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FATIGUE BEHAVIOR OF  
A COPPER BASED SHAPE MEMORY ALLOY

BY

Inyoung Song

A THESIS

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## ABSTRACT

### FATIGUE BEHAVIOR OF A COPPER BASED SHAPE MEMORY ALLOY

BY  
Inyoung Song

Fatigue properties of Cu-62.78 Zn-3.75 Al-0.01 Zr shape memory alloy with different annealing times were studied by using R. R. Moore type rotating fatigue testing machine. The fracture mode was also studied by using optical and electron microscopes. S-N curves were obtained at room temperature and at  $-81^{\circ}\text{C}$ . The fatigue life in the martensitic state increased as annealing time decreased in the austenitic state. In the austenitic phase the fatigue resistance showed little dependence on the annealing time, and the results generally showed very low fatigue life. Fatigue fracture predominantly occurred along grain boundaries of austenitic phase, whereas some took place through grains. The longer fatigue life was obtained with finer grain size in the martensitic state.

## ACKNOWLEDGEMENTS

I would like to dedicate this work to my late mother. I wish to extend my sincere appreciation to Dr. C. M. Hwang for his advice and support. Thanks are due to Dr. C. M. Wayman for the materials supplied ,and to my family and to my fellow students for their encouragement and the help. Finally, I thank God through Jesus Christ, for none of this would have been possible without His guidance.

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## I. INTRODUCTION

Greninger and Mooradian<sup>1</sup> introduced the shape memory effect in 1938. However, only recently work has begun on applications.<sup>2</sup> The shape memory effect was observed in other alloys and these phenomena were reported with different names.<sup>3,4,5,6</sup> Of these materials, most applications depend on Ni-Ti type and Cu based alloys. Especially, the Cu based alloys are relatively inexpensive to produce and fabricate into numerous forms.

The shape memory effect occurs at temperatures where the martensitic phases, caused by either thermal stress reorientation or by mechanical stress, are stable in the absence of stress; that is, the memory effect is observed upon heating.

Another way of shape memory effect, shape recovery, takes place when stress induced martensite phase transforms to parent phases in the thermally unstable state upon unloading. Such mechanisms give rise to high strains up to 15 % without any conventional plastic deformation such as slip or twinning.<sup>7</sup> Consequently, fatigue resistance is expected as Melton et. al.<sup>20</sup> reported. Recent works on the various alloys, Cu-Al-Ni,<sup>8,9,10</sup> Ni-Ti,<sup>11</sup> Au-Cd,<sup>12</sup> Cu-Zn-Al,<sup>13</sup> show some results of good fatigue life.

Recently, Janssen et. al.<sup>13,14</sup> reported that grain refinement together with a sharp texture improve quite effectively the fatigue properties. So that several attempts<sup>14-18</sup> have been made to refine the grain sizes to avoid grain coarsening, which generally is generated in the manufacturing process, leads to the result of weakening in the mechanical properties of the materials.

In addition to these attempts, Sugimoto et. al.<sup>19</sup> reported that introducing small amount of Ti to Cu-Al-Ni shape memory alloy was effective in improving not only the brittle condition but also the grain refinement. Addition of Ti resulted in fine precipitates in the B<sub>2</sub> matrix so that the grain boundary segregation of impurities was suppressed due to the strong affinity with oxygen. The same mechanism was found from the addition of Zr in improving the brittleness.

In the present study, the alloy Cu-Zn-Al-Zr was used to investigate the fatigue behavior relative to grain size and annealing time either in the martensitic condition or in the B<sub>2</sub> condition with low cyclic loading. The fatigue substructures and microstructures were examined by optical and electron microscopes.

## II. EXPERIMENTAL PROCEDURES

A Cu-26.78 Zn-3.75 Al-0.01 Zr alloy was used in this investigation. The alloy data are shown in the table 1. To compare grain sizes, small bulk specimen were annealed in the evacuated quartz tube for 5 minutes at 800°C. This specimen was then polished using 5  $\mu\text{m}$  alumina powder. The polished surface was etched by 30 % nitric acid etchant. The surface was photographed at 100 times magnification. The same specimen was again evacuated and then charged into the furnace for 5 minutes at 800°C. The quenching process using ice water was followed immediately and the etched portion was photographed with 100 times magnification to compare the grain size. The same procedure, evacuation, heating, quenching and photographing, was iterated by changing only the specimen holding times, 30 minutes and 60 minutes, in a furnace as in the Fig.1.

For the fatigue test, the samples were machined to make the rotating beam specimen. Fig.2 shows specimen specification. The specimens were heat treated on the conditions used for grain observation for the B<sub>2</sub> structure. All specimens were polished on the middle portion to avoid error of crack initiation from scratches.

