JOURNAL DE PHYSIQUE IV
Colloque C7, supplément au Journal de Physique III, Volume 5, novembre 1995

Fatigue of High Purity Copper Wire

N. Tanabe, A. Kurosaka*, K. Suzuki and O. Kohno

Materials Research Laboratory, Fujikura Ltd. 1-5-1, Kiba, Koto-ku, Tokyo 135, Japan
** Advanced Technology R&D Center, Fujikura Ltd. 1-5-1, Kiba, Koto-ku, Tokyo 135, Japan*

Abstract The fatigue properties of 7N, OFC and Tough-Pitch copper wires were evaluated by a rotational bending method. 7N copper wires, having $RRR_{300K/4.2K}$ of 7600, were produced by electro-refining, vacuum melting, zone-refining followed by suitable drawing processes, using 4N class commercial OFC plates as the starting material. The fatigue failure cycles of 7N copper increased with an increased pause period imposed during the fatigue test after 5×10^6 cycles. Remarkable recrystallizations initiated from the wire surface occurred in 7N copper after the pause period in the fatigue test. Strains accumulated during fatigue are considered to act as the driving force. It is expected that 7N copper can recover from fatigue by itself during practical service use and show excellent fatigue resistance.

1. INTRODUCTION

The annealing properties of copper change greatly when it is purified up to 6 or 7N level [1, 2]. The annealing temperature of 7N class high purity copper is less than 100°C and not only recovery of point defects or dislocations but also recrystallizations occur even at room temperature after cold work [3]. The activation energy for recrystallization [2-7] in 7N class zone-refined copper was estimated as 0.82 eV [2] from relations between tensile strength and measuring temperature. This is very low compared with the 1.82 eV [7] of usual Tough Pitch copper. This characteristic of high purity copper makes it appreciable as material for electric wires and flexible printed circuits (FPC), because it can be "self-annealed" and its electrical and mechanical properties can modify themselves after fabrication.

We further expected that high purity copper would have excellent fatigue resistance. Defects and strains accumulated by fatigue could rectify themselves by recrystallization. In this study, the fatigue properties of 7N class zone-refined copper wire were investigated.

2. EXPERIMENTAL PROCEDURES

2.1 Purification and wire drawing

The details of fabrication of the high purity copper wires have been described elsewhere [2]. 4N class commercially available oxygen free copper (OFC) plates were used as the starting material. A Φ16.5 mm rod was produced by electro-refining in a sulphuric acid bath, vacuum melting under $5x10^{-2}$ Pa and zone-refining with a zone transfer rate of 50 mm/hr. The rod was swaged to 5.0 mm and drawn to 2.0 mm in diameter. Etch-cleanings were conducted in the procedure in order to remove contaminations embedded in the wire surface during the cold work. The sample wire showed $RRR_{300K/4.2K}$ of 7600 (value after size effect correction). According to GDMS analyses, the nominal purity of the refined copper wire was in 7N class (hereafter we refer to purified copper as 7N copper).

2.2 Fatigue tests

Table 1 Tensile strength of sample wires.

Sample wires Φ2.0 mm	As cold worked		As annealed*
	Reduction (%)	Tensile strength (MPa)	Tensile strength (MPa)
Tough Pitch Cu	93.4	456	229
OFC	93.4	452	228
7N-Cu	98.5	422	189

*500℃ x 2hrs

Φ2.0 mm cold worked wires of the 7N copper, OFC and Tough Pitch copper were presented for fatigue testing. The OFC and Tough-Pitch copper wires were drawn from Φ8.0 mm wire rods. The tensile strength of sample wires is shown in Table 1. Fatigue tests were carried out at 25°C in an air-conditioned room by a rotational bending method shown in Fig.1. In the test, a constant bending stress is applied repeatedly to a testing length of a wire until it breaks by failure. Relations between load and applied stress are given by the following equation,

$$\sigma_w = 16LW/\pi d^3$$

where

σ_w	applied stress
W	load
L	span of bending beam (testing length)
d	diameter of a sample wire.

