

THE PROPERTIES OF WHISKERS

É. M. NADGORNÝĀ

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INTRODUCTION

THE growth of crystals in the form of filaments has been familiar for a very long time,^[1] but the investigation of the properties of filamentary crystals or "whiskers" did not begin practically until 1952, when their extraordinary mechanical properties were first observed.^[2] Whiskers have been studied very intensively in recent years; they have been the subject of two international conferences,^[3,4] and many meetings, and their properties have been discussed in more than 200 scientific publications. Whiskers of 30 elements and more than 50 compounds have thus far been grown, and work is being done continually to produce and study whiskers of additional substances.

This great interest is accounted for by several properties of whiskers, which encompass a very wide range of materials—metals, semiconductors, and ionic, molecular, and organic crystals. At the present time the term "whisker" is applied only to crystals with small diameters of the order of a micron and exhibiting certain unusual properties, especially large elastic deformation. This suggests immediately that whiskers represent a practical realization of the properties of perfect crystal lattices, which have been predicted theoretically but could not previously be checked experimentally. The validity of these predictions can be determined only by a detailed investigation of whiskers.

The most characteristic properties of whiskers are:

- 1) very large elastic deformation up to several per cent;
- 2) a different form of the stress-strain and bending curves in the plastic region than for ordinary crystals;
- 3) several peculiarities of phase transitions;
- 4) slower rates of oxidation, evaporation, and dissolution;
- 5) high coercive force (for iron), several thousand times greater than that observed ordinarily.

The optical and electrical properties of whiskers have been studied much less, so that we can expect to find new features of these properties in whiskers.

The present review covers mainly the results obtained in investigations of whiskers performed during the last few years and published after the appearance of a previous review in 1959.^[5] Earlier data will sometimes be presented for the purpose of maintaining continuity. In contrast to^[6] whisker growth will not be discussed especially, since the amount and im-

portance of the data in this branch of the subject warrant a separate review.

I. MECHANICAL PROPERTIES OF WHISKERS

1. **Elastic and plastic deformations.** The mechanical properties of whiskers are usually studied by obtaining stress-strain diagrams, using special apparatus for the extension, bending, and twisting of whiskers.^[6-11] The initial portion of a strain or bending curve exhibits a large region of elastic deformation, extending hundreds of times beyond the maximum elastic deformation of ordinary single crystals. Thus the yield point of whiskers approaches the lower limit of theoretical shear strength, equal to $\frac{1}{30}$ of the shear modulus, or even sometimes exceeds the latter. The largest elastic deformations and the corresponding stresses for whiskers of several different substances are given in the table. The elastic deformation of a whisker of any substance is ordinarily at least of the order of 1%.

Not all the investigated whiskers have the same high degree of strength; a considerable spread of the strength is associated with the conditions of production and with the structure and diameters of the crystals. The greatest strength is found in individual "unique" specimens, while most whiskers possess less strength, yet still considerably exceeding the strength of ordinary bulk crystals. Almost all of the investigated materials exhibit a clear relation between the strength and diameters of whiskers.^[15] When the yield point has been reached there are three possibilities: a whisker will exhibit either brittle fracture, plastic deformation, or creep. At room temperature brittle fracture occurs in the whiskers of Si, Te, MoO₃, Al₂O₃, and SiO₂; at lower temperatures this occurs in Zn and Cd. Very thin whiskers of copper and particularly of iron sometimes fracture after very small plastic deformation that is unobserved in the appropriate diagram but is discovered from the presence of necking at the fracture.

For the whiskers of substances that deform plastically the stress-strain curves usually resemble Fig. 1. Here point 1 is the yield stress σ_1 , which is followed by an abrupt decrease of the stress to point 2. Beginning with point 2, plastic deformation takes place at a stress σ_2 about 30–100 times smaller than σ_1 . The flow stress remains constant except for small abrupt changes not shown in the diagram. Plastic deformation usually occurs through the propagation of slip

Substance	Method of production	Method of testing	Maximum elastic deformation, %	Stress, kg/mm ²	Reference
1. Fe	Halide reduction	Tensile	4.9	1350	6
2. Cr	Separation from solid phase	Bending	3.8	800	12
3. Cu	Halide reduction	Tensile	3.8	450	13
4. Si	The same	"	3.6	680	14
5. Al ₂ O ₃	Oxidation in gaseous phase	Bending	3.0	1500	15
6. NaCl	Evaporation through a membrane	Tensile	2.6	100	16
7. C	In a high-pressure arc	"	2.0	2000	17

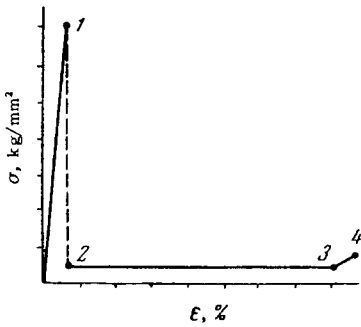


FIG. 1. Typical stress-strain diagram of a whisker in the elastic and plastic regions.

bands (Lüders bands) that are visible on the surface; these require the stress σ_2 for their generation or propagation. The plastic deformation sometimes reaches 60–80%. Slight hardening (point 3) precedes fracture. We thus see that the strength of whiskers diminishes abruptly after the onset of plastic formation. Graphite whiskers are an exception to this rule.^[27] If the bending of a graphite whisker results in a kink, which is evidence of plastic deformation, the whisker will lose none of its strength after being straightened out. This is accounted for by the very

great anisotropy of graphite and by the special structure of graphite whiskers, which consist of cylindrical layers twisted in the form of a cigar; the [0001] axis is perpendicular to the whisker axis. Local deformation does not result in fracture, but only in the wrinkling of a single layer.

The stress-strain curves of copper also sometimes exhibit, after the onset of plastic deformation, a more pronounced alternation of the rise and fall of the stress; this is associated with the impeding of flow at any obstacles.^[18] Figure 2 shows examples of the propagation of plastic deformation in copper and zinc whiskers. With increasing diameter of a copper whisker σ_2 diminishes somewhat; at the same time there is increased likelihood that a few Lüders bands will be generated. Sometimes slip in Lüders bands occurs in two different systems (Fig. 2c).

The mechanism of plastic deformation propagation in whiskers is still not entirely understood. Brenner^[18] has suggested that the development of Lüders bands in silver and copper whiskers proceeds through the double cross-slip of dislocations. However, Charsley^[28] has

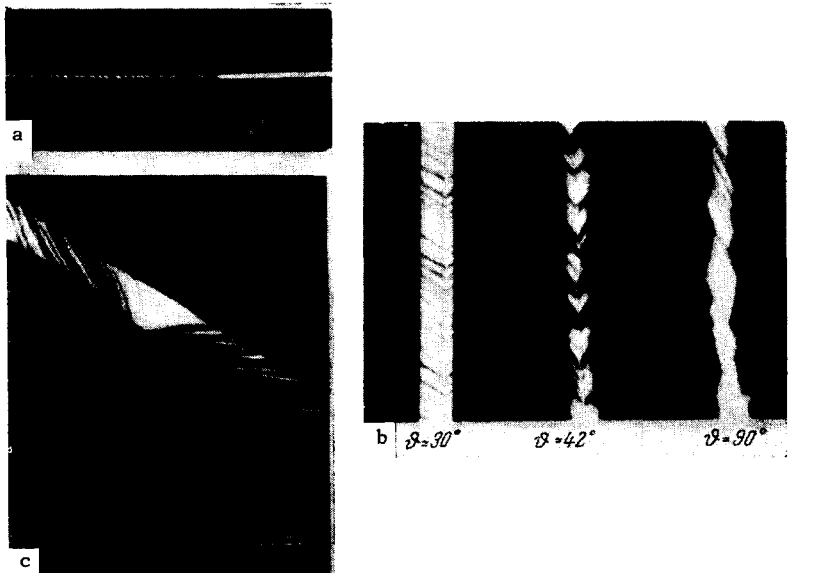


FIG. 2. Propagation of plastic deformation in whiskers. a) Lüders band in a thin copper whisker;^[15] b) Lüders bands in zinc whiskers with different orientations (φ is the angle between the whisker axis and the c axis^[15]); c) two intersecting Lüders bands in a thick copper whisker.^[15]

observed that in copper whiskers slip can occur in a single system; if slip occurs in several systems it proceeds much more intensely in one of these, while single cross-slip occurs in the other systems, i.e., double cross-slip does not necessarily occur. He therefore believes that Lüders bands are propagated through the concentration of stresses between parts of a whisker subjected to shear and undeformed parts.

