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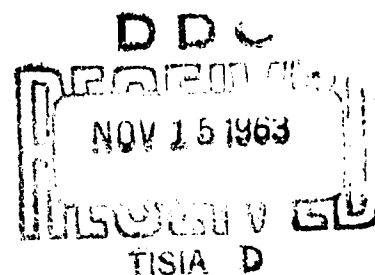
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POLISHES AND ETCHES FOR TIN TELLURIDE,
LEAD SULFIDE, LEAD SELENIDE, AND
LEAD TELLURIDE

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POLISHES AND ETCHES FOR
TIN TELLURIDE, LEAD SULFIDE, LEAD SELENIDE, AND LEAD TELLURIDE

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ABSTRACT: A review is given of chemical and electrolytic polishes and dislocation etches for SnTe, PbS, PbSe, and PbTe, covering the period from 1907 to 1962. Recipes, conditions, and results are described. Satisfactory polishes for all compounds except PbS and etches for all except SnTe are included.

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POLISHES AND ETCHES FOR TIN TELLURIDE, LEAD SULFIDE, LEAD
SELENIDE, AND LEAD TELLURIDE

This report represents a study of chemical and electrolytic polishes and dislocation etches for SnTe, PbS, PbSe, and PbTe made at the U. S. Naval Ordnance Laboratory, White Oak, Maryland, during the periods, April-August 1959 and April 1961-May 1963. It is for information only and is published under FR-46.

The author wishes to express thanks to his supervisor, Dr. Bland B. Houston, Jr., for helpful advice and for the photographs shown in Figures 3 and 4.

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Captain, USN
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LOUIS R. MAXWELL
By direction

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(Note the five-sided and possibly eight-sided pits.)

Figure

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Chapter I

INTRODUCTION

This report describes chemical and electrolytic polishes and etches for SnTe, PbS, PbSe and PbTe.

The polishes are for removing damaged surface layers formed by grinding or mechanical polishing, for removing pits caused by etching, and for producing smooth, shiny surfaces. Damage-free surfaces such as these, are necessary for measuring the optical reflectivity which is an important tool for the study of properties of solids.

The etches are for producing pits where dislocations intersect the surface of the crystal. These pits are observed along grain boundaries, traces of active slip planes, and at points randomly distributed over the surface. The density of the pits, the average grain size, etc., give an estimate of the quality of the crystal.

Descriptions of the polishes and etches are given in Chapters II and III of this report. Those not originally developed by the author were tested, and the results, together with some additional tests on the author's polishes and etches, are given in the paragraphs entitled "Test Results."

Chapter II

POLISHES

A. Tin Telluride

FAUST AND SAGAR.¹ After the surface is first given a high mechanical polish, chemical polishing is carried out at room temperature in a solution consisting of 1 part of HF, 3 parts of HNO₃, and 6 parts of glacial acetic acid. All parts are by volume. A mirror-like surface, free of stains is obtained after polishing for about 10 seconds. This solution is a modification of an etch published by Dash^{2,1} for producing dislocation etch pits on silicon.

Test Results. Both 'pulled' crystals and castings were tested. The samples were ground on a glass plate with a water slurry of Carborundum No. 95 grit aluminum oxide optical finishing powder. They were then polished, first with a distilled-water suspension of gamma alumina on a Buehler Rayvel cloth, and then with Buehler's Metadi $\frac{1}{4}$ μ diamond polishing compound lubricated with their Automet lapping oil on a nylon cloth. The polishing compound was removed from the surfaces of the samples by wiping them with lens paper soaked in acetone.

The specimens were immersed in the polishing solution (in a small polyethylene beaker) for 10 sec., at 25°C, rinsed with distilled water followed by acetone, and carefully dried on lens paper. The fine lines remaining after mechanical polishing were not removed after 10 sec. of chemical polishing. Also, a very thin film or a slightly hazy appearance was often visible at a magnification of 9X. The samples that were castings contained more than one phase. Their surfaces were preferentially attacked by the polishing solution so as to form mosaic-like ridges of lines that are believed to be the location of one or more of these phases. (See Fig. 1.)

The scratches remaining after mechanical polishing could be removed by 3-5 min. of chemical polishing. However, the surface sometimes had a heavier film (and sometimes brown stains) than that found after the 10 sec. treatment. A

procedure was found that frequently reduced and sometimes eliminated the film and stain. The specimen was immersed in the polishing solution at 25° and the latter was stirred by gentle swirling. After 5 min., the solution was gradually diluted while swirling, with twice its volume of glacial acetic acid. The sample was then rinsed with distilled water followed by acetone and carefully dried on lens paper. A shiny surface was obtained. Very fine lines were observed along some large-angle grain boundaries, and at a magnification of 9X there were many raised specks distributed randomly over the surface.

In order to find the optimum ratio of acetic acid: HNO_3 :HF, various ratios of the concentrated acids were tested. Best results were obtained, however, by using the composition given by Faust and Sagar, described above.

FINN.³ The samples are immersed for 15 min. at room temperature in a solution consisting of 20 ml. of 15% NaOH and 20 ml. of saturated $\text{Na}_2\text{S}_2\text{O}_8$; thereafter they are rinsed, first with 10% H_2O_2 and then with distilled water. The procedure was reported to give good grain boundary definitions with some polishing.

Test Results. Both 'pulled' crystals and castings were tested. They were all p-type.

The $\text{Na}_2\text{S}_2\text{O}_8$ was purified by filtering a saturated aqueous solution twice through a fine-porosity, sintered-glass Büchner funnel, diluting the solution to twice its volume with reagent grade methanol, and filtering off the white crystalline precipitate. The precipitate was washed with methanol and dried first in air, and then over NaOH in a desiccator.

