Evaluation of Dislocation Structure in Tensile and Fatigue Deformed Steels by Magnetic Measurement

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Magnetic properties are compared with total dislocation density that considered the dislocation structure change comprehensively in tensile and fatigue deformed commercial steels. Coercive field increases little with the fatigue cycles and seems to be saturated in the middle and last stages of metal fatigue. The coefficient of magnetic susceptibility \( c \) increases over all stages of the fatigue tests and is sensitive to degradation in comparison with \( H_C \). These properties are influenced from total dislocation density, and independent on deformation way. The magnetic-structure-sensitive properties would be useful for evaluation of dislocation structure change, and applied nondestructive evaluation method with development of the correct \( B–H \) curve measurement way.

KEY WORDS: fatigue; dislocation density; coercive field; magnetic susceptibility; nondestructive evaluation.

1. Introduction

Steel is the most important material for constructions and machines because mechanical properties of steels can be changed with relatively little effort by heat treatments, alloy elements addition and processing. Steel structures are designed in a cautious manner with the factor of safety to obviate destruction by overload applied. However, fatigue breakdown is happened occasionally at a stress concentration zone.

Fatigue damage accumulates in steel structures under long-time operation. Low-cycle fatigue at a stress concentration zone is the major reason for fatigue breaking. It is very important to evaluate the accumulated fatigue damage in steel structures for vouching for its safety. The nondestructive evaluation methods of fatigue damage have been turned into actual utilization, for example an ultra sonic and an eddy current method. These methods give the information about the nucleation and the development of microcracks.

Another evaluation method, the magnetic measurement evaluates indirectly the microstructure change. Magnetic properties are received a great amount of influence from a microstructure of ferromagnetic material. It is possible to evaluate the accumulated fatigue damage by magnetic measurement nondestructively. An example of nondestructive evaluation method is the direct measurement in Charpy impact test pieces. There is some problem in nondestructive evaluation method using magnetic measurement, which is, for example, the influence of leakage magnetic field.

The influence of microstructure change on magnetic properties have been studied by many researchers. The magnetic parameter, coercive field arises from magnetic domain wall motion. And the other parameter, the coefficient of magnetic susceptibility \( c \) arises from the magnetic moment rotation. Takahashi and others have been reported the coefficient \( c \) at low magnetic field is sensitive to the microstructure change due to tensile deformation in iron and a low alloy steel.

The relationship between the coefficient \( c \) and the density of homogeneously distributed dislocations in tensile deformed iron and a low alloy steel, has been reported in Ref. 5. In general known, the microstructure change in the fatigue deformed material is different from the tensile deformed one. Due to fatigue deformation, the cellular dislocation structure is formed without dislocation density increasing. It is necessary for evaluating the fatigue damage to clarify the influence of the cellular dislocation structure forming on magnetic properties.

The purpose of the present study is to clarify the dependence of the magnetic properties, coercive field and the coefficient \( c \) on microstructure change due to tensile and low-cycle fatigue deformation by the direct observation.

2. Relationship between Dislocation Density and Magnetic Properties

In ferromagnetic metals, domain walls are pinned by lattice defects. Coercive field is the critical magnetic field as domain walls getting into motion. The relationship between coercive field and pinning force against on domain wall motion is written as

\[
H_C = \frac{1}{2M_s F \cos \phi} (\Sigma_{\text{wall}}) \quad \text{...............(1)}
\]
$M_s$ is a saturation magnetization, $F$ is an area of domain wall at unit volume, $\varphi$ is an angle between magnetic field direction and domain wall, $\Sigma$ is a resultant pinning force on domain wall. The pinning force on domain wall is generated by lattice defects that have stress field around itself. Dislocation has long-range stress field, and acts as a pinning site against domain wall displacement. Thus, coercive field is increased due to increase dislocation density.\(^1\)\(^2\) The relation between coercive field $H_c$ and dislocation density $\rho$ is given by $H_c \propto \rho^{1/2}$. Another lattice defects, grain boundary and precipitation also act pining sites, too.

