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Faulting in Sodium Azide

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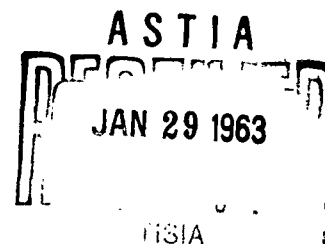
Measurement of Stacking Fault Probabilities in Bulk Specimens

by

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Faulting in Sodium Azide

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In a recent paper Keating and Krasner⁽¹⁾ reported measurements of X-ray powder pattern diffraction line shapes and positions from sodium azide (NaN_3) after various treatments. In particular, they noted that after deformation, the line broadening was consistent with that expected from the formation of deformation stacking faults, but that the anticipated shifts in the peak positions could not be detected. The purpose of this communication is to suggest a possible explanation.

Sodium azide is rhombohedral, but the structure may also be described as hexagonal with ABCABC.....stacking, as in FCC close-packed metals. The analysis of the diffraction effects due to stacking faults was given by Keating and Krasner, and is similar to that for FCC structures⁽²⁾. Since there is a unique slip plane in sodium azide, no correction is needed for a multiplicity of slip planes and the powder pattern analysis is thus simpler than for the FCC case.

If only intrinsic (or deformation) stacking faults are formed upon deformation or irradiation, then the application of the analysis by Keating and Krasner would be valid. However, if extrinsic (or double-deformation) faults are also produced, then the expected diffraction effects would have to be re-examined. Published work to-date indicates that in metals, intrinsic faults predominate. However, in other materials, the stacking fault energy of intrinsic and extrinsic faults may be of about equal magnitude (and possibly even less for the extrinsic faults). Such a situation appears to prevail in Si⁽³⁾, where intrinsic and extrinsic faults form in

about equal amounts when the material is deformed in a suitable manner. Since sodium azide has a unique slip plane, the mode of deformation would not be too important in producing both types of faults if their energies were approximately equal.

The diffraction effects from extrinsic faults in FCC structures have recently been calculated by Johnson⁽⁴⁾. He found that although the same reflections were shifted and broadened as for intrinsic faulting, the shift produced by a low density of extrinsic faults was in the opposite direction to that produced by intrinsic faults. If α' is the probability of an intrinsic fault and α'' is that of an extrinsic fault, then for small α the peak shift depends upon the difference $\alpha' - \alpha''$ and the broadening depends upon the sum $\alpha' + \alpha''$ ⁽⁵⁾. Consequently, for $\alpha' = \alpha''$, no net peak shift will be observed and the broadening will be double that from intrinsic faults only.

Thus on the basis of the supposition that (i) extrinsic stacking faults are at least as likely to form in sodium azide as intrinsic stacking faults and that (ii) the X-ray diffraction analysis for extrinsic stacking faults is applicable, one can explain qualitatively the results of Keating and Krasner.

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Measurement of Stacking Fault Probabilities in Bulk Specimens

H. M. Otte,* D. O. Welch* and G. F. Bolling**

Recently Welch and Otte (1962) pointed out that residual elastic strains can affect measurements of the stacking fault probability, α , in face-centered cubic metals. The purpose of this note is to consider one case in detail, where strains were not examined, and to draw attention to some precautions which must be taken in interpreting the measured shifts of X-ray reflections.

In the investigation of zone-refined lead by Bolling et al. (1961), bulk specimens were examined at 4.2°K and 77°K. The deformation at temperature was performed by scraping the surface of the specimens (gouging). Even though the nature and distribution of any residual lattice strains would be quite complex and certainly would depend on the exact manner of deformation, it is possible that a residual strain predominantly of one sign was left in the specimens. This strain would lead to a shift in the position of each X-ray reflection depending on the scattering angle and the elastic constants. Since lead is more anisotropic elastically than the other f.c.c. metals, it turns out that the effect of residual lattice strains is relatively most important for lead.

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When residual elastic strain and faulting are both considered, the following equation should be used to calculate α ,

$$\delta\Delta 2\theta_{p-q} = K_{p-q} \sigma + H_{p-q} \alpha. \quad (1)$$

Here, $\delta\Delta 2\theta_{p-q}$ represents the change in separation between the p^{th} and q^{th} X-ray reflections; H_{p-q} as defined by Wagner (1957), are coefficients relating the change in separation to the faulting probability; and K_{p-q} are coefficients relating the change to the residual stress, σ . The constants K_{p-q} are derived simply from the differential form of Bragg's law and the definition $\epsilon_p = K_p \sigma$; i.e.,

$$K_{p-q} = - \frac{180}{\pi} 2(K_p \tan \theta_p - K_q \tan \theta_q) \quad (2)$$