The SnTe samples were ground with a water slurry of Carborundum No. 95 grit aluminum oxide optical finishing powder, first on a glass plate and then on a piece of twill jean cloth.^a They were cleaned by rinsing with water followed by soaking a few minutes in acetone. The ground samples were immersed in Finn's solution at room temperature for 15 min., rinsed with a 10% aqueous solution of H_2O_2 followed by distilled water, and dried on lens paper.

^a K. S. 2423 twill jean cloth, Exeter Manufacturing Co., Inc., Exeter, N. H.

The surface obtained by this procedure was partially covered with a thin, light-colored film. When the H_2O_2 rinsing step was omitted, the film was not observed. The surface then had a clean, uniformly etched appearance. Large-angle grain boundaries were sharply defined. The samples that were castings contained more than one phase. Their surfaces were preferentially attacked so as to form mosaic-like ridges that are assumed to be the location of one or more of these phases. No polishing action was detected on either the pulled crystals or on the castings.

B. Lead Sulfide

BREBRICK AND SCANLON.⁴ Polishing is carried out at 50°C in a freshly prepared mixture of concentrated acids consisting of 30 parts of HCl, 10 parts of HNO_3 , and 1 part of acetic acid. The sample is then rinsed for a few minutes in 10% acetic acid at room temperature to remove a slight film that is formed during the polishing process. It was reported that a sanded surface can be converted in a few minutes to a mirror-like surface by this procedure.

Test Results. An attempt was made to polish the {100} surfaces of several kinds of PbS crystals: (1) n-type, obtained from natural sources; (2) n-type, grown by the Bridgman-Stockbarger technique; and (3) p-type, similarly grown. The surfaces were first ground on No. 600 grit silicon carbide paper which was lubricated with water. They were then soaked for 10 min. in the polishing solution at 50°; soaked in 10% acetic acid at 25° until the film formed during the polishing step was removed (5-15 min.); rinsed with water, and then finally dried on lens paper.

The acetic acid attacked the film, first along the edges of the crystal, and then gradually worked inward, initially leaving a fairly shiny surface. But, by the time the film had been completely removed, a gray tarnish had developed which seemed to grow darker during the subsequent steps. The scratches formed during grinding were, for the most part, removed. Under a magnification of 480X, the surface showed a fine roughness.

C. Lead Selenide

TOMPKINS AND JOHNSON.⁵ The sample is first ground with 600 mesh silicon carbide and then polished with powdered chalk. It is then polished electrolytically in a solution consisting of 40 ml. of glycerol, 10 ml. of HNO_3 , and 10 ml. of glacial acetic acid, for 2 min. at 50° . The sample serves as the anode. The potential across the cell is 6 volts, and the initial and final current densities are 0.30 and 0.10 amp./cm.², respectively. The heavy layer of selenium formed during electropolishing is removed with a solution of KCN. The final surface was reported to be specular. Although it was also specular under a microscope, very shallow undulations could be seen, and linear depressions were observed along crystal boundaries.

Tompkins and Susman⁶ have published a modification of this polish. The sample is first ground on wet No. 600 grit silicon carbide paper, then with No. 600 grit silicon carbide powder in water, and finally with No. 900 grit alundum powder in water. It is polished with a fine cloth impregnated with magnesium oxide. It is then polished electrolytically in a solution consisting of 42 ml. of anhydrous glycerol, 8 ml. of concentrated HNO_3 , and 10 ml. of glacial acetic acid for 1.0-1.5 min. at 45° . The sample serves as the anode. The potential across the cell is 10 volts, and the initial and final current densities are approximately 0.3 and 0.1 amp./cm.², respectively. The sample is then thoroughly washed with distilled water and swabbed with a saturated aqueous solution of KCN to remove the layer of red selenium formed during electropolishing. The surface was reported to be specular both visually and under the microscope.

Test Results. Some n- and p-type PbSe castings were ground with a water slurry of carborundum No. 95 grit aluminum oxide optical finishing powder on a glass plate. After rinsing with water and drying on lens paper, they were polished with magnesium oxide (Buehler's Magomet Polishing Compound, lubricated with water) on a Buehler Microcloth. They were rinsed with water, soaked a few minutes in acetone, and dried on lens paper.

Electropolishing was carried out using the sample to be polished as the anode and a strip of platinum foil as the

cathode. The electrolytes were prepared from reagent grade chemicals. Concentrated acids were used.

a. The samples were electropolished by Tompkins and Johnson's procedure, thoroughly rinsed with water, and the thick layer of material formed during electropolishing was removed by swabbing with cotton soaked in a saturated aqueous solution of KCN. Thereafter, the samples were rinsed with water and dried on lens paper. This procedure produced a shiny surface. At a magnification of 9X, the surface had an 'orange peel' appearance and some barely visible patches of whitish film. At a magnification of 240X, the surface was smooth. These results were obtained whether or not the electrolyte was stirred during electropolishing.

b. When the samples were similarly electropolished, swabbed, etc., using Tompkins and Susman's procedure, their surfaces were partly shiny and partly etched. Shiny surfaces, free of etching, were obtained when there was a potential of 6 rather than 10 volts across the cell during electropolishing. At a magnification of 9X, the surfaces had a pronounced 'orange peel' appearance, and at 240X, they were smooth, except for shallow undulations on some of the samples. These results were obtained whether or not the electrolyte was stirred. Raising the electrolyte temperature from 45 to 50° did not seem to materially effect the results.

COATES.^{7,8} This is a material removal etch that was also reported to produce a polished surface.

The samples are ground with 4F carborundum and polished at 35° in a freshly prepared solution consisting of 5 volumes of aqueous KOH (saturated at 20°), 1 volume of 30% H₂O₂, and 5 volumes of ethylene glycol. The solution is good for about 15 min. of polishing, after which it should be replaced or a few drops of H₂O₂ solution added. The sample is agitated by moving it slowly backwards and forwards through the solution. If its surface becomes dull, the rate of agitation should be increased. If the temperature rises too high or if the concentration of H₂O₂ is too great, the surface may become pitted or may become colored reddish-brown. After polishing, the samples are rinsed with distilled water.

