NATIONAL BUREAU OF STANDARDS REPORT

9482

TAGGING OF METAL OBJECTS

By

Theodora A. Welsko
David B. Ballard
Melvin R. Meyerson

To

United States Arms Control and Disarmament Agency

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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I. Introduction

The United States Arms Control and Disarmament Agency expressed a need for a means of tagging items in delivery systems which are frozen in number and characteristics. Two types of tags were required: (1) a type that could be applied directly to the system by observers "in situ"; and (2) a type that would be given to custodians by observers for application by custodians, termed "transmittable." The nature of the transmittable tag requires that it be easily and quickly identified as the tag designated for a particular piece of hardware at some date in the future. However, the non-transmittable tag is best defined as a fingerprint of a specific area on a section of the hardware which cannot be removed without destroying the integrity of the hardware; nor can the tagged section be safely transferred to a different item of hardware. This would prevent forgery by moving a known or suspected fingerprinted segment from one item to another. The fingerprint, by its very nature, could not be duplicated in any known way by man.

The feature that has been chosen as the fingerprint

characteristic is the microstructure or, in some cases, the macrostructure of a piece of metal in a precisely defined location. Microstructure is defined for purposes of this report as the structure of a metal as viewed at magnifications above X10. Macrostructure is defined as the structure seen at magnifications less than X10. The general microstructural characteristics of a metal component depend on the composition of the material, heat treatment given it, and the method by which the component was formed. But the specific characteristics of a metallic microstructure as defined by grain size and shape, phases, inclusions, etc., in one location on a particular component is unique. No two metal grains are likely to have exactly the same size, shape, and relationship to the surrounding grains. In addition, particles of various phases and inclusions will not be found in exactly the same configuration in any other area. An example of a microstructure with these unique features is shown in Figure 1. The specimen is cast iron, and the distincitive features of the microstructure are the white cementite particles, the dark gray graphite flakes and the gray and white areas that look like fingerprints. The "fingerprint" structure is a phase called pearlite. Examination of any area on the same specimen will reveal pearlite, cementite, and graphite flakes; but, in no other area on the specimen or on any other specimen

of cast iron will the areas of cementite, pearlite, and graphite have the same size, configuration, or relationship to each other. This microscopic area identifies the specimen on which it is found as assuredly as a fingerprint identifies a specific person.

Included in this report are studies of materials and components that were obtained from the Martin Company and from Aeroject General Corporation. These were examples of materials and components that have been used on items in missile delivery systems. Of the materials submitted, extensive studies were made on 7075 aluminum, a wrought aluminum alloy; on type 347 stainless steel, a ferritic alloy; and on Ti-6Al-4V, an alpha-beta titanium alloy.

Although the microstructure in a fingerprint area could not be reproduced intentionally, those characteristics which serve to identify the hardware could be altered or destroyed. Under extreme conditions of environment, such as exposure to extreme heat or to a corrosive atmosphere for an extended period of time, the fingerprint characteristics might be changed. However, if the identifying features of a fingerprinted area should be destroyed in this manner, most likely the same conditions that destroyed the features of the fingerprint would adversely affect the remainder of the hardware. It is important that the inspectors choose areas on the

various items of hardware which are critical enough that the environmental degradation of a fingerprint would be synonymous with the impairment of the entire item of hardware.

The fingerprint may also be destroyed by deliberate marring. Such a procedure does not seem to be in the best interests of anyone and therefore does not constitute a serious problem.

II. Technology — Methodology — Techniques Involved

The general techniques involved in examining and recording metallic microstructures are tools that metallurgists have been using for many years in the laboratory. Several modifications were employed to enable hardware to be inspected without removal from its site. Before an area can be examined, its surface must be prepared. Preparation involves polishing the metal surface; which can be done by mechanical, chemical, or electrolytic means, or by a combination of them; and subsequent etching of the polished surface, either chemically or electrolytically. With some materials, it is not necessary to etch the metal surface; the unetched surface, when it is properly polished, contains enough features to make a good fingerprint. The identifiable characteristics revealed with the aid of optical microscopy after suitable preparation include the size, shape, and configuration of grain boundaries, inclusions, precipitates, and particles of differing phases.

Preparation of a small spot on the surface of a large object at its site need not be difficult. Polishing and etching procedures, if necessary, can be carried out by one person either by hand or with the aid of portable equipment. Once a spot is satisfactorily prepared and located, it either can be photographed with the use of a portable microscope or

replicated with the use of a organic solvent and cellulose tape. The resultant photomicrograph or replica can then be taken to the laboratory to be studied and recorded for future comparisons. The degree to which one can identify fingerprint areas depends entirely on the magnification at which the characteristic structure is best identified. Some fingerprint characteristics, such as the weldment which will be discussed later, could be identified at a magnification of ten diameters; most fingerprints, however, will be of polished areas which can best be identified at magnifications in the range of one-hundred to five-hundred diameters. In any case, a photomicrograph or replica displaying the characteristics of the microstructure of an object in a defined area would serve as a permanent record of the area studied and, by extension, a permanent record of the object of which that structure is a permanent part.

III. Preparation of Metal Objects "In Situ"

To prepare and examine metal objects "in situ" certain modifications of the grinding, polishing, and etching techniques normally used in metallography are required. These changes are readily accomplished with the use of commercially available equipment listed in Table 1.

Polishing specimens for metallographic examination in the laboratory begins with hand grinding the specimen on silicon carbide abrasive papers of progressively finer grit. This procedure must be duplicated in the field, except that the selected area would be more efficiently ground by machine. The Portamet Spot Polishing shown in Figure 2 is well suited to the kind of grinding necessary here. Either abrasive discs of silicon carbide impregnated paper or a coarse grit diamond polishing compound on a disc can be used.

In many ways, diamond grinding and polishing compounds are preferred for field work. The cutting action of the diamond particles reduces the amount of heat that is evolved during grinding and polishing. In addition, techniques using diamond are usually much faster than procedures using other abrasive materials. The grinding operation should remove any deep surface scratches or oxidation that might be present. The resultant surface should be uniformly scratched and have a metallic lustre.

After completion of the grinding step, final polishing can be done in several ways. The simplest, but not necessarily the quickest, would be to continue mechanical polishing with still finer abrasives. Abrasives and lubricants normally used for final polishing include magnesium oxide in slightly acidified distilled water, aluminum oxide in distilled water, and diamond polishing paste in kerosene or mineral oil. Again, the Portamet can be used with good results. Whenever mechanical polishing or grinding is done, it is essential that the working surface be thoroughly cleaned of all metal particles and excess abrasive accumulated from the prior step before the next finer abrasive is used. Cleaning a small area on a large surface can generally be accomplished with the use of a plastic squirt bottle and cotton swabs. An area should be swabbed with water or an organic solvent until it looks clean, and then two or three times more.

Chemical polishing, in which chemical action is used to dissolve uneven surfaces, functions very well if the chemical composition and mechanical condition of the material are known precisely and the surface has been properly ground. Under these circumstances, the solution rate and the final results are predictable; otherwise they are not. In those instances where information is not available concerning the exact composition and treatment of materials used in the hardware,

chemical polishing should not be used. Chemical etching, however, can still be put to good use. The action of swabbing a polished area with an appropriate chemical solution, usually quite dilute, will result in the dissolution of some phases faster than others. In a single phase material, the grain boundaries are attacked much faster than the interiors of the grains. The resultant microstructure will clearly delineate the separate phases and grains. In order for chemical etching to be effective, it is usually necessary to know only the main components of the alloy.

Electro-polishing is a quick method of polishing most materials and, unless the presence of a low electrical voltage is objectionable, it would be the most suitable method. Electro-polishing may be considered as electro-plating in reverse. An electrolyte suitable for the material to be polished is chosen; the area to be polished is made anodic to the polishing device and metal ions flow from the metal surface, through the electrolyte, to the polishing device, which is cathodic. What remains at the anode is a smooth, polished surface. Since the voltage and current density are critical, an electropolishing device such as the Ellopol (shown in Figure 3) can be used to great advantage. Generally, electropolishing is far less time consuming than mechanical polishing, and the area that has been polished can be quickly

etched by lowering the voltage to approximately one-tenth of the polishing voltage.

The quality of the final surface depends on the effectiveness of each step in the preparation procedure, and especially on the earlier grinding steps, as is shown in the series of photographs of the alpha-beta titanium alloy of Figure 4. The surface shown in 4(a) was hand ground using silicon carbide abrasive papers. Hand grinding was quite tedious and, obviously, not too effective. 4(b) was polished with the Portamet using only one grinding step with a very fine silicon carbide paper. 4(c), which is a virtually perfect area, was polished using a coarse and then a finer abrasive on the Portamet. All three areas were subsequently polished and etched electrolytically with the Ellopol.

Close inspection of some hardware similar to that which will be encountered in the field indicated that some areas of the hardware involved could be fingerprinted without any surface preparation at all. Tool markings such as those shown in Figure 5 or the pattern formed by the solidification of weld or brazing material, as is illustrated on the torus shown in Figure 6 can be observed with the naked eye and replicated or photographed with the aid of a hand carried camera "in situ." Other areas, such as the attach bracket shown in Figure 7 could be fingerprinted with very little

preparation. In the laboratory, an attempt was made to examine areas A and B, shown on the bracket. The Portamet was used to grind these until they were in sufficiently good condition to proceed with electro-polishing. After electro-polishing, both areas were pitted too badly to make a good fingerprint. Accidentally, the electro-polishing wand wandered over to area C, around the hole that had been drilled for a threaded section. The photomicrograph in Figure 8 shows the result, a perfectly prepared, cast aluminum section. Cast materials are notorious for the ease with which pits are formed in them during metallographic preparation.

In order to prepare areas on hardware for metallographic fingerprints, "in situ", inspectors should be familiar with the preparatory characteristics of the materials that are likely to be encountered. Most of the materials will be high strength, light weight, corrosion resistant alloys. The following paragraphs discuss peculiarities in the behavior of three groups of materials that are likely to be met. Figures 8 and 9 show microstructures of some materials that are likely to be encountered.

(1) Wrought aluminum alloys. These are the easiest of the aluminum alloys to prepare. Grinding can usually be done quickly, because the wrought aluminum alloys seldom develop corrosion pits. Instead, a thin, even oxide coating forms immediately on exposure of aluminum to the atmosphere; the

oxide often interferes with etching procedures. For the purposes of this investigation, etching of the wrought aluminum alloys was not necessary; because the alloys contain numerous particles of various phases that can be observed and fingerprinted without etching.

The aluminum alloys can be ground and polished with diamond compound starting with a coarse grit and finishing with the finest, quarter-micron size, or they can be effectively finished with a thick slurry of magnesium oxide powder dissolved in distilled water to which a small amount of hydrochloric acid has been added. The addition of the acid prevents the formation of magnesium carbonate by the combination of magnesium oxide and carbon dioxide taken from the air; magnesium carbonate is an extremely hard, coarse abrasive.

(2) Stainless Steels. There are three groups of stainless steel alloys, austenitic, ferritic, and martensitic, each of which has very distinct characteristics, and it is not likely that an inspector would know whether the alloy were ferritic or martensitic. The austenitic type could be distinguished from the other two with the aid of a small magnet. Such distincition could be made in the field with a portable hardness tester if the martensitic stainless steel had been hardened, since its hardness is greater than that of

the ferritic steel. Austenitic stainless steel is non-magnetic or very slightly so, whereas the other two types respond strongly to a magnet. All steels can be effectively ground and polished with diamond paste. Because the alloys are harder than aluminum alloys, the various polishing steps take longer, but a good final polish is not difficult to obtain. Etching procedures vary somewhat with the various types of stainless steels; in keeping with this fact, the resultant structures vary considerably. Alloys of the single phase, austenitic or ferritic types have large grains which differ only in size and shape. However, microstructures of hardened martensitic stainless steels are so complex and difficult to resolve that it would be advisable to use inclusions that are visible in the unetched condition as fingerprint characteristics. Most of the stainless steel alloys are so corrosion resistant that electrolytic etching is essential. Once the area is polished and etched, the high degree of inherent corrosion resistance will inhibit deterioration of the revealed microstructure over long periods of time. In the experimental work, the stainless steels were most frequently etched with a 10% solution of oxalic acid or with Marbles reagent which contains hydrochloric acid and cupric chloride.

