### ⊙ 研究論文

### A Study on the Fatigue Properties of Ti – Ni Shape Memory Alloys

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Ti - Ni계 형상기억 합금의 피로특성에 관한 연구 김 영식\* · 미야자끼 수이찌\*\*

**Key words**: Rotary bending fatigue test, Strain amplitude, Failure life, Deformation behavior, Martensite inducing stress, Transformation enthalpy, DSC measurement

#### **Abstract**

The effects of strain amplitude, test temperature and stress on the fatigue properties for Ti – Ni wires were investigated using a rotary bending fatigue tester specially designed for wires. The fatigue test results were discussed in connection with the static tensile properties. The DSC measurement was conducted after fatigue test in order to clarify the change of transformation behavior due to the progress of fatigue.

Under the temperature below or near the Af, the strain amplitude( $\epsilon_a$ ) – failure life (Nf) curve showed to be composed of three straight lines with two turning points. Of the 2 turning points, the upper one was coincident with the elastic limit strain and the lower one with the proportional limit strain. With rising of the test temperature above Af, the pattern of  $\epsilon_a$  – Nf curve changed gradually to composition of 2 straight lines. The  $\epsilon_a$  – Nf curve shifted depending on test temperature. In the short and medium life zones, the higher the temperature was, the shorter the fatigue life. However, in the long life zone, above the Af temperature, the fatigue life was not affected by the temperature. The transformation enthalpy measured after fatigue test was dependent on Nf,  $\epsilon_a$ , and test temperature.

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### 1.INTRODUCTION

The Ti – Ni alloy subjected to cyclic deformation changes deformation behavior and finally leads to fatigue fracture. Therefore, it is very important to understand the fatigue characteristics of Ti – Ni alloy in order to utilize many application ideas.

Several papers have been reported so far on the fatigue properties of the Ti – Ni alloy in state of plate or bar[1 – 6]. However, the data on the Ti – Ni shape memory alloy wire that is used in most applications such as the fields of sensor, actuator and medicine are scarcely available except only one for the present[7]. Even the only one paper did not reveal the difference in fatigue characteristics between the ordinary material and shape memory alloy wire. The shape memory alloy wire is expected to show different fatigue characteristics from the ordinary materials because of its transformation behavior in deformation process.

Moreover, the cyclic bending deformation properties are very important in applications of the shape memory alloy wire because the components made of that wire receive cyclic bending deformation in many cases. However, the fatigue properties have so far mainly been tested under tension – compression conditions; the fatigue properties of the Ti – Ni alloy wire under cyclic bending deformation have not been revealed sufficiently.

Besides, the change of the cyclic tensile deformation behavior accompanied by increasing of the cyclic number has been investigated in limited cyclic number[8-9]; no results on the damage of the shape memory effect due to the cyclic bending deformation at various temperatures have yet been published.

The purpose of the present paper is to clarify these problems. In the present paper, a rotary bending fatigue tester specially designed for wires was used in order to investigate the effects of strain amplitude, test temperature and stress on the fatigue properties for Ti – Ni wire. The fatigue test results were discussed in connection with the static tensile test results. Moreover, the change of transformation behavior accompanied by the progress of fatigue under various loading conditions was clarified by DSC test.

### 2. Experimental details

The compositions of the alloy used were Ti-50.9at%Ni. The ingots of the alloy were hot drawn, followed by cold drawn by 30% to wires 1.0 mm diameter. The specimens were cut to 100mm length for rotary bending test and 40mm length for tensile test. The wire specimens were annealed at 673K for 3.6 ks followed by water quenching. After the heat – treatment, the specimens were polished lightly by emery papers to remove the lightly oxidized surface layer, and then electropolished. The transformation temperatures of the prepared specimens appeared to be as follows by DSC test:  $R^*=310$ K, Ms=223K,  $A^*=288$ K,  $AR^*=313$ K. Af=323K

