DISLOCATION STRUCTURE AND NON-PROPORTIONAL HARDENING OF TYPE 304 STAINLESS STEEL

S. Kida¹, T. Itoh², M. Sakane³, M. Ohnami¹ and D. F. Socie³

¹Department of Mechanical Engineering, Faculty of Science and Engineering, Ritsumeikan University, 1916 Noji-cho Kusatsu-shi, Shiga, 525-77, Japan
²Department of Mechanical Engineering, Faculty of Engineering, Fukui University, 9-1 Bunkyo 3-chome Fukui, 910, Japan
³Department of Mechanical and Industrial Engineering, University of Illinois, 1206 West Green Street, Urbana, IL 61801, USA

Received in final form 19 May 1997

Abstract—This paper describes the microstructure of Type 304 stainless steel after cyclic loading at room temperature under tension-torsion non-proportional strain paths. The degree of cyclic non-proportional hardening is correlated with changes in the dislocation substructure. Dislocation cells, dislocation bundles, twins and stacking faults are all observed. The type of microstructure formed and resultant stress response is dependent on the degree of non-proportional loading and strain range. Cyclic stress range was uniquely correlated with mean cell size.

Keywords—Multiaxial fatigue; Non-proportional loading, Cyclic plasticity, Dislocation structures.

INTRODUCTION

Many practical applications such as the nuclear vessel of a fast breeder reactor have non-proportional stresses and strains under the combination of thermal and mechanical loading. Type 304 stainless steel is known as a material which shows a significant additional cyclic hardening under non-proportional loading in comparison with proportional loading. Recent studies have shown that the degree of the additional cyclic hardening is material dependent. Doong et al. [1] reported the relationship between the microstructure and additional cyclic hardening behavior of 1100 aluminum alloy, oxygen free pure copper and Type 304 and 310 stainless steels. They reported that no additional hardening occurred in aluminum alloy but significant additional hardening in stainless steel. Non-proportional cyclic hardening was reported for pure copper. They discussed the microstructure change with proceeding cycles in detail for a limited number of strain paths. Cailletaud et al. [2] compiled much of the published data and concluded that the main parameter governing the degree of non-proportional hardening in solid solution material is the ease of cross slip. Itoh [3,4] studied non-proportional cyclic hardening of Type 304 stainless steel, pure copper, pure nickel, pure aluminum and 6061-T6 aluminum and reported that the degree of additional cyclic hardening is related to the stacking fault energy of the material. For a material with a low stacking fault energy, such as Type 304 stainless steel, planar slip occurs and results in a large amount of additional cyclic hardening. This is caused by the interaction of many slip systems. Materials with a high stacking fault energy such as pure aluminum and 6061-T6 aluminum alloy deform by wavy slip. These materials do not show additional cyclic hardening during non-proportional loading. The difference in the additional hardening behavior between high and low stacking fault energy materials is to be related to the microstructure of the material but extensive and systematic studies have not yet been reported.

Several investigators have examined the dislocation structure for room temperature tests. Doquet
[5] reported twin deformation as a primary deformation mechanism under non-proportional loading for binary Co33Ni. She reported that the increase in the amount of twin deformation is a cause of additional cyclic hardening during non-proportional loading. Jiao et al. [6] examined alloy 800 H and observed deformation twins and suggested that the formation of twins depends not only on the shear stress but also on the normal stress acting on the slip plane. McDowell et al. [7] found that the heterogeneity of ε-martensite and other planar slip deformation products (e.g. α'-martensite) are a function of a non-proportionality in 304 stainless steel. They found that the homogeneity and morphology of the deformation products is of key importance. Cailletaud et al. [2] observed ladders, veins or dislocation cell structures with loose outlines in uniaxial specimens but walls, mazes, cells, and, above all, abundant micro-twinning for non-proportionally loaded specimens of Type 316 stainless steel. Twinning is not an easy deformation mode in 316 steel at room temperature. The critical shear stress needed to induce twinning was reached because of the additional hardening during the non-proportional tests. Doong et al. [1] found single slip structures under proportional loading of both 304 and 310 stainless steel. Multi-slip structures such as cells and labyrinths were found for non-proportional loading. At high temperatures, Hishino et al. [8] observed the dislocation structure of Type 304 stainless steel cyclically loaded at 823 K and have discussed the relationship between the dislocation structure and hardening behavior. They concluded that anisotropic hardening is caused by the directionally developed cell formation and isotropic hardening by the formation of round-shaped cells.