In addition, dislocations bring a local anisotropy. Magnetic moments around dislocation are deviated from magnetic field direction through magneto-elastic coupling. When magnetization process proceeds by mainly magnetic moments rotation, magnetic susceptibility $\chi$ is a parameter of local anisotropy around dislocation, and given by

$$\chi = \frac{c}{H^3} \quad \quad (2)^{1,2}$$

$H$ is an external field. The coefficient $c$ indicates a state of internal stress, and has the dependence on dislocation density. Kronmüller and others have reported that the coefficient $c$ at near saturation above 1 000 Oe is proportional to shear stress $\tau$ in tensile deformed Ni, Co and Ni-20%Co single crystals at work hardening stage II.\(^3\)\(^4\) From this result, the $c \propto \rho$ is derived. In addition, the relation $c \propto \rho$ is valid at low magnetic field near 100 Oe in iron and low alloy steel.\(^5\)

### 3. Experimental Procedures

Test materials are two kinds of low alloy steels, JIS SFVQ-1A steel for tensile deformation, and JIS SM490YA steel for fatigue deformation. SFVQ-1A steel is usually used in pressure vessels. SM490YA steel is used for welded structures. The specimen of SM490YA steel was toughened up fatigue strength by shot peening similar as a practical material. The chemical compositions of materials are shown in Table 1. These two materials have different grain size, which are 10 $\mu$m for SFVQ-1A steel and 15 $\mu$m for SM490YA steel. Sheets of materials were cut out by electric discharge method into deformation test specimens as shown in Fig. 1. Tensile and fatigue deformation were carried out at room temperature. Tensile deformation was carried out with a strain rate of 0.2%/min. Fatigue deformation is under tension stress with frequency of 5 Hz. Mechanical property change due to fatigue deformation was measured by the Vickers hardness test. Magnetic measurement and dislocation structure observation were carried out after deformation. Deformed specimens were cut out parallel to the stress-applying axis into doughnut-shape for magnetic measurement and disk-shape for dislocation density observation. The doughnut-shape specimen was of 10 mm outside diameter and 5 mm inside diameter for tensile deformed specimens, 15 mm and 7 mm for fatigue deformed specimens. The excitation and search coil were wound on the doughnut-shape specimens. Magnetization curve was measured by the magnetic fluxmeter at room temperature. The disk-shape specimens of both steels were polished mechanically down to 40 $\mu$m, and electro polished with 10% perchloric acid and 90% acetic acid mixed electrolyte. Dislocation structure was observed by TEM at 300 kV acceleration voltage.

### 4. Results and Discussions

#### 4.1. Magnetic Properties for Tensile and Fatigue Deformed Steels

SFVQ-1A steel was tensile deformed differently unto several stresses between the yield stress of 450 MPa and tensile strength of 580 MPa. $B$–$H$ curves for as received and tensile deformed specimens were measured. The magnetic properties, coercive field and the differential magnetic susceptibility are derived from $B$–$H$ curves. The coefficient is calculated from the differential susceptibility according to Eq. (2) within a limited magnetic field region. This equation is not valid under 40 Oe and over 100 Oe.\(^5\) The changes of magnetic properties due to tensile deformation are shown in Fig. 2. Coercive field is increased linearly with increasing true stress, and this relation is equal to the case of other ferromagnetic metals.\(^1\)\(^4\) The coefficient $c$ is increased nonlinearly, the dependence of the magnetic properties on true stress, which can be written as

$$H_c \propto \sigma, \quad c \propto \sigma^2$$

Secondly, the change of magnetic properties due to fatigue deformation is discussed. Fatigued material was SM490YA steel, which has 460 MPa yield stress and 570 MPa tensile strength. The stress amplitude of low-cycle fatigue deformation was 472 MPa. The fatigue life $N_f$ was $10^7$ cycles at

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#### Table 1. The chemical compositions of SFVQ-1A steel and SM490YA steel.

| Elements (wt. %) | C | Si | Mn | P | S | Ni | Mo | Cr | Fe |
|-----------------|---|----|----|---|---|----|----|----|----|
| SFVQ-1A steel   | 0.18 | 0.18 | 1.50 | 0.004 | <0.001 | 0.89 | 0.12 | 0.12 | bal. |
| SM490YA steel   | 0.17 | 0.35 | 1.44 | 0.015 | 0.03 | - | - | - | bal. |
this stress amplitude from $S-N$ line as shown in Fig. 3. Applied cycles on specimens were between from 0.2 to 0.9 the ratio $N/N_f$. Fatigued specimens were hardened as shown in Fig. 4. At the initial stage of fatigue $N/N_f \leq 0.2$, specimen was hardened rapidly, and then, the hardness seemed to be saturated or increased slightly at the middle and final stages.