The moduli K_r can be calculated from the elastic constants under the two simplified extremes of constant strain or constant stress existing throughout the specimen. Greenough (1952) gives the equations for the strain $\epsilon_{hk\ell}$ in the $hk\ell$ direction and it follows that

$$\left. \begin{aligned} K_r [\epsilon = \text{const.}] &= \frac{-(C_{11} + 4C_{12} - 2C_{44})}{2(C_{11} + 2C_{12})(C_{11} - C_{12} + 3C_{44})} \\ \text{and } K_r [\sigma = \text{const.}] &= S_{12} + \frac{(S_{11} - S_{12} - \frac{1}{2} S_{44})(h^2 k^2 + k^2 \ell^2 + \ell^2 h^2)}{(h^2 + k^2 + \ell^2)^2} \end{aligned} \right\} \quad (3)$$

where C_{ij} and S_{ij} are the elastic constants and $r = h^2 + k^2 + \ell^2$. In most cases, however, the best agreement with observation is not obtained under these extreme assumptions; better agreement is obtained if the

actual residual strain distribution is considered to be somewhere between the limits of isotropic stress and isotropic strain, (Greenough, 1952; Welch and Otte, 1962). The K_r for this assumed situation is chosen here to be the average of the values of K_r in equation (3).

The stress and the faulting probability can both be derived from equations (1) if the change in separation between two pairs of X-ray reflections are measured. For the zone-refined lead such changes were measured at 4.2°K; the reflections involved were the 111, 200, and 220, lines 3, 4 and 8 respectively (Bolling et alia, 1961). The value of the coefficients to be used for lead examined with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418\text{\AA}$) are given in Table I.

Calculated values for the stacking fault probability and the residual stress are given in Table II. The usual assumption of zero residual strain ($K=0$), gives values of α very close to those derived at the assumed extreme of isotropic strain. However, the stress values for both seem unreasonable; $\sigma=0$, or -6 and -8 Kg/mm^2 . At the other assumed extreme, of isotropic stress, the value of α has been appreciably diminished and it is also somewhat diminished under the assumed, mean case. The four values of stress from -2 to -4 Kg/mm^2 fall below the stress at the beginning of necking observed in single crystals of lead deformed in tension at 4.2°K, (Bolling et al., 1962); and because it is not unusual to obtain high values of stress in compression these values must be considered reasonable. Since the deformation was by gouging, any residual strain in the direction of the surface normal would most likely have been predominantly posi-

tive, reflecting a transverse compressive stress in the deformed surface. The calculated stresses are therefore of the expected sign.

In the investigation at 4.2°K, specimens of aluminum and a Cu-30Zn alloy were also deformed by gouging. Unfortunately, the results from neither material can be used to clarify the effects of residual strain. Aluminum is quite isotropic elastically and there would have been little effect. The brass on the other hand is anisotropic elastically, but it faults profusely and the changes in line separation due to the deformation faults would most likely have masked any effects of residual strain due to the anisotropy.

Other experiments (Welch and Otte, 1962) have shown that residual elastic strains can make a significant contribution to the peak shifts in an anisotropic f.c.c. alloy. It can also be concluded here that the contribution is measurable in lead if the averaging assumption holds true, and quite significant, if the extreme of isotropic stress holds true. In the work performed by Bolling et al. (1961) the $\delta A_{2\theta_{4-3}}$ was primarily relied upon; it is however advisable in this sort of experiment to examine as many X-ray peaks as possible in order to take account of the contribution due to residual strains. Finally the precaution should be added that, even though the use of filings provides an averaging that should eliminate the extreme of isotropic stress, it is not clear that the effects of residual elastic strain have been experimentally shown to be negligible.

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TABLE I

Coefficients and Data Used in Equations (1) for Lead at 4.2°K *

<u>Quantity</u>	<u>4-3</u>	<u>8-4</u>	<u>Units</u>
First cold work $\delta\Delta 2\theta$	-0.03 ₀ (± 0.01)	0.02 ₃ (± 0.02)	°(2 θ)
Second cold work $\delta\Delta 2\theta$	-0.04 ₈ (± 0.01)	0.03 ₈ (± 0.02)	°(2 θ)
H _{p-q}	-3.7	4.5 **	°(2 θ)
K _{p-q} Constant strain	0.00053	0.00181	°(2 θ)mm ² /Kg
K _{p-q} Constant stress	0.00930	-0.00445	"
K _{p-q} Mean Case Average of above	0.00491	-0.00132	"

* The data is taken from Bolling et al (1961). A lattice constant $a_0 = 4.916$ Å and the elastic constants for lead at 4.2°K, given by Waldorf (1960), were used.

** The value of H₈₋₄ was incorrectly given by Bolling et al (1961) as 7.8, but was not significantly used.

TABLE II

Calculated Values of Stacking Fault Probability and Residual Stress- First Cold Work -

Quantity	Assumptions				
	$\epsilon=0$	$\epsilon=\text{constant}$	$\sigma=\text{constant}$	mean Case	
α	0.008 0.005 } 0.006 _s	0.007	0.003	0.004	
$\sigma, \text{Kg/mm}^2$	0	-6	-2	-3	

- Second Cold Work -

α	0.013 0.008 } 0.010 _s	0.012	0.005	0.007	
$\sigma, \text{Kg/mm}^2$	0	-8	-3	-4	