It was reported that thin slices of PbSe single crystals treated as described above, can be reduced to a thickness of $1\ \mu$ and are quite flat. The surfaces thus produced have a good polished appearance.

Test Results. In order to minimize the thermal decomposition of the H_2O_2 , the solution was prepared by mixing and cooling the ethylene glycol and saturated KOH before adding the H_2O_2 . It was prepared just before using. The solution was tested on n- and p-type material at 25 and 35-40°. Both fresh cleavage surfaces and those obtained by grinding on No. 600 grit silicon carbide paper, lubricated with water, were used. The former were immersed in the solution for 2 min. and the latter for 10. Agitation was produced by moving the samples slowly back and forth. After removal from the solution, they were rinsed with water and carefully dried on lens paper.

At a magnification of 480X, many square, shallow, flat-bottomed pits were seen; only a few were pyramidal. Some of the pits were so shallow that they were just barely visible. Yellow-brown stains were present on the surfaces of some of the samples; these were removed upon dipping in 1:1 acetic acid for a few seconds and rinsing with distilled water. This clean surface was shiny, but sometimes had a slightly etched appearance.

D. Lead Telluride

DE NOBEL.⁹ After scouring and polishing, a plate of p-type PbTe is treated with a 22% HNO_3 solution (time and temperature not specified). A white layer is reportedly formed on the surface. The sample is then treated with a freshly prepared, boiling solution of 5% NaOH and 5% $Na_2S_2O_4$, to obtain a shining surface free from tellurium and tellurium oxide layers.

Test Results. The procedure was tested at 25 and 40°C. The length of the acid step was varied from 1 to 5 min., and that of the NaOH/ $Na_2S_2O_4$ step from 10 sec. to 5 min. The samples used were n- and p-type castings and p-type crystals prepared by the 'pulling' technique. The castings were polycrystalline, contained more than one phase, and in the case of the p-type samples, generally had an n-type core. The 'pulled' crystals were mildly polycrystalline and contained only one phase.

A surface of each specimen was ground on No. 600 grit silicon carbide paper lubricated with water and on dry No. 4/0 grit emery paper. They were cleaned after each step by rubbing with lens paper soaked in acetone. Following grinding, the surfaces were polished using Buehler's Metadi $\frac{1}{4}$ μ diamond polishing compound lubricated with their Automet lapping oil on a nylon cloth. The polishing compound was removed by carefully wiping with lens paper soaked in acetone.

The white layer (reported by de Nobel) obtained by the acid treatment, was not observed. On p-type samples, a black layer was formed instead, while on n-type a gray one tended to form, particularly when the acid treatment was carried out at 40°. At this temperature, the color difference was sufficiently sharp to indicate clearly the locations of the n- and p-type regions (observed on p-type castings containing n-type cores). The NaOH/Na₂S₂O₄ treatment removed neither the black nor the gray layers. These layers, however, could be mostly rubbed off with lens paper, leaving a slightly rough surface having a dull metallic appearance. It was noticed that on p-type castings having an n-type core, the core was somewhat smoother than the remainder of the surface.

SCHMIDT (A).¹⁰ The surface to be electropolished is ground on No. 600 grit silicon carbide paper lubricated with water. It is then cleaned with benzene to remove traces of abrasive and preheated at 70° in a drying oven to reduce the possibility of cracking by thermal shock during electropolishing. The electrolyte is a mixture of 100 ml. of 85% H₃PO₄ and 10 g. of CrO₃ (both reagent grade). Electropolishing is carried out between 90 and 105°. The sample to be polished serves as the anode and a strip of tantalum as the cathode. The current density is about 3 amps./cm², requiring a cell potential of 6-12 v. The sample is agitated continuously. After polishing, it is rinsed in warm water (70°) and dried in a stream of air. The procedure was reported to produce a mirror-like polished surface free of macroscopic defects or waves.

Tilly¹¹ made some slight modifications in Schmidt's procedure. After the sample has been ground on 700 mesh carborundum powder, it is ultrasonically cleaned with carbon tetrachloride. Upon removal from the electrolyte after electropolishing, it is immediately dipped into alcohol at 70° to suppress the formation of tenacious oxide layers. The

white scum which forms is wiped off with cotton wool, and the sample is washed in a stream of cold water. The author recommended that the electropolishing process be carried out in an inert atmosphere to avoid oxidation, and reported that electropolishing for 4-6 min. is sufficient to produce a mirror-like finish. Macroscopic cracks, believed to be caused by thermal shock or growth fissures, are often present.

Test Results. Using Schmidt's procedure, a number of n- and p-type samples (crystals prepared by the 'pulling' technique and castings) were electropolished. Usually 15-30 sec. was needed to remove the scratches that had been formed during the grinding process. To obtain a current density of 2-3 amps./cm.², a potential of approximately 20 volts across the cell was required. This high current density caused the anode to become noticeably hotter than the solution, producing a gradual rise in the temperature of the solution.

The electropolished samples frequently had black or brown stains and whitish patches of film on them. Tilly's practice of dipping the samples in 70° ethanol (instead of water) immediately following electropolishing tended to reduce or eliminate the black stains. Subsequent immersing in 1:10 HNO₃ for 5 sec. tended to remove the whitish film patches and any dark stains that were present. (Sometimes they were completely removed and sometimes not.) Although the polished surfaces were usually shiny, they generally had a fine orange-peel texture, and at a magnification of 240X, they usually had a somewhat rough appearance. Even when the precautions suggested by Schmidt were followed, macroscopic cracks, presumably initiated by thermal shock, formed in almost every sample.

