Gigacycle Fatigue Properties of V-Added Steel with an Application of Modified Ausforming

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This report reveals gigacycle fatigue properties for a modified-ausformed V-added steel with the chemical composition of 0.3 C-0.3 Si-1.0 Cr-0.7 Mo-0.3 V in mass %. Modified-ausformed and oil-quenched steels were prepared for fatigue tests, followed by tempering at 400°C and 600°C. The tensile strengths of the 600°C tempered steels were almost equal to those of the 400°C tempered versions because of secondary hardening due to fine precipitation of vanadium carbides. The fatigue properties of the 600°C tempered version of oil-quenched steel (QT600) showed little difference from the 400°C tempered version (QT400) in spite of the fine precipitation of vanadium carbides. The modified-ausformed steels (AF400 and AF600) revealed higher fatigue limits at $5 \times 10^9$ cycles than the oil-quenched versions (QT400 and QT600), although the difference between AF400 and AF600 was small. The remarkable difference between AF400 and AF600 was fatigue strengths at around $10^6$ cycles, i.e. the fatigue strength of AF600 at those cycles was higher than that of AF400. Based on the above results, the effect of the fine precipitation of vanadium carbides was small on the gigacycle fatigue properties, while modified-ausforming could improve those properties. On the other hand, the multiple effects of the fine precipitation and modified-ausforming was large on the fatigue strength at around $10^6$ cycles.

Key Words: Iron and Steel, Fatigue, Fractography, Gigacycle, Modified-Ausforming, Fine Vanadium Carbide, Fish-Eye Fracture

1. Introduction

Weight-saving of automotive components and machine parts is needed for reasons of energy saving and consequent CO2 reduction, and as a result, the demand for higher-strength and -performance iron and steel materials is continuing to grow. However, in high-strength steels with a strength level over 1 200 MPa, internal fractures occur with initiation sites at inclusions and matrix cracks(1), (2), causing the problem called gigacycle fatigue when the number of cycles exceeds $10^7$. It is known that the fatigue limits of steels are usually approximately one-half those of tensile strengths(3). When internal fractures occur, fatigue strength drops significantly below these levels.

According to Murakami et al., optically dark area (ODAs)(4), which are observed microscopically as dark areas, are present on the fracture surfaces around the inclusion at the initiation site of this type of internal fracture. ODA formation is suspected as being one of the causes of reduction in fatigue strength, and its connection with hydrogen embrittlement has also been suggested.

The authors’ research group has been proceeding with research in another direction: the application of modified ausforming, a type of thermomechanical treatment, to overcome internal fracturing. It has been reported that modified ausforming provides a martensitic structure with fine, uniform blocks and is effective in improving the delayed fracture properties caused by hydrogen embrittlement(5), (6). This suggests the feasibility of suppressing ODA formation by means of modified ausforming, as a result of which improvements in the internal fracture property can be expected. Actually, with SCM 440 steel(7), (8), SUP 12 steel(9), and Fe-C-Si-Mn steel(10) treated with modified ausforming, ODA formation was suppressed, leading to improvements in the delayed fracture property.

In addition, a research result has recently been reported in which the hydrogen-trapping effect of finely precipitated vanadium (V) carbides improves the delayed fracture property(11), (12). Therefore, improvement in fatigue properties of high-strength steels owing to fine V carbides is also expected from the viewpoint of suppress-
ing ODA formation due to the hydrogen-trapping effect.

The hydrogen-trapping effect of fine V carbides was also investigated in the current study in addition to modified ausforming which has been the focus of research so far. Materials were prepared in the study for a total of 4 conditions, combining modified ausforming treatment applied/not applied, fine V carbides present/absent, and giga-cycle fatigue properties up to $5 \times 10^9$ cycles. The results are described in this report.

2. Experimental Methods

2.1 Test material

The test material is a low-alloy steel, whose chemical composition is shown in Table 1, with a carbon (C) content of 0.3 mass % and a V content of 0.35 mass % added.