Microstructural studies of additional non-proportional cyclic hardening have been limited to a small number of strain paths so that the results of these studies are rather qualitative. Little quantitative discussion has been reported. This paper studies the microstructure and cyclic stress-strain relationships obtained at room temperature under 14 non-proportional strain paths for Type 304 stainless steel, and will discuss the relationship between stress response and cell structure quantitatively.

**EXPERIMENTAL PROCEDURE**

The material tested is Type 304 stainless steel that received a solution treatment at 1373 K for 1 h. Hollow cylindrical specimens with 9 mm I.D., 12 mm O.D., and 4.6 mm gage length were employed in this study. Strain controlled cyclic loading tests at a Mises' effective strain rate of 0.1%/s were carried out at ambient temperature. Testing details are reported by Itoh et al. [9]. Figure 1 shows the 14 proportional and non-proportional loading histories employed, where ε and γ are the axial and shear strains, respectively. Case 0 is a push-pull proportional test which is the basic data for examining the microstructure. Case 0 testing was carried out at strain ranges between 0.5 and 1.5%. Case 5 is also a proportional test as in Case 0, but is a combined push-pull and reversed torsion test. The other cases are non-proportional tests in which the severity of non-proportional loading is determined by the strain history. In all the tests except for Case 0, axial and shear strain ranges were 0.5 and 0.8% Mises' equivalent strain.

One cycle is defined here as a full straining for both axial and shear strain. All the tests except Cases 3, 4 and 13 were counted as one cycle and these tests were counted as two cycles for a full straining along the strain path chosen in Fig. 1. The number of cycles to failure (Nf) was defined as the cycle at which the axial stress amplitude decreased to 5% of the saturation stress in tension.

After the cyclic loading tests, thin foils of 3 mm diameter were cut from specimens away from cracks by a wire cutter to observe the microstructure. They were polished down to about 0.2 mm in thickness with emery papers and were jet-electropolished in acetic and perchloric acid. A JEOL JEM-100C (100 kV) was used to observe the microstructure and diffraction pattern.
During non-proportional loading, stress and strain amplitudes vary with time, so that the principal strain and stress ranges must be defined. In a previous paper [9], the authors have proposed a definition of the maximum principal strain and stress ranges for non-proportional loadings and this paper follows that definition.

The maximum principal strain range, $\Delta \varepsilon_1$, is defined as,

$$\Delta \varepsilon_1 = \max [\varepsilon_{\text{max}} + \cos(\xi(t))\varepsilon_1(t)]$$

In this equation, $\varepsilon_1(t)$ is the maximum absolute value of principal strain at time $t$ and

$$\varepsilon_1(t) = |\varepsilon_1(t)| \quad \text{for} \quad |\varepsilon_1(t)| \geq |\varepsilon_3(t)|, \quad \varepsilon_1(t) = |\varepsilon_3(t)| \quad \text{for} \quad |\varepsilon_1(t)| \leq |\varepsilon_3(t)|$$

where $\varepsilon_1(t)$ and $\varepsilon_3(t)$ are the maximum and minimum principal strains at time $t$, respectively. Figure 2 is a polar figure of $\varepsilon_1(t)$ schematically showing $\varepsilon_1(t)$, $\xi(t)$ and $\Delta \varepsilon_1$. In Eq. (1), $\varepsilon_{\text{max}}$ is the maximum value of $\varepsilon_1(t)$ in a cycle and $\xi(t)$ is the angle between $\varepsilon_{\text{max}}$ and $\varepsilon_1(t)$ directions. Thus, $\Delta \varepsilon_1(t)$ is determined by the two strains, $\varepsilon_1(A)$ and $\varepsilon_1(B)$, and by the angle between the two strain directions in Fig. 2, where $A$ is the time giving $\varepsilon_{\text{max}}$ and $B$ the time maximizing the strain range in Eq. (1).
The maximum principal stress range, $\Delta \sigma_1(t)$ has a similar definition to $\Delta \varepsilon_i$,

$$\Delta \sigma_1 = \sigma_i(A) + \cos(\xi(B))\sigma_i(B)$$

where $A$ and $B$ correspond with those defined for the maximum principal strain range.