The rotary bending tester is composed of a motor with a speed control unit, a light sensor for measuring the number of revolution, a chamber containing silicone oil and an electric heater with a temperature control unit, etc. as shown in figure 1. Both ends of a wire specimen were connected to the motor and the light sensor, respectively. The specimen is cyclic loaded by rotating as bent with a certain arc radius made by both fixing sides. The cyclic loading strain is changeable by changing of the arc radius of the specimen. For testing at various temperatures, the specimens were kept in

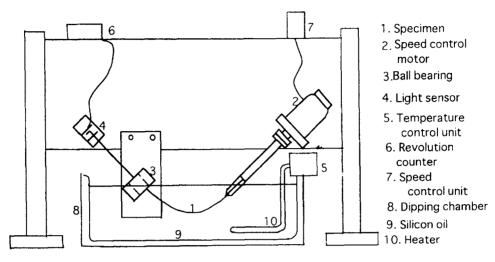


Fig. 1 Schematic figure of rotary bending fatigue tester for SMA wire

silicone oil tank where temperature was controlled by the electric heater and stirring fan. The rotating speeds of the specimens were kept constant at 400r.p.m.

Moreover, the tensile properties were investigated for clarifying the relationship between the static deformation behavior and fatigue properties using a Shimadzu Autograph AG10kND Instron type tester with a heating chamber.

Furthermore, the DSC measurements were conducted on the specimens after the fatigue test in order to investigate the change of transformation behavior due to the progress of fatigue.

### 3. Results and discussion

# 3.1. Deformation behavior at various temperatures.

Figure 2 shows stress – strain curves of the specimen at various test temperatures. General features of the curves with rising of the test temperature are as follows: (1) R phase trans-

formation occurs only at 293K which is below Af temperature; (2) martensite inducing and proportional limit stresses increase straight; (3) elastic and proportional limit strain are constant above Af temperature, 323K; (4) residual strain increases with rising of the temperature above Af temperature. Of these features, matters of (2),(3),(4) were investigated in detail at the temperature range from 223K to 423K. Figure 3 shows the variety of the critical stress for inducing martensite( $\sigma_{\mathtt{m}}$ ) and proportional limit(Pr) with rising of the temperature. Both stresses increase almost in proportion to temperature. Besides, the difference between both stresses becomes larger with rising of the temperature.

The proportional limit is considered to be the point of which a local transformation starts at stress concentration part such as grain boundaries or Ti3Ni4 inclusions. The overall transformation starts at the apparent critical applied stress. The stresses for inducing the local and overall transformation increase together in same proportion with rising of the temperature. As a consequence, the difference between

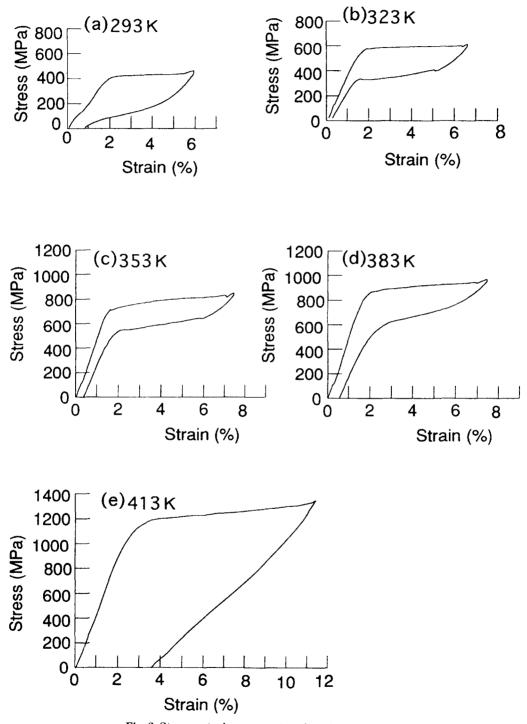


Fig. 2 Stress - strain curves at various temperatures.

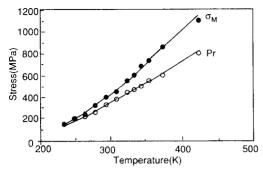


Fig. 3 Effect of test temperature on the stress for inducing martensite( $\sigma_M$ )and the proportional limit(Pr).

