

Density and Hardness Changes Produced by Plastic Deformation in KCl Crystals*

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A study has been made of the density changes which accompany plastic deformation in compression of potassium chloride crystals and of their annealing. Also hardness changes, their annealing, and slip-line formation have been investigated. Up to about 11% deformation, the slip is predominantly simple with some admixture of double slip, and no change of density is observed. At 11% deformation, multiple slip presumably sets in accompanied by a rapid decrease of density. The rate of formation of vacancies is $3.02 \times 10^{17} \text{ cm}^{-3}$ per percent deformation. A comparison of this value with the results of the experiments of Ueta and Känzig leads to the ratio of about four vacancies per electron trap. Vacancies seem to have no measurable influence on hardness which thus appears to be primarily associated with changes in the dislocation density or their configuration during plastic deformation.

INTRODUCTION

THE question of the detailed mechanism of plastic deformation of single crystals has a long history, and a number of models and approximations, often of only qualitative significance, has been proposed. In particular, while the simple slip is reasonably well understood in terms of the dislocation model, such phenomena as multiple slip and work hardening still pose many problems. According to the dislocation model in simple slip, there should be little work hardening, mostly caused by exhaustion of Frank-Read sources, and the stress-strain curve should be linear.¹⁻³ In multiple slip, characterized by slip on intersecting families of planes, work hardening should be strong⁴ since it is caused by increasing intersections of dislocations, formation of sessile defects such as Cottrell dislocations (these probably do not exist in alkali halides), and other defects. Here the stress-strain curve is roughly parabolic as predicted by theory.⁵⁻⁷

In alkali halides, slip occurs on $\{110\}$ planes in $\langle 1\bar{1}0 \rangle$ directions, and thus, according to the maximum-resolved-shear law, there should be always at least two sets of planes on which the resolved shear stress is the same. In spite of this, Pratt⁸ reported single slip in sodium chloride crystals in compression. He found also that in some cases, even when the slip in these crystals did occur on two intersecting families of planes, one of these then became temporarily inactive. During this interval, the stress-strain curve was linear. This can be

otherwise described as nonequal work-hardening of the two slip systems. Since intersection of dislocations acting on different slip planes may lead to the formation of a large number of vacancies and interstitials, one would expect that conductivity, ease of coloration, and density of alkali halides would behave differently in simple and in multiple slip. Here the energy of formation of vacancies and interstitials not only makes the intersection of dislocations difficult, but also produces obstacles which may impede the motion of other dislocations. The well-known experiments of Gyulai and collaborators^{9,10} on conductivity of sodium chloride already point in this direction, although some objections to such an interpretation have been raised by Tyler.¹¹ In metals, similar phenomena have been observed by many, for instance by Masima and Sachs on alpha brass.¹²

An important recent work on the production of vacancies by plastic deformation is that of Ueta and Känzig¹³ who measured the number of electron traps generated by various degrees of plastic deformation, with the following results: Crystals which have been colored additively contain essentially only F -centers and if such crystals are irradiated with F -light, electrons are released and these can then be captured by other kinds of traps. If the trap is a negative-ion vacancy, another F -center will be formed, but if the trap is anything else, for example an M - or R -center, then the number of F -centers will decrease. Therefore, the initial bleaching rate of the F -band for a given intensity of bleaching light, a given concentration of F -centers, and a given thickness of crystal will be proportional to the number of unfilled traps other than negative-ion vacancies. By determining the initial bleaching rate and the final absorption coefficient for an additively colored crystal of a given thickness and degree of coloration, they determined the proportion-

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¹ Maddin, Mathewson, and Hibbard, *Trans. Am. Inst. Mining Met. Engrs.* **175**, 86 (1948).

² E. N. Andrade and C. Henderson, *Trans. Roy. Soc. (London)* **A244**, 177 (1951).

³ F. Rohm and A. Kochendörfer, *Z. Metallk.* **41**, 265 (1950).

⁴ R. W. K. Honeycombe, *J. Inst. Metals* **80**, 45 (1951).