Figure 1 shows changes in Vickers hardness of the test material with a diameter of 13 mm when tempered at each temperature between 300°C and 650°C after oil-quenching from 920°C×45 min. The test material shows a monotonic decrease in hardness at tempering temperatures from 300°C to 500°C, and a secondary hardening, i.e., a subsequent increase in hardness, at temperatures above 550°C that peaks at around 600°C. This is caused by the precipitation of solid-solution V as fine V carbides (called "VC" hereinafter) at 550 – 600°C. The modified ausforming-treated material prepared under the conditions described below also showed a trend similar to that of the oil-quenched material.

Table 2 Heat treatment conditions

| Material | Quenching | Tempering          |
|----------|-----------|--------------------|
| AF600    | Modified-ausforming | 600°C for 90 min, water-cool |
| QT600    | 920°C for 45 min, oil-cool | 600°C for 90 min, water-cool |
| AF400    | Modified-ausforming | 400°C for 90 min, water-cool |
| QT400    | 920°C for 45 min, oil-cool | 400°C for 90 min, water-cool |

Heat treatment conditions for the test material are shown in Table 2. Two conditions, modified-ausforming treatment and ordinary oil-quenching, were employed for quenching, and 2 conditions, 600°C and 400°C, for tempering. Thus, a total of 4 kinds of materials were prepared. In the modified ausforming treatment, as shown in Fig. 2, 14-mm square bar stocks were austenitized at 920°C, air-cooled down to 830°C, wrought by caliber-rolling at a 30% reduction in area into bars 13 mm in diameter, and were immediately water-cooled. In the ordinary oil-quenching, bars 13 mm in diameter were quenched from 920°C×45 min. With respect to the 2 conditions for tempering, 600°C and 400°C, the former is the temperature where the secondary hardening peak was obtained due to precipitation of fine VCs, and the latter is the condition for obtaining the hardness at a level similar to that of the material tempered at 600°C without involving precipitation of the fine VCs. These materials are termed as follows: for the modified ausforming-treated material, AF600 for the 600°C-tempered type and AF400 for the 400°C-tempered type; for the conventionally oil-quenched material, QT600 for the 600°C-tempered type and QT400 for the 400°C-tempered type.

2.2 Structural observation

Structural observations were made using an optical microscope in planes parallel to the rolling direction. After the observation surfaces of each material were mirror-finished, microstructural observations were made after etching with 3% Nital, and observations of prior-austenite grain boundaries were made after etching with saturated picric acid solution.

2.3 Fatigue tests

Fatigue tests were performed using 3 types of testing machines: an ultrasonic fatigue-testing machine with a frequency of 20 kHz; a rotating bending fatigue-testing...
machine with a frequency of 120 Hz; and a servo hydraulic testing machine with a maximum frequency of 50 Hz. Appropriateness of the ultrasonic fatigue test was confirmed, in that no frequency effect is observed even at 20 kHz in the low-stress and long-life region where internal fracture takes place\(^{(13)}\). Therefore, on the basis on the high-and-low frequency hybrid test method\(^{(14)}\) where only the low-stress and long-life region is tested by an ultrasonic fatigue-testing machine, gigacycle fatigue tests up to \(5 \times 10^9\) cycles were performed by choosing the most appropriate machines from the three. The stress ratio, \(R\), was \(-1\) for all the fatigue tests, and the test environment was in air at room temperature.

Figure 3 shows the specimen geometries. Specimens with a minimum diameter of 4 mm as shown in (a) were used for the rotating bending test and the servo hydraulic test, and specimens with a minimum diameter of 3 mm as shown in (b) were used for the ultrasonic test.