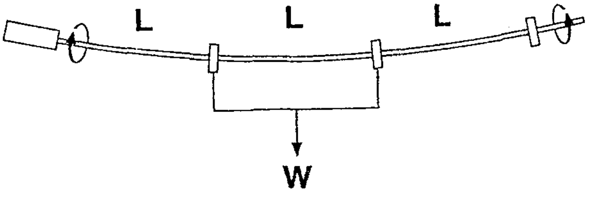

Fig.1 Schematic representation of rotational bending fatigue test.

The length of a sample wire was 350 mm and L of the wire was 100 mm. The rotation rate was fixed at 4000 rpm (corresponding to 67 Hz). Relations between applied stress (MPa) and induced strain (μ ST) at the surfaces of sample wires were directly measured by strain-gauges as follows,

$\varepsilon_w = 8.88 \cdot \sigma_w$ (7N copper)

$\varepsilon_w = 7.34 \cdot \sigma_w$ (OFC)

$\varepsilon_w = 6.95 \cdot \sigma_w$ (Tough Pitch copper)

3. RESULTS

3.1 Recrystallizations at room temperature

Fig.2 shows microstructures of longitudinal sections of Φ2.0 and 0.50 mm 7N copper wires aged for 1 day and 4 months at room temperature after drawing. Remarkable recrystallizations are observed in samples after 4 months. Fig.3 shows changes of tensile strength in the wires as a function of time after drawing. It decreased to almost full-annealing level after 2 months due to recrystallizations. The rate of recrystallization was higher in the Φ0.50 mm wire, because of the greater strains accumulated during cold work.

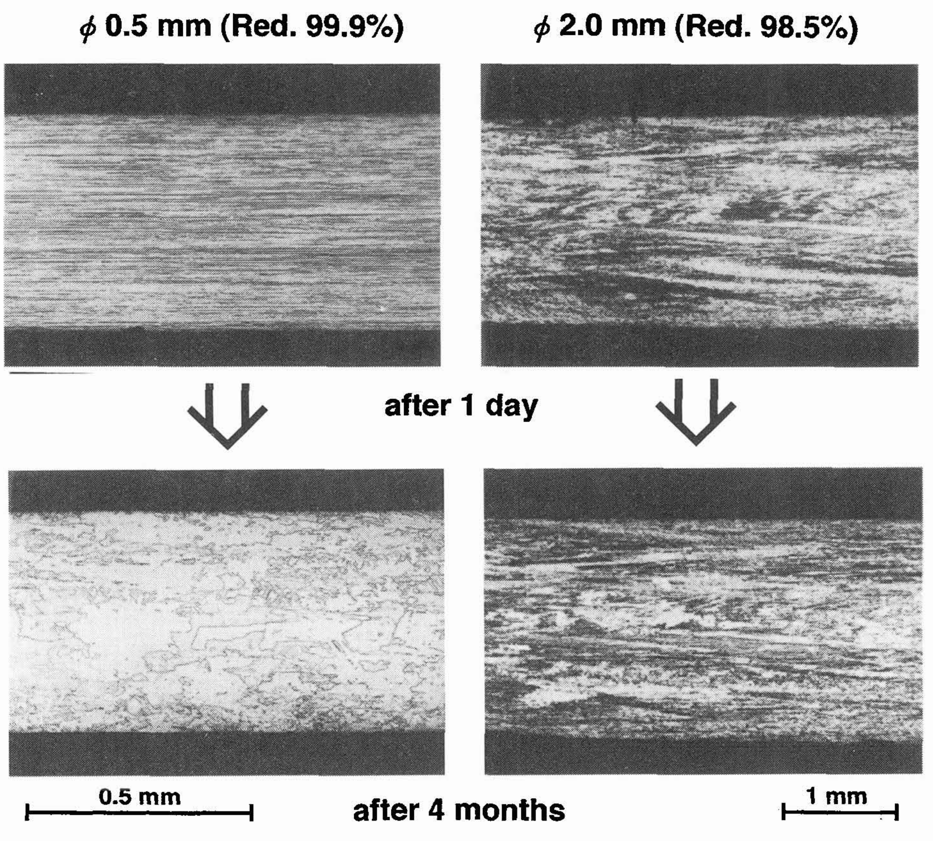

Fig.2 Microstructures of longitudinal sections of Φ 2.0 and 0.50 mm 7N copper wires aged for 1 day and 4 months at room temperature after drawing.

