NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL MEMORANDUM

No. 1072

THE SURFACE STRUCTURE OF GROUND METAL CRYSTALS

By W. Boas and E. Schmid

Naturwissenschaften
Vol. 20, Nos. 22/24, May 27, 1932

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL MEMORANDUM NO. 1072

THE SURFACE STRUCTURE OF GROUND METAL CRYSTALS*

By W. Boas and E. Schmid

The changes produced on metallic surfaces as a result of grinding and polishing are not as yet fully understood. Undoubtedly there is some more or less marked change in the crystal structure, at least, in the top layer. Hereby a diffusion of separated crystal particles may be involved, or, on plastic material, the formation of a layer in greatly deformed state, with possible recrystallization in certain conditions. Czochralski verified the existence of such a layer on tin micro-sections by successive observations of the texture after repeated etching (reference 1); while Thomassen established, roentgenographically by means of the Debye-Scherrer method, the existence of diffused crystal fractions on the surface of ground and polished tin bars, which he had already observed after turning (on the lathe). (Thickness of this layer ~ 0.07 mm) (See reference 2.) Whether this layer borders direct on the undamaged base material or whether deformed intermediate layers form the transition, nothing is known. One observation by Sachs and Shoji (reference 3) simply states that after the turning of an α-brass crystal the disturbance starting from the surface, penetrates fairly deep (~ 1 mm) into the crystal (proof by recrystallization at 750°).

For a systematic investigation of the grinding effect the roentgenographic method, briefly described in the following, seemed suitable. The method consists of the recording of Laue-reflection diagrams with X-rays of specially long wave lengths - that is, X-rays suitable for the study of the structure of thin sections. (See reference 4.) If, in addition, diffractions lie at great angles of deviation for the characteristic radiation of the employed anticathode, the

---

same arrangement makes it possible to detect fine-grained
layers or such with ordered texture by the appearance of
Debye-Scherrer concentric rings or layer lines. In this
manner the grinding effect can be followed by a series of
records after etching with varying degree of severity.

The metals used in the present study included aluminum,
copper, tin, cadmium, and antimony. The crystals appeared
in form of cylindrical bars of about 4 millimeters diameter.
First, a Laue reflection diagram of each of the explored
crystals was taken, and the crystal then ground as nearly as
possible perpendicular to the employed ray direction. The
little bars were imbedded in Picein wax and ground about 0.5
millimeter parallel to the longitudinal axis on a rotating
disk (emery paper No. 2, then reground with No. 1G), and
then polished with alumina No. 2 or 1 on a slowly rotating
disk. The X-rays were obtained from commercial tubes with
Cu and Mo anticathode (Müller-Metalix tubes (exposure:
4 hr at 24 kv effective and 20 ma)). After etching the spec-
imens were carefully returned to the same position, with the
cut surface perpendicular to the incident ray, before the
tube. The distance of the specimen from the perforated film
pack was 35 millimeters. Before discussing the obtained di-
agrams, the possible dispositions of diffraction lines on
the refraction diagrams for the simplest cases are briefly
represented:

1. <u>Unoriented multicrystal</u>.- The heterochromatic radia-
tion results in a general blackening, the radiation typical
of the appearance of evenly blackened Debye-Scherrer rings,
which, owing to the great angles of deviation, usually ex-
hibit a cleavage (K_α-doublet).

2. <u>Monocrystal</u>.- The heterochromatic radiation yields a
Laue reflection diagram on which the diffractions, emanating
from planes of one zone, lie on a hyperbola branch or on a
straight line passing through the optical center. The char-
acteristic radiation yields, in the general case, no diffrac-
tion lines on the static crystal.

3. <u>Crystal aggregate with simple fiber texture and axis
of fiber perpendicular to the ray</u>.- The heterochromatic radi-
ation yields diffused curves, resulting in the repeatedly
described "radial diagrams." (See reference 5.) The charac-
teristic radiation yields the Polany layer lines. Whereas
the blackening on the diffused strips is produced by diffrac-
tion of polychromatic Roentgen rays on the same plane, the
diffractions on the layer lines stem from different planes

but identical wave length. In the equator, zone circle, layer line, and diffused heterochromatic radiation band coincide, and even in its proximity (~20° vertical angle) the different curves are still so closely adjacent that here also a separation encounters difficulties.