⁵ N. F. Mott, *Phil. Mag.* **43**, 1151 (1952).

⁶ J. S. Koehler, *Phys. Rev.* **86**, 52 (1952).

⁷ H. G. Van Bueren, *Acta Met.* **1**, 464 (1953).

⁸ P. L. Pratt, *Acta Met.* **1**, 103 (1953).

⁹ Z. Gyulai and D. Hartly, *Z. Physik* **51**, 378 (1928).

¹⁰ Z. Gyulai and J. Boros, *Math. u. naturw. Anz. ungar. Akad. Wiss.* **59**, 115 (1940).

¹¹ W. W. Tyler, *Phys. Rev.* **86**, 801 (1952).

¹² M. Masima and G. Sachs, *Z. Physik* **51**, 321 (1928).

¹³ M. Ueta and W. Känzig, *Phys. Rev.* **97**, 1591 (1955).

ality constant between the initial bleaching rate and the absolute number of traps, which, in turn, was determined by subtracting the number of F -centers remaining after bleaching from the number present before bleaching. Additively colored crystals were thus deformed to various degrees and bleached by F -light, the initial bleaching rates giving the number of electron traps. A plot of their results is given in Fig. 1; we shall compare it with ours later.

It should be also remarked that the hardening influence of vacancies in alkali halides finds an apparently indirect confirmation in the experiments of Podashevsky,¹⁴ Westervelt,¹⁵ and others as discussed by Seitz.¹⁶ They found that hardness, yield stress, and fracture strength of alkali halides increased after x-ray or ultraviolet irradiation. Such irradiation is known to produce a high concentration of vacancies and an accompanying change of density.¹⁷⁻²¹ Seitz suggests that the hardness change is caused by clusters of vacancies of both signs which condense near dislocations during irradiation and during the bleaching that may follow. If this is so, as Pratt²² has pointed out, a sufficient excess of vacancies, not absorbed as F -centers and V -centers, must be formed during the irradiation to saturate the dislocation atmospheres, since no further increase in the hardening is observed during the generation of vacancies by bleaching. The lack of any yield-point phenomena argues against vacancy locking of dislocations as a strong contribution to the hardening. A calculation²² shows that few excess vacancies are created by x-irradiation and, since it was shown earlier

that F -centers have no effect on hardness, the indications are that vacancies themselves may have little effect on hardness. On the other hand, vacancies are believed¹⁶ to be formed through the climb of edge dislocations during x-ray irradiation, and this can be a powerful source of hardening since it would be a disturbance of the equilibrium dislocation array. Hardening of this nature would not be effected by bleaching, as the vacancies are known to diffuse away from the dislocations to form F -centers²³ and to cluster on bleaching so that the dislocations cannot climb back to their original positions.

In view of these various results and interpretations, it seemed of interest to make a study of density changes, hardening, and slip-line formation which accompany plastic deformation of a well-known alkali halide such as potassium chloride.

EXPERIMENTAL PROCEDURE

The change in density of potassium chloride was determined by obtaining the difference in suspension temperatures between a deformed and an undeformed crystal. Hutchison, Johnston,²⁴ and others developed this method which was adapted by Estermann, Leivo, and Stern¹⁷ to measurements on KCl. 1-3-dibromopropane was used as the suspension liquid since near room temperature it has the same density as potassium chloride, and it satisfies the further requirement of not reacting chemically with the crystals. Although, in principle, this method is quite simple, it has several important experimental aspects which require special attention. These are described in some detail below.

(a) The Crystals

Previous experience indicated that crystals which were in contact with air during or after the cutting process did not yield reproducible density values. It appears that any moisture on the surfaces of the crystals or in the liquid can cause its hydrolysis. This produces HBr which acts as a catalyst in promoting a halogen ion exchange between KCl and the 1-3-dibromopropane. To avoid these difficulties, the cleaving was done in a glove box filled with dry helium at a slight positive pressure.

