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전기비저항 측정에 의한 구리와 구리합금의 미시적 열화평가

Evaluation of Microscopic Degradation of Copper and Copper Alloy by Electrical Resistivity Measurement

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초 록 본 연구에서는 직류 4단자 전위차법을 이용한 전기비저항을 측정하여 반복피로손상을 받은 구리와 구리합금의 미시적 열화를 평가하였다. 서로 매우 다른 적층결함 에너지를 갖는 구리(Cu)와 구리합금(Cu-35Zn)에 대해 반복피로손상을 가하고 이들 재료에서 발달한 전위구조와 전기비저항 간의 관계를 연구하고자 하였다. Cu는 전위셀 하부구조를 형성하였지만, Cu-35Zn 합금은 피로사이클에 따라서 전위밀도는 증가하고 평면배열의 전위구조를 형성하였다. 전기비저항은 두 재료 모두에서 피로변형 초기 단계에서 급격하게 증가하였다. 더욱이, 피로시험 후 구리는 약 7 % 그리고 구리 합금은 약 6.5 % 변하였다. 이러한 일관적인 결과들로부터, 반복적인 피로에 의해 발달한 전위 셀구조는 평면배열의 전위구조보다도 전기비저항에 매우 민감한 것으로 판단된다.

주요용어: 전위구조, 전기비저항, 적층결함, 피로

Abstract In the present study, the microscopic degradation of copper and copper alloy subjected to cyclic deformation has been evaluated by the electrical resistivity measurement using the DC four terminal potential method. The copper (Cu) and copper alloy (Cu-35Zn), whose stacking fault energy is much different each other, were cyclically deformed to investigate the response of the electrical resistivity to different dislocation substructures. Dislocation cell substructure was developed in the Cu, while the planar array of dislocation structure was developed in the Cu-35Zn alloy increasing dislocation density with fatigue cycles. The electrical resistivity increased rapidly in the initial stage of fatigue deformation in both materials. Moreover, after the fatigue test it increased by about 7 % for the Cu and 6.5 % for the Cu-35Zn alloy, respectively. From these consistent results, it may be concluded that the dislocation cell structure responds to the electrical resistivity more sensitively than the planar array dislocation structure evolved during cyclic fatigue.

Keywords: Dislocation Structure, Electrical Resistivity, Stacking Fault, Fatigue

1. Introduction

Structural materials subjected to cyclic loading change their mechanical properties at the beginning of the fatigue process, and then eventually fail. Many previous observations indicate that both dislocation multiplication and point defect production are important aspects of fatigue deformation (Lindgren et al., 2008; Weidner et al., 2010; Schapink and Jong, 1964).

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It is evident that for a characterization of the fatigue state, particular emphasis has to be laid on point defects as well as on the knowledge of the dislocation structure (Grobstein et al., 1991; Kim et al., 2008). Such changes in mechanical properties are well known to closely relate with the changes in dislocation density and configuration. Therefore, the structural components being used under fatigue stresses should regularly undergo safety evaluation. Moreover, an understanding of the evolution of fundamental microstructural changes, such as dislocation density and substructure, is important for the nondestructive evaluation during the plastic deformation process.

