth rate depends on the stress level applied. In the 1960's, Paris examined a number of alloys and realized that crack growth rate against range of stress intensity factor gave the relation as [53]:

$$\frac{da}{dN} = C(ΔK)^n$$  \hspace{1cm} (1-1)

where the parameters $C$ and $n$ are properties of the material, $da$ is the change in crack length, and $dN$ is the change in the number of cycles, $ΔK$ is the change in the stress intensity factor which is dependent on stress and crack length. As a result, the relation of the number of cycles of failure $N_f$, to the size of the initial default length $a_0$, and the critical crack length $a_C$ can be written as:

$$N_f = \int_0^{N_f} dN = \int_{a_0}^{a_C} \frac{da}{C(Δ\sqrt{πa})^n} = \frac{1}{Cn\pi(Δσ)^n} \int_{a_0}^{a_C} \frac{da}{a^{2n}}$$ \hspace{1cm} (1-2)

where $N_f$ is an estimate of the number of cycles to failure, and $ΔK = Δ\sqrt{πa}$.

It must be noted that Paris law, for the first time, made it possible to make a quantitative prediction of residual life for a crack of a certain size. Now it is well known that $·$ also depends on several other factors such as stress ratio, loading frequency, loading sequence, and environment conditions. In the last few decades, it has been modified to take into account more other factors to use in the special loading conditions such as Walker’s equation, Forman’s equation and Elber’s equation [1] [2] [3] [17] [18].
1.5.3 Transition from the crack initiation to propagation stage

As mentioned above, the fatigue life was described as consisting of a crack initiation stage and propagation stage (Fig. 1.17). The definition of transition from the initiation stage to the crack propagation is difficult even cannot really be given in quantitative terms. According to Schijve [3], the definition is that the first initiation stage is over when microcrack propagation is no longer depend on the material microstructure, or surface conditions for a polished specimen. The sizes of the microcrack at the transition from the initiation stage to the crack propagation stage differ significantly with the types of materials. The transition depends on microstructural barriers to be overcome by a growing microcrack, and these barriers are not the same in all materials. The transition also implies the change from microstructurally short crack to short crack (about 200 µm to 1mm) which is no longer sensitive to microstructure, but the crack is still short and influenced by full opening and closing at the bottom of the cycle. Fig. 1.18 provides a schematic comparison between stress range and crack length, on a log-log scale as first presented by Kitagawa and Takahashi [8], in which $a_1$ is a size comparable to the scale of some microstructural dimension, for example the grain size and $a_2$ is typically between 200 µm to 1 or 2mm [3] [18].

![Fig. 1.17. Various stages of the fatigue life redrawn from [3].](image)
Fig. 1.18. Schematic of Kitakawa-Takahashi diagram showing the relationship between stress range and crack length [18].

The crack initiation stage includes the initial microcrack propagation. Because the growth rate is still low, the initiation stage may cover a significant part of the fatigue life. This is illustrated by the generalized picture of crack propagation curves presented in Fig. 1.19 which schematically shows the crack propagation development as a function of the percentage of the fatigue life consumed [3]. There are three curves in Fig. 1.19, all of them in agreement with crack initiation in the very beginning of the fatigue life, however, with different values of the initial crack length. The lower curve corresponds to microcrack initiation at a perfect surface of the material; the middle curve represents crack initiation from an inclusion; the upper curve is associated with a crack starting from a material defect which should not have been present, such as defects in a welded joint.

From Fig. 1.19, it is clear that only cracks starting from macro defects can have a detectable macrocrack length immediately. The two lower crack propagation curves illustrate that the major part of the fatigue life is spent with a crack size below 1 mm, i.e. with a practically invisible to bare eyes crack size. It is also possible that cracks do not always grow until failure as shown with dotted lines in Fig. 1.19. It implies that there must have been microstructural barriers in the material which stopped crack propagation.
1.6 Fatigue under variable-amplitude loading

Constant-amplitude (CA) fatigue loading is defined as fatigue under cyclic loading with constant amplitude and a constant mean load. However, it was well known that various structures in service such as in airplanes and in pressurized water reactors are in practice subjected to variable-amplitude (VA) loading, which can be a rather complex load-time history [2] [18].

Fatigue behavior of metallic materials under variable amplitude loading has been addressed in many papers over the last decades. Some of them pay special attention to the simulation of representative load histories of defined random loading processes. Others analyzed the effect of overloads and their distribution throughout the load history of the crack propagation life. There are also many articles that present numerical or analytical models to simulate the crack propagation under this type of loading. However, all these current damage accumulation rules proposed are based on phenomenological approaches. So fatigue behavior under VA loading remains an open problem and fatigue life prediction is obviously more complex than that under CA loading.
At first, in order to calculate the fatigue life of a part under variable amplitude loading using constant amplitude fatigue life data, Miner popularized a rule [7] that had first been proposed by Palmgren [54]. The rule states that where there are $k$ different stress magnitudes in a spectrum, $S_i (1 \leq i \leq k)$, each contributing $n_i(S_i)$ cycles, then if $N_i(S_i)$ is the number of cycles to failure of a constant reversal stress $S_i$, failure occurs when

$$\sum_{i=1}^{k} \frac{n_i}{N_i} = D,$$

is originally proposed to be 1 and experimentally found to be between 0.7 and 2.2. Usually for design purposes, is still assumed to be 1. This can be thought of as assessing what proportion of life is consumed by reversal stress at each magnitude then forming a linear combination of their contribution.

Though Miner's rule is a useful approximation in many circumstances, some results of VA fatigue tests clearly indicated that Miner’s rule does not give accurate and reliable predictions on fatigue lives. The shortcomings can be understood. The most important one is that it does not consider the effect of overload or high level stress/strain amplitude on crack initiation and propagation.

Conventional knowledge in crack growth behavior shows that in metallic materials it is expected a decrease in crack growth rate immediately after the overloading. This idea, stemming from Elber’s model, was the first to employ the concept of crack closure to explain retardation in fatigue crack growth following overloading [55]. It was discovered that overloading produced compressive residual stresses, which acted on fatigue crack surfaces causing crack closure. As a result, subsequent tensile loading will see a portion of this tensile load consumed in overcoming the crack closure caused by residual compressive stresses ahead of the crack tip and crack growth retardation is achieved assuming that crack growth would not take place when a crack is closed. It was also suggested that the primary mechanisms of closure are reasoned to be associated with the wedging crack face asperities (roughness induced closure) and more importantly closure induced by cyclic plasticity in the crack wake [1] [3] [18].

So up to now, from the macroscopic point of view, it is well recognized that the occurrence of positive overloads or the alternance of high to low load sequences had a strong effect on the crack propagation behavior leading to favorable crack propagation retardation in certain cases.
1.7 Quantitative characterization of dislocation and its effect on LCF behavior

According to the literature, there were some reports about the correlation between dislocation substructure evolution and fatigue behavior of stainless steels. The quantified dislocation characteristics could be considered as possible candidates for prediction of fatigue failure. However, there is not any comprehensive study about this quantitative evaluation. There were a number of studies which examined the dislocation substructure by qualitative methods based on direct observation. Pham et al. [56] reported that in the as-received condition, dislocations are mainly present in the form of planar structures, e.g. regular arrays of dislocations, stacking faults. At the beginning of cyclic loading, grain-to-grain misorientations inducing plastic strain incompatibilities between grains, i.e. at an inter-granular scale, result in a significant increase in dislocation density close to the grain boundaries to preserve the continuity of material. In contrast, there is a slight increase in dislocation density inside grains. The grain-to-grain strain incompatibilities are relieved thanks to the activity of secondary slip (including cross slip) upon further loading. Secondary slip activity promotes dislocation interactions, resulting in: (1) an increase in the annihilation rate of dislocations, leading to a slight decrease in dislocation density; and (2) the rearrangement of dislocations to form lower and more stable energetic configurations, i.e. dislocation high/low density regions, to further relieve the grain-to-grain strain incompatibilities. Dislocation structures, i.e. dislocation high/low density regions, are strongly dependent on imposed plastic strain. At the end of life, dislocation structures are less well organized in lower strain amplitude tests. This is because the planar character of dislocation motion persists to a higher life fraction, and the tendency of the formation of well-organized wall/channel structures is increasingly diminished. Although the development of dislocation structures relieves the grain-to-grain incompatibilities, it raises other strain incompatibilities between dislocation high/low density regions on a finer scale, i.e. intra-granular scale. The stabilized condition of these strain incompatibilities is established upon further loading once dislocations seek their most stable configuration under a given testing condition [56].

