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Development of a real-time stereo TEM

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We have developed a real-time stereo transmission electron microscope (TEM) with tilting illumination. This system allows us to observe three-dimensional (3-D) images directly with a video-rate of 1/30 s. The system comprises two electrostatic deflectors. The first, included in the illumination system, is able to illuminate a specimen at two oblique stereoscopic angles (left and right of the optical axis) up to $\pm 2.3^{\circ}$. The second deflector, in the imaging system, is used to correct the image separation caused by defocusing of the tilted illumination. These deflectors are operated in synchronization with an NTSC video signal output from a CCD camera to record left projections on odd fields and right projections on even fields. The time series of stereo pairs is transferred to a 3-D display that enables viewing of the 3-D images without special glasses. Real-time observation of ZnO particles demonstrated that 3-D images were clear even while moving the specimen. We applied this system to observing dislocations in an Al thin film.

1. Introduction

Obtaining three-dimensional (3-D) information with a transmission electron microscope (TEM) is valuable in materials science and has been applied to studies of dislocations, voids and precipitates. Since the 1960s, stereoscopy, which allows 3-D information to be taken from two projected images of a specimen that is tilted (stereo pair), has been carried out to investigate the 3-D distribution of defects in crystalline materials [1, 2]. Recently computer tomography (CT), which reconstructs a detailed 3-D image from several tens of images of a gradually tilted specimen has been applied to characterization of microelectronic devices [3]. These techniques can be applied only to stable specimens because the speed of image recording is limited by the mechanism of specimen rotation and CT requires time to perform 3-D reconstruction from the acquired images.

Extending TEM into real-time observation is important in materials science. By changing specimen conditions such as straining, heating and cooling using specialized specimen holders we can understand the function of the specimen *in situ* from dynamic observations of the specimen response. For example, Saka *et al.* have demonstrated real-time observation of dislocations with a video camera, and were able to clarify the behavior of the dislocations and measure their velocity [4].

Real-time TEM 3-D observation would offer a novel technique for materials characterization. In terms of obtaining qualitative 3-D information in real time, the stereo method is advantageous because 3-D images can be observed using only two images. The stereo pair is usually taken by tilting the specimen obliquely to the right and left by 2–7°. This range gives a convergence semi-angle consistent with the human eye. It is convenient to perform such real-time observations using a video camera system. In order to obtain stereo pairs at the National Television System Committee (NTSC) video-rate of 1/30 s, the tilting angle of the specimen stage must be switched within about 1 ms after the 15 ms exposure. However, the ordinary tilting mechanisms of TEM specimen stages cannot

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meet this requirement. Tilting of the incident electron beam can allow more rapid and accurate control than tilting of the specimen stage, and the time to record stereo pairs can be greatly reduced. Typke *et al.* [5] proposed an electron lens system for stereo observation with tilting illumination, and Pawley [6] proposed TEM stereoscopy in which the incident angle is switched in synchronization with the NTSC video signal. Fan *et al*. [7] reported a TEM system that could display stereo pairs every 5 s. These reports demonstrated only the principle, but the real-time 3-D TEM has not yet been practically realized.

We have previously reported a real-time stereo laser optical system with tilting illumination in which we successfully observed, in real-time, moving latex particles and a living plankton on a 3-D display [8].

In this paper, we report the development of a real-time stereo TEM, which enables direct observation of a time series of 3-D images at the video-rate of 1/30 s.

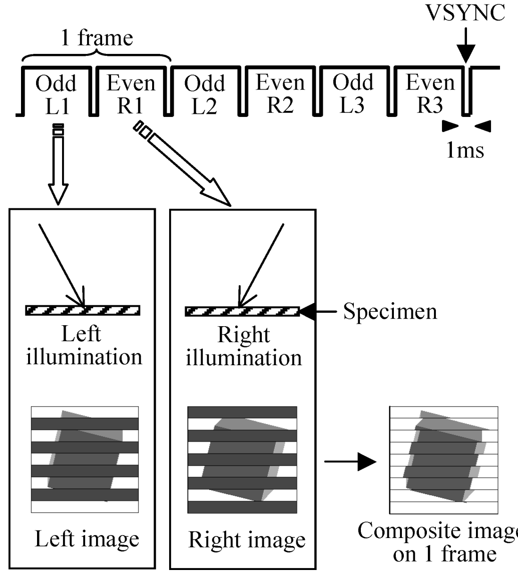

Figure 1 Principle of real-time stereoscopy.