### 2.4 Fracture surface observation

Fracture surface observations were made using a field emission type of scanning electron microscope (FE-SEM). When internal fracture was noted, the initiation sites were analyzed by means of an attached energy-dispersive X-ray analyzer (EDAX), and the types of initiation sites were identified. In addition, for internal fractures, ODA observations were made under a microscope based on Murakami et al.'s method\(^{(4)}\).

### 3. Experimental Results

#### 3.1 Mechanical properties and structural observations

Table 3 shows mechanical properties. JIS No.14A specimens with a parallel portion diameter of 3.5 mm were used for tensile testing. Hardness testing was performed using a Vickers hardness tester at a force of 98 N. No significant difference is observed in the mechanical properties between the modified ausforming-treated material and the conventionally oil-quenched material when tempered under the same conditions. Additionally, a comparison between materials tempered at 600°C and 400°C showed that for both the modified ausforming-treated and the oil-quenched material, the tensile strengths of the materials tempered at 400°C were higher.

Figure 4 shows optical micrographs of the nital-etched surfaces of AF600 and QT600. Both take on a tempered martensitic structure, and AF600 has a more uniform structure than QT600. AF400 and QT400 showed a similar pattern.

![Fig. 4 Microstructure of nital etched surface](image_url)

| Material | 0.2% proof stress \(\sigma_{0.2}\) (MPa) | Tensile strength \(\sigma_t\) (MPa) | Elongation \(\delta\) (%) | Reduction of area \(\psi\) (%) | Vickers hardness HV |
|----------|-----------------|-----------------|-------------|-----------------|----------------|
| AF600    | 1432            | 1501            | 14          | 63              | 482            |
| QT600    | 1332            | 1446            | 13          | 64              | 464            |
| AF400    | 1530            | 1611            | 10          | 63              | 486            |
| QT400    | 1434            | 1594            | 9           | 65              | 491            |

![Table 3 Mechanical properties](image_url)
materials tempered at 400°C showed a similar pattern.

3.2 Fatigue test results

Figures 6 through 8 show S-N curves. Open symbols are for the results obtained by the rotating bending and the servo hydraulic testing machine. Since no difference was observed in fatigue life between the two testing machines, no distinction is made in the symbols employed. The subscript “I” means that the specimen was confirmed to show an internal fracture. However, in the rotating bending test, not only initiation sites of internal fracture are concentrated in the vicinities of specimen surfaces, but also fracture surfaces close to specimen surfaces were damaged in some cases. Therefore, internal and surface fractures were not clearly distinguishable in such cases. The subscript “I” is not affixed in such cases. Additionally, symbols with arrows plotted at 10^8 cycles are for specimens terminated without fracture in the rotating bending fatigue test. Solid symbols are for results obtained in the ultrasonic fatigue test.

Figure 6 compares QT600 and QT400, both conventionally oil-quenched, to verify the hydrogen-trapping effect of finely precipitated VCs. Although QT600 appears to have a slightly higher fatigue strength in the short-life region below 10^6 cycles, the difference between the two is small on the whole. Both QT600 and QT400 showed internal fractures and a similar level of fatigue limits at 5 × 10^9 cycles as obtained by the ultrasonic fatigue-testing machine.

Figure 7 is a comparison of AF400 and QT400 for verification of the effect of the modified ausforming treatment alone. Although there is no difference in fatigue strength in the short-life region below 10^6 cycles between the two, AF400 showed a higher fatigue strength in the long-life region above 10^6 cycles. QT400 showed internal fractures and a fatigue limit of 620 MPa at 5 × 10^9 cycles. AF400, on the other hand, showed no internal fractures except in one specimen, and a fatigue limit of 800 MPa at 5 × 10^9 cycles, which is higher than that of QT400.