Price^[29,30] has carefully studied plastic deformation in the extension of zinc whiskers and platelets with their axes lying in the basal plane ($\varphi = 90^\circ$ in Fig. 2b) and having less than 0.5μ thickness. (This limitation on thickness was imposed because, in addition to plotting the stress-strain curve, the motion of dislocations was observed in an electron transmission microscope.)^[31] The direction of elongation was always parallel to the (0001) plane. Therefore basal slip could not occur, and plastic deformation developed either by pyramidal slip with the formation of Lüders bands perpendicular to the axis of extension or by the creation and propagation of a twin. Sometimes both types of deformation occurred together in a crystal.

In the first case pyramidal slip occurred in the system (11 $\bar{2}$ 2) [$\bar{1}\bar{1}$ 23]. The macroscopic yield stress representing approximately 0.2% deformation for platelets and 1% for whiskers, corresponds to the appearance of a very narrow plastic deformation zone a few microns wide (Fig. 3). This zone contains traces of pyramidal slip and dislocations of three kinds. Long dislocations with the Burgers vector $1/3\langle\bar{1}\bar{1}20\rangle$ and lying mainly in the basal plane are easily pinned by obstacles, so that their motion produces only a very insignificant portion of the plastic



FIG. 3. Electron micrograph showing narrow deformed zone immediately after its formation in a zinc whisker. The zone boundaries are indicated by arrows. Pyramidal slip traces and dislocations are visible within the zone.^[30]

deformation. The deformation occurs mainly through the formation and motion of short $\langle\bar{1}\bar{1}23\rangle$ screw dislocations progressing from the upper to the lower surface of the crystal (Fig. 3). These dislocations induce pyramidal slip and sometimes leave behind long narrow $\langle\bar{1}\bar{1}23\rangle$ loops resulting from large jogs in these dislocations; these loops subsequently become circular with the same Burgers vector. Loops with the Burgers vector $\langle 0001 \rangle$ can also result from loops of the first type in a dislocation reaction. Both types of loops impede the motion of dislocations. With increasing deformation the loop density grows until all dislocations are pinned, and further shear will occur at the edge of the deformed zone, where there are few loops. The deformation zone is thus expanded along the crystal, and new Lüders bands appear on the surface.

If a crystal is unloaded and kept at room temperature for about an hour, flow is renewed at the same stress as previously, but new slip lines appear inside the deformed zone. This results from the fact that at room temperature loops are reduced in diameter through climb (since they are prismatic) and disappear in approximately an hour. (Judging from the activation energy of this process, vacancy diffusion occurs between loops and the crystal surface.) Consequently, moving dislocations do not encounter obstacles and further deformation is possible so long as the number of loops is insufficient to obstruct dislocations. New slip traces are thus produced in the deformed zone.

Twinning, the second kind of deformation, is encountered more frequently in crystals of this orientation; twinning occurs in the (10 $\bar{1}$ 2) plane and [10 $\bar{1}$ 1] direction. Figure 4 is a typical stress-strain curve of these crystals. Point 1 represents the nucleation of a narrow wedge-shaped twin on one of the lateral faces (Fig. 5) at the critical shear stress, which varies from 10 kg/mm^2 for large platelets to 60 kg/mm^2 for whiskers. A twin always appears at the location of a higher stress concentration. This twin is then propagated through the crystal under a lower stress (Fig. 4). When the twin reaches the opposite side of the crystal the twin boundaries begin to move

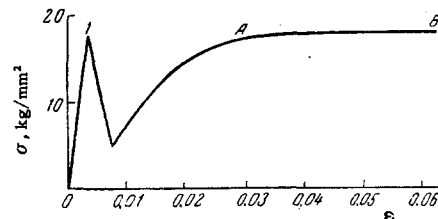


FIG. 4. Typical stress-strain curve for a zinc platelet which is deformed by twinning. The axis of the platelet lies in the basal plane. Twin nucleation occurs at point 1. The twinning lamella propagates along the platelet in the region AB, where the stress usually is considerably below the twin nucleation stress.^[30]

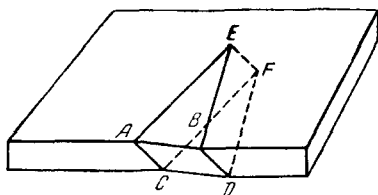
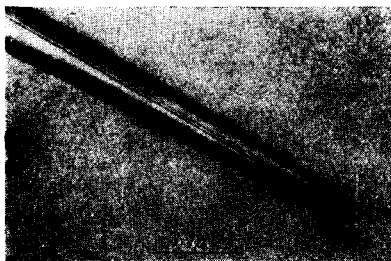


FIG. 5. Schematic diagram of a wedge-shaped twin nucleating in a zinc platelet, and electron micrograph of its tip. The platelet occupies the entire field of view.^[30]



in opposite directions along the length of the crystal at an almost constant stress varying from 2 to 20 kg/mm² in different samples and usually less than the nucleation stress. (In Fig. 4 the propagation stress of the twin is almost equal to the nucleation stress.)

The propagation of a twin results from the production and motion of twinning dislocations. The propagation velocity is completely determined by the deformation rate. It was sometimes possible to convert an entire crystal to a twin orientation before fracture. More frequently, fracture occurred earlier, either following the formation of a crack in the twin or after appreciable basal glide with the appearance of many dislocations on the glide plane (which is possible due to the crystal reorientation), leading to shear between parts of the crystal. An analysis of the conditions for the creation of stress concentrations and for twin nucleation shows that although the critical shear stress for twinning, as determined experimentally from the load, cross section, and orientation, differs for each tested crystal, yet when the concentration effect of the surface defect in the region of twin nucleation is taken into account, the critical shear stress for twinning is always of the same order of magnitude, about 50 kg/mm² on the average. This value is in good agreement with Orowan's theory^[32] of the homogeneous nucleation of a twin lamella. Therefore both twinning and slip in whiskers begin at near-theoretical stresses.

2. Creep and fatigue in whiskers. There have not been many investigations of creep in whiskers. Creep has been observed in whiskers of silicon,^[7,10] zinc,^[19] and iron.^[10,15] Creep is not ordinarily observed at room temperature if the creep stress does not exceed the yield stress σ_1 . Cabrera and Price^[19] have sometimes observed creep in the elastic region of zinc whiskers after about 1% deformation, but before the onset of flow. The initial creep rate increased with the stress and was of the order 10^{-3} per hour at about 10 kg/mm², but dropped to zero after a few hours. No recovery occurred after unloading; the creep was completely irreversible. Following the termination of

creep the yield stress of zinc whiskers was sometimes doubled. However, some whiskers exhibited no creep even after a lengthy stressed period. The microcreep in the elastic region was found to result from the presence of dislocations.^[20] The observed creep results from the motion of these dislocations under a stress. After the dislocations emerge from the crystal or reach a more stable position, creep is terminated; a higher stress is then required to initiate plastic flow in the same whisker. The dislocations may have originated during growth or, as is more probable, as a result of insufficiently careful handling. When no dislocations were originally present in a crystal, creep would not appear during several hours of loading.

Creep in silicon whiskers has occurred at 800°C under a stress of about $\frac{2}{3}\sigma_1$.^[7] The creep rate increased with time, reaching its maximum in about a minute, after which it decreased to zero in a few minutes. The general character of this creep resembles that observed in zinc,^[19] and the same cause can be assigned.

After the yield point is reached in iron whiskers without fracture, further plastic deformation occurs through creep. Since in iron whiskers having diameters smaller than 6 μ the deformation is highly localized and it is difficult to produce plastic deformation without breaking the whisker, larger diameters were used in the investigation of creep.^[15] The creep curves were plotted under constant load, with the plastic deformation measured as a function of time. The creep curves for different whiskers were not entirely alike, but usually exhibited three regions like ordinary bulk crystals—the region of diminishing creep rate, that of a constant rate, and that of accelerated creep. Figure 6 shows creep in an iron whisker of 14 μ diameter at 13 kg/mm². In the linear region the creep rate $\dot{\epsilon}$ depends exponentially on the creep stress σ . During creep Lüders bands are propagated from the nucleation site of the first localized shear (Fig. 7, a and b), and shear lamellae are also formed in separate regions (Fig. 7c). Shear occurs in several slip systems.