Single crystals, measuring approximately 1 in. \times 1 in. \times 1 in., obtained from the Harshaw Chemical Company, were cleaved into four 1 in. \times 0.5 in. \times 0.5 in. crystals. The crystals were then placed in a press, in a holder, in such a way that they would be compressed in the long direction. To prevent barreling, very smooth $\frac{1}{2}$ -in. thick plate-glass surfaces lubricated with water were used to press upon the specimen. The readings of two Federal

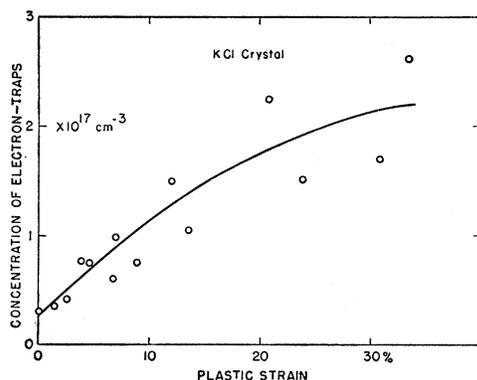


FIG. 1. Concentration of electron traps in KCl as a function of deformation (after Ueta and Känzig¹⁸).

¹⁴ M. N. Podashevsky, *Physik. Z. Sowjetunion* **8**, 81 (1935).

¹⁵ D. R. Westervelt, *Acta Met.* **1**, 755 (1953).

¹⁶ F. Seitz, *Revs. Modern Phys.* **26**, 7 (1954).

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¹⁸ H. Witt, *Nachr. Akad. Wiss. Göttingen, Math.-physik Kl.* No. 4, p. 17 (1952).

¹⁹ K. Sakaguchi and T. Suita, *Technol. Repts. Osaka Univ.* **2**, 177 (1952).

²⁰ C. R. Berry, *Phys. Rev.* **98**, 934 (1955).

²¹ W. J. Leivo and R. Smoluchowski, *Phys. Rev.* **93**, 1415 (1954).

²² P. L. Pratt, *Report of the Conference on Defects in Crystalline Solids, Bristol, 1954* (The Physics Society, London, 1955), p. 402.

²³ D. L. Dexter, unpublished work—mentioned by Seitz (reference 16, p. 82).

²⁴ C. A. Hutchison and H. J. Johnston, *J. Am. Chem. Soc.* **62**, 3165 (1940). D. A. Hutchison and H. L. Johnston, *Phys. Rev.* **62**, 32 (1942). D. A. Hutchison, *Phys. Rev.* **66**, 144 (1944); *J. Chem. Phys.* **13**, 383 (1945).

0.001-in. dial indicators were averaged to eliminate effects due to cocking of the holder.

(b) Preparation of the Suspension Liquid

The treatment of the 1-3-dibromopropane consisted of mixing it very thoroughly with sulfuric acid in a separatory funnel. Repeating this process until the sulfuric acid separates as a clear phase insures the removal of any reactive materials from the liquid. Traces of the acid are eliminated by washing with a saturated solution of sodium bicarbonate and the liquid is then given a prolonged washing in distilled water. The subsequent drying process involved intimate mixing with activated alumina in a slowly revolving container followed by vacuum distillation in which only the middle fraction was kept. A drop of mercury introduced into the liquid helped to stabilize it by precipitating any HBr or Br₂ which may have formed by the action of light or heat.

(c) The Suspension Cell

The cell was made with a long neck which would fit through a hole in the bottom of the helium-filled glove box. In this position, the cap could be removed and first the liquid and then the crystals could be put into the cell without exposure to air. It was further found very helpful to make flat walls on that part of the cell where the actual suspension took place. This made it possible to identify every crystal in the presence of a weak red light. A U-bend in the neck of the cell served as a dry resting place in which the crystals could be outgassed by wrapping the U-bend with heating tape and heating between 60°C and 80°C. About five minutes were allowed for the crystals to cool before tilting the cell and sliding them into the liquid. After the measurements of the suspension temperatures had been made on one set of crystals, they were poured together with the liquid into a small arm on the side of the cell where they sank to the bottom, thereby allowing the liquid to be poured back into the cell proper. This made the cell ready for further measurements on other crystals.

