The ratio of hexagonal to cubic packing in complex polytypes or mixtures may be measured approximately by intergrating all of the specular and diffuse diffraction intensity from, and including, the first line, 10.0, up to, but not including the second, $00.1+10.\frac{1}{3}$ (unit layer indices), and taking the ratio of this to the integrated intensity of the second line (Smith, 1955). The change of this ratio with change in the ratio of packing is assumed to be the same as for mechanical mixtures of hexagonal ($2H$) and cubic ($3C$) material. Using this method, the weight percentage of cubic packing in the two starting materials and the two impacted materials were calculated. The results are:

	Weight % cubic packing (approximate)
Hexagonal phase	0
Same after hammering	61
Cubic phase	93
Same after hammering	76

Paterson (1952) calculated that deformation faulting of a cubic close-packed structure should cause broadening of the diffraction spectra, and certain shifts of peak angles. The simplest shifts are of the 200 reflection to smaller Bragg angle, and of 400 to larger angle. A measurement of the cubic starting material and the same after hammering showed that the 200 reflection was broadened $0{\cdot}44°$ (2θ) and shifted $0{\cdot}1°$ (2θ) to lower angle. Similarly, the 400 reflection was broadened $1{\cdot}0°$ (2θ) and shifted $0{\cdot}1°$ (2θ) to higher angle. Therefore the sense of the shifts of diffraction angle are in accord with deformation faulting deduced from the qualitative change in the diffraction pattern itself. However, the large amount of broadening suggests that in part this may be due to small size of the crystals in the hammered material.

Assuming tentatively that all of the broadening of the second diffraction spectrum ($00.1+10.\frac{1}{3}$) is due to the size effect, and using an approximate method of calculation given by Klug and Alexander (1954), the mean size of particles is 240 Å in the material originally cubic, and 330 Å in the material originally hexagonal.

The grain size is too small for optical resolution, but examination of electron photomicrographs of the two hammered specimens indicated that the mean sizes are at least five times the above calculated values. Evidently much of the broadening of the diffraction spectra is due to the size effect, but some is due to deformation faulting (glide twinning).

We conclude that impact grinding of zinc sulphide randomizes the structure by deformation faulting and, independent of the starting material (cubic or hexagonal), a similar product is obtained.

References

Bridgman, P. W. (1939). *Amer. J. Sci.* **237**, 7.

Frey, F. (1948). *Ann. Phys., Lpz.* **2**, 147.

Fuller, M. L. (1929). *Phil. Mag.* (7), **8**, 658.

Klug, H. P. & Alexander, L. E. (1954). *X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials*, chap. 9. London: Chapman and Hall.

Kullerud, G. (1953). *Norsk geol. Tidsskr.* **32**, 61.

Paterson, M. S. (1952). *J. Appl. Phys.* **23**, 805.

Schleede, A. & Gantzchow, H. (1923). *Z. phys. Chem.* **106**, 37.

Short, M. A. & Steward, E. G. (1955). *Acta Cryst.* **8**, 733.

Smith, F. G. (1955). *Amer. Min.* **40**, 658.

Strock, L. W. (1955). *Sylvania Techn.* **8**, 71.

Acta Cryst. (1956). **9**, 822

Crystal data for two new coordination compounds.

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Murmann (1956) has recently prepared two new coordination compounds: bis-(3-amino-3-methyl-2-butanone oxime)nickel(II) perchlorate and the corresponding palladium compound. The similarity of oscillation, Weissenberg, and precession photographs for crystals of the two compounds indicates they are isomorphous. The space group is $P2_1/c$. For the nickel compound

$$a = 13{\cdot}0\pm0{\cdot}1,\ b = 12{\cdot}75\pm0{\cdot}1,\ c = 20{\cdot}9\pm0{\cdot}1\ \text{Å},$$
$$\beta = 105°\ 59'\pm30'\ .$$

For the palladium compound

$$a = 13{\cdot}0\pm0{\cdot}1,\ b = 12{\cdot}85\pm0{\cdot}1,\ c = 21{\cdot}2\pm0{\cdot}1\ \text{Å},$$
$$\beta = 105°\ 52'\pm30'\ .$$

Each unit cell contains eight formula weights (calculated from density measurements: 7·95).

Crystals grown from water solution are elongated in the a direction. All of the crystals examined were twinned. The twinning does not become obvious in the form of 'spot splitting' on Weissenberg photographs about a until the second level. The first-level photographs appear normal since $10l$ spots from one individual lie very nearly half way between $10l$ spots from the other, and remaining $1kl$ spots are nearly coincident in pairs; the zero-level appears altogether normal. One twinned individual can be isolated fairly well by cutting with a razor blade.

Although several interesting questions could be answered by complete structure determinations of these compounds, the apparent necessity for three-dimensional methods from the outset has decided us against making the attempt.

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Reference

Murmann, R. K. (1956). *J. Amer. Chem. Soc.* To be published.