ANOMALOUS WHISKER GROWTH

by

GLEN EUGENE HARLAND, JR.

B. S., Kansas State University
of Agriculture and Applied Science, 1958

A THESIS

submitted in partial fulfillment of the
requirements for the degree

MASTER OF SCIENCE

Department of Physics

KANSAS STATE UNIVERSITY
OF AGRICULTURE AND APPLIED SCIENCE

1960
# TABLE OF CONTENTS

INTRODUCTION ......................................................... 2

PROPOSED DESCRIPTION OF GROWTH ................................. 4

EXPERIMENTAL GROWTH PROCEDURE ................................. 10

The Procedure For Obtaining The I<sub>3</sub> ions In The Saturated KI Solutions 11

ANALYSIS ............................................................. 13

X-Ray Technique .................................................... 13

Rotation Pattern ................................................... 13

Laue Method ......................................................... 21

Optical Technique .................................................. 21

RESULTS .............................................................. 22

Growth ............................................................... 22

Analysis ............................................................. 22

CONCLUSION ........................................................ 31

ACKNOWLEDGMENT ................................................... 33

REFERENCES ......................................................... 34
INTRODUCTION

During the past few years, a great deal of experimental work concerned with the growth and perfection of crystals has been undertaken in the field of solid state physics. A particular type of crystal that has been of interest is the single crystal, in the form of a filament. This filamentary type of growth is given the name of "whiskers".

The primary purpose in working with the whisker type of growth is to study imperfections and dislocations in the crystal structure. Due to the small volume of the whisker, only a few dislocations are present.

For many centuries crystals have been assumed to have a definite atomic lattice structure. However, it was not until the early part of the twentieth century that a method was available to study the structure precisely. This was the utilization of x-ray diffraction introduced by Fredrick, Knipping, Laue, and Bragg.

In much x-ray diffraction work, the crystal structure is assumed to be a perfect geometrical lattice, with atoms residing at the intersection of lattice lines; however, it has been known for some time that actual crystals are not perfect but contain several types of imperfections. There are three main classes of imperfections; point imperfections, line imperfections, and surfaces of imperfection. Point imperfections include vacancies, impurity atoms, and interstitial atoms. Line imperfections are generally associated with dislocations. Surfaces of imperfection are associated with grain boundaries and surfaces of misfit. This report will be concerned with the line imperfection type associated with an edge and screw dislocation.

Dislocations are characterized by the amount of misfit of the crystal lattice around the line imperfection. The amount of the misfit is called the Burgers vector of the dislocation. An edge dislocation is a dislocation in
which the Burgers vector is perpendicular to the line imperfection. A screw dislocation is one in which the Burgers vector is parallel to the line imperfection. Thus, a dislocation may be a pure screw, pure edge, or a mixed dislocation containing components of both the screw and edge dislocation.

The alkali halide crystals have face centered cubic crystal lattices and are thus ideal for imperfection studies; i.e., the finding of distortions from the ideal lattice. A particular alkali halide, potassium iodide (KI), will be reported on in this paper.

Single KI crystals in the form of whiskers are grown from an aqueous solution in the following manner. A saturated solution of KI is obtained by adding KI crystals to distilled water until the solution will no longer dissolve the KI crystals. Then a slightly porous ceramic material is placed into the saturated KI solution for a period of two to three hours and allowed to absorb the KI solution. After the ceramic is well soaked, it is removed from the KI solution and placed within a container, where the ceramic is allowed to evaporate very slowly. After the ceramic is left under the container for a period of two to three days, it is observed that a filamentary type growth has been produced on the surface of the ceramic. Often the resulting growth is straight whiskers up to several millimeters in length and a few microns in maximum cross section dimension.

The theory of the filamentary crystal growth is given by Sears. He theorizes that the whisker growth takes place about an axial screw dislocation in the whisker. When a screw dislocation meets a surface normally, a ridge runs out from the point of emergence to the edge of the crystal. This step is self-perpetuating in the sense that it is not destroyed when atoms are deposited along it. Instead, it winds itself into a spiral, producing a growth pyramid on the surface of the crystal, on which deposition of the
material atoms or molecules can take place. Hence, the whisker is elongated in the direction of the axial screw dislocation.

