

A REVIEW: HOLZ lines and lattice parameter determination

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ABSTRACT

In convergent-beam electron-diffraction patterns at a zone axis orientation, the bright-field disc is often crossed by a set of fine dark lines - the HOLZ lines. These lines are formed because the diffracted beams are divided into well separated Laue zones. The lines are valuable for the determination of the *local* lattice parameter of the crystal in the small volume traversed by the electron beam. This is a tutorial review that summarizes the usefulness and limitations of the method.

INTRODUCTION

The discs in a zone-axis convergent-beam pattern (particularly the bright-field disc) are frequently crossed by a set of fine dark lines called HOLZ lines (Figure 1). Apart from their part in the general analysis of convergent-beam patterns, for example their contribution to symmetry determination, these lines are valued for their sensitivity to variation in lattice parameter. The measured positions of the lines can be used to determine local lattice parameter or strain in the sample. This paper aims to be a tutorial review of this subject.

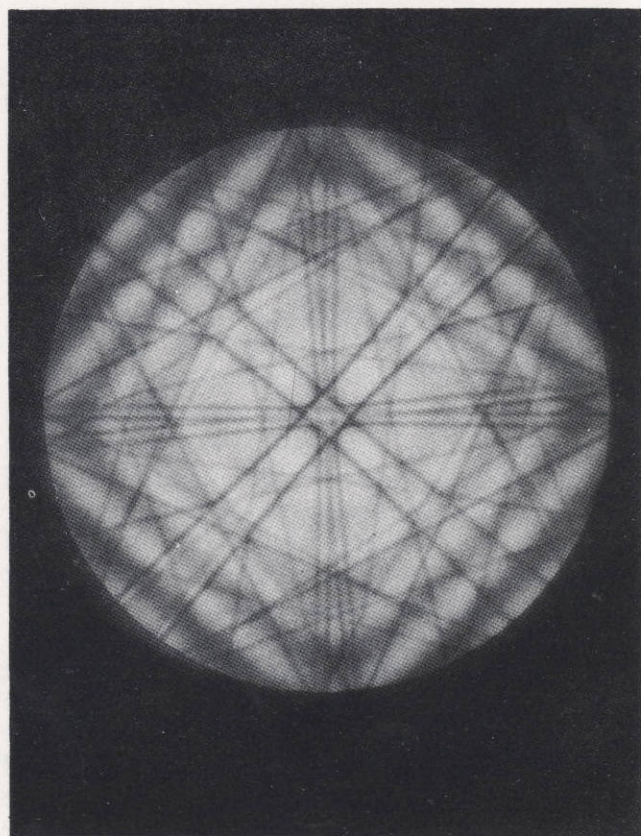


Figure 1.

Bright-field disc of a convergent-beam pattern of silicon at the [100] zone axis. 100 kV, sample at liquid nitrogen temperature.

KEY WORDS

Convergent-beam diffraction, HOLZ lines, lattice parameter, strain.

ORIGIN OF THE LINES

Bragg's law expresses the result that for each set of planes in a crystal there will be a diffraction peak at a position given by

$$2d \sin \Theta = \lambda$$

or more conveniently in three dimensional notation

$$\mathbf{k}' - \mathbf{k} = \mathbf{g}.$$

It is one of the most important characteristics of electron diffraction, as opposed to x-ray or neutron diffraction, that diffraction occurs for a relatively wide range of angles about the exact Bragg angle. This is why, in a convergent-beam pattern, the diffracted intensity fills the whole area of the disc. However, the width of the diffraction peak about the Bragg angle is not a constant. It is determined by the "strength" of the diffracting planes. If the crystal potential is expressed as a sum of Fourier components

$$V = \sum V_g \exp -2\pi i \mathbf{g} \cdot \mathbf{r}$$

then we can associate each component, V_g , with a set of planes. As far as electron diffraction is concerned, V_g is the strength of the diffraction of the planes g . Depending on the arrangement of the atoms in the unit cell, the V_g will vary, but overall the values of the V_g will drop off uniformly from the origin because the scattering of a single atom decreases with increasing scattering angle.

So, as g becomes larger, there is a tendency for the diffraction to become more and more localized, to occur within a smaller range of angles about the Bragg angle. Thus for a large enough g , a convergent-beam disc will not fill with intensity but show only a bright line along the position of the Bragg condition.

When a crystal is oriented so that the electron beam is parallel to a zone axis, the diffraction pattern is naturally divided into Laue zones (Figure 2). As can be seen from the Ewald sphere construction, the zones correspond to the intersection of the Ewald sphere with successive planes in the reciprocal lattice. This geometry produces a situation in which there is a sharp break in the magnitudes of the g 's. The reflections in the zero layer (the zero-order Laue zone - those reflections in the reciprocal lattice plane through

the origin that make up the group of reflections near the direct beam) all have small values of g , while reflections in the first-order Laue zone have much larger values of g . There is a distinct gap between the two groups. For many zone axes in many materials, this gap coincides with the range of g that carries the width of the diffraction peak from wide to narrow. Wide and narrow are not absolute terms of course. Here wide