NORR.¹² The surface to be electropolished is ground on No. 600 grit silicon carbide paper lubricated with water, after which it is washed with water and thoroughly wiped with lens paper. The electrolyte is prepared by dissolving 20 g. of KOH pellets in 45 ml. of distilled water. To this are added while stirring, 35 ml. of glycerol and 20 ml. of ethanol. The chemicals are reagent grade. The cell container is a 250 ml. beaker. The cathode is a piece of platinum foil, 2 to 20 cm.², and the anode is the PbTe to be electropolished, with its ground surface oriented horizontally. The electrolyte is stirred with a magnetic stirrer at the rate of 75-200 r.p.m. and the anode is slowly rotated around its vertical axis at about 4 r.p.m.

to prevent the formation of flow lines on its surface. The voltage drop across the cell during electropolishing is 6 volts and the current density is about 0.2 amp./cm.². (Since the surface area to be polished has generally varied with different samples, the cell voltage, rather than the current density, has proven to be the more convenient parameter to control.) The temperature of the electrolyte is about 25°. The anode is electropolished until the surface is smooth and free from scratches (generally 5-15 min.). It is then rinsed with water, immersed a few seconds in 1:10 HNO₃, rinsed with distilled water, and carefully dried on lens paper.

The surface produced by this electropolishing procedure has proven satisfactory for reflectivity measurements used for the calculation of the carrier effective mass. The procedure has been successful on both single-crystalline and polycrystalline p-type PbTe. Under a magnification of 9X, a few tiny raised specks of PbTe could be seen occasionally on the surface. With material that contained more than one phase, the surface was shiny but not smooth.

A graph of current density vs. cell voltage did not show the pronounced plateau frequently observed with successful electropolishing solutions. During electropolishing, a thin film was sometimes visible, but it disappeared when the cell was disconnected or when the anode was withdrawn from the solution. Consistent with the presence of the film were the facts that, (1) the vertical and horizontal surfaces of the anode were polished equally well, (2) the cell current did not vary significantly when the cathode area was increased from 2 to 20 cm.², and (3) the cell current did not vary significantly when the anode-cathode distance was increased by a factor of 7. A possible explanation of these observations is that an anode film, rather than the electrolyte, was the current-limiting unit within the cell during electropolishing.

Test Results. Using the above procedure, similar results were obtained on single-phase, polycrystalline, n-type material.

SCHMIDT (B).¹³ The sample is mounted in a jig and abraded flat, first on No. 400 grit aluminum oxide paper followed by No. 600 grit silicon carbide paper, both lubricated with water, and finally on No. 4/0 grit French emery paper. The samples are thoroughly cleaned between grinding stages, and care is taken not to overload the Nos. 600 and 4/0 grit papers.

Using a figure eight, 1-2 inch stroke, the sample is rubbed 2-3 min. on twill jean cloth stretched over a flat glass plate and saturated with a 1:1 mixture of glacial acetic acid and 30% H_2O_2 and Linde A abrasive.

The sample is then rubbed 2-3 min. on twill jean cloth stretched over a flat glass plate and saturated, as before, with the acetic acid-hydrogen peroxide mixture, but without the Linde A abrasive. Following polishing, it is immediately rinsed, first with warm water, and then with acetone or ethanol.

It was recommended that when undesirable films are formed on the polished samples, the latter be immersed 3-4 min. in a 1:1 mixture of glacial acetic acid and 30% H_2O_2 , then swabbed with water, and finally polished by the procedure of the second twill jean step, above.

It was reported¹³ that back-reflection Laue photographs showed these surfaces to be free of strain. They were highly reflective, with no fine grain ripple, and were visibly smoother and flatter than those obtained by electrolytic polishing.

Test Results. The samples were first abraded flat on a glass plate using a water slurry of Carborundum No. 95 grit aluminum oxide optical finishing powder, then ground on dry No. 4/0 grit emery paper, and finally cleaned with water followed by acetone. The flat surfaces had areas of 1-2 cm.². The samples were p-type, single-phase castings.

Using a figure eight, 1-2 inch stroke, the samples were polished for 3 min. on a piece of twill jean cloth^a (on a flat glass plate) sprinkled with Linde A^b abrasive and saturated with a 1:1 mixture of glacial acetic acid and 30% H_2O_2 . The procedure was carried out at 25°. The samples were then rinsed with distilled water and wiped with a piece of lens

^a K. S. 2423 twill jean cloth, Exeter Manufacturing Co., Inc., Exeter, N. H.

^b Fine Abrasive, Type A-5175, Linde Co., Division of Union Carbide Corp., New York 17, N. Y.

paper soaked in acetone. The surfaces were rather shiny, but had many fine scratches.

The samples were then polished for 3 min. on a second piece of twill jean cloth (on a flat glass plate) saturated with the acetic acid-hydrogen peroxide solution, but without the Linde A abrasive. The procedure was carried out at 25°. Immediately thereafter, they were rinsed, first with warm distilled water, and then with acetone.

The surfaces thus obtained were mostly covered with a blue-black film. The film-free areas were highly reflective and visibly flatter than surfaces produced by electrolytic polishing. At a magnification of 9X, a very fine ripple could be detected. A similar surface, but with no blue-black film, was obtained by using a polishing solution containing 50 ml. of glacial acetic acid + 50 ml. of 30% H_2O_2 + 5 g. of tartaric acid.

Chapter III

DISLOCATION ETCHES

A. Tin Telluride

Neither a thorough search of the literature nor many hours of work in the laboratory has enabled the author to find a satisfactory dislocation etch for SnTe.

B. Lead Sulfide

COATES.^{7,8} The etching solution is a 1:1:1 mixture of aqueous KOH (saturated at 20°), 30% H₂O₂, and ethylene glycol. The etching temperature was not given.