The determination of the Burgers vector associated with the screw dislocation of a whisker has been given by Dragsdorf and Webb.

It has been noted that a few of the whiskers obtained from the preceding type of whisker growth were not straight, but crooked. This irregular whisker growth is given the name of anomalous whisker growth. Several people have proposed theories as to why such anomalous growth occurs. One theory by Amelinckx, suggested that the screw dislocation could shorten and partially relieve the line tension and produce this helical growth. Frank has proposed that the anomalous growth is due to a circular movement of a catalyst on the tip of the whisker. None of these theories fully explain the observed anomalous growth.

PROPOSED DESCRIPTION OF GROWTH

It is the purpose of this paper to explain the anomalous growth by a different theory. This theory proposes that it is the interaction between a pair of dislocations which causes the anomalous growth and not a single screw dislocation. It is proposed that a growing whisker takes on an edge component in addition to the screw dislocation present. The edge component will have a Burgers vector which is perpendicular to both the screw dislocation of the whisker and the direction of growth. In this manner, the resulting dislocation will have the two components of the screw and edge dislocation, and the two related Burgers vectors will add vectorially to give a resultant Burgers vector which is in a different direction than the component vectors, and also, in a different direction than the whisker growth (see figure 1).

The preceding discussion is used to explain the anomalous growth in the following manner. As the growing whisker elongates in the direction of the
screw dislocation, the center of the growth spiral will move across the growth surface of the growing whisker (see plate II). When the center of the growth spiral meets the side of the growing whisker, the direction of growth is changed since the growth site will be on the edge of the growing whisker. The Burgers vector of the screw and edge dislocation will then have a new direction which is different than the original direction. The new direction of growth will be in the direction of the new screw dislocation. The resultant of the two Burgers vectors in the new growth direction will again give rise to a dislocation which moves across the growth surface of the crystal as it elongates. The growth spiral will again intercept the edge of the growing whisker providing a new direction of growth. The preceding process continues and a uniform helical whisker should result (see plate III).
EXPLANATION OF PLATE II

Pictorial description of the dislocation moving across the growth surface of the growing whisker.
EXPLANATION OF PLATE III

Pictorial description of helical whisker growth. The straight line intersecting the sides of the whisker and then changing direction represents the path of the resultant Burgers vector (line imperfection).
EXPERIMENTAL GROWTH PROCEDURE

The edge component of the dislocation which interacts with the screw dislocation to produce the anomalous whiskers is manufactured by the introduction of foreign molecules into the lattice of the whisker. The foreign molecules or atoms that are placed in the crystal lattice are different in size and shape than those constituting the usual lattice. In making this addition, the normal sequence of building up the lattice is interrupted as the vapor deposition takes place upon the growth surface of the whisker. This could give rise to the edge component which could explain the anomalous growth.

If only one impurity atom is placed into the lattice, then a uniform helical whisker should result. If more than one impurity atom settles on the growth step of the whisker as it is growing, then an anomalous growth will result that is not uniform.

The anomalous growth that is observed indicates that the addition of impurities to a growth site causes this growth rather than the ceramic base from which the whisker grows. This is shown by the fact that a whisker may start growing straight, then suddenly will form a helical or irregular growth. Also, it is observed that sometimes the helical whisker growth will stop and the whisker will start growing straight, or take off in a new direction, which indicates that another impurity atom has been introduced into the lattice of the growing crystal in such a manner as to cause the lose of the edge component.

In the experiments to be described here the foreign material that is introduced into the KI whisker to produce the edge dislocation is the \( I_3^- \) (triiodide) ion. The \( I_3^- \) ion is introduced into the growth step of the growing whisker. Because the \( I_3^- \) ion is larger in size than the \( I^- \) ion,
an interruption of the normal growth pattern of the KI whisker occurs. This causes the crystal to form an imperfection in the lattice and gives rise to the edge component of the dislocation.