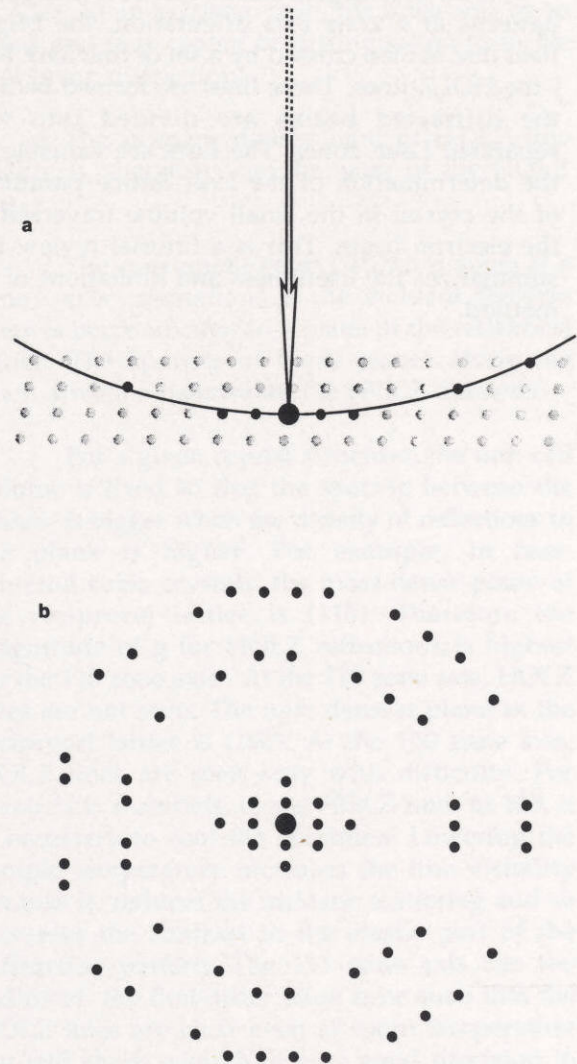


Figure 2.

a) Schematic diagram of the Ewald sphere construction at a zone axis orientation. At the origin, the sphere is tangent to a plane of the reciprocal lattice. The reflections from that plane form the zero-order Laue zone.

b) Schematic diagram of the diffraction pattern corresponding to the situation shown in a).

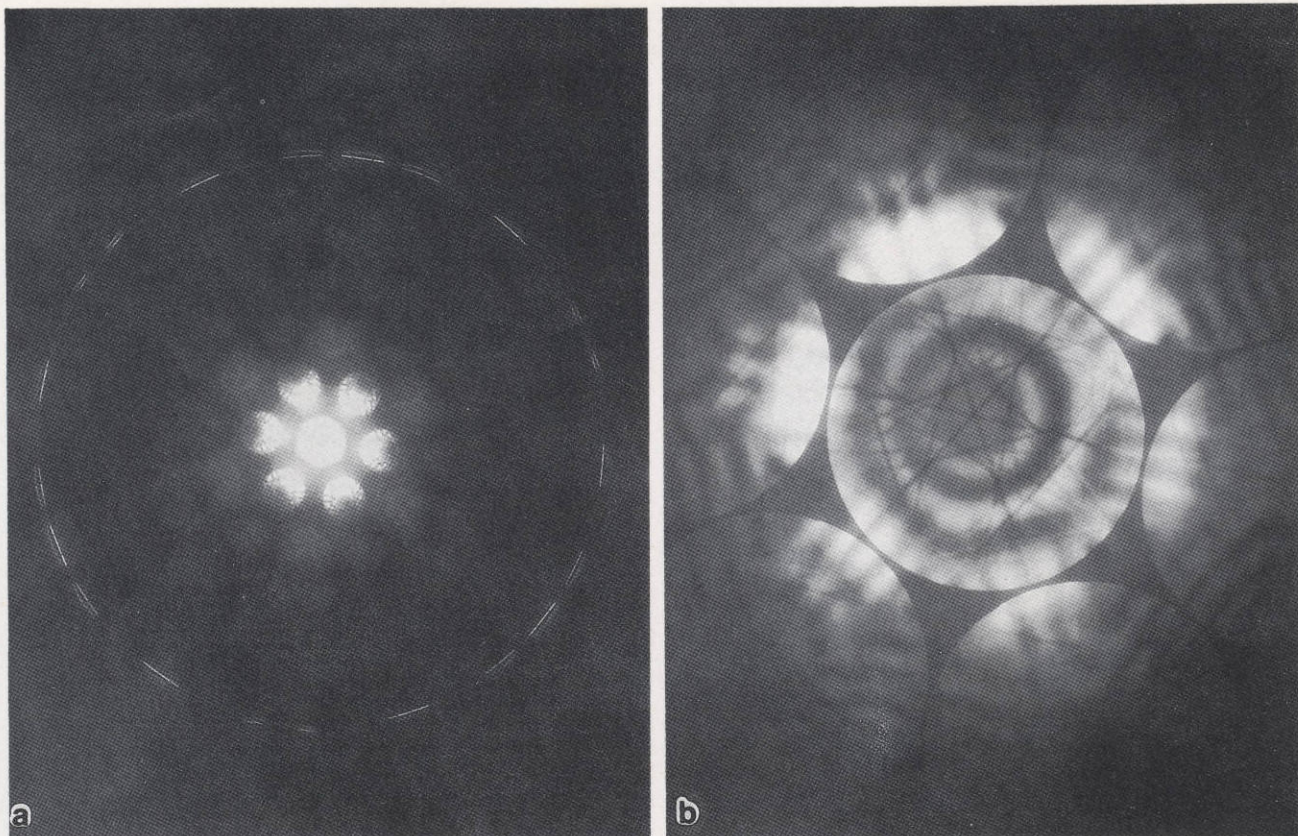


Figure 3.