Those as quenched specimens were applied with

different loads on R. R. Moore type high speed fatigue machine during cooling by dry ice to get the martensitic state condition. In this test the voltage of the testing machine was set at constant value to minimize the deviations of strain which might be generated from the different straining speed.

The loads applied were determined in the range of linear region, elastic region, on the stress-strain curve. (Fig.3) This stress-strain curves were obtained from the tensile test by using floor type Instron. The fatigue fractured surface was observed by Hitachi S-415A scanning electron microscope.

Crack propagation mode was obtained from the microcrack observation by using optical microscope when the specimen testing was interrupted at the time of half of the usual fracture cycles in the lowest load.



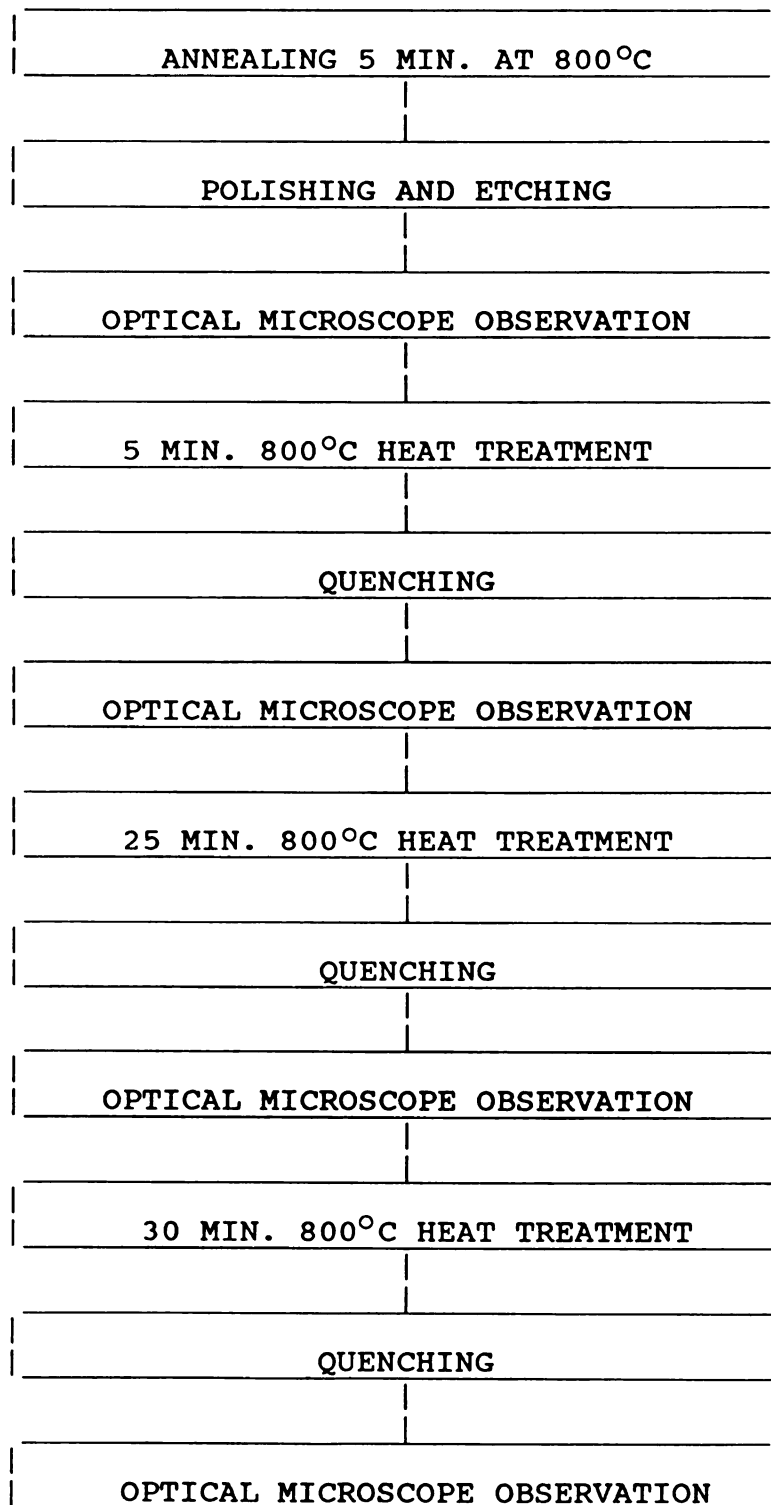
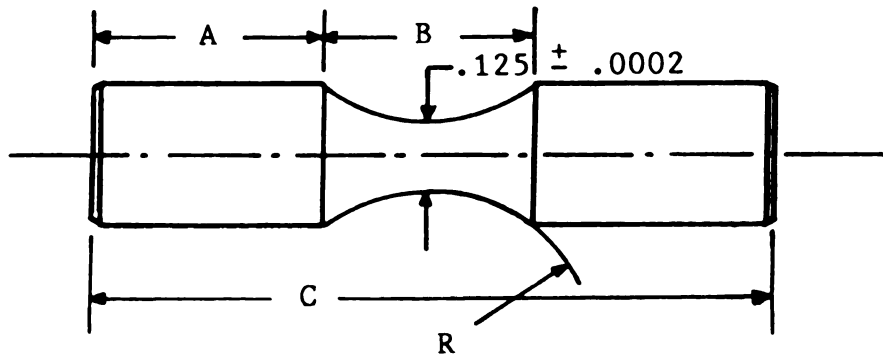