(3) Titanium alloys. Titanium alloys are quite difficult to polish by mechanical means. They tend to pit badly

and smearing of the surface during the grinding and polishing steps causes the microstructure to become obscured. For this reason, it is advisable to polish and etch titanium alloys electrolytically after they have been carefully ground mechanically. As with the stainless steels, the corrosion resistance of the alloys makes them difficult to etch chemically by any except very strong acid solutions.

After an area has been selected and prepared for metallographic examination, the problem of obtaining an identifiable fingerprint may be solved by either of two techniques. A photographic negative can be made with the aid of a portable microscope which has an eyepiece camera attachment or plastic replicas can be made of the entire fingerprint area. The replicas could then be processed and photographed in a laboratory removed from the site of the hardware with a conventional metallograph.

IV. Obtaining a Photomicrograph "In Situ"

A small, lightweight microscope that could be mounted on a tripod and used in any position is essential. The experimental work was done with the portable microscope shown in Figure 10, which must be used on a horizontal surface; however, it would not be difficult to adapt a microscope of this type for vertical viewing. Once the fingerprint area was located and brought into focus, the eyepiece of the microscope would be removed and replaced with a camera attachment in which the film has the same focal plane as the eyepiece lens of the microscope. The eyepiece camera shown with the microscope can be used only with Polaroid film, but similar camera attachments are available for use with small size sheet film or with conventional roll film. The instrument contains its own light source and therefore must have a source of suitable electrical current, 110 V.A.C. as now constructed. It is important that the location of the fingerprint area be precise if photography "in situ" is to be used, because a 4-inch by 5-inch negative of an area photographed at 100 diameters will cover an area of only 0.04 inches by 0.05 inches and a 35 mm roll film negative will cover still less area. Therefore, for subsequent examinations of the same spot, this size of spot must be located precisely. Techniques for relocation are described in section VI.

The photomicrograph shown in Figure 11 was originally taken using the eyepiece camera shown and Polaroid film. The area photographed is the chemically milled surface of a wrought aluminum alloy. No attempt was made to prepare the surface in any way. The structure observed is the pitted surface that is produced by the chemical milling process. This type of surface would be an adequate fingerprint when viewed at magnifications of 50X or greater.

V. Replicating "In Situ" - Identification by Replica Technique

The use of a soft material pressed against the master or original surface to make a copy is not new. It is essentially the same process used in making phonograph records, coins, plastic parts, die castings, etc. The use of a thin plastic replica for electron microscopy provides for the examination of surfaces at high magnification with good fidelity. It also provides an excellent means for identifying surfaces at lower magnification. It enables a field inspector to easily and quickly duplicate a surface in the field and bring the replica back with him for subsequent examination in a laboratory. It has the additional advantage of being able to reveal surface features in areas inaccessible to a microscope or camera. The following technique is a modification of cellulose acetate negative replication.

Plastic Replica Technique

This replication technique can be used to duplicate the surface details of many materials that are not soluble in the solvent in which the plastic is dissolved. The materials investigated were not affected by the solvent. The following steps have been found to produce satisfactory specimen replicas of materials and surfaces found in missile structures.

(1) Clean the surface with acetone or other solvent to

remove paints, loose particles, oils or moisture. A further cleaning step would be accomplished by discarding the first replica. By so doing, the plastic replica will remove loose dirt particles.

(2) Cut a piece of plastic film about 1-inch square. While holding the plastic with tweezers, moisten the film with a few drops of acetone, shake off the excess, add more acetone and let soften until the film bends under its own weight. This time interval of this procedure depends on the thickness of the film; with 5 mil tape, it generally requires 1/2 minute.

(3) If possible, a drop of acetone should be put on the surface to be replicated just prior to placing the softened film against it. Good contact is obtained by gently pressing the film with an art gum eraser having a piece of saran wrap wrapped around it. Small entrapped air bubbles should be removed by pressing towards the outer edge. Remove eraser and saran wrap after approximately 30 seconds.

(4) Allow film to dry for at least 45 minutes, drying time may be shortened if a warm air supply is carefully applied to the film. After the film is dry remove it by gently lifting on the corners. On longer drying times it may detach by self-contraction. Approximately 3-5% shrinkage has been noted as the film dries. Small bubbles may form during drying if the specimen temperature increases during the

procedure.

(5) Attach the film, impression side up, to a glass microscope slide with scotch tape. The mounted films may be stored in the microscope slide box until preparation for metallizing. Length of storage under ambient conditions seems to be infinite.

(6) The mounted replicas may be metallized (at 90^{o} with respect to surface) with aluminum in a metal evaporator. The replica surface then can be examined by eye or by optical microscope at magnifications up to 500X.

A completed replica of a weld is attached as Figure 12a. A photograph of the weld is shown in 12b. The crystalline structure and arc strikes in the weld area may be compared directly. In Figs. 13a and 13b these same features may be seen in great detail at slightly higher magnification.

Figures 14a through 17b compare surfaces of several materials on a macroscale as reproduced by macrophotography and replicas. Faithful replication of fine microstructural details on a polished and properly etched metal surface are shown in Figs. 18a and 18b.

The advantages of replicas as a means of reidentification of materials and surfaces representative of missile structures are many and are considered to be extremely important. The simplicity of the technique enables inexperienced personnel

to make satisfactory replicas after a few hours training. The kit of inexpensive materials required consists of 1) a roll of cellulose acetate plastic tape, 2) a small bottle (2 oz.) of acetone with dropper, 3) art gum eraser, 4) small scissors, 5) 1 x 3 inch microscope slides, 6) microscope slide box, 7) tweezers. All of the above items are readily available and the total cost should not exceed $5.00. An obvious advantage is the light weight and portability of several hundred replicas. A unique feature is the ability to replicate a surface that isinaccessible by direct observation, (photography or microscope). The total time required for one replica (depending on the solvent used and thickness of plastic) should not exceed one hour, but this does not include the time spent in removing paint or replacement of a coating. On bare metal surfaces, the most desirable surface, there is no alteration or damage to even highly polished surfaces. After shadowing with a metal, the finished replica can be compared stereoptically with the original surface if the area has sufficient accessibility for a small folding stereoscope. Also, the bare plastic replicas may be mounted between glass and projected as a slide for audience viewing.

The disadvantages of the replica process would of course depend on the plastic and solvent used. Most quickly drying

solvents such as acetone are inflammable, toxic, or on painted surfaces the solvent might dissolve the paint. Satisfactory replicas cannot usually be obtained from objects completely submerged, because of the drying process required. If precise measurements are to be made from the replicas, allowance for shrinkage must be made. A small inconvenience is the reversal of the features of the replica as compared with the original surface, i.e., indentations on the component are hills on the replica. The temperature limit of cellulose acetate is approximately 70C. Above this temperature fine details of the prior surface are obliterated. Two other plastics, cellulose acetate butyrate and cellulose acetate propionate were tried. These produced satisfactory replicas, but the solvents used required longer drying times, (2 hours). Polyethylene may be moulded by heat, but in order to retain the fine details it should be subsequently cooled to room temperature under pressure to give intimate contact with the original surface.

VI. Selecting and Relocating the Fingerprint Area

Selecting the area to fingerprint is one of the most indefinite and difficult tasks that faces the inspector. Areas on attachment brackets, nozzles, and engine assemblies are among the most suitable for identification, provided that those brackets, nozzles and engine assemblies are permanently fixed to the piece of hardware they will serve to identify. If they can be removed and attached to another piece of hardware, then the fingerprinting process will become useless. If data is available concerning the size, shape and type of materials used in the various pieces of hardware, the inspector will be able to select areas to be fingerprinted in advance and the problem will be simpler. However, if no such data is available, it will be extremely difficult for the inspector to select appropriate areas without some knowledge of the materials and fabrication processes that are likely to be used in manufacturing hardware such as he must fingerprint.

After an area has been selected and fingerprinted, it is essential that future inspectors be able to locate the same area. Fingerprinted areas can be located by at least three methods: 1) measurements from fixed points can be given to define the fingerprinted areas, 2) the hardware can be marked with a punch or a scribe to indicate which areas have been fingerprinted, 3) an overall photograph of a large portion of

the missile can be made to include the fingerprint area, and the area marked on the photograph. On an object that is nearly symmetrical, it is sometimes quite difficult to describe small areas verbally. However, symmetry can be used advantageously if it is interrupted by one or more irregularities. For example, consider a missile of very large diameter that is cylindrical in shape over most of its length. It will almost certainly have a bracket to which instruments will later be attached or a hole through which they will be mounted on the interior of the missile. Any deviation from symmetry can define a point from which the missile may be divided into quadrants with the deviant at the north pole. Fingerprint areas can then be located by giving measurements in degrees or in inches about the circumference and by specifying how far above or below the point of reference they are located. With a system such as this, there is a distinct advantage in using exactly the same areas as fingerprints for all of the hardware of one type. A template could be made to locate this spot on any one type of hardware.

An alternative method is to actually put small marks on the hardware. A spring loaded punch or a small scribe such as that shown in Figure 19 can be used. This method is more exact in that the marks could define a microscopic area that would be precisely the area that had been fingerprinted. It

is particularly suitable if "in situ" photography is to be used instead of the replica technique.

VII. Effect of Time on Prepared Areas

A study was made on change occurring in a fingerprinted area between the time that it is first prepared and fingerprinted and the time when another inspector attempts to verify the spot at a later time. Specifically, a study was made to determine what effect a normal indoor atmosphere would have on polished specimens of materials used in hardware construction. No attempt was made to study these materials in extremely corrosive environments or at very high temperatures, because hardware of the type that will be fingerprinted is generally handled and stored with extreme caution. Figure 20 shows before and after photographs of two of the alloys, 7075 aluminum and 347 stainless steel. All of the materials were sufficiently corrosion resistant that little or no change in appearance was observed after several months exposure.

VIII. Effect of Repolishing Prepared Areas

In the event that some corrosion should occur, or if the identifying area should become badly scratched, the area could be repolished slightly and an attempt made to relate it to the area previously observed. The extent to which the same area can be detected after surface layer removal depends on the characteristics of the material in question. Generally, the size of the identifying features that are observed determines the depth to which the material can be repolished and still be identified as being the same area that was previously observed. Depth studies of wrought aluminum, ferritic stainless steel, and an alpha-beta titanium alloy are shown in Figures 21, 22 and 23. A knoop hardnessindentation was used to indicate the area that was to be examined for structural changes at deeper surfaces. It was the same area used for the time tests. After the specimen was initially polished and photographed, it was exposed to an indoor atmosphere for several months. Because no attempt was made to protect its surface mechanically, fine scratches and water spots were observed at the second examination. Before any structural changes were studied, an attempt was made to polish away the deeper scratches. In order to do this, it was necessary to polish 0.00024 inches of material from the sample. Comparison of the first and third photomicrographs on the left in

Figure 21 will reveal that the sizes and shapes of the light gray second phase particles change considerably in this depth. However, if one observes the black particles, which are inclusions, it becomes obvious that both areas are the same. Although the inclusions, too, changed in size and shape slightly, their relationship to each other is fingerprint enough for identification.