a) Short-camera length convergent beam pattern from silicon at the [111] zone axis. 100 kV (approx.). b) The same, but at a longer camera length to show the central part of the pattern in more detail. For each dark line in the bright-field disc there is a corresponding bright line in the first-order Laue zone.

means of the order of the smallest Bragg angle in the crystal. In such a case, the illumination will fill a convergent-beam disc. Correspondingly, narrow means a good deal smaller than the diameter of a convergent-beam disc. In such cases the illumination will be confined to a narrow line within the disc (Figure 3).

The bright line in the reflection from the first-order Laue zone removes electrons from the corresponding position in the discs of the zero order reflections - in particular from the bright-field disc - so that a dark line is produced. As indicated above, the Laue zones are numbered starting with zero for the layer through the origin. All Laue zones other than the zero-order zone are known collectively as higher-order Laue zones, hence the acronym HOLZ. The zero-order Laue zone is sometimes referred to, for reasons of euphony, as the zero layer.

Dynamical effects introduce complications into this picture but leaving them aside for the moment, we have a clear and rather simple picture of the origin of HOLZ lines. Each line corresponds to diffraction into a particular reflection in a higher-order Laue zone. For each reflection in the HOLZ ring itself, there is a dark line in the bright-field disc in a position matching the position of the HOLZ reflection in the HOLZ disc (Figure 4). In this model, the position of each line is predicted directly from Bragg's law.

MEASURING LATTICE PARAMETER

In a more conventional method of measuring lattice parameter, whether by x-rays or by electrons, the lattice parameter is determined by measuring the Bragg angle. More precisely, the angle measured is the angle between the incident beam and the diffracted beam, which is twice the Bragg angle. The equivalent measurement here would be to measure the distance between the line in the bright-field disc and the line in the corresponding HOLZ reflection. However, in this case we can make a great improvement in the precision of the result by making the measurement differently.

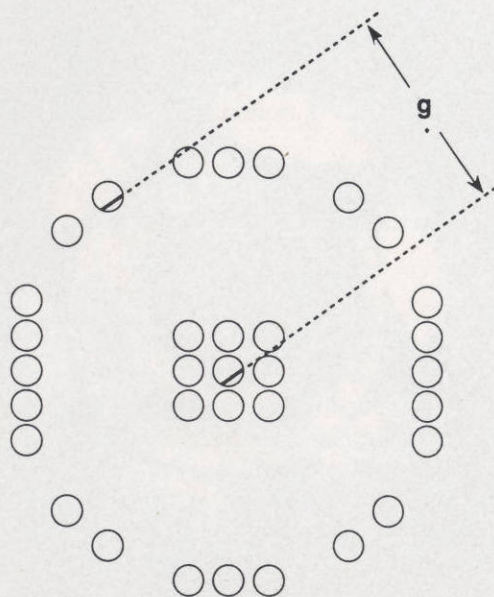


Figure 4.

Schematic diagram to show that the HOLZ line in the bright-field disc lies in the same position as the line in the HOLZ reflection (even if the disc itself is not normally visible for the reflections in the HOLZ ring). The separation between the two lines is g (if the diffraction pattern is treated as in reciprocal space) or $2\Theta_B$ (if the pattern is treated as in angular space).

The principle is to measure the position of a single line - HOLZ line - with respect to a nearby reference point. Not only is it more precise to do it this way, but it also avoids systematic errors and the correspondingly awkward calibration problems. Normally we are looking for a small change in lattice parameter, or what is equivalent, the strain in the crystal. In this case it is not a good idea to measure the distance between the line in the HOLZ reflection (sometimes called the excess line) and the corresponding line in the bright field disc (sometimes called the deficit line), because then we are trying to measure a small change in a large distance. This is intrinsically imprecise but also introduces additional errors because such a measurement requires a short camera length diffraction pattern and such patterns are very liable to have substantial distortions in them.

Conversely, if the position of the line in the bright field disc is determined with respect

to a local point of reference, these difficulties are avoided. The change in lattice parameter is measured precisely because the shift is measured directly. Distortions are not a problem because the measurements are all made close to the center of the pattern where distortions are negligible.

The point of reference used to determine the position of the HOLZ line could be the zone axis. The zone axis is clearly identifiable and would thus make a convenient point from which to measure. However, it is generally hard to locate the zone axis (the center of the pattern) with sufficient accuracy especially when the symmetry of the pattern may be broken by the very strain that we wish to determine. A better way turns out to be to use, not a fixed reference, but the positions of the HOLZ lines with respect to each other (Figure 5).