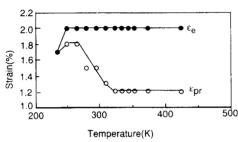


Fig. 4 Elastic limit strain( $\epsilon_e$ )and proportional limit strain ( $\epsilon_{nr}$ )as function of temperature

both stresses becomes larger at higher temperature than lower one.

Figure 4 shows the variety of the elastic limit  $strain(\mathcal{E}_e)$  and proportional limit  $strain(\mathcal{E}_{pr})$  as the function of temperature. Although the elastic limit and the proportional limit stresses are in proportion with the temperature as shown in figure 3, the  $\mathcal{E}_e$  and  $\mathcal{E}_{pr}$  show to be constant at 2% and 1.2% respectively, above Af temperature, 323K . The cause of such phenomenon is due to the change of Young's modulus according with the temperature as shown in figure 5. Figure 5 shows that the Young's modulus increases up to  $5.5 \times 10^4 \mathrm{MPa}$  straightly with rising of the temperature. Therefore, the elastic limit and the proportional limit strains remain constant above 323K.

Figure 6 shows the temperature dependence of relative residual strain  $\mathcal{E}_r/\mathcal{E}_{max}$ , where  $\mathcal{E}_r$  is

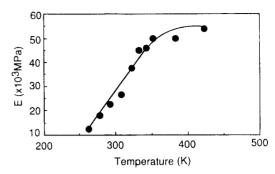


Fig. 5 Temperature dependence of Young's modulus

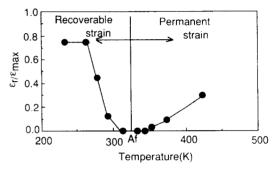


Fig. 6 The ratio of residual strain( $\mathcal{E}_{\mathbf{r}}$ ) to maximum strain( $\mathcal{E}_{\mathbf{max}}$ ) as a function of temperature.

the residual and  $\epsilon_{max}$  the maximum strain. The relative residual strain decreases abruptly to zero around the Af temperature, and it increases with rising of the temperature above Af. The residual strain below Af is the recoverable strain by heating and that above Af is the permanent strain. The permanent strain is due to slip deformation that is induced because the stress for inducing martensite approaches to the value of critical stress for the slip as the temperature becomes high.

The above features discussed in figure 3,4,5 and 6 are expected to affect markedly the fatigue properties.

### 3.2. Fatigue life curves at various temperatures.

Figure 7 shows the cyclic strain amplitude(  $\epsilon_a$ ) – fatigue life(Nf) curves at various tempera-

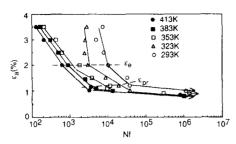


Fig. 7 Strain amplitude( $\varepsilon_a$ ) - fatigue life(Nf) curves at various temperature.

tures. Under a temperature below 383K, the curve is consist of three straight lines with different slopes. There are two intersections, one is between the short and medium life zone, and the other between medium and long life zone. Of these intersections, the upper one was verified to be coincident with the elastic limit strain( $\varepsilon_e$ ) and the lower one with the proportional limit strain( $\varepsilon_{pr}$ ) shown in figure 4. With rising of the test temperature over 383K, the curve shows to be composed of two lines with one turning point.

At the temperature of 293K and 323k, the slope of line over  $\mathcal{E}_{e}$  is very steep, the center one between the two limit strains is gentle, and that below  $\mathcal{E}_{pr}$  is very even. The temperature of 323k is the Af temperature of the specimen as mentioned previous. From this result, therefore, it is known that the change of cyclic strain at the region of martensite transformation above the  $\mathcal{E}_{e}$  does not change the fatigue life under the temperature to the Af. Moreover, the decreasing of the cyclic strain increases the fatigue life at the region between  $\epsilon_e$  and  $\epsilon_{nr}$ . Besides, below the  $\epsilon_{pr}$ , the very small change of cyclic strain changes markedly the fatigue life and hence, the  $\boldsymbol{\epsilon}_{pr}$  is almost same as fatigue limit strain.