The Procedure For Obtaining The $I^-_3$ ions In The Saturated KI Solutions

Measured amounts of iodine ($I_2$) in the solid form are placed in containers filled with distilled water. Solid ($I_2$) iodine is slightly soluble in water at room temperature (.029 gm. per 100 ml). The dissolving of a few milligrams of iodine in 400 cubic centimeters of distilled water takes several weeks, at room temperature. A definite known number per unit volume of $I_2$ molecules that can be varied by further dilution of the iodine and water solution is then available for use.

The $I_2$ molecules obtained in the preceding manner can be used to produce the edge component of the dislocation.

The $I^-_3$ ions are produced by adding $I_2$ molecules to a saturated KI solution. $I_2$ molecules form with the $I^-$ ions of the KI solution to give the $I^-_3$ ions according to the chemical equation; $I_2 + I^- \rightarrow I^-_3$

The process proceeds in the direction of the arrow in the chemical equation. The reason for this is that the concentration of the $I^-$ ions is very much greater than the concentration of $I_2$ molecules placed in the KI solution. This gives an equilibrium constant for the equation that is very large and the process proceeds quickly as shown. Therefore, if a few $I_2$ molecules are placed in a saturated KI solution, $I^-$ ions will immediately form with the $I_2$ molecules to give the $I^-_3$ ions.

Only one $I^-_3$ ion is needed to replace an $I^-$ ion in each whisker to grow the helical whisker. Therefore, in a saturated solution of KI in which the ceramic is soaked are placed a number of $I^-_3$ ions corresponding to the number of whiskers that can be grown from the KI solution. This gives a one to one
correspondence if each I$_3^-$ ion goes into a single whisker. The number of whiskers that can be grown from a given volume of solution is calculated by finding the average volume of a single whisker and dividing this volume of an average whisker into the volume of KI in the solution. Then, one I$_3^-$ ion is placed into the solution for each whisker obtainable. Table 1 on page 13, lists the various concentrations of I$_3^-$ ions introduced into several 25 ml samples of KI with the resulting type of growth obtained from the ceramics. It is noted that in the region of small concentrations of I$_3^-$ ions per whisker, there is very little or no anomalous growth resulting on the ceramic.

Table 1, listing the sample number and type of growth present for a given amount of I$_2$ added, shows the following results.

For the samples in which a large number of I$_2$ molecules were added giving the I$_3^-$ ions, the resulting growth was mainly anomalous. The anomalous growth had no definite pattern or type and was crooked throughout. This is an indication that a large number of I$_3^-$ ions were present when the whisker was growing. Each time an I$_3^-$ ion was crystallized into the growth step, the direction of the growth changed.

For the samples from three through nine, it was noticed that many anomalous and some straight growths resulted. Many of the anomalous growths were helical in nature which indicated that only one I$_3^-$ ion was being introduced into the lattice for each whisker.

For samples ten through twelve, the resulting growths were primarily straight, with only a few anomalous type growths resulting. This was to be expected in that the number of I$_3^-$ ions placed in the solution was smaller than the number of whiskers which could have been grown from the KI in solution.

The preceding results clearly demonstrated that the effect of adding foreign molecules or ions into the lattice of the growing crystal was to
cause anomalous growth. In particular, this indicated that the anomalous or helical growths resulting were due to the replacing of $I^-$ by $I_3^-$ ions in the lattice.

**ANALYSIS**

The helical growths that resulted from the above mentioned experiments were analyzed by x-ray and optical methods.

X-ray techniques were used to determine the helical growth axis and the Burgers vector of the dislocation. The combination of x-ray and optical techniques provided a method to determine the crystal growth axis.

**X-ray Technique**

The whiskers that have been grown from a ceramic base were carefully detached from the base and mounted on thin glass rods with duco cement. Glass rods were used since glass does not give an x-ray pattern that interferes with the crystal x-ray pattern. A cobalt target tube was used to produce the x-ray radiation. Due to the small size of the crystals, the time required to obtain x-ray patterns that were suitable for analysis was from ten to thirty hours.

