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Vacancy Generation in Aluminum Single Crystal with a Low Dislocation Density

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Vacancy generation process in nearly perfect aluminum single crystal was investigated by means of a shape of helical dislocation by X-ray diffraction topography, using a translation type high temperature Lang camera. Helical dislocations as a vacancy source were observed after temperature rise. The shape of helix at initial stage of the appearance looked like a coil spring. But the shape changed to the helix reflected the crystal structure of specimen with the lapse of time. Under-saturation of vacancy in the specimen was estimated from the size of helix. In the results, most of vacancies were emitted from dislocations existed in as-grown nearly perfect crystals, and it takes several hours to achieve the equilibrium concentration of vacancy after a temperature rise.

§1. Introduction

Thermal generation of vacancies in metals have been investigated by several workers,[1,2] and it is generally accepted that predominant source of vacancy is dislocations in usual metal crystal[2] and interstitial dislocation loops in perfect crystal.[3] However, details of the generation is not clear, for example the type of dislocation as a vacancy source was not specified in the majority of the studies, because of the difficulties in the experimental techniques and the preparation of specimen.

We reported already the nucleation and growth process of helical dislocation.[4] Therefore, the present work has been planned with the aim to make clear the vacancy source in nearly perfect metal crystals using a helical dislocation as a sensor of under-saturation of vacancy. So, X-ray topograph was taken using a one-scan type Lang camera at elevated temperature after sudden temperature rise on an aluminum single crystal with a low dislocation density.

§2. Experimental procedures

Specimen used in the present work was an aluminum single crystal with a low dislocation density which was grown from zone-refined poly-crystalline aluminum. This single crystal was prepared in the following way: Zone-refined aluminum of which residual resistance ratio was 1.1×10^4 without size correction was rolled down to 0.5 mm

in thickness and shaped into 5 mm in width. After pre-annealing at 250°C for 60 minutes in air, these plates were strained 2.0% by extension and cut out about 50 mm in length. Then the plates were electro-polished to remove the oxide layer on the surface, and were converted into single crystal under vacuum by a travelling furnace. The travelling speed of the furnace was 60 mm/h and the maximum temperature was 600°C. After single crystals were grown, the furnace was stopped and the crystals were cooled down to room temperature for about 50 hours. Furthermore, single crystals were annealed cyclically six times between 250°C and 150°C at the heating and cooling rates of 25°C/h in vacuum. By this treatment, the dislocation density of the specimen decreased to about 1×10^3 cm^{-2}.

This specimen was mounted in a electric furnace which was set up on the goniometer-head of X-ray Lang camera. And synchronous motor for the traverse on the camera was replace by stepping motor in order to reduce the travers speed of the camera. The traverse speed could be varied from 0.1 mm/h to 0.5 mm/s. Therefore, the specimen and nuclear plate on the camera are translated without scanning during the exposure. The method of moving without scanning enables us to follow the change of the lattice defects. Datails of the apparatus will be published in elsewhere. A characteristic X-ray used in this work was AgKα_1 radiation. The topographs were exposed on Illford L-4 nuclear plate with emulsion thickness 50 μm and were taken in (111) reflection.

In the present investigation, the specimen was heat-treated according to the following schedule: The temperature of specimen was rised from room temperature to 250°C with the heating rate of 500°C/h at the beginning, and three X-ray topographs were taken in a series holding the temperature at 250°C, and then cooled down to 240°C with the cooling rate of 100°C/h, and again one topograph was taken at the temperature.

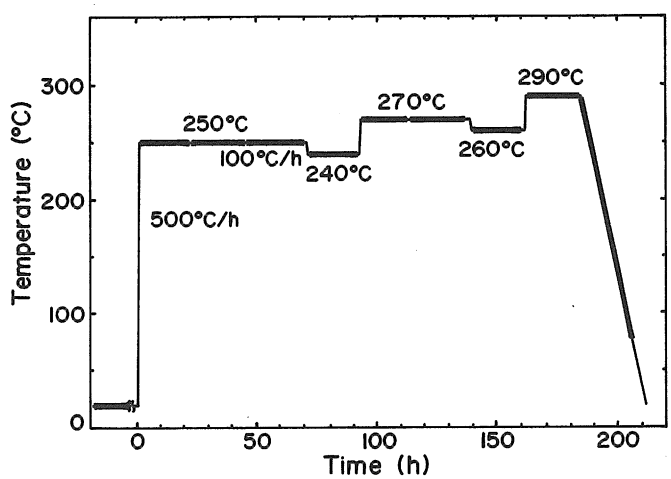

Fig. 1. Temperature-time diagram during the heat-treatment of the present experiment. The periods for X-ray exposure are indicated with thick lines.

These procedures were performed on after another as shown in Fig. 1. Thick lines in the figure show the exposure time for the X-ray topographs.

§3. Experimental results

An example of the translation topograph of Lang is shown in Fig. 2. This topograph was taken at 270°C after temperature rise from 240°C with the heating rate of 500°C/h. Many helical dislocations are observed in this topograph. The shape of helix at initial stage of the appearance looks like a coil spring. But the shape changes to the helix reflected the crystal structure of specimen at the last stage of the exposure because of the line tension of helix. The growth of helix is considered to come from the vacancy emission from it, because of the deficiency of vacancy from the thermal equilibrium concentration due to the temperature rise of specimen. The growth

