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Magnetic Domain Patterns on Single-Crystal Iron Whiskers

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Ferromagnetic domain patterns on single-crystal whiskers of pure iron have been studied by using the Bitter powder technique. Patterns have been observed on both magnetized and unmagnetized specimens and in both cases straightforward interpretations of the patterns can be given. Whiskers with axes along the [111] and [100] directions have been studied and observations indicate that crystals with perfect geometry have a very simple domain structure in the unmagnetized state.

INTRODUCTION

WHISKERS grown by hydrogen reduction of ferrous chloride[1] form strain-free single crystals of pure iron. Under certain conditions whiskers attain diameters up to several hundred microns and lengths of the order of 1 cm. Many of these whiskers are found to have mirror-like surfaces which offer excellent possibilities for the study of ferromagnetic domains. In thick whiskers of this type the [111] direction of growth seems to predominate but whiskers growing along [100] directions have also been found.

EXPERIMENTAL PROCEDURES

Domain patterns were observed by means of the Bitter[2] powder technique using a colloidal suspension of Fe_3O_4.

Two methods of applying the colloidal solution to the crystal specimen were used. In the first method the crystal was immersed for $1\frac{1}{2}$ hours in a very light straw-colored suspension of magnetite particles in a $\frac{1}{4}\%$ Aerosol[3] solution. Upon removal, rapid drying took place, leaving a permanent record of the domain boundaries. Low-strength solutions appear to give the best domain patterns on unmagnetized crystals. However, reduction of the aerosol concentration below $\frac{1}{6}\%$ causes flocculation of the suspended magnetite. In the second method, the crystal was mounted so that it could be observed in the microscope with about an 0.004-inch layer of the colloidal solution over the surface. The fitting which was used was arranged so that the whisker could be laid on one face of a soft-iron pole piece. Magnetization was accomplished by varying the distance of an Alnico magnet from the opposite pole face. For these immersed observations a considerably denser suspension of magnetite in a $\frac{1}{2}\%$ Aerosol solution was used, the color being a transparent amber.

The colloidal solution was prepared by precipitating Fe_3O_4 from a solution of ferrous and ferric chlorides with NaOH in the manner described by Elmore.[4] The precipitate settled quickly to the bottom and the clear liquid was decanted off. This precipitate was thoroughly washed and then added to a $\frac{1}{2}\%$ Aerosol solution. The pH of the solution was adjusted to seven by adding sufficient NaOH (or HCl). The Fe_3O_4 particle density can be adjusted by the volume of $\frac{1}{2}\%$ Aerosol added. Most of the domain patterns were photographed using dark-field illumination; however, some bright-field photographs are also shown. None of the photographs has been retouched.

WHISKERS GROWING PARALLEL TO THE [111] DIRECTION

Whiskers growing in the [111] direction have a hexagonal cross section and terminate at the tip in three {100} surfaces forming a cube corner. Figure 1 shows a schematic diagram of a whisker of this type. The six sides of the whisker are equivalent {110} surfaces and intersect the cube faces at the tip along six cube edges. In the unmagnetized state only one of

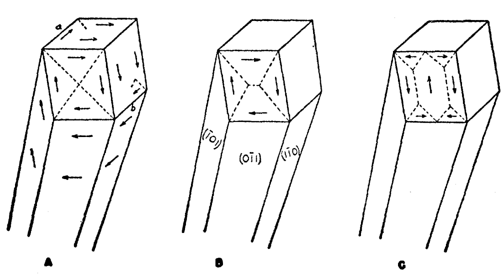


FIG. 1. Diagram of whiskers grown in a [111] direction. Directions of magnetization are indicated for the patterns shown in Figs. 2 and 3.

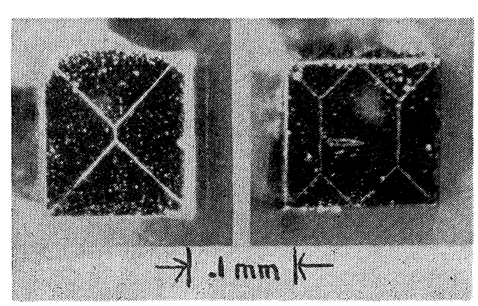


FIG. 2. Two examples of the domain pattern which appears on one of the cube faces at the tip of whisker grown in a [111] direction. These patterns are on two different whiskers.