**Rotation Pattern.** A rotation method was used to find the helical growth direction of the whisker. In taking a rotation pattern, the whisker to be analyzed was placed in a Weissenberg camera in such a way that the incident x-ray beam was perpendicular to the helical or straight axis of the whisker. The whisker was rotated about this axis (see plate IV). The rotation pattern gave the periodicity (see plate V) of the crystal along the helix, and is explained in the following manner.
Table 1

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Number of I₂ Molecules Added Per 25 ml. KI Saturated Solution</th>
<th>Type of Growth Observed From Ceramics</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.4 X 10&lt;sup&gt;11&lt;/sup&gt;</td>
<td>Mainly crooked growth with a few straight present.</td>
</tr>
<tr>
<td>2</td>
<td>4.2 X 10&lt;sup&gt;13&lt;/sup&gt;</td>
<td>For samples 3 to 9 there was a large percentage of anomalous and helical growth present, and only a few straight filaments.</td>
</tr>
<tr>
<td>3</td>
<td>5.3 X 10&lt;sup&gt;12&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>5.3 X 10&lt;sup&gt;11&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>5.3 X 10&lt;sup&gt;10&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>6.3 X 10&lt;sup&gt;9&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>10.4 X 10&lt;sup&gt;9&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>5.2 X 10&lt;sup&gt;8&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>2.6 X 10&lt;sup&gt;8&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>1.6 X 10&lt;sup&gt;8&lt;/sup&gt;</td>
<td>Mostly straight growth, a few crooked growth.</td>
</tr>
<tr>
<td>11</td>
<td>5.2 X 10&lt;sup&gt;7&lt;/sup&gt;</td>
<td>Mostly straight growth, a few crooked growth.</td>
</tr>
<tr>
<td>12</td>
<td>5.2 X 10&lt;sup&gt;6&lt;/sup&gt;</td>
<td>Mostly straight growth.</td>
</tr>
<tr>
<td>13</td>
<td>None</td>
<td>Mostly straight growth.</td>
</tr>
</tbody>
</table>

The average number of whiskers which can be grown from a 25 ml saturated KI solution is 4.4 X 10<sup>8</sup>.
For any x-ray diffraction work, the Laue equation must be satisfied.

\[ S - S_0 = \lambda H. \]  

(1)

\( S \) is a unit vector in the direction of the diffracted radiation, \( S_0 \) is a unit vector in the direction of the incident radiation, \( \lambda \) is the wavelength of the x-ray radiation used, and \( H \) is the reciprocal lattice vector given by

\[ H = h \mathbf{b}_1 + k \mathbf{b}_2 + l \mathbf{b}_3, \quad \text{or} \]

\[ |H| = \frac{1}{d}. \]

\( \mathbf{b}_1, \mathbf{b}_2, \) and \( \mathbf{b}_3 \), are unit cell vectors in reciprocal space. \( h, k, \) and \( l \), are integers representing the reciprocals of the fractional intercepts which the lattice plane nearest and parallel to that passing through the origin makes with the crystallographic axes, \( d \) is the planar spacing.

The Laue equation is applied to the x-ray diffraction which results from the rotation of the crystal about its helical axis (see plate IV). Taking the scalar product of the Laue equation with the periodicity axis vector \( (A) \) of the single crystal shown in plate V, the following will result.

\[ (S - S_0) \cdot A = \lambda H \cdot A. \]

Where \( A = m_1 \mathbf{a}_1 + m_2 \mathbf{a}_2 + m_3 \mathbf{a}_3 \), or

\[ |A| = (m_1^2 a_1^2 + m_2^2 a_2^2 + m_3^2 a_3^2)^{1/2}. \]

\( m_1, m_2, \) and \( m_3 \), are integers representing the number of unit cells along a unit cell axis. \( \mathbf{a}_1, \mathbf{a}_2, \) and \( \mathbf{a}_3 \), denote unit cell vectors of the three crystal axes.