(d) Measurements of Suspension Temperatures

Suspension temperatures were measured with a Beckmann thermometer immersed in an insulated water-bath, controlled to 0.002°C, with windows for observing the crystals. Although the thermometer could be read to 0.001°C, the suspension temperatures were actually determined only to within 0.005°C to 0.01°C which corresponds to an error in relative density measurements of about $12 \times 10^{-6} \text{ g cm}^{-3}$ or 2.0×10^{17} vacancies per cm³. The greatest limitations on the accuracy were the effects due to viscosity, the presence of density gradients due to slight residual differences in composition, and slight thermal fluctuations. To minimize these effects, the temperature was raised in increments of only 0.005°C every hour and the actual suspension tem-

perature corresponded to a suspension for at least an hour. Although the thermal fluctuations could be cut down to less than 0.002°C for any short time interval, it appeared that a reproducibility of the suspension temperatures to 0.01°C from day to day was possible if a temperature fluctuation of 0.004°C was allowed with a frequency of around two times a minute.

Densities of crystals subjected to three or four different degrees of plastic deformation were measured in each experiment. The center of each deformed crystal was cleaved out of the sample and as many as twenty crack-free pieces were formed. Three or four of these pieces chosen to represent each specimen were placed in the suspension cell together with at least three, undeformed, freshly cleaved crystals. As a means of identification, all pieces which came from a given deformed crystal were of the same shape. This practice was also followed for the undeformed crystals. In addition to all of the above-mentioned requirements, the suspension temperature for a given deformed crystal was considered to be the temperature at which two or more nontouching samples remained suspended at a given point for at least an hour. This eliminated the effect of accidental cracks and defects. Further, the accuracy of measurement was increased by having a large number of crystals in the liquid at the same time since in this way several comparisons could be made simultaneously.

(e) Measurements of Hardness

In addition to density, hardness was also measured by using a Tukon Tester with a 136° diamond indenter. Crystals were mounted on 1-in. diameter aluminum base with a rubber cement. The size of the indentations (about 70 microns) was so small that they were unaffected by the variation in the curvatures of the deformed crystals. A five-gram weight was used because preliminary measurements showed that it gave the most reproducible values.

RESULTS AND DISCUSSION

(a) Change of Density on Plastic Deformation

Twenty-eight crystals were deformed to various degrees, and the density changes were measured on at least two samples from each crystal. Table I lists the results, including the approximate time between deformation and the density determination. There is indication that some of the density change annealed out at room temperature, especially at the higher degrees of deformation. Figure 2 is a plot of all the points in Table I which correspond to two or more crystals. Some of the scatter is reduced if only points corresponding to measurement of the density change, taken within one day of the plastic deformation, are taken into account.

Assuming that the density change is a linear function of the degree of deformation, an application of the least-

squares method indicates that the change in density starts at about 11% plastic deformation with a slope of 0.0122°C per percent deformation. This corresponds to a density change of $18.8 \times 10^{-6} \text{ g cm}^{-3}$ per percent deformation or to the production of 3.02×10^{17} vacancies per cm^3 per percent deformation on the assumption that the vacancies have the same size as the ions.

(b) Annealing of Density Change

In addition to density measurements of deformed crystals, annealing of the density changes was studied. This was done on crystals deformed 21.2% in which the density change resulting from plastic deformation was $1.85 \times 10^{-4} \text{ g cm}^{-3}$. The annealing, at temperatures under 400°C, was made in a Pyrex tube in a helium atmosphere. Higher-temperature anneals were carried out with the crystals in a clear quartz boat which was placed in a ceramic tube and constantly flushed with dry helium. The temperature was raised and lowered at a rate of 5°C/min. The crystals were given 10-minute anneals at 200°C, 240°C, 280°C, 310°C, and 350°C. All of the anneals gave no measurable change in density except the one at 350°C which gave a complete return to the original density within the accuracy of the

TABLE I. Change of suspension temperature (°C) as function of deformation. Asterisks indicate measurements made between one and two weeks after the compression; all others were made on the same day.