Subsequently, the specimen was polished in five-minute intervals using magnesium oxide in acidified distilled water on microcloth. An automatic polishing device was employed to insure that the polishing variables remained consistent. Figure 21 shows photomicrographs of the variations in microstructure observed after polishing for 10 minutes (0.00029" deep), 20 minutes (0.00039"), and 30 minutes (0.00048").

Similar procedures were used in the depth examinations of ferritic stainless steel (Figure 22) and Ti-6Al-4V (Figure 23) alloys. At first the stainless steel sample was etched with oxalic acid solution; but because the results could not be duplicated accurately, etching was discontinued. In the 347 stainless steel studied, inclusions proved to be better fingerprints than grain configurations. Polishing was done by hand using 1/4 micron diamond paste on microcloth for one minute intervals.

It was necessary to etch the titanium alloy, Ti-6Al-4V, with Keller's etch, a dilute mixture of hydroflouric, nitric, and hydrochloric acids, before an easily identifiable fingerprint could be seen. The polishing, in five minute intervals, was carried out on the automatic polisher with no additional load using the finest aluminum oxide abrasive and distilled water on microcloth.

IX. Comparison of Photographs and Photomicrographs

Photographic negatives or positives taken at different times can be superimposed on each other to identify the areas as the same. If they are of the same area and magnification, they will fall into alignment, even though the sizes and shapes of some features will have changed. Those features that have disappeared, as well as new features that have appeared, will be outlined. An instrument such as a stereoscope can be used for a less quantitative identification. Two negatives, placed side by side in a stereoscope will be superimposed on each other by a system of mirrors and lenses. Their similarities and differences will become evident as soon as they are aligned. If the depth of the surface removal in a particular area does not exceed the limit beyond which the fingerprint features in that material can be identified, then the extent to which the area can be identified depends on the accuracy of the photographic techniques used.

A refinement of the stereoscope, called the Hinman Collator, would be useful for fast comparison of two photomicrographs taken at separate times. It can be used either with negatives or prints. In the Hinman Collator, illustrated in Figure 24, one views two photographs through two separate optical systems—one with each eye. Lights blink on and off,

alternating from one photograph to the other. If both photographs are exactly alike, and properly aligned, no variations will be observed. However, differences that are present between the photographs become immediately obvious and appear to be in motion. An inclusion on one photomicrograph that was not contained on the other would appear to be a flickering dot. With a little practice on this equipment, positive identification could be established even if slight changes in structure have been produced by repolishing. The manufacturer has expressed a willingness to design and build a desk model version of this apparatus.

X. Transmittable Tags

A complex weldment of several different nickel and stainless steel alloys has been selected for use as a transmittable tag. These particular materials were selected primarily because they were on hand, but other combinations would be equally effective, provided that they are corrosion resistant and easy to weld to one another. The weldments shown in Figure 25 are two different arrangements of the same four rods—1 1/8 inch diameter 16-2 stainless steel (martensitic), 3/4 inch diameter "K" Monel with a knurled surface, 5/8 inch diameter 13-2 stainless steel (martensitic), and 5/8 inch diameter 304 stainless steel (austenitic) with a threaded surface. These were welded with Inco-Weld "A" Electrode, a nickel-chromium-cobalt welding material.

When rods such as these are welded together, the weld metal forms a distinctive pattern as it solidifies along the length of the rod. This pattern is positive identification in itself. When the rods have been welded into a bar and sliced, no two slices will have the same configuration when viewed in cross section. The sliced weldments can be metallographically polished and etched with chemical reagents to reveal the microstructure. This microstructure, plus the configuration of the assembly, plus the surface characteristics of each bar make an absolutely non-reproducible combination.

The degree of the accuracy with which one can identify the weldments depends on the magnification at which they have been photographed. The series of photographs shown in Figures 25 and 26 illustrates this.

Although no identification other than the structure of the tags would be necessary, a convenient method of identifying such tags for the inspectors would be to stamp serial numbers of several digits on them. These numbers would then serve as marks to define areas for micro-identification purposes. For example, if "A1064" were the serial number stamped on a tag, the areas bounded by A, 0, and 6 could be selected as identification areas. Photomicrographs of these areas could be recorded and compared with micrographs taken at a later date to verify the identity of that specific tag.

A more subtle means of identifying the tags would be to stamp serial numbers on their surfaces and then grind away the stamped surface. Then, although no serial number would be visible to the naked eye, if the surface were treated with an appropriate etchant, the numbers that had been previously stamped on the tag would reappear. When the tag is originally stamped, the surface is marked, but the metal underneath the stamping is also work hardened. Etching the metal surface after the visible stamping marks have been removed causes the

work hardened areas to become darker; they contain more energy, therefore they react to the etchant faster than the surrounding material. If this method is used, it is suggested that softer materials be selected for the weldments. No illustrations of the technique are shown, because, under laboratory conditions, serial numbers could not be punched in the nickel-stainless steel alloys with sufficient force.

XI. Equipment Survey

All of the equipment used in the simulated field work is commerically available (Table 1). Most of it can be obtained from several supplies of metallurgical laboratory equipment; therefore, where a supplier's name is given in Table 1 it was the source of supply used and not necessarily the only source. The exception is item 6, Hinman Collator, which is personally built and sold only by Mr. Johnson.

XII. Summary and Conclusions

The fingerprinting methods described herein are deemed to be a feasible means of tagging metal objects "in situ." The methods outlined, when they are applied properly, are considered foolproof, positive methods of identification. The features that have been chosen as "fingerprints," particularly those characteristics which can be seen only at micromagnifications, cannot be forged by man.

Techniques have been developed in the laboratory for the preparation and fingerprinting of metal objects that are considered feasible for "in situ" examinations. During the experimental work, every attempt was made to simulate "in situ" conditions. However, it is felt that all possible problems concerning this type of hardware cannot be anticipated and that the techniques described ought to be field tested before they are applied on a widespread basis. The techniques have been developed keeping in mind the fact that they might be applied by personnel without formal university training in the art and science of metallography. The required skills can be taught to untrained inspectors in from 4 to 6 weeks. The use of the replica technique is viewed as a breakthrough in inspection, for with a minimum of equipment inspections can be made quickly and easily in areas that otherwise would be inaccessible or difficult by other means.

The use of a complex weldment as a transmittable tag is considered the simpliest possible means of identifying a tag absolutely and very quickly. There is no doubt that an inspector could verify the identity of such a tag "in situ."

Table 1. Equipment Described other than that Used for Replication Studies.

	Item	Number Required	Manufacturer	Estimated Cost	Remarks
1.	Portamet spot polisher including power pack, and polishing discs (silicon carbide papers and microcloth).	1	Buehler Ltd. 2120 Greenwood St. Evanston, Illinois	$950	Additional acessories for using the spot polisher in a vertical position are available. Item can be battery operated.
2.	Ellopol electroytic polishing apparatus, including power pack.	1	Schueler & Company 250 West 15th St. New York 11, N. Y.	$850	Can be adapted for battery operations.
3.	Portable microscope	1	Bausch & Lomb Optical Co. Rochester 2, New York	$400	Can be adapted for battery operation.
4.	Eyepiece camera equipped with Polaroid-Land cameral back. A .35 mm cameral back.	1	Bausch & Lomb Optical Co.	$302 A. $40	
5.	Stereoscope	1	Bausch & Lomb Optical Co.		
6.	Hinman Collator	1	Arthur W. Johnson 1600 Grace Church Road Silver Spring, Md.	$6000	Conversations with Mr. Johnson indicated that he would be willing to design a smaller, portable version if this were desirable.

Table 1. Continued

Item	Number Required	Manufacturer	Estimated Cost	Remarks
7. Metallographic Preparation materials.	3	Amplex Corporation	$100	
a. Diamond polishing compound in 5 gram applicators. 1/4 micron size; 3 micron size; 9 micron size				
b. Alumina abrasive powder, .3 micron	8 oz.	A. Buehler	$2.50	
c. Magnesium oxide powder	8 oz.	Fisher Scientific Ins.	$4.00	
8. Photographic supplies				
a. Polaroid film 2 1/2 x 3 1/4" size Type 37	12 rolls	Polaroid-Land Corp.	$18.00	

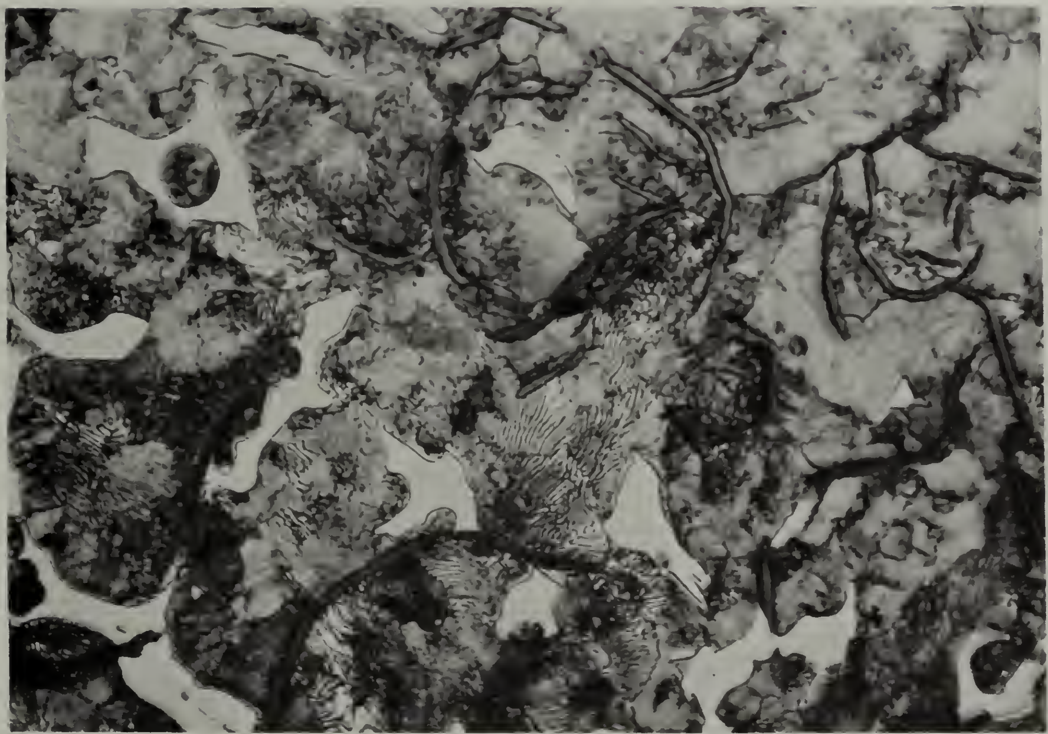


Figure 1. A "fingerprint" of a cast iron sample. On no other sample will the white cementite phase, the dark graphite flakes, and the lamellar pearlite (which most resembles a "fingerprint") have the same size, shape, and interrelationship. Photographed at X 500, after etching with 1% nital.

Figure 2. The Portamet Spot Polisher is light weight and versatile in its ability to mechanically polish various surfaces. The center shaft both rotates and inscribes a circle which becomes the polished area.