The evolution of material microstructure during cyclic loading at ±0.7% fatigue strain is summarized with its corresponding cyclic deformation response in Fig. 1.20 [56]. During cyclic hardening, dislocation density significantly increases, in particular in regions close to grain boundaries (Fig. 1.20 (a,b)). At the end of cyclic hardening, dislocation-dense sheets are
observed (Fig. 1.20 (c)). Dislocations then rearrange during the softening stage due to the strong activation of secondary slip, finally resulting in the formation of dislocation walls/channels (Fig. 1.20 (d)). Upon further loading, the strong multiple slip activity at this strain amplitude is responsible for the development of the wall/channel structure into a cellular structure towards the end of life (Fig. 1.20 (e,f)). The activity of secondary slip systems creates more connections between walls, resulting in a labyrinth structure (Fig. 1.20 (g)). Towards the end of fatigue life, the cellular structure consequently becomes more equiaxed. Cellular structures more effectively confine the movement of mobile dislocations than wall/channel structures, thereby gradually compensating for the softening effect caused by the formation of channels and the activity of PSBs which were observed in the later part of the softening response stage (Fig. 1.20 (h)).

Paul et al. [57] tried to quantify the dislocation density and compare LCF life with dislocation density difference as shown in Fig. 1.21. The strain amplitude dependence of microstructural evolution implies that changes in internal stresses during cyclic loading are also different for different imposed strain amplitudes. Consequently, if one wants to verify the effectiveness of a proposed evolutionary constitutive model with the parameter identification being made at another strain amplitude condition, it is necessary to compensate the identified values of model parameter for the strain amplitude dependence of microstructural evolution.
The change in total dislocation density from the beginning of the test compared to the start of the test ($DD$ is dislocation density, $\delta DD$ is the change in dislocation density) showed a strong correlation with the LCF life. This behavior is independent of the deformation mode. In other words, if the steel exhibits the strain-induced martensite transformation (SIMT), this relationship between dislocation density change and LCF life remains the same. Thus, the LCF life is dictated by the overall dislocation density irrespective of the deformation mode. We can conclude then that for austenitic alloys, the optimum microstructure for resistance to LCF is one in which the dislocation density remains the same or rises in response to cyclic loading, and attainment of this behavior could be either by SIMT, slip deformation, or a combination of these two [57]. Since LCF lifetime is known to be strongly correlated with material plasticity, it is commonly found that more ductile materials show better LCF behavior [58].

As shown by one example [56], Transmission electron microscopy (TEM) has been used to characterize dislocations of cyclically loaded materials [59] [56] [60] [61] [62] [63]. However, when it comes to quantitative measurement of dislocation characteristics, it is difficult to measure the dislocation density or other parameters of dislocations in tangled structures, such as dislocation cell walls, via TEM. Also, Electron backscatter diffraction (EBSD) has been applied for studying dislocations of cyclically loaded alloys [64] [65] [66] [67]. Kernel average misorientation (KAM) is used to observe geometrically necessary dislocations (GNDs). However, for determining the relationship between the dislocation density and the LCF life, not only GNDs but also statistically stored dislocations (SSDs) should be considered. Recently,
X-ray diffraction line-profile analysis (DLPA) has been applied for characterizing dislocations in various cyclically loaded alloys [68] [69] [70]. By using X-ray DLPA, the dislocation density, dislocation arrangement, average crystallite size, and dislocation character (edge/screw) can be determined. In previous studies [68] [69] [70], X-ray DLPA was used to compare the dislocation characteristics before and after LCF of alloys. Kishor et al. [70] investigated the effects of the fatigue loading parameters on the dislocation characteristics of cyclically loaded steel via X-ray DLPA. Thus, X-ray DLPA is one of the most promising techniques for characterizing dislocations.

1.9 Aim of the work

The main objective of the present study was to determine the relationship between the quantitative dislocation parameters of fatigued stainless, namely dislocation density, dislocation arrangement, and crystallite size, and the LCF behavior by using the X-ray DLPA method. The effect of the work hardening of the materials on the relationship between the LCF life and the dislocation density was also investigated. Additionally, the changes of the dislocation parameters with different stress amplitudes were examined, and the evolution of the dislocation parameters with respect to the number of cycles was studied for different stainless steels. Also, the effect of work hardening mechanisms on the LCF life and the contributions of austenite phase and SIMT phenomenon to the work hardening by using the X-ray DLPA and EBSD methods. In view of the effect of work hardening mechanisms, a metastable austenitic stainless steel was compared with a stable austenitic stainless steel.
Chapter 2 Comparison between dislocation evolution in cyclically loaded stable austenitic and ferritic stainless steels
2.1 Introduction

Fatigue failures are widely studied because it accounts for 90% of all service failures due to mechanical causes. Among them, austenitic and ferritic stainless steels are commonly subjected to cyclic loading in industrial applications. Because of the low stacking-fault energy of austenitic stainless steel, the cross-slip of the dislocations in austenitic SS is difficult during plastic deformation, compared with that in ferritic SS. The cross-slip difficulty leads to low annihilation of dislocations, resulting in a higher dislocation density in austenitic SS compared with ferritic stainless steel. The high dislocation density is coincident with significant monotonic tensile elongation in austenitic stainless steel. It is commonly used that the low-cycle fatigue life is considered to be related to the tensile elongation; however, studies have shown that the relationship between the LCF life and the tensile elongation is not necessarily strong. Additionally, with the increase in the dislocation density and elongation, the maximum tensile work hardening of austenitic stainless steel increases. Therefore, to elucidate the effect of the maximum tensile work hardening of austenitic and ferritic stainless steels on the LCF life, measuring the dislocation density of cyclically loaded austenitic and ferritic SSs with different LCF lives is a viable approach.

The main objective of the present chapter was to determine the relationship between the dislocation density and the LCF life by using the X-ray DLPA method which was explained in the first chapter. The effect of the maximum tensile work hardening of the materials on the relationship between the LCF life and the dislocation density was also investigated. Additionally, the changes of the dislocation parameters with different stress amplitudes were examined, and the evolution of the dislocation parameters with respect to the number of cycles was studied for both austenitic and ferritic SSs.

2.2 Experimental

2.2.1 Materials

The materials under study were AISI 430 and AISI 316L. The Chemical compositions of stainless steels under study are shown in Table 2.1.
Table 2.1. Chemical compositions (mass%) of the materials under study in this chapter.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISI 430</td>
<td>0.017</td>
<td>0.14</td>
<td>0.65</td>
<td>0.28</td>
<td>15.90</td>
<td>-</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>AISI 316L</td>
<td>0.017</td>
<td>0.69</td>
<td>1.22</td>
<td>12.08</td>
<td>17.48</td>
<td>2.05</td>
<td>0.31</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Fig. 2.1 shows tensile stress-strain curves of stainless steels, tested at strain rate of $7.16 \times 10^{-4}$ s$^{-1}$ [71] at ambient temperature. The yield points of the AISI 430 and AISI 316L are comparable, however, the yield point of AISI 304 is lower than the other stainless steels. AISI 316L exhibits a higher maximum work hardening and higher elongation than AISI 430 under tensile loading.