| Percent deformation | Change in suspension temperature | Number of crystals suspended at the same temperature |
|---------------------|----------------------------------|--|
| 5.4 | 0 | 5 |
| 6.6 | 0 | 3 |
| 7 | 0 | 4 |
| 10 | 0 | 3 |
| 13.1 | 0.045 | 4 |
| 13.4 | 0.02 | 2 |
| | 0.045 | 1 |
| | 0.06 | 1 |
| 13.4 | 0.035 | 1 |
| | 0.04 | 2 |
| | 0.06 | 1 |
| 14.2 | 0.025 | 3 |
| 14.5 | 0.01 | 2 |
| 14.8 | 0.05 | 2 |
| 15.8 | 0.05 | 3 |
| 16 | 0.03* | 1 |
| | 0.05 | 5 |
| 16.3 | 0.75 | 2 |
| 16.9 | 0.05 | 2 |
| | 0.08 | 1 |
| 17.1 | 0.05 | 1 |
| | 0.065 | 2 |
| 17.2 | 0.10 | 2 |
| | 0.115 | 2 |
| 17.8 | 0.105 | 2 |
| 17.9 | 0.065 | 2 |
| 18.3 | 0.10 | 5 |
| 18.7 | 0.09* | 2 |
| 18.7 | 0.09* | 2 |
| | 0.10* | 2 |
| 21.2 | 0.120 | 2 |
| 21.2 | 0.09* | 2 |
| | 0.11* | 2 |
| 24 | 0.12* | 4 |
| 27.3 | 0.10* | 4 |

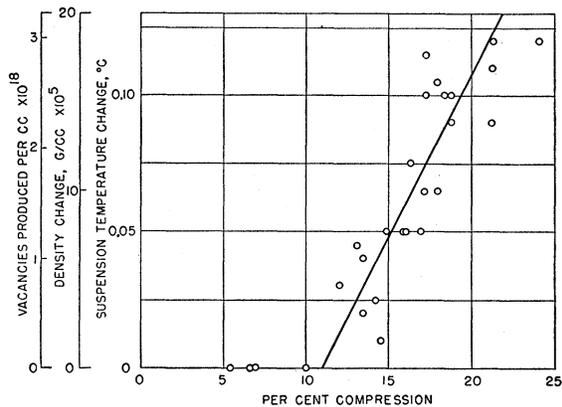


Fig. 2. Production of vacancies, changes in density, and changes in suspension temperature of KCl as functions of deformation.

measurement. The 350°C anneal was repeated with a crystal which had been deformed 17.2%, and the same results were obtained, i.e., the original density completely returned after the anneal.

(c) Mode of Deformation

The fact that there was always considerable hardening present, i.e., stress increased with strain, indicates that none of the crystals deformed exclusively by pure simple slip. On the other hand, density measurements, visual observations of slip lines and mode of cleavage described below indicate that in the first phase of deformation in any localized region only one slip system was operative. One may satisfy all these observations by concluding that at low deformations there was a mixture of predominantly simple slip and of some double slip similar to Pratt's results discussed earlier, but the amount of the latter was insufficient to show up as new slip lines or as changes of density. At higher deformations only double or, in general, multiple slip occurred.

(d) Slip Line Observations

Slip in NaCl-type crystals occurs on {110} planes in <110> directions. It follows that slip lines observed on a single-crystal parallelepiped, whose faces are the usual {100} cleavage faces, are of the "screw type," i.e., parallel to the Burgers vector, if they are at 45 degrees to the cube edge; and they are of the edge type, i.e., perpendicular to the Burgers vector, if they are parallel to the cube edge. Visual observations of the crystals used in the compression experiments described above indicate that up to about 15% deformation, slip lines corresponding to only one slip system were visible. Around this degree of compression a large number of new closely spaced edge-type slip lines appeared between the screw-type slip lines of the original slip system, i.e., on one pair of faces of the parallelepiped. Curiously enough, no corresponding effect was observed on the other faces of the crystal, i.e., no new screw slip

lines appeared among the old edge slip lines up to the highest deformations used (27%). It seems as if the slip planes active in the initial slip blocked the motion of screw components of the Frank-Read loops generated in the new slip system but did not interfere with the edge components of these loops.