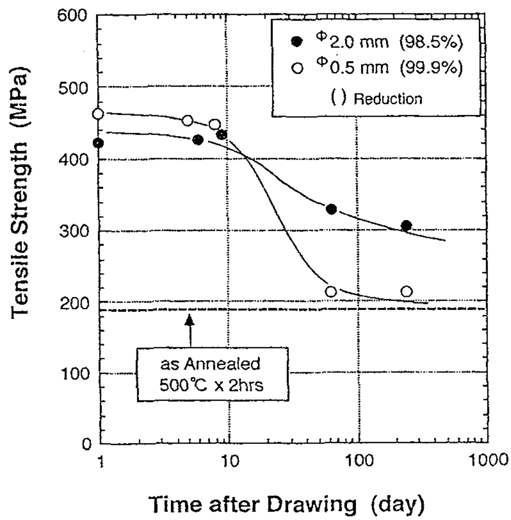

Fig.3 Tensile strength of Φ 2.0 and 0.50 mm 7N copper wires as a function of aging time at R.T. after drawing.

3.2 Fatigue properties

Fig.4 shows S-N curves (relations between applied stress and fatigue failure cycles) of Φ2.0 mm cold worked 7N, OFC and Tough-Pitch copper wires measured within 2 months after drawing. The fatigue limit stress of 7N copper was obtained as 53.9 MPa, which was the smallest value of the samples, because 7N copper is rather soft and bends easily under a bending stress compared with other samples. Fig.5 shows rearranged relations of Fig.4 as relations between induced strain and fatigue failure cycles. In the relations, the fatigue properties are almost the same as for the other samples.

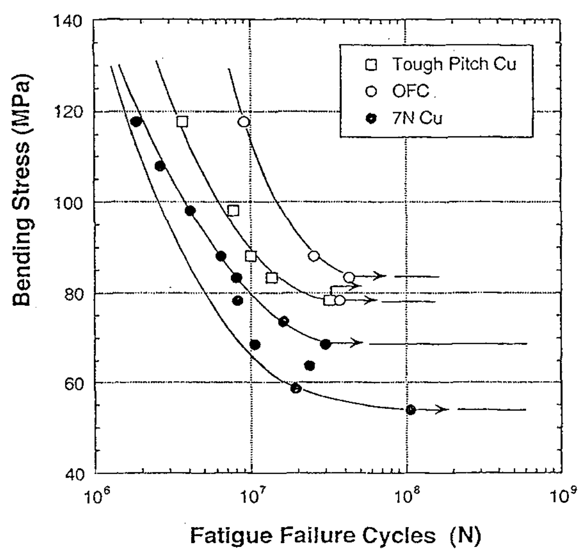

Fig.4 S-N curves of Φ 2.0 mm cold worked 7N, OFC and Tough Pith copper wires.

In the fatigue property mentioned above, we did not find any strong points for 7N copper. But we found that remarkable recrystallizations occurred in 7N copper wires after fatigue tests. They initiated from the wire surface where the largest strains are induced by bending. The wire rotation rate of 4000 rpm was chosen as the lowest rate of our testing machine. But in these conditions, the operation period for 10^7 cycles, which is regarded as infinite for fatigue cycles in general, is only 1.7 days. It is higher than recrystallization rate at room temperature.

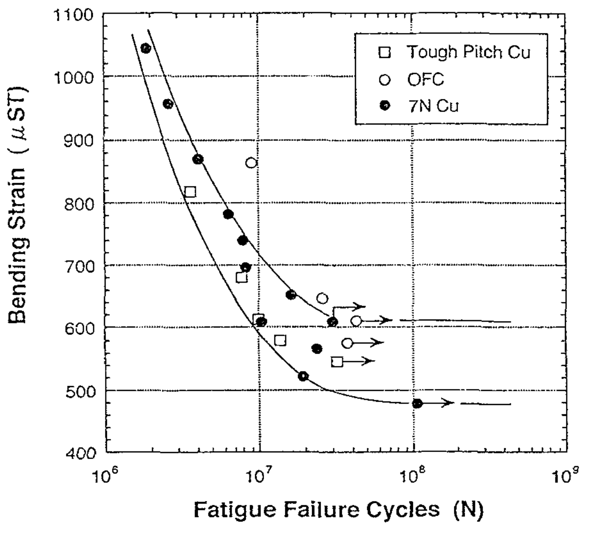

Fig.5 Re-arranged relations of Fig.4 as relations between induced strain and fatigue failure cycles.