2. Method

2.1. Principle of real-time stereoscopy

The development of this system enables the display of stereo pairs at a time resolution of 1/30 s using the NTSC video signal from a video camera. One frame of the NTSC signal is composed of 525 horizontal scanning lines, and displays scanning lines of odd numbers (odd field) and even numbers (even field) alternately every 1/60 s. The field is switched by a periodic pulse signal, called the vertical synchronizing signal (VSYNC), included in the NTSC composite signal. The incident beam that illuminates the specimen is tilted to the right and left alternately every 1/60 s in synchronization with VSYNC. Switching of the incident angle must be finished within 1 ms of the VSYNC input. One stereo pair is recorded per frame by illuminating the specimen from the left for odd fields and from the right for even fields as shown in Fig. 1. Although the vertical resolution is reduced by half compared to ordinary video images, stereo pairs can be obtained at the video rate. A time series of stereo pairs is then transferred to a 3-D display.

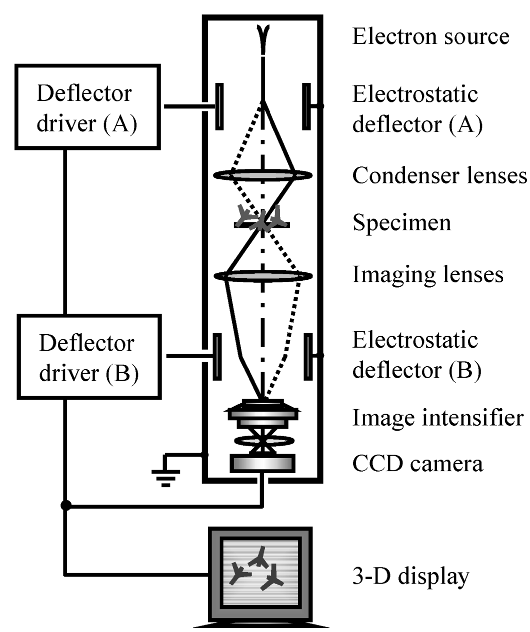

Figure 2 A schematic diagram of the real-time stereo TEM system developed.

2.2. System

The real-time stereo TEM has been developed using a 200 kV TEM (Hitachi HF-2000). Fig. 2 shows a schematic diagram of the real-time stereo TEM. The following four improvements from the basic system enabled us to obtain the stereo impression directly using the real-time stereoscopy with tilting illumination.

1. An electrostatic deflector (A) was installed above the illumination lenses for tilting illumination.

Switching of the incident angle must be finished within 1 ms after the 15 ms exposure. Because the response speed of magnetic deflectors originally employed in TEM was too slow, a new electrostatic deflector was installed. The incident angle is switched by reversing the polarity of the voltage of the deflector electrode in synchronization with VSYNC, which is extracted from the NTSC composite signal by a sync-separator IC (LM-1881) included in the deflector driver (A). The deflector driver (A) supplies the deflector (A) at a maximum voltage of ± 450 V. The condenser lenses are adjusted so that the deflector is located in the plane conjugate with the specimen plane, and the same area on the specimen is illuminated obliquely from the left and right. The incident angle required to obtain a sufficient stereo impression is greater than $\pm 2^\circ$. Because the maximum incident angle obtained only with the deflector (A) is about $\pm 0.8^\circ$, the condenser lenses are used in an image reduction mode to provide angular magnification of more than 25 times.

2. Another electrostatic deflector (B) was installed near the intermediate image plane of the objective lens.

Tilting illumination causes image blurring due to spherical aberration. Appropriate defocusing (underfocusing) improves the image quality. When the focus of the objective lens deviates from the Gaussian image plane, however, the corresponding left and right images are formed a large distance apart. The electrostatic deflector (B) compensates for the image separation and enables free focusing with the objective lens. The voltage supplied to deflector (B) is controlled by deflector driver (B) in the same way as deflector (A) and is adjusted in proportion to the amount of image separation. The image positions of the left and right images are corrected until the two images are overlaid, after the objective lens is focused on the objective plane of interest. Adjusting the image position facilitates the perception of a 3-D image, and reduces stress on the eyes.

3. A charge coupled device (CCD) video camera was used to record the time series of stereo pairs.

To record the stereo pairs by our method, it is necessary to record the right and left images independently in the corresponding fields. However, a vidicon-type image pickup tube often causes a long lag. For example, when a Newvicon tube was used in the base video camera system (Gatan 622), 20% of the residual image intensity remained even after three fields. This lag due to the image pickup tube was a severe hindrance to independent recording of right and left images. Thus, the Newvicon tube, which along with a scintillator and an image intensifier composed the original video recording system, was replaced with a CCD video camera, SONY XC-ST70, which avoids the lag.

