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The Dislocation Loop Density Dependence on the Thickness of a TEM Sample

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1. Introduction

We measured density of dislocation loops at Fe and Fe-1.0% Cu samples irradiated with electrons at a high voltage electron microscope (HVEM). The loop density increased as the fluence of electrons increased. Because the dislocation loop density is a very important parameter at the radiation hardening, accurate determination of loop number densities have a special meaning. The TEM is a unique technique, with which a shape and a microstructure of defect clusters can be observed through images. Most radiation defects are dislocation loops. When the size is below 5 nm, loops with **g•b**=0 are often not invisible and loops with **g•b**≠0 may also show very weak contrast under weak beam imaging conditions. A special method such as black-white contrast analysis should be used for the determination. This method can be applied to black-white lobe images obtained under dynamical two beam contrast conditions. The measurement of dislocation loop density was checked through an image simulation technique, TEMACI which was developed by Zhongfu Zhou, at Oxford University.

2. Methods and Results

In this report, the measured density of defect clusters is shown. The samples were pure Fe and Fe-1.0 Cu. They were irradiated with accelerated electrons at an HVEM. And the results from a computer simulation are showing that the intensity of dislocation loop image changes according to the position of loops in the direction of the sample depth. Our simulations also show the the resolution limit affects the analysis of radiation hardening.

2.1 Measurement of the defect cluster densities at HVEM

The irradiation of electrons to 99.98% pure Fe and Fe-1.0 % Cu alloy samples was performed at theHVEM in KBSI. The energy of electrons was 1.25 MeV and the electron flux was 4.7 x 10^{23} e/m^2·sec. The TEM images were captured through a CCD camera with a time interval of 1/30 s without binning. The thickness of samples were measured with the EELS method and got from a following equation.

$$t = \lambda \ln\left(\frac{I_l}{I_0}\right)$$

t : thickness,
λ : average mean free path
I_l : Intensity in the low-loss portion
I_0 : Intensity under the zero-loss peak

The density of defect clusters was determined by the counting the defects in TEM images got through athe CCD camera and printed out. The density was calculated from the counted numbers divided with the area of a photograph and the thickness got from the EELS data. Fig. 1 shows a captured image from the video. The defect density is shown in Fig.2

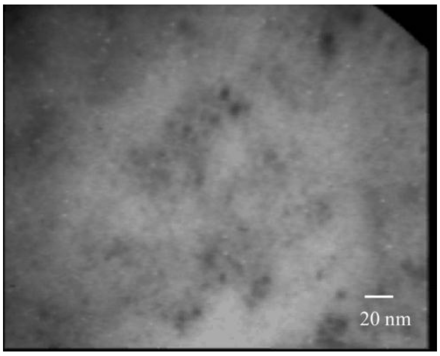

Fig. 1. Captured image from a CCD camera at HVEM showing defect clusters formed in Fe-1.0% Cu.

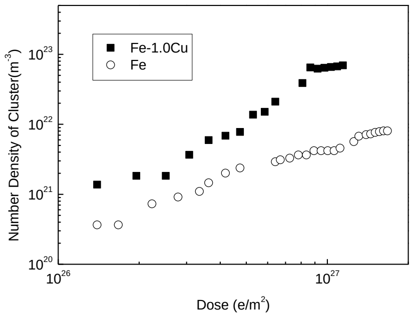

Fig. 2. Dose dependence of cluster number density in Fe and Fe-1.0% Cu irradiated with electrons.

The density of defects increases as theelectron dose increases. Fig. 2shows that more defects are formed at Fe-1.0% Cu than at the pure Fe. This means that Cu affects the radiation embrittlement through the more defect formation. At the same irradiation environment, the higher defect density makes the higher irradiation hardening and then results in severe radiation embrittlement.

2.2 TEM image intensity as a function of depth

Image contrast was simulated as a function of depth where the defects existed. Using TEMACI TEM Amplitude Contrast Imaging)[1], the intensity of dislocation loop image was simulated. TEMACI which is a computer program for dislocation loop simulation of weak beam diffraction contrasts. When we select two beam dynamical contrast condition in this program, we can obtain the same effect as images applied with Howie-Whelan equations. Image intensities can be calculated with Howie-Basinski equations as follows.