3.3 Effects of recrystallization on fatigue

In order to investigate the effects of recrystallization on fatigue properties, we set a pause period in the fatigue test as shown in Fig.6. When the fatigue cycle reached 5x10^6, the test machine operation was stopped for 1, 10 and 30 days as a pause period. During this period the samples were kept at 25°C in the air conditioned room. After the period, the test machine operation was started again and continued until fatigue failure occurred. Tests were carried out on 7N and Tough Pitch copper wires at stresses calculated to give the same strains of 700 μST at each wire surface.

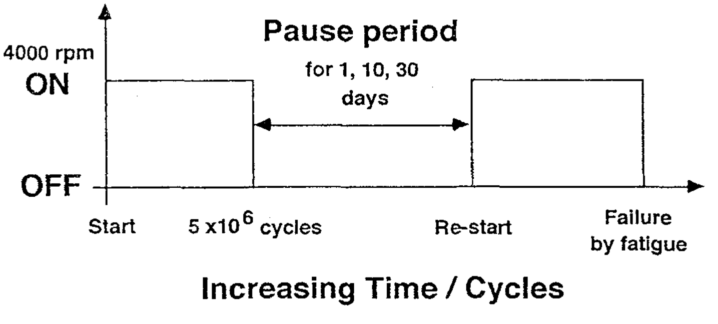

Fig.6 A pause period settled in the fatigue test.

Fig.7 shows relations between total fatigue failure cycles and the pause period. In 7N copper, the fatigue failure cycle increased up to twice with an increasing pause period, although no increase was recognized in Tough Pitch copper. Fig.8 shows microstructures of longitudinal sections of 7N and Tough Pitch copper wires which have spent 10 days as a pause period after the initial fatigue cycles. Remarkable recrystallizations were observed in the surface area of 7N copper. On the other hand, fibre structures formed by drawing were still observed in the core area and in all areas of Tough Pitch copper. Fig.9 shows hardness distributions of sample wires. During the pause period, hardness of the surface area in 7N copper decreased to full-annealing level, while no change was observed in Tough-Pitch copper. The results of the hardness measurements correspond to the microstructures shown in Fig.8.

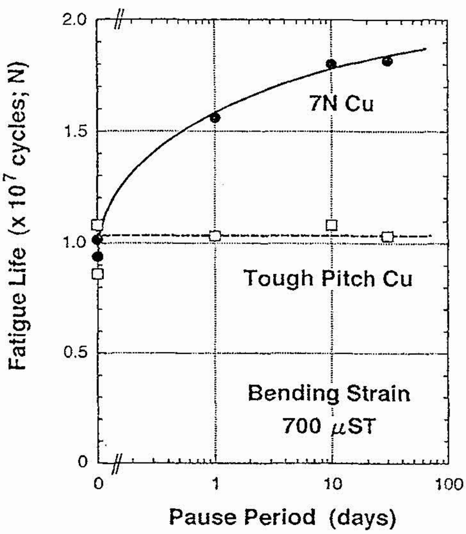

Fig.7 Relations between total fatigue failure cycles and a pause period.

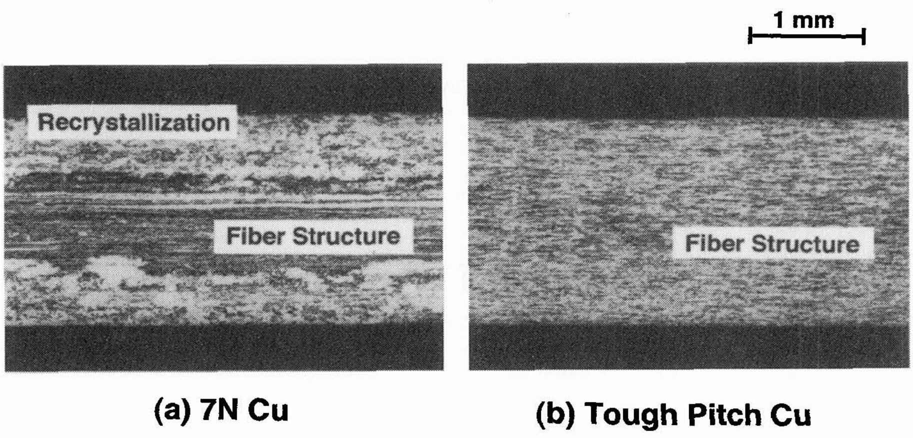

Fig.8 Microstructures of longitudinal sections of (a)7N and (b)Tough Pitch copper wires aged for 10 days at R.T. as a pause period after $5x10^6$ fatigue cycles.