![Tensile stress-strain curves](image)

Fig. 2.1. True stress–strain curves from the monotonic tensile test for AISI 430 and AISI 316L.

2.2.2 Fatigue test

Fig. 2.2 shows the geometry of the fatigue specimen used for the fatigue tests. The fatigue tests were performed with $\frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} = 0.1$, at a frequency of 10 Hz. Two alloys were tested under cyclic loading with different numbers of cycles and stress amplitudes ($\sigma_a = \frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{2}$), as shown in Fig. 2.3. To study the effect of the number of cycles on the fatigued stainless steels, the fatigue testing was stopped at different numbers of cycles for each sample during cycling.
and just before fracture. For all the stress amplitudes, the $\sigma_{max}$ was larger than 400 MPa. Notably, the fatigue lives were similar for AISI 316L and AISI 430 at a stress amplitude of 250 MPa, despite differences in the maximum work hardening and elongation of stainless steels under tensile loading. The dashed line in Fig. 2.3 was calculated using the Basquin law [72] to determine the stress amplitude for $10^7$ cycles:

$$\sigma_a = \sigma_f'(N_f)^b,$$

where $\sigma_f'$ is the fatigue-strength coefficient given by extrapolation of the S–N curve to the first half-cycle, $b$ is the exponent of the S–N curve, and $N_f$ is the number of cycles until fracture. According to calculations, the stress amplitude at the fatigue limit for both the AISI 316L and AISI 430 at $10^7$ number cycles was approximately 110 MPa, in spite of the significant differences in the maximum work hardening and elongation in the tensile test between the AISI 316L and AISI 430.

Fig. 2.2. Geometry and dimensions (in mm) of the fatigue-test specimen. The cross section of the fatigue specimen was obtained by the transverse cutting from the middle of the specimens. The surface of the cross section (hatched surface) of the fatigue specimen was subjected to EBSD and XRD measurement.
Fig. 2.3. S–N diagram showing the conditions for the fatigue test at which the fatigue test was stopped during cycling for (a) AISI 316L, (b) AISI 430.

### 2.2.3 Microstructural characterization

After the cyclic loading was stopped, the cross section of the samples was obtained by transverse cutting from the middle of the samples (see Fig. 2.2). The surface of the cross section of the samples was characterized using X-ray diffraction (XRD) and EBSD measurements. The surface of the cross section of the fatigued samples was prepared via polishing. Before the XRD measurement, electropolishing was performed at 30 V for 2 min using a solution containing 90% acetic acid and 10% perchloric acid as an electrolyte to remove the mechanically polished layer followed by ultrasonic cleaning in ethyl alcohol [73] [74]. The XRD patterns were measured using a Bragg-Brentano diffractometer (D8 Advance,
Bruker), which can obtain a high flux and has a high resolution. The measurements were performed with CuKα\textsubscript{1} radiation, which was monochromated by a Johansson monochromator, with an operation voltage of 40 kV and a tube current of 40 mA. The maximum of all the peaks was considered 10,000 counts to obtain 1% error [74]. Six diffraction peaks were analyzed for each SS: 111, 200, 311, 222, 400, and 331 for AISI 316L and 110, 200, 211, 220, 310, and 222 for AISI 430. As an example of using the CMWP analysis of obtained XRD pattern, which was explained in the previous chapter, the obtained dislocation parameters for a fatigued AISI 316L sample at $\sigma_a = 250$ MPa and $10^3$ cycles are shown in Fig. 2.4.

![XRD pattern](image)

<table>
<thead>
<tr>
<th>$\rho$ (m$^2$)</th>
<th>$R_e$ (nm)</th>
<th>$M$</th>
<th>$q$</th>
<th>$&lt;X&gt;_{area}$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.1×10$^{14}$</td>
<td>15</td>
<td>0.44</td>
<td>2.21</td>
<td>135</td>
</tr>
</tbody>
</table>

**Fig. 2.4.** CMWP analysis of the XRD patterns of the fatigued AISI 316L at $\sigma_a = 250$ MPa and $10^3$ cycles.

To obtain EBSD images, the electropolished surfaces of the samples were prepared with a colloidal silica slurry 20 nm in size, followed by ion-milling surface preparation. The EBSD measurements were performed to analyze the KAM by using two scanning electron microscopes (SEM Hitachi SU5000 and JEOL JSM-6610LA) equipped with an EDAX OIM system. The measurements were performed on the surface of the cross section of the fatigued samples with the step size of 0.5 µm.
2.2.4 X-ray diffraction line-profile analysis

The convolution multiple whole profile (CMWP) method [75] [76] [77] [78] was employed to analyze the XRD line profiles. The obtained line profiles were fitted using a theoretical profile, which was the result of the convolution of the crystallite size, strain due to dislocations, and instrumental profiles. The Fourier transform equation [78] for the theoretical strain profile is as follows:

\[
I_{\text{strain}} = \int_0^\infty \exp\left\{-2\pi^2 L^2 g^2 \langle \epsilon^2_{g,L} \rangle \right\} \exp(2\pi i L s) dL,
\]

where \( L \) is the Fourier variable, \( g \) is the absolute value of the diffraction vector, and \( \langle \epsilon^2_{g,L} \rangle \) is the mean-square strain. The intensity profile \( s \) can be expressed as

\[
s = \frac{2\sin\theta}{\lambda} - \frac{2\sin\theta_B}{\lambda},
\]

where \( \lambda \) is the wavelength of the X-rays, and \( 2\theta \) and \( 2\theta_B \) are the scattering angle and the exact Bragg position, respectively. For dislocated crystals, the mean-square strain is related to the Wilkens function, \( f\left(\frac{L}{R_e}\right) \) [79]:

\[
\langle \epsilon^2_{g,L} \rangle \cong \frac{\rho b^2}{4\pi} \langle C \rangle f\left(\frac{L}{R_e}\right),
\]

where \( \rho \) and \( b \) are the density and Burgers vector of the dislocations, respectively, and \( \langle C \rangle \) is the average contrast factor. The \( f\left(\frac{L}{R_e}\right) \) function describes the \( L \) dependence of the mean-square strain with \( \frac{L}{R_e} \), where \( R_e \) is the effective outer cutoff radius of the dislocations.

The shape analysis of the XRD peaks indicates the coherently scattering domain size (hereinafter referred to as the crystallite size). The crystallite size given by X-ray DLPA provides the subgrain or cell size bounded by small-angle grain boundaries or dipolar walls [78] [80]. By assuming a lognormal distribution of the crystallite size, one can determine the theoretical size profile [81]:

\[
I_{\text{size}} = \int_0^\infty T \frac{\sin^2(Tm)}{(\sigma^2)^2} \text{erfc}\left(\frac{\log(Tm)}{\sqrt{2}\sigma}\right) dT,
\]

where \( T \) is the crystallite size, and \( \log m \) and \( \sigma \) are the median and variance of the lognormal size distribution, respectively. In the present case, the area-weighted crystallite size (\( \langle X \rangle_{\text{area}} \)) is used [80]:

40
< X >_{area} = m \exp(2.5 \sigma^2), \hspace{1cm} (2-6)

The dimensionless \( M \) value, which describes the dipole character of the dislocation arrangement, is obtained via normalization of \( R_e \) by the average distance \( (1/\sqrt{\rho}) \) between dislocations, \( M = R_e \sqrt{\rho} \). For a strong dipole character, i.e., when \( R_e \) is smaller than the average dislocation distance, \( M \) is smaller than unity \( (M < 1) \). In contrast, when \( R_e \) is larger than the average dislocation distance, \( M \) is larger than unity \( (M > 1) \). Additionally, the peak broadening due to dislocations depends on the relative orientations between their Burgers and line vectors, as well as the diffraction vector, which is taken into account by the dislocation contrast factors \cite{75} \cite{82} \cite{83}. The contrast factors corresponding to one particular \( hkl \) can be averaged over the permutations of these \( hkls \) \cite{84}. In the case of cubic polycrystalline materials, the average dislocation contrast factor is determined using the following equation:

\[
\langle C \rangle = C_{h00} \left[ 1 - q \left( \frac{h^2k^2+k^2l^2+l^2h^2}{h^2+k^2+l^2} \right) \right], \hspace{1cm} (2-7)
\]

where \( C_{h00} \) is the average dislocation contrast factor for \( h00 \)-type reflections, and \( q \) is related to the fraction of the edge/screw character of the dislocations \cite{82} \cite{83} \cite{85}.