Eisner^[10] has observed creep in silicon and iron whiskers during twisting tests. Figure 8 shows hysteresis in iron whiskers following twisting beyond the

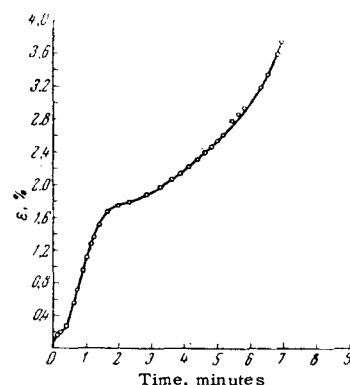


FIG. 6. Creep in an iron whisker of 14 μ diameter under the stress $\sigma = 13$ kg/mm² after plastic deformation at 38 kg/mm². In the linear region the creep rate is exponential: $\dot{\epsilon} = 10^{-7} \exp(0.56\sigma)$.^[15]

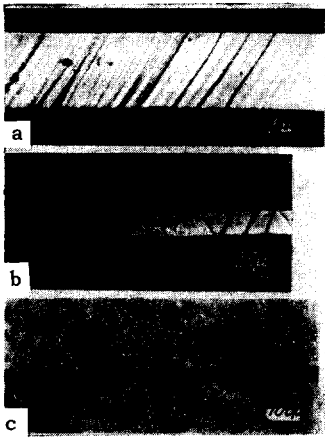


FIG. 7. Propagation of a plastic deformation in an iron whisker during creep. a) Plastic deformation $< 0.5\%$; b) plastic deformation $\approx 4.0\%$; c) plastic deformation $\approx 10.0\%$ (transmitted light).^[15]

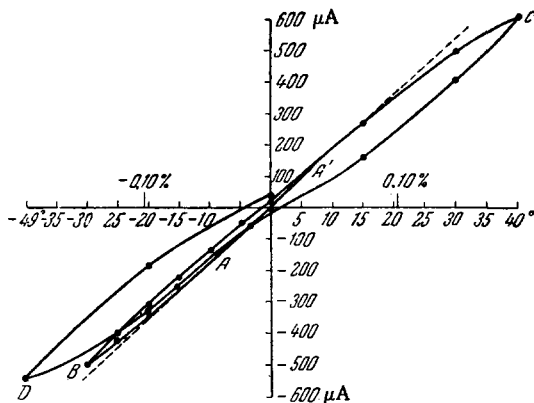


FIG. 8. Twisting of a square iron whisker (6.1μ sides). The ordinate represents the current in the deflection coils inducing twisting and is proportional to the twisting moment; the abscissa is the angle of twist, which is proportional to the deformation. Plastic deformation begins after the elastic region AA'.^[10]

elastic region AA'. The creep rate of silicon whiskers at room temperature was of the order of 0.01% per hour, which was greatly exceeded by the approximately 0.2% per hour rate in iron whiskers (at point C). No plastic deformation was observed in thin silicon whiskers having diameters smaller than 3μ . From the shape of the relation between the twisting moment and the twist deformation Eisner concludes that the investigated whiskers contained dislocations able to move even at room temperature, whereas in silicon whiskers they move only in an outer layer far from the crystal axis. This conclusion conflicts with earlier data for silicon whiskers,^[7] where no dislocation motion was observed at room temperature. From a study of the twisting of copper and iron whiskers Brenner^[9] concludes that the shape of the deformation curve did not result from the creep but from the effect of an oxide film on the crystal surface.

Whiskers possess high fatigue strength as well as high static strength. Eisner^[25] studied cantilever bending of copper whiskers with diameters under 3μ . The fatigue tests began with bends of from 0 to 1% deformation. Then, if plastic deformation had not

occurred (i.e., no kink appeared), the deformation was increased in steps of 0.3% and was maintained during 10^6 cycles at each step. This discontinuous loading procedure was used because there is a spread of the yield points of the whiskers. It was found that some whiskers remain elastic in bend tests up to 4% static deformation, corresponding to a stress of 470 kg/mm^2 for a whisker having its axis along $[110]$. Kinking occurs after a large number of cycles (up to 10^5) at the given deformation. When whiskers were tested statically and remained loaded during a time exceeding the total time of fatigue cycles, no kink appeared unless it was produced immediately after loading. This indicates that the dynamic strength is less than the static strength, i.e., the processes during fatigue tests induce plastic deformation of the whiskers.

Oding and Kop'ev^[26] have studied fatigue in copper and iron whiskers with diameters from 3 to 12μ . They tested two groups of whiskers, with high ($> 1\%$) and low ($\leq 0.1\%$) yield points, respectively, as determined by static bending. The whiskers were tested under a cyclical load up to 0.8% elastic deformation. The first (stronger) group withstood more than 10^7 cycles at 60 – 120 kg/mm^2 without kinking. The fatigue curve of the second group showed a fatigue limit at about 0.17% deformation.

3. Temperature and impurity effects. There have been many studies of the influence of temperature on the mechanical properties of whiskers. In the most detailed investigation silicon whiskers^[7] having diameters from 16 to 28μ were bent in the range from room temperature to 1000°C . At room temperature brittle fracture occurred under a mean stress of about 200 kg/mm^2 . Above 500°C plastic deformation occurred; the yield stress dropped to 50 kg/mm^2 at 800°C . The authors conclude that the high strength of silicon whiskers at room temperature does not indicate that they are perfect or dislocation-free. This was confirmed by direct experiments; dislocations were introduced into a silicon whisker (by plastic deformation at a higher temperature), which was then tested at room temperature without exhibiting any change in its strength. In addition, rods of the same dimensions as the whiskers (produced by gradual etching down of a large crystal) exhibited almost the same high strength. Therefore the whiskers are stronger than ordinary crystals because of their smaller dimensions, which reduce the number and influence of any defects that can cause stress concentrations. Dislocations, even if they are present, play no part, since they are pinned by oxygen and boron impurities. However, these conclusions regarding silicon cannot be extended to whiskers of other substances because of the special nature of such crystals as silicon. Peierls-Nabarro forces in silicon are probably much stronger than in ordinary metals. Also, a comparison of the yield stresses of whiskers and

rods shows that whenever dislocations play a part in plastic deformation and can move, i.e., at high temperatures, these two types of crystals do not exhibit identical behavior and the rods are far from possessing the same high strength as whiskers. For example, at 800°C the yield stress of whiskers is 50 kg/mm², while that of rods is the same as for ordinary silicon crystals, which is about 10 kg/mm². It should be noted that the difference between silicon rods and whiskers will possibly exist even for smaller diameters; in this case, as Gordon pointed out,^[14] silicon whiskers have considerably greater strength (up to 680 kg/mm²) at room temperature.

The preliminary data of Brenner^[15] for copper and iron whiskers show that some of these can withstand considerable elastic bending for a long time at temperatures up to 900°C. This indicates negligibly small creep at these temperatures, whereas other whiskers exhibited rapid plastic deformation above 350°C. The time preceding plastic deformation decreased as the temperature rose. The nature of the ambient was also of considerable importance; in a vacuum of from 10⁻⁵ to 10⁻⁶ mm Hg, kinking occurred less frequently than after heating in a quartz tube in the presence of hydrogen. Iron whiskers were also subjected to tensile tests in a stream of hydrogen up to 450°C. An oxide film was formed, producing interference colors but not affecting strength at room temperature. The whiskers were first deformed to about 1% at room temperature, in order to determine their strength, and were then further deformed at higher temperatures. Beginning at 150°C most of the whiskers fractured at very much lower stresses than those required at room temperature. Fracture occurred in a narrow localized region; the separate fragments then withstood a much higher temperature before breaking. All of this indicates a spread of the strength characteristics of different whiskers at high temperatures, similar to that existing at room temperature and caused by the defects that induce fracture.

The tensile strength of sapphire (Al₂O₃) whiskers was tested from room temperature to 2000°C as a function of time and of the ambient.^[21] In a short time of about 1 sec the average strength decreased almost linearly with temperature from 630 kg/mm² at 25°C to 105 kg/mm² at 1965°C. (At room temperature the greatest strength exhibited by a sapphire whisker is 1500 kg/mm².^[15]) Under static loads the strength was time-dependent, decreasing by approximately 10% for each order of magnitude of time. Brittle fracture occurred except at the very highest temperatures. The time elapsed before fracture is independent of the atmosphere (oxygen or hydrogen), so that the time dependence is not associated with the ambient. This strong temperature-and-time dependence of sapphire whisker strength cannot be accounted for by Griffith's theory of fracture through crack growth.