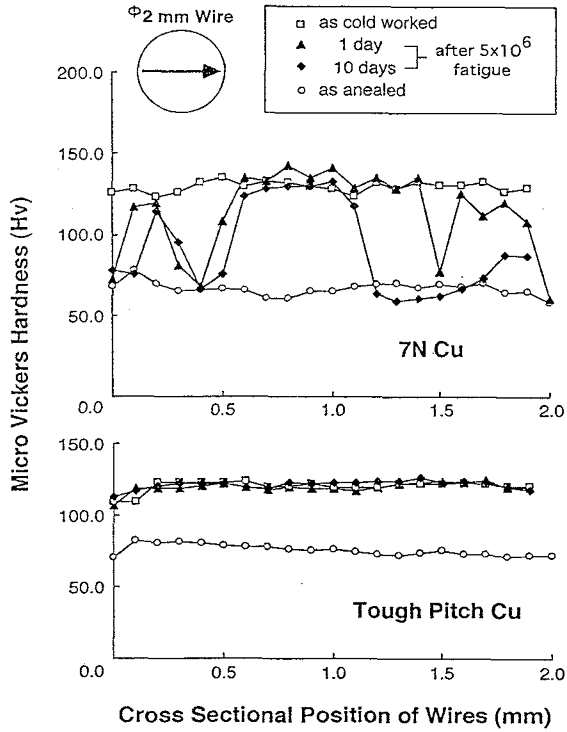

Fig.9 Hardness distributions of wire cross-section in 7N and Tough Pitch copper wires.

4. DISCUSSION

Recrystallizations and consequent softening were observed in 7N copper wire aged at room temperature after cold work. The extent depended on reduction or strains induced by cold work. Recrystallizations at the wire surface where the largest strain was induced during fatigue testing and a significant increase of fatigue life as a result were also observed in fatigue tests performed at room temperature. It is considered that strains accumulated during cold work or fatigue enhance recrystallization in 7N copper. The rate of recrystallization is high enough compared with the service life of the material. This character gives a unique property to 7N copper. It can recover from fatigue by itself during use and show excellent fatigue resistance.

5. CONCLUSION

From fatigue tests carried out by the rotational bending method at room temperature on 7N, OFC and Tough-Pitch copper wires, the following conclusions were obtained.

(1) The fatigue failure cycles of 7N copper increased with an increasing pause period imposed during the fatigue test.

(2) Remarkable recrystallizations initiated from the wire surface occurred in 7N copper after the pause period in the fatigue test.

(3) It is considered that strains accumulated during fatigue act as the driving force and 7N copper recovers from fatigue by the effect of recrystallization.

References

[1] M. Kato: "Application of Ultra-High-Purity Copper for Electric Wires and Conductors", Proc. of Ultra High Purity Base Metals (UHPM94), Kitakyusyu Fukuoka Japan, May 1994, (Jpn. Inst. Metals, Sendai, Japan, 1995), p441.

[2] A. Kurosaka, N. Tanabe, O. Kohno and O. Osanai: "High Purity Copper Wires", Proc. of Ultra High Purity Base Metals (UHPM94), Kitakyusyu Fukuoka Japan, May 1994, (Jpn. Inst. Metals, Sendai, Japan, 1995), p446.

[3] T. Nara and Y. Yamada: J. Jpn. Inst. Metals, 24(1960)744.

[4] J. S. Smart, A. A. Smith and A. J. Phillips: AIME, 143(1941)272.

[5] B. F. Decker and D. Harker: Trans. AIME, 188(1950)887.

[6] V. A. Phillips and A. Phillips: J. Inst. Metals, 81(1952/53)185.

[7] R. Urao, M. Aoyama, M. Kaminaga, M. Oonuki and Y. Miyake: J. Jpn. Inst. Metals, 54(1990)1030.