### 2.3 Results and discussion

#### 2.3.1 Dislocation evolution versus number of cycles

For quantitative analysis of the dislocation characteristics of the cyclically loaded AISI 316L, DLPA was performed for the full width at half maximum (FWHM) of each XRD peak. Fig. 2.5 shows the change in the FWHMs of all the peaks at \( \sigma_a = 250 \) MPa and different numbers of cycles for the cyclically loaded AISI 316L. There were two stages in the variation of the FWHMs with respect to the number of cycles: an increasing stage and a constant stage.
Fig. 2.5. Changes in the FWHMs of the fatigued AISI 316L with respect to the number of cycles for all six reflections at $\sigma_a = 250$ MPa.

The evolution of the dislocation parameters versus the number of cycles at $\sigma_a = 200$ and 250 MPa is shown in Fig. 2.6. The error bars are related to the fitting errors of the dislocation parameters, which do not consider sampling errors. As the cycling proceeded in the early stage, the dislocation density increased (stage I). The area-weighted crystallite size results show that crystallite size decreased with the increasing number of cycles during stage I. The $M$ parameter remained constant ($M < 1$) during cycling in stage I for both stress amplitudes, indicating that the dislocations were correlated in stage I.

In stage II, for both stress amplitudes (in Fig. 2.6), all the dislocation parameters ($\rho$, $M$, and $<X>_{area}$) remained constant. According to Fig. 2.6 (a), the dislocation density in stage II at 250 MPa was higher than that at 200 MPa. Additionally, a larger stress amplitude led to a smaller crystallite size in stage II, which agrees with previous studies [86] [87]. Further, stage II started at a larger number of cycles at 200 MPa than at 250 MPa.
Fig. 2.6. Evolution of the (a) dislocation density, (b) dislocation arrangement, and (c) area-weighted crystallite size ($<X_{area}>$) for the fatigued AISI 316L with respect to the number of cycles at $\sigma_a = 200$ and 250 MPa. The fitting error bars were calculated using error estimates for the dislocation characteristic evaluations.
Fig. 2.7 shows the EBSD results for fatigued specimens at $\sigma_a = 200$ and 250 MPa at different numbers of cycles during stage II (see Fig. 2.6). The KAM map characterizes the local misorientation gradient induced by GNDs. The KAM maps were compared with the inverse pole figure maps parallel to the loading direction, revealing that there was no relationship between the crystallographic orientation of the grain toward the loading axis and the KAM value. Further, there was no discernible change in the KAM values at different numbers of cycles. This indicates that the GND density remained almost constant at each stress amplitude with the increasing number of cycles in stage II. Moreover, as expected from Fig. 2.7, Fig. 2.8 shows that the distribution of the number fraction of KAM did not vary during cycling, indicating that the GND density did not change. Fig. 2.6 shows that the dislocation density ($\rho_{(GND+SSD)}$) was almost constant in stage II, suggesting that not only the GND but also the SSD density remained constant during stage II.
The dislocation density, dislocation arrangement, and crystallite size of the cyclically loaded AISI 430 at a stress amplitude of 250 MPa and different numbers of cycles are shown in Fig. 2.9. The evolution of the dislocation characteristics with respect to the number of cycles is similar to that for the cyclically loaded AISI 316L. In stage II, the dislocation density,
dislocation arrangement, and area-weighted crystallite size remained constant with increasing cycles, similar to the results for the AISI 316L. In stage I, the dislocation density increased, and the area-weighted crystallite size decreased. The dislocation density in AISI 430 was lower than that in the AISI 316L (Fig. 2.6 (a)) at a stress amplitude of 250 MPa. This is because of the easy cross-slip of the dislocations in AISI 430. Easy cross-slip of dislocations leads to greater annihilation of dislocations and thus a lower dislocation density in AISI 430. Compared with the AISI 316L (Fig. 2.6 (b)) at a stress amplitude of 250 MPa, the $M$ parameter at a small number of cycles in stage I was higher than unity for AISI 430, indicating that the correlations between dislocations was weak. That is, AISI 430 required a larger number of cycles to form strong correlations between dislocations ($M < 1$). These weak correlations between dislocations and the slow decrease in the $M$ parameter are related to the low dislocation density of AISI 430. Additionally, stage II started at a larger number of cycles for AISI 430 than for AISI 316L.

![Graph](image_url)

**Fig. 2.8.** KAM numerical distribution of the fatigued AISI 316L on KAM maps at different stress amplitudes and numbers of cycles during the constant stage of dislocations.
Fig. 2.9. Evolution of the (a) dislocation density, (b) dislocation arrangement, and (c) area-weighted crystallite size of the fatigued AISI 430 with respect to the number of cycles at $\sigma_a = 250$ MPa. The fitting error bars were calculated using error estimates for the dislocation characteristic evaluations.
2.3.2 Change in dislocation parameters with different stress amplitudes

Fig. 2.10 shows the dislocation arrangement and area-weighted crystallite size versus the dislocation density for fracture points of the cyclically loaded AISI 316L and AISI 430 at different stress amplitudes, compared with the case of monotonic tensile loading at different flow stresses. The results of tensile loading for both stainless steels are also shown in Fig. 2.10 [88]. The $M$ parameter of cyclic loading was smaller than that for tensile loading for both SSs. This small $M$ parameter originates from the well-developed dislocation cell walls [59] [56] in cyclically loaded SSs, which are the responsible for the constant stage in the cyclic response. The dislocation cell walls confined the movement of dislocations during stage II [63] [89] (Figs. 2.6 and 2.9), resulting in almost constant dislocation parameters. On the other hand, the area-weighted crystallite size under cyclic loading was comparable to the tensile-loading results. Therefore, it can be concluded that the $M$ parameter is related to the cyclic response of AISI 316L and AISI 430.
Fig. 2.10. Comparison between cyclic loading and monotonic tensile loading [85] for AISI 316L and AISI 430: (a) dislocation arrangement versus dislocation density; (b) area-weighted crystallite size versus dislocation density. The points of cyclic loading are related to the fracture points at different stress amplitudes.

Fig. 2.11 shows the proportion of screw dislocations to total dislocations with respect to the LCF life. The screw-dislocation percentage for AISI 316L was calculated using the $q$ parameter results; the values of $q$ varied between 1.55 (pure edge) and 2.35 (pure screw). These two values were determined according to the elastic constants of AISI 316L [86] [90]. The possible range of the $q$ value for the AISI 430, which was calculated using the elastic constants of body-centered cubic Fe, was between 1.36 (pure edge) and 2.68 (pure screw) [86] [91] [92]. Because of the easy cross-slip of the dislocations in AISI 430 compared with the AISI 316L, the dislocation mobility of AISI 430 was likely to be higher than that of AISI 316L, resulting in greater annihilation of screw dislocations by the cross-slip in AISI 430. The higher frequency of annihilation of screw dislocations at a shorter LCF life for AISI 430 was related to the increased amplitude of the stress applied to the material. On the other hand, for the AISI 316L, the screw-dislocation fraction remained almost constant with the increase in the LCF life, and the increase in the stress amplitude did not lead to the annihilation of screw dislocations.
Fig. 2.11. Proportion of screw dislocations to total dislocations versus the LCF life for the AISI 316L and AISI 430. The fitting error bars were calculated using error estimates for $q$ parameter evaluations.