Test Results. In order to minimize the thermal decomposition of the H₂O₂, the ethylene glycol and the saturated KOH solution were mixed and cooled before the H₂O₂ was added. The solution was prepared just before using. The PbS samples were either obtained from natural sources (n-type only) or were grown by the Bridgman-Stockbarger technique (both n- and p-type). Etching was carried out on fresh cleavage surfaces at 25 and 35°, with and without agitation. Agitation was produced by moving the sample slowly back and forth through the solution. After etching for 5 min., the samples were rinsed with water and carefully dried on lens paper.

Etch pits were formed in straight lines (and sometimes in bands) running in the two <100> directions, at points randomly distributed over the surface, and along small-angle grain boundaries. For revealing dislocations, the etch had the following disadvantages: (1) a thin, yellow-brown film was usually formed which covered most of the surface (easily visible at a magnification of 240X); (2) generally, the pits were not sharply defined, and they were sometimes partially obscured by the surface film; and (3) the surface often had areas that were either resistant to or roughened by the etch.

BREBRICK AND SCANLON.⁴ Samples are etched by treating them for 1-10 min. in freshly prepared solutions consisting of 1 part of concentrated HCl and 3 parts of a thiourea solution (100 g./l.), at a temperature of 60°. Sharp, square, pyramidal pits were reported.

Franklin and Wagner¹⁴ using this etch on fresh cleavage surfaces of a number of PbS samples (both n- and p-type) observed that two types of pits, A and B, were formed.

The predominate one, Type A, was pyramidal, more distinct, and usually had a greater width and depth than Type B. Its sides were generally parallel to the $\langle 100 \rangle$ directions of the crystal. The pits were 3-5 μ wide after a 60-sec. etch at 59°. They occurred along small-angle grain boundaries, at points distributed randomly over the surface, and along traces of active slip planes. Variations in the ratio of thiourea solution to hydrochloric acid in the etch caused a change in the orientation of the pits. When the ratio was 1:1, 2:1, or 4:1, the sides of the pits were parallel to the $\langle 110 \rangle$ directions, while with a ratio of 3:1, they were generally parallel to the $\langle 100 \rangle$.

The Type B pits were almost always square and usually flat-bottomed. They occurred singly, in pairs, and overlapping in rows extending in the $\langle 100 \rangle$ directions. The sides of the pits were almost always parallel to the $\langle 110 \rangle$ directions.

The etching of the two surfaces formed by the cleaving of a crystal produced etch pits at corresponding points on the two surfaces.

Test Results. Samples on n- (natural) and p-type material (prepared by the Bridgman-Stockbarger technique) were etched for 2 min. at 60°C, rinsed with distilled water, and carefully dried on lens paper. The results were generally similar to those of Franklin and Wagner, described above. A few 5-sided, pyramidal pits were observed on some of the n- and p-type crystals (see Fig. 2); and a few cone-shaped pits, that appeared to be 8-sided, were seen on one n-type sample. Only a small number of typical Type B pits were observed on our n-type crystals, possibly because of the very high density of pyramidal pits on this material. (The pit density was

considerably higher than on our p-type samples.) A large number of the pits on the n-type material had their sides parallel to the $\langle 110 \rangle$ directions of the crystal.

C. Lead Selenide

BREBRICK AND SCANLON.⁴ Samples are etched by treating them for 1-10 min. in a freshly prepared solution consisting of 1 part of concentrated HCl and 3 parts of a thiourea solution (100 g./l.), at a temperature of 60°. The formation of etch patterns was reported.

Test Results. An attempt was made to etch both n- and p-type PbSe. Fresh cleavage surfaces were immersed in the solution at 60° for 5-30 min., rinsed with water, and dried on lens paper. The thin film that was generally present was removed by swabbing with cotton dipped in benzene.

After etching for 30 min., fairly sharp, square, pyramidal pits about $1\frac{1}{2} \mu$ wide were formed. Their sides were parallel to the $\langle 110 \rangle$ directions of the crystal. Pits were obtained along small-angle grain boundaries, along traces of active slip planes, in curved bands, and at points distributed randomly over the surface.

COATES, LAWSON, and PRIOR.⁸ Using Coates' material removal etch (see Sect. II-C-COATES), these authors produced pits on PbSe single crystals that may be dislocation etch pits. The exact conditions for pitting were not given, but it was stated that it is produced when the etch is abnormally vigorous. Further, it was indicated that the vigorousness of the etch is increased by raising its temperature above 35°, by increasing the concentration of H_2O_2 , and by increasing the agitation.

From the photograph published in their report, the pits appeared to be round; part of them were arranged in straight lines, some parallel and some perpendicular to each other.

Igaki and Ohashi¹⁵ have published a study in which this etch was used. Judging from their report and photographs, etch pits were formed along small-angle grain boundaries, along traces of active slip planes, and at points randomly distributed over the surface.

Test Results. Two etches, A and B, were tested. The first (Coates' material removal etch) was prepared by adding 5 volumes of ethylene glycol to 5 volumes of the saturated KOH, cooling to 25°, and then adding 1 volume of 30% H₂O₂. The second (B) was similarly made, except that 1.5 volumes of 30% H₂O₂ were used (the additional H₂O₂ was added to make the etch more vigorous in an attempt to get good dislocation etch pits). The etches were prepared just before using.

Fresh cleavage surfaces of n- and p-type material were immersed in the solutions at 35-40° for 2 min. Agitation was produced by moving the samples slowly back and forth. After removal from the solution, they were rinsed with water and carefully dried on lens paper.

At a magnification of 480X, many square, shallow, flat-bottomed pits were seen; only a small number were pyramidal. Some of the pits were so shallow that they were only barely visible. Yellow-brown stains were occasionally present on the surfaces of the samples. These were removed by dipping in 1:1 acetic acid for a few seconds and rinsing with distilled water. No material difference between the action of etches A and B was noted.

