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ELECTRON DIFFRACTION BY PERFECT VOID LATTICE

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Electron diffraction patterns from void lattice produced by high-temperature high-dose irradiation have been discussed. Intensities of extra reflections caused by perfect void lattices have been compared with those of matrix lattice reflections, where the size and the spatial arrangement are choosen as the parameters.

1. Introduction

Observation on voids have recently been made extensively from scientific and practical interests. Void nucleation and void growth is, for scientific interest, the direct and typical consequence of the interaction among vacancies, interstitials, dislocations and impurities. For practical interest, void formation in the results of high-tempetature high-dose irradiation brings volume increasing and changes mechanical properties in metals. They are the serious problems in the materials employed to commercial-scale fast reactors.

Cawthorne and Fulton [1] first found radiation-induced voids by stainless-steel. Successively many detailed observations in many metals in various conditions have been performed. The first observation of periodic void arrays was performed by Evans [2] in molybdenum irradiated with 2 MeV nitrogen ions.

Althouth voids can be formed, regardless of the kind of irradiation, by high-temperature high-dose irradiation, the interaction kinetic between defects and irradiation elements such as neutrons, electrons and charged-particles is different.

For observation of void lattice, electron microscopy, electron diffractoin and X-ray small-angle scattering techniques are available. Electron microscopy is advantageous for investigation of direct and whole aspect and is employed most commonly. Electron microscopy has some insufficency, however, for discussing spatial distribution of such as lattice parameters, size and imperfection. Sass and Eyre [3] succeeded to observe void arrays in pure molybdenum and TZM alloy (Mo, 0.5%Ti, 0.1%Zr) using electron diffraction technique and determined the lattice parameters of the void lattice. The advantage of this technique is as follows: direct determination of lattice parameter is, of course, of practical applications. Ditection of small defects, which are invisible by direct imaging techniques, can also be possible by this technique. Furthermore spatial distribution or imperfection of defect clusters

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may be measured.

The symmetry of the void lattice coincides with that of matrix lattice. The mechanism of the lattice formation has been discussed in mainly two ways. The first one, proposed by Malen and Bullough [4)5)], Stoneham [6)] and Tewary and Bullough [7)], depends on the elastic interaction of voids and leads to an energy minimum when voids are arranged in a lattice. The other idea by Evans [8)] is that voids nucleate on an impurity superlattlce existing prior to the irradiation. More recently, Foreman [9)] has pointed out that anisotropy of interstitial movement in metals, along certain crystal directions leading to formation of void free and void rich channels in these directions. The coincidence of the void superlattice structures with those of matrix (e.g. b.c.c. in Mo, f.c.c. in Ni) is simply a consequence of the difference in anisotropy in b.c.c. and f.c.c. lattice.

2. Intensity Equations

The intensity from a crystal can be written in free electron unit as

$$I(\boldsymbol{s}) = |F_{total}(\boldsymbol{s})|^2. \tag{1}$$

Here, $F_{total}(\boldsymbol{s})$ is the crystal structure factor of the overall crystal, and is given by

$$F_{total}(\boldsymbol{s}) = \sum_n F_n(\boldsymbol{s}) \exp(i\boldsymbol{s}\cdot\boldsymbol{R}_n), \tag{2}$$

where $F_n(\boldsymbol{s})$ is the crystal structure factor of the n-th unit cell, and

$$F_n(\boldsymbol{s}) = \sum_j f_j \exp(i\boldsymbol{s}\cdot\boldsymbol{r}_j), \tag{3}$$

while f_j and $\boldsymbol{r}_j$ the atomic scattering factor and the position vector, respectively. The scattering vector $\boldsymbol{s}$ and the lattice position vector $\boldsymbol{R}_n$ are respectively as follows

$$\boldsymbol{s} = 2\pi \sum_{i=1}^{3} h_i \boldsymbol{a}_i^*,$$

$$\boldsymbol{R}_n = \sum_{i=1}^{3} n_i \boldsymbol{a}_i. \tag{4}$$

The variable designated by h_i is a Miller index, while $\boldsymbol{a}_i$ and $\boldsymbol{a}_i^*$ are the unit cell vector and the reciprocal lattice vector, respectively.