If one cleaves a crystal deformed in compression, along a plane perpendicular to the direction of compression, then the cleavage plane is bent. On the other hand, cleaving along a cube plane containing the direction of compression and the Burgers vector of the active slip system yields a flat cleavage plane as if the crystal were not deformed at all. This result confirms the deduction made by Nye²⁵ that the curvature of a plane is given by nb , where n is the number of dislocations, of the same sign, parallel to the plane and all of the Burgers vectors b . It follows also that whenever double slip occurs in the alkali halides, the cleavage planes will never be flat. If, before cleaving, a deformed crystal is annealed, then the cleavage planes, instead of being bent, consist of flat segments inclined at small angles to each other. One might interpret this as an evidence of the formation of large subgrains due to polygonization.

Crystals which are short rather than long in the direction of compression deform with the formation of deformation bands parallel to the direction of compression. The deformation band boundary is a $\{110\}$ plane which contains the compression direction. The slip systems operative on the two sides of the boundary are different but not arbitrary: The deformation band boundary bisects the angle between the two slip planes and also the angle between the two slip directions. The deformation boundary is made up of dislocations lying in $\langle 111 \rangle$ -type directions (which are the intersection lines of the two slip planes) forming an angle $35^\circ 16'$ with the Burgers vectors, i.e., they are partly screw-type, partly edge-type.

(e) Hardness Measurements

Hardness tests were made on crystals that were (a) undeformed; (b) deformed 21.2% and unannealed; (c) deformed 21.2%, annealed at 350°C for 10 minutes with a rise of 5°C/min, and cooled at 5°C/min; (d) deformed 21.2%, heated to 750°C at 5°C/min, and immediately cooled at 20°C/min; (e) prepared as in group (c) and rotated by 45° to check for a possible error due to anisotropy. Since hardness number has no particular physical significance, Table II lists the lengths of the diamond indentations in microns. The higher the hardness, the shorter the indentation. Apparently there is no significant change in hardness after a 10-minute anneal at 350°C and only a slight softening upon heating to 750°C at 5°C/min and immediately cooling at 20°C/min. The softened crystal showed a "polygonized" cleavage plane as discussed

above. All these observations were made on a crystal deformed 21.2%.

The main results of the present investigation are as follows:

1. Judging by the amount of strain-hardening, none of the crystals deformed exclusively by a pure simple slip.

2. Up to a deformation of about 11% compression, there was presumably a mixture of predominantly simple slip with some double slip, with no accompanying measurable density change or new slip lines. This region is characterized by a low rate of production of vacancies and a probably large increase of the density of dislocations.

3. At about 11% deformation, multiple slip sets in accompanied by a rapid decrease of density of the crystals. The rate of this decrease is $18.6 \times 10^{-6} \text{ g cm}^{-3}$, or 3.02×10^{17} vacancies cm^{-3} per percent deformation. This assumes that all interstitials which may be formed in this process, and which one expects to be highly mobile at these temperatures, are already absorbed by dislocations and subgrain boundaries. The above number of vacancies is in agreement with the value 2×10^{17} to 2×10^{18} vacancies cm^{-3} at 12% deformation predicted by Seitz on the basis of the experiments of Gyulai and collaborators.

4. Ueta and Känzig's results, which are illustrated in Fig. 1, lead to the value of 0.77×10^{17} electron traps produced per cm^3 per percent deformation, and at the critical deformation of 11% the total number of traps reaches the value of about 1.2×10^{17} per cm^3 . If one assumes that the ratio of the number of traps to the number of vacancies is of the order of unity, then this concentration of traps would not be easily observable as a change of density in the experiments here reported. This is in agreement with experiment.