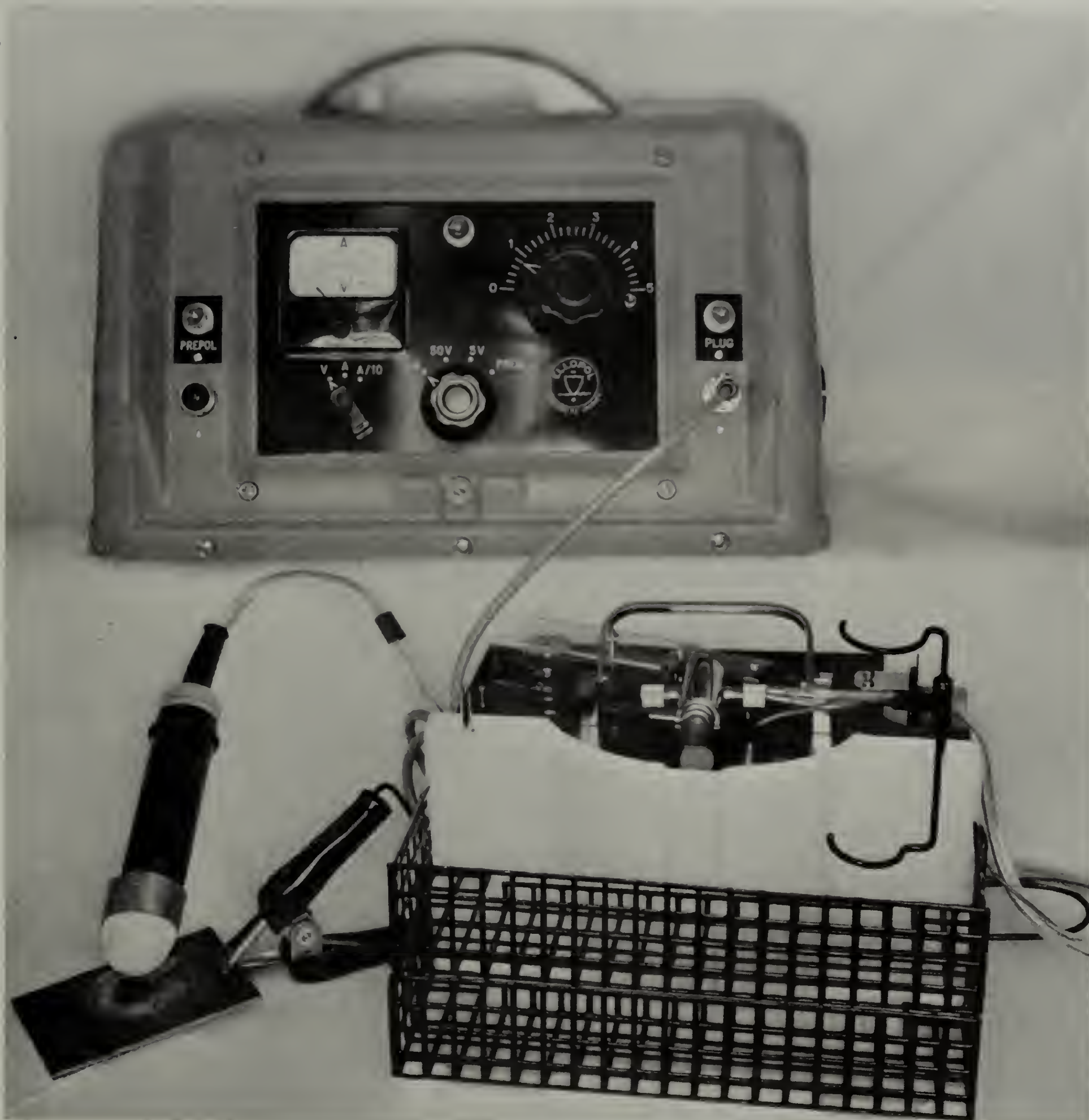
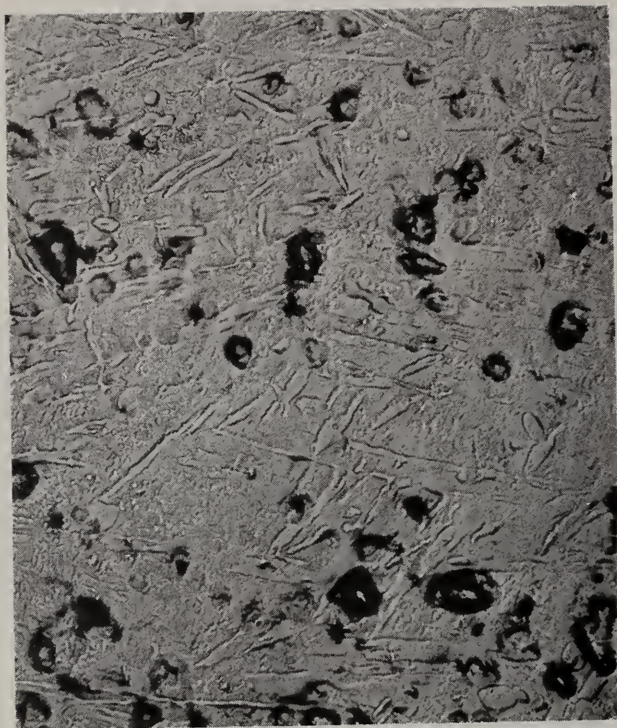
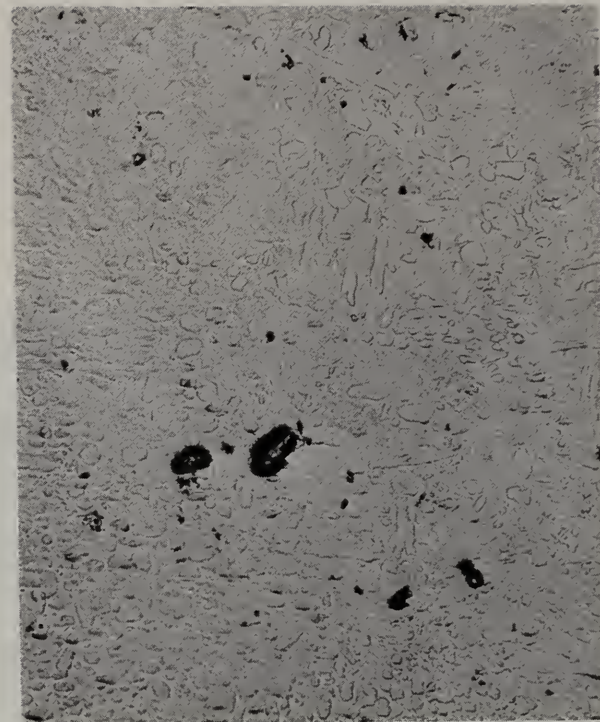


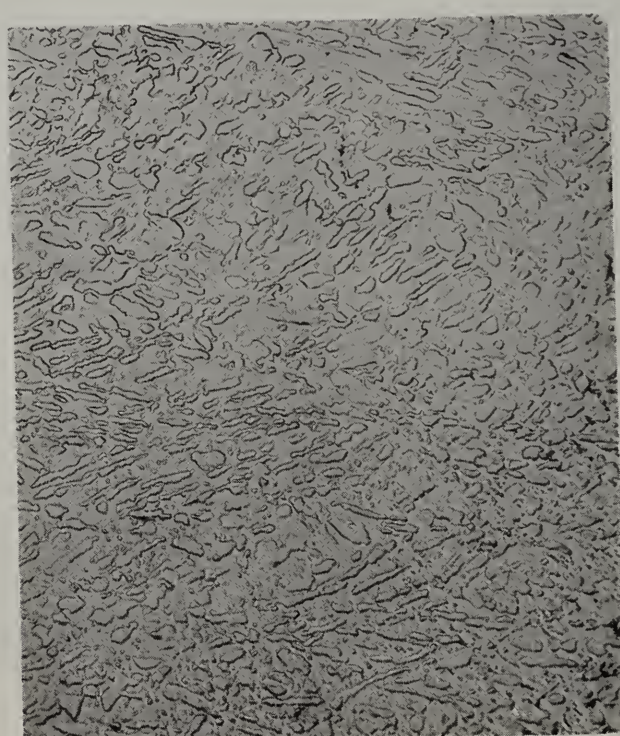
Figure 3. Ellopol electrolytic polishing apparatus, including power pack.



a



b



c

Figure 4. Three separate areas on an alpha-beta titanium alloy. (a) was hand ground; (b) was ground with the Portamet using only fine abrasive paper; and (c) was ground using the Portamet with coarse, then fine paper. All three areas were polished and etched with the Ellopol. X 100.

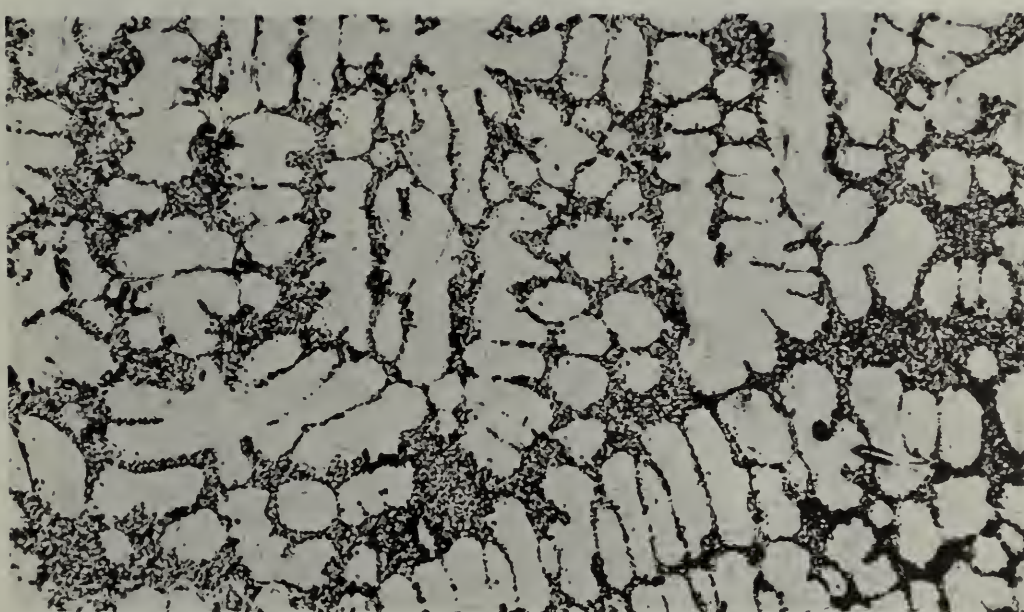
a

b

Figure 5. (a) shows the aft skirt of a Polaris missile, X 1. An arrow indicates the tool marks which are shown in greater detail (X 10) in (b).

