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Electron Diffraction and Microscopic Study on the Faulted Structure of Martensite in Evaporated Fe-Ni Alloy Films

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Evaporated films produced from Fe-17.2 at% Ni alloy were austenitized by heating in a hydrogen stream, and cooled in the liquid nitrogen to produce the b.c.c. martensite. The repeated twins were observed in electron micrographs. Line breadth analysis of electron diffraction patterns gives the mean strain $(7.2\pm0.7)\times10^{-3}$. The effective particle size and probability of faulting on the (211) plane of martensite were also estimated. A considerable extinction of the diffraction intensity was observed. There was a distinct discrepancy between the effective particle size and the effective thickness of crystal estimated from the intensity. This was interpreted by considering the repeated twin structure of martensite.

Recent electron microscope observations by Pitsch[1], Nishiyama *et al*[2]., and Kelley and Nutting[3] revealed that repeated twins are produced on the (211) plane of b.c.c. martensite crystal. The present author studied martensite films of evaporated Fe-Ni alloy by electron diffraction and microscope. The mean strain and fault probability were estimated from the electron diffraction line broadening. A considerable extinction of diffraction intensity was observed and it was interpreted by considering the repeated twin structure of martensite.

Fe-17.2 at% Ni alloy was deposited in thickness of 500Å on the cleavage face of rock-salt. In order to austenitize, the films were heated in a stream of purified hydrogen at 650°C for 10 min. Martensite films were obtained by cooling these specimens in the liquid nitrogen. Electron diffraction patterns were taken by a camera operated at 63 kV (wave-length $\lambda=0.045$Å), and electron micrographs by a JEM microscope operated at 80 kV. Intensity curves were obtained according to the usual microphotometric method.

Photo. 1 reproduces an electron diffraction pattern of martensite. The broad Debye rings due to martensite (b.c.c.) are observed together with the sharp rings due to retained austenite (f.c.c.). Photo. 2 is an electron micrograph of martensite showing repeated twin structure. The observed twin widths are in a range between 30 and2 00Å. The mean width is 60Å.

When both the strain and particle size are responsible for the broadening of diffraction

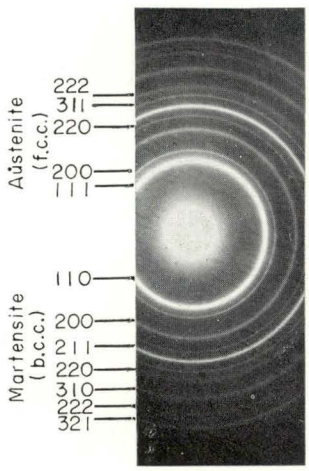

Photo. 1. Electron diffraction pattern of b.c.c. martensite. Sharp rings are due to retained austenite (f.c.c.).

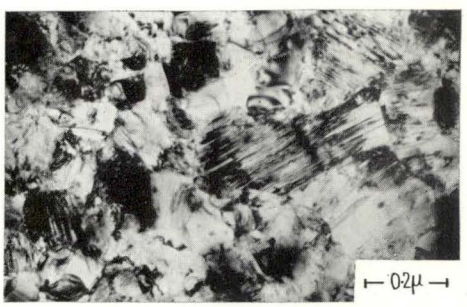

Photo. 2. Electron micrograph of martensite.

line, the interrelation between them is known to be expressed by the Hall's formula[4]. The applicability of the formula was discussed by Kuriyama[5]. When the stacking faults on the (211) plane of b.c.c. crystal are also responsible for the line broadening, the Hall's formula can be expressed by

$$\frac{B\cos\theta}{\lambda}=\frac{1}{2D_{\text{eff}}}+E\frac{h_0}{a}, \qquad (1)$$

where

$$\frac{1}{D_{\text{eff}}}=\frac{1}{D}+\frac{1}{D_F}=\frac{1}{D}+\frac{1.5\alpha+\beta}{a(u+b)h_0}. \qquad (2)$$

Here, B is the line breadth, $E/2$ the mean strain, $h_0^2=h^2+k^2+l^2$, a the lattice constant, D_{eff} the effective particle size, D the average domain size, D_F the fictitious particle size, α and β are probabilities of the deformation fault and of the twin fault, $u+b$ the number of reflections contributing to a Debye ring, and $L_0=-h-k+2l$. These notations are due to Warren[6].