Silicon whiskers^[7] deformed at high temperatures before flow begins sometimes exhibit partial recovery of the yield point after aging for two hours at 800°C. The highest flow stress after such recovery was about half of the original yield stress. The authors of^[7] therefore conclude that impurities affect the plastic deformation of silicon whiskers. No recovery of the yield point has been observed in whiskers of other materials.^[15,19] Figure 9 shows the stress-strain curves of a copper whisker before and after annealing; no recovery is apparent.

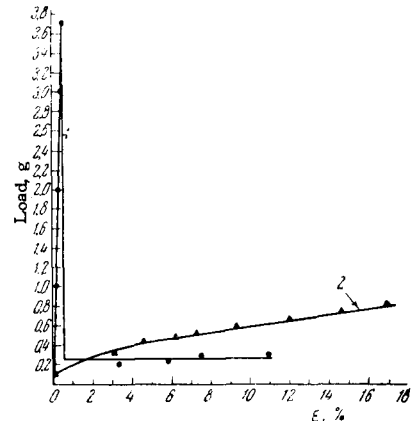


FIG. 9. Effect of annealing on a plastically deformed copper whisker. 1—Original stress-strain curve; 2—after one-hour anneal of plastically deformed crystal at 670°C.

Only iron, zinc, and cadmium whiskers have been tested at low temperatures. In iron whiskers tested at 77°K Coleman^[22] observed thin straight shear lines in {110} planes, twinning in {112} planes, and brittle fracture in {100} planes. Etching can reveal dislocations appearing after deformation at 77°K as well as at room temperature. Preliminary data^[23] are also available for the twisting of iron whiskers from room temperature down to 20°K; strong temperature dependence of the critical shear stress was observed.

Zinc and cadmium whiskers, which are plastic at room temperature, exhibit brittle fracture in the basal plane at low temperatures (for zinc below 160°K and for cadmium below 20°K).^[24] The breaking strength is almost twice as high as the yield stress at room temperature; the strength is thus very highly temperature-dependent.

Phase transitions have been observed at certain temperatures in whiskers of zinc sulfide, iron, cobalt, and titanium. In zinc sulfide whiskers^[33] the transition temperature rose by 350°C. In iron and cobalt whiskers^[15] the transition temperature was 910 and 410°C, respectively, as for ordinary crystals. In iron whiskers the α phase was superheated not more than 10–20°C. When silicon from the quartz heating tube entered the whiskers during the heating process the transition temperature was elevated by 200°C.

The phase transformation of iron is accompanied by the plastic deformation of the whiskers, some of which exhibit numerous kinks (Fig. 10). Following the transition the smooth surfaces of other whiskers having larger diameters become irregular. The whiskers



FIG. 10. Deformation of an iron whisker after a phase transition.^[15]

become polycrystalline, and after selective^[13] or thermal^[34] etching grain boundaries and mosaic and dendritic structures are visible. Only the thinnest iron whiskers, having diameters of about 0.1μ were not deformed in a phase transition.^[15] It was also shown that iron whiskers grown by vapor deposition above 950°C remain straight after cooling. It is suggested that these whiskers grow in a low-temperature b.c.c. structure.

Cobalt whiskers obtained by hydrogen reduction of cobalt bromide above the transition temperature usually contain two phases at room temperature,^[15] although whiskers having a pure hexagonal structure are also found. These whiskers remain straight and undeformed. The existence of two phases does not affect the strength; the elastic deformation of such two-phase cobalt whiskers attains 1%.

In the field-emission study of very thin whiskers (diameters $50\text{--}200\text{ \AA}$) of different substances grown directly in a field-electron emission microscope it was observed^[35] that iron and titanium whiskers exist at room temperature in both the high-temperature phase (f.c.c. iron and b.c.c. titanium) and in the usual low-temperature phase (b.c.c. iron and hexagonal titanium). Figure 11 shows field-electron emission patterns of iron and titanium whiskers in these phases at 300°K . With heating above the transition point the low-temperature phase is transformed into the high-temperature phase. The anomalous existence of high-temperature phases at low temperatures is probably a surface-energy effect, because of the high surface-to-volume ratio of these thin whiskers. Other peculiarities of these whiskers can exist, in addition to those

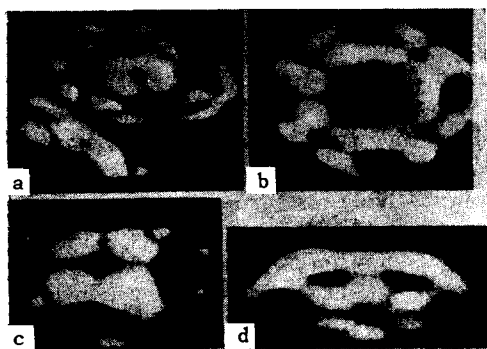


FIG. 11. Field-electron emission pattern of iron and titanium whiskers. a) b.c.c. Fe whisker with $[100]$ axis; b) f.c.c. Fe whisker with $[100]$ axis; c) hexag. Ti whisker with $[1000]$ axis; d) b.c.c. Ti whisker with $[110]$ axis.^[35]

noted in^[35], when compared with ordinary bulk crystals and with more usual whiskers having diameters of the order 1μ .

We have already noted the conclusion reached in^[7] regarding the influence of impurities on the mechanical properties of silicon whiskers. Indeed, the spectrographic analysis and the conductivity data for these whiskers indicate the existence of boron and oxygen impurities. Whiskers of other materials also are not usually of very high purity. For example, according to Brenner^[15] copper whiskers grown by copper iodide reduction contain about 0.003% silver, while iron whiskers grown by iron bromide reduction contain about 0.01% copper. A measurement of residual resistivity indicates approximately the same impurity content in zinc and cadmium whiskers grown by vapor deposition. By using purer original materials whiskers with a small impurity content can be grown. When the silver content in copper whiskers was reduced below $10^{-4}\%$ their strength was not affected.

In some experiments silver was introduced into copper whiskers^[15] either by heating at 750°C in a vacuum into which silver vapor is admitted, or by the addition of a silver halide to the original copper iodide from which the whiskers were grown. In two whiskers containing about 1 atom % silver introduced by the first method no change of strength was observed. The average strength of ten whiskers grown by the second method, containing a few percent of silver at about the solubility limit of silver in copper, exhibited $\frac{1}{3}$ of the strength of uncontaminated whiskers. The usual polygonal cross sections of these whiskers became circular. Therefore, impurities dissolved in copper whiskers do not affect their strength, but impurities present during growth can greatly reduce strength.

It was shown in^[79] that strong whiskers having large diameters up to about 1 mm can be produced by hydrogen reduction of a mixture of copper and iron chlorides. The structure of these whiskers was unusual; the central portion consisted of a single metal while the outer shell was a dense mechanical mixture of very small particles of both metals. The high strength of these whiskers can be attributed to these separate particles acting as obstacles to dislocation motion.

The effect of impurities introduced in order to change the growth processes of sodium and potassium chloride whiskers has been noted in^[3].

Impurities can thus sometimes diminish or even enhance whisker strength, but the high strength inherent in whiskers certainly does not depend essentially on impurities.

II. MAGNETIC AND ELECTRIC PROPERTIES OF WHISKERS

The magnetic properties of whiskers have been studied considerably more thoroughly than their elec-

tric properties. The favorable lateral surfaces and simple faceting of whiskers make them ideal objects for the study of simple domain structure and the kinetics of the movement of domain walls. This leads to new information about the magnetic properties of whiskers and about their surface states. The colloidal powder technique can be applied very simply to whiskers, and many other magnetic techniques have also been developed. Iron and cobalt whiskers are usually studied.

An ideal iron single crystal magnetized in the [100] direction should remain magnetized until an opposing magnetic field attains the theoretical coercive force, which is about 560 Oe at 25°C.^[36] In actuality, the coercive force for pure iron samples lies between 0.01 and 1 Oe. This discrepancy results from the nonuniform nucleation of domains around different surface defects, where local magnetic fields exceed the applied field. The homogeneous nucleation of a domain wall requires a strong magnetic field, but after its formation the wall can move in a very weak applied field under 0.008 Oe.