4. New 3-D displays were used to give observers 3-D images directly without any special glasses and to aid in real-time analysis of 3-D images.

Parallax illumination and field-lens 3-D displays were adopted. The basic principles of these displays are well covered in the literature [9]. Here, we describe only parallax illumination for a display from Dimension Technologies Inc. (DTI 2015XLS).

Fig. 3a shows a schematic diagram of this display. The left and right images for a time series appear on the odd and even columns, respectively, of a liquid crystal display (LCD), as shown in Fig. 3b. These images are emitted to the eyes through a special illumination plate. This illumination plate generates a lattice of bright vertical lines. With these lines as backlights, left and right images on the LCD are projected separately at a suitable distance of 60 cm from the LCD. In this viewing zone, humans can perceive a stereo impression. This system also allows for more than one person to observe the same 3-D images simultaneously because this display provides two or more viewing zones as the broken lines in Fig. 3a illustrate. As mentioned in Section 2.1, the vertical resolution of the left and right images is reduced by half. The horizontal resolution is also reduced by half in this display because the left and right images are staggered horizontally as shown in Fig. 3a to project the left and right images to the corresponding eyes. As a result, the left and right images are displayed as in the checkered flag pattern shown in Fig. 3b.

3. Experimental

The voltages of the electrostatic deflector (A) and the NTSC composite video signal were measured simulta-

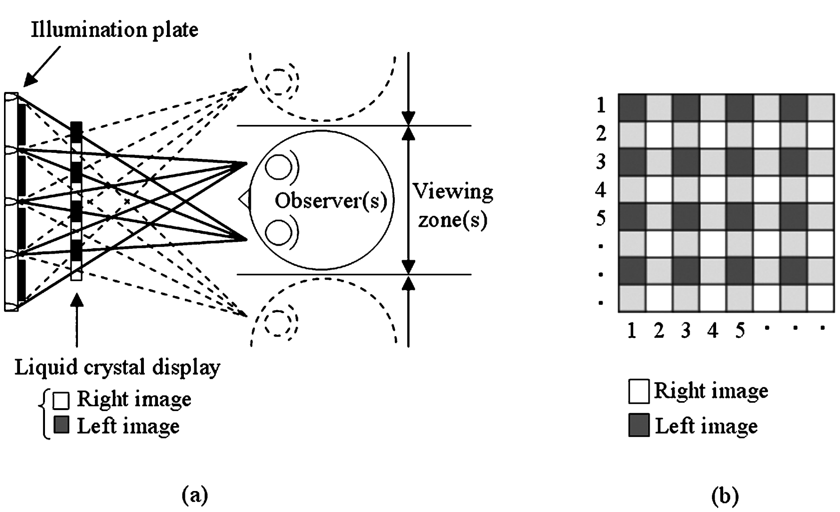

Figure 3 A schematic diagram of the 3-D display (DTI 2015XLS). (a) Basic principle of parallax illumination. (b) Arrangement of the right and left images on the liquid crystal display.

neously with an oscilloscope. The angle of the incident electron beam was determined from the shift in the 000 diffraction spot in the diffraction mode while changing the deflector voltage. In this experiment, Deflector (B) was not used.

Observation using the real-time stereo TEM was demonstrated with ZnO particles. The ZnO particles were 100 nm–1 μm in diameter with a tetrapod habit and were prepared by burning Zn powder in air with collection on a holey carbon film for TEM.

Additionally, dislocations were observed in a stainless steel thin film and an Al thin film as trial applications. Both specimens were prepared by jet electropolishing. A double-hole objective aperture was used to obtain bright field images. The diameter of the hole corresponds to 20 mrad and the centre of the holes from the optical axis corresponds to 40 mrad.

4. Results and discussion

4.1. Measurement of applied voltage to deflector (A) and incident angles

Fig. 4 shows the voltages of electrostatic deflector (A) (solid line) and the NTSC composite video signal from the CCD camera (broken line). It is confirmed that the polarity of the deflector voltage was reversed within 1 ms of switching the field. Fig. 5 shows multiple-exposed 000 diffraction spots taken while changing the voltage applied to deflector (A). The nine spots correspond to deflector voltages of 0 V, ± 100 V, ± 200 V, ± 300 V and ± 450 V, respectively. The maximum obtainable angles were ± 40 mrad ($\pm 2.3°$) measured from the shift in the 000 spots. This value corresponds to the convergence angle of human eyes when observing an object from 80 cm away.