A non-proportional factor, $f_{NP}$, was proposed by the authors to express the severity of non-proportional loading [9].

$$f_{NP} = \frac{\pi}{2T_{\text{max}}} \int_0^T (|\sin(\xi(t))| |\varepsilon_i(t)|) \, dt$$

where $T$ is the time for a cycle shown in Fig. 1. The value of $f_{NP}$ is zero under proportional loading and is the range of $0 \leq f_{NP} \leq 1$ under non-proportional loading. As shown in Eq. (4), $f_{NP}$ is a function of only the applied strain history to avoid the need to compute stresses and plastic strains.

The authors [9] have proposed the following strain parameter to correlate non-proportional LCF lives,

$$\Delta \varepsilon_{NP} = (1 + \alpha f_{NP}) \Delta \varepsilon_i$$

where $\alpha$ is a material parameter related to the additional hardening of the material under non-proportional loading, and $\alpha$ is defined as the ratio of stress range under non-proportional circular loading in $\gamma/\sqrt{3-\varepsilon}$ plot to that under proportional loading at the same Mises equivalent strain. The value of $\alpha$ becomes larger for lower stacking fault energy materials [1–4]. Murakami et al. [10] showed that this parameter will decrease with increasing temperatures. For Type 304 stainless
Dislocation structure and non-proportional hardening of Type 304 stainless steel 1379

steel at room temperature, \( \alpha \) takes the value of 0.9. Benallal and Marquis [11] show a small strain range dependence of \( \alpha \) but here we take it as a constant. The term \( (1 + \beta f_{NP}) \) accounts for the additional cyclic hardening observed during non-proportional cyclic loading and is similar to damage parameters that are based on the product of stress and strain range. The advantage of this parameter is that it does not require a sophisticated transient cyclic plasticity model to obtain the stress ranges.

EXPERIMENTAL RESULTS AND DISCUSSION

A complete tabular listing of all test data is available in [9]. Table 1 is a condensed version that gives the stress and strain ranges, fatigue lives and computed non-proportional loading factor for the 14 strain paths in Fig. 1. Mean cell size is also included. Fatigue lives for cases 0–13 significantly depend on the strain history. Rotating principal strain directions in tests such as Cases 8–10, 12 and 13 yields the largest reduction in fatigue lives by as much as a factor of 10. In Cases 6–9, steps in the path can have a large influence on fatigue lives when the number of steps is small and the path length is large. Thus, Case 6 shows a small reduction in fatigue life as the strain history is nearly proportional loading because of the small step length. Figure 3 correlates LCF lives with principal stress range. The figure shows that a significant additional hardening occurs under non-proportional loadings. Greater additional cyclic hardening results in smaller fatigue lives. Thus, an estimate of additional hardening is necessary for predicting fatigue lives under non-proportional loading in the LCF regime. Figure 4 correlates the non-proportional LCF lives with equivalent strain given in Eq. (5). Most of the data are correlated within a factor of two scatter bands.

Figure 5 shows the relationship between the stabilized axial and shear stresses for twelve of the fourteen loading histories. The stress response for Case 8 is the mirror image of Case 9 and was omitted from the figure. In the figures, solid lines are the results at \( \Delta \varepsilon = 0.5\% \), and dashed lines are the results at \( \Delta \varepsilon = 0.8\% \). The shear stress scale has been plotted as one half of the axial stress scale so that stresses can easily be compared on the basis of maximum shear stress. Comparing the equivalent stresses in Table 1 for Cases 1 and 2 with Case 0 at \( \Delta \varepsilon = 0.5 \) and 0.8\% shows significant non-proportional hardening due to the change of principal strain direction at zero strain. The normal stress–shear stress relationship of Case 1 is different from that of Case 2 which shows that a fully reversed straining has a different influence on stress response from a zero-to-maximum straining, and the former strain history causes greater additional hardening than the latter one. This additional hardening also results in a lower fatigue life for Case 2. The shape of the \( \tau–\sigma \) plot of Cases 3 and 4 is similar to that of Cases 1 and 2, respectively, after giving a rotation of 45° to the former two cases. However, the stress amplitude of Cases 3 and 4 is larger than that of Cases 1 and 2 because the shear and axial strains are applied simultaneously resulting in a cyclic strain range in the former two cases that is larger by about 1.4 times.