[1] S. S. Brenner, Acta Metallurgica 4, 62 (1956).
[2] F. Bitter, Phys. Rev. 38, 1903 (1931).
[3] Aerosol is a trade name of the American Cyanamid Company for sodium dioctyl sulfosuccinate.
[4] W. C. Elmore, Phys. Rev. 54, 309 (1938).

the three cube faces at the tip of the whisker develops a complete symmetrical domain pattern. The three types of patterns generally observed in this case are shown in Figs. 2 and 3. The pictures shown in Fig. 3 are the three cube faces forming the end of a single whisker. A cross of the type shown only develops on a whisker having a perfect cube at the tip. The short diagonal boundaries shown on the other two faces of the figure are commonly observed. In the unmagnetized state the six {110} faces of the whisker do not develop domain patterns of any type.

These observations lead to an interpretation of the domain arrangement in the unmagnetized whisker. The {110} faces are thought to be the surfaces of single domains magnetized in a [100] direction parallel to the surface. These six domains produce flux closure around the crystal as shown in Fig. 1(A). The six walls forming the domains near the surface in the main body of the crystal would then coincide with the three cube diagonal planes which are parallel to the growth axis of the whisker. The domain arrangement near the center is not indicated by these observations but probably remains quite simple.

The domains in the main body of the whisker and the domains formed in the cube at the end of the whisker maintain flux closure as shown in Fig. 1(A) except along the cube edges *a* and *b*. If domain walls were formed along these two edges they would have a discontinuous normal component of magnetization across the boundary. Actual observation shows that additional domains form along these edges as shown in Fig. 3. The formation of these domains, however, does not entirely eliminate boundaries with a noncontinuous normal component. It appears in these experiments that magnetite particles do not collect along boundaries of this type as evidenced by the sudden termination of the developed boundary on one cube face and the poorly defined boundary on the other.

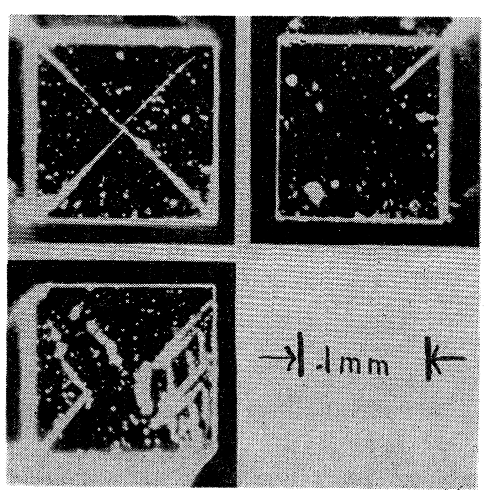


FIG. 3. Domain patterns on the three cube faces at the tip of a single unmagnetized whisker grown in the [111] direction.

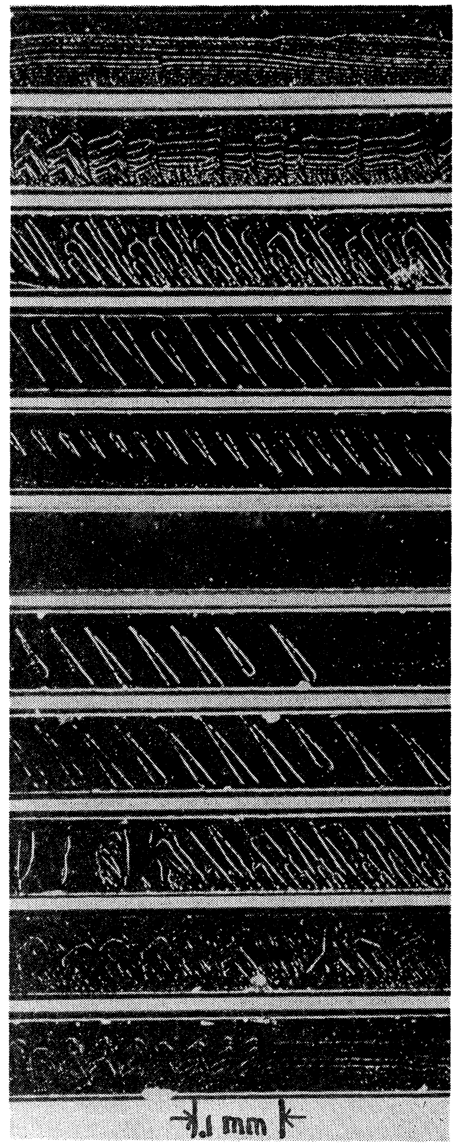


FIG. 4. Series of domain patterns obtained with a magnetic field applied normal to a {110} surface of a crystal grown in the [111] direction. The applied field is zero in the center photograph and increases in opposite directions to 1800 oersteds in the top and bottom photographs.