In figure 1 are reproduced several of the diagrams as obtained on a tin crystal ground approximately parallel to the base surface after variously deep etching. The record obtained on the undamaged original crystal merely showed the sextuple arrangement of the Laue spots due to the ray direction and resembled figure 1(e) very closely. After grinding, the Laue diffraction lines disappear and two distinct Debye-Scherrer rings (relating to planes (1015) and (1232)) appear. (See fig. 1(a).) Blackened maximums are plainly visible at various points of the rings. Polishing the ground surface does not change the character of the reflection diagram. (See fig. 1(b).) On etching the polished cut 0.015 millimeter, the concentric rings disappear, and the previously merely suggestive blackened maximums appear as heavy monochromatic diffractions which can be combined with the layer lines (fig. 1(c). Further etching of the ground surface causes, at first, little change. Slowly the intensity of individual diffraction spots decreases considerably (fig. 1(d)), and, after disappearance of the blackened areas due to the Cu radiation, the Laue reflection pattern of the original crystal emerges again. (See fig. 1(e).)

One seemingly feasible interpretation of the secured diagrams is as follows: at the surface of the crystal cut there is a fine-grained layer, in which - recognizable on the dissolution of the Debye-Scherrer circles in single diffractions - recrystallization has taken place. This layer, 0.015 millimeter, at the most, in thickness, is followed by a zone in which different lattice positions exist simultaneously, which is indicative of a deformation. Measurement of the vertical angle of the layer lines (50 mm) yielded here for the identity period in the grinding direction a value of 2.6 Å, which corresponds with the atom distance on a diagonal axis of type I. The absence of diffused heterochromatic radiation curves, moreover, points to the fact that rotations of the base about the grinding direction are present only in small measure. The present orientation multiplicity thus obviously varies but little from the orientation of the original crystal. The thickness of this transition zone, which represents the connection between the fine-grained surface layer and the undamaged base crystal, is about 0.16 millimeter. In contrast to the very abrupt transition in the

surface layer, the change-over in the base crystal is largely progressive.

All the other test series confirmed the existence of the fine-grained surface layer and of the disturbed intermediate layer in ground metal crystals. Tin crystals ground parallel to the hexagonal axis manifest differences in respect to the crystal ground perpendicular to the hexagonal axis to the extent that the thickness of the layers appears to be perceptibly greater. It required a 0.030-millimeter etching to make the Debye-Scherrer circles for the surface zone disappear; a 0.015-millimeter etching left them plainly visible (cf. fig. 2a). The diffusions caused by heterochromatic radiation diffraction (cf. fig. 2b) is, further, suggestive of a very much greater multiplicity of positions than in the first discussed example. The tests with tin therefore clearly point to an influence of the crystal orientation on the grinding effect. By parallel position of the translation elements to the plane and direction of grinding, the disturbances introduced by grinding are manifestly very small. As to the existence and type of a grinding texture possibly occurring in the multicrystal, the tests so far, in which the grinding direction always coincided with the direction of translation, permit of no conclusions.

The grinding surface of the analyzed cadmium crystal was by no means as simply oriented as on the tin specimen. The fine crystalline surface layer was evidenced by profound general blackening and complete absence of diffraction spots (Mo-anticathode). In consequence, the thickness of the layer could be ascertained only by the reappearance of Laue spots which emerged after 0.020-millimeter etching with about half intensity. Accordingly, 0.020 plus half the half-value thickness (for ~ I Å) can be regarded as approximate indication of the layer thickness (table 1). (See reference 6.) The deformation of the intermediate layer shows particularly plain through the heavy diffusion of the diffractions of figure 3a. (The close agreement between zone hyperbola and diffused heterochromatic radiation curve is clearly evident. The differences in direction are recognizable by careful examination.) It requires a 0.54-millimeter etching to bring out the undisturbed Laue reflection diagram of the original crystal again. (See figure 3b.)

TABLE 1

HALF-VALUE THICKNESS
(in mm)

λ in $\overset{\circ}{A}$	Al	Cu	Zn	Cd	Sb
0.5	1.3	0.040	0.046	0.074	0.081
1.0	.17	.0059	.0067	.011	.012
1.5	.056	.016	.018	.0037	.0040
2.0	.025	.0071	.0081	.0018	.0020

On the antimony crystal the Laue diffraction lines of the base crystal have not completely disappeared after grinding, but they have after polishing. The thickness of the surface layer was likewise estimated from the re-appearance of the Laue diffractions and the half-value thickness, because of the absence of Debye-Scherrer rings.

On a ground copper crystal the thickness of the fine-grained top layer could be directly determined again from the disappearance of the Debye-Scherrer rings of the (204) and (313) planes. A recrystallization of the surface layer, such as observed on tin, does obviously not occur. The utter diffusion of the K_α-doublet points to marked deformations. (See fig. 5.)

On aluminum, the fine crystalline surface layer produced by grinding, is also manifested by the Debye-Scherrer rings of plane (511) or (333), and (422). (See fig. 6.) No recrystallization was observed; the K_α-doublet is plainly split, which, however, on aluminum, should not be interpreted as an absence of deformations in the grains. In consequence of the low absorption of the material the Laue diagram of the base crystal is also recognizable at the same time. Surprisingly, this diagram hardly permits of an inference of transition zone. Etching the ground surface yields, other than the disappearance of the Debye-Scherrer rings and intensification of the Laue spots, no changes in the diagram worth mentioning. A subsequent test with very small etching stages yielded a thickness of 0.015 millimeter of fine-grained surface layer, from the disappearance of the Debye-Scherrer rings. From

the disappearance of the slight diffusion of the Laue spots, it may be inferred here that the thickness of the deformed layer is approximately twice as great.

