TABLE I. Summary of error analysis of x-ray divergent beam method using an as-grown, zone-refined tungsten single crystal.

{hkl}	{321}	{222}	{310}	{220}	{211}
No. of observations	24	15	28	11	7
$\bar{d}$	0.84601	0.91381	1.00085	1.11912	1.29230
$\bar{\sigma}_d$	0.00008	0.00026	0.00029	0.00036	0.00039
$\bar{\sigma}_d/\bar{d}$, %	0.009	0.028	0.029	0.032	0.030
σ_d^*	0.00016	0.00028	0.00061	0.00076	0.00083
$1/\sigma_d^{*2}$	4.0829×10^7	1.2958×10^7	0.2675×10^7	0.1740×10^7	0.1254×10^7
a'	3.16547	3.16561	3.16496	3.16534	3.16549
$\bar{\theta}$	65.57	57.45	50.32	43.49	36.59

It should be understood that the σ_d values from which $\bar{\sigma}_d$ was obtained are a measure of the precision of the technique, that is, they express the experimental error of each measurement of d spacing. The $\bar{\sigma}_d$ should be compared to the σ_d^* values listed in row 6 of Table I which are the standard deviations of d spacings of the $\{hkl\}$ forms. σ_d^* measures, therefore, more than the experimental error and its physical significance seems to be intimately related to the residual strain distribution in the as-grown crystal. It will be noted from Fig. 6 that both $\bar{\sigma}_d$ and σ_d^* decrease with increasing Bragg angle θ and that for all $\{hkl\}$ forms, excepting {222}, the σ_d^* are nearly twice as large as the corresponding $\bar{\sigma}_d$ values.

The precision determination of the lattice parameter was carried out by adopting a sequence of steps which will be outlined presently.

(1) It may be seen from Table I that a considerable number of determinations of d spacings has been carried out for each $\{hkl\}$ form investigated. From each d value of an $\{hkl\}$ form, the lattice parameter a' was computed using the relation $a'=d\cdot(h^2+k^2+l^2)^{\frac{1}{2}}$.

(2) To each a' of an $\{hkl\}$ form thus obtained a value of the Nelson–Riley function, $\frac{1}{2}[(\cos^2\theta/\sin\theta)+\cos^2\theta/\theta]$, was assigned. The θ value used for this computation corresponded to the d spacing from which each a' was originally derived.

(3) Since the error in the computation of d and therefore the error in a' diminishes with increasing θ (Fig. 6), a statistical weight was assigned to each a' which is proportional to $1/(\sigma_d^*)^2$ (see Appendix A).

(4) Employing a method of least squares, the lattice parameter a_0 was obtained by extrapolation of the Nelson–Riley plot of the weighted a' values. The slope of this least-squares line is then given by

$$b=\sum n_i w_i a_i'(x_i-\bar{x})/\sum n_i w_i(x_i-\bar{x})^2$$

and the y intercept by

$$a_0=(\sum w_i a_i'/\sum w_i)-b\sum x_i w_i/\sum w_i,$$

where $x_i=\frac{1}{2}[(\cos^2\theta_i/\sin\theta_i)+\cos^2\theta_i/\theta_i]$ (the value of the Nelson–Riley function), $w_i=[1/(\sigma_{d_i}^*)^2]/[\sum 1/(\sigma_{d_i}^*)^2]$ (the statistical weights), $\bar{x}=\sum n_i w_i x_i/\sum n_i w_i$.

The following results were obtained:

$$b=-3.88\times10^{-4}\pm0.56\times10^{-4}$$
$$a_0=3.16554\pm0.00002\text{ Å}.$$

The lattice parameter a_0 was corrected for refraction by adding to it a term $a_0(1-n)$, where n is the coefficient of refraction.[9] For tungsten and using CuK_{α_1} radiation ($\lambda=1.54051$ Å) the correction factor for refraction was 157×10^{-6} Å. Since the ambient temperature during the experiments was 28°C, a temperature correction was also applied[9] using the expression

$$a_2=a_1+\alpha a_1(T_2-T_1),$$

where a_1 and a_2 are the lattice parameters at temperatures $T_1=28$°C and $T_2=25$°C, respectively, and α, the coefficient of expansion of tungsten, is 4.6×10^{-6} °C^{-1}. With these corrections applied the lattice parameter a_0 is 3.16566 Å at 25°C.

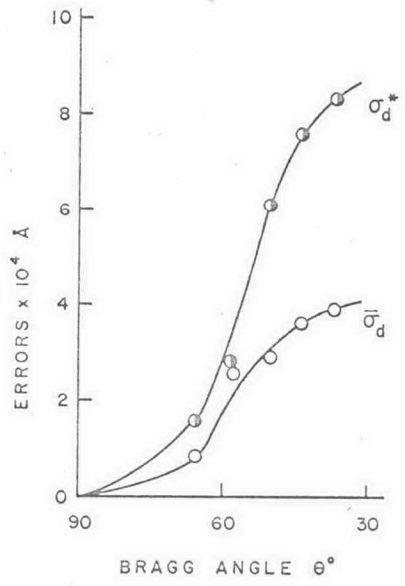

FIG. 6. Dependence of the d-spacing errors on the Bragg angle.

[9] *International Tables for X-Ray Crystallography* (Kynoch Press, England), Vol. 3.