In early work in this field, it was noted that if two lines intersect at a small angle (and if a change in lattice parameter causes them to move in opposite directions) the point of intersection moves much farther, for a given change in lattice parameter, than the lines themselves. Thus a measurement of the distance between two intersection points, appropriately chosen, would give a more precise value than a direct measurement of the line shift. Nowadays it is more usual, indeed it is almost universal, to simulate the pattern of all the lines in the HOLZ disc with a computer program. [1] The advantage of this technique in addition to its convenience is that it allows us to obtain the best fit while varying all the unit cell parameters, something that would be extremely difficult making measurements between intersections (Figure 6).

HOLZ LINES VERSUS KIKUCHI LINES

A HOLZ line that occurs in the disc of a convergent-beam diffraction pattern is formed at the locus of positions at which Bragg's law is satisfied for a set of planes in the crystal. In this respect HOLZ lines are similar to some other lines that appear in electron diffraction. The Kikuchi lines that are formed in the diffusely scattered inelastic background of electron diffraction patterns also lie at the locus of Bragg reflection for the atomic planes. In Tanaka patterns (or wide-angle convergent-beam patterns), there are also lines at these positions. They are distinct from Kikuchi lines in that inelastic scattering is not involved and in that

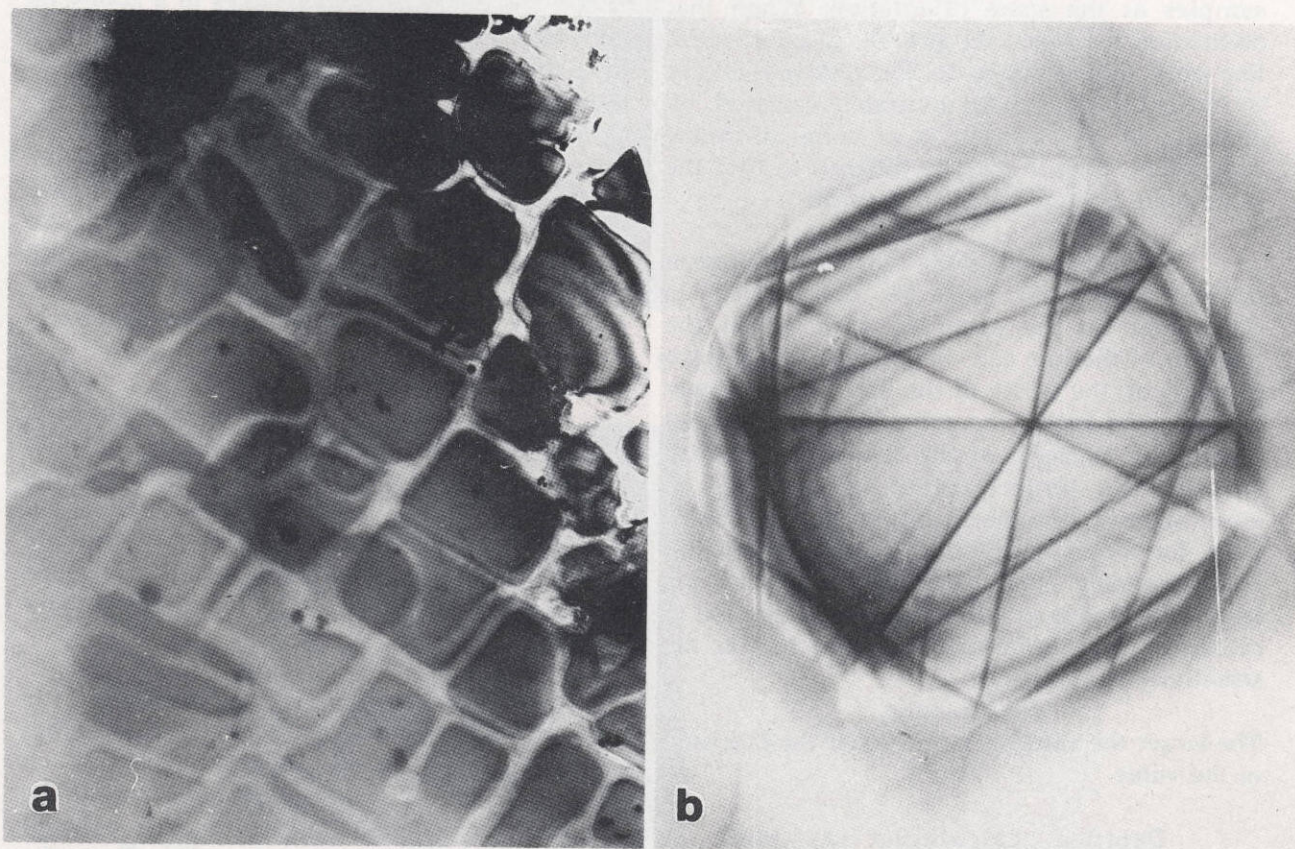


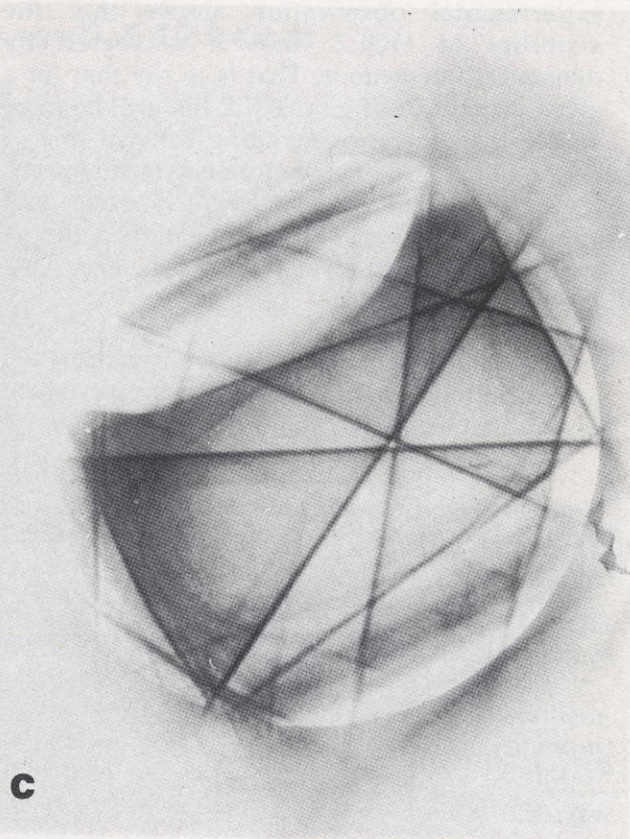
Figure 5.

a) TEM micrograph of a nickel-based superalloy. Two phases are present: the matrix, γ , and the ordered cuboid precipitates, γ' . b) Bright-field disc of a convergent-beam pattern from the γ' , c) The same but from the γ phase, 100 kV. The shift of the lines between the two patterns was used to show a lattice parameter change of about 0.5% between the phases. (Courtesy M.S. Dean, M. Sc. thesis, University of Bristol 1977).

the symmetries are different. The present author has used the term Bragg lines to distinguish these lines from Kikuchi lines.

Since all of these lines fall in positions determined by the same construction, it is appropriate to ask why HOLZ lines are favored over other such lines for measurement of strain and lattice parameter. There are three reasons for this.

1 In order to compare one sample with another it is important to be able to set the



samples at the same orientation. When that orientation is a zone axis, as is the case for HOLZ line work, this is easy to do. The zone axis is easy to recognize and can be accurately centered. By contrast, if the sample is at some arbitrary orientation, then it may be difficult to find the same angle again in the next specimen.