Summing up, it may be stated that the grinding and polishing of metal crystals results in the formation of two layers in the metal: a fine crystalline thin surface layer and a deformed transition layer about 10 times as thick, which establishes the connection with the undamaged base crystal. The data in table 2 give an approximate idea of the thickness of the layers.

TABLE 2

STRATIFICATION RESULTING FROM GRINDING METAL CRYSTALS

Metal		Thickness of fine crystallized surface layer (mm)	Thickness of deformed intermediate layer (mm)
Al		0.015	~0.03
Cu		.005	~.02
Zn	∥ base	.015	.16
	⊥ base	.030	.30
Cd		.025	.46
Sb		.020	>.10

A noticeable effect on the layer thickness appears to be attributable to the crystal orientation, which is plausible in view of its significance for the transportation possibilities. The differences in the translation possibilities might also be responsible for the very much shallower depth of penetration of the disturbances on the cubic metals.

It suggests itself to extend these tests to include multi-crystalline base material – and also other chip-raising processes – so that the question regarding the existence of textures in the outer layers of the piece of the metal could be treated along with it. (Addendum by correspondence:

R. C. French (reference 7) recently investigated the changes
induced by grinding and polishing by means of high-speed
electrons. The grinding of a copper block brought out a
widening of the diffraction circles; after polishing, the
original diffraction rings had disappeared and two wide rings
appeared at a different spot. French interprets this as a
grain destruction, or, in support of Beilby's theory, as a
transition in an unoriented state. Against this last inter-
pretation, however, F. Kirchner (reference 8) objects.)

Translation by J. Vanier,
National Advisory Committee
for Aeronautics.

REFERENCES

1. Czochralski, J.: Metall u. Erz, 13, 381, 1916.

2. Thomassen, L.: Z. Metallkunde, 15, 306, 1923.

3. Sachs, G., and Shoji, H.: Z. d. Physik, 45, 776, 1927.

4. Boas, W., and Schmid, E.: Metallwirtschraft, 10, 917,
      1931. (Prof. Ewald reminds of the E. Keller (Annalen
      d. Physik, 46, 13, 1915) discussion of Laue reflection
      diagrams on diamonds.)

   Regler, F.: Z. d. Physik, 74, 547, 1932.

5. Hupka, E.: Z. d. Physik, 14, 623, 1913.

   Nishikawa and Ashara: Phys. Rev., 15, 38, 1920,
      and particularly

   Polanyi, M.: Z. d. Physik, 7, 149, 1921. For position
      of plate between X-ray tube and specimen, see

   Regler, F.: Z. d. Physik, 71, 371, 1931.

6. Siegbahn, M.: Spectroscopy of X-Rays. 2d ed., 1931.

7. French, R. C.: Nature, 129, 169, 1932.

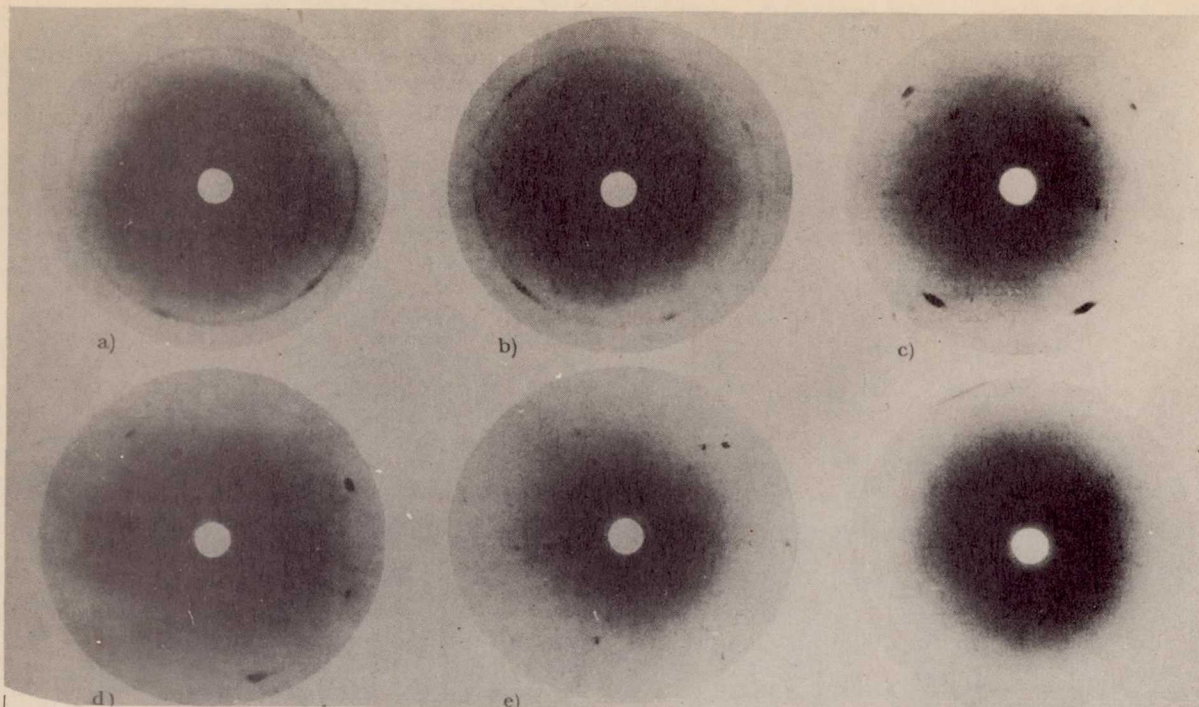8. Kirchner, F.: Nature, 129, 545, 1932.