\[ S \cdot A - S_0 \cdot A = \lambda (h \mathbf{b}_1 + k \mathbf{b}_2 + l \mathbf{b}_3) \cdot (m_1 \mathbf{a}_1 + m_2 \mathbf{a}_2 + m_3 \mathbf{a}_3). \]

\( S_0 \cdot A = 0 \) since the incident beam is perpendicular to the rotation axis.

\[ S \cdot A = |S||A| \cos (90 - B) = A \sin B, \]

where the angle \( B \) is described on plate IV.
Therefore;

\[ A \sin B = \frac{\lambda(hm_1 + km_2 + lm_3)}{\sin B}, \]

or

\[ A = \frac{\lambda(hm_1 + km_2 + lm_3)}{\sin B} = \frac{\lambda}{\sin B}, \quad (4) \]

where \( N = hm_1 + km_2 + lm_3 \)

(5)
is always equal to some integer.

Equation four will give the periodicity \((A)\) of the crystal in a direction normal to the incident beam.

Since the values of \( a_1, a_2, \) and \( a_3 \), are equal and known for KI, equation three will give an equation relating \( m_1, m_2, \) and \( m_3 \).

\[ m_1^2 + m_2^2 + m_3^2 = \left( \frac{A}{a} \right)^2 \quad (6) \]

By analysis of the diffraction spots on the rotation film, according to the \((hkl)\) values of the plane causing each spot, equation five can be solved to account for all diffraction spots. When this occurs, the helical growth axis will be the \((m_1, m_2, m_3)\) values of equation five.

The preceding can be seen by direct analysis of plate V,

\[ \cos(A, a_1) = \frac{m_1a_1}{A} = \frac{m_1a}{A} = m_1c \]

\[ \cos(A, a_2) = \frac{m_2a_2}{A} = \frac{m_2a}{A} = m_2c \]

\[ \cos(A, a_3) = \frac{m_3a_3}{A} = \frac{m_3a}{A} = m_3c \]

The three preceding equations are the direction cosines of the helical growth axis with respect to the three crystal axes. Hence, \( m_1, m_2, \) and \( m_3 \), will be the Miller indicies of the helical growth axis and are usually specified in terms of \((hkl)\) values.
EXPLANATION OF PLATE IV

Helical sample in the rotation camera setup.
PLATE IV

Helical Axis (A)

Diffraction Spot

Helical Sample

Film Holder

Glass Mount
EXPLANATION OF PLATE V

Single crystal in the rotation camera setup showing periodicity.
The third dimension of the crystal is perpendicular to and into the surface of the paper.
\[ A = m_1 a_1 + m_2 a_2 + m_3 a_3 \]

\[ |A| = \left( m_1^2 a_1^2 + m_2^2 a_2^2 + m_3^2 a_3^2 \right)^{\frac{1}{2}} \]
**Laue Method.** The Laue technique can be used to determine the Burgers vector of the dislocation in a whisker. Eshelby predicted that a whisker containing an axial screw dislocation should be twisted by an amount

\[ \alpha = \frac{kb}{A}, \]

where \( k \) is a constant depending upon the geometry of the whisker, \( b \) is the Burgers vector of the dislocation, \( A \) is the cross-sectional area of the whisker, and \( \alpha \) is the twist in radians per unit length. This lattice twist can be calculated by measuring the angle \( \sigma \) in which the equatorial Laue spots are tilted in the x-ray Laue pattern. This work is reported by Dragsdorf and Webb. The Burgers vector will be given by

\[ b = A C \tan \sigma, \]

where \( C \) is a constant depending upon the camera geometry.