Electrical resistivity resulted from conduction electron scattering is affected by phonon scattering, impurities and lattice defects. In particular, a defect on an atomic scale (i.e., impurity atom, vacancy and dislocation) has been found to be a predominant microstructural factor affecting electrical resistivity (Hummel, 1985; Fukai, 2008). Schafler et al. observed that the electrical resistivity increased with respect to strain in large cold worked pure iron and derived directly dislocation density from X-ray diffraction analysis (Schafler et al., 1997). Eikum and Hlowech reported that during fatigue the specimen resistance increased to a saturation value of about approximately 100 nΩcm at strain amplitude of 0.21% (Eikum and Holwech, 1968). Jongenburger showed that the electrical resistivity of metals is affected by the presence of point defects, i.e., vacancies, and that dislocations can act as sinks for these vacancies (Jongenburger, 1953). Hence, the recovery of electrical resistivity might be expected if vacancies are annihilated due to the diffusion to dislocations. Since the process of dislocations ascending from their slip planes involves the migration of vacancies to the edges of the half planes, it would be expected that an ascending dislocation would be accompanied by a decrease in the electrical resistivity, and initially attributable to the presence of vacancies. Seitz interpreted the increase in the electrical resistivity of metals during plastic deformation in terms of the generation of lattice vacancies due to moving dislocations (Seitz, 1952). In addition, Byeon et al. represented that the electrical resistivity of ferritic Cr-Mo rotor steel decreased rapidly in the initial stage of isothermal aging heat treatment due to the depletion behavior of solid solution element in matrix (Byeon et al., 2002). But, they did not consider the effect of dislocation recovery during isothermal aging at high temperature since this ferritic steel is a typical bainite structure having lath subgrains and high dislocation density.

Most of all, these literatures contain little data concerning the dislocation and point defect structure of metals after cyclic deformation and only show phenomenological correlation between deformation and electrical resistivity. In addition, they were lacking the explicit relationship between microstructural evolution and electrical resistivity. In the present study, microscopic degradation of the cyclically deformed microstructures of Cu and Cu-35Zn alloy was conducted in order to investigate the electrical resistivity responses to the different dislocation substructures that evolved during fatigue damage.

2. Experimental Procedure

2.1 Preparation of Samples

The materials used for this study were 99.9% pure polycrystalline Cu and Cu-35Zn alloy. All specimens were machined to have a gage length of 12 mm, and were annealed in an argon atmosphere for one hour at 450℃ for the Cu and at 600℃ for the Cu-35Zn alloy. The final grain size analyzed using an average linear intercept method and tensile strength of the specimens were about 41 ㎛, 261 MPa for Cu and 54 ㎛, 348 MPa for Cu-35Zn alloy, respectively.

The fatigue experiments were performed under constant strain amplitude at room temperature using a servohydraulic fatigue testing machine. A triangular wave signal was used for the constant strain rate of 6×10^{-4}/s and the cycles to failure (N_f) defined as the number of cycles at which the saturation stress decrease to 20%. Subsequently, all cyclically deformed specimens were cut so the stress axis would be perpendicular to the plane of observation. All thin foils were prepared by electropolishing in a solution of 33% nitric acid and 67% methyl alcohol at a temperature of -25℃ and a voltage of 2 V using a Struers Tenupol jet polisher. The foils were examined using a JEOL 1200EX transmission electron microscope. All TEM images were taken under bright field conditions.

2.2 Electrical Resistivity Measurement

Electrical resistivity was measured using the DC four terminal potential method (Nahm et al., 2001). Measurement was carried out at room temperature consistently fixed at 20℃. A computer-controlled direct current source was used to supply current ±1 A to the specimen and the voltage was measured to 1 nV using nano-voltagemeter. As the interval between voltage output leads can lead an error in electrical resistivity values, a fixture with constant terminal interval was used. A sheet-type specimen of 10 mm in length, 10 mm in width, and 1 mm in thickness was used to measure electrical resistivity. The thickness of the specimen was plane paralleled to within ±2 ㎛ and the specimen have been electropolished to produce clean surface. The electrical resistivity in this experiment was a value averaged over five measurements.

3. Results and Discussion

For the annealed single-phase fcc (face centered cubic) polycrystalline Cu and Cu-35Zn alloy, the cyclic deformation response, as depicted by the cyclic hardening curve, is shown in Fig. 1 for a specimen tested under total strain amplitude of 7×10^{-3}. Both materials exhibited cyclic hardening curves that consisted of an initial rapid hardening stage. Typical strain hardening was observed to be most pronounced during the early stage of cycling at both test materials, where after the strain hardening became saturated within initial thirty to forty cycles.