FIG. 1 GRAIN SIZE OBSERVATION FLOW DIAGRAM



DIA. D	A	B	C
.230	$\frac{31}{32}$	$\frac{3}{4}$	$2\frac{11}{16}$

FIG. 2 ROTATING BEAM FATIGUE SPECIMEN

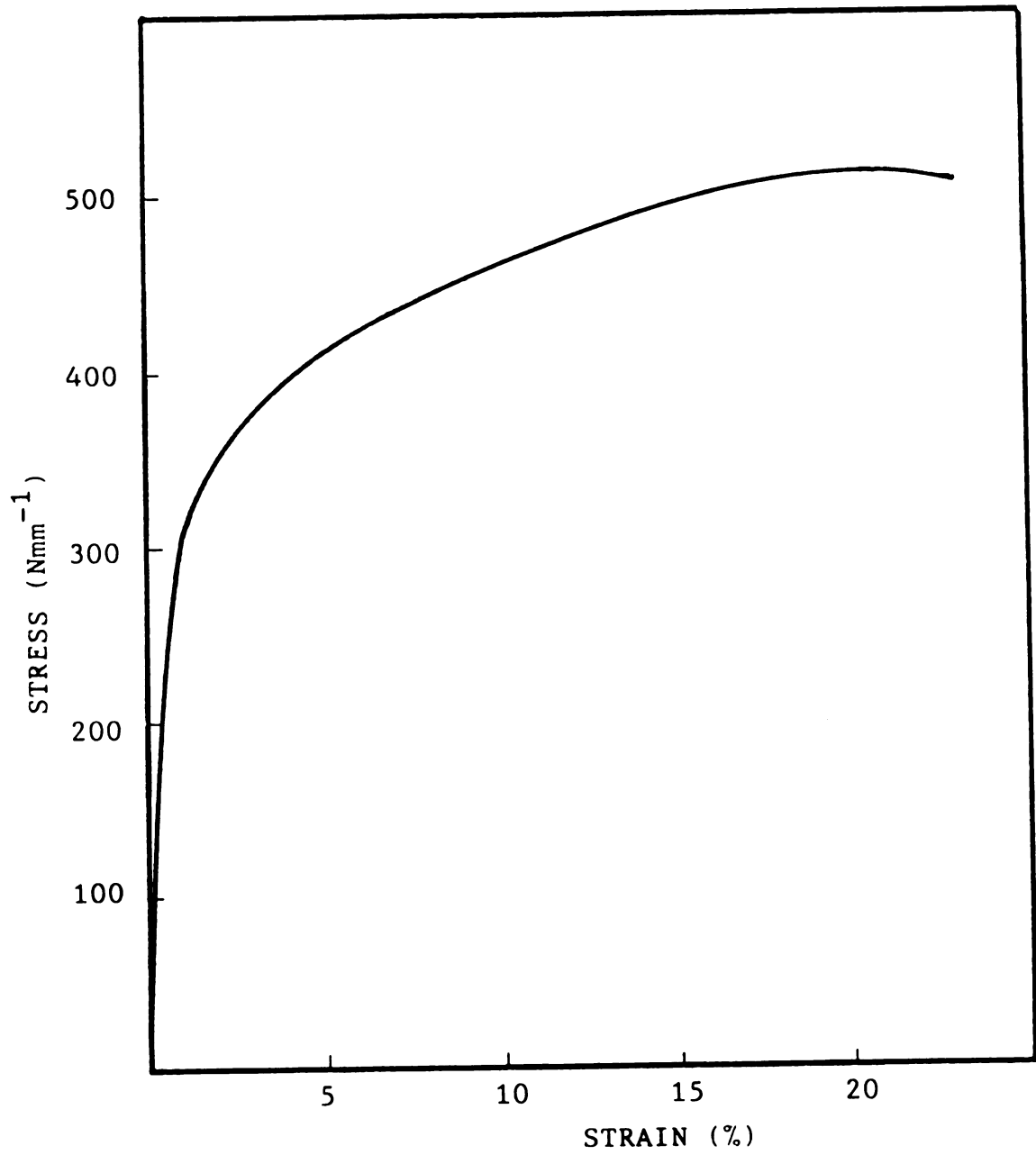


FIG. 3 STRESS-STRAIN CURVE OF Cu-Zn-Al-Zr

TABLE 1 ALLOY DATA

COMPOSITION		Wt %		Ms	STRUCTURE
Cu	Zn	Al	Zr	°C	
69.37	26.78	3.75	0.01	-8	B <sub>2</sub>

TABLE 2 GRAIN SIZES RELATIVE TO ANNEALING TIME

ANNEALING TIME	TEMP.	GRAIN SIZE
5 MIN	800 °C	40 μm
30 MIN	800 °C	60 μm
60 MIN	800 °C	70 μm

### III. RESULTS AND DISCUSSION

#### Grain Size Observation

The grain size is expressed as the function of annealing time. Fig.4 shows second phases with slow cooling after 5 minutes annealing at 800°C. Fig. 5 shows the B<sub>2</sub> structure of parent phase. From the Fig.5 to Fig.7, the grain growth is shown as the annealing time increases. The grain size was determined by the linear intercept method.<sup>21,22</sup> The measured grain sizes were 40 μm, 60 μm and 70 μm for the aging time 5, 30 and 60 minutes, respectively as in the table 2.

#### Fatigue Test

Fig.8 shows S-N curves of Cu-Zn-Al-Zr for fatigue fractured samples in the martensitic state. The fracture cycles decreased as the heat treatment time increased so grain growth resulted. No fatigue limit is observed in the test. The annealing time dependence of fatigue life of martensitic phase is shown in the Fig.9. For the 97 MPa stress fatigue cycle is almost linealy proportional to the annealing time. So the fatigue cycle is expected to be increased with decreasing annealing time which leads to fine grains. The stress for fracture in around 10<sup>5</sup> cycles