A simple method of visualizing the degree of non-proportionality is useful when interpreting the stresses in Fig. 5. If an ellipse is drawn so as to circumscribe the entire stress path, non-proportionality can be thought of as the ratio of the minor axis to the major axis. In Case 5, the minor axis of an ellipse circumscribing the loading history is small which corresponds to a low degree of non-proportionality. The degree of non-proportionality increases in going from Case 5 to Case 10. This is easily visualized as an increasingly circular ellipse circumscribing the stress history. The size of the ellipse also increases as the degree of non-proportionality increases. Case 5 is proportional loading where the normal stress amplitude \( \sigma \) is the same as the shear stress amplitude \( \sqrt{3} \tau \) since the normal strain equals the shear strain on Mises basis. Comparing the
### Table 1. Summary of the test results together with the stress and strain parameters

| Strain Path (CASE) | Cycles to Failure $N_c$ | Axial Strain $\Delta e$ | Shear Strain $\Delta \gamma$ | Axial Stress $\Delta \sigma$, MPa | Shear Stress $\Delta \tau$, MPa | Equivalent Stress, MPa | Cell Size d, mm | Nonproportional Factor $f_{np}$ |
|--------------------|-------------------------|-------------------------|-------------------------------|---------------------------------|-------------------------------|------------------------|-----------------|-------------------------------|
| 0                  | 49000                   | 0.050                   | 0                             | 530                             | 0                             | 558                    | 2.178           | 0                             |
| 0                  | 23400                   | 0.065                   | 0                             | 580                             | 0                             | ***                    | ***             | 0                             |
| 0                  | 71000                   | 0.080                   | 0                             | 630                             | 0                             | 633                    | 2.209           | 0                             |
| 0                  | 15000                   | 1.000                   | 0                             | 730                             | 0                             | 735                    | 1.803           | 0                             |
| 0                  | 17000                   | 1.13                    | 0                             | 730                             | 0                             | 743                    | 1.613           | 0                             |
| 0                  | 690                     | 1.20                    | 0                             | 805                             | 0                             | 802                    | 1.207           | 0                             |
| 0                  | 540                     | 1.50                    | 0                             | 825                             | 0                             | 824                    | 1.046           | 0                             |
| 1                  | 95000                   | 0.50                    | 0.87                          | 685                             | 395                           | 683                    | 1.584           | 0.34              |
| 2                  | 20000                   | 0.50                    | 0.87                          | 670                             | 355                           | 666                    | 1.406           | 0.34              |
| 3                  | 24000                   | 0.50                    | 0.87                          | 670                             | 420                           | 892                    | 0.912           | 0.39              |
| 4                  | 34000                   | 0.50                    | 0.87                          | 790                             | 395                           | 884                    | 1.975           | 0.39              |
| 5                  | 17500                   | 0.50                    | 0.87                          | 485                             | 185                           | 583                    | 1.645           | 0.00              |
| 6                  | 97000                   | 0.50                    | 0.87                          | 500                             | 240                           | 624                    | ***             | 0.10              |
| 7                  | 18000                   | 0.50                    | 0.87                          | 530                             | 285                           | 637                    | 1.959           | 0.20              |
| 8                  | 20500                   | 0.50                    | 0.87                          | 760                             | 410                           | 948                    | 1.405           | 0.77              |
| 9                  | 29500                   | 0.50                    | 0.87                          | 780                             | 370                           | 799                    | 1.275           | 0.77              |
| 10                 | 26000                   | 0.50                    | 0.87                          | 765                             | 400                           | 898                    | 1.111           | 0.77              |
| 11                 | 14400                   | 0.50                    | 0.87                          | 570                             | 280                           | 595                    | 1.611           | 0.46              |
| 12                 | 47500                   | 0.50                    | 0.87                          | 660                             | 360                           | 670                    | ***             | 0.77              |
| 13                 | 32000                   | 0.50                    | 0.87                          | 655                             | 360                           | 663                    | 1.491           | 0.77              |
| 1                  | 14000                   | 0.80                    | 1.39                          | 950                             | 530                           | 960                    | 0.897           | 0.34              |
| 2                  | 21000                   | 0.80                    | 1.39                          | 860                             | 490                           | 860                    | 1.370           | 0.34              |
| 3                  | 820                     | 0.80                    | 1.39                          | 975                             | 545                           | 1237                   | 0.503           | 0.39              |
| 4                  | 900                     | 0.80                    | 1.39                          | 1010                            | 520                           | 1114                   | ***             | 0.39              |
| 5                  | 32000                   | 0.80                    | 1.39                          | 590                             | 250                           | 732                    | 1.418           | 0.00              |
| 6                  | 26000                   | 0.80                    | 1.39                          | 670                             | 320                           | 770                    | 1.174           | 0.10              |
| 7                  | 17000                   | 0.80                    | 1.39                          | 735                             | 390                           | 822                    | 1.339           | 0.20              |
| 8                  | 470                     | 0.80                    | 1.39                          | 1055                            | 560                           | 1046                   | 0.759           | 0.77              |
| 9                  | 660                     | 0.80                    | 1.39                          | 1075                            | 600                           | 1070                   | ***             | 0.77              |
| 10                 | 320                     | 0.80                    | 1.39                          | 1060                            | 555                           | 1172                   | 0.649           | 0.77              |
| 11                 | 12000                   | 0.80                    | 1.39                          | 850                             | 500                           | 924                    | 1.001           | 0.46              |
| 12                 | 710                     | 0.80                    | 1.39                          | 940                             | 510                           | 960                    | ***             | 0.77              |
| 13                 | 10000                   | 0.80                    | 1.39                          | 965                             | 550                           | 1025                   | 1.048           | 0.77              |
Dislocation structure and non-proportional hardening of Type 304 stainless steel