Figure 4 shows a series of photographs obtained by applying a field normal to a {110} surface of a whisker. The five photographs below the center represent successive increases in field strength in the positive direction up to a value of about 1800 oersteds. The five photographs above the center photo were obtained by applying the field in the negative direction up to the same maximum value.

As the field is increased from zero, dagger-like domains appear on the surface, usually starting from one edge. Similar "dagger" domains were observed and studied as "lozenges" by Paxton and Nilan[5] on poly-

[5] W. S. Paxton and T. G. Nilan, J. Appl. Phys. 26, 994 (1955).

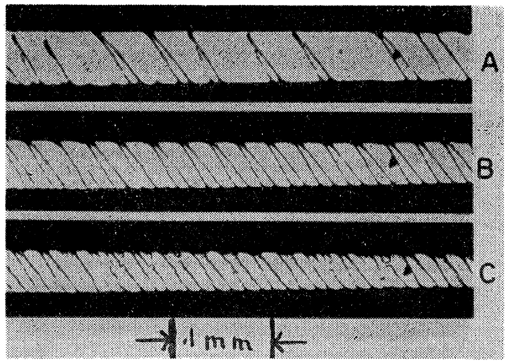


FIG. 5. Details of dagger domains on a {110} surface with changes in applied normal field. Field increases from A to C.

crystalline silicon steel. Photographs in the series show some daggers originating away from the edge; however, this does not commonly occur. With further increase in the normal field the dagger domains move across to the other edge of the {110} surface. Figure 5 shows particular details of the formation and growth of the dagger domains. Photograph A shows the domain pattern for a value of the field just beyond the critical value for formation of the daggers. If the field is reduced below this critical value the daggers recede toward the edge and suddenly disintegrate. Under increasing field, dagger domains form until there is an even spacing of domains on the surface as shown in photograph B. Further increase in field causes the dagger domains to expand in width while maintaining an equal spacing as shown in photograph C. Increasing the field beyond this point causes the pattern to break up into a horizontal domain pattern. This process is shown in the last three photographs of Fig. 4. Reversal of the field direction causes the dagger pattern to form from the opposite edge of the {110} surface being viewed. At high fields in the reversed direction the horizontal domains again form.

Observations were also made simultaneously on the top {110} surface and the two adjacent {110} surfaces inclined at 60°. Application of the field normal to the top surface caused dagger domains to form on the top surface, but no domain patterns were observed to form on either inclined surface.

From these observations a possible interpretation can be given for the formation of dagger domains. As discussed previously, each {110} face forms the surface of a single domain when the whisker is in the unmagnetized state. Figure 6(A) represents the application of a field in the positive direction normal to the top surface. In this case dagger domains form from the front edges of both the top and bottom {110} crystal faces. The upper dagger domains are apparently extensions of the adjacent domain bounded by the $(0\bar{1}1)$ face and are therefore magnetized in the [100] direction. The lower dagger domains are correspondingly extensions of the domain bounded by the $(1\bar{1}0)$ face and are therefore magnetized in the $[00\bar{1}]$ direction. The resultant magnetization due to the formation of the two sets of dagger domains is thus in the direction of the field.

Reversal of the field causes dagger patterns to form from the rear edges of the top and bottom faces as shown in Fig. 6(B). The patterns now represent the extensions of the domains bounded by the two rear {110} surfaces and again give the crystal a resultant magnetization in the direction of the applied field. On {110} faces the dagger domains are approximately parallel to a [100] direction and make an angle of 60° with the edge of the crystal.