NORR.¹⁶ The etching solution is prepared by dissolving 10 g. of KOH in 10 ml. of distilled water and then adding 1.0 ml. of glycerol. After the solution has cooled to room temperature, 0.5 ml. of 30% H₂O₂ is added. Reagent grade chemicals are used throughout. Fresh cleavage surfaces of PbSe are etched in the solution for 2 min. at 25°, after which they are rinsed with water and carefully dried on lens paper. It was recommended that the surfaces to be etched be freshly cleaved because they are very susceptible to mechanical damage and chemical contamination.

The etch produced results (see Fig. 3) qualitatively very similar to those obtained on LiF by Gilman and Johnston¹⁷ (using their "A" etch) and on PbTe by Houston and Norr.¹⁸ Sharp, square, pyramidal pits were formed on both n- and p-type material. The pits were 2-6 μ across, and their edges were parallel to the <100> directions of the crystal. They were obtained along small-angle grain boundaries, along the traces of active slip planes, and at points distributed randomly over the surface. When previously etched material

was lightly deformed and etched again, new pits were produced which appeared to be due to dislocations introduced by the deformation. When the two surfaces formed by the cleavage of a crystal were etched, their pit patterns were approximate mirror images of each other, except in areas which showed traces of active slip planes.

D. Lead Telluride

COATES.^{7,8} The etching solution is prepared from 10 volumes of aqueous KOH (saturated at 20°), 1 volume of 30% H₂O₂ solution, and 10 volumes of ethylene glycol.

Tilly¹¹ used this etch on surfaces that had been freshly electropolished by a modification of Schmidt's procedure (see Sect. II-D-SCHMIDT (A)). It was recommended that both the polishing and etching be carried out in an inert atmosphere to avoid oxidation and to obtain considerably improved etching. It was reported that well-defined dislocation etch pits were obtained. A photograph accompanying the report shows such pits on an electrolytically polished (115) surface, and the report gives the impression that dislocation etch pits can be formed on all crystallographic planes by this method.

Test Results. In order to minimize the thermal decomposition of the H₂O₂, the etching solution was prepared by mixing and cooling the ethylene glycol and saturated KOH before adding the H₂O₂. It was prepared just before using. Fresh cleavage surfaces and freshly electropolished surfaces of n- and p-type PbTe were immersed in the solution at 25° for 5 min., rinsed with water, and carefully dried on lens paper.

Etching fresh cleavage surfaces of p-type PbTe produced sharp, square, pyramidal pits about 3 μ wide. The pits were formed along small-angle grain boundaries, traces of active slip planes, and at points distributed randomly over the surface. Their sides were parallel to the $\langle 100 \rangle$ directions. When previously etched material was lightly deformed and etched again, new pits were produced which appeared to be due to dislocations introduced by the deformation. These pits formed a grid of lines parallel to the $\langle 100 \rangle$ directions. Stained spots were occasionally seen on the surface.

Similar etching of fresh cleavage surfaces of n-type material produced like results, except that the pits were slightly smaller and the pit density was generally much larger. In some areas the density was so high that the individual pits could not be resolved. Etched n-type material tended to have more stained spots than the p-type.

Etching surfaces that had been freshly electropolished by the procedure given in Section II-D-NORR generally produced sharp, square, pyramidal pits, 2-4 μ wide on both n- and p-type material. The pits were formed along small-angle grain boundaries, traces of active slip planes, and at points distributed randomly over the surface. They were formed on surfaces approximately parallel to the cleavage planes, only. As the surfaces departed from $\{100\}$, the pits became distorted and finally disappeared.

Tilly's procedure, described above, was not tested.

HOUSTON AND NORR.¹⁸ Two etches were reported which are substantially the same chemically, since I_2 quickly forms IO_3^- in strong alkali.

The first is prepared by dissolving 5 g. of NaOH in 10 ml. of distilled water, and while the solution is still hot, 0.2 g. of I_2 is added. The final solution is colorless. Fresh cleavage surfaces of p-type PbTe are etched by treating them with the solution for 5 min. at 94-98°. They are then rinsed with distilled water and carefully dried on lens paper.

The second etch is prepared by dissolving 5 g. of NaOH in 10 ml. of 0.5% $NaIO_3$ solution. The etching procedure is the same as before. Reagent grade chemicals are used in both solutions.

The pits and pit patterns produced were qualitatively very similar to those obtained on LiF by Gilman and Johnston,¹⁷ using their "A" etch. They were sharp, square, and pyramidal, and their sides were parallel to the $\langle 100 \rangle$ directions of the crystal. Pits occurred along small-angle grain boundaries, traces of active slip planes and at points distributed randomly over the surface. When previously etched material was lightly deformed and etched again, new pits were produced which appeared to be due to dislocations introduced by the

deformation. When the two surfaces formed by the cleavage of a crystal were etched, their pit patterns were almost perfect mirror images of each other, except in areas which showed traces of active slip planes. (See Fig. 4.)

Test Results. A piece of n-type PbTe (mildly polycrystalline) was cleaved, forming two new surfaces. The first was etched with Houston and Norr's 'iodate etch,' following the method given above. The second was etched by the procedure of Coates, as previously described. The pits formed by Houston and Norr's etch were roundish, shallow, and poorly defined, while those by Coates' were sharp, square, and pyramidal. Also, the latter appeared to produce a higher pit density. The failure of Houston and Norr's etch on n-type material and the success of Coates' thereon have been confirmed by additional experiments.

NORR.¹² Either a fresh cleavage surface or a freshly electropolished surface may be etched by this process. In the latter case, a surface parallel to a cleavage plane is prepared and electropolished as described in Section II-D-NORR. Then (without removing it from the cell) the sample is etched by reducing the potential across the cell to 1 volt for 15 sec. After removal from the cell, it is rinsed with water, immersed a few seconds in 1:10 HNO₃, rinsed with distilled water, and carefully dried on lens paper.