A cell structure is observed in Case 0, Fig. 6(b) where the mean cell diameter is around 1 μm. Cell formation was also observed in the specimens cyclically loaded at large strain ranges (>1%) in Case 0. Dislocation bundles which indicates the cluster of dislocations were observed at low strain ranges (<0.8%) in Case 0.

Cell structures, twins and stacking faults were observed in Case 1, Fig. 6(c), but only twins and stacking faults were observed in Case 3, Fig. 6(d). No clear cell formation was found in Case 3,

OBSERVATIONS OF DISLOCATION STRUCTURE

Additional hardening has been reported to have a close connection with dislocation structure [9], but there have been few systematic and quantitative studies of the relationship between the microstructure and additional hardening. Figure 6(a)–(j) show the microstructure observed by TEM. Figure 6(a) shows the dislocation structure before testing where the dislocation density is very low and no specific substructure is identified.

A cell structure is observed in Case 0, Fig. 6(b) where the mean cell diameter is around 1 μm. Cell formation was also observed in the specimens cyclically loaded at large strain ranges (>1%) in Case 0. Dislocation bundles which indicates the cluster of dislocations were observed at low strain ranges (<0.8%) in Case 0.

Cell structures, twins and stacking faults were observed in Case 1, Fig. 6(c), but only twins and stacking faults were observed in Case 3, Fig. 6(d). No clear cell formation was found in Case 3,
and dislocation bundles were observed. Many stacking faults occurred before cell formation and they appear to hinder the cell formation in Case 3. The number of stacking faults in Case 3 is larger than that in Case 1. The phasing of the applied strains produces larger stress and strain ranges for Case 3.
Fig. 6. Microstructures observed by TEM. (a) Before test; (b) Case 0, $\Delta \varepsilon = 1.0\%$; (c) Case 1, $\Delta \varepsilon = 0.8\%$; (d) Case 3, $\Delta \varepsilon = 0.8\%$; (e) Case 5, $\Delta \varepsilon = 0.8\%$; (f) Case 7, $\Delta \varepsilon = 0.8\%$; (g) Case 9, $\Delta \varepsilon = 0.8\%$; (h) Case 10, $\Delta \varepsilon = 0.8\%$; (i) Case 11, $\Delta \varepsilon = 0.8\%$; (j) Case 13, $\Delta \varepsilon = 0.8\%$. 
Nishino et al. [8] reported that a ladder of maze structure was a common structure for Type 304 stainless steel in proportional straining and a cell structure was primarily found in the non-proportional straining like Case 1 at high temperature. At room temperature, however, cell structures formed and no ladder or maze structures were observed in Case 0 loading. This difference in dislocation structure between room and elevated temperatures results from the difference in the thermal activation. At elevated temperatures, dislocations slide more easily to form a structure of low elastic energy by the assistance of thermal activation so that a ladder or maze structure, which is a lower elastic energy microstructure than the cell, was found. In Case 5, which is a proportional straining, Fig. 6(e), cell structures were observed. A deformation twin boundary was also observed at the center of the photograph. Diffraction patterns were taken for all of the dislocation structures but it was difficult to distinguish between stacking faults and martensite that was observed by McDowell et al. [7]. Case 7, Fig. 6(f), shows a coherent boundary of an annealing twin at the center of the photograph. It’s role is similar to a grain boundary for the deformation microstructure.