5. As the region of multiple slip sets in, the vacancies are produced at a rate about four times higher than the electron traps. From this point on, with increasing deformation, the efficiency of trap formation seems to fall off. This apparent contradiction, with the sudden onset of a high rate of production of vacancies, is to be

TABLE II. Lengths (in microns) of diamond indentations in KCl crystals deformed 21.2% and variously annealed.

| Undeformed | Deformed unannealed | Deformed and annealed at 350°, 10 min | Deformed and heated to 750°C | Maximum effect of anisotropy on crystal deformed, annealed at 350°C |
|--------------|---------------------|---------------------------------------|------------------------------|---|
| 96 | 75 | 80 | 79 | 83 |
| 90 | 77 | 78 | 76 | 85 |
| 91 | 80 | 76 | 74 | 83 |
| 92 | 75 | 74 | 78 | 81 |
| 95 | 77 | 78 | 77 | 83 |
| 90 | 76 | 76 | 76 | 83 |
| 88 | 77 | 79 | 75 | 81 |
| 88 | 78 | 76 | 80 | 83 |
| 91 | 77 | 79 | 82 | 83 |
| 95 | 78 | 77 | 81 | 84 |
| Average 90.5 | 77.0 | 77.5 | 78.0 | 82.8 |

²⁵ J. F. Nye, Acta Met. 1, 153 (1953).

interpreted as an evidence of the progressive formation of larger clusters of vacancies which are poor electron traps. It should be kept in mind too that each electron trap which is not an F -center must have at least two vacancies (the F' -center is not stable at room temperature). Ueta and Känzig also observed the formation of the M and R bands during plastic deformation and the growth of the R band up to 48 hours after plastic deformation. Thus if one considers that there are some free positive-ion vacancies, the ratio of about four vacancies to one trap is not out of line.

6. The annealing experiments indicate that the presence of vacancies has no measurable effect on hardness: The vacancies anneal out completely at 350°C, while hardness remains unaltered up to about 750°C annealing temperature. In addition there was no correlation between the change of density and amount of work done in deforming the crystal, which probably reflects the fact that only a small amount of energy of deforma-

tion goes into creation of vacancies.²⁶ It is thus quite likely that hardness is associated with a disturbance of the dislocation pattern, especially of the Frank-Read sources, during the process of deformation and not with the presence of vacancies as slip obstacles. This is in agreement with some of the experiments discussed in the introduction. It should be remembered, of course, that one has to be careful in drawing conclusions from hardness, an essentially surface property, about internal phenomena.

7. Formation of deformation bands was analyzed in terms of the operating slip systems and the structure and orientation of the deformation band boundary.

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The authors wish to thank Professor F. Seitz for his interest and valuable discussions during the progress of this work.

²⁶ F. Seitz, *Advances in Physics* (Taylor and Francis, Ltd., London, 1952), Vol. 1, p. 43.

Effects of Contamination on the Characteristic Loss Spectrum of Tungsten*

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Two types of contamination, the adsorption of a monolayer of atmospheric gas and the deposition of carbonaceous material under electron bombardment, were found to affect the characteristic electron energy-loss spectrum of tungsten. Spectra were obtained by analyzing with a 127° electrostatic spectrometer the energy distribution of 850-volt electrons scattered by the target through 90°. The deposition of the carbonaceous material was associated with the growth of the carbon Auger peak. Each type of contamination could be removed by heating the target in vacuum to a suitable temperature.

INTRODUCTION

THE results of electron scattering experiments at low energies are markedly affected by the occurrence of two common types of surface contamination of the specimen. One type arises from the adsorption of a monolayer of residual atmospheric gas, and many workers¹ in this field have recognized the need for thorough outgassing of the specimen. Some direct effects of this contamination have been reported by Harrower.² The other type of contamination, which has also been

observed in many systems,³⁻⁵ is the deposition of carbonaceous material on those portions of the specimen irradiated by the electron beam.

The purpose of this paper is to show how these two types of contamination influence the characteristic energy-loss spectrum⁶ of tungsten.

APPARATUS

The specimen used in these experiments, a tungsten wire 0.01 in. in diameter, was the target for primary electrons from a Pierce-type electron gun.⁷ Scattered

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⁶ L. Marton, *Revs. Modern Phys.* **28**, 172 (1956).

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