The fatigue life curves for Cu and the Cu-35Zn alloy are shown in Fig. 2. A power law type that follows the Manson-Coffin law can be well fitted to the experimental data.

$$\varepsilon_{ap} = \varepsilon_f'(2N_f)^c \qquad (1)$$

where ε_{ap} is the plastic strain, ε_f' the fatigue ductility coefficient, N_f the cycles to failure and

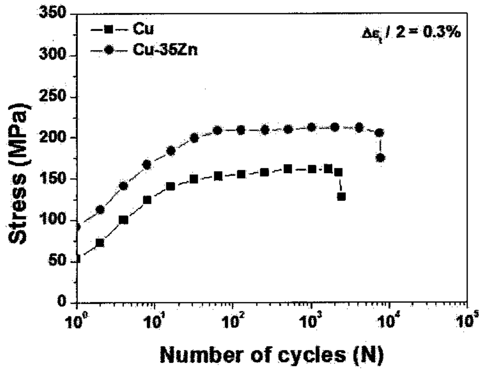
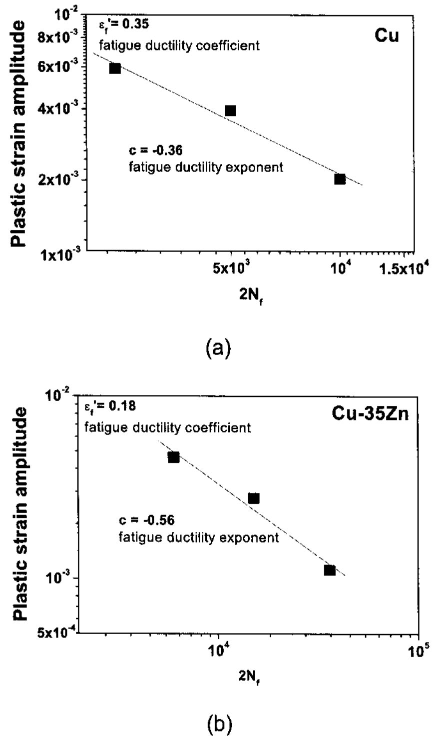


Fig. 1 Fatigue hardening curves in test with constant total strain amplitude at a strain rate of $\dot{\varepsilon} = 6\times10^{-4}$/s

Table 1 Comparison of monotonic and cyclic stress-strain behavior for incremental step test of Cu and Cu-35Zn alloy at a strain rate of $6\times10^{-4}s^{-1}$

	UTS	YS	c	e'f	Strength coefficient		Strain hardening	
					monotonic	cyclic	monotonic	cyclic
Cu	205	40	-0.36	0.35	305	334	0.38	0.12
Cu-35Zn	311	94	-0.56	0.18	260	388	0.20	0.1



(a)

(b)

Fig. 2 Fatigue ductility coefficient and fatigue ductility exponent of Cu and Cu-35Zn alloy

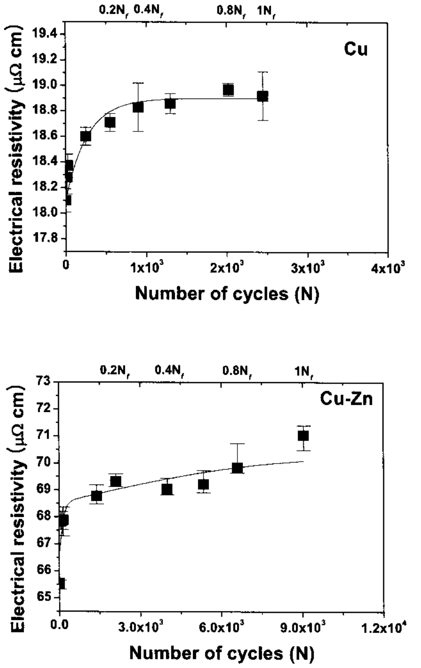
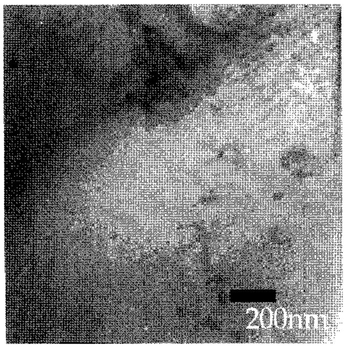
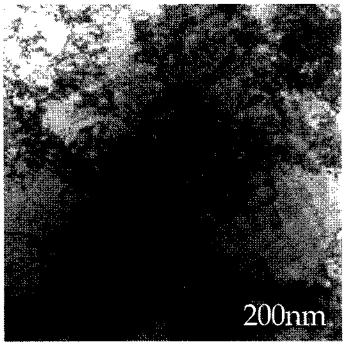