**Optical Technique**

Optical measurements can be combined with the x-ray results to determine the crystal growth axis. The whiskers were examined with a Leitz microscope, to determine such things as the length of one complete cycle of growth, diameter of the helix, and the angle the growth axis made with the helical growth axis. Both transmitted and reflected light microscopy were used. The crystal growth axis is calculated by combining the direction of the helical growth axis with the observed angle between the helical growth axis and one of the helix sides in the following manner:

\[ H_1 = (h_1 b_1 + k_1 b_2 + l_1 b_3), \]  

(6) is the reciprocal lattice vector of the helical axis.

\[ H_2 = (h_2 b_1 + k_2 b_2 + l_2 b_3), \]  

(7) is the reciprocal lattice vector of the crystal growth axis.
Taking the dot product of 6 and 7 gives

\[ H_1 \cdot H_2 = (h_1 b_1 + k_1 b_2 + l_1 b_3) \cdot (h_2 b_1 + k_2 b_2 + l_2 b_3) \]
\[ = |H_1||H_2| \cos (H_1, H_2) \quad \text{or} \]
\[ \cos (H_1, H_2) = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\sqrt{h_1^2 + k_1^2 + l_1^2} \sqrt{h_2^2 + k_2^2 + l_2^2}} = \cos \theta. \quad (8) \]

RESULTS

Growth

The results of the anomalous growth from the controlled experiment in which different numbers of \( I^- \) ions were placed into various saturated solutions of KI are given in table 1. The resulting anomalous growth obtained from the ceramics indicated the effect of the impurities introduced into the growing whiskers. If too many impurities were present when growth was taking place, an undefined type of anomalous growth resulted. If there were a small number of impurities present at the time of growth, the resulting growth was primarily straight. However, if there was an approximate one to one correspondence of the impurities present with the number of whiskers which could be grown from the KI solution, then the resulting growth was helical in nature.

Analysis

The tabulated analysis of the helical whiskers is given in table 2. Column one gives the number of the helical samples analyzed. Column two gives the equation for the layer lines of the rotation pattern, which relates the planes causing each diffraction spot to the \( N \) value of the layer line. The coefficients of the \( hkl \) terms in column two give the \( hkl \) values of the helical growth direction repeated in column four. Column six gives the angle between the helical growth direction and the crystal growth direction, obtained by
optical methods. The combining of the data in column six and column four with equation eight gives the crystal growth axis tabulated in column five. Column seven gives the results of equation four. Column eight gives the tabulated values of the crystal periodicity according to equation two. Column nine gives the pitch of each helical whisker obtained by optical methods.

It is noticed in column five that the primary crystal growth axis is [110] for the helical whiskers. This is to be expected in that this is a habit direction of growth for KI crystals.

No definite results were obtained from the Laue analysis.

The optical analysis can be summed up best by looking at some helical growth. A few of the resulting helical growths are given in plates VI, VII, and VIII with explanations.
### Tabulated Results of Analysis

<table>
<thead>
<tr>
<th>Whisker Number</th>
<th>Results of Rotation Pattern</th>
<th>Laue Spot Tilt</th>
<th>Helical Growth Crystal Growth Axis [hkL]</th>
<th>Helical Growth Crystal Growth Axis [hkL]</th>
<th>$m_1^2 + m_2^2 + m_3^2$ A units</th>
<th>Pitch of Helix in mm</th>
<th>Solution from which growth occurred</th>
</tr>
</thead>
<tbody>
<tr>
<td>KI-97</td>
<td>$2h - 2k + L = N$</td>
<td>$60^\circ$</td>
<td>221</td>
<td>___*</td>
<td>9</td>
<td>21.3</td>
<td>9</td>
</tr>
<tr>
<td>KI-109</td>
<td>$4h + 5k + 4L = N$</td>
<td></td>
<td>454</td>
<td>111</td>
<td>18</td>
<td>57</td>
<td>55.0</td>
</tr>
<tr>
<td>KI-114</td>
<td>$5h + 5k + 6L = N$</td>
<td></td>
<td>556</td>
<td>110</td>
<td>40</td>
<td>86</td>
<td>65.6</td>
</tr>
<tr>
<td>KI-115</td>
<td>$5h + 2k + 4L = N$</td>
<td></td>
<td>524</td>
<td>101</td>
<td>20</td>
<td>45</td>
<td>47.3</td>
</tr>
<tr>
<td>KI-116</td>
<td>$5k + 2L = N$</td>
<td></td>
<td>052</td>
<td>110</td>
<td>52</td>
<td>29</td>
<td>39.3</td>
</tr>
<tr>
<td>KI-119</td>
<td>$5h + 6k + 5L = N$</td>
<td>$45^\circ$</td>
<td>565</td>
<td>110</td>
<td>30</td>
<td>86</td>
<td>65.1</td>
</tr>
<tr>
<td>KI-122</td>
<td>$3h + k + L = N$</td>
<td></td>
<td>311</td>
<td>010</td>
<td>75</td>
<td>11</td>
<td>23.0</td>
</tr>
<tr>
<td>KI-123</td>
<td>$2h + k + 5L = N$</td>
<td>$74^\circ$</td>
<td>215</td>
<td>011</td>
<td>38</td>
<td>30</td>
<td>38.5</td>
</tr>
</tbody>
</table>