As mentioned in Section 2.2, the condenser lenses are used on the condition that the image of the beam source is reduced and formed in the near side of the specimen. The

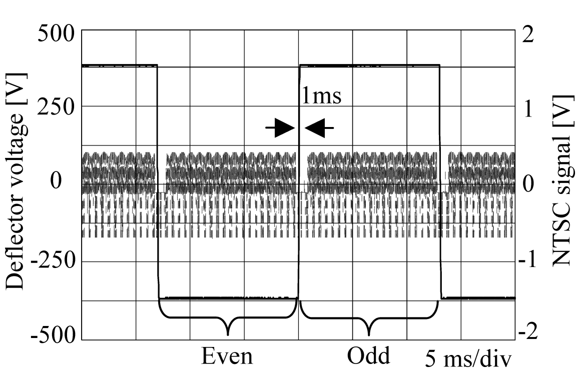

Figure 4 The voltage applied to the electrostatic deflector (A) and the NTSC composite video signal from the CCD camera. The deflector voltage is shown with a solid line and the NTSC signal is shown with a broken line. The horizontal axis indicates the time, and one scale division corresponds to 5 ms.

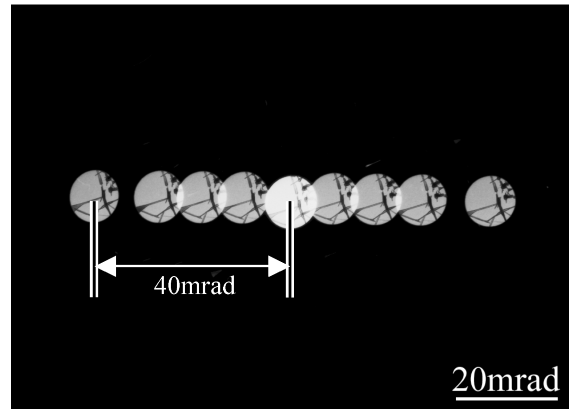

Figure 5 The multiple-exposed 000 diffraction spots taken while changing the voltage applied to the deflector (A). The spot at the centre of the figure corresponds to a voltage of 0 V.

specimen is illuminated by the beam that radiates from the source image. This means the incident angle is slightly different in each point in the field of view. Therefore, the centre spots appear as disks corresponding to the diameter of the selected-area aperture and the bright-field images of the specimen are formed on the centre spots. In Fig. 5, the spread angle of the illumination beam was 9 mrad. This spread did not influence the image quality when we observed at the magnification of 100,000 times or more.

4.2. Real-time stereo observation of ZnO particles

Fig. 6a shows a stereo pair of ZnO particles recorded on one frame with incident angles of $\pm 2.3°$. In the enlarged image (Fig. 6b), it is obvious that the different images are recorded on alternate odd and even lines. Fig. 6c shows the stereo pair separated from Fig. 6a. Mixed images, those including information from both sides, are not seen in the stereo pair, indicating that deflectors (A) and (B) work completely in synchronization with VSYNC. A strong stereo impression is obtained from stereo viewing of Fig. 6c using the cross-eye method.

4.3. Application to observation of dislocations

Fig. 7a shows a time series of stereo pairs of dislocations in the Al thin film, taken with incident angles of $\pm 2.3°$ and a double-hole objective aperture. The specimen's orientation was adjusted so that the diffraction vector of $\mathbf{g} = (1\ 1\ 1)$ is excited similarly with left and right illumination as shown in Fig. 7b. We succeeded in observing dislocations in real time while moving the specimen stage. The dislocations themselves did not move during this experiment. The same dislocations are seen in the right and left images of each stereo pair and almost no variation in diffraction contrast is recognized. Some dislocation lines