FIG. 4 SEM MICROGRAPH OF Cu-Zn-Al-Zr WITH SLOW COOLING AFTER 5 MIN. ANNEALING AT  $300^{\circ}\text{C}$



FIG. 5 OPTICAL MICROGRAPH B2 PARENT PHASE OF Cu-Zn-Al-Zr  
ANNEALED FOR 5 MIN. AT 800°C (X 100)

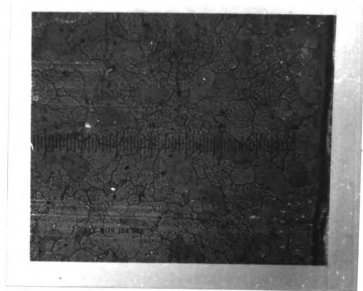


FIG. 6 OPTICAL MICROGRAPH OF Cu-Zn-Al-Zr ANNEALED FOR 30 MIN. AT 800°C (X 100)



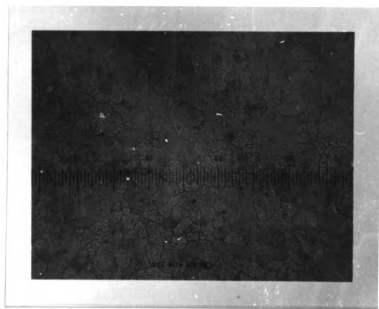


FIG. 7 OPTICAL MICROGRAPH OF Cu-Zn-Al-Zr ANNEALED FOR 60 MIN. AT 800°C (X 100)

is very low in the load less than 100 MPa. As Melton and Mercier<sup>20</sup> pointed out that this is an order of magnitude lower than the others reported for Cu-Zn-Al strip specimens tested in pulsating tension.<sup>7</sup>

This is explained by two facts. Wield and Gillam<sup>24</sup> said that a round specimen has more provable grains of several orientations in a given cross section than a strip one does. Also it is reported that round tensile specimens of polycrystalline Cu-Zn alloy show higher yield stress and higher work hardening rate than flat specimens.<sup>25</sup> This leads that more permanent deformation is caused by tension-compression cycling rather than pulsating tension.

It is apparent from the comparison that 5 min. 30 min. and 60 min. heat treated samples have lower order of fracture cycle in the austenitic state than in the martensitic structures. This is well agreed with the other workers.<sup>13,14,17,23,27</sup> The samples in the austenitic state show almost the same order of fatigue life with annealing time. This follows that the primary role to increase fatigue life depends on not the grain size but the martensitic structure.