WHISKERS GROWING PARALLEL TO A [100] DIRECTION

Whiskers growing in a [100] direction have also been investigated in both the unmagnetized and magnetized

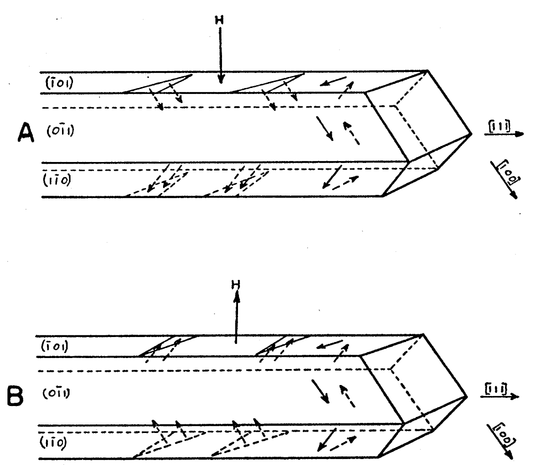


FIG. 6. Diagrams indicating directions of magnetization in dagger domains formed when field is applied normal to a {110} surface.

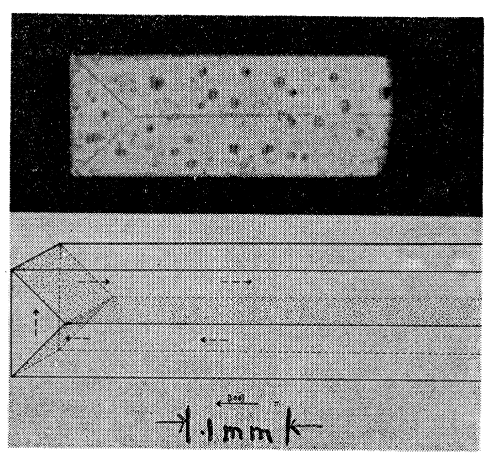


FIG. 7. Domain pattern observed on a {100} surface of a whisker having a [100] axial direction.

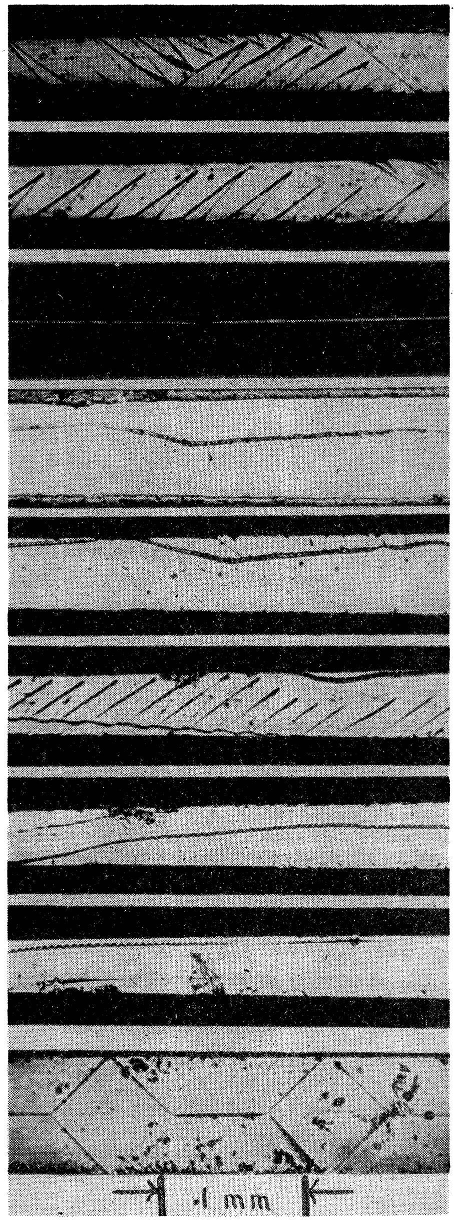


FIG. 8. Series of domain patterns obtained with a magnetic field applied normal to a {100} surface of a crystal grown in a [100] direction.