The mean strain $E/2$ was determined from the multiple orders of reflections such as (200)-(400) and (211)-(422) using single crystalline patterns of martensite. For powder patterns, the line breadths of higher order reflections could not be measured accurately. Table I shows the results. The obtained mean strain $(7.2\pm0.7)\times10^{-3}$ is close to the strain in the quenched steel (8×10^{-3})[4]. The r.m.s. strain was also calculated according to the quadratic formula:

$$\left(\frac{B\cos\theta}{\lambda}\right)^2=\frac{1}{C^2}+E^2\left(\frac{h}{a}\right)^2. \qquad (3)$$

(r.m.s. strain: $E/\sqrt{2\pi}$). This is also tabulated for comparison. The mean value is nearly equal to the value obtained by Eq. (1).

Table I. Mean strain in the martensite crystals.

Reflections	Mean strain (Eq. (1))	r.m.s. strain (Eq. (3))
(200)-(400)	$(6.9\pm0.9)\times10^{-3}$	$(7.6\pm0.1)\times10^{-3}$
(211)-(422)	$(7.5\pm0.5)\times10^{-3}$	$(6.4\pm0.1)\times10^{-3}$
Mean	$(7.2\pm0.7)\times10^{-3}$	$(7.4\pm0.6)\times10^{-3}$

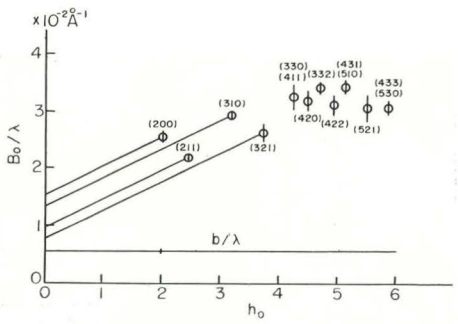

Fig. 1. Plots of B_0/λ against $h_0=\sqrt{h^2+k^2+l^2}$. $(B_0-b)/\lambda$ at $h_0=0$ gives $\frac{1}{2}D_{\text{eff}}$.

Fig. 1 shows plots of B_0/λ for the powder pattern against h_0, where B_0 is the observed line breadth. The line b/λ corresponds to the instrumental breadth. This breadth was determined from the breadth of the trace of direct electron beam on a photographic plate moved rapidly and from the amount of beam deflection due to the alternating leakage magnetic field. Using the mean strain obtained before, the extrapolation $h_0\to0$ of observed line breadth was carried out for (200), (211), (310) and (321) reflections. Obtained values for the effective particle size, fictitious particle size, and fault probability are tabulated in Table II. For the calculation of D_F and $1.5\alpha+\beta$, the average domain size D was assumed to be 500Å, which was estimated from the amount of deposition. The accuracy of D_{eff} depends not only on the accuracy of measurement of the line breadth, the mean

Table II. Effective particle size D_{eff}, fictitious particle size D_F and fault probability $1.5\alpha+\beta$.

Reflection	D_{eff} (Å)*	D_F(Å)*	$1.5\alpha+\beta$ $(\times10^{-2})$*
(200)	50	60	4
(211)	100	200	2
(310)	60	70	3
(321)	200	400	1

* $D=500$ Å Mean $(2.5\pm0.6)\times10^{-2}$

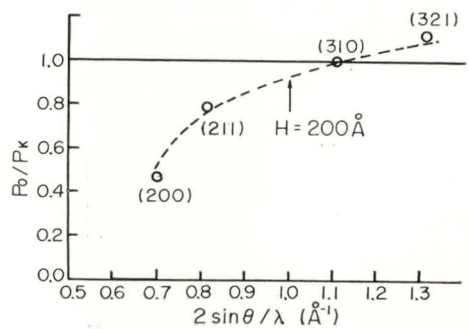

Fig. 2. Ratio of the observed intensity P_0 to the calculated P_K. The plots are normalized at (310) reflection.

strain and the instrumental breadth, but also on the degree of applicability of the Hall's formula. In the present case the average distance between faults estimated from these values (46±10Å) is in accordance with the mean twin width observed in the electron micrographs in the experimental error.

An intensity ratio P_0/P_K is plotted in Fig. 2, where P_0 is the observed integrated intensity per unit length of Debye ring and P_K the calculated intensity taking the temperature effect into consideration. The plots are normalized at (310) reflection. A considerable extinction effect can be seen to take place. The effective thickness of crystal H corresponding to this extinction was estimated according to the theory of primary extinction. When we assume H=200Å, the calculated intensity ratio coincides very well with the observed as shown in Fig. 2 and in Table III.

Table III. Extinction coefficient.*

Reflection	$f_{hkl}(D_{\text{eff}})$	f_{hkl} (H=200Å)	$f_{0,hkl}$	H/D_F
(200)	0.94 (0.97)	0.39 (0.51)	0.46	3
(211)	0.78 (0.80)	0.59 (0.78)	0.79	1
(310)	0.97 (*1.00*)	0.76 (*1.00*)	*1.00*	3
(321)	0.83 (0.85)	0.84 (1.10)	1.12	0.5

* $f_{hkl}(D_{\text{eff}})$: extinction coefficient corresponding to the thickness D_{eff}.

f_{hkl} (H=200Å): extinction coefficient corresponding to the thickness H=200Å.