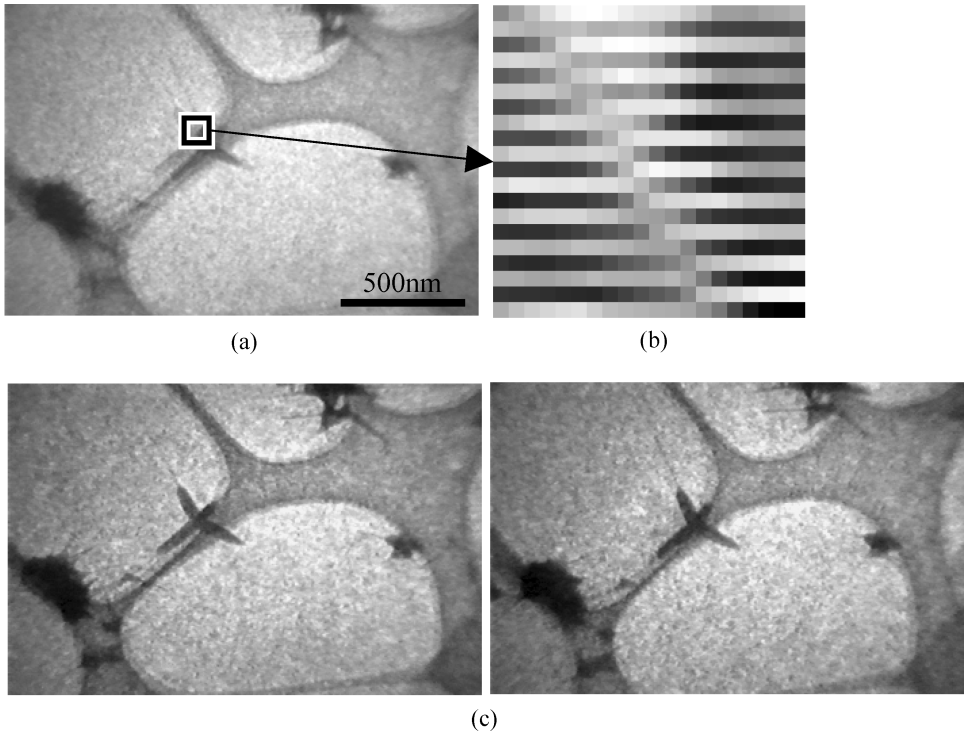

Figure 6 Stereo pair of ZnO particles taken with incident angles of $\pm 2.3^{\circ}$. (a) The composite stereo pair on one frame. (b) An enlargement of the enclosed area in (a). In the image, the two different images appear alternately. (c) The stereo pair separated from Fig. 6a. With this pair, a stereo impression is obtained using the cross-eye method.

in the first stereo pair of Fig. 7a are shown as solid lines. It can be seen that the parts indicated by arrows come from the background to the surface when observing with cross-eye method.

For effective stereo observation of dislocations in crystalline materials, the same diffraction condition must be maintained while a stereo pair of dislocations is recorded. In other words, the tilt direction of the incident beam must be arranged to be parallel with the Kikuchi line of interest. The developed system might encounter difficulties in adjusting the diffraction condition because the tilt direction is restricted by the positions of the two holes in the objective aperture. With a specimen of known orientation and a rotation specimen holder, real-time stereo observation could be performed more easily.

4.4. Criterion for recording high-speed motion without afterimages

It should be noted that the tracks of movement appear in the stereo pairs as afterimages when a high-speed motion is recorded by a video camera. The existence of afterimages makes it impossible to obtain depth information for objects from the stereo pairs. When a CCD video camera is used, the afterimage of a moving object can be suppressed using the following condition for an object travelling at a speed V along the horizontal direction of the CCD, within the length of one pixel.

$$T \leq \frac{0.2s}{mMV} \quad (1)$$

where T is the time resolution of the CCD video camera, s is the horizontal size of the CCD, m is the number of horizontal pixels of the CCD, and M is the image magnification on the CCD. The factor of 0.2 means tracking of the movement is allowed to extend over two pixels with a probability of 20%. Substituting the experimental parameters for the CCD camera ($T = 1/30$ s, $s = 8.8$ mm and $m = 640$ pixels), the following relation is obtained.

$$V \leq 82.5/M \ [\mu\text{m/s}] \quad (2)$$

This means that a movement of $V \leq 16.6$ nm/s can be observed at $M = 5000$ (ignoring the factor of 0.2 in Equation 1, $V \leq 83$ nm/s). We may be able to observe 3-D motion of dislocations considering reports on typical dislocation velocities for metal and semiconductor thin films [4, 10].