Dislocation walls are observed in the right of the photograph, and a cell structure is found in the left of the photograph. In Case 7, columnar cells were formed and Fig. 6(f) shows the two different sections of the columnar grains; the left is the normal section to columnar axes and the right is the parallel section of them. In Case 7, the maximum shear strain direction changed its direction, so that dislocations would easily rearrange to columnar structure by the cross steps.

Cases 5–10 all have the same maximum shear strain ranges. Cases 8–10 have a rectangular or box strain history. In these strain paths, the maximum shear strain direction rotates continuously, so that many slip systems operate. The additional hardening was most significant in these strain paths. Cases 11–13 are also rectangular strain paths but the phasing of the strains is such that the maximum shear strain range is smaller than Cases 5–10. In Case 9, Fig. 6(g), cell boundaries are not clear, but many dislocations exist even in the cells. The maximum shear stress direction rotates continuously in Case 9, so many slip planes operate and interact, which results in the significant additional hardening.

In Cases 10 and 11, Figs 6(h) and (i), many stacking faults or martensite was observed. There is little difference between a stacking fault and martensite formation. A stacking fault forms when one atomic plane is dropped off and when more than two atomic planes have a disordering it is a different structure which may be martensite. Since Type 304 stainless steel is a material of low stacking fault energy, slip is planar and there are many partial dislocations which make a stacking fault between them. Long stacking faults exceeding several subgrains in length were formed in Case 10 with short stacking faults formed within cells in Case 11. The long stacking faults were formed by the severe box non-proportional straining and which hindered the cell formation, while, in Case 11, the cells were formed earlier than the stacking faults and stacking faults were stopped by the cell boundaries. For Case 13, fine cells are found and they are rather close to subgrain since the cell boundaries are rigid and misorientation angle between cells is rather large. This strain path made resulted in clear cells and rigid cell boundaries.

Figure 7 is a microstructure map showing the cell, dislocation bundle and stacking fault boundaries as functions of maximum principal strain range and non-proportional factor for all the strain paths. In the figure, solid symbols indicate tests in which only cells were observed, while open symbols represent tests in which cells and other dislocation structures were found. Asterisks indicate tests where stacking faults were observed and the number at the data indicates the strain path number shown in Fig. 1. This figure shows that stacking faults were observed in almost all the tests and did not depend on the principal strain range and non-proportional factor. Type 304 is a low stacking fault energy material and a dislocation easily splits into partial dislocations, making a stacking fault between them. A partial dislocation glides on the slip plane, and a stacking
fault arises between the partial dislocations. Many stacking faults seem to be generated by this mechanism. There is a critical combination of strain range and non-proportional factor for forming cells indicated by the solid line. In the region above the line, the microstructure is dominated by cells with other microstructures in addition to cells observed for the test conditions below the solid line.