2 The precision with which the change in lattice parameter may be determined is dependent on the accuracy with which the position of the line can be judged. This in turn depends on the width of the line. The narrower the line, the better the precision. As indicated above, the strength of the line is, in general, smaller for larger values of g . This is advantageous because it implies that the finest lines, for which the position can be determined most accurately, are just those lines for which g is large. If a change in line position (Δg) is used to calculate a change in lattice parameter (Δa), the sensitivity is increased:

$$\Delta a/a = -\Delta g/g$$

The larger the value of g , the better the precision of the value.

Detailed calculation as well as experimental observation shows that the visibility of HOLZ lines is increased by dynamical interactions. That is to say that for a given magnitude of g , a HOLZ line will be more visible than the corresponding Kikuchi line or Bragg line. [2] This enhancement factor means that the HOLZ lines used at a zone axis typically have a g that is longer than the g 's that could be used away from a zone axis. Thus the precision is higher.

3 The same dynamical interaction that gives better precision in strain measurement, means that more reflections are within the useful

range. The density of HOLZ lines in a zone-axis pattern is higher than the density of lines in a Kikuchi pattern or in a Tanaka pattern. Moreover, the lines will all have g 's of similar magnitude and that magnitude will be near the optimum value for lattice parameter determination (i. e. the maximum that will give visible lines). Very often at a zone axis, there are many visible lines so that there are many parameters (the line positions) to fit to the unknowns (the unit cell dimensions and angles).

DYNAMICAL EFFECTS

The enhancement of the visibility of lines at the zone axis mentioned above has the advantage that it improves the precision of the measurement. However the same dynamical interaction produces an important complication. Where the dynamical interactions are strong, the position of the line is not that predicted by Bragg's law. The line is shifted.

This would appear to invalidate the method. However it can be shown that, at a given zone axis, all the lines are displaced as if Bragg's law is still valid but for a value of the electron wavelength (i. e. microscope voltage) which is not the true value. [3] Thus the microscope voltage has to be treated as an adjustable parameter rather than as a known value. The microscope voltage that is used in the kinematical calculation depends on the material, on the zone axis, and on the order of the Laue zone (i. e. the second-order lines - if visible - would need to be simulated using a different voltage from the first-order lines). Provided that these conditions are taken into account, dynamical effects do not restrict the ability of the technique to measure changes of lattice parameter. The method remains a precise method of determining strain in the crystal.