The cyclic strain hardening always appears during the initial stage of the fatigue test though it is only effective within 100 cycles.<sup>26</sup> This cyclic hardening

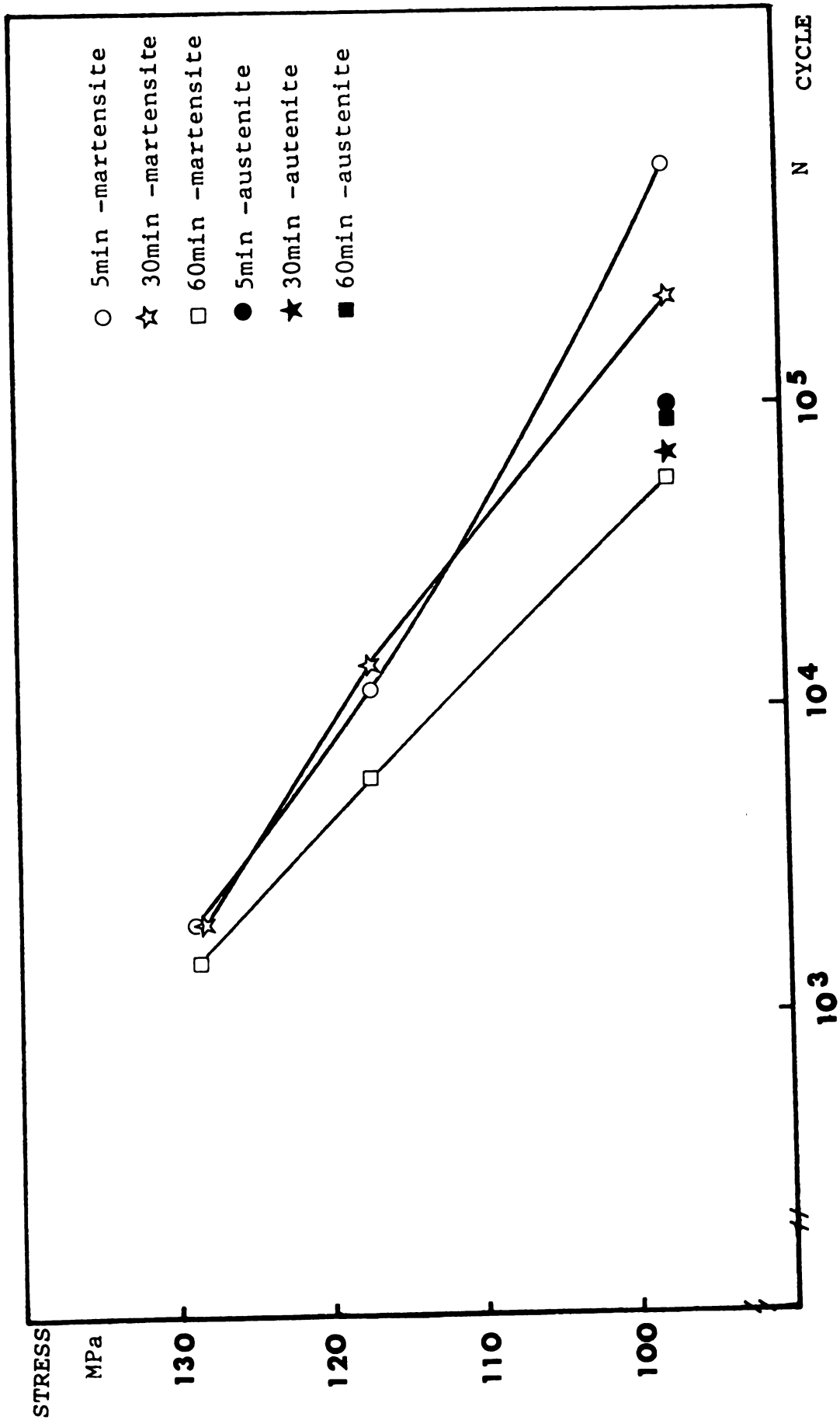


FIG. 8 FATIGUE LIFE OF Cu-Zn-Al-Zr RELATIVE TO STRESS AMPLITUDE

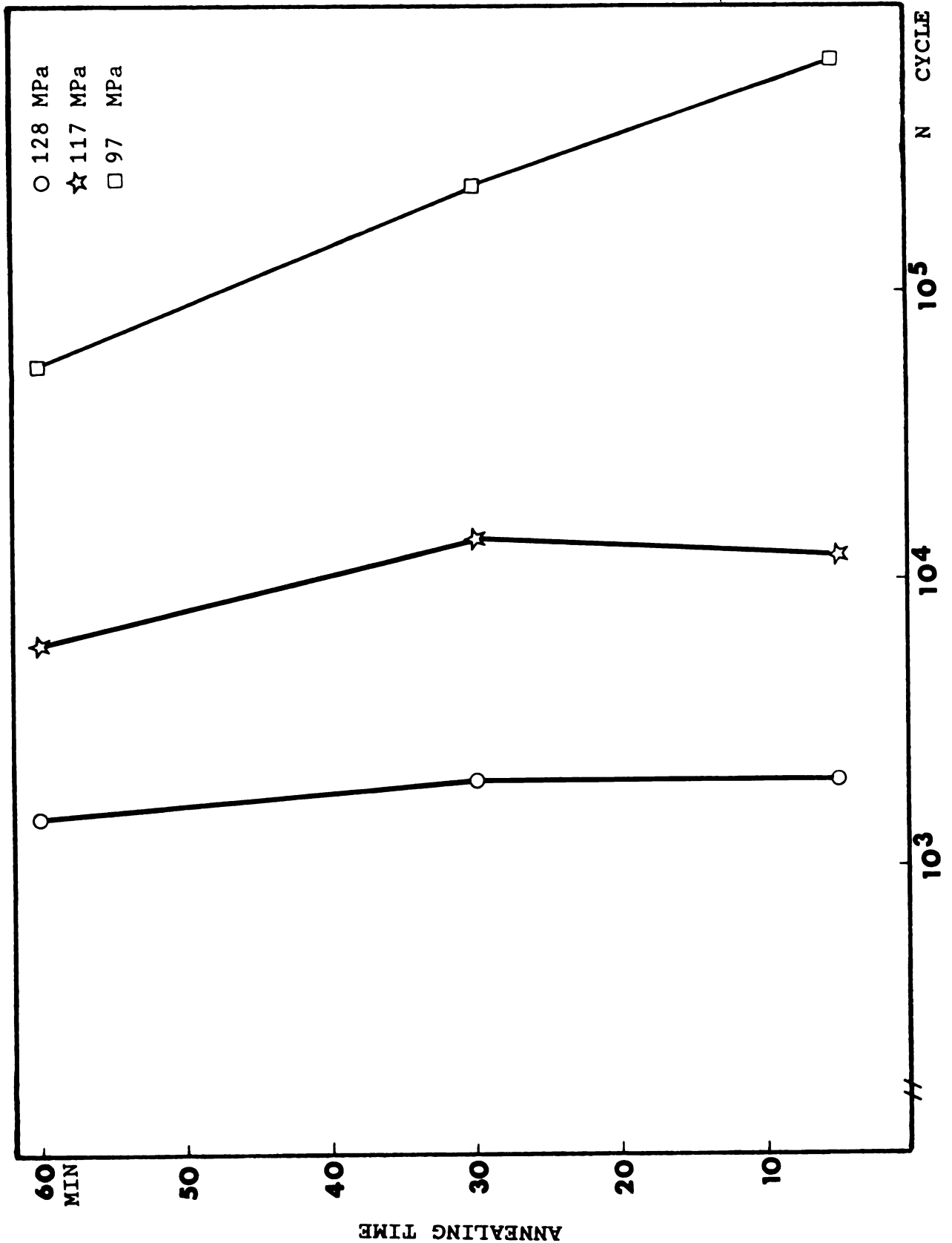


FIG. 9 FATIGUE LIFE OF MARTENSITIC Cu-Zn-Al-Zr RELATIVE TO ANNEALING TIME

occurs with the interactions between matrix dislocations and martensite plates as well as the other intersecting martensite plates generated.<sup>14</sup> Janssen et. al.<sup>27</sup> said that the cyclic strain hardening occurs much more extent in the coarse grained martensite specimens than in the fine grained ones.

It is assumed that cyclic strain hardening does not affect much to the fatigue resistance; that is, the longer fatigue life in the martensitic state than in the austenitic state is not due to the cyclic strain hardening. It is likely that the deformation of materials is associated with stress induced martensite in which dislocation piles up the interchange of martensite variants and the displacement of internal defect boundaries in the martensite, as the stress concentrates on.<sup>6</sup> The irreversible stress induced martensite increased with the increase of fatigue cycle such that the density of dislocation increased. This gives rise to incomplete stress relaxation upon unloading.<sup>26</sup>

As the microcrack localized by stress relief, the reduction of cross sectional area is resulted so that the stress level comes to go up.<sup>26</sup> Accordingly, as far as the applied stress does not exceed a critical value, the microcrack propagation does not occur for the elastic deformation and longer fatigue life will be expected in the