Figure 8 shows the relationship between the mean cell size and the maximum principal stress range for all the tests where the cell structure was observed. The mean cell size was determined by the Heyn method (JIS G0552), observing 3 or 4 locations of each specimen. Maximum principal stress range and mean cell size can be approximated by a straight line for all of the strain histories. The relationship is,

$$\Delta \sigma = m \times d^n$$  \hspace{1cm} (8)

The values of $m$ and $n$ are 975 MPa and $-0.57$, respectively when $d$ is measured in $\mu$m. The value of exponent is close to $-1/2$, so that the Hall–Petch relationship holds in proportional and non-proportional loadings. As shown in Fig. 7, various microstructures are formed under non-proportional loading. However, the results in Fig. 8 indicate that the additional hardening in non-proportional loading is mainly caused by reduction of cell size. The severe interaction of slip systems under non-proportional loading reduces the cell size and results in the additional hardening. The results also imply that microstructures other than cell structure have almost no influence on the additional hardening.

**CONCLUSIONS**

(1) Dislocation substructures observed under non-proportional loading were associated with cells, stacking faults, twins and bundles.

(2) A microstructure map was proposed that show conditions for forming cells and stacking faults as functions of the maximum principal strain range and a non-proportional factor. There exists a critical boundary for forming cells. Stacking faults were observed in almost all the proportional and non-proportional tests.
The principal stress range was uniquely correlated with the mean cell size and is independent of the strain loading path which indicates that the additional hardening was mainly associated with a reduction of cell size.

Acknowledgement—The authors express their gratitude to Dr Kei Ameyama, the associate professor of Ritsumeikan University, for assisting with the TEM observations. Prof. D. F. Socie was supported by the U.S. Department of Energy, Division of Materials Science grant DE-F602-96ER45439 through the University of Illinois at Urbana-Champaign, Fredrick Seitz Materials Research Laboratory.

REFERENCES

1. S. H. Doong, D. F. Socie and I. M. Robertson (1990) Dislocation substructure and non-proportional hardening. J. Engng Mater. Technol. 112(4), 456–465.
2. G. Cailletaud, V. Doquet and A. Pineau (1991) Cyclic multiaxial behavior of an austenitic stainless steel: microstructural observations and macromechanical modeling. In: Fatigue Under Biaxial and Multiaxial Loading, ESIS 10 (Edited by Kussmaul et al.), 131–149.
3. Itoh, M. Sakane, M. Ohnami and K. Ameyama (1992) Effect of stacking fault energy on cyclic constitutive relation under nonproportional loading. J. Soc. Mater. Sci., Japan 41(468), 1361–1367 (in Japanese).
4. T. Itoh, M. Sakane, M. Ohnami and K. Ameyama (1992) Additional hardening due to nonproportional cyclic loading—a contribution of stacking fault energy. MECAMAT'92, Proceedings International Seminar on Multiaxial Plasticity, Cachan, France (Edited by Benallal et al.), 43–50.
5. Doquet (1992) Deformation twinning and cyclic behaviour of a CoNi alloy under multiaxial loading. MECAMAT'92, Proceedings International Seminar on Multiaxial Plasticity, Cachan, France, 51–66.
6. F. Jaio, W. Osterle, P. D. Portella and J. Ziebs (1995) Biaxial path dependence of low cycle fatigue behavior and microstructure of alloy 800 H at room temperature. Mater. Sci. Engng A196, 19–24.
7. D. L. McDowell, D. R. Stahl, S. R. Stock and S. D. Antolovich (1988) Biaxial path dependence of deformation substructure of Type 304 stainless steel. Metallurgical Transactions A 19A, 1277–1293.
8. S. Nishino, N. Hamada, M. Sakane, M. Ohnami, N. Matsumura and M. Tokizane (1986) Microstructural study of cyclic strain hardening behavior in biaxial stress state at high temperature. Fatigue Fract. Engng Mater. Struct. 9(1), 65–77.
9. Itoh, M. Sakane, M. Ohnami and D. F. Socie (1995) Nonproportional low cycle fatigue criterion for Type 304 stainless steel. ASME J. Engng Mater. Technol. 117(3), 285–292.
10. S. Murakami, M. Kawai, K. Aoki and Y. Ohmi (1989) Temperature dependence of multiaxial non-proportional cyclic behavior of Type 316 stainless steel. J. Engng Mater. Technol. 111(1), 32–39.
11. A. Benallal and D. Marquis (1987) Constitutive equations for nonproportional cyclic elasto-viscoplasticity. J. Engng Mater. Technol. 109(4), 326–336.
12. D. F. Socie (1987) Multiaxial fatigue damage models. J. Engng Mater. Technol. 109(3), 293–298.