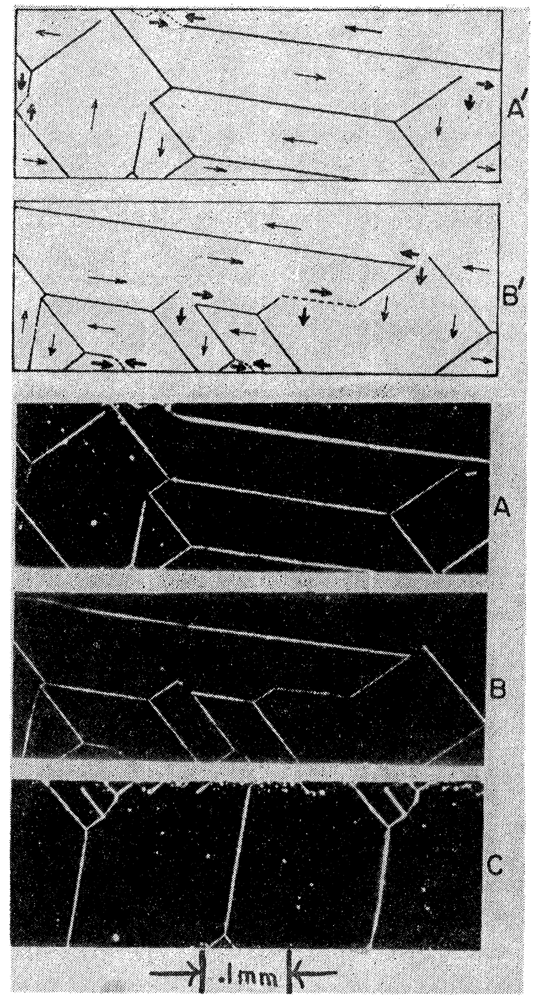


FIG. 9. Domain patterns remaining on a {100} surface after several magnetizations. A' and B' are interpretations of the photographs A and B.

cases. Figure 7 shows the pattern obtained on an unmagnetized crystal. This whisker terminated in a flat mirror surface making 90° angles with the sides of the whisker. After immersion in the colloid, identical domain patterns developed on two opposite sides of the whisker, one of which is shown in Fig. 7. The other two sides and the end developed no patterns indicating that the three domains observed pass completely through the crystal. This simple domain structure which produces flux closure in the crystal has also been observed on a number of other whiskers growing in a [100] direction. Simple domain structure and flux closure of this type have also been observed on silicon iron crystals by Williams and Shockley[6] and by Williams as reported by Kittel.[7]

Figure 8 shows a series of photographs obtained by applying a magnetic field normal to a {100} face of a whisker which had grown in a [100] direction. The third photograph from the top shows the domain pattern obtained when the crystal was in its original unmagnetized state. The single domain wall running down the middle of the crystal is similar to the no-field pattern observed on the cubic whisker shown in Fig. 7.

Application of a weak field in the positive direction (into the surface) causes the center domain boundary to move toward one edge of the crystal as shown in the fourth and fifth photographs of Fig. 8. Reversal of this weak field causes the boundary to move in the opposite

[6] H. J. Williams and W. Shockley, Phys. Rev. **75**, 178 (1949).
[7] Kittel, Revs. Modern Phys. **21**, 541 (1949).

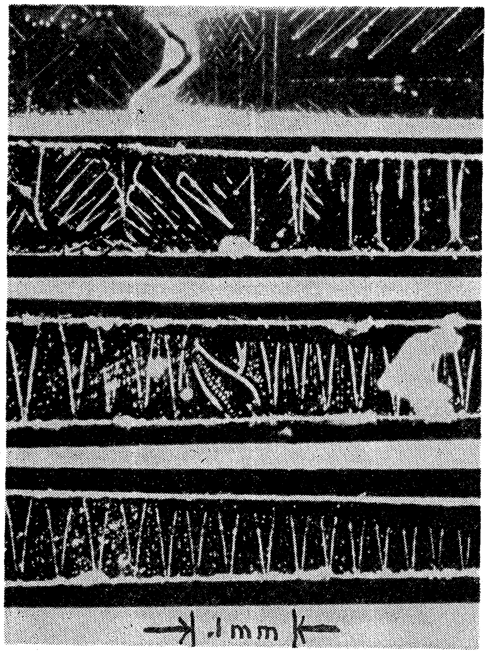


FIG. 10. Series of domain patterns on a {100} surface as the normal field is varied between 0 oersteds (top) and 1800 oersteds (bottom).

direction although this is not shown in the figure. As the field is increased beyond a certain critical value, dagger-like domains making a 45° angle with the edge of the crystal begin to form. Reversal of the field also causes dagger-like domains to form at a certain value of the field, however this pattern does not necessarily form from the opposite edge. As the normal field is increased, the dagger pattern disappears and long parallel domain walls form moving in from one edge of the crystal as shown in the seventh and eighth photographs of the series. The last photograph in Fig. 8 shows the domain pattern after the field has been applied several times and then removed. This remaining domain pattern changes slightly after each application of the field, but still retains the simple geometrical structure of straight lines and rectangles.