The magnetic parameters for fatigue deformed specimens are shown in Fig. 5. The dependence of coercive field on fatigue cycle was similar to the hardness. The increasing amount of coercive field at the initial stage was 90% of the total increase. On the other hand, the dependence of the coefficient $c$ on fatigue cycle was different from one of the hardness. The coefficient $c$ was rapidly increased at the initial stage of fatigue similar to the hardness, and then, at the middle and final stage, clearly increased. The dependence of magnetic parameter would originate dislocation structure change.

4.2. Microstructure in Tensile and Fatigue Deformed Steels

Dislocation structures of tensile and fatigue deformed specimens are shown in Fig. 6. In as received specimens of both steels, dislocations distributed homogeneously in a wide area (Figs. 6(a), 6(b)). As received SFVQ-1A steel has been cellular structure. In tensile deformed specimens, dislocation tangling, cellular structure forming, and dislocation increasing in cell interior were observed (Figs. 6(b), 6(c)). The average diameter of cell blocks was 0.5 μm. The cellular structure forming was observed in the fatigue deformed specimens, too. At the initial stage of fatigue ($N/N_f = 0.2$), cell walls were formed and dislocation was increased in the cell interior. The dislocation structure at the middle and final stage of fatigue was changed little (Figs. 6(e), 6(f)). The shape of cell block was ellipsoid, 1 mm length and 0.5 mm width. (In many cases of metal fatigue, cell wall and cell block are called as sub-boundary and sub-grain respectively.) There are two types of dislocations in deformed specimens, homogeneously distributed and cell wall structuring dislocations. To discuss quantitatively the influence of the dislocation structure change on the magnetic properties, it is necessary to consider the influence of the cell wall.

The simple analysis is attempted to clarify the influence of the cell wall in this study. The analysis is the comparison between magnetic parameters and total dislocation density. The total dislocation density is calculated from the dislocation density within cell interior, $\rho_{in}$, at cell wall, $\rho_{nw}$, and the
area fraction of cell wall, $f_w$ with the equation of
\[ r_w = (1 - f_w) r_{in} + f_w r_w \]........................................(3)

The dislocation density in the cell interior $r_{in}$ is measured by the Keh method. This method gives the dislocation density from the number of intersections of dislocation lines and grid lines, according to the equation of
\[ r_{in} = \frac{n_h}{l_h + n_v / l_v} \].........................................(4)

where $n$ is the number of intersections, $l$ is the length of mesh and $t$ is the thickness of sample. The grid lines are ten horizontal and vertical lines. The thickness of sample is 200 nm from equi-thick fringes. $r_{in}$ is calculated from the total length at a unit area and the average thickness of cell walls.

\[ \theta = h_{edge}^2 + 2h_{screw}^2, \quad h = \frac{b}{2 \tan(\theta/2)} \]........................................(5)

$\theta$ is the angle of crystal orientation between neighboring cell blocks. Tilt and twist angle are generated by an alignment of edge dislocations and a pair of alignment of screw dislocations. The angle between neighbor cells is measured with the Kikuchi map. A measured example is represented in Fig. 7. There are two cell blocks separated by the cell wall at the center of this figure. The measuring positions of the Kikuchi map are indicated by dots. The electron beam diameter is as same as the diameter of dots, which is 50 nm. $f_w$ is calculated from the total length at a unit area and the average thickness of cell walls.

\[ \rho_{in}, \rho_w, \text{ and } f_w \] was measured for 20 pictures taken from one TEM sample. These dislocation parameters are shown in Fig. 8. The measured values included measurement error that originated from variation of measuring value across microscopic field. The measurement error was large in the tensile deformed specimen because it is difficult to count tangling dislocations individually. $\rho_{in}$, $\rho_w$, and $f_w$ were increased with increasing true stress in the tensile deformed specimens (Fig. 8(a)). The value of $f_w \rho_w$ was larger than $(1 - f_w) \rho_{in}$ accounts for 90% of $\rho_t$. The total dislocation density is plotted in accordance with the Bailey–Hirsch equation
\[ \sigma \propto \alpha b^2 \cdot \rho_t^{1/2} \]........................................(6)

in Fig. 8(b). $\alpha$ is a constant between 0.1 and 1, $\mu$ is the shear modulus, $b$ is the magnitude of the Burgers vector. In this result, $\alpha$ was 0.4. Same behavior of $\rho_t$ was obtained in tensile deformed Fe–3%Si by Griffiths and Riley. In the fatigue deformed specimens (Fig. 9), $\rho_w$ was increased rapidly at the initial stage of fatigue due to plastic deformation. After the initial stage, $\rho_{in}$ and $f_w$ stayed constant, only $\rho_w$ was increased as shown in Fig. 9. The percentage of $f_w \rho_w$ in $\rho_t$ was 50–75%.