The dislocation density of the AISI 316L and AISI 430 exhibited a linear correlation with the LCF life in a double-logarithmic graph, as shown in Fig. 2.12 (a). The LCF life decreased with the increase in the dislocation density, and the slopes differed between the AISI 316L and AISI 430. The dislocation density for the AISI 316L was higher than that for the AISI 430, which is related to the differences in the work hardening of the SSs. Fig. 2.12 (b) shows the log–log linear relationship between $\rho/\Delta\sigma_t$ and the LCF life, which is independent of the work hardening of the SS. The maximum tensile work hardening, $\Delta\sigma_t$, is the difference between the true ultimate stress and the true yield stress (see Fig. 2.1). When the dislocation density was normalized by the maximum tensile work hardening, $\Delta\sigma_t$, the effect of the dislocation density on the LCF life was normalized, as shown in Fig. 2.12 (b). It may be assumed that the log–log linear relationship between $\rho/\Delta\sigma_t$ and the LCF life is related to the similar yield strengths of the two SSs. However, very few cycles are needed to start plastic deformation compared with the LCF life (in the present cases, $>10^4$ cycles) [58]; thus, the yield stress cannot affect the LCF life. In AISI 316L, the correlation between dislocations is strong, owing to the high work hardening and high dislocation density. The strong correlation
between dislocations increases the accumulation of dislocations in well-developed dislocation cell walls, which increases the potential sites for crack propagation; thus, the LCF life decreases.

![Graph](image)

**Fig. 2.12.** Log–log relationship between (a) the dislocation density and the LCF life for each SS; (b) \( \rho / \Delta \sigma_{ts} \) and the LCF life, regardless of the material type. \( \Delta \sigma_{ts} \) is the maximum work hardening of the AISI 316L and AISI 430. The fitting error bars were calculated using error estimates for the dislocation density evaluations.
2.4 Summary

(1) There is a log–log linear correlation between the LCF life and the dislocation density normalized by the maximum tensile work hardening for the AISI 316L and AISI 430.

(2) The proportion of screw dislocations in the AISI 430 decreases with the decrease in the LCF life because of the annihilation of screw dislocations due to the easy cross-slip of dislocations. However, in AISI 316L, the dislocation character is hardly affected by the LCF life, owing to the difficulty of the cross-slip of dislocations.

(3) The dislocation arrangement parameter of cyclic loading is smaller than that of tensile loading for AISI 316L and AISI 430. The area-weighted crystallite size of cyclic loading is comparable to that of tensile loading. Thus, the dislocation arrangement parameter is related to the cyclic response for AISI 316L and AISI 430.

(4) According to the X-ray DLPA results, the variation of the dislocation characteristics with respect to the number of cycles exhibits two stages: I) the variation of the dislocation characteristics and II) a constant stage. The dislocation density, dislocation arrangement parameter, and crystallite size do not change in stage II.

(5) The GND density and SSD density remain constant during stage II.

(6) AISI 316L and AISI 430 exhibit the same trend with regard to the changes in the dislocation parameters. Because of the easy cross-slip and the resulting greater annihilation of dislocations in AISI 430, the dislocation density in AISI 430 is lower than that in AISI 316L. A larger number of cycles is needed to form a strong correlation between dislocations in AISI 430 than in AISI 316L.
Chapter 3 Effect of work hardening mechanisms in unstable and stable stainless steels on low-cycle fatigue behavior
3.1 Introduction

During cyclic loading, austenitic stainless steels with compositions in the range where austenite, δ-ferrite and martensite are in equilibrium in the Schaeffler diagram [93] can be expected to be work hardened via the contribution of nucleation of martensite as well as the usual work hardening mechanism by dislocation slip. The work hardening of metastable austenitic stainless steel is controlled by the dislocation density, and by the volume fraction of α’-martensite which mainly generates in cyclic deformation. During high cycle fatigue (HCF), it has been reported that if the transformation was triggered after fatigue crack initiation, the fatigue life was enhanced, while if triggered before initiation, the fatigue life was reduced [57] [94] For the low-cycle fatigue (LCF), it has not been completely understood how to affect strain-induced martensite transformation (SIMT) on the LCF life in different fatigue stress amplitudes. The LCF life is generally considered to be related to the tensile test results via elongation owing to the improvement in the ductility by SIMT phenomenon [95] [96] [97]; however, some studies have shown that the relationship between the LCF life and the tensile elongation is not necessarily strong [57] [98]. On the other hand, it was reported that the plastic deformation and related work hardening mechanisms of the stainless steel including the contribution of SIMT phenomenon to the work hardening likely affect the fatigue life [57]. Therefore, to elucidate how plastic deformation work hardening mechanisms and affect the LCF life, measuring the contributions of austenite phase and SIMT phenomenon to the work hardening via measurement of dislocation density and volume fraction of α’-martensite in a cyclically loaded metastable austenitic stainless steel with different LCF lives is the appropriate approach.

The main objective of this chapter was to determine the effect of work hardening mechanisms on the LCF life and the contributions of austenite phase and SIMT phenomenon to the work hardening by using the X-ray DLPA and EBSD methods. In view of the effect of work hardening mechanisms, a metastable austenitic stainless steel was compared with a stable austenitic stainless steel. Also, the evolution of the dislocation parameters with respect to the LCF life in different stress amplitudes was studied for both stainless steels.
3.2 Experimental

3.2.1 Materials

The materials under study in this chapter were AISI 316L and AISI 304. The Chemical composition, \(M_{d30}\) temperatures and SFEs of stainless steels under study are shown in Table 3.1.

**Table 3.1.** Chemical composition (mass%), \(M_{d30}\) temperatures, and SFEs of the materials.

<table>
<thead>
<tr>
<th></th>
<th>Chemical composition (mass%)</th>
<th>(M_{d30}) (°C)</th>
<th>SFE (mJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>C 0.017, Si 0.69, Mn 1.22, Ni 12.08, Cr 17.48, Mo 2.05, Cu 0.31, N 0.18, Fe Bal.</td>
<td>-86.4</td>
<td>34.5</td>
</tr>
<tr>
<td>304</td>
<td>C 0.038, Si 0.51, Mn 0.91, Ni 8.1, Cr 18.28, Mo 0.25, Cu 0.33, N 0.04, Fe Bal.</td>
<td>32.9</td>
<td>23.5</td>
</tr>
</tbody>
</table>

The susceptibility to the formation of deformation induced \(\alpha'\)-martensite is mainly influenced by the chemical composition [99] [100]. The following equations [99] [101] for calculating \(M_{d30}\) temperature threshold and stacking fault energies (SFEs) were used:

\[
M_{d30} (°C) = 413 - 462(C+N) - 9.5Ni - 13.7Cr - 8.1Mn - 18.5Mo - 9.2Si, \tag{3-1}
\]

\[
SFE (mJ/m^2) = -7.1 + 2.8Ni + 0.49Cr + 2.0Mo - 2.0Si + 0.75Mn - 5.7C - 24N, \tag{3-2}
\]

At \(M_{d30}\) temperature, by definition, 50\% \(\alpha'\)-martensite is formed as a result of 30\% deformation [99] [101]. These equations were used, because the reported chemical compositions of the austenitic stainless steels [99] [101] were similar to the chemical composition of the austenitic stainless steels under investigation. Table 3.1. The calculated SFE for AISI 316L is higher than that for AISI 304. The calculated \(M_{d30}\) for AISI 316L is significantly lower than that for AISI 304, meaning the high possibility of martensite phase transformation at the room temperature.