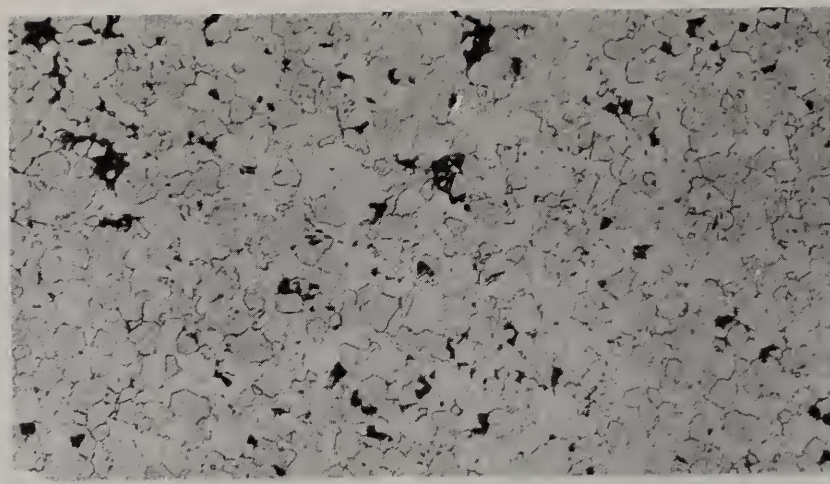
Figure 6. Section of engine torus showing welded joint. Approximately X 1.

a

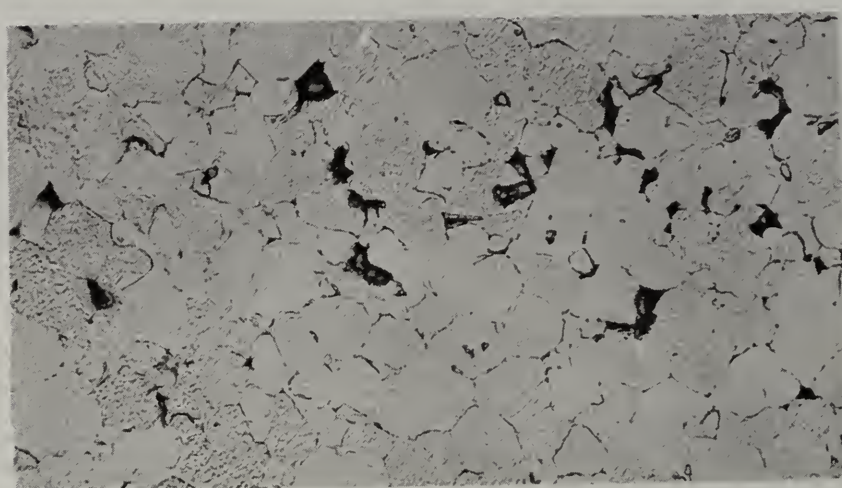


b

Figure 7. An attempt was made to electrolytically polish the cast aluminum attach bracket shown in (a). Areas A and B could not be polished successfully, but C electropolished very nicely without any prior preparation as shown in (b). (b X 100)



a



b

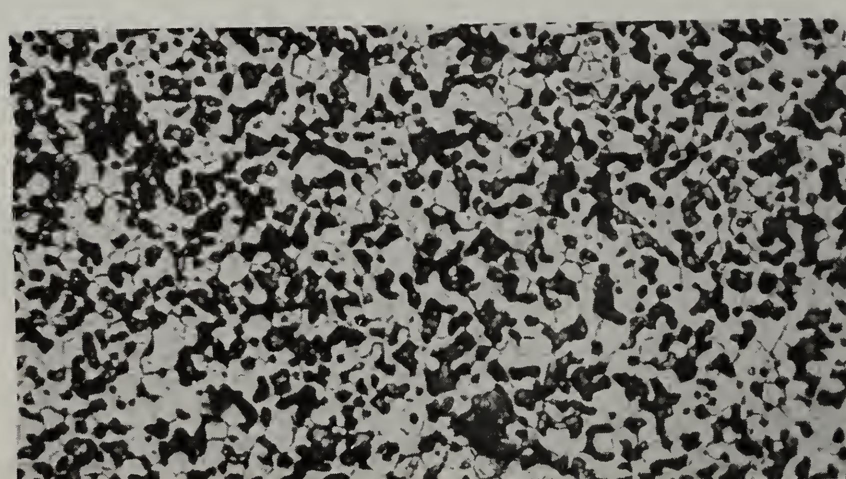
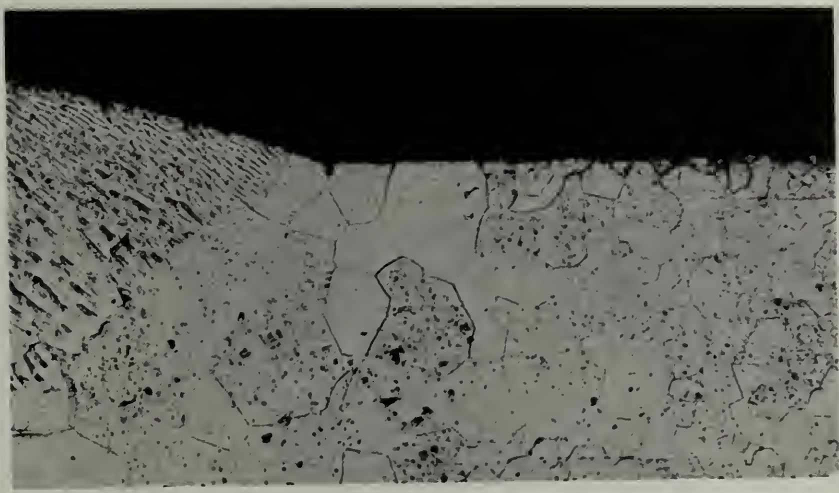


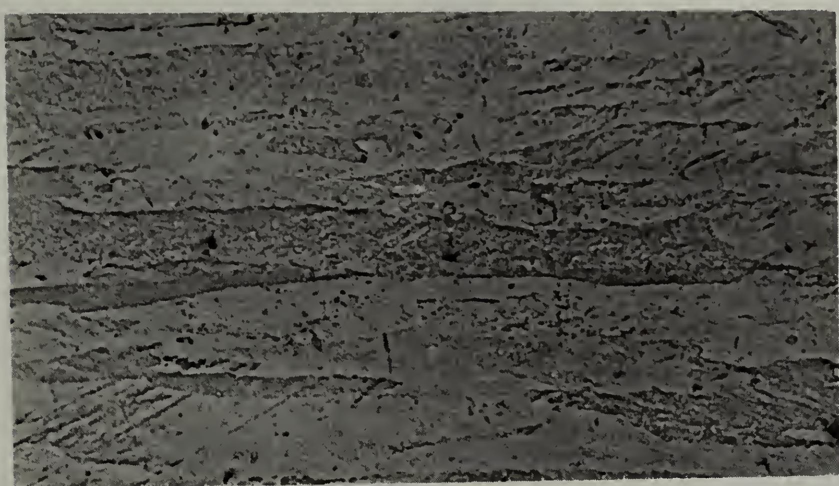
Figure 8. Microstructures that are likely to be encountered in tagging various items of hardware.

a and b. Flame sprayed tungsten showing the vacant sites that are normal for the material. a is at X 200; b, at X 500; both were etched with $HF:2HNO_3$: 6 latic acid. c is a silver-tungsten alloy, unetched at X 500.



a

b



c

Figure 9. Typical microstructure of materials used in metal components.

a and b. Columbium strip showing part of a weld in (a). a. X 100; b. X 500.
c. Titanium nozzle material, etched with Keller's reagent, X 200.

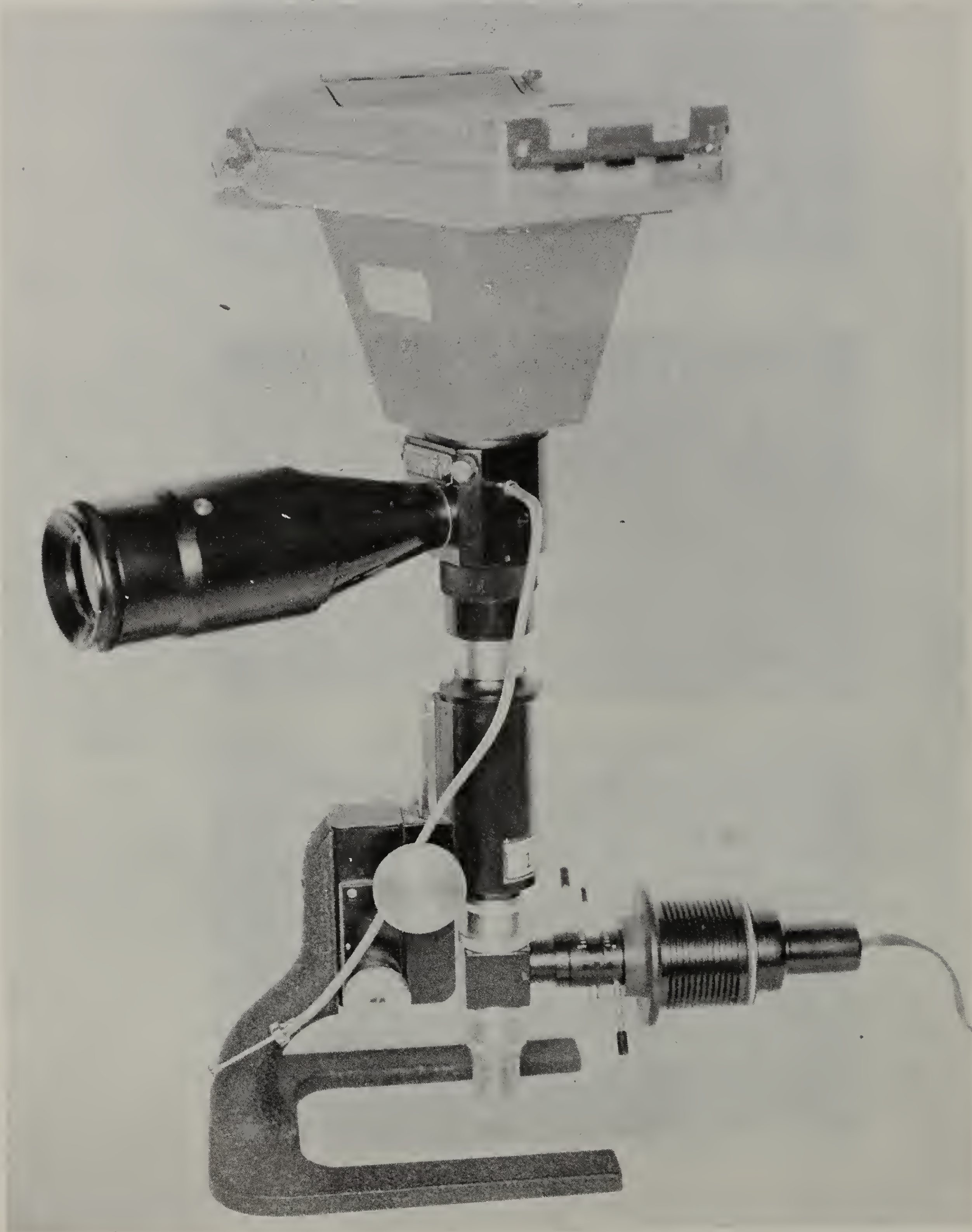


Figure 10. Portable microscope used in laboratory examination shown with the eyepiece camera and the Polaroid film back that was used.

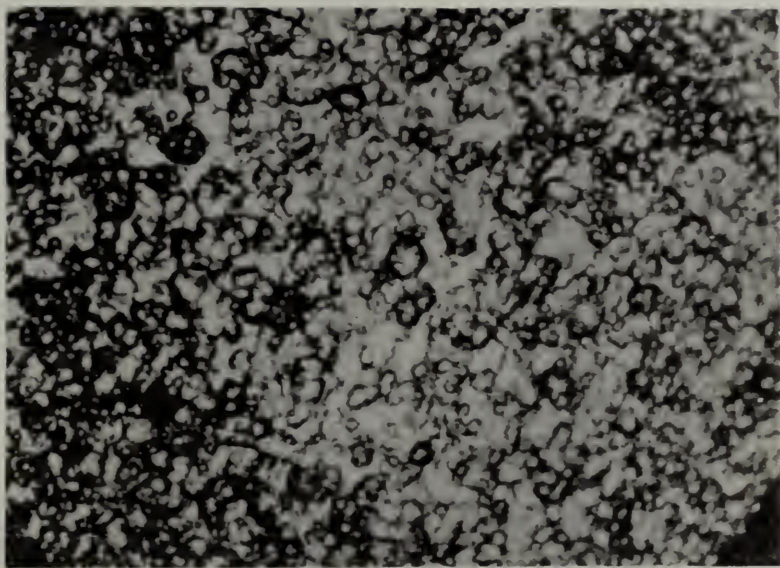


Figure 11. Photograph of chemically milled wrought aluminum alloy as it appeared to the eyepiece camera. No preparation, X 50.

a.

b.