Figure 7 also shows that the fatigue life is strongly dependent on the temperature to 323k, Af temperature at the short and medium life zone. Over the 323K, the slopes of life lines are more gentle and the temperature dependence is not so remarkable. Moreover, at the region below  $\epsilon_{pr}$ , the temperature dependence does not appear at all test temperature range except 293K.

The causes of which the temperature dependence is small and the slope is more gentle over Af temperature are probably due to large slip deformation occurring at high temperature. Finally, the fatigue life will become almost insensitive to test temperature and the pattern of the curves change as in ordinary materials above the Md point, which no martensitic transformation occurs. The fatigue life curve obtained at 413K which consists of two lines with one turning point is presumably the above case.

Figure 8 shows the stress amplitude( $\sigma_a$ ) – fatigue life(Nf) curves at various temperatures. Except the case of 413K, each curve consists of three straight lines having two intersections. The two intersections correspond to the critical stresses for inducing martensite( $\sigma_m$ ) and proportional limit( $\sigma_{pr}$ ), respectively. In the region of M – phase over the  $\sigma_m$ , the fatigue life shows to be changeable under almost constant stress amplitude. The fatigue life increases with decreasing the stress amplitude in the region between the  $\sigma_m$  and  $\sigma_{pr}$ . Moreover, in the

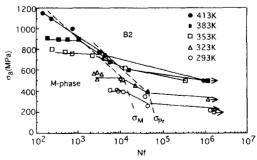


Fig. 8 Stress amplitude( $\sigma_a$ ) - fatigue life(Nf) curves at various temperature.

region of B2 phase below the  $\mathcal{E}_{pr}$ , the small change of stress amplitude results the remarkable change of fatigue life, and hence, the  $\mathcal{E}_{pr}$  is almost coincident with the fatigue limit.

Figure 8 also shows that the fatigue limit increases with rising of the temperature to 353K and the temperature dependence of fatigue limit scarcely appears over 353K. The result under 413K shows that the curve consists of two lines with one intersection as in ordinary materials. Such a phenomenon at 413K and over 353K is probably due to slip deformation as discussed in figure 7.

# 3.3 Transformation behavior due to progress of fatigue.

The damage of the shape memory effect caused by the cyclic loading will be estimated by measuring the change of the transformation enthalpy. Figure 9 shows the comparison of

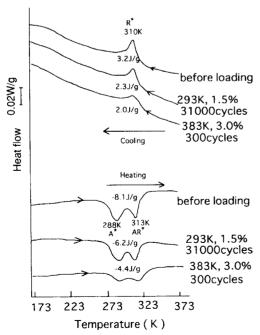


Fig. 9 Comparison of DSC curves measured in Ti - 50.9at%Ni wires failured under various loading conditions.

DSC curves measured in the fresh specimen with the 2 fatigue fractured specimens by the different cyclic loading. The loading conditions of the 2 fractured specimens are that the  $\mathcal{E}_a$  is 1.5%, N is  $3.1 \times 10^4$  cycles at 293K and the  $\varepsilon_a$  is 3.0 %, N is  $3 \times 10^2$  cycles at 383K. The top and bottom curves were measured during cooling and heating, respectively. As shown in this figure, the peaks appear at same temperature in all cases but the area of peak point shows to be smaller in cyclic loaded specimens than fresh specimen. Of the 2 loaded specimens, the area is smaller in case of loading condition of the 3.0%,  $3x10^2$  cycles at 383K. From these results, it is known that the transformation point does not change by the cyclic loading, but the quantity of transformation enthalpy decreases with the number of cyclic loading, especially, under the high temperature and large strain amplitude.

