JOURNAL OF APPLIED PHYSICS VOLUME 35, NUMBER 11 NOVEMBER 1964

Strain and Precision Lattice Parameter Measurements by the X-Ray Divergent Beam Method. I*

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(Received 20 April 1964)

An X-ray method is described which permits a precise determination of the interplanar spacings in a crystal. The method is capable of disclosing small differences in interplanar spacings even between various sets of (*hkl*) planes of the same form and, therefore, serves as a basis for a stress–strain analysis. An error analysis of this method shows that the minimum measurable strains in the lattice, expressed as $\Delta d/d$, are a function of the diffraction angle θ. For θ varying from 35° to 65°, the minimum measurable strains vary from 0.03% to 0.009%.

A precision determination of the lattice parameter of a zone-refined tungsten crystal yielded $a_0=3.16566\pm0.00002$ Å. Limitations and possible applications of the method to problems in physical metallurgy and solid state physics are presented.

1. INTRODUCTION

A STRAIN analysis was recently developed by which the principal strains in a crystal can be determined provided the changes of interatomic spacings of more than six independent (*hkl*) reflections are recorded.[1] Such data are conveniently provided by the back-reflection divergent x-ray beam method. By means of this method pseudo-Kossel patterns are obtained which consist of ellipses corresponding to the reflections from a number of individual (*hkl*) planes. Changes in the interatomic spacings Δd_{hkl} induced by mechanical or chemical processes are manifested sensitively by changes in the parameters of the ellipses. By measuring the Δd values of several (*hkl*) reflections and referring them to the corresponding d spacings of the initial state, strains $\Delta d_{hkl}/d_{hkl}$ are obtained which are used as the raw data for the strain analysis.

For cubic crystals the strain analysis has been recently further developed so as to yield a complete stress–strain analysis.[2] In addition to the stress–strain configuration of the crystal, the stored elastic energy can also be measured and the magnitude and direction of the maximum shearing strain in any desired plane can be determined. Since this stress–strain analysis represents a powerful research tool for various problems in physical metallurgy and solid-state physics[1–6] and since this analysis is based on the strain data supplied by the divergent beam method, it is only fitting to inquire what precision can be obtained by this technique. This paper deals, therefore, with the new experimental developments—the precision and the advantages and limitations of the x-ray divergent beam method.

It should be clearly understood that the precision requirements for the stress–strain analysis based on the divergent beam method are much more stringent than those usually associated with conventional strain analyses based on other x-ray methods, viz., powder method. In the latter method the strains are usually obtained from extrapolated lattice parameter measurements which, of course, can also be obtained by the divergent beam method. However, if one uses extrapolation techniques in the divergent beam method for the sole purpose of obtaining a precision value for the lattice parameter, one surrenders a unique advantage which the method offers; it is an important feature of this method that each set of planes of a form in a cubic crystal gives rise to a separate ellipse. If small strains are introduced into a crystal, the size of the ellipses will change. One of them, for example, pertaining to the (*hkl*) reflection may expand, while another one, pertaining to the ($\bar{h}kl$) reflection, may contract. These changes in the dimensions of the ellipses are directly related to the changes in d spacings and the modifications of the various ellipses of {*hkl*} reflections are therefore directly related to the strain configuration of the crystal. In the powder method, on the other hand, the various (*hkl*) reflections of a form will be recorded on a single line and in the case cited above will give rise to line broadening and thus to the usual complications associated with this effect.

Since the experimental differentiation between various (*hkl*) planes of a form is fundamental for an effective stress–strain analysis, this paper will be principally concerned with the precision that can be obtained from measurements of the interatomic spacings of a number of {*hkl*} forms. In addition to this investigation of strain measurements, a complete analysis of precision lattice parameter measurements will also be offered.

* This work was sponsored by the Aeronautical Systems Division, U. S. Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, under contract AF33(657)-10872.

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