The investigation of domain wall motion in iron whiskers has shown^[37] that if we exclude the base from which the whiskers grow, where large distortions exist, we can attempt to observe a near-theoretical coercive force for individual whiskers. This was confirmed by coercive-force measurements on iron whiskers having different diameters and different degrees of perfection. It was found that the magnetic field required to reverse the magnetization of a given whisker region depends on the diameter and varies along the crystal (Fig. 12). The colloidal technique can be used to discover various defects reducing the coercive force. Figures 13 and 14 show domain nucleation around defects originating in growth (Fig. 13) or introduced artificially (Fig. 14). Most of the minima in Fig. 12 are associated with surface defects.

The coercive force decreases as whisker thickness increases; in the thickest whiskers the coercive force was that of an ordinary crystal. The highest value attained in thin whiskers was 480 Oe, which is only 15% under the theoretical value. Smaller values have been

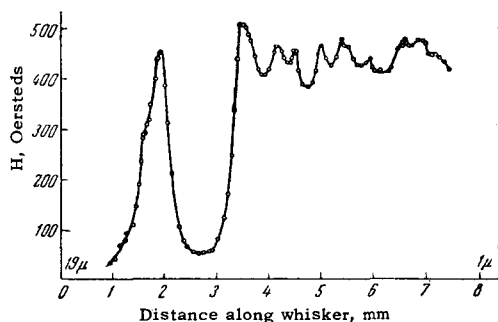


FIG. 12. Variation of the applied magnetic field required to reverse magnetization along an iron whisker (length 8.4 mm, base diameter 19 μ , tip diameter 1 μ).^[36]

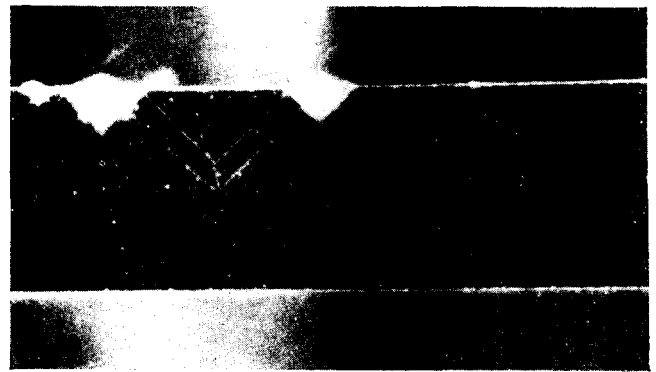
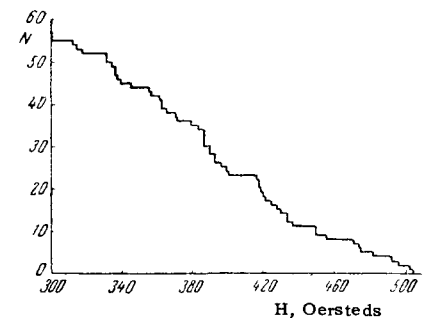


FIG. 13. Nucleation of domain walls around a surface defect arising during growth.^[15]



FIG. 14. Nucleation of domain walls around an artificially introduced defect.^[15]

FIG. 15. Number of tested iron whiskers containing regions where magnetic reversal occurred in magnetic fields greater than the indicated field.^[36]



observed much more frequently; for example, the result for 50 samples was 300 Oe (Fig. 15).

Because of the extremely high coercive force the velocity of domain wall motion along the whisker axis can attain 50 km/sec;^[37] because of domain nucleation at defects the highest velocity observed in ordinary samples is 0.5 km/sec. The changes of slope and discontinuities along the curve of domain wall motion vs magnetic field have not been accounted for.

Rodbell^[38,39] has studied ferromagnetic resonance in iron whiskers with [100] orientation at 9000 Mc in the temperature range from -196 to 850°C. Two resonance modes and a temperature dependence of resonance line width were discovered. The whiskers have a resonance line at least one order of magnitude narrower than has hitherto been observed in ordinary iron crystals. Since defects can induce disorder of the spin system accompanied by line broadening and at the given frequency the magnetic field penetrates the crystal to about 1000 Å, the resonance line width also depends on the perfection of the crystal surface.

The structure of domains formed in whiskers is very simple and is largely in accord with modern magnetic theory. Figure 16 shows domains in iron whiskers and their motion in a field.^[40] Domains in cobalt whiskers have been studied in^[41]; here the domain structure was found to differ from that in large samples. The dependence of domain width on whisker diameter obeys a $2/3$ -law rather than the theoretical $1/2$ -law predicted by Landau and Lifshitz and by Kittel.

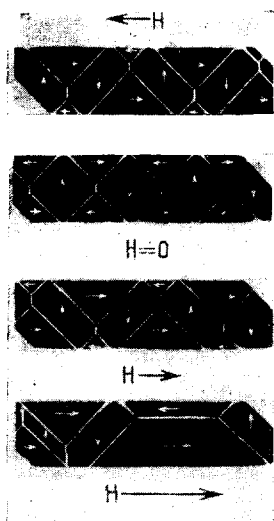


FIG. 16. Changes in the domain structure of an iron whisker in a magnetic field.^[40]

Besides the colloidal magnetite technique, the longitudinal magneto-optic Kerr effect has also been used to observe the domain structure of iron whiskers.^[42,43] The domain structure is preserved even in high magnetic fields up to 6000 Oe, the hitherto assumed saturation point. Above 2000 Oe the domains preserved at the whisker tip were magnetized perpendicular to the whisker axis on all faces. The domain width decreases as the field is enhanced. The investigators attribute these results to high localization of the magnetic field around the sharp angles and edges of the tip.

The regularities of the magnetic properties of whiskers are thus very similar in character to the variations of mechanical properties; this applies particularly to the spread in the magnitude of the coercive force and its dependence on whisker diameter and location in the whisker. However, the magnetic properties have a smaller spread than the strength, depend less on the diameter, and are in closer agreement with theory than the mechanical properties. These results are probably associated mainly with the fact that the effective size of a defect for the magnetic properties is about 1000 Å, but is considerably smaller for the mechanical properties. The smaller defects will clearly exist much more frequently. Also, whiskers containing large defects are more easily sorted out because of the optical imperfection of the whisker surfaces.

Experimental confirmation has recently been obtained^[44,45] for the quantum-mechanical prediction that electron interference fringes will be shifted due to magnetic flux in a magnetized iron whisker. A conical whisker tip changes the magnetic flux along the whisker; this tilts the fringes. For example, in the case of an iron whisker of 1μ diameter with tip narrowing of the order 10^{-3} the tilt of the fringes will be about 10° . Therefore the variation of the interference pattern along a whisker can indicate both the geometrical shape of the whisker and the magnetic flux in very small regions. The development of this method should furnish further details regarding whisker surfaces.

Shoenberg has observed the de Haas-van Alphen effect in copper whiskers;^[46] here the diamagnetic susceptibility oscillates as the magnetic field is varied. This effect was not previously observed in monovalent metals, and existed only in whiskers having a single orientation, with axes along [111].

The electric properties of whiskers, with the exception of resistivity, have not been studied. Resistivity at room temperature and at liquid helium temperatures have been measured in whiskers of zinc, cadmium, silver,^[47] copper,^[47,48] and iron,^[15] but the results are only provisional in character. The effect of large elastic deformation up to 2% on resistivity has been studied in copper, silver, iron, and zinc whiskers.^[49] The resistivity depended linearly on the deformation, although there were also some small second-order effects. The conductivity of tellurium whiskers at room temperature was found to be higher than in tellurium crystals with larger diameters (from 0.1 to 0.01 mm). In addition, tellurium whiskers exhibited a broader temperature region where a transition occurs from impurity conductivity to intrinsic conductivity.^[95]

III. SURFACE PROPERTIES AND STRUCTURE OF WHISKERS

1. Surface properties. The lateral surface of a whisker, according to the existing growth theory, should not contain regions that could serve as growth nuclei (i.e., steps, emerging dislocations etc.), since otherwise ordinary three-dimensional crystals would grow instead of whiskers. This means that, as a general rule, the surfaces of whiskers are more nearly perfect than those of ordinary whiskers. Microscopic examination reveals the smoothness of whisker surfaces. If the same criterion were applied to the surfaces of ordinary crystals as to those of whiskers, it is likely that no crystals would be acceptable for testing because of their poor surface state. The pliability of whiskers and the careful handling required because of their small size help to preserve the perfection of their surfaces after growth. This affects both the strength and surface properties of whiskers.