* Whisker was lost before optical picture was taken.

+ Possibility of either 101, 111, or 211 axis.
EXPLANATION OF PLATE VI

Reflected light microscope pictures of various potassium iodide helices

<table>
<thead>
<tr>
<th>Location</th>
<th>Code</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper Left</td>
<td>KI - 123</td>
<td>360 X</td>
</tr>
<tr>
<td>Upper Right</td>
<td>KI - 122</td>
<td>136 X</td>
</tr>
<tr>
<td>Lower Left</td>
<td>KI - 110</td>
<td>136 X</td>
</tr>
<tr>
<td>Lower Right</td>
<td>KI - 110</td>
<td>360 X</td>
</tr>
</tbody>
</table>
EXPLANATION OF PLATE VII

Reflected light microscope pictures of various potassium iodide helices

<table>
<thead>
<tr>
<th>Location</th>
<th>Sample</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>UPPER LEFT</td>
<td>KI - 116</td>
<td>136 X</td>
</tr>
<tr>
<td>UPPER RIGHT</td>
<td>KI - 118</td>
<td>136 X</td>
</tr>
<tr>
<td>LOWER LEFT</td>
<td>KI - 114</td>
<td>136 X</td>
</tr>
<tr>
<td>LOWER RIGHT</td>
<td>KI - 115</td>
<td>136 X</td>
</tr>
</tbody>
</table>
**EXPLANATION OF PLATE VIII**

Reflected light microscope pictures of various potassium iodide helices

<table>
<thead>
<tr>
<th>Location</th>
<th>Description</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper Left</td>
<td>KI - 115</td>
<td>360 X</td>
</tr>
<tr>
<td>Upper Right</td>
<td>KI - 107</td>
<td>136 X</td>
</tr>
<tr>
<td>Lower Left</td>
<td>KI - 111</td>
<td>136 X</td>
</tr>
<tr>
<td>Lower Right</td>
<td>KI - 119</td>
<td>136 X</td>
</tr>
</tbody>
</table>
CONCLUSION

The results (see table 1) of the growth experiment, in which impurities were placed into the lattice of a growing whisker show that there is a definite correlation between the type of growth which resulted and the number of impurities that were placed in the solution from which the whiskers grew. In particular, the impurities placed in the solution are associated with anomalous growth.

The analysis of the helical growth indicates that the proposed theory of the helical whisker growth by a mixed dislocation containing both a screw and edge component is quite reasonable. The helical whiskers are found to be single crystals in which the crystal growth occurs primarily along one of the (110) directions of the crystal.

Some of the results tabulated in table two are worthy of extra attention. It is noticed that whiskers KI-114 and KI-119 have the same crystal growth axis [110] and the same [hkl] values of the helical axis, and are grown from the same solution (Mixture 9).

The majority of whiskers analyzed were grown from solution number 7. This would indicate that the number of impurities present in the KI solution was optimum for the growth of helical whiskers. The number of whiskers which could be grown from a 25 ml saturated KI solution is approximately $4.4 \times 10^9$. The number of impurity atoms placed in solution number 7 was $10.4 \times 10^9$. This is close to a one to one correspondence of the number of whiskers obtainable from the 25 ml KI solution and the number of impurities present. This gives good experimental agreement with the proposed growth theory.