### 4.3. Relation between Magnetic Properties and Dislocation Structure

The relations between magnetic parameters and the total dislocation density $\rho_t$ in tensile and fatigue deformed steels are discussed in this section. The magnetic parameter, coercive field $H_c$ that is largely-concerned with the mobility of the domain wall is increased due to tensile and fatigue de-
formation, and depends on the total dislocation density. The relation between $H_C$ and $\rho_T$ is given by

$$H_C = H_{C0} + \gamma_{HC} \rho_T^{1/2} \quad \text{..................(7)}$$

$H_C = 8.9 \times 10^{-6} \rho_T^{1/2}$ (Tensile deformation)

$H_C = 7.0 \times 10^{-6} \rho_T^{1/2}$ (Fatigue deformation)

as shown in Fig. 10. This result was similar to the other results, which discussed the relation between $H_C$ and the shear stress $\tau$. It is of interest that the slopes $\gamma_{HC}$ are same in both steels even though the deformation way is different. In fatigue deformed steel, the grouped dislocation is multiplied at only cell wall. On the other hand, the homogeneously distributed dislocation is multiplied due to tensile deformation. This result indicates the influence of grouped dislocations and homogeneously distributed dislocations on coercive field would not be much difference. Coercive field is given by Eq. (1), when the pinning force is the maximum resultant force from lattice defects. The stress field around dislocation is changed with decreasing in neighboring dislocation distance. The stress field around cell wall is decays exponentially with the distance from cell boundary. The stress field of aligned dislocations is counteracted each neighboring dislocations. However, at the close area of cell wall, the amplitude of stress field is same as single dislocation. Therefore, the pinning force from cell wall structuring dislocation would be same as a single dislocation.

The coefficient $c$ is increased due to deformation. The relation between the coefficient $c$ and dislocation density could be given by

$$c = c_0 + \gamma_c \rho_T \quad \text{..................(8)}$$

$c = 9.3 \times 10^4 + 1.9 \times 10^{-5} \rho_T$ (Tensile deformation)

$c = 4.6 \times 10^4 + 4.2 \times 10^{-5} \rho_T$ (Fatigue deformation)

as shown in Fig. 11. The slope of coefficient $c$, $\gamma_c$ in fatigue deformed specimens was twice as in tensile deformed one. Cell wall brought significant increase for the coefficient $c$ because the total dislocation density was increased due to only $\rho_{w}$ increasing in fatigue deformed specimens. The influence of cell wall would be divided into the two factors. One factor originates from the stress field as before mentioned. Another factor originates from the crystal orientation. Differential susceptibility was increased by magnetization direction change at whole magnetic field as shown in Fig. 12. The magnetization was calculated by the balance between the magnetocrystalline anisotropy energy...
Ea and the torque E_H with the next equation

\[ \frac{\partial E_a}{\partial H} - \frac{\partial E_H}{\partial H} = 0 \]

The coefficient c became large with forming cellular structure by rotating the average magnetization direction of specimen in the easy magnetic direction. These two factors would increase the coefficient c, thus the slope γ of fatigue deformed specimens is larger than tensile deformed specimens.

5. Conclusions

The total dislocation density has been calculated by using measured \( \rho_{\text{cell}} \), \( \rho_{\text{cell}} \), and \( f_{\text{cell}} \). The dislocation density at cell interior and cell wall was increased by tensile deformation, but in fatigue deformed steel only at cell wall. We have also discussed the relation between the magnetic properties and the total dislocation density. The magnetic properties, coercive field and the coefficient c were measured for tensile and fatigue deformed steels. In both steels the relations \( H_c \propto \rho_t^{1/2} \) and \( c \propto \rho_t \) were derived. These properties are influenced from the total dislocation density, and independent on the deformation way. From above results, magnetic measurement would be useful for evaluation of dislocation structure change, and applied nondestructive evaluation method with development of the correct B–H curve measurement way.

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