Fig. 3.1 shows tensile stress-strain curves of stainless steels, tested at strain rate of 7.16\times10^{-4} s\(^{-1}\) [71] [102] at ambient temperature. The tensile elongations of AISI 304 and AISI 316L were almost comparable. It should be noted that AISI 304 showed a higher maximum work hardening than AISI 316L under the tensile loading in spite of the lower yield point of AISI 304.
Fig. 3.1. True stress–strain curves from the monotonic tensile test for AISI 316L and AISI 304.

3.2.2 Fatigue test

Fig. 3.2 shows the geometry of the fatigue specimen used for the fatigue tests. The fatigue tests were performed with $\frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} = 0.1$, at a frequency of 10 Hz. Two alloys were tested under cyclic loading with different numbers of cycles and stress amplitudes ($\sigma_a = \frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{2}$), as shown in Fig. 3.3. To study the effect of the number of cycles on the fatigued stainless steels, the fatigue testing was stopped at different numbers of cycles just before fracture (Fig. 3.3). For all the stress amplitudes, the maximum stresses for AISI 304 and AISI 316L were larger than 300 MPa and 400 MPa, respectively.

Fig. 3.2. Geometry and dimensions (in mm) of the fatigue-test specimen. The cross section of the fatigue specimen was obtained by the transverse cutting from the middle of the specimens. The surface
of the cross section (hatched surface) of the fatigue specimen was subjected to EBSD and XRD measurement.

![Fig. 3.3](image-url)  
**Fig. 3.3.** S–N diagram showing the conditions for fatigue fracture points and stopped points just before fracture for AISI 316L and AISI 304.

### 3.2.3 Microstructural characterization

After the cyclic loading was stopped, the cross section of the samples was obtained by transverse cutting from the middle of the samples (see Fig. 3.2). The surface of the cross section of the samples was characterized using XRD and EBSD measurements. The surface of the cross section of the fatigued samples was prepared via polishing. Before the XRD measurement, electropolishing was performed at 30 V for 2 min using a solution containing 90% acetic acid and 10% perchloric acid as an electrolyte to remove the mechanically polished layer followed by ultrasonic cleaning in ethyl alcohol [73] [74]. The XRD patterns were measured using a Bragg-Brentano diffractometer (D8 Advance, Bruker), which can obtain a high flux and has a high resolution. The measurements were performed with CuKα₁ radiation, which was monochromated by a Johansson monochromator, with an operation voltage of 40 kV and a tube current of 40 mA [74]. The maximum of all the peaks was considered 10,000 counts to obtain 1% error. Six diffraction peaks were analyzed for AISI 316L: 111, 200, 311,
222, 400, and 331. Nine diffraction peaks of AISI 304 were analyzed: 111, 200, 311, 222 and 400 for γ phase, and 110, 211, 220 and 310 for α′-martensite phase. As an example of using the CMWP analysis of XRD pattern, which was explained in the previous chapter (section 2.2.4), the obtained dislocation parameters for a fatigued AISI 316L sample at $\sigma_a = 375$ MPa and $4.1\times10^3$ LCF life are shown in Fig. 3.4.

![CMWP analysis of XRD patterns](image)

<table>
<thead>
<tr>
<th>Phase</th>
<th>$\rho$ (m$^2$)</th>
<th>$R_e$ (nm)</th>
<th>$M$</th>
<th>$&lt;X&gt;_{area}$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>γ - austenite</td>
<td>$4.8\times10^{15}$</td>
<td>2.53</td>
<td>0.17</td>
<td>68.8</td>
</tr>
<tr>
<td>α′ - martensite</td>
<td>$5.1\times10^{15}$</td>
<td>2.09</td>
<td>0.15</td>
<td>70.1</td>
</tr>
</tbody>
</table>

**Fig. 3.4.** CMWP analysis of the XRD patterns of the fatigued AISI 304 at $\sigma_a = 375$ MPa and $4.1\times10^3$ LCF life.

To obtain EBSD images, the electropolished surfaces of the samples were prepared with a colloidal silica slurry 20 nm in size, followed by ion-milling surface preparation. The EBSD measurements were performed to analyze the KAM by using two scanning electron microscopes (SU5000 JSM-6610LA) equipped with an EDAX OIM system. The measurements were performed on the surface of the cross section of the fatigued samples with the step size of 0.2 $\mu$m.
3.3 Results and discussion

Fig. 3.5 shows EBSD phase maps for fatigued AISI 304 at different LCF lives. The $\alpha'$-martensite phase is shown in green, and the austenite phase is shown in red. It can be seen that there is a small fraction of martensite at $N_f = 4.8 \times 10^4$. However, at $N_f = 1.8 \times 10^4$, there is a higher fraction of $\alpha'$-martensite in the microstructure. With decreasing the LCF life, the fraction of martensite was found to be a higher value.

Fig. 3.5. EBSD phase maps for cyclically loaded AISI 304 at different LCF lives ($N_f$); (a) $4.8 \times 10^4$, (b) $1.8 \times 10^4$, (c) $1.1 \times 10^4$ and $4.1 \times 10^3$. The loading direction was perpendicular to the observed surfaces. Martensite is shown in green, and austenite is shown in red.

Changes in the full width at half maximums (FWHMs) of 200 peak of $\gamma$ phase and 110 peak of $\alpha'$ phase with respect to LCF life were shown in Fig. 3.6 (a). The results indicate the increasing trend of FWHM of 200$_{\gamma}$ with decreasing the LCF life for AISI 304 and AISI 316L,
which means the broadening occurred. In the lower LCF lives for both stainless steels, the increments of FWHM of $200\gamma$ were small values. The FWHMs of $200\gamma$ in AISI 304 were higher than those in AISI 316L, showing higher differences between FWHMs of AISI 304 and AISI 316L in lower LCF lives than an LCF life. Since FWHMs of $110\alpha'$ showed that $\alpha'$ phase slightly deformed compared with $\gamma$ phase, the strain and dislocation induced by the cyclic loading are mainly stored in the $\gamma$ phase. Here, the diffraction peak corresponding to the $\alpha'$ phase was not reported at stress lower than 200 MPa, which is related to weak 110 peak. The dislocation density of $\gamma$ phase increased with decreasing the LCF life in AISI 304 and AISI 316L, Fig. 3.6 (b), presenting slight increases in dislocation density of both alloys lower than an LCF life. Dislocation densities of $\gamma$ phase of AISI 304 in lower LCF lives than an LCF life were significantly higher than those of AISI 316L where higher $\alpha'$-martensite fraction was observed. Fig. 3.7 presents kernel average misorientation (KAM) map of fatigued AISI 304 in different LCF lives. The KAM map characterizes the local misorientation gradient induced by GNDs. With decreasing LCF life, the KAM value increased and the heterogeneity of misorientation distribution in austenite phase also increased which is consistent with the result reported by Schayes et al. [66]. Also, the higher dislocation density accompanied by higher $\alpha'$-martensite fraction in AISI 304 is because of accommodating a higher plastic deformation predominantly in $\gamma$ phase surrounding $\alpha'$-martensite. This is consistent with the result reported by Miyamoto et al. [103]. These higher values of dislocation density in shorter LCF lives in AISI 304 were related to the increased amplitude of the stress applied to the materials. There is also a correlation between fatigue stress amplitude results and dislocation densities of $\gamma$ phase. This does not agree with the previous results in sec. 3.1, showing a negative effect which can be considered on LCF life for a stable austenitic stainless steel with a higher work hardening. Therefore, another contribution can be supposed in this improved fatigue behavior, namely the contribution of $\alpha'$-martensite to the work hardening.
Fig. 3.6. (a) Comparison of $\gamma$ and $\alpha'$ phases by using FWHM of 200 and 110 reflections with respect to the LCF life; evolution of (b) dislocation density in austenite phase, (c) dislocation arrangement parameter (M) in austenite phase and (d) crystallite size in austenite phase with respect to LCF life for AISI 304 and AISI 316L. The fitting error bars were calculated using error estimates for the dislocation characteristic evaluations.
Fig. 3.7. Kernel average misorientation map showing distribution of misorientation in austenite phase; (a) \(4.8 \times 10^4\) and (b) \(1.1 \times 10^4\). The loading direction was perpendicular to the observed surfaces.