Figure 8 compares AF600 and AF400 for verifying the synergistic effect of the fine VCs and modified ausforming. Since heat generation became a problem at and over stress amplitudes of σ_a = 960 MPa in the ultrasonic fatigue test of AF600, tests in high-stress regions were problematic. Therefore, a small number of results obtained by the ultrasonic fatigue test for AF600 do not overlap with the results obtained by the rotating bending or servo hydraulic fatigue test. However, for the other materials, results obtained by the ultrasonic fatigue test overlap with those obtained by the rotating bending or the
servo hydraulic fatigue test, indicating that an appropriate fatigue limit at $5 \times 10^9$ cycles is obtained for AF600 as well. AF600 showed high fatigue strengths on the whole, although some specimens showed internal fractures. Although it showed a fatigue limit of 820 MPa at $5 \times 10^9$ cycles, which is at a similar level to that of AF400, its fatigue strength at around $10^6$ cycles was higher.

3.3 Fracture surface observations

Figure 9 shows typical FE-SEM images of internal fracture surfaces. Fracture surfaces obtained by the ultrasonic fatigue test are shown in (a) for QT600, (b) for QT400, and (c) for AF600. However, (d) for AF400 shows a fracture surface obtained in the servo-hydraulic test since it did not show internal fractures in the ultrasonic fatigue test. Initiation sites (a) through (c) appear to be matrix cracks\(^{(1,2)}\) since inclusions were not found on these fracture surfaces, and an EDAX analysis of the initiation sites showed Fe alone. However, a previous report\(^{(7)}\) has revealed that, when a hard TiN inclusion is an initiation site, separation from the matrix is not accompanied, unlike the case for Al$_2$O$_3$, and therefore, only a part of TiN or minor quantities appear on the fracture surface in many cases. Consequently, it is possible that TiN inclusions are hiding immediately beneath the initiation sites shown in (a) through (c). An Al$_2$O$_3$ inclusion was detected at the initiation site on the fracture surface shown in (d). Although typical examples of fracture surfaces where no inclusions were found in (a) through (c), some fracture surfaces of AF600, QT600, and QT400 also showed Al$_2$O$_3$ inclusions in the vicinities of the initiation sites as in (d). Regarding the ratio of such surfaces, the ratio of fracture surfaces supposedly initiated by matrix cracks or by TiN inclusions as shown in (a) through (c) to ones initiated by Al$_2$O$_3$ in-
Fig. 10 Typical OM fractographs of fish-eye. (a) is QT600 broken at 3.1×10^9 cycles at 610 MPa in an ultrasonic test. (b) was AF600 broken at 6.1×10^8 cycles at 850 MPa in an ultrasonic test.

Inclusions with a diameter of approximately 20 µm as shown in (d) were fifty-fifty. In this regard, however, the only fracture surface of AF400 which clearly showed internal fracture was the one shown in Fig. 9 (d).

Figure 10 shows optical micrographs taken on the same fracture surfaces with the same magnification as those of the SEM images shown in Fig. 9 (a) and (c). Both QT600 in (a) and AF600 in (b) showed ODAs, and the former had larger ODAs than the latter. In addition, QT400 also showed an ODA whose size was equivalent to that of QT600.

4. Discussion

4.1 Influences of fine VCs and modified ausforming on the internal fracture property

Tsuchida et al.\cite{15} found that the hydrogen-absorption amount of the material tempered at 600°C is significantly greater than that tempered at 400°C as a result of hydrogen-absorption tests of a V-added steel with approximately the same composition as that of the current test material and similarly oil-quenched as the current one. Additionally, it was confirmed as a result of TEM observations that fine VCs several nanometers in size in addition to coarse VCs precipitated in the material tempered at 600°C, whereas only coarse undissolved VCs are observed in the material tempered at 400°C. Tsuchida et al. concluded from these results that the fine VCs observed in the material tempered at 600°C act effectively as hydrogen-trapping sites.