FIG. 10 ROOM TEMP. OPTICAL MICROGRAPH SHOWING INTERGRANULAR FRACTURE OF FATIGUED Cu-Zn-Al-Zr IN THE MARTENSITIC STATE  
-AFTER  $1 \times 10^5$  CYCLE ON THE STRESS OF 97 MPa

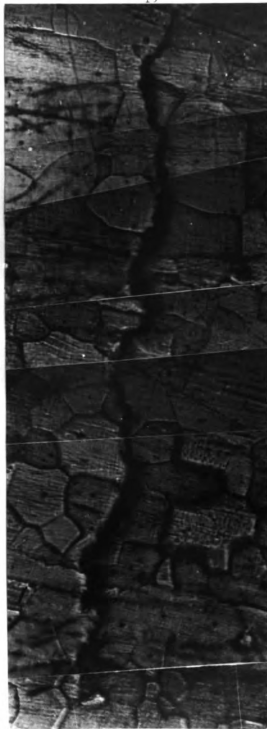


FIG. 11 MAGNIFIED ROOM TEMP. MICROGRAPH SHOWING INTERGRANULAR  
FRACTURE OF FATIGUED Cu-Zn-Al-Zr IN THE MARTENSITIC  
STATE  
- AFTER  $1 \times 10^5$  CYCLE ON THE STRESS OF 97MPa

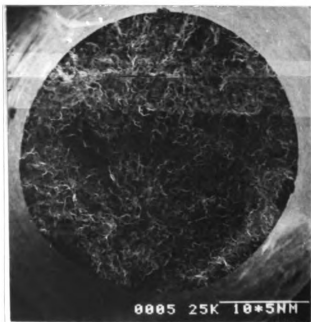
martensitic state.

The microcrack is initiated along the grain boundaries as Fig. 10 shows. Fig.11 is the magnified picture of Fig.10 and some parts exhibits the transgranular mode. This transgranular cracking, indicated as white lines in the Figures 15, 16 and 17, the fatigue striations, is consistent with Janssen<sup>14</sup> who said that some of crack propagation occurs transcrystalline for the martensite samples. Fig. 12, 13 and 14 show SEM photographs taken from fractured surfaces of fatigue tested specimens and these pictures show that the fracture was caused by fatigue not by tensile load as Gough<sup>28</sup> illustrated. All of these show the fatigue fracture mode no matter in the what kind of state, martensite or austenite, it was fractured; that is, there was no sign of the original necking and subsequent separation at the center, followed by a sliding of shearing action at the outside, giving rise to the usual cup and cone failure. In both state of specimens fracture took place along the B<sub>2</sub> grain boundaries, but somewhat did through the grains, transgranular fracture.

The ductile and intergranular fracture is dominant on the specimens fractured in the martensitic state.<sup>6</sup> The possible reasoning is that the martensite variants are interchanged with stress concentration and this yields microcrack initiation around grain boundaries as Oshima<sup>26</sup>



a



b

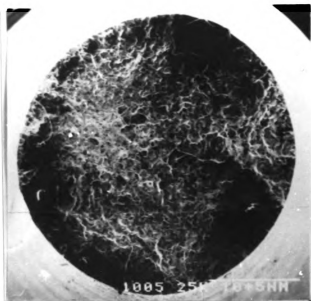
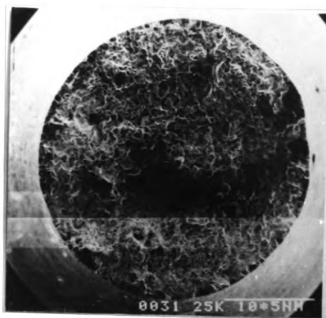


FIG. 12 SEM MICROGRAPH OF FRACTURED SURFACES OF  
Cu-Zn-Al-Zr  
(a) FRACTURED IN THE MARTENSITIC STATE-ANNEALED  
FOR 5 MIN. AT 800°C  
(b) FRACTURED IN THE AUSTENITIC STATE-ANNEALED  
FOR 5 MIN. AT 800°C

a



b

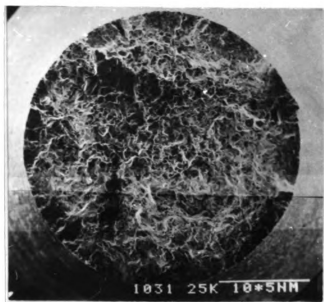
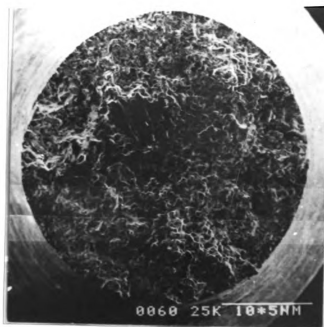