Fig. 3.6 (c) shows \(M\) parameter of AISI 316L is higher than that of AISI 304. There were larger \(M\) parameters than unity for both alloys in the high LCF lives. In the lower LCF lives, \(M\) parameters are smaller than unity for both stainless steels. This is consistent with the observation of dislocation substructure reported in refs. [56] [103]. The small \(M\) parameter originates from the well-developed dislocation cell walls [56] [62] [63] in a cyclically loaded stainless steel, which are responsible for the constant stage in the cyclic response according to the result in sec. 3.1 [74]. The dislocation cell walls confined the movement of dislocations, resulting in almost constant dislocation parameters during cycling [62] [63] [68]. The smaller \(M\) parameter of AISI 304 implies that there is stronger correlation between dislocations in AISI 304 compared with AISI 316L.

The change in crystallite size with respect to the LCF life, Fig. 3.6 (d), showed the smaller crystallite size for AISI 304 compared with AISI 316L which usually indicates higher local stress and dislocation density [104], therefore, there is a correlation between dislocation density and crystallite size. This correlation also was reported in chapter 2. However, in the
In the present case, crystallite size and dislocation density in the high LCF lives are not correlated where there were smaller crystallite sizes for AISI 304 than AISI 316L in spite of almost comparable dislocation density. The differences between crystallite sizes of AISI 304 and AISI 316L were higher in the lower LCF lives. Since the calculated SFE for AISI 316L is higher than that for AISI 304 (see Table 2.1), the effect of stacking fault can be considered on the crystallite size of fatigued AISI 304 and AISI 316L. The stacking fault probability ($\alpha$) is expressed by the diffraction angle ($2\theta$) and the following equation in angular dispersive diffraction [105]:

$$\Delta(2\theta_{200} - 2\theta_{111})^\circ = -6.2\alpha,$$

(3-3)

where $2\theta_{200}$ and $2\theta_{111}$ are the gravity centers of XRD peaks. The deformation stacking fault probabilities for the fatigued specimens were calculated from the peak shift analysis taking the corresponding annealed samples as standard. Fig. 3.8 shows changes in the stacking fault probability ($\alpha$) in different LCF lives for AISI 304 and AISI 316L, indicating that the stacking fault probability for AISI 304 is higher than that for AISI 316L. The stacking fault probability results show that higher probability of stacking fault in AISI 304 than AISI 316L has a contribution to smaller crystallite size for AISI 304 than that for AISI 316L.

![Fig. 3.8. Stacking fault probability of austenitic phase for AISI 304 and AISI 316L.](image)

The relationship between obtained dislocation densities and work hardening can be explained by the Bailey- Hirsch equation [106]:

$$\text{Equation}$$
\[ \sigma_y = \sigma_0 + T\alpha Gb \sqrt{\rho}, \]  
(3-4)

where \( \sigma_0, T, \) and \( G \) are the friction stress, Taylor factor, and shear modulus, respectively. \( \alpha \) is a geometrical factor that depends on the type and arrangement of the interacting dislocations. Based on the dislocation density \( (\rho_0) \) and yield stress of lowest fatigue stress amplitude \( (\sigma_a = 150 \text{ MPa}) \) in the range of LCF life, the work hardening amount \( (\Delta \sigma) \) can be expressed as follows.

\[ \Delta \sigma = T\alpha Gb \sqrt{\rho} - \sqrt{\rho_0}, \]  
(3-5)

In the present work, \( T \) is assumed to be 3. The shear modulus of AISI 304 is considered to be 79 GPa [107]. The following equation presents the relationship between total work hardening \((\Delta \sigma)\) of AISI 304 containing two phases with the work hardening of each phase \((\Delta \sigma_\gamma \text{ or } \Delta \sigma_{\alpha'})\).

\[ \Delta \sigma = X_\gamma \Delta \sigma_\gamma + X_{\alpha'} \Delta \sigma_{\alpha'}, \]  
(3-6)

where \( X_\gamma \) and \( X_{\alpha'} \) are volume fractions of \( \gamma \) and \( \alpha' \) phases. Fig. 3.9 (a) shows the relationship between yield stress of fatigued specimens and LCF life of AISI 304 and AISI 316L. The yield stresses of the fatigued specimens were calculated by using measured Vickers microhardness (HV0.5kgf) and LCF life via three-time relationship \( (\sigma_y = \frac{HV}{3}) \) [108] [109] [110] [111]. These yield stresses of fatigued specimens can be considered as criteria for considering the effect of work hardening on the LCF life by subtracting yield stress of the lowest fatigue stress amplitude in the range of LCF life from the yield stresses. In the lower LCF lives, the yield stress of fatigued AISI 304 was higher than that of fatigued 316L, showing a correlation with the results of dislocation densities as presented in Fig. 3.6 (b). Also, the difference between yield stresses of two stainless steels increases with decreasing the LCF life, as shown in Fig. 3.9 (a).

For calculation of contribution of austenite phase to the work hardening in AISI 304, \( \alpha \)-factor is calculated based on the comparing the results of yield stress obtained by hardness with using eq. (3-5) for AISI 316L in the high and low LCF life. Fig. 3.9 (b) shows the contributions of \( \alpha' \) and \( \gamma \) phases to the work hardening \((X_\gamma \Delta \sigma_\gamma \text{ or } X_{\alpha'} \Delta \sigma_{\alpha'})\) of fatigued AISI 304 compared with the work hardening of AISI 316L. The contribution of \( \alpha' \) phase to the work hardening was calculated by subtracting the \( X_\gamma \Delta \sigma_\gamma \) term from the work hardening calculated based on microhardness results. The \( X_\gamma \Delta \sigma_\gamma \) term of work hardening of AISI 316L calculated based on the yield stresses values resultant from microhardness results. The
difference between work hardenings of austenite phase in AISI 304 and AISI 316L in the higher LCF lives is larger than the differences between dislocation densities of austenite phase in AISI 304 and AISI 316L. This difference is related to the using microhardness result for calculation of work hardening in AISI 316L. In the lower LCF lives, the contribution of $\alpha'$ phase to the work hardening of fatigued AISI 304 increased with decreasing the LCF life, while the contribution of $\gamma$ phase slightly changed. It can be suggested that the work hardening comprises contributions of $\alpha'$ and $\gamma$ phases to the work hardening of AISI 304, showing a correlation with the LCF life. The contribution of $\alpha'$-martensite phase to work hardening was maximized in the lower LCF lives for AISI 304. Thus, the higher work hardenings of AISI 304 than those of AISI 316L in the lower LCF lives were related to increasing the contribution of $\alpha'$-martensite phase to the work hardening. This higher work hardening in the lower LCF lives in AISI 304 had a good correlation with the fatigue stress amplitude results which was shown in Fig. 3.3. The contribution of $\alpha'$-martensite phase to the work hardening led to the same LCF life for AISI 304 and AISI 316L when the fatigue stress amplitude for AISI 304 was considerably higher than that for AISI 316L. This implies that the higher work hardening induced by SIMT phenomenon can improve LCF behavior of AISI 304 compared with AISI 316L in the lower LCF life via increasing the fatigue stress amplitude. On the other hand, in the high LCF life when the $\alpha'$-martensite had a small contribution to the work hardening, the fatigue stress amplitudes of AISI 304 were slightly higher than those of AISI 316L. This is in good agreement with several reports showing a slight improvement in LCF life in steels containing SIMT phenomenon at low stress/strain amplitudes (in the range of LCF), with explanations that suggest a likely influence of additional strain accommodation via a slight increase in work hardening [112] [113].
The austenite in AISI 304 steel is metastable, and the system can lower its energy by transformation to martensite. The difference in energy between the austenite and martensite, $\Delta G$, is the energy required to nucleate martensite. For the present study, the application of stress provides an additional mechanical driving force for martensite formation. The critical mechanical driving force ($\Delta G_{mech}$) must be met for the transformation from austenite to martensite to be possible. The monotonic testing results showed a higher monotonic work hardening for AISI 304 compared with AISI 316L, which is consistent with the result reported by Sugimoto et al. [114], showing that the SIMT effect is markedly improved monotonic work hardening. The SIMT effect also improves LCF behavior where there is progressive