Meanwhile, current study, as shown in Fig. 6, indicated that whether VCs (= hydrogen-trapping sites) are present or absent has no significant influence on the internal fracture property. It was expected at the beginning that the internal fracture property is improved owing to suppression of formation of ODAs, which are suggested to be influenced by hydrogen embrittlement due to the hydrogen-trapping effect. However, no such effect was observed. Furthermore, in the fracture surface observations, an ODA as shown in Fig. 10 (a) was observed in the vicinity of the initiation site of internal fracture in QT600 as well as in the samples in which fine VCs were absent. The ODAs of QT600 had a similar size to those of QT400, and there was no difference in the appearance of the ODAs between the two. These results indicate that the hydrogen-trapping effect caused by VCs has minimal influence on the internal fracture property.

However, Urushibara et al.\cite{16} evaluated the delayed fracture susceptibility using the slow strain rate technique (SSRT) of a 0.3 mass % V-added material with the basic composition of SCM 440 by applying repeated stresses in advance after hydrogen charging. As a result, they reported that the delayed fracture susceptibility increases with increasing application of repeated stresses, and that the reason for this is that the hydrogen-trapping action is weak: hydrogen is released owing to the application of repeated stresses. The possibility of trapped hydrogen being released due to repeated stresses cannot be ruled out in the current study, either.

As for the influence of modified ausforming, it was confirmed anew to improve the internal fracture property. Table 4 shows a summary of the fatigue limits at 5×10^9 cycles, which shows that the fatigue limit of AF400 at 5×10^9 cycles is higher by approximately 200 MPa than that of QT400. This is caused by the fact, illustrated in Fig. 7, that the fatigue strength of QT400 decreases due to internal fractures, whereas the internal fractures are almost expelled in AF400. Actually, the fatigue limit of AF400 at 5×10^9 cycles was 0.5 σ_B, as shown in Table 4. This improvement in the internal fracture property is assumed to be caused by suppression of ODA formation as a result of the modified ausforming treatment. Although the mechanism of suppression of the ODA formation through modified-ausforming has not yet been clarified, it is thought that the uniformization and refining of the martensitic structure are involved.

In this regard, however, the synergistic effect of the fine VCs and modified ausforming on the internal fracture property was not significant. Because the fatigue limit of...
Table 4 Summary of fatigue tests results

| Materials | Fatigue strength at $10^7$ cycles | Fatigue strength at $5 \times 10^6$ cycles |
|-----------|----------------------------------|-----------------------------------------|
|           | $\sigma_w$ (MPa) | $\sigma_w / \sigma_y$ | $\sigma_w$ (MPa) | $\sigma_w / \sigma_y$ |
| AF600     | 970                | 0.65                     | 820              | 0.55                     |
| QT600     | 800                | 0.55                     | 600              | 0.42                     |
| AF400     | 800                | 0.50                     | 800              | 0.50                     |
| QT400     | 740                | 0.46                     | 620              | 0.39                     |

AF600 at $5 \times 10^6$ cycles was at a similar level to that of AF400, as shown in Table 4, the effect of the fine VCs superimposed on the modified ausforming did not further improve the internal fracture property.

4.2 Influences of the VCs and the modified ausforming on the surface fracture property

A significant difference between AF600 and AF400 is the fatigue strength in the finite life region as shown in Figs. 7 and 8. Although the fatigue limit at $5 \times 10^6$ cycles does not show a big difference between AF600 and AF400, a comparison of fatigue strengths at around $10^6$ cycles shows that AF600 has markedly higher strength. Figure 11 shows a plot of all the fatigue test results obtained in the current study in the S-N diagram with a vertical axis in terms of stress amplitude, $\sigma_a$, normalized by a tensile strength, $\sigma_B$. Figure 11 also shows the results of axial-load fatigue tests of quenched-and-tempered (tempered at 550–650°C) low-alloy steels (SCM 435, SCM 440, SNC 631, and SNCM 439) taken from the Fatigue Data Sheets(17) published by the National Institute for Materials Science (NIMS). It is understood from Fig. 11 that AF600 shows an extraordinarily high fatigue strength at $10^6$ – $10^7$ cycles in comparison with other materials.

