

Figs. 1-3 have been interpreted in terms of a hexagonal form of the  $\omega$ -phase.

The results of the present experiments raise the question as to the mechanism which leads to different populations of four  $\omega$ -phase variants. Electron diffraction patterns similar to that shown in Fig. 1 were obtained from all thick regions of the specimens. No evidence was obtained from these patterns to suggest that in these regions the population of the four  $\omega$ -phase variants was anisotropic. Figs. 2 and 3 were obtained from thin regions of the specimens where the  $\omega$ -phase variants show marked anisotropy in population. This indicates that the anisotropy may arise when an isotropic specimen (in the sense of equal population of the four  $\omega$ -phase variants) is thinned. Presumably preferential reversion of some variants of the  $\omega$ -phase to the parent  $\beta$ -phase occurs as a result of stress relaxation of the thin areas of the previously deformed foils.

None of the work published purporting to show that  $\omega$  has a cubic structure [11-14] is inconsistent with the hexagonal interpretation. The results of the present experiments are, however, inconsistent with the proposed cubic structure. Thus it must be concluded that the cubic interpretation of the omega phase structure is not valid.

### Acknowledgements

The authors are indebted to Mr S. Chinniah for photographic work and to the National Research Council of Canada for research funds.

### References

1. B. S. HICKMAN, *J. Mater. Sci.* **4** (1969) 554.

2. S. L. SASS, *Acta Metallurgica*, **17** (1969) 813.
3. M. J. BLACKBURN and J. C. WILLIAMS, *Trans. Met. Soc. AIME*, **242** (1968) 2461.
4. J. C. WILLIAMS and M. J. BLACKBURN, *Trans. ASM* **60** (1967) 373.
5. W. G. BRAMMER, JUN, and C. G. RHODES, *Phil. Mag.* **16** (1967) 477.
6. D. J. COMMETTO, G. L. HOUZE, JUN, and R. F. HEHEMANN, *Trans. Met. Soc. AIME*, **30** (1965) 233.
7. B. A. HATT and J. A. ROBERTS, *Acta Metallurgica* **8** (1960) 575.
8. J. M. SILCOCK, *Acta Metallurgica* **6** (1958) 481.
9. B. A. HATT, J. A. ROBERTS, and G. I. WILLIAMS, *Nature (Lond.)* **180** (1957) 1406.
10. J. M. SILCOCK, M. H. DAVIES, and H. K. HARDY, A Symposium, Monograph and Report Series, No. 18, p. 93 (London, Inst. of Metals, 1956).
11. E. U. LEE, *J. Appl. Cryst.* **3** (1970) 413.
12. C. HAMMOND, The Morphology of the Omega Phase, Presented at the International Conference on Titanium in London (May 23, 1968).
13. A. E. AUSTIN and J. R. DOIG, *Trans. Met. Soc. AIME* **209** (1957) 27.
14. W. M. PARRIS, C. M. SCHWARTZ, and P. D. FROST, Precipitation Hardening and Embrittlement of High-Strength Titanium Alloys, WADC Technical Report 54-355 (1955) Part II.
15. M. K. KOUL and J. F. BREEDIS, *Acta Metallurgica*, **18** (1970) 579.

Received 6 December 1971

and accepted 10 February 1972

T. S. LUHMAN

A. E. CURZON

Physics Department

Simon Fraser University

Burnaby 2

British Columbia, Canada

### Scanning electron microscope selected-area channelling patterns: dependence of area on rocking angle and working distance

In the selected-area channelling pattern (SACP) method in which the electron probe is made to rock about a "point" on the specimen surface, [1] the minimum selected-area size  $d$  is given in theory by  $d = \frac{1}{2}C_s\phi^3$ , where  $C_s$  is the spherical aberration coefficient of the final lens, and  $2\phi$  is the total angle of rock. The value of  $C_s$  depends on the working distance ( $WD$ ) of the final lens,

and so  $d$  depends on both  $2\phi$  and  $WD$ .

For many investigations, it is necessary for  $d$  to be small, e.g. in the 1 to 10  $\mu\text{m}$  range, and the conditions favouring this are small  $2\phi$  and small  $C_s$ . The value of  $2\phi$ , the angular width of the SACP, cannot generally be decreased much below 8 to 9° if useful patterns are to be obtained. However, the value of  $C_s$  can be decreased by using small values of  $WD$ , i.e. placing the specimen close to the final lens. It was for this reason that the SACP studies made during the last few years at the Westinghouse Research Laboratories [2, 3] were mostly performed with a

true  $WD$  of 2 mm. It was then found experimentally that for  $2\phi = 9^\circ$ , a value of  $d = 4$  to  $5 \mu\text{m}$  was routinely obtained (without applying any corrections to compensate the spherical aberration of the final lens).