Figure 12. Girth weld in titanium. Comparison of replica and direct photography.
a. Replica of titanium on a girth weld. Cellulose acetate tape 0.0015" thickness was used and afterwards vapor coated with aluminum. b. Photo of the original part. Sample weld is typical of the surface found on 1/8" thick titanium sheet. X 1.

Figure 13a. Original titanium girth weld in original part. X 5.

Figure 13b. Shadowed replica of the titanium weld. X 5.

Note that replica is a mirror image of the original.

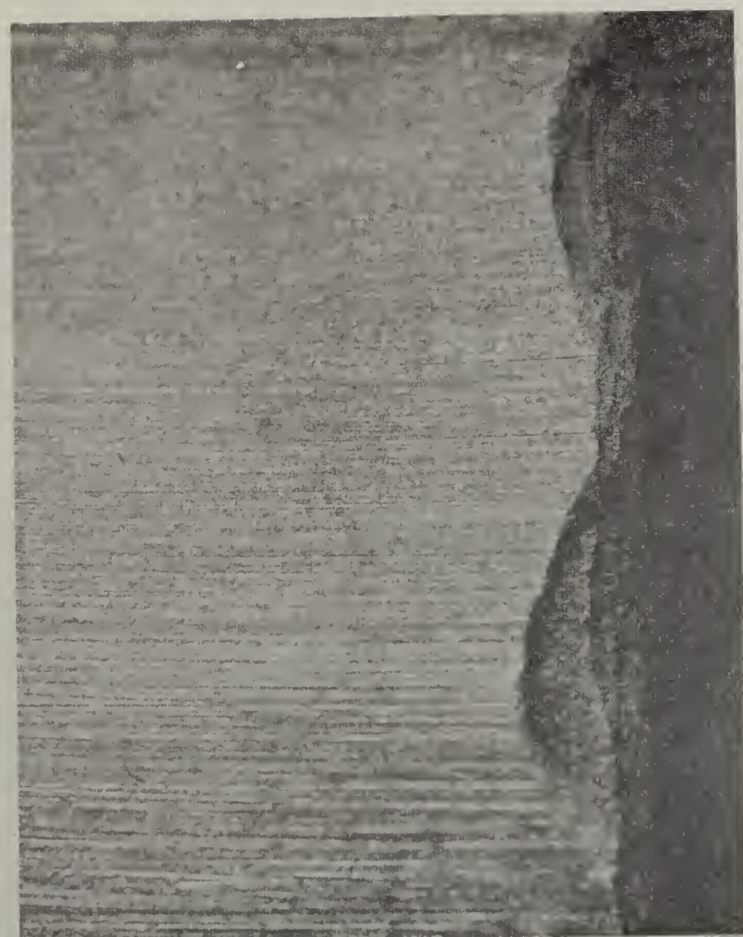


Figure 14a. Sample of tungsten flame sprayed graphite on exit cone insert. (original part). X 5.

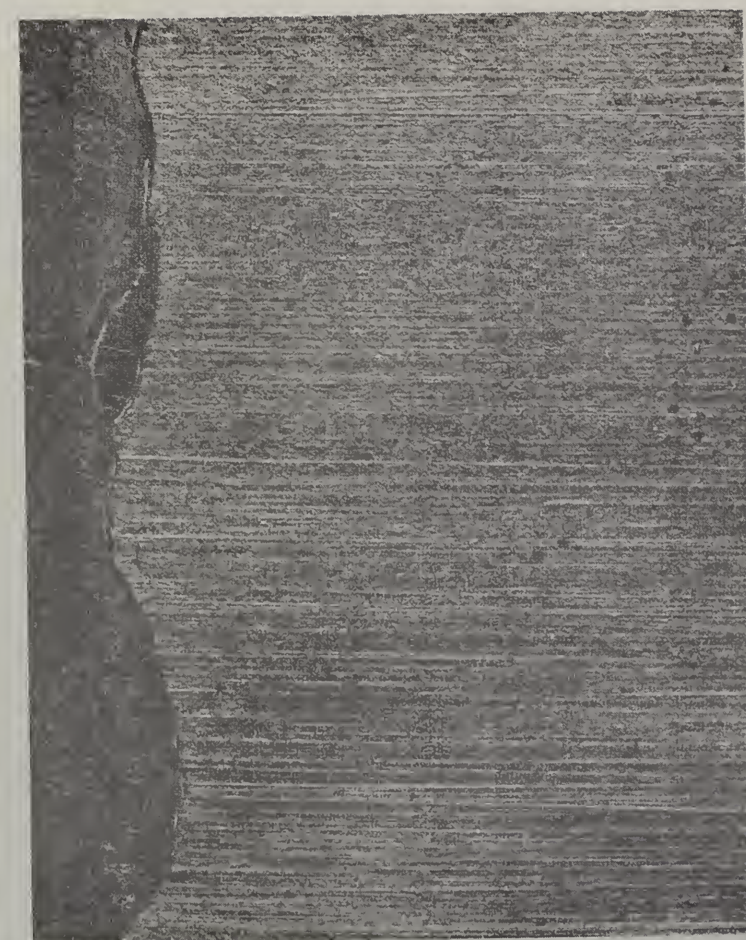


Figure 14b. Replica of the surface duplicates accurately the grinding marks. X 5.

Figure 15a. Three marks in the corner of a destruct mount were noted on the original part. X 5.

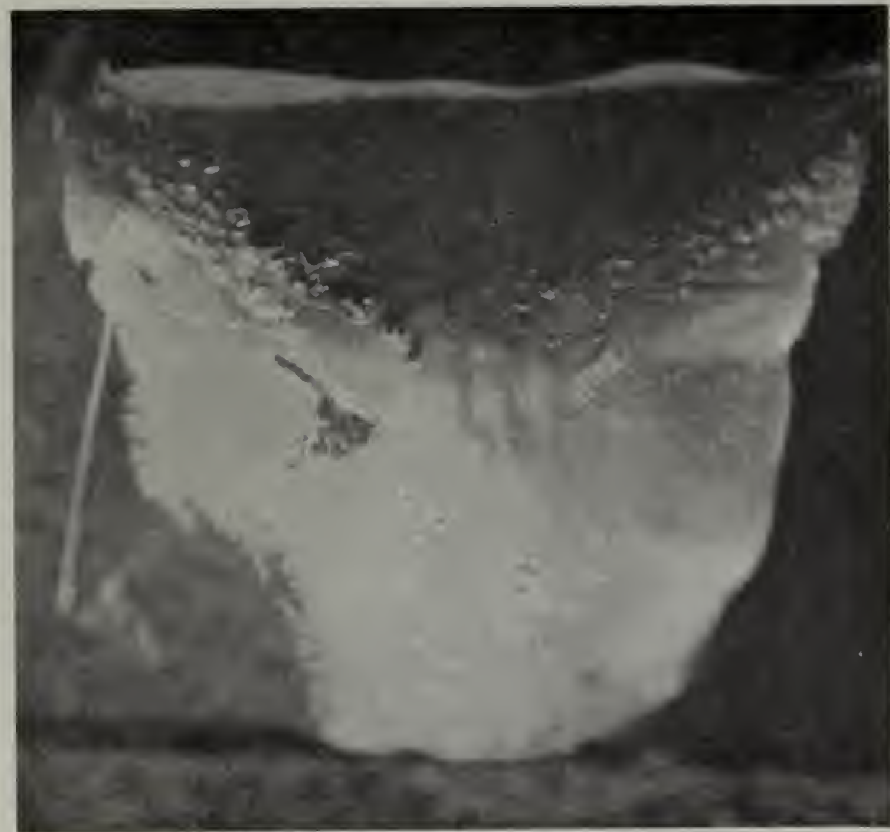


Figure 15b. The same marks are duplicated in the shadowed replica. X 5.

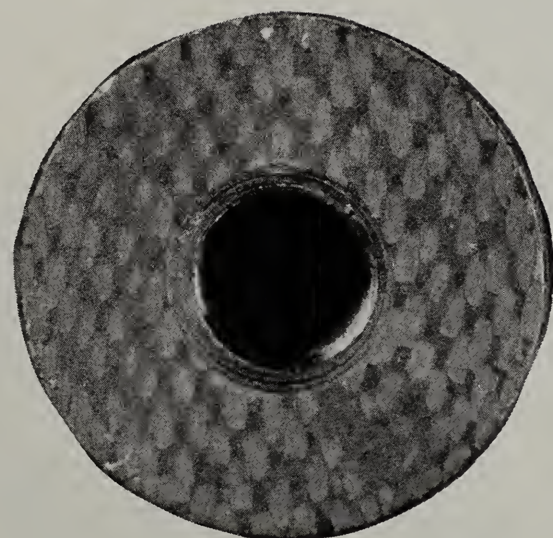


Figure 16a. The pattern of marks in the above photo was noted on the surface of the hardness mounting boss coated with No. 40 zinc chromate primer. X 5.

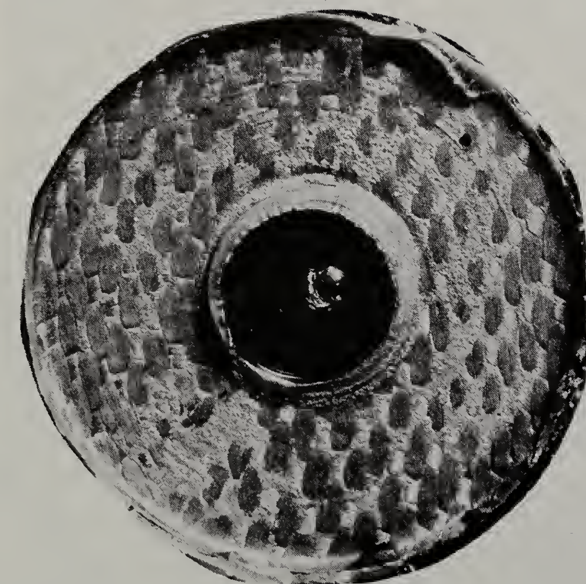


Figure 16b. Replica of boss surface.

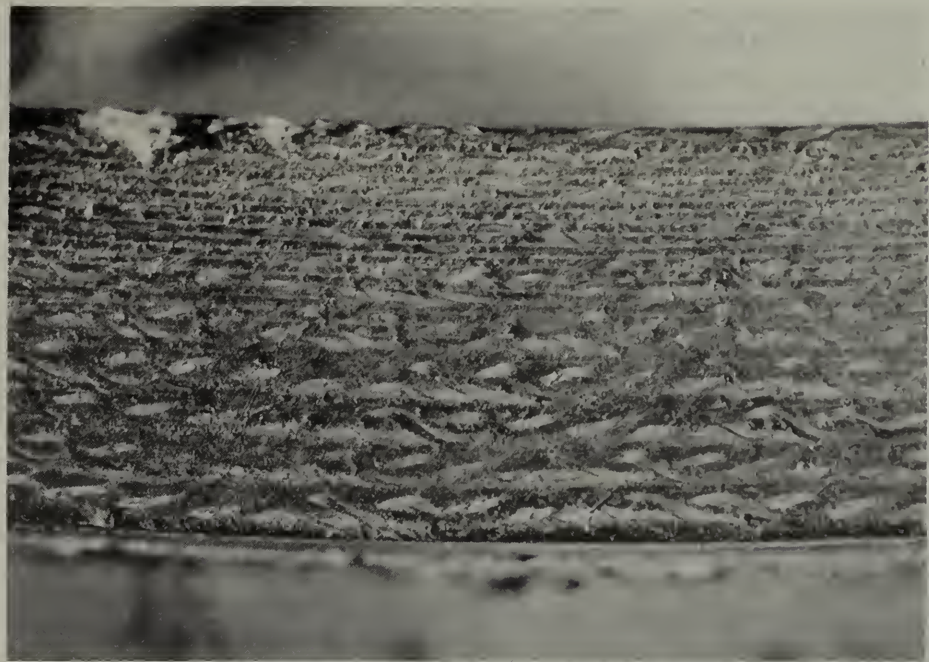


Figure 17a. Sample of silica fabric from aft nozzle exit cone. X 5.