If the surface to be etched is a fresh cleavage one, it is etched in the manner described above, except that the electrolytic polishing step is omitted.

This electrolytic etching procedure produced pits on p-type PbTe, but only on planes approximately parallel to the cleavage planes. The pits were generally square and pyramidal and were 3-13 μ across. As the surfaces departed from the {100} planes, the pits became distorted and finally disappeared. The diagonals of the pits were parallel to the <110> directions. Pits occurred along small-angle grain boundaries, traces of active slip planes, and at points distributed randomly over the surface. When previously etched material was lightly deformed and etched again, new pits were produced which appeared to be due to dislocations introduced by the deformation.

Test Results. A 'pulled' crystal of PbTe, containing p-n junctions, was ground, electropolished 15 min., and electro-etched 15 sec. by the procedures described above (see Sects. II-D-NORR and III-D-NORR). The pits formed were sharp, pyramidal, and fairly square. They occurred along small-angle grain boundaries, traces of active slip planes, and at points randomly distributed over the surface. Pitting on the n- and p-type areas appeared to be identical except that the pit density tended to be higher in the n-type. As the surface departed from the $\{100\}$ plane, the pits became distorted and finally disappeared. The distorted pits observed on this crystal are similar to those shown in Fig. 5.

Chapter IV

SUMMARY

Descriptions of chemical and electrolytic polishes and dislocation etches, together with tests made thereon by the author, have been given for SnTe, PbS, PbSe and PbTe. Five were developed by the author, two were obtained from private communications, and the remainder were found by a search of the literature.

The polishes and etches found most satisfactory are given in Tables 1 and 2.

A polish for PbS and a dislocation etch for SnTe are still needed. Also, improved polishes for SnTe and PbSe and a room temperature dislocation etch for PbS would be helpful.

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**This reference is now unclassified. (See letter from W. D. Lawson, Airborne Radar, Royal Radar Establishment, Great Malvern, Worcs., England, to Charles V. Kincaid, NOL Representative in the United Kingdom, dated 19 Aug 1963. A Xerox copy of this letter is filed in the office of the Solid State Division, Applied Physics Department, U. S. Naval Ordnance Laboratory, White Oak, Md.)

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Table 1

SELECTED POLISHES

Material	Recipe and Conditions	Reference To Text
SnTe	6 vols. glacial acetic acid 3 vols. concd. HNO_3 1 vol. concd. HF 25° Gently stirred 5 min. At the end of the 5 min., the polishing soln. is gradually diluted (with stirring) with 2X its vol. of glacial acetic acid.	pp. 2-3
PbS	Nil	
PbSe	40 ml. glycerol 10 ml. glacial acetic acid 10 ml. concd. HNO_3 50° Anode: PbSe Cathode: Pt foil Cell potential: 6 v. Cell current density: 0.30 amp./cm²(initial) 0.10 amp./cm²(final) 2.0 min. The thick film formed during electro-polishing is removed by swabbing with cotton soaked in satd. KCN.	5-6

Table 1 (Cont.)

Material	Recipe and Conditions	Reference To Text
PbTe	45 ml. distilled H ₂ O 20 g. KOH pellets 35 ml. glycerol 20 ml. ethanol 25° Anode: PbTe Cathode: Pt foil Cell potential: 6 v. Cell current density: 0.2 amp./cm ² Stirred 5-15 min.	pp. 9-10
	<u>Step No. 1</u> Rub sample on Linde A abrasive on twill jean cloth satd. with a soln. composed of 50 ml. glacial acetic acid 50 ml. 30% H ₂ O ₂ 5 g. tartaric acid 25° 2-3 min. <u>Step No. 2</u> Rub sample on twill jean cloth satd. with the above soln., omitting the abrasive. 25° 2-3 min.	10-12

Table 2

SELECTED DISLOCATION ETCHES

Material	Recipe and Conditions	Reference to Text
SnTe	Nil	-
PbS	1 part concd. HCl 3 parts aq. thiourea (100 g./l.) 60° 1-10 min.	pp. 14-15
PbSe	10 ml. distilled H ₂ O 10 g. KOH 1.0 ml glycerol 0.5 ml. 30% H ₂ O ₂ 25° 2 min.	16-17
PbTe	10 vols. aq. KOH (satd. at 20°) 10 vols. ethylene glycol 1 vol. 30% H ₂ O ₂ 25° 5 min.	17-18

Table 2 (Cont.)

Material	Recipe and Conditions	Reference to Text
PbTe (cont.)	45 ml. distilled H ₂ O 20 g. KOH pellets 35 ml. glycerol 20 ml. ethanol 25° Anode: PbTe Cathode: Pt foil Cell potential: 1 v. Stirred 15 sec.	pp. 19-20

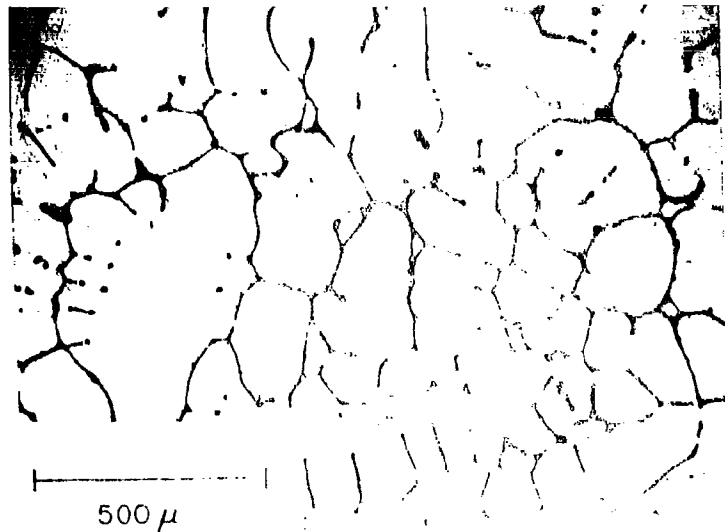


FIG. 1 RIDGES FORMED BY FAUST AND CASALI'S POLISHING SOLUTION ON A p-TYPE TIN TELLURIDE CASTING.