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**Fig. 3.9.** (a) Yield stress ($\sigma_y$) of fatigued stainless steels calculated based on measured Vickers hardness versus LCF life and (b) each term of eq. (3-6) versus LCF life.
transformation during loading such as in high stress amplitudes, as occurs during monotonic deformation [57] [114]. We also note here that from a plasticity perspective, the SIMT effect is always competing with dislocation slip, and in all cases dislocation motion will be preferred if it requires the smaller applied stress until the $\Delta G_{mech}$ can be provided [57]. Since in high fatigue stress amplitudes, the $\Delta G_{mech}$ for martensite transformation can be met more easily during cycling, the possibility of progressive transformation during cycling increased; thus, the contribution of $\alpha'$-martensite phase to the work hardening increased. This progressive transformation leads to similar work hardening for high fatigue stress amplitude and monotonic deformation. However, in the high LCF lives (lower stress amplitudes), this critical mechanical driving force ($\Delta G_{mech}$) cannot be easily met. Therefore, in high fatigue stress amplitudes, SIMT effect led to improve the LCF behavior of AISI 304 significantly (higher fatigue stress amplitude for AISI 304) compared with AISI 316L owing to additional strain accommodation via increasing the contribution of SIMT phenomenon to the work hardening.

### 3.4 Summary

The low-cycle fatigue (LCF) behavior of a metastable and a stable stainless steel has been studied. The main results are summarized as follows:

1. In spite of almost comparable tensile elongations in AISI 304 and AISI 316L, the fatigue stress amplitude for AISI 304 was higher than that for AISI 316L in the almost same LCF life.

2. The dislocation density of $\gamma$ phase in AISI 304 was higher than that in AISI 316L. The difference between dislocation densities of AISI 304 and AISI 316L increased in the lower LCF lives.

3. In the lower LCF lives, the contribution of $\alpha'$ phase to the work hardening of fatigued AISI 304 increased with decreasing the LCF life, while the contribution of $\gamma$ phase to the work hardening slightly changed.

4. The higher work hardening induced by SIMT phenomenon improved LCF behavior of AISI 304 compared with AISI 316L in the lower LCF life. This improved behavior was shown in increasing the fatigue stress amplitude while the LCF lives of both stainless steels are same.

5. In high fatigue stress amplitudes (lower LCF lives), the mechanical driving force for martensite transformation can be met more easily during cycling and therefore, the possibility of progressive transformation and contribution of $\alpha'$-martensite phase to the work hardening
during cycling increased. This progressive transformation leads to similar work hardening for high fatigue stress amplitude and monotonic deformation. In the high LCF lives (lower stress amplitudes), this critical mechanical driving force cannot be easily met.
Chapter 4 Conclusions
4.1 Conclusions

We characterized the dislocations quantitatively, that effectively play role in LCF behavior of stainless steels. It was found that X-ray DLPA is one of the most promising techniques for characterizing dislocations. As results of X-ray DLPA, the evolution of dislocation density, dislocation arrangement and crystallite size versus number of cycles was elucidated. It was shown that there are two stages for evolution of mentioned dislocation parameters during cycling: 1- hardening; 2- stabilization of dislocation parameters. The dislocation density, dislocation arrangement parameter, and crystallite size do not change in stage II. By using combination of X-ray DLPA and EBSD, it was found that the GND density and SSD density remain constant during stage II. AISI 316L and AISI 430 exhibit the same trend with regard to the changes in the dislocation parameters. Because of the easy cross-slip and the resulting greater annihilation of dislocations in AISI 430, the dislocation density in AISI 430 is lower than that in AISI 316L. A larger number of cycles is needed to form a strong correlation between dislocations in AISI 430 than in AISI 316L. There is a log–log linear correlation between the LCF life and the dislocation density normalized by the maximum tensile work hardening for the AISI 316L and AISI 430. The proportion of screw dislocations in the AISI 430 decreases with the decrease in the LCF life because of the annihilation of screw dislocations due to the easy cross-slip of dislocations. However, in AISI 316L, the dislocation character is hardly affected by the LCF life, owing to the difficulty of the cross-slip of dislocations. The dislocation arrangement parameter of cyclic loading is smaller than that of tensile loading for AISI 316L and AISI 430. The area-weighted crystallite size of cyclic loading is comparable to that of tensile loading. Thus, the dislocation arrangement parameter is related to the cyclic responses of AISI 316L and AISI 430.

The LCF behaviors of a stable and a metastable austenitic stainless steel were studied via EBSD and X-ray DLPA. In spite of almost comparable tensile elongations in AISI 304 and AISI 316L, the fatigue stress amplitude for AISI 304 was higher than that for AISI 316L in the almost same LCF life. The dislocation density of γ phase in AISI 304 was higher than that in AISI 316L. By studying the effect of SIMT phenomenon on LCF life and its related work hardening, it was found that the higher work hardening induced by SIMT phenomenon improved LCF behavior of AISI 304 compared with AISI 316L in the lower LCF life. This improved behavior was shown in increasing the fatigue stress amplitude while the LCF lives of both stainless steels are same. In high fatigue stress amplitudes (lower LCF lives), the mechanical driving force for martensite transformation can be met more easily during cycling.
and therefore, the possibility of progressive transformation and contribution of $\alpha'$-martensite phase to the work hardening during cycling increased. This progressive transformation leads to similar work hardening for high fatigue stress amplitude and monotonic deformation. In the high LCF lives (lower stress amplitudes), this critical mechanical driving force cannot be easily met. The difference between dislocation densities of AISI 304 and AISI 316L increased in the lower LCF lives where AISI 304 showed a higher fatigue stress amplitude than that for AISI 316L. This improvement in fatigue life while there is higher work hardening does not agree with the result reported in the first comparison. By considering the contributions of each phase in AISI 304 on the work hardening, it was found that the contribution of $\alpha'$ phase to the work hardening of fatigued AISI 304 increased with decreasing the LCF life in the lower LCF lives, while the contribution of $\gamma$ phase to the work hardening slightly changed. The different mechanism of work hardening, namely SIMT phenomenon, is the main reason of different effect of work hardening in AISI 304.
References


[32] S. Heino and B. Karlsson, "Cyclic deformation and fatigue behaviour of 7Mo0.5N superaustenitic stainless steel-slip characteristics and