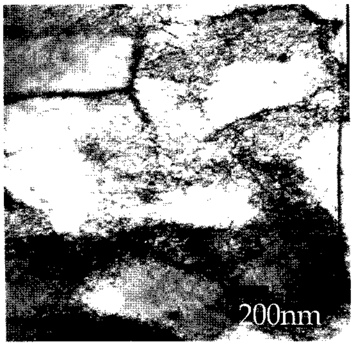
Fig. 3 Electrical resistivity as a function of number of cycles in fatigue damaged Cu and Cu-35Zn alloy



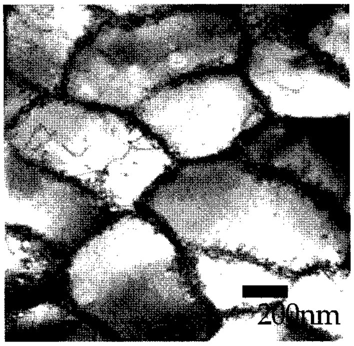
(a) as-annealed



(b) 0.1N_f



(c) 0.4N_f



(d) 0.8N_f

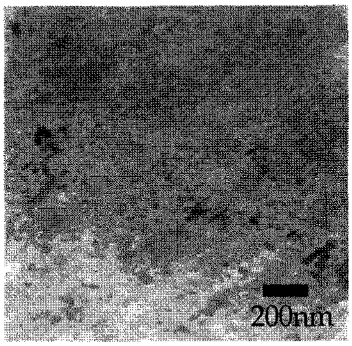
Fig. 4 TEM micrographs showing dislocation substructure of Cu under the strain amplitude of 0.3%; formation of cell structure

c the fatigue ductility exponent.

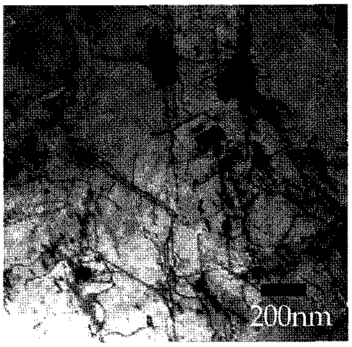
The fatigue ductility coefficients for Cu and Cu-35Zn alloy were 0.35 and 0.18, with fatigue ductility exponents of -0.36 and -0.56, as evaluated using the least-squares fit, respectively. Table 1 compares the monotonic and cyclic stress-strain curves for incremental step tests of Cu and Cu-35Zn alloy.

Fig. 3 shows the change of electrical resistivity with the life fraction. Resistivity rapidly increases in the initial fatigue life and then change little thereafter. In general, the atomic scale defects act as predominant scattering centers of conduction electron influencing on electrical resistivity (Rossiter, 1987). In this respect, the increase of electrical resistivity with increasing fatigue life fraction is thought to be closely related to the dislocation evolved during cyclic deformation. Scattering centers other than dislocations are, for the most part, not directly observable, so that the additional evidence is necessary before the resistivity introduced by deformation can be attributed with certainty to particular defects.