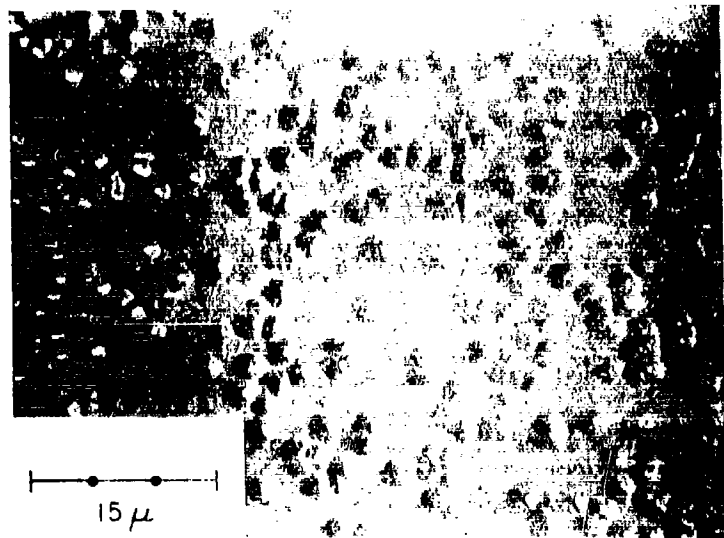


FIG. 2 DISLOCATION ETCH PITS FORMED BY BIRCHBICK AND SCANLON'S ETCHING SOLUTION ON THE SURFACE OF n-TYPE LEAD SULFIDE. MOST ARE TRIANGULAR AND POSSIBLY EIGHT-SIDED PITS.

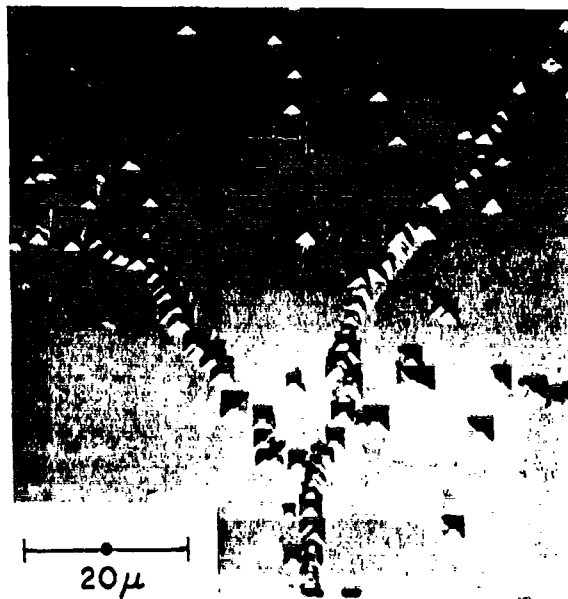


FIG. 3 DISLOCATION ETCH PITS FORMED BY NORR'S
ETCH ON A CLEAVAGE SURFACE OF n-TYPE
LEAD SELENIDE.



FIG. 4 DISLOCATION ETCH PIT PATTERNS ON THE TWO SURFACES FORMED BY THE CLEAVAGE OF A p-TYPE LEAD TELLURIDE CRYSTAL. THE SURFACE WITH THE SMALLER PITS WAS ETCHED BY HOUSTON AND NORR'S 'IODINE ETCH' AND THE ONE WITH THE LARGER BY THEIR 'IODATE ETCH'.

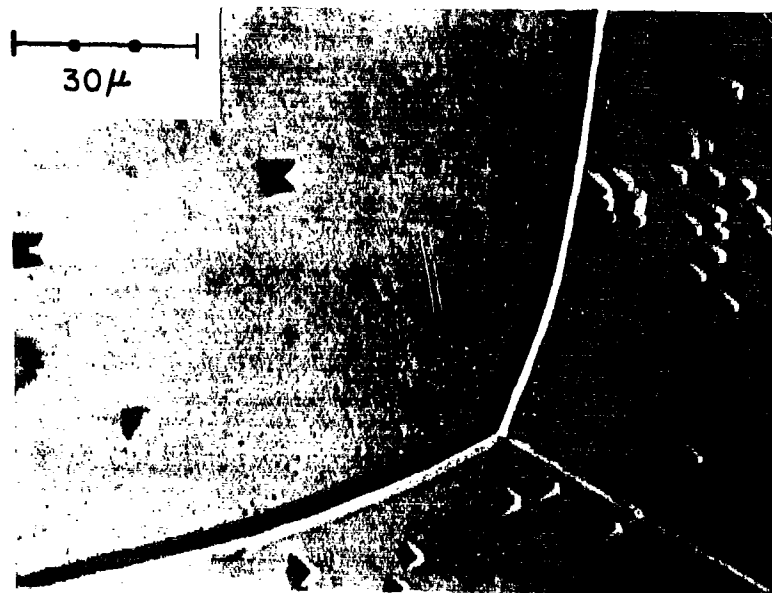


FIG. 5 JUNCTION OF THREE GRAINS IN A p-TYPE LEAD TELLURIDE CRYSTAL. THE SURFACE OF GRAIN A IS PARALLEL TO THE (100) PLANE, WHILE THE SURFACES OF GRAINS B AND C DEVIATE SOMEWHAT FROM THIS PLANE. THE CRYSTAL SURFACE WAS PREPARED BY GRINDING ON NO. 600 GRIT SILICON CARBIDE PAPER LUBRICATED WITH WATER, FOLLOWED BY ELECTRO-POLISHING AND ELECTROETCHING BY NORR'S PROCEDURE.

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