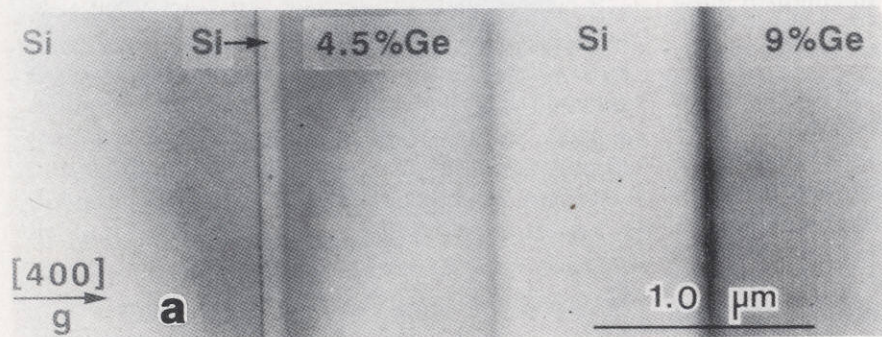
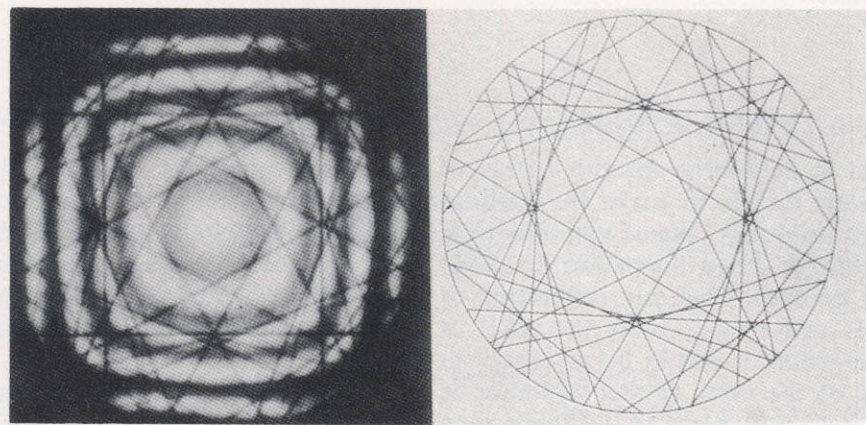
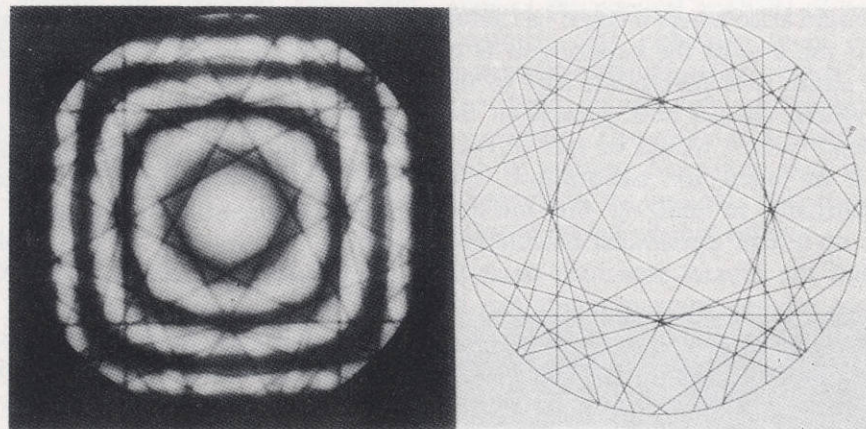


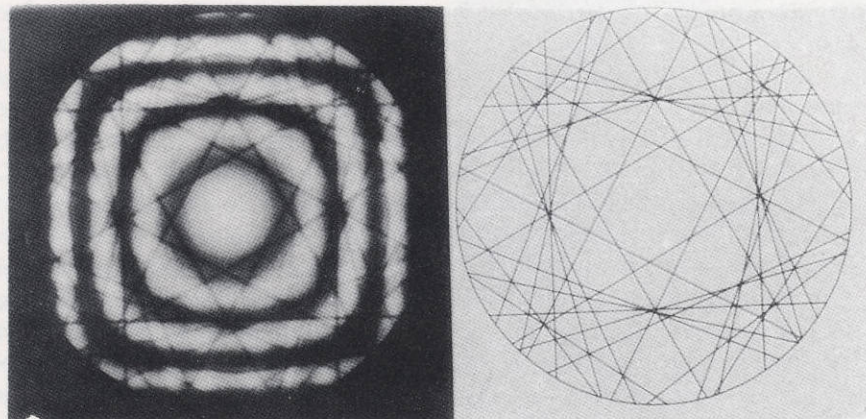
Figure 6 a) TEM micrograph of a cross-section of a thin film structure. The substrate, of pure silicon, is on the left. The third layer is of silicon alloyed with 4.5% germanium. The lattice parameter of the alloy would be different from that of pure silicon but, parallel to the interface, it is constrained to be the same as that of silicon because of the continuity of the planes.

**b**

$$a=b=c=0.54294\text{nm}$$

**c**

$$a=0.54335\text{nm} \quad b=c=0.54294\text{nm}$$

**d**

$$a=0.5441\text{nm} \quad b=0.54294\text{nm} \quad c=0.5441\text{nm}$$

Figure 6.

b) Bright-field convergent-beam disc from the pure silicon some way from the interface. To obtain the computer match the lattice parameter from x-ray diffraction is assumed and the microscope voltage treated as adjustable.

c) Bright-field convergent-beam disc from the alloy taken close to the interface with the silicon. The strain due to the substrate introduces a tetragonal distortion. The lattice parameters are obtained using the same voltage as for a), not considered adjustable now.

d) Bright-field convergent-beam pattern from the alloy further from the interface. Surface relaxation of the stresses changes the strain from tetragonal to orthorhombic. (Courtesy C.J. Humphries et. al. see Ultramicroscopy 26: (1988) 13-24).