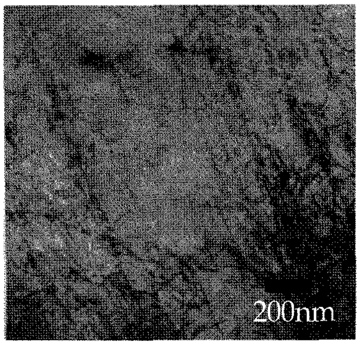
Fig. 4 and 5 show the change in dislocation substructures with fatigue cycles in the Cu and Cu-35Zn alloy. The type of dislocation substructure typical for the whole volume of the fcc specimens depends very strongly on the strain amplitude, the stacking fault energy, and, to a limited extent, the temperature (Madhoun et al., 2003). Feltner and Laird presented a simple schematic diagram relating the types of internal substructures to these parameters (Feltner and Laird, 1967). In their diagram, the high stacking fault energy metals exhibit cell structure in the high amplitude region, but for the low stacking fault energy metals, the typical substructure features a planar array distribution of dislocation. Cu is wavy slip material where cross slip takes place easily as a result of a high stacking fault energy, and multiple slip leads to the decomposition of the persistent slip band into cellular bands, and then to a cell structure at a high strain amplitude (Eikum and Holwech,



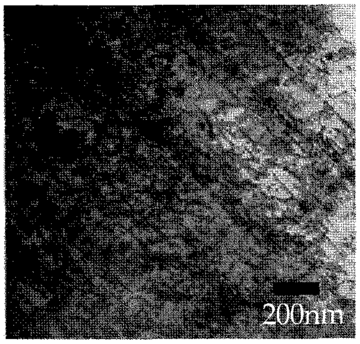
(a) as-annealed



(b) 0.1N_f



(c) 0.4N_f



(d) 0.8N_f

Fig. 5 TEM micrographs showing dislocation substructure of Cu-35Zn alloy under the strain amplitude of 0.3%; formation of planar structure

1968; Jongenburger, 1953). On the other hand, the Cu-35Zn alloy is a planar slip material where it is difficult for cross slip to occur due to the low stacking fault energy (Lukáš and Klesnil, 1973).

In this study, the development of dislocation cell substructure in Cu and increased dislocation density in Cu-35Zn alloy without change of configuration are evident. The same results reported by previous researchers were obtained. The dislocation density on the cell structure cannot be reliably determined by means of thin foils because the dislocation density in the cell walls is too high, although the cell size decreased slightly with the fatigue life fraction.

Dislocation interaction produces a jog and lattice vacancy or interstitial were created by the motion of the jog. As a screw dislocation moves through its slip plane it will, in general, encounter a forest of screw dislocations and many intersections will occur. Some of these will produce vacancy jogs and others will be interstitial jogs. Seeger estimated that the energy required to form a vacancy or an interstitial atom at a jog in an fcc metal are 4.8eV for an interstitial atom and 0.7eV for a vacancy, respectively (Seeger, 1955). It has been put forward for lattice vacancies being energetically more favorable than interstitials. Therefore, the conduction electron scattering effect arising form dislocation and vacancy, which created by dislocation interaction during the fatigue process, caused the increase of electrical resistivity in the cyclic deformation of Cu and Cu-35Zn alloy.

Summary

The microstructural changes due to low cyclic fatigue damage were evaluated by using electrical resistivity measurement.

Dislocation cell substructure was developed in Cu, while planar array of dislocation structure was developed in Cu-35Zn alloy increasing dislocation density with fatigue cycles. The electrical resistivity increased rapidly in the initial stage of fatigue deformation in both materials. Moreover, after the fatigue test it increased by about 7 % for the Cu and 6.5 % for the Cu-35Zn alloy, respectively. From these results, it may be concluded that the dislocation cell structure responds to electrical resistivity more sensitively than the planar array dislocation structure evolving during cyclic fatigue.

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