It has been found that the rates of dissolution, evaporation, and oxidation of whiskers are considerably slower than for ordinary crystals. Copper and silver whiskers produced by hydrogen reduction of their halides^[50] were found to dissolve considerably more slowly than solid metal in 40% nitric acid. The dissolution starts at the whisker tip. When a whisker is bent in the acid, rapid dissolution begins at the bend and leads finally to breaking at the same point.

Iron whiskers have been oxidized^[15] by heating up to 500°C in a stream of oxygen. Figure 17 shows the oxidation rate of a single whisker (the time dependence of the square of the oxidized layer thickness d). The oxidation rate is 50 times slower than for high-purity polycrystalline iron. It was found that oxidation often proceeds especially intensely at individual points, probably at defects (Fig. 18b).

The evaporation of whiskers also proceeds considerably more slowly. For example, hydroquinone whiskers, which ordinarily sublime at room temperature, were stored unchanged for several months in air.^[51] A reduced evaporation rate of methaldehyde whiskers has also been observed.^[52] The 7 μ diameter of a copper whisker heated to 1000°C did not change appreciably in 30 minutes,^[15] although, judging from the vapor pressure, it should have been diminished by 3 μ ; a copper wire heated together with the whisker evaporated much more rapidly.

Very interesting behavior of whiskers has been observed in electrodeposition.^[53] Thin copper whiskers having diameters less than 10 μ were immersed in an electrolyte and were connected to the cathode, as is always done in electroplating. Copper was not deposited on the whiskers until the overvoltage reached 1000 mV, whereas deposition on thick whiskers and

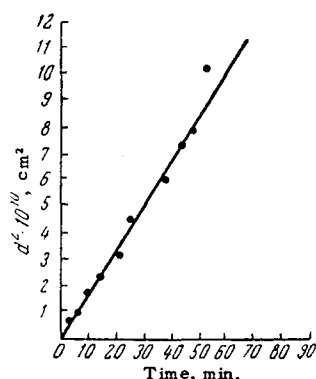


FIG. 17. Oxidation rate of iron whiskers at 500°C.^[15]



FIG. 18. Cross section of an oxidized iron whisker.^[15]

on a copper wire began at an overvoltage of about 10 mV. At higher overvoltages deposition began at separate points on the whisker surface with the formation of copper lumps rather than in a continuous layer. With a reversal of polarity in order to start the dissolution of copper these lumps are dissolved leaving pits; the whisker then breaks. Preferential dissolution beginning at the tip was not observed. This probably indicates that the sites where the lumps grew were imperfect.

The surfaces of growing crystals sometimes exhibit pits, overgrowth, spotty corrosion products etc. which can be observed in an optical microscope. Such crystals usually have lower strength and are customarily not tested. Defects outside the limit of resolution are smaller but have a decisive effect on whisker strength. Unfortunately, the character of these defects is almost unknown because direct investigations have not been performed. Price^[29,30] has found that these defects in very thin zinc crystals (less than 0.5 μ diameter) are growth steps and corrosion traces on the whisker surface. Surface defects can act as sources for the inhomogeneous generation of dislocations, initiating plastic deformation. The conditions of growth strongly influence the strength and surface state of whiskers. For example, the spread of strength values is always greater for whiskers produced by the reduction of metal halides having a large amount of impurities diffusing along the surface of a growing whisker than for whiskers produced by the vapor deposition of pure material. In a study of the growth and structure of iron whiskers Weik^[54,55] noted that the conditions of whisker production affect both the surface structure and strength.

It is interesting to determine how whisker strength is affected by surface films of oxides and other materials, since we know that surface films enhance strength.^[58] In air whiskers are probably entirely covered with an oxide film, with the exception of gold and ionic crystals. Films on iron^[56] and zinc^[30] whiskers have been observed experimentally. Since decreasing whisker diameter is accompanied by increasing strength due to the film, the strength of whiskers has been attributed to this effect.^[19]

Conflicting results have been obtained regarding the role of surface films. Weik^[55] covered iron whiskers with a nickel film about 100 atomic layers thick and observed approximate doubling of their strength (up to 300 kg/mm²). Cabrera and Price^[19] oxidized zinc whiskers by heating them in an oxygen atmosphere and found that their strength was doubled for an optimum oxide thickness of the order of tens of angstroms; whisker strength was reduced by further oxidation. Brenner^[15] heated iron whiskers until an oxide film was formed and produced interference colors, but detected no change of strength. The same negative result was obtained with silicon whiskers heated to 800°C.^[7]

If the presence of a film causes high strength, its absence should change the properties of whiskers. However, whiskers of gold^[57] and ionic crystals^[16] also possess high strength and properties identical with those of other whiskers. Webb and Stern^[59] showed by direct experiments that whisker strength is not affected by the removal of a film. An oxidized surface film was completely removed electrochemically from the surfaces of copper and silver whiskers. The bending strength was measured before and after removal of the film. It was found that the yield stress remains practically unchanged within the 30% spread of the measurements. No systematic increase or decrease of whisker strength was observed following the removal of the oxide films.

It is entirely possible that the influence of surface films on strength, as noted in^[19] and^[55], resulted from imperfection of the whiskers. This follows particularly from the low strength of the iron whiskers^[55] and from the presence of moving dislocations^[20] causing microcreep in the elastic region of the zinc whiskers. A film on these whiskers can interfere with plastic deformation and impede dislocations, thus increasing strength. A film can have no influence on very pure whiskers.

Surface-active substances such as mercury^[80] and a solution of oleic acid in vaseline oil do not affect the yield stress of zinc whiskers with diameters less than 10μ . The influence of these materials appears at a later stage of deformation and consists of changing the character of the deformation after plastic flow begins.*

Several properties of whisker surfaces are therefore essentially different from those of ordinary crystals as a result of the greater perfection of the former. This is reflected in their high strength.

2. Structure of whiskers. Following the first investigations of whiskers it was suggested that their extraordinary properties are associated in some way with high three-dimensional perfection, i.e., by the absence of dislocations or by a small number insufficient to induce multiplication.^[2] Small size itself results in a smaller probability of encountering defects; for example, if dislocations are uniformly distributed in the original crystal with a density of 10^6 dislocations per cm^2 they will be 10μ apart; a whisker with a diameter smaller than 10μ might therefore contain no dislocations. If this conclusion is correct, small crystals produced by etching down ordinary larger crystals should have the same properties as whiskers, but this has been disproven experimentally.^[7] It is more likely that whiskers are more nearly perfect than ordinary crystals because of certain peculiarities of their growth process.

It is also not clear what number of dislocations will make a whisker lose its strength. It is frequently as-

sumed that the presence of only a few dislocations will reduce crystal strength drastically from its theoretical level to the observed low yield stress. In this case a whisker should contain not a single dislocation in order to have the observed high strength.