In order to determine absolute values of lattice parameter, that is to make the method accurate as well as precise, one of two options is available, although they have rarely been undertaken in practice. They are calibration or computation. In order to calibrate the effect of dynamical interaction, it would be necessary to have a sample of the same crystal structure as the sample under study (and at the same zone axis) but when the lattice parameter is known (e. g. from x-ray diffraction). This may seem to negate the whole point of the exercise but circumstances could arise where it could be useful. For example, if the aim of the experiment is to determine the strain in a precipitate due to the surrounding matrix, then a bulk sample of the precipitate phase - which would be free of such strain - could be used for calibration. The alternative approach to making the method accurate is to do a full dynamical calculation. This means that the crystal structure must be known, whereas relative measurements can be made even for unknown phases. Until recently to do such calculations in full would have seemed too big a proposition. Now the full simulation can be done in an hour or so on a good work-station, so that it is quite reasonable to do it. [4].

An additional complication, due to the effects of dynamical diffraction, is that the HOLZ ring may show, not a single line in each reflection, but more than one. This is not usually a problem because, in most cases, only a single set of lines is visible in the bright-field disc. In the HOLZ reflection the lines are bright against a background that has very low intensity, thus two lines can be seen even when one is much brighter than the other. On the other hand, in the bright-field disc, the HOLZ lines are dark on a bright background. The contrast of the lines is very weak. The drop in intensity at the line is only a small fraction of the total intensity. In this case the visibility of the line depends critically on how strong the line is. Only the strongest line is generally visible.

CHOICE OF ZONE AXIS

Not all convergent beam patterns show HOLZ lines. Indeed, many do not. Two factors determine HOLZ line visibility: geometry and order. The strength of a HOLZ line depends on the magnitude of V_g (and the dynamical enhancement). V_g in turn depends on the magnitude of g

(and structure factor effects). Thus, if the zone axis is such that the magnitude of g for the HOLZ reflections is too large, the HOLZ lines will not be visible because the contrast will be too weak. Conversely, if the orientation is such that the magnitude of g is smaller, the precision of the determination will be poor, in part because g is smaller and in part because the lines are broader. In an extreme case, the lines will be so broad that they cannot be distinguished from the zero-layer diffraction.

The primary determinant of HOLZ line quality is, therefore, the diameter of the HOLZ ring.

The convergent-beam pattern is taken at a zone - axis orientation, so the incident electron beam is perpendicular to a plane of the reciprocal lattice. The spacing of these planes along the beam direction determines the HOLZ diameter.

For a given crystal structure, the unit cell volume is fixed so that the spacing between the planes is bigger when the density of reflections in the plane is higher. For example, in face-centered cubic crystals, the most dense plane of the reciprocal lattice is (110). Therefore the magnitude of g for HOLZ reflections is highest for the 110 zone axis. At the 110 zone axis, HOLZ lines are not seen. The next densest plane in the reciprocal lattice is (100). At the 100 zone axis, HOLZ lines are seen only with difficulty. For many f.c.c. materials, to see HOLZ lines at 100, it is necessary to cool the specimen. Lowering the sample temperature increases the line visibility because it reduces the inelastic scattering and so increases the contrast in the elastic part of the diffraction pattern. The 111 zone axis has the radius of the first-order Laue zone such that the HOLZ lines are clear even at room temperature but still sharp enough to give good precision in the determination of the lattice parameter. For this reason this orientation is frequently seen in publications that use HOLZ line analysis. Because 100 is a difficult zone axis, the nearby 411 zone axis is often used in the case of samples oriented close to 100.

In general the practical rule is to pick a zone axis that gives the largest diameter for the first-order Laue zone that still gives visible lines in the bright field disc. The choice of microscope

operating voltage is relevant. A lower voltage gives a larger electron wavelength and a smaller radius to the Ewald sphere. This reduces the diameter of the HOLZ ring. Raising or lowering the microscope operating voltage can therefore be used to change the visibility of the lines if necessary.

The other factor that affects the visibility of the lines is the degree of order in the crystal. When the temperature of the specimen is raised, the effect is more dramatic on high-order reflections (large g) than on low. This is because the high g reflections correspond to a small planar spacing and the atom motion due to lattice vibrations is larger as a fraction of the spacing between the planes. In a similar way, the effect of disorder in the crystal is to displace atoms from their sites (in the case of disorder, the displacement is constant while, in the case of thermal vibration, the displacement varies with time) and this displacement will have a more dramatic effect when it is large compared to the spacing of the planes. Disorder will wash out large angle diffraction effects including HOLZ lines. If HOLZ lines are invisible when they might otherwise be expected, one possible explanation is that there is some form of disorder in the crystal.

SURFACE RELAXATION

HOLZ line analysis leads to a measurement of the lattice parameter or strain in the region of the sample that the electron beam traverses. Of necessity this region is part of a thin foil, suitable for transmission electron microscopy. For some applications this is what is wanted. However in most cases, the measurement that is needed is the strain that was present in the bulk material prior to thinning for microscopy. In preparing a thin section from a bulk sample, two new surfaces are introduced and there can be no component of stress normal to these surfaces. Prior to the thinning, however, there would have been (in general) stress across these planes. The relaxation of these components of stress, as the new surfaces are exposed, will produce deformation in the sample - its state of strain will not be the same in the thin foil as it was in the bulk.