Now consider a crystal containing void lattice. For simplicity, we treat the case where the number of cells excluded constant for every void and is m^3. The lattice parameter $\mu\boldsymbol{a}$ and the total number of void cells M^3 are introduced in the followings. In this case

$$F_{total}(\boldsymbol{s}) = T(\boldsymbol{s}) \sum_{k=0}^{M-1} \exp(i\boldsymbol{s}\cdot\boldsymbol{R}_k) \left\{ \sum_{n=0}^{\mu-1} F(\boldsymbol{s}) \exp(i\boldsymbol{s}\cdot\boldsymbol{R}_n) - \sum_{n=0}^{m-1} F(\boldsymbol{s}) \exp(i\boldsymbol{s}\cdot\boldsymbol{R}_n) \right\}. \tag{5}$$

Here, $\boldsymbol{R}_n$ is the displacing of the n-th matrix cell in a void cell and $\boldsymbol{R}_k$ that of the void cell in the crystal. $T(\boldsymbol{s})$, derived from the symmetry of the void lattice, corresponds the unitary structure factor of the void cell and is written by

$$T(\boldsymbol{s})=F(\mu\boldsymbol{s})/f. \tag{6}$$

For simplification of the representation, the case of (h00) reflection will be considered. Hence, eq. (5) is given as follows:

$$F_{total}(h)=\sum_{k=0}^{M-1}\exp(2\pi ikh)\left[\frac{F(h)F(\mu h)}{f}\,\frac{\sin\pi\mu h}{\sin\pi h}\exp\{2\pi i(\mu-1)h\}\right.$$
$$\left.-\frac{F(h)F(\mu h)}{f}\,\frac{\sin\pi mh}{\sin\pi h}\exp\{2\pi i(m-1)h\}\right]. \tag{7}$$

The term in the bracket of the right hand side is the crystal structure factor of the void lattice F_V. Finally the intensity is represented as

$$I(h)=|F_V|^2\sum_{k=-(M-1)}^{M-1}(M-|k|)\exp(2\pi ikh)$$
$$=\left|\frac{F(h)F(\mu h)}{f}\right|^2\left(\frac{\sin\pi M\mu h}{\sin\pi\mu h}\right)^2\left(\frac{\sin\pi\mu h}{\sin\pi h}-\frac{\sin\pi mh}{\sin\pi h}\right)^2. \tag{8}$$

3. Discussion

The values of the void size ma and the lattice parameter μa change variously with irradiation conditions such as temperature and dose. The order of magnitude ma is 30 Å and that of μa is 200 Å. From eq. (8), the intensity of a fundamental reflection is given by

$$I_f=M^2(\mu-m)^2|F|^2. \tag{9}$$

and that of a extra reflection caused by void lattice is

$$I_e=M^2m^2|F|^2. \tag{10}$$

From eqs. (8) and (9), it is immediately estimated that the order of magnitude I_e/I_f is 1×10^{-2}. Since m and μ are not so large, diffraction intensities of extra reflections decrease monotonically with increasing h and reach zero when h becomes $1/m(\sim0.1)$. A schematic feature is shown in Fig. 1.

Images of void array obtained by an electron microscope may not be clear. They often include lack of voids or varieties in void size. Moreover, Sass and Eyre [3] have observed only the first-order extra diffraction spots. They have interpreted this fact as follows: degree of short range disorder in the arrays could be a contributory factor in reducing the intensity of higher-order reflections and then higher-order reflections could not be observed. For the determination of these parameters, the intensity distribution

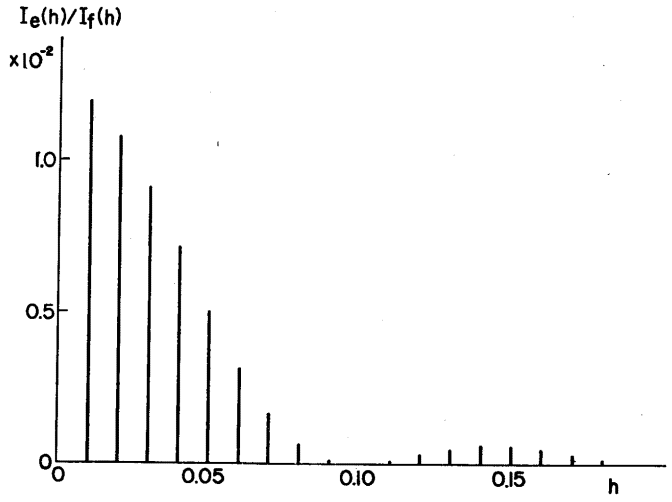

Fig. 1. Schematic intensity spectrum of extra reflections normalized by I(000). Here, $m=10$ and $\mu=100$ are choosen.

on the reciprocal lattice space is required. The electron diffraction technique, however, is not suitable for quantitative measurement of a intensity. X-ray small-angle scatting technique has been used for the parameter determination by Kulcinski, Mastel and Kissinger [10]. They have compared the parameters thus obtained with those from electron microscopy.

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