The wave function of the electron ($\Psi(\mathbf{r})$) can be written as a sum over the diffracted beams $\phi_g(\mathbf{r})$ in the Bloch-wave form[2].

$$\Psi(\mathbf{r}) = \sum_{\mathbf{g}} \phi_{\mathbf{g}}(\mathbf{r}) e^{2\pi i(\mathbf{k}+\mathbf{g}+\mathbf{s_g})\cdot\mathbf{r}} \tag{1}$$

k : the wave vector of electrons incident on the foil.

Sg : the excitation error for the beam with diffraction vector **g.**

The crystal potential is evaluated using the deformable ion approximation.

$$\mathbf{V}(\mathbf{r}) = \sum_{\mathbf{g}} \mathbf{V_g} e^{2\pi i \mathbf{g}\cdot(\mathbf{r}-\mathbf{R}(\mathbf{r}))} \tag{2}$$

R(**r**) : the field of atomic displacements around a defect.

$$\frac{\partial \Phi_{\mathbf{g}}}{\partial \mathbf{z}} = \frac{2\pi i}{\beta_{\mathrm{g}}}(\mathbf{k}+\mathbf{g}+\mathbf{s_g})\cdot \mathbf{s_g}^{(\mathbf{R})}\Phi_{\mathbf{g}} - \pi i \sum_{\mathbf{g}'}(1-\delta_{\mathbf{gg}'})\frac{\mathrm{U}_{\mathbf{g}-\mathbf{g}'}}{\beta_{\mathbf{g}}}\Phi_{\mathbf{g}'} \tag{3}$$

$\beta_g = (\mathbf{k}+\mathbf{g}+\mathbf{s_g})$

In Eq. (3), we can obtain the form of Howie-Whelan equations through considering only two beams of the transmitted and the diffracted .

Fig. 3 shows the image intensity contrast as a function of depth in a Cu. The contrast means the ratio of the maximum intensity of dislocation loop to the intensity of the background. The contrast oscillates in accordance with the position in the direction of depth. Roughly at depths 1/4 nξ_g, the intensity shows maximum or minimum, namely layer structure. ξ_g is the extinction distance. There are the layer structures at the top surface and the bottom surface. The transition from first to second layer occurs at about 1/3 ξ_g. The ξ_g of Cu is about 336 nm.

2.3 Density of dislocation loops

As shown in Fig. 3, all of dislocation loops can be observed at a TEM. The visibility depends on the resolution limit of a TEM and based on the assessment of images by eye. The dislocation loops at the layer near the top surface and the bottom surface can be observed, but those at the center or at 1/4ξ_g, 3/4 ξ_g, 5/4ξ_g --- are not visible

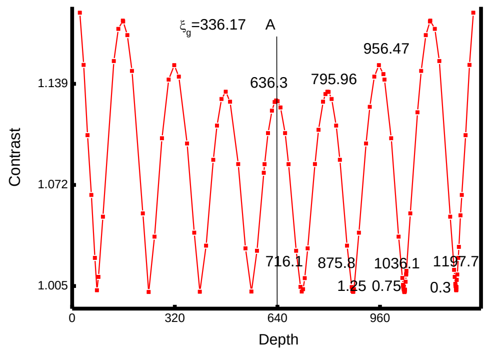

Fig. 3. Dislocation loop contrast as a function of depth in a Cu sample. Thickness : 1,275 nm, Voltage : 100 keV, Burgers vector : $1/2[01\bar{1}]$, **n :** $(01\bar{1})$, **g** : 200, **z** : [011] and loop radius : 2.4 nm

2.4 The effect of dislocation loop density on the hardening

According to the Orowan hardening mechanism, an increase in yield strength depends on the density of dislocation loops as following:

$$\Delta\sigma_y = M\alpha\mu b(Nd)^{1/2} \tag{4}$$

where M, α, μ, N and *d* are the Taylor factor, barrier strength of obstacles, the shear modulus of the matrix, the Burger's vector of moving dislocation, the density of obstacles and the mean diameter of obstacles. When this model is used for the matrix damage, a special attention should be paid to the application of a measured dislocation loop density. Direct input of the measured value from TEM analysis data affects the Taylor factor.

3. Conclusion

We could evaluate the effect of the defect position at the TEM sample on the image contrast. The measurement of defect density should be made with a special consideration at the effect of depth in samples

ACKNOWLEDGEMENT

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