One way in which this effect can be handled is to make a model of the elastic state (stress/strain) of the bulk and from that to

predict the corresponding state in the thin film and to compare that with the experimental result. This is difficult and less than satisfactory because of the possibility that the solution is not unique.

EXPERIMENTAL LIMITS

A. Thickness

The detail in convergent-beam patterns develops only in specimens of a certain thickness. Very thin samples produce patterns in which the intensity in the discs is uniform. HOLZ lines appear clearly in foils that are somewhat thicker than those that give zero-layer detail, since the relevant extinction lengths will be greater. Conversely, if the sample is too thick, the HOLZ-line contrast will be lost in the diffuse scatter. Nonetheless there is a fairly broad range of specimen thickness for which good patterns can be obtained. Typically a sample thickness of about 100 nm is suitable.

B. Diameter

The size of the region that contributes to the pattern is the size of the focused beam plus any spreading of the beam in the specimen. For a sample on the order of 100 nm thick, the beam spreading will be perhaps 20 nm. Therefore there is no point in trying to work with a beam that is much smaller than this. Indeed, it may be desirable to use a larger beam - this gives more intensity and makes the experiment easier. There is a balance to be struck; a larger beam makes the experiment easier, while smaller beams tend to produce patterns of better quality.

C. Uniformity of strain

In principle there is no limit on the magnitude of the strain that can be measured (except that the sample must be able to support it). There is a limit on the uniformity of the strain. For the convergent-beam pattern to have a clear set of sharp lines, the strain must be constant within the volume that the beam traverses. Any strain gradient in that volume will produce a degeneration of the pattern that will make the strain difficult or impossible to measure. This is true whether the strain gradient is long range or localized. Defects in the crystal can produce such localized strain gradients. For example, dislocations have been shown to produce splitting and blurring of the HOLZ lines. [5].

D. Precision

How accurately the measurements can be made depends on the quality of the patterns (how fine the lines are) and how delicately the positions of the lines can be matched to the computer output. Characteristic values for the precision that can be attained lie in the range 10^{-3} to 2×10^{-4} . The best values may only be obtained by cooling the sample.

SUMMARY

HOLTZ lines are used for the determination of *local* values of the lattice parameters of the sample; if global values were required, x-ray methods would be preferred. The technique is particularly good at determining how the lattice parameter varies from place to place within the same sample or between different specimens of the same structure. Such local variation can arise from two main causes. The change can be due to local changes in composition or due to the stresses imposed on the region in question by the remainder of the sample. In both cases the technique can be very valuable although, in both cases, there are reasons to treat the data with caution.

When there is a local variation in composition, the shift of the HOLTZ lines has two components: the shift due to the change in lattice parameter and the shift due to the change in dynamical diffraction effects resulting from the composition change itself. Fortunately, for many systems and for reasonable changes in composition, the latter effect is small. [6] A naive interpretation of the data as resulting solely from the changes in lattice parameter will often give a satisfactory result.

When the changes are due to stress, as already noted, the problem is that the lattice parameter that is measured will be changed by the surface relaxation. (Lattice parameter variation due to change of composition may be unaffected by surface relaxation since there may be no stresses to relax). The technique is nonetheless valuable in this case provided that the data are interpreted taking this into account.

Like all techniques, HOLTZ line analysis has its limitations and problems. Despite them it remains a uniquely powerful method for the measurement of lattice parameter on the nanometer scale.

ADDITIONAL BIBLIOGRAPHY

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The basic papers on HOLTZ-line analysis are Jones, Rackham and Steeds[11] and Buxton.[12]

A good and comprehensive bibliography of convergent-beam diffraction was published recently.[13] It lists many papers on HOLTZ line methods and applications to problems in materials science.

ACKNOWLEDGMENTS

My recent interest in HOLTZ lines has been stimulated by discussions with David Bird and Dick James to whom my thanks are due. This work was funded by the Materials Research Laboratory grant through the U.S. Department of Energy, Division of Materials Research DEFG-02-91ER45439.

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1. The author is aware of perhaps a dozen programs that have been written to simulate HOLTZ line patterns in the kinematical approximation. They are rather easy to write; many people write their own. Two widely available packages of programs for electron microscopy include HOLTZ line routines. They are "EMS" and "Diffract". Details of EMS are given in:

Stadelmann, P.A. (1987) *EMS - a software package for electron diffraction analysis and HREM image simulation in materials science. Ultramicroscopy* **21**: 131-146.

For further information contact:
EMS: Stadelmann, P.A., I2M-EPFL, Swiss Federal Institute of Technology, CH-1015 Lausanne, Switzerland
Diffract: Schlienger, M.E., Virtual Labs, 37 Highland Court, Ukiah, CA 95482, USA.

For a note concerning errors in such programs see:

Eades, J.A., Moore, S., Pfullmann, T. and Hangan, J. (1992) *Errors in HOLTZ-line simulations*, EMSA 1184-1185 (a more complete version of this paper has been submitted to Microscopy Research and Technique).

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