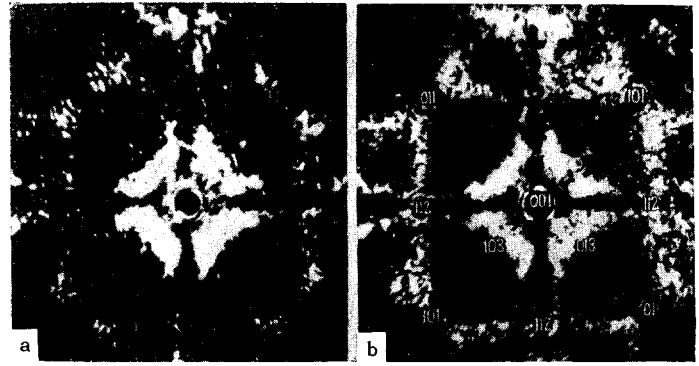
We shall now review some of the principal facts concerning the structure of whiskers. According to the theory of whisker growth a screw dislocation is to be found along the whisker axis. After Eshelby showed^[60] that the axial twist of the whisker lattice is more pronounced for whiskers with smaller radii, whiskers of more than ten materials were subjected to x-ray investigation.^[61-63] Twisting of the lattice was found only in whiskers of sapphire, palladium, tin, and sodium chloride. In addition to this twisting corresponding to the Burgers vector of a single dislocation in the growth direction, very large twisting is sometimes observed corresponding to the total Burgers vector (about 100 Å). Some whiskers of silver,^[72] copper, palladium,^[73] potassium bromide,^[74] and sodium chloride^[75] grow in the form of spirals corresponding externally to Eshelby twisting. The Burgers vector of the corresponding dislocations can be computed from the geometry of the spiral and is also found to be very large.^[73,75] However, the x-ray investigation of some whiskers showed^[62] that this external form is not necessarily associated with axial twisting of the lattice and can be attributed to some characteristics of growth. No twisting of the lattice was detected in whiskers of the other materials, but this does not necessarily indicate that such whiskers contain no axial dislocation. It is possible that two dislocations (or another even number) with like signs are directed along the axis; the result would then be zero twist. A study of whisker structure by the x-ray diffraction microscopy technique of Lang^[64] has shown that sodium chloride whiskers^[62] often contain dislocations parallel to the axis, as is predicted by the growth theory. Figure 19 shows a sodium chloride whisker containing two dislocations along its axis.^[78] A more detailed study has shown that in the thinnest NaCl whiskers there are no axial dislocations. A similar result was obtained from the study of NaCl whiskers decorated with gold.^[66] Different techniques have thus been used to show that crystals having diameters greater than 10μ often contain one or more axial dislocations of growth. These same techniques do not lead to a clear answer in the case of thinner whiskers because of their low resolution (of the order of 5μ).



FIG. 19. X-ray diffraction pattern of a sodium chloride whisker containing two axial dislocations. (200) reflection. 100 \times magnification.^[78]

*The absence of the Rebinder effect in whiskers can be evidence not only of surface perfection but also of the absence of dislocations or their motion below the yield point.^[80]

FIG. 20. Field emission pattern of the tip of an iron whisker having two axial screw dislocations. a) Original crystal; b) after evaporation of 20 atomic surface layers and annihilation of dislocations.^[70]



The structure of whiskers must therefore be studied with higher resolution. Field-emission microscopes have been used to study the structure of a whisker tip down to atomic resolution.^[67] By applying an electric field it is possible to remove successive layers from the tip in order to study the bulk structure of the crystal. The first work of this kind was done by Gomer,^[68] who observed the field-electron emission patterns of many whiskers. Mercury whiskers were studied in the greatest detail. Through the application of an electric field that extends the whiskers, rotation of the emission pattern was observed, in accordance with Eshelby's calculations.^[60] Gomer therefore concluded that even the thinnest mercury whiskers contain an axial screw dislocation. However, a more precise calculation^[69] revealed extreme disagreement with Gomer's values for the rotation rate. Müller^[70] used a helium field-ion microscope, giving the highest resolution hitherto attained, to produce emission patterns of iron whiskers at 21°K. Figure 20a shows one of these patterns for a whisker with its axis along [100]; the white dots correspond to individual atoms and a configuration resembling two horseshoes is seen in the center. Müller suggests that two screw dislocations of opposite signs are located very close to each other, with a separation of about 100 Å. After the application of an electric field corresponding to a stress of about 1000 kg/mm² and the removal of about 20 layers of iron atoms, the dislocations were annihilated, probably through climb (Fig. 20b).

The question regarding the existence of axial growth dislocations in whiskers has still not been answered, at least for whiskers with radii of about 10μ. It has been shown theoretically^[71] that in very thin whiskers the axial screw dislocation is unstable and can emerge on the whisker surface at the growth temperature. For example, this is possible in copper whiskers with diameters under 0.01μ. Indeed, a study of zinc whiskers in an electron transmission microscope^[29] failed to detect axial growth dislocations.

Whiskers, as perfect crystals, should contain no dislocations other than one or a few axial growth dislocations, which should not affect their mechanical properties. Investigations have shown, however, that the situation is considerably more complicated. Just

as in the case of the axial dislocation, the structure of whiskers depends essentially on their diameter.

Whiskers having diameters of about 50μ can be studied by means of the etch-pit technique, as has been done by Coleman^[76] for iron whiskers and by Amelinckx^[66] for sodium chloride whiskers. However, whiskers of such large diameters possess low strength, so that their structure is not typical of "true" whiskers; on the other hand, it is difficult to investigate whiskers of smaller diameters. Gorsuch^[77] used an x-ray double-crystal spectrometer to measure the density and distribution of dislocations in the tips of iron whiskers. Many whiskers having diameters under 10μ were found to be perfect, containing no dislocations. The width of the rocking curve of these whiskers did not exceed 50 sec of arc. Tilt and twist boundaries were found in some of the whiskers.

Amelinckx^[66] decorated sodium chloride whiskers with gold, as is done with normal bulk crystals. Some whiskers were found to be decorated uniformly throughout their entire volume, thus indicating that they contain a large number of dislocations. In other whiskers a decorated structure could be distinguished, and one or more axial dislocations and complex dislocation networks were observed. The thinnest whiskers remained entirely undecorated, which would indicate the absence of dislocations. Amelinckx believes, however, that this may simply indicate that the given technique is unsuitable for thin whiskers.

Webb^[65] has made a detailed investigation of the structure of sodium chloride whiskers using the etch-pit technique, x-ray measurements of lattice twist, and x-ray diffraction microscopy. The greatest merit of his work lies in the fact that he compared the structure with the bending strength of the whiskers. All of these whiskers can be divided into three groups. The thinnest whiskers, having cross-sectional areas less than 100μ², exhibited high strength up to 1% elastic deformation and their fracture often occurred without appreciable plastic deformation. These whiskers contained only one or two straight axial dislocations traversing the entire crystal. These dislocations were of the mixed type, usually having the Burgers vector $a/2 [110]$. The largest sodium chloride whiskers with cross-sectional areas greater than 1000μ² were weak;

their elastic limit did not exceed 0.01%, and they exhibited considerable plastic deformation before breaking. The dislocation structure was complicated, with the frequent occurrence of dislocations having a large number of branchings and intersections. The whiskers with intermediate cross sections varied in strength. Low strength was observed in the case of whiskers with complicated dislocation structure or with more than very few straight axial dislocations, except when such axial dislocations were close to the whisker axis. (In the latter case they cannot affect the bending strength, which is therefore often observed to be somewhat greater than the tensile strength of whiskers.) Whiskers having a simple structure (with one or two axial dislocations) were strong, like the thin whiskers in the first group.

A direct relationship therefore exists between the dislocation structure of whiskers and their strength; the number and locations of dislocations determine whisker strength. However, certain results obtained in the same work cannot be understood completely. It appears that whiskers containing one or two straight axial dislocations are just as strong as others containing no dislocations, and it is entirely unclear why five or ten such dislocations will greatly reduce strength. This can possibly be accounted for by the shortcomings of the employed techniques, since x-ray diffraction only resolves dislocations at least 5μ apart. It is entirely possible that the presence of a few dislocations results in their intersection, curving, and the formation of jogs without detection by this technique but inducing plastic deformation under low stresses. Whisker structure must be investigated by improved techniques.

The best existing technique for studying dislocation structure is transmission electron microscopy,^[31] which permits the observation of dislocations and their motion under stresses using very high magnification. A disadvantage is the necessity of using thin foils not more than 0.5μ thick, so that whiskers of the normal sizes cannot be studied. As already mentioned, Price studied the structure of very thin zinc whiskers and platelets not more than 0.5μ thick. He found that these whiskers contain no dislocations if handled with sufficient care to avoid introducing dislocations. Plastic deformation of these whiskers was always initiated by stress concentrations around surface defects or cemented areas, leading to the creation of a narrow zone of plastic deformation^[29] or a wedge-shaped twin.^[30] The whisker strength decreased for larger surface areas, probably because of the higher probability of surface defects.

The foregoing technique can unfortunately not be applied to whiskers having large diameters; therefore the relation between the strength and structure of the latter has not been determined completely. The existing data regarding the strength of whiskers, the spread of strength values, and the dependence on diameter can only be accounted for by such a relationship. These data show that whiskers contain a certain number of

defects which cause varying degrees of damage, inducing flow or fracture. These defects are located either in the interior (structural defects) or on the surfaces of whiskers. This is shown clearly by experiments on the retesting of a copper whisker after removal of the portion where flow began (i.e., of the most harmful defect), after which the yield stress was doubled.^[18] Several investigators later confirmed these results for whiskers of different materials.