It would clearly be valuable when either designing or performing SEM experiments involving the SACP method if values of  $d$  were known in advance for a wide range of values of  $2\phi$  and  $WD$ . The purpose of the present communication is to report such values that have been experimentally measured.

The SEM used was a Cambridge Instrument Company Stereoscan Mark 2A. Typical electron probe parameters at the specimen were: spot size  $-1 \mu\text{m}$ , beam divergence  $-2 \times 10^{-3}$  radians, beam current  $-4 \times 10^{-9}$  A. The SACP method was set up in the standard manner [1], and a carbon replica of an optical diffraction grating with line spacing  $0.88 \mu\text{m}$  was used as the specimen. A through-focus series of photographs was obtained by advancing the specimen along the electron optical axis of the SEM in steps of typically  $20 \mu\text{m}$  at a time using the appropriate micrometer movement, and keeping the final lens

excitation current constant. From an analysis of the distortions of the lines in the SACP images, extremely accurate plots of the electron ray paths were obtained. The results revealed the precise position for the minimum selected-area size, and gave directly the value of  $d$ , for this particular  $2\phi$  and  $WD$ . The procedure was then repeated for other values of  $2\phi$  and  $WD$ . The method will later be fully described elsewhere.

The manner in which  $d$  was found to depend on  $2\phi$  and  $WD$  is shown in Fig. 1. Two scales are given for  $WD$ . "A" is the reading shown on the  $WD$  meter, while "B" is the true  $WD$  measured from the specimen to the final lens plate, which is the lower pole-piece of this lens. Two scales are also given for the final lens current. "C" is for 30kV type Stereoscans (irrespective of the voltage used), and "D" is for 20kV type Stereoscans (irrespective of the voltage used). Typical values of  $d$  for different operating conditions taken from Fig. 1 are as follows. For  $2\phi = 9^\circ$ , and for true  $WD = 1$  and 6 mm, then  $d = 3$  and  $20 \mu\text{m}$  respectively. For  $2\phi = 7^\circ$ , the corresponding values are 1.3 and  $9 \mu\text{m}$  respectively, and for  $2\phi = 13^\circ$ , the corresponding values are 9 and  $60 \mu\text{m}$  respectively. The considerable decrease in  $d$  on going from a true  $WD = 6$  mm, typical of that mostly used hitherto, to a true  $WD = 1$  mm, is immediately apparent.

The availability of such information regarding  $d$  not only enables the feasibility of particular experiments to be determined in advance, but helps in selecting the optimum electron probe size when using the SACP method. For example, for a true  $WD = 9$  mm and  $2\phi = 9^\circ$ , then  $d = 40 \mu\text{m}$ . In this case, the probe size could be set at, say,  $10 \mu\text{m}$ , rather than the  $1 \mu\text{m}$  often used, without significantly changing the value of  $d$ . Hence, either the resulting increase in electron beam current could be accepted, or the beam divergence decreased, both improving the SACP quality.

**Acknowledgements**

The authors wish to thank Drs D. C. Joy and C. G. van Essen for useful discussions.

**References**

1. C. G. VAN ESSEN, E. M. SCHULSON, and R. H. DONAGHAY, *Nature* **225** (1970) 847, and *J. Mater. Sci.* **6** (1971) 213.
2. G. R. BOOKER, Proc. 4th SEM Symposium, Chicago (IIT Research Institute) (1971) p. 465.

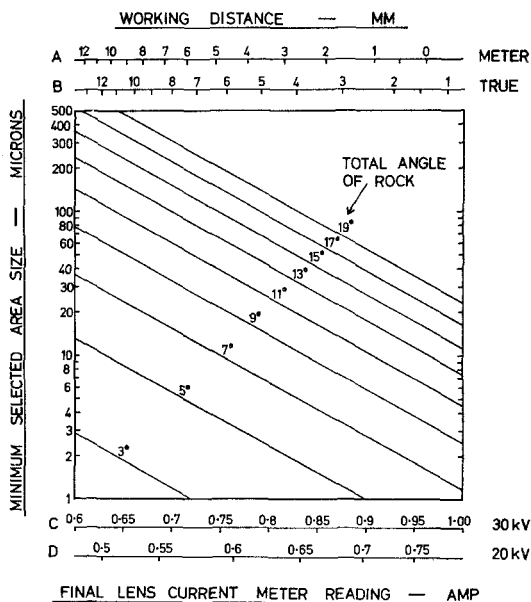


Figure 1 Experimental results showing how the minimum selected area size ( $d$ ) varies with the working distance of the final lens ( $WD$ ) and the total angle of rock of the electron beam ( $2\phi$ ). Results include some interpolation and extrapolation.