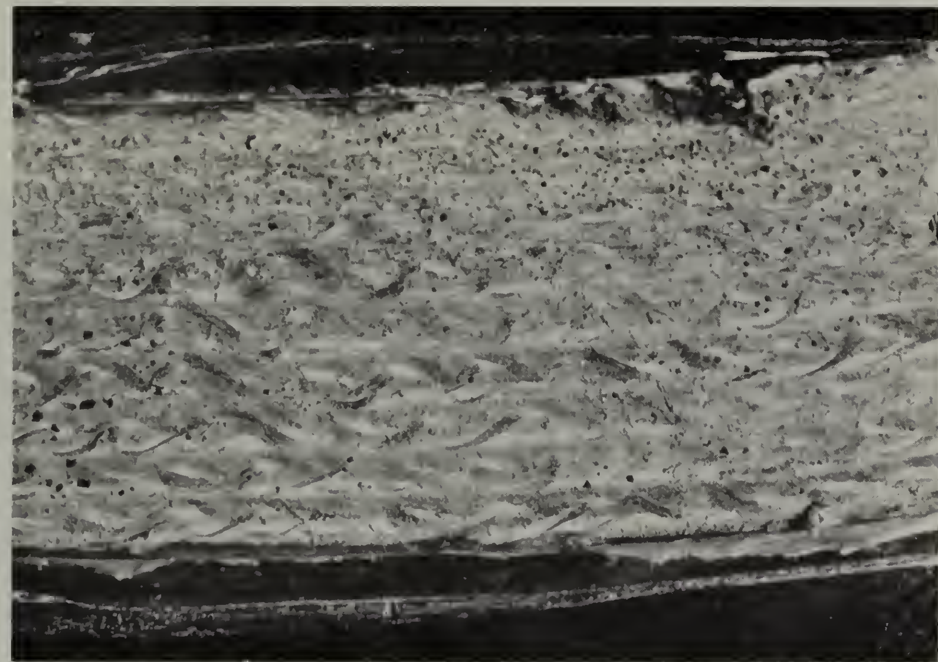


Figure 17b. The replica of the fabric surface duplicates weave pattern. The small black dots indicate porosity. X 5.

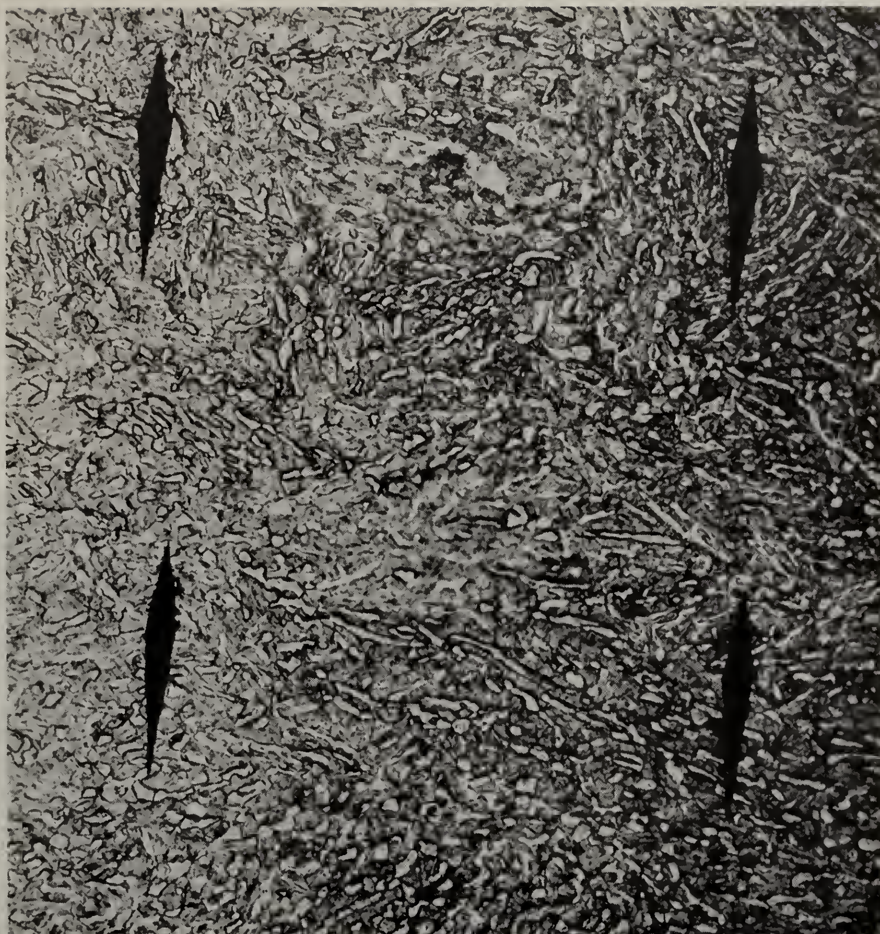


Figure 18a. The microhardness impressions used as an index mark assist in the location of the microfeatures in an etched sample of titanium. X 200.

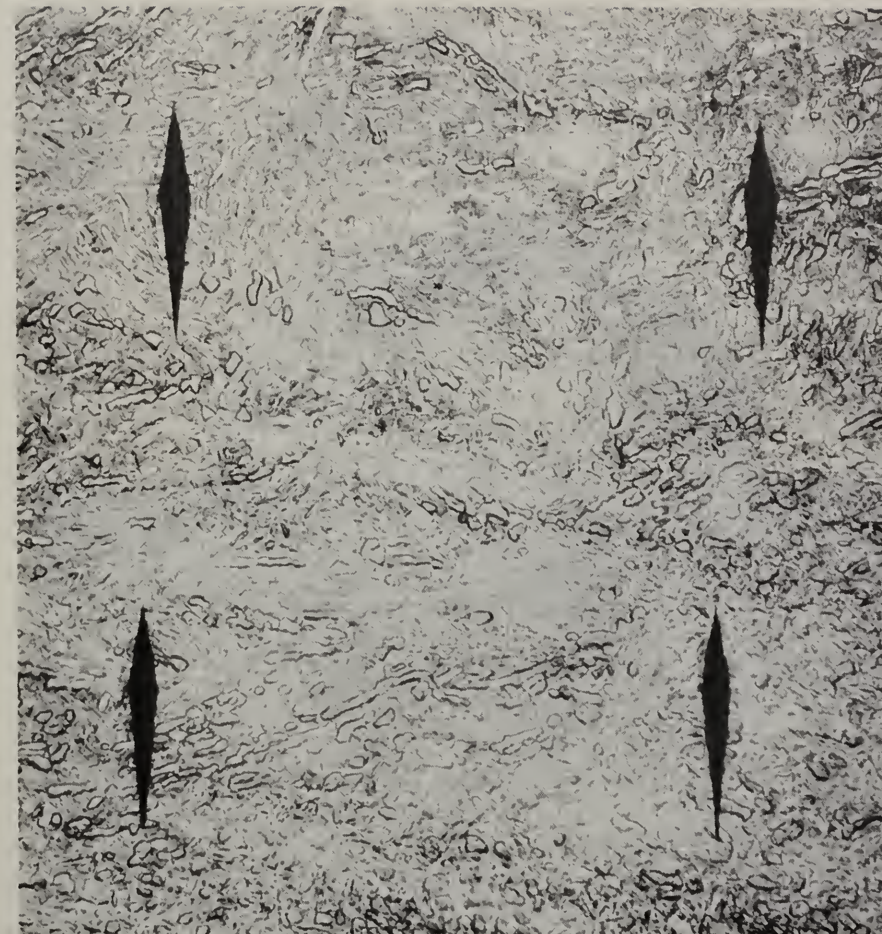


Figure 18b. Shadowed replica of identical area. X 200.

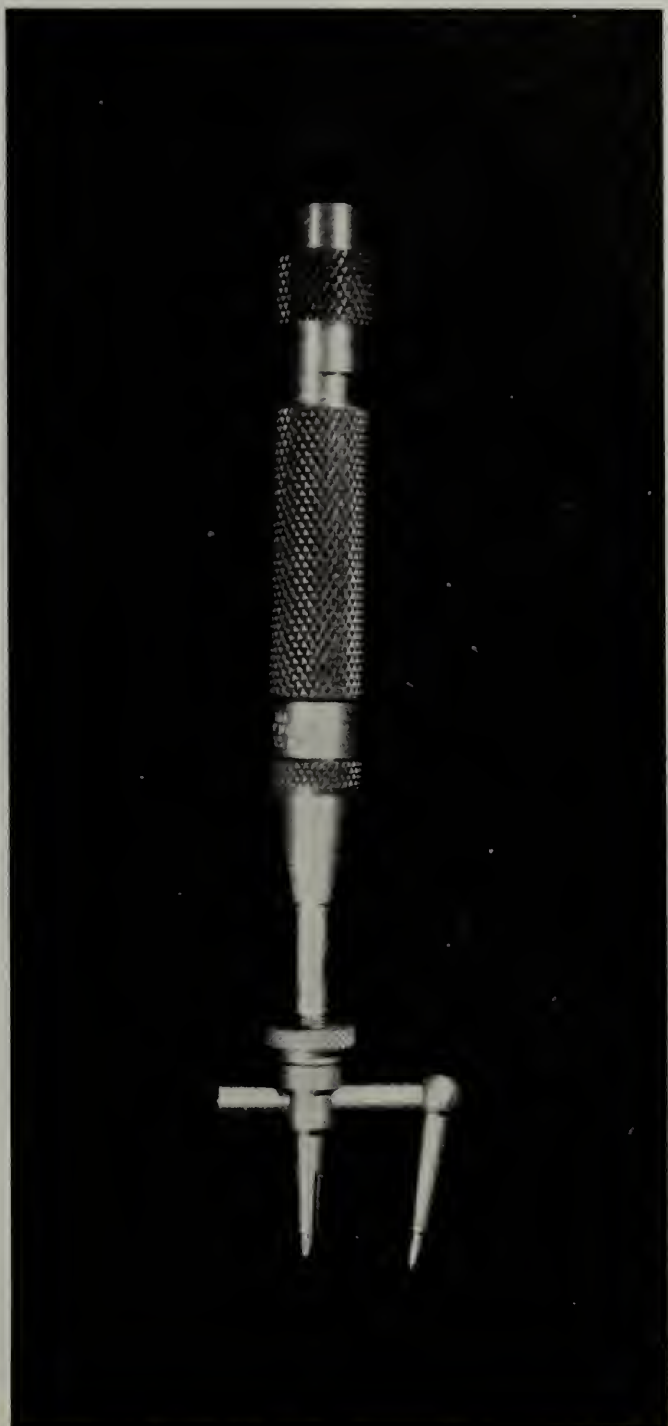


Figure 19. Punch that could be used to identify finger-printed area. Approximately X 1.