Examples of the domain patterns which remain on {100} surfaces after the field has been removed are also shown in Fig. 9. Photographs *A* and *B* show the same area of a crystal surface after successive application and removal of the field in the positive direction. Photograph *C* is the pattern remaining on the same surface but at a different point along the whisker. These patterns indicate that the whisker has grown with two {100} surfaces along an axis inclined at about 8° to a [100] direction. The long parallel domain boundaries can be interpreted as 180° boundaries while the rectangular and triangular domains have 90° boundaries. Diagrams *A′* and *B′* in Fig. 9 give the directions of magnetization in the domains shown in photographs *A* and *B*. Several sections of the domain boundaries in these patterns have not attracted magnetite particles. If one compares the directions of magnetization assumed in the accompanying diagrams with the sections where the lines have not developed it appears that magnetite particles have not collected along boundaries across which the normal component of magnetization is not continuous.

The change in direction of magnetization across these domain walls is indicated in the diagram by the heavy black arrows. From these observations it would appear that as the field is removed, systems of domains form at various points along the crystal in order to give flux closure within the crystal. When the field reaches zero these systems are unable to match up, and perfect closure does not occur in all areas. Consequently a few boundaries are formed with a noncontinuous normal component of magnetization across them.

The series of photographs in Fig. 10 shows another example of patterns observed on the surface of a whisker with a rectangular cross section. The top photograph shows the domain pattern on the surface with no applied field. However, this observation was made after one application of the field. Tree patterns very similar to those observed by Williams, Bozorth, and Shockley[8] on surfaces inclined slightly to a {100} surface are seen in one area of this crystal. This whisker was slightly imperfect and the exact orientation of the surface was not determined. The three lower photographs of Fig. 10 show domain patterns which develop as the normal field is applied and increased to a value of 1800 oersteds.

SUMMARY

These experiments indicate that single-crystal iron whiskers offer an excellent method for the study of ferromagnetic domains. A very simple domain structure has been found to exist in the unmagnetized state of whiskers grown in the [111] and [100] directions. Experimental evidence indicates that domain walls exist which do not attract magnetite particles. Domain walls of this type always appear to have a discontinuous normal component across the boundary. Domain patterns developed under the influence of an applied field have been studied and in the case of {110} faces can be readily interpreted.

[8] Williams, Bozorth, and Shockley, Phys. Rev. **75**, 155 (1949).

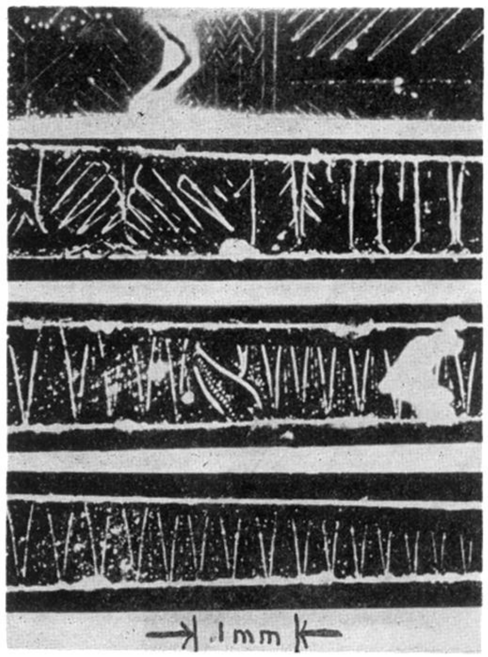
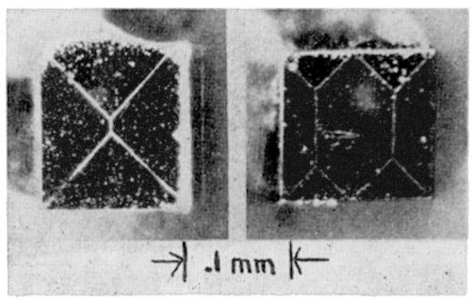


FIG. 10. Series of domain patterns on a {100} surface as the normal field is varied between 0 oersteds (top) and 1800 oersteds (bottom).