3. Strength of whiskers and normal crystals. Despite the meager data available regarding the relation between whisker structure and strength, it can be concluded that whisker strength is related to their bulk and surface perfection. Bulk perfection means the complete absence of dislocations or the presence of only a small number. Surface perfection means the absence or low effectiveness of different kinds of stress concentrators (surface defects). A combination of both factors results in the extremely high strength, equal to the theoretical strength, of some individual whiskers. The spread of strength values is due to random departures from perfection; the increase of strength with diminishing diameter results from the smaller probability of encountering defects as the whisker surface is reduced.

The presence of a few dislocations will not necessarily impair the strength of a whisker. For example, one or two axial dislocations^[65] can be present without participating in plastic deformation during tensile or bending tests. Gliding dislocations that moved during the microcreep of zinc whiskers^[19] did not reduce their strength; on the contrary, the whiskers were afterwards usually found to be stronger. In normal crystals the motion of dislocation loops on glide planes results in dislocation multiplication and macroscopic plastic deformation.^[89]

The foregoing differences in the behavior of dislocations can be associated with the peculiarities of dislocation structure in whiskers. It is known that the most important causes of dislocation multiplication are jogs (the cross-slip mechanism), the restraining of dislocations (mechanisms associated with the creation of stress concentrations around dislocation clusters) and the pinning of dislocations (the Frank-Read mechanism). Because of the low dislocation density and high homogeneity of whiskers we can conclude that in small-diameter whiskers these multiplication mechanisms are unimportant; for example, dislocations in whiskers have few jogs, do not intersect etc. On the other hand more importance attaches to surface defects, which serve as stress concentrators and induce the inhomogeneous generation of dislocations. The observation of the onset of plastic deformation in very thin zinc whiskers by transmission electron microscopy^[29,30] has shown that the first traces of plastic deformation always occurred near stress concentrations (surface defects, growth steps, oxidized regions) or near clamps.

In ordinary large crystals (probably in all except silicon crystals at room temperature) surface defects, which are stress concentrators, play a less important role, since there are enough other causes of dislocation movement and multiplication. Orowan^[82] has collected a large amount of data showing that the yield stress of plastic materials is not determined by the presence of surface defects. Almost no success was achieved in raising the yield stress of metals by removing surface defects. This is probably accounted for by the fact that bulk defects continue to be effective after the removal of surface defects. This is not contradicted by the abundant experimental data showing the important influence of the surface on the character of dislocation motion, obstruction, and generation.^[81]

Surface defects should be important in two cases: when the whiskers are dislocation-free, or when they are perfectly brittle (which means that no plastic deformation occurs both as shown by the stress-strain curve and by the microscopic observation of dislocation motion). Both conditions have been attained thus far only in single crystals of silicon and germanium. At room temperature the dislocations in these crystals are motionless. The development of the single-crystal growing technique and the analysis of dislocation sources during crystal growth have led to the production of large dislocation-free crystals of silicon and germanium.^[83]

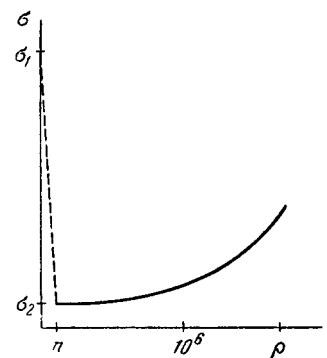
It has been found that, independently of the existence of dislocations, following careful surface treatment (mechanical and chemical polishing) silicon whiskers of up to 0.5 mm diameter have been bent elastically about 2% at room temperature (at approximately 350 kg/mm²) without fracture.^[84] Also, above about 500°C, when dislocations can begin to move in silicon, plastic deformation begins under low stresses if the crystal contains dislocations even if the surface is as perfect as previously. In this case the yield stress does not exceed 1 kg/mm² at 900°C. If there are no dislocations, the whisker is considerably stronger, withstanding above 20 kg/mm² without plastic deformation at the same temperature. Poor surface treatment of dislocation-free crystals or the intentional introduction of surface defects immediately lowered the yield point, probably because of the introduced dislocations. At this temperature^[7] silicon whiskers exhibit a yield stress of about 45 kg/mm²; the difference by a factor of more than two can be attributed to the smaller whisker surface and therefore the smaller probability of encountering an effective surface defect.

It has been possible to isolate regions containing no dislocations or dislocation sources in lithium fluoride crystals having a low dislocation density.^[85] Pressure on these regions with a thoroughly cleaned glass ball produced stresses of about 75 kg/mm² without plastic deformation or the appearance of dislocations.

All the aforementioned cases of high strength, including whiskers, are associated with high degrees of perfection. By an opposite procedure crystal imperfection is enhanced in a definite manner. In this way obstacles to dislocation motion are created, thus elevating the yield stress but without generating microcracks or premature embrittlement. The increase in the number of dislocations and the creation of complex dislocation configurations, the breaking up of subgrains and their misorientation by different kinds of thermal and mechanical treatments, the accumulation of vacancies and impurity atoms around dislocations, the decomposition of supersaturated solid solutions with the disperse precipitation of the second phase—all of these represent an incomplete and very schematic list of the factors that cause the obstruction of dislocation motion in crystals.^[86] Thin metallic films produced by vapor deposition have high strength.^[87] This results from a high concentration (up to 10¹¹ per cm²) of dislocations and other defects formed by quenching during deposition, which greatly impede dislocation motion and multiplication.

The experimental data obtained from the study of these two extreme cases of structural perfection have provided somewhat more precise knowledge regarding the relation between the shear strength σ and dislocation density ρ (Fig. 21). (Only active dislocations are considered.) The strength of a perfect crystal where $\rho < n$ (n is one or a few times unity) equals the theoretical strength multiplied by a coefficient $k < 1$ allowing for a surface effect. In the absence of surface defects producing a stress concentration we have $k = 1$. When $\rho = n$ the strength decreases to σ_2 , the stress required for the motion of separate dislocations; in plastic metallic crystals this stress is extremely small,^[88] probably less than 1 g/mm². Increasing dislocation density (above about 10⁶–10⁸ per cm²) is accompanied by growing strength. Thus the two extreme points on the curve in Fig. 21 correspond to two possible ways of attaining high strength in crystals.

FIG. 21. Breaking strength σ as a function of dislocation density ρ . n is of the order of one or a few times unity. $\sigma_1 = k\sigma_{\text{theor}}$, where $k < 1$ is a coefficient representing the surface effect; σ_2 is the stress required to overcome the resistance to the motion of separate dislocations.



CONCLUSION

The data now available permit a more definite explanation of the extraordinary properties of whisk-

ers.^[5] Two possible approaches to the study of whiskers must be clearly distinguished. On the one hand, whiskers, being perfect or nearly perfect crystals, make it possible to study the properties of an ideal lattice undergoing large elastic deformation, under high mechanical stresses, with rapidly moving domain walls etc. This is especially important for structure-sensitive properties. On the other hand, the study of instances in which whisker properties are not extraordinary makes it possible to understand why this occurred in each given case, i.e., to account for the low strength of ordinary crystals. This means that relatively weak, as well as strong, whiskers must be studied. Investigators of crystals generally have a very subjective point of view and use different criteria (such as shape, surface state etc.) to select only strong whiskers, while completely ignoring the second approach. The causes of low whisker strength are seldom studied although this is a very important topic.

The investigation of whiskers can be used to indicate the most general conditions under which ordinary crystals can also possess a high degree of strength. For example, a brittle crystal must have a perfect surface, while a plastic crystal must also contain few or no dislocations. Of course this does not preclude high strength resulting from the impeding of dislocation movement.

The investigation of whiskers is seen to be of great scientific and practical importance for understanding the nature of strength and plasticity. On the other hand, direct technical applications are also possible. It has been suggested that germanium whiskers can be used in highly sensitive strain gauges.^[90] It is more difficult to make use of the high strength of whiskers because of the small size of those produced at the present time; the techniques used in growing whiskers must be improved. More practical suggestions^[91,92] envision their use as structural materials prepared either by compression and sintering, as in powder metallurgy, with the preservation of their high strength, or by the use of a binder (resembling plastic-impregnated fibred glass). Work in this direction has already been started.^[93,94]

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