Figure 1 (a to e).- Tin crystal-plane of slide $\infty\|(0001)$. Grinding direction $\infty\|(1120)$. Cu-anticathode: a) ground, b) polished, c) .015 mm etching, d) .065 mm etching, e) .17 mm etching.
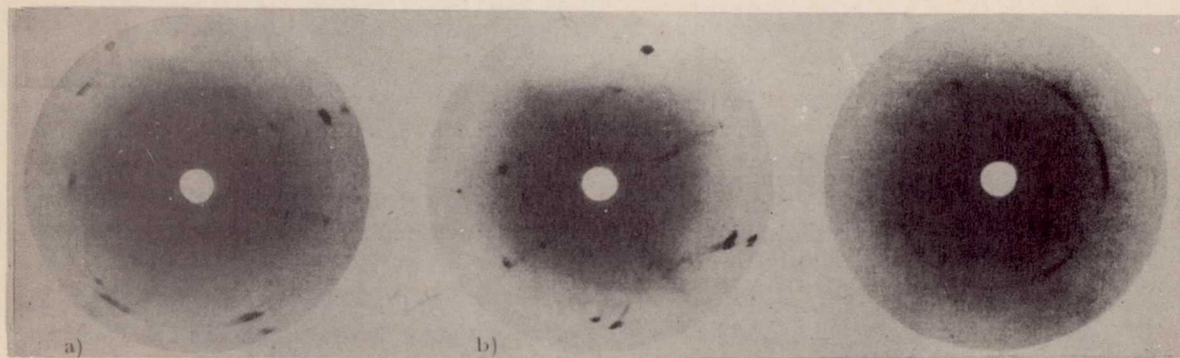
Figure 4.- Antimony crystal, ground (Mo-anticathode).

Figure 2 (a and b).- Tin crystal-plane of slide $\infty\perp(0001)$. Grinding direction $\infty\|(1120)$. a) etched .015 mm, b) etched .065 mm.

Figure 5.- Copper crystal, ground (Cu-anticathode).

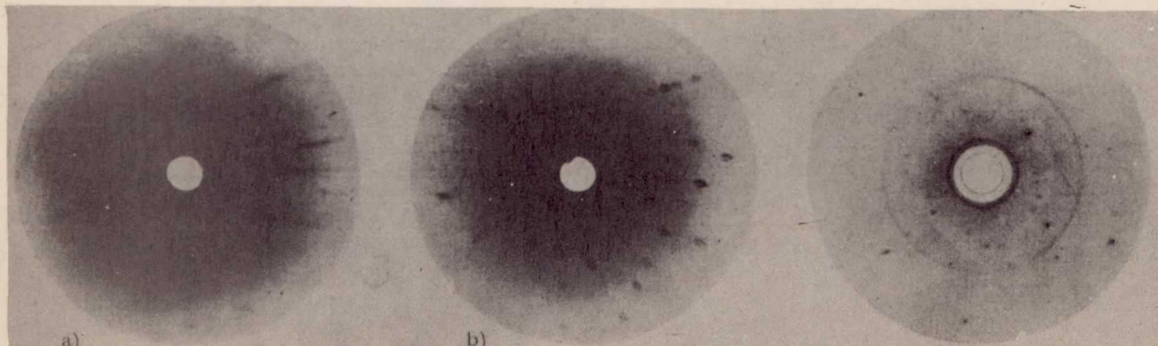Figure 3 (a and b).- Cadmium crystal (Mo-anticathode) a) etched .060 mm, b) .54 mm.

Figure 6.- Aluminum crystal, ground (Cu-anticathode).