$f_{0,hkl}=(P_0/P_K)_{hkl}/(P_0/P_K)_{310}$.

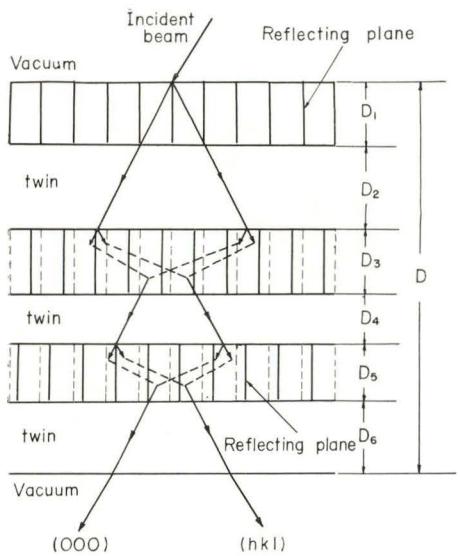

Fig. 3. Schematic representation of the twin structure and diffraction of electrons.

The discrepancy between D_{eff} and H can be considered to be due to the repeated twin structure of martensite. Fig. 3 illustrates the structure schematically. The plates of thickness D_1, D_2 etc. are piled up and the adjacent plates are in the twin relation. If the first plate diffracts electrons, the second does not, the third does, and so on. The plates are considered to be coherent. Since the reflecting planes of the third plate are displaced relative to the reflecting planes of the first, some phase shifts of electron waves take place at their entrance to the third plate. Similar situation occurs when the waves enter into the fifth, the seventh etc. These phase shifts affect the intensity distribution of electron waves. On an average, however, the effect of phase shifts on the integrated intensity of Debye ring disappears. Therefore, the effective thickness of crystal responsible for the intensity is a sum of the thickness of twin plate satisfying the Bragg condition, i.e.

$$H=D_1+D_3+D_5+\cdots\approx(1/2)D\,.$$

The average thickness of plates is equal to the fictitious particle size D_F, which is responsible for the line broadening. This consideration interprets the experimental results. A preliminary calculation based on the dynamical theory proved the above consideration.

The actual structure of martensite, however, is much more complicated than the present model. Especially, the large internal strain may distort the twin plates, and the secondary extinction may take place. An application of the Darwin's theory of secondary extinction[7] led to the result that the average angle of inclination between plates is about one degree, which is considered to be rather large. However, a theory considered both the primary and secondary extinctions will give a more satisfactory interpretation of the diffraction of electrons in the martensite crystal.

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Direct Observation of Crystal Imperfections in KCl Single Crystals by Electronmicroscope

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The processes of change of thin KCl single crystal due to electron irradiation were studied by means of electronmicroscope and electron diffraction. As the result, it was found that by weak irradiation loop structures first appeared and then disappeared showing a lot of small bright specks over the whole crystal. At this stage each diffraction spot had the streaks along <100> directions. By intense irradiation bright squares appeared immediately without showing loop structures. At this stage the diffraction pattern was perfect N-pattern. By using replica technique, it was confirmed that loop structures and large squares were not due to surface structures. By using dark-field method, it was found that the loop structures and bright squares were caused by Bragg reflection. Thus it was proved that the loop structures and bright squares correspond to voids and cubic cavities in the crystal, respectively.

Mechanism of coagulation of vacancies in KCl with electron irradiation was discussed in detail.

1. Introduction

Direct observations of the rolled metallic foil thinned by chemical or electrolytic etching were made electronmicroscopically by Hirsch and others[1]. Jones and Mitchell[2] and Amelinckx and others[3] observed the decorated dislocations in the silver halide and additively coloured alkali halide under light microscope. Hibi and Ishikawa[4] and Hibi and Tomiki[5] made electronmicroscopic observations of coloured alkali halide crystals by using replica technique. Later, the present authors[6] also succeeded in verifying the process of crystallite growth due to X-ray irradiation and crystallite disappearance due to natural bleaching, by using successive replica technique on the definite position of KCl single crystal. It seems to be of much significance to observe directly such changes by using such a thin alkali halide crystal, which makes the use of replica unnecessary. From this point of view, crystal imperfections in KCl single crystal prepared from its water solution were directly observed by means of electronmicroscope and electron diffraction.

2. Experimental method

Thin KCl single crystal obtained by the vacuum drying method already reported[7] was used in this experiment. Successive observations were made under HS-6 electron microscope operating at 50KV soon after the pre-