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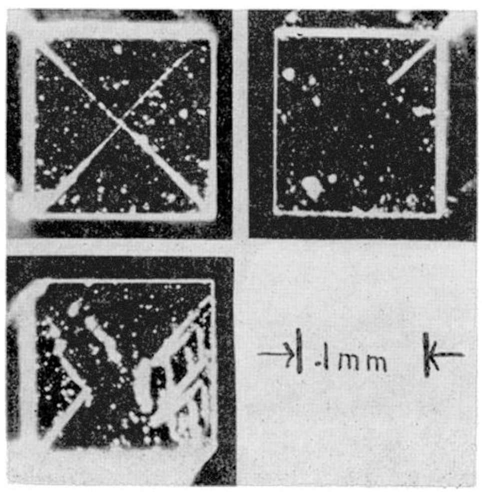


FIG. 3. Domain patterns on the three cube faces at the tip of a single unmagnetized whisker grown in the [111] direction.

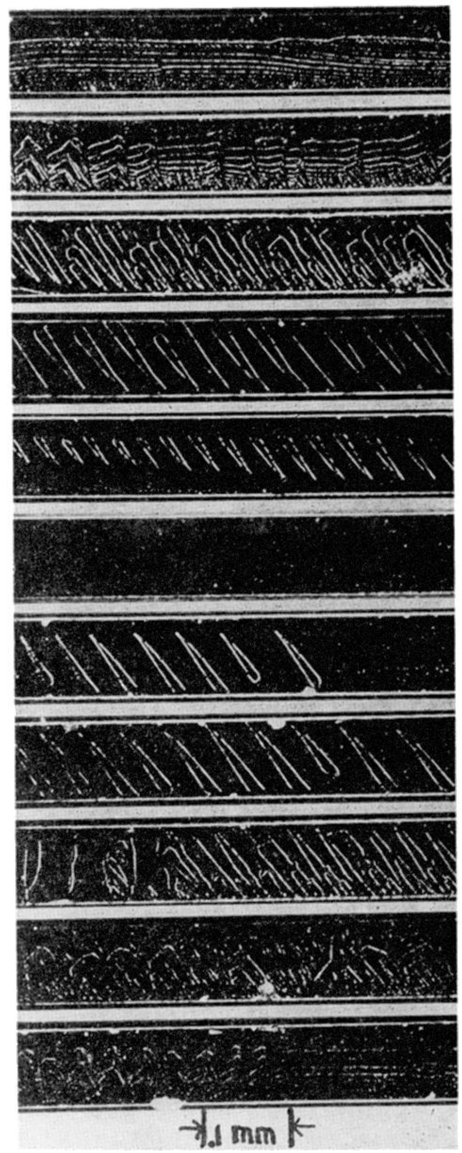


FIG. 4. Series of domain patterns obtained with a magnetic field applied normal to a {110} surface of a crystal grown in the [111] direction. The applied field is zero in the center photograph and increases in opposite directions to 1800 oersteds in the top and bottom photographs.

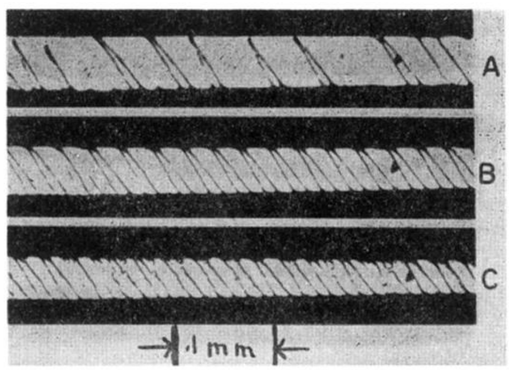
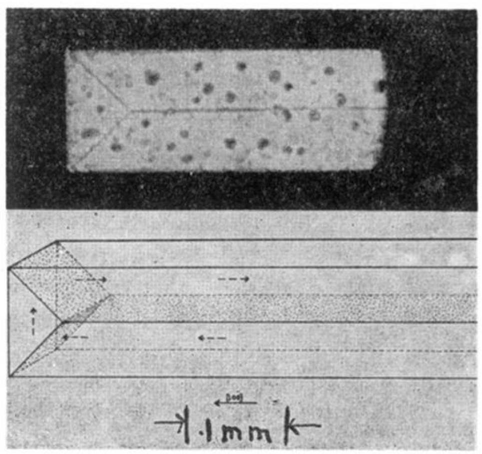


FIG. 5. Details of dagger domains on a {110} surface with changes in applied normal field. Field increases from A to C.