It was expected that the lattice twist associated with a growing whisker could explain the three dimensional helical growth; however, the lattice twist
was calculated for several helical whiskers by measurement of the equatorial Laue spot tilt from the Laue patterns, and the calculations show that the lattice twist alone will not account for the large three dimensional growth.

It is reasonable to assume that the three dimensional helical growth must be due to a more complicated form of edge component, instead of the single edge dislocation proposed. A more complicated edge component could be one in which there are two components of the edge dislocation. The resulting Burgers vector for such a dislocation could be visualized as being made up of three components, as in an x, y, z coordinate frame.
ACKNOWLEDGMENT

The author wishes to express his appreciation for the assistance and guidance of Doctor R. Dean Dragsdorf in carrying out this project.
REFERENCES


(7) Frank, Lake Placid Conference on Dislocations. Lake Placid, New York. 1956


ANOMALOUS WHISKER GROWTH

by

GLEN EUGENE HARLAND, JR.

B. S., Kansas State University
of Agriculture and Applied Science, 1958

A THESIS

submitted in partial fulfillment of the
requirements for the degree

MASTER OF SCIENCE

Department of Physics

KANSAS STATE UNIVERSITY
OF AGRICULTURE AND APPLIED SCIENCE

1960
The work reported here is concerned with the growth of anomalous whiskers, that is, helical and irregular but essentially single crystal whiskers. It is the purpose of this paper to describe how the anomalous whiskers can be obtained experimentally, and to formulate a growth mechanism to explain the growth.

It is proposed that the anomalous whisker growth is due to the interaction between a pair of dislocations and not the single screw dislocation which is used to explain the regular whisker type crystal growth. It is proposed that for helical growth a growing whisker takes on an edge component in addition to the usual screw dislocation present. The edge component will have a Burgers vector which is perpendicular to both the screw dislocation and the direction of growth. In this manner, the resulting dislocation will have the two components of the screw and edge dislocation. The two related Burgers vectors will add vectorally to give a resultant Burgers vector which lies in a direction different from the component vectors, and also, in a direction different from the whisker growth. The direction of whisker growth is always along the direction of the screw component of the dislocation.

The edge component of the dislocation which interacts with the screw dislocation to produce the anomalous whiskers is produced by the introduction of foreign molecules into the lattice of the whisker. The foreign molecules or atoms that are placed in the crystal lattice are different in size and shape than those constituting the usual lattice. In making this addition, the normal sequence of building up the lattice is interrupted as the vapor deposition takes place upon the growth surface of the whisker. It is here assumed that this interruption gives rise to the edge component which explains the anomalous growth.
The foreign material that was introduced into the KI whisker to produce the edge dislocation was the $I_3^-$ (triiodide) ion. The $I_3^-$ ions are produced by adding $I_2$ (iodine) molecules to a saturated KI solution. $I_2$ molecules form with the $I^-$ ions of the KI solution to give the $I_3^-$ ions according to the chemical equation $(I^- + I_2 \rightarrow I_3^-)$.

Eight helical whisker growths that resulted from experiment were analyzed by x-ray and optical methods. X-ray techniques were used to determine the helical growth axis and the Burgers vector of the dislocation. The combination of x-ray and optical techniques provided a method to determine the crystal growth axis.

The results of the growth experiment, in which impurities were placed into the lattice of the growing whisker show a definite correlation between the type of growth that resulted and the number of impurities that were placed in the solution from which the whiskers grew. In particular, it seems apparent that the impurities placed in the solution must be associated with the anomalous growth.

The analysis of the helical growth indicates that the proposed theory of the helical whisker growth by a mixed dislocation containing both a screw and edge component is quite reasonable. The helical whiskers were found to be single crystals in which the crystal growth occurred primarily along one of the $[110]$ directions of the crystal.