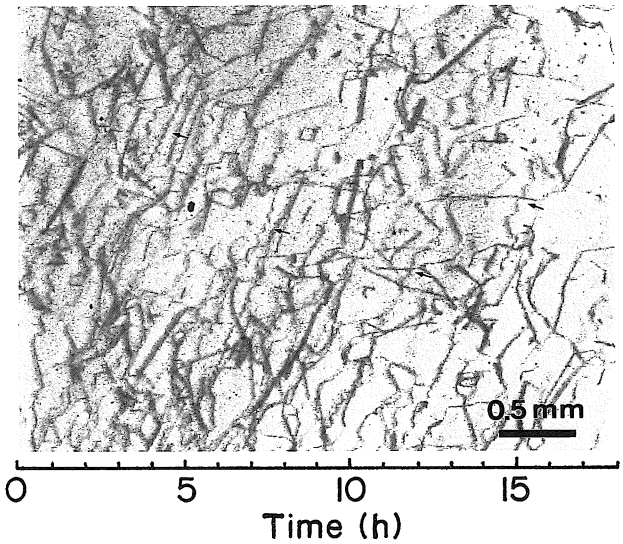

Fig. 2. Translation topograph of aluminum single crystal with the time scale showing the change of the shape of helical dislocations.

Table I. Radius and pitch of the helical dislocation corresponding a time after a temperature rise.

Time (h)	Radius (μm)	Pitch (μm)
5.0	24	60
8.3	26	78
11.6	33	128
15.6	35	118
16.0	40	198

mechanism and the configuration of helix were already published.[4] In this topograph, the radius and pitch of helix increase with the aging time. As a typical example, the radius and pitch of helix indicated by arrow in Fig. 2 and the corresponding time are tabulated in Table I.

§4. Discussion

We evaluate the degree of under-saturation of vacancy C/C_0 in the specimen using the values of radius r and pitch λ of helix shown in Table I. It was pointed out by Hirth and Lothe that the degree of super- or under-saturation of vacancy necessary for the growth of helix is estimated from the equilibrium condition between the line tension of helix and the chemical potential of excess or deficient vacancy as follows,[5]

$$\ln\left(\frac{C}{C_0}\right)=\frac{2\pi\Omega\phi}{bkT\left[(2\pi r)^2+\lambda^2\right]^{1/2}}, \tag{1}$$

where Ω is the atomic volume, b is the magnitude of the Burgers vector, ϕ is the line tension of helix and is expressed by

$$\phi=\frac{Gb^2(1+\upsilon)}{4\pi(1-\upsilon)}\ln\frac{\lambda}{3b},$$

and G is the shear modulus and υ is Poisson's ratio. The degree of under-saturation of vacancy necessary for the growth of helix was calculated. The under-saturation in the specimen are shown in Fig. 3. The degree of under-saturation attains to unity within 5

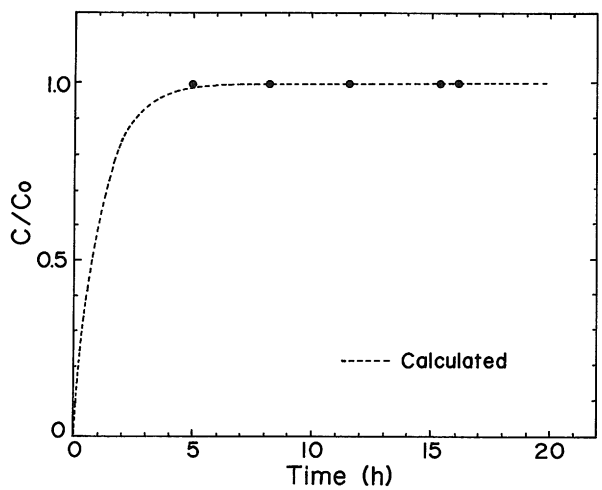

Fig. 3. Degree of under-saturation of vacancy estimated from the size of helical dislocation after a temperature rise. Dotted line shows a degree of under-saturation calculated from eq. (2).

hours after temperature rise. In other word, vacancy concentration in the specimen has achieved the equilibrium one within 5 hours. Since it is seems that the main source of vacancy in the specimen is pre-existence dislocations, change of vacancy concentration can be treated as the vacancy diffusion in a cylinder where vacancies are emitted from a dislocation along the center axis of the cylinder. The radius of cylinder r_s is $1/\sqrt{\pi N_d}$ where N_d is the dislocation density. Using Ham's expression in this approximation,[6] the concentration C is expressed as follows,

$$C=C_i+\Delta C\left[1-\exp\left(-\frac{t}{\tau}\right)\right] \tag{2}$$

$$\tau=\frac{r_s^2}{2D(T)}\left(\ln\frac{r_s}{r_0}-\frac{3}{5}\right),$$

where C_i is the initial concentration of vacancy, ΔC is the difference between the equilibrium concentration and the real concentration. $D(T)$ is the diffusivity of a vacancy, and is expressed by

$$D(T)=D_0\exp\left(-\frac{E_m}{kT}\right).$$

Here, E_m is the migration energy of vacancy and r_0 is the radius of dislocation core. The calculation was performed using $E_m=0.65$ eV,[7] $D_0=0.176$ cm^2/s,[8] $N_d=3\times10^3$ cm^{-2} and $T=270$°C.

Concentration of vacancy after the temperature rise from 240°C to 270°C was estimated using equation (2), and the result as a degree of under-saturation C/C_0 are shown by dotted line in Fig. 3. It is noted that vacancy concentration in the specimen increases rapidly and attains the equiribrium concentration a few hours after the temperature rise. And this is consistent with the experimental facts that the under-saturation of vacancy estimated from the size of helix is nearly unity at several hours after the temperature rise. Therefore, the predominant vacancy source in nearly perfect crystal is also dislocations presented in as-grown crystal, if interstitial loops as a vacancy source are not appear. And it takes several hours to achieve the equilibrium concentration of vacancy after a temperature rise in nearly perfect metal crystals.

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