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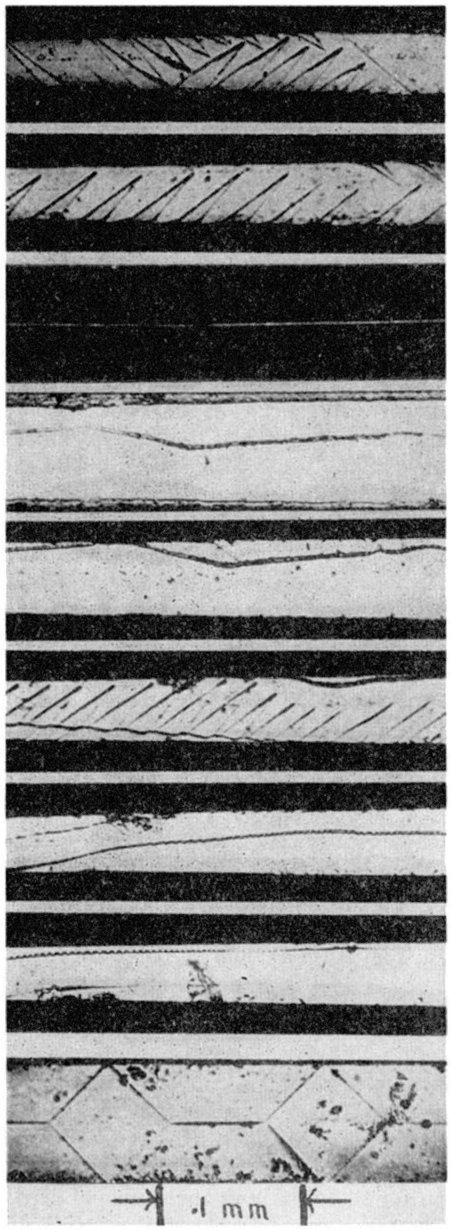
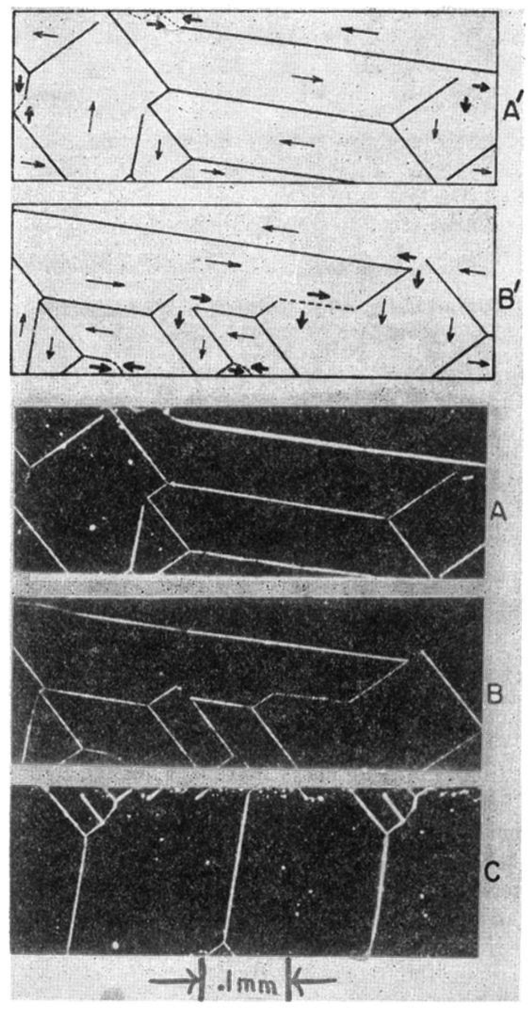


FIG. 8. Series of domain patterns obtained with a magnetic field applied normal to a {100} surface of a crystal grown in a [100] direction.



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