7. DISCUSSION OF RESULTS

The newly developed stress–strain analysis[1,2] is based on the ability of the divergent beam method to record sensitively the strains in a crystal in terms of the changes of d spacings of the various (hkl) reflections. The question that arises is: What is the precision of strain measurement that can be obtained by the divergent beam method?

It is quite evident that the strains $(\Delta d/d)$ smaller than the experimental error in d spacing, given by $\bar{\sigma}_d$, cannot be measured. Focusing our attention on Table I and Fig. 6, we note that $\bar{\sigma}_d$ is a function of the Bragg angle θ, declining with increasing θ. It can be seen that for planes belonging to the {211} form in tungsten crystals, the minimum value of $\bar{\sigma}_d$ is 0.00039 Å and, therefore, strains smaller than 0.030% (=0.00039/1.29230) cannot be measured. For the {321} form the precision improves to 0.009% strain. It follows from the dependence of $\bar{\sigma}_d$ on θ that if small strain variations are to be measured for a given crystal orientation, the radiation used should be such as to yield the maximum number of ellipses in the vicinity of the center of the film.

For the successful application of the stress–strain analysis it appears useful to construct at the outset a curve such as that shown in Fig. 6, which defines the limits of the largest experimental error, namely, that of $\bar{\sigma}_d$. It is significant that in this error analysis the σ_d* values which represent the standard deviations of the d spacings of the $\{hkl\}$ forms turned out to be nearly twice as large as the corresponding experimental errors $\bar{\sigma}_d$. This difference between σ_d* and $\bar{\sigma}_d$ is interpreted to be due to small residual strains which the divergent beam method seems to be capable of detecting even in as-grown, zone-refined crystals.

The decline of σ_d* and $\bar{\sigma}_d$ with increasing θ is due to a number of factors that are common to all diffraction methods in which the highest precision of measurements is obtained from lines with the largest diffraction angles. They need not be discussed here. One factor, however, is peculiar to the divergent beam method and arises from the method of measurement of the diffraction profiles. As the α value of the reflecting (hkl) planes increases (decreasing θ), the recorded ellipses are further removed from the center of the film. The decrease in the angle between the diffracted rays and the film causes an increase in the width of the recorded line. Since this line broadening results in a flattening of the diffraction maximum, an error in measurement is introduced, due to the increased uncertainty of the exact peak-to-peak distance of the line profiles.

It is interesting to compare the results of our precision lattice parameter measurement with the results obtained from an international project conducted by the International Union of Crystallographers Commission on Crystallographic Apparatus.[10] The result of the present investigation for tungsten is $a_0=3.16566 \pm 0.00002$ Å (the standard error of the mean based on 85 observations), whereas the average value obtained from the International Union of Crystallographers project, in which 16 laboratories participated, was $a_0=3.16522 \pm 0.00009$ Å.

The difference of 0.0004 Å between the values of the lattice parameter can be satisfactorily explained if one considers the impurity content of the samples studied. The material used in this investigation was a zone-refined crystal containing virtually no substitutional impurity atoms and having 2 ppm oxygen, 9 ppm carbon, 4 ppm nitrogen. On the other hand, the sample studied by the International Union of Crystallographers Commission was a polycrystalline specimen of 99.27% to 99.92% purity. Optical spectrographic examination showed small amounts of Ca, Mg, Si, B, and Cr. A wet chemical analysis showed 0.19% Fe_2O_3 and 0.06% SiO_2.[10] Substitution of W atoms by the impurities in concentrations such as these would give rise to a decrease of the lattice parameter and bring the value of a_0 published by the International Union of Crystallographers very close to that found in the present investigation.

8. APPLICATIONS AND LIMITATIONS OF THE METHOD

The stress–strain analysis based on the divergent beam method has already been applied to a number of problems in physical metallurgy and solid state physics. In all these problems the aim was to obtain quantitative information about the structural changes induced by lattice defects insofar as they manifest themselves by the concomitant changes in the strain distribution. Thus the method has been applied to the analysis of the strain distribution in the age-hardening of an Al–3.85% Cu crystal.[1] It was also applied to the study of ordering in the Cu–50% Au alloy,[2] where tetragonality strains induced by the ordering process have been analyzed by this method.

The divergent beam method becomes a particularly powerful research tool when correlated with the study of lattice defects by transmission electron microscopy.[2,3,6] As practiced in this laboratory, the single-crystal specimen is first studied by the x-ray method to obtain the stress–strain configuration. Subsequently the specimen is thinned down and viewed by transmission electron microscopy.

This combination of methods was also employed in the study of neutron-irradiated quartz when the change in strain distribution was analyzed as a function of radiation dosage[3,6] and in the study of the yield phenomena of refractory metal crystals where the slip activity in the pre-yield, microstrain region was explored.[4] It is currently applied in such diversified investigations as the study of the strain distribution

[10] W. Parrish, Acta Cryst. 13, 838 (1960).