3. R. STICKLER, C. W. HUGHES, and G. R. BOOKER,  
*ibid.* p. 473.

Received 20 December 1971  
and accepted 24 February 1972

G. R. BOOKER  
*Department of Metallurgy*  
*University of Oxford*  
*England*

R. STICKLER  
*Westinghouse Research Laboratory*  
*Pittsburgh, Pennsylvania, USA*

### *Scanning electron microscope selected area channelling patterns from 1 micron specimen areas*

In the scanning electron microscope (SEM) selected-area channelling pattern (SACP) technique, the beam is rocked about a point on the specimen surface. This rocking action is achieved by the combined action of the scan coils and final lens as described by van Essen, Schulson and Donaghay [1]. In this condition, the smallest area which can be selected is determined by the spherical aberration of the final lens and the total included angle of rock. This limitation has been discussed from a theoretical standpoint by van Essen *et al* [2], and a careful series of practical measurements have been made by Booker and Stickler [3] to determine the variation in the selected area as a function of both angle of rock and specimen working distance.

To reduce the area below this limit set by aberrations, it is necessary to provide some form of correction signal to either the scan-wave forms or to the lens. The most practicable scheme is that suggested by van Essen [4] in which the final lens is dynamically focused. Since at any one time only one scan-ray orientation is passing through the lens, it is possible to vary the strength of the lens as a function of time so as to keep the crossover point of the scan rays exactly at the desired point. This is difficult to do successfully when using standard Cartesian scanning because the angle of scan changes from a maximum to a minimum at the line rate. The Cartesian scan is therefore replaced by a spiral scan with a frame repetition rate of approximately 5 sec and a circular frequency of 50 Hz. The angle of incidence in this mode increases linearly with time during each 5 sec frame scan period, and the necessary correction need only be supplied at the same rate. The system is set up by observing a piece of fine mesh grid (12  $\mu\text{m}$  repeat) in the selected area mode and adjusting the correction amplitude until the distortion due to the spherical aberration of the lens is at a

minimum. Once set the correction need not be adjusted again.

The other lenses in the system are set so as to produce a probe size of approximately 0.5  $\mu\text{m}$ , a beam divergence of  $3 \times 10^{-3}$  radians, and a beam current of  $3 \times 10^{-9}$  A at the specimen. These values are chosen to give a pattern of adequate resolution together with sufficient beam current to provide the necessary signal to noise ratio to record the low level channelling contrast. For a source of given brightness no one of these figures can be improved without worsening the others. By changing the gun from a tungsten hair-pin source (of brightness  $5 \times 10^4$  A  $\text{cm}^{-2}$   $\text{sr}^{-1}$  at 20 kV) to a pointed filament ( $2 \times 10^5$  A  $\text{cm}^{-2}$   $\text{sr}^{-1}$  at 20 kV) or a field emission source ( $10^7$  A  $\text{cm}^{-2}$   $\text{sr}^{-1}$  at 20 kV), it would be possible to increase the beam current, or reduce the probe size or beam divergence.

For a number of investigations at present underway in the Department, it was important for SACP's to be obtained with a total angle of rock of  $9^\circ$  and from areas only 1  $\mu\text{m}$  across, a combination not hitherto achieved. In order to do this, it was decided (a) to work with the smallest selected-area size possible that could be obtained without corrections by working with the shortest practicable final-lens working distance, and then (b) to use spiral scan and apply dynamical focusing as described above.

It was found that a true working distance of 1 mm could be used as long as the specimen current was taken for the image signal, thereby avoiding the difficulty of collecting sufficient emitted electrons from a rather restricted geometrical configuration. From the results of Booker and Stickler [3] (see Fig. 1 of the previous paper in this journal), for a total rocking angle  $2\phi = 9^\circ$  and a true working distance  $WD = 1$  mm, the selected-area size is  $d = 3$   $\mu\text{m}$ . This value was closely obtained in practice in the present work.

The spiral scan system and circuitry used to subsequently apply the correction were those described by van Essen [4]. However, special