FIG. 13 SEM MICROGRAPH OF FRACTURED SURFACES OF  
Cu-Zn-Al-Zr  
(a) FRACTURED IN THE MARTENSITIC STATE-ANNEALED  
FOR 30 MIN. AT 800°C  
(b) FRACTURED IN THE AUSTENITIC STATE-ANNEALED  
FOR 30 MIN. AT 800°C

a



b

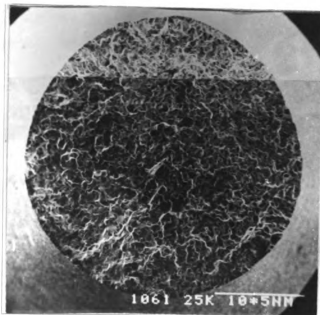


FIG. 14 SEM MICROGRAPH OF FRACTURED SURFACES OF  
Cu-Zn-Al-Zr  
(a) FRACTURED IN THE MARTENSITIC STATE-ANNEALED  
FOR 60 MIN. AT 800°C  
(b) FRACTURED IN THE AUSTENITIC STATE-ANNEALED  
FOR 60 MIN. AT 800°C

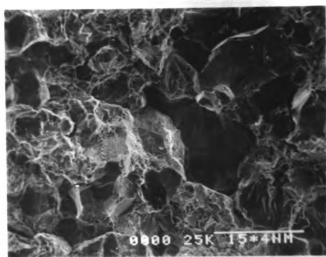


FIG. 15 SEM MICROGRAPH OF FATIGUE FRACTURED SURFACE OF  
Cu-Zn-Al-Zr ANNEALED FOR 60 MIN. AT 800°C SHOWING  
GRAIN BOUNDARY SEGREGATION  
- 97 MPa IN THE MARTENSITIC STATE

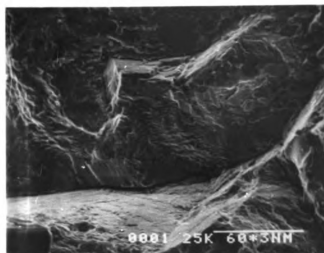


FIG. 16 MAGNIFIED SEM MICROGRAPH OF FATIGUE FRACTURED SURFACE OF Cu-Zn-Al-Zr ANNEALED 60 MIN. AT 800°C SHOWING GRAIN BOUNDARY SEGREGATION  
- 97 MPa IN THE MARTENSITIC STATE

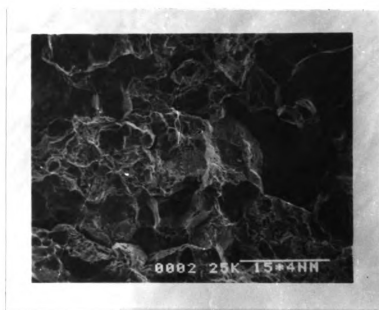


FIG. 17 SEM MICROGRAPH OF FRACTURED SURFACE OF Cu-Zn-Al-Zr  
ANNEALED FOR 60 MIN. AT 800°C SHOWING MIXED  
CRACKING  
- 97 MPa IN THE MARTENSITIC STATE

said. The martensite almost disappears after cracking, proving that accommodated internal stresses exist along grain boundaries.

Fig.15 and Fig.16 show that the main mode of crack propagation to fracture is intergranular type though some of the transgranular mode is shown in the Fig.15. The fatigue striations are shown in the Fig.16. In the Fig.17, mixed type of crack mode is shown when the surface is viewed from the different angle from the Fig.15. and some of the grain coalescence phenomena are seen as annealing time increased.

## SUMMARY

- (1) Austenite grain sizes after 5 minutes, 30 minutes and 60 minutes annealing time at 800°C are 40  $\mu\text{m}$ , 60  $\mu\text{m}$  and 70  $\mu\text{m}$ , respectively.
- (2) Fatigue behavior of the martensitic phase of the Cu-Zn-Al-Zr alloys studied by rotating bending testing shows fatigue lives of around  $10^5$  cycles at stresses of less than 100 MPa. No fatigue limit is obtained in the both phases, martensite and austenite.
- (3) The longer fatigue life showed in the martensitic state than in the austenitic state. As the annealing time is decreased, the fatigue cycle is increased.
- (4) Fatigue substructure of the parent phase showed brittle type fracture.
- (5) Most fatigue fracture takes place along  $B_2$  grain boundaries showing intergranular cracking.



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