5. Conclusion

We have developed real-time stereo TEM using tilting illumination. This system enables direct observation of

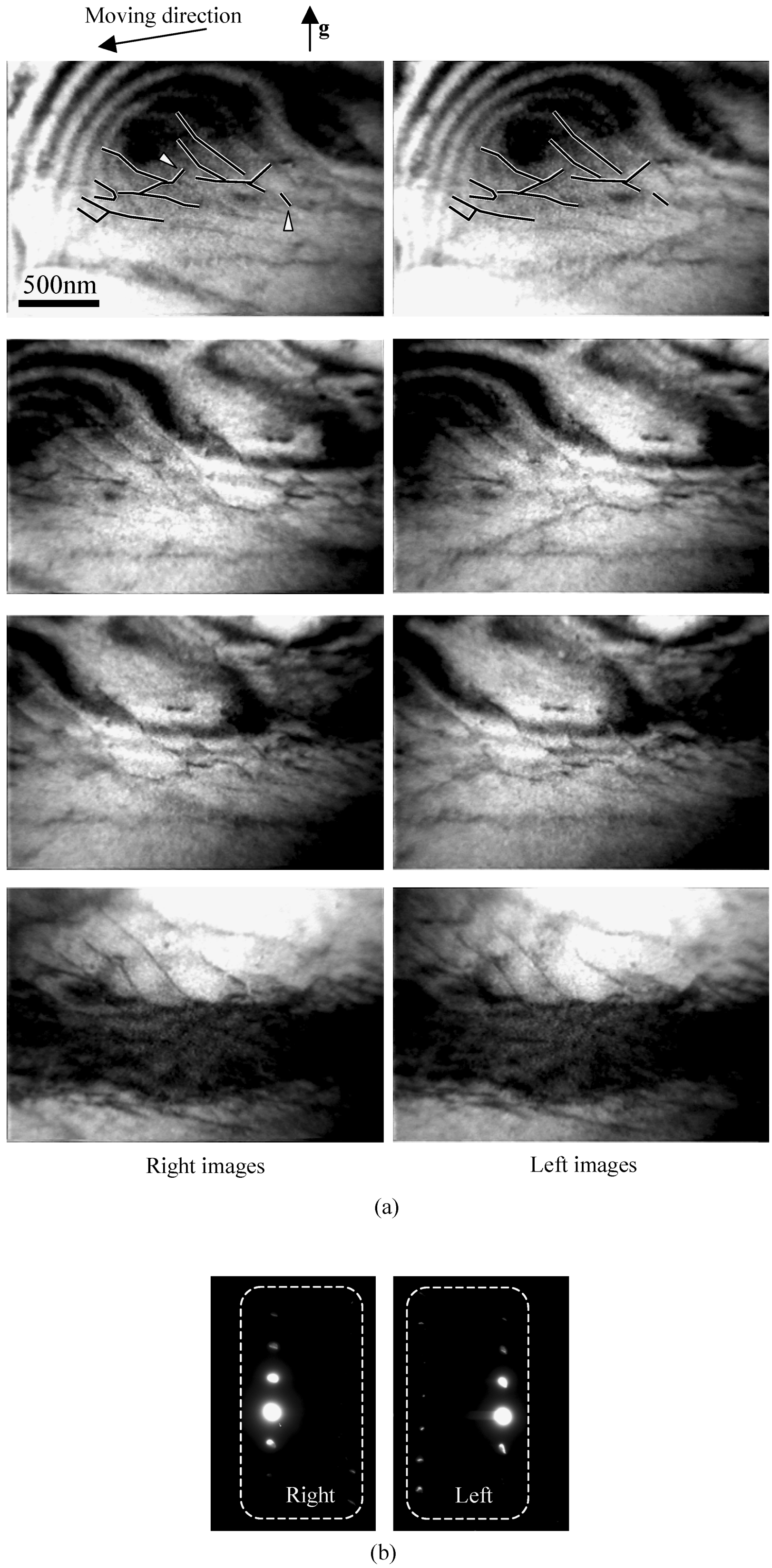

Figure 7 Real-time observation of dislocations in an Al thin film. (a) The time series of stereo pairs. The two arrows at the upper-left indicate the direction in which the specimen stage was moved and the direction of the diffraction vector **g**. The typical dislocation lines are shown in the first stereo pair by the solid line. A stereo impression is obtained with the cross-eye method. (b) Diffraction patterns taken with right illumination and left illumination for the observed area.

3-D images, displaying a time series of stereo pairs at the NTSC video-rate of 1/30 s. The incident beam is tilted obliquely left and right up to 2.3° by the electrostatic deflector in the illumination system and the image separation between the left and right images caused by defocusing is corrected by another electrostatic deflector in the imaging system. Real-time observation was realized for ZnO particles. The 3-D images were obtained with the 3-D display even with movement of the specimen. We also succeeded in observing dislocations in an Al thin film. Adjustment of the diffraction condition will be much easier with a specimen that has a known crystal orientation. This system will enable dynamic observations of defects in crystalline materials for *in situ* experiments.

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