Figure 10 shows the relationship between the number of cycles to failure(Nf) and the transformation enthalpy( $\Delta H$ ) measured in the fractured specimen. The  $\Delta H$  was measured in the fractured specimens under the strain amplitude of 3.0%, 1.5% and 0.8% at 293K and 383K, respectively. In this figure, the 3.0% is the over elastic limit( $\epsilon_{pr}$ ), 1.5% is the over proportional limit( $\epsilon_{pr}$ ), and 0.8% is the below  $\epsilon_{pr}$ , under

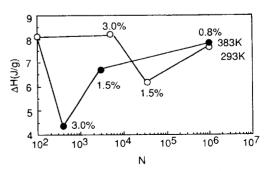


Fig.10 Effect of number of cycles(N) on the transformation enthalpy( $\Delta h$ )in fatigue tested specimen.

both 293K and 383k as shown in figures 2 and 3.

Under the 293K, the  $\Delta H$  of the specimens fractured under the 3.0% and 0.8% show to be the almost same as that of the fresh specimens. However, in the specimen fractured under the 1.5% strain amplitude at  $3x10^4$  cycles, the  $\Delta H$  decreases to 6.2 J/g from 8.1J/g of the fresh specimen.

On the other hand, under the 383K, the  $\Delta H$  decreases most in the specimen fractured at  $3x10^2$  cycles receiving 3.0% strain amplitude. Second is the specimen fractured at  $2x10^3$  cycles receiving 1.5%, and the specimen receiving 0.8% strain amplitude does not change the  $\Delta H$ , until  $10^6$  cycles.

From the above results, it is known that under the strain amplitude below the proportional limit, the damage of shape memory effect almost does not occur to  $10^6$ cycles of cyclic number, regardless of the temperature. However, under the strain amplitude over the proportional limit which the local transformation and/or slip deformation probably starts depending on the temperature, the damage changes along with the temperature, strain amplitude, and number of cycles.

Under 293K being below Af, the damage is largely dependent on the cyclic numbers, and is scarcely on the strain amplitude. However, under 383K being over Af, the damage changes predominantly by the strain amplitude.

The cause of above difference accompanied by the temperature can be explained by the residual strain discussed in figure 6. Below the Af, the residual strain after every cycle of even the large strain amplitude is recoverable, and hence, it is not accumulate to occur the damage. However, above the Af, the residual strain after every cycle is the permanent and hence, it is accumulated every cycle to occur damage.

### 4. Conclusions

The fatigue tests were conducted at various temperatures for Ti – 50.9at%Ni wires using a rotary bending fatigue tester specially designed for wires. The test results were discussed in connection with the static deformation behavior. Moreover, the damage of the shape memory effect due to the progress of fatigue was investigated by the DSC measurement after fatigue test. The results obtained are as follows:

- 1) Above the Af temperature, the elastic limit strain and the proportional limit strain appear to be almost constant regardless of the temperature, while the elastic limit and the proportional limit stresses increase in proportion with test temperature in the range from 233k to 423k.
- 2) Under the test temperature below or near the Af, the strain amplitude  $(\mathcal{E}_a)$  fatigue life(Nf) curve was found to consist of 3 straight lines with 2 turning points. Of the 2 turning points, the upper one was coincident with the elastic limit strain and the lower one with the proportional limit strain. However, with rising of the test temperature over Af, the pattern of the curves changed gradually to the composition of 2 straight lines as in ordinary materials.
- 3) The  $\epsilon_a$  Nf curves shifted depending on test temperature. In the short and medium life zones, the higher the temperature was, the shorter the fatigue life appeared. However, in the long life zone, above the Af temperature, the fatigue life was hardly affected by the temperature.
- 4) The stress amplitude( $\sigma_a$ ) Nf curve show ed different appearances at B2 and M phase region respectively. In the B2 region, Nf increas ed with decreasing of the  $\sigma_a$  down to fatigue limit, while Nf increased under constant  $\sigma_a$  in the M phase region. Moreover, the fatigue

limit increased with the rising of the test temperature.

- 5) The fatigue limit appeared to be almost coincident with the proportional limit stress.
- 6) The damage of shape memory effect due to the progress of fatigue is largely dependent on the cyclic number, scarcely on the strain amplitude below Af temperature, while it is affected predominantly by the strain amplitude above Af.

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