This marked increase in the finite life fatigue life of AF600 can be explained by the improvement in its surface fracture property. Figure 12 is a schematic S-N diagram. The concept has been firmly established(18) that S-N curves exist independently for surface and internal fractures as shown in Fig. 12 (a), and the two knees observed in an S-N curve of a high strength steel are brought about by superimposition of these two curves. Assuming that the S-N curve for the internal fracture remains unchanged, as shown by the solid line in Fig. 12 (b) and that the one for the surface fracture alone, shown by the broken line, increases with the rise of the curve for AF400 to that for AF600, the fatigue life increases as a result of the superimposition of the two. It is understood in this case that, by comparing fatigue lives at the same stress, the fatigue life is extended even if internal fractures take place. Actually, the ratio of the fatigue strength at $10^7$ cycles, $\sigma_{W7}$, of AF600 shown in Table 4 to the tensile strength, $\sigma_B$, is approximately 0.65, which is higher than 0.5 for steels showing ordinary surface fracture.

Improvements such as these in the surface fracture property of AF600 are attributable to the synergistic effect of the fine VCs and modified ausforming. However, the effect of the fine VCs here is not the same as the hydrogen-trapping sites but has a precipitation-hardening effect. Additionally, a careful look at Fig. 11 reveals that QT600, which was conventionally oil-quenched, also has...
a higher fatigue strength at around 10^6 cycles than the material tempered at 400°C. Consequently, the precipitation-hardening due to the fine VCs alone has the effect of improving the surface fracture property, and this effect appears to be enhanced by combination with modified ausforming.

Hayakawa et al. (19) reported, by observing the structures of a tempered SCM 440 steel in detail using an atomic force microscope (AFM) which was modified-ausforming treated, that not only martensite blocks but also carbides are finer and more uniform than those of an conventionally quenched and tempered material. With respect to AF600 as well in the current study, there is a possibility of a change in the VC precipitation condition in addition to uniformization of blocks as a result of the modified-ausforming treatment, and such a change is considered to have contributed to improvement in the surface fracture property. Whatever the case, a detailed structure survey is now needed.

As described above, the surface fracture property of AF600 with precipitation-hardening by fine VCs and modified ausforming combined is of immense interest. However, the internal fracture and the surface fracture were not distinguished in some cases of rotating bending fatigue tests in the current experiment, and therefore, the surface fracture property has not been argued in a strict manner. It is therefore necessary to carry out experiments again and to conduct a study on the surface fracture property of the materials prepared in the current study, focusing on AF600. Thus, although the experiment focusing on the surface fracture property and the detailed study on structures remain as issues for the future, it can be concluded that combining modified ausforming and the fine precipitates described in the current study effectively improves fatigue properties.

**5. Conclusions**

Fatigue tests were performed of modified ausforming-treated and conventionally oil-quenched V-added steel. Studies were undertaken on the effects of the modified ausforming, the hydrogen-trapping effect of the fine VCs, and the combined effect of these on fatigue properties. The results obtained are as follows;

1. QT600, an oil-quenched material, showed a fatigue strength at 5 x 10^6 cycles similar to that of QT400, and did not show the influence of the hydrogen-trapping effect of the fine VCs influencing the internal fracture property.

2. AF400, an modified-ausforming treated material, showed improved fatigue strength at 5 x 10^6 cycles over that of QT400, and improvement in the internal fracture property owing to the modified ausforming.

3. Although the fatigue strength at 5 x 10^6 cycles of AF600, an modified-ausforming treated material, was at a similar level to that of AF400, its fatigue strength at 10^7 cycles was significantly improved.

4. As for the internal fracture property, the modified ausforming showed an improvement effect, but the fine VCs did not.

5. With respect to the surface fracture property, a significant improvement was observed due to the synergistic effect of the modified ausforming and the precipitation-hardening due to the fine VCs.

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