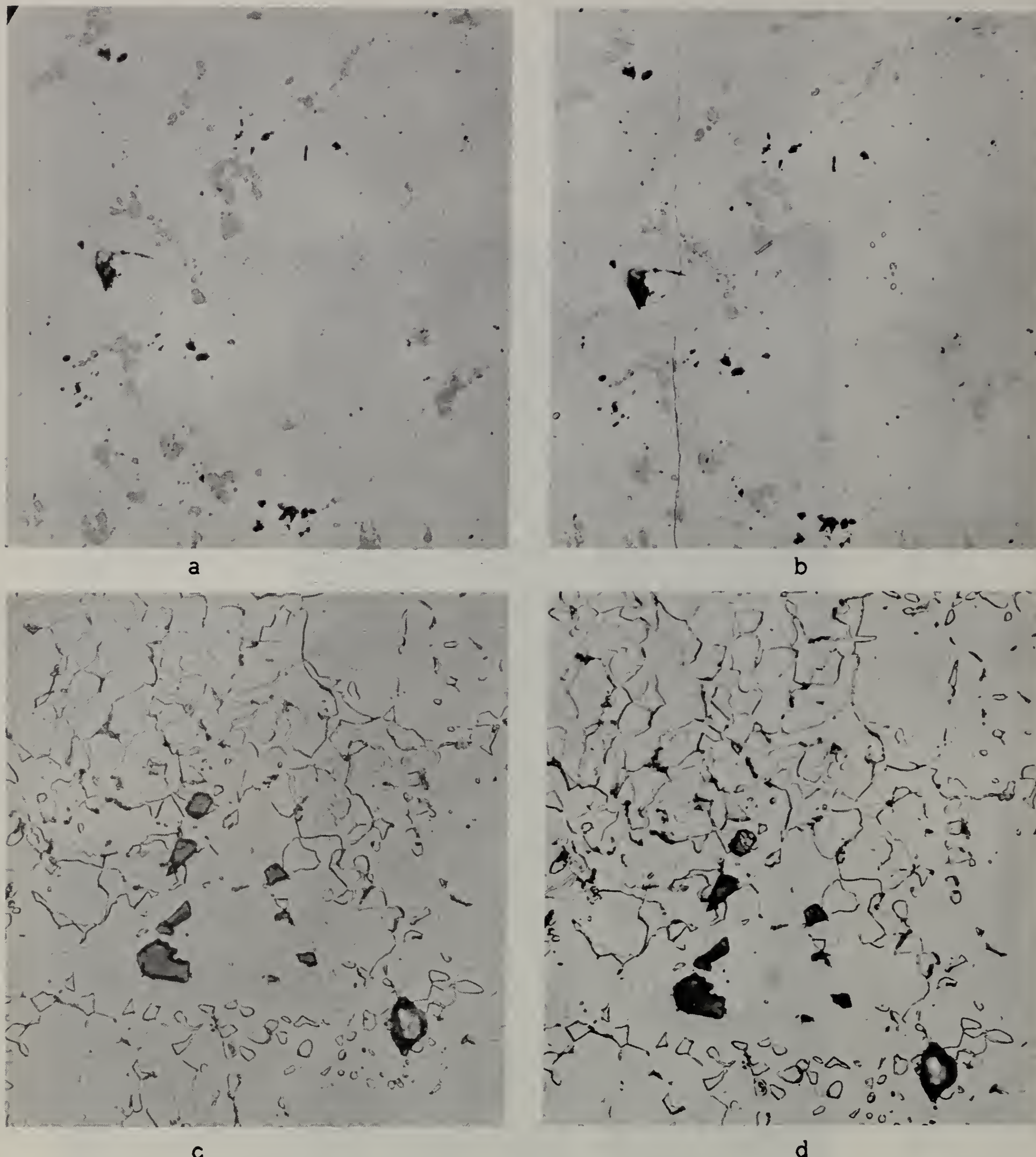


Figure 20. The effects of exposing 7075 aluminum and 347 stainless steel to an indoor atmosphere for three months are illustrated above. (a) and (c) were photographed immediately after the specimens were prepared; (b) and (d) were photographed three months later. Stains and an apparent increase in the size of some inclusions were the consequences of exposing 7075 aluminum. The stainless steel was not affected by the exposure.

a and b. 7075 aluminum, unetched. X 200.

c and d. 347 stainless steel, etched electrolytically with 10% oxalic acid. X 500.

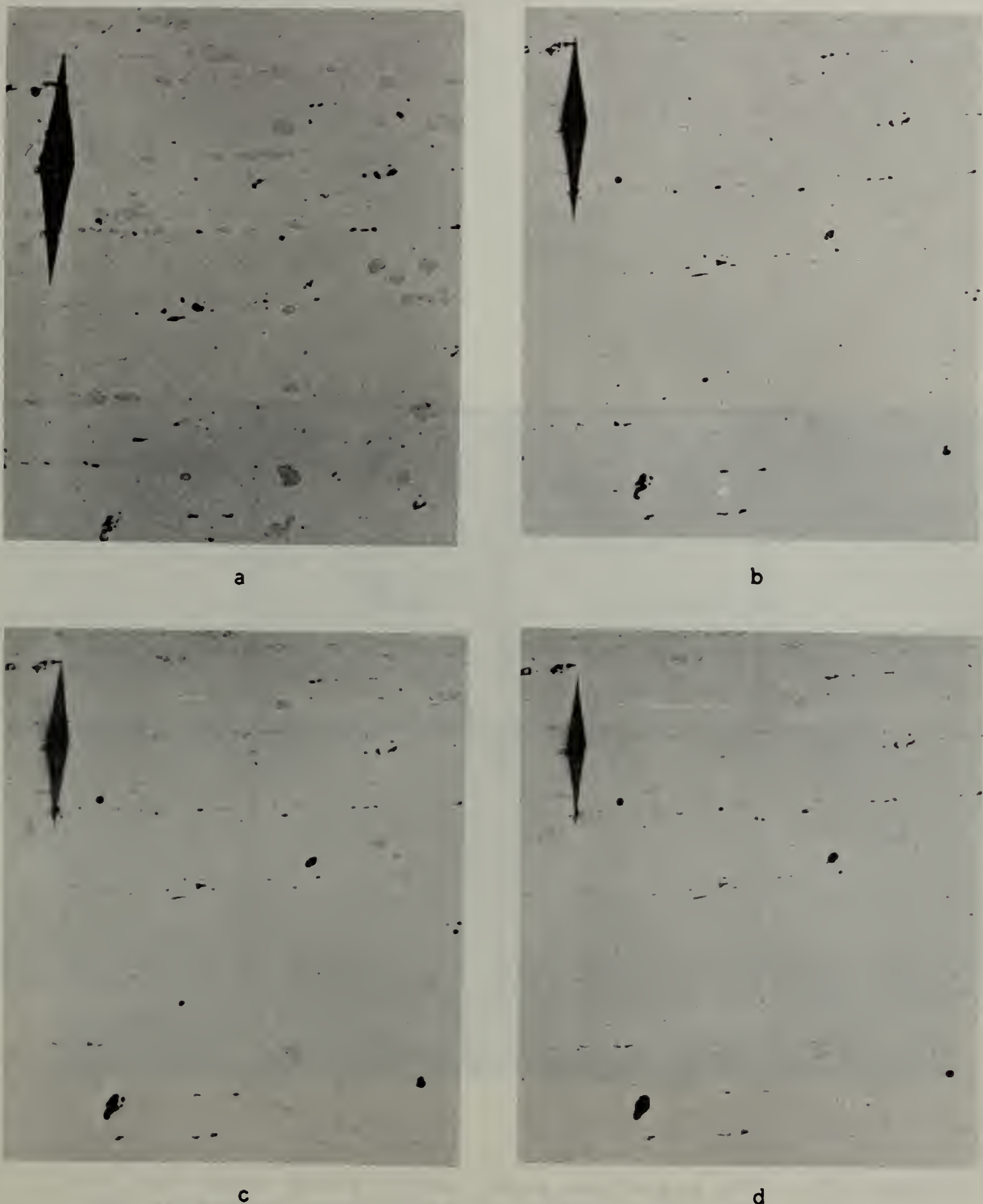


Figure 21. Depth study of 7075 aluminum alloy. As the material was removed, sizes and shapes of the second phase particles change slightly. Unetched.

a. Original surface. X 200.
b. Ten minute polish, 0.00029" deep.
c. Twenty minute polish, 0.00039" deep.
d. Thirty minute polish, 0.00048" deep.

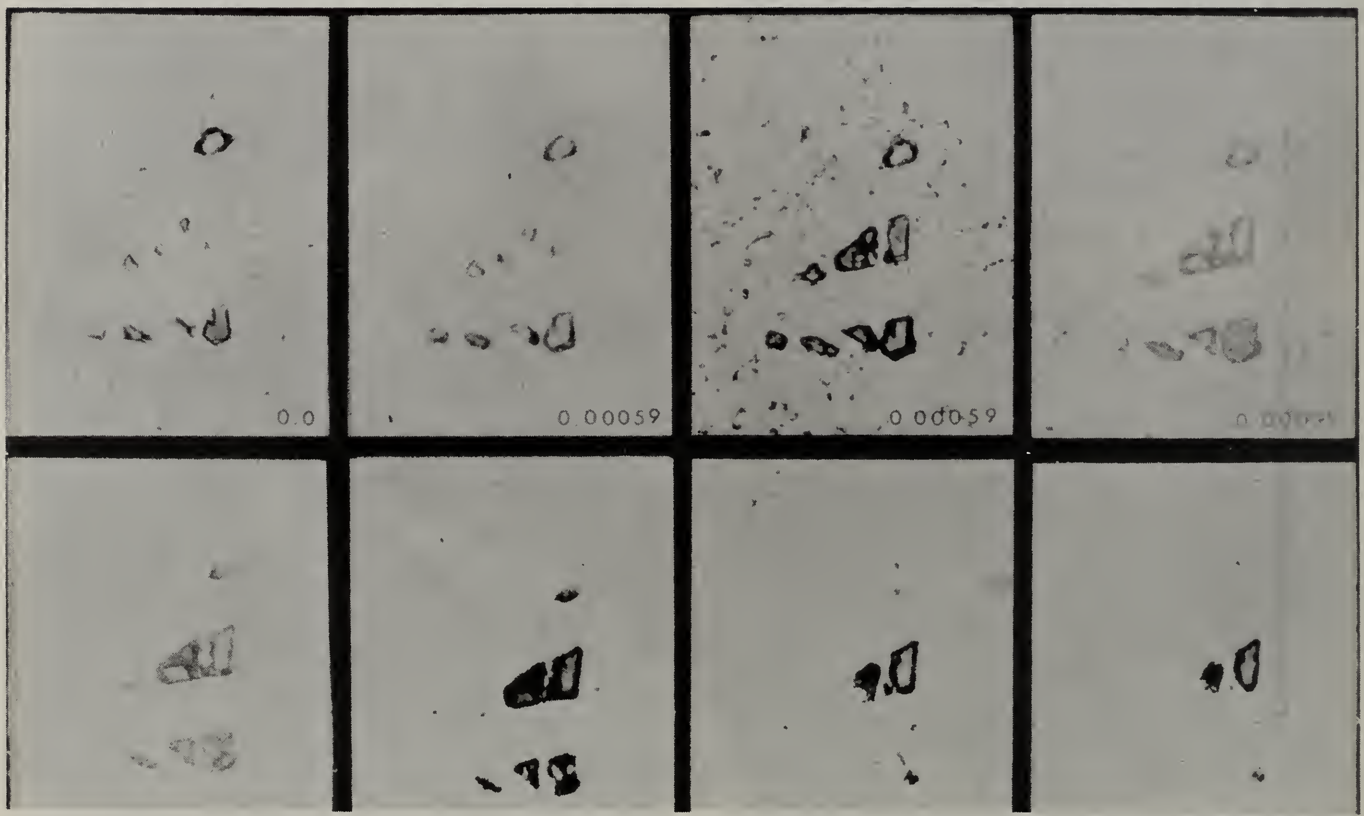


Figure 22. The stainless steel depth study shows how the sizes and shapes of inclusions change as the surface moves deeper into the material. The first and third micrographs from the left in the top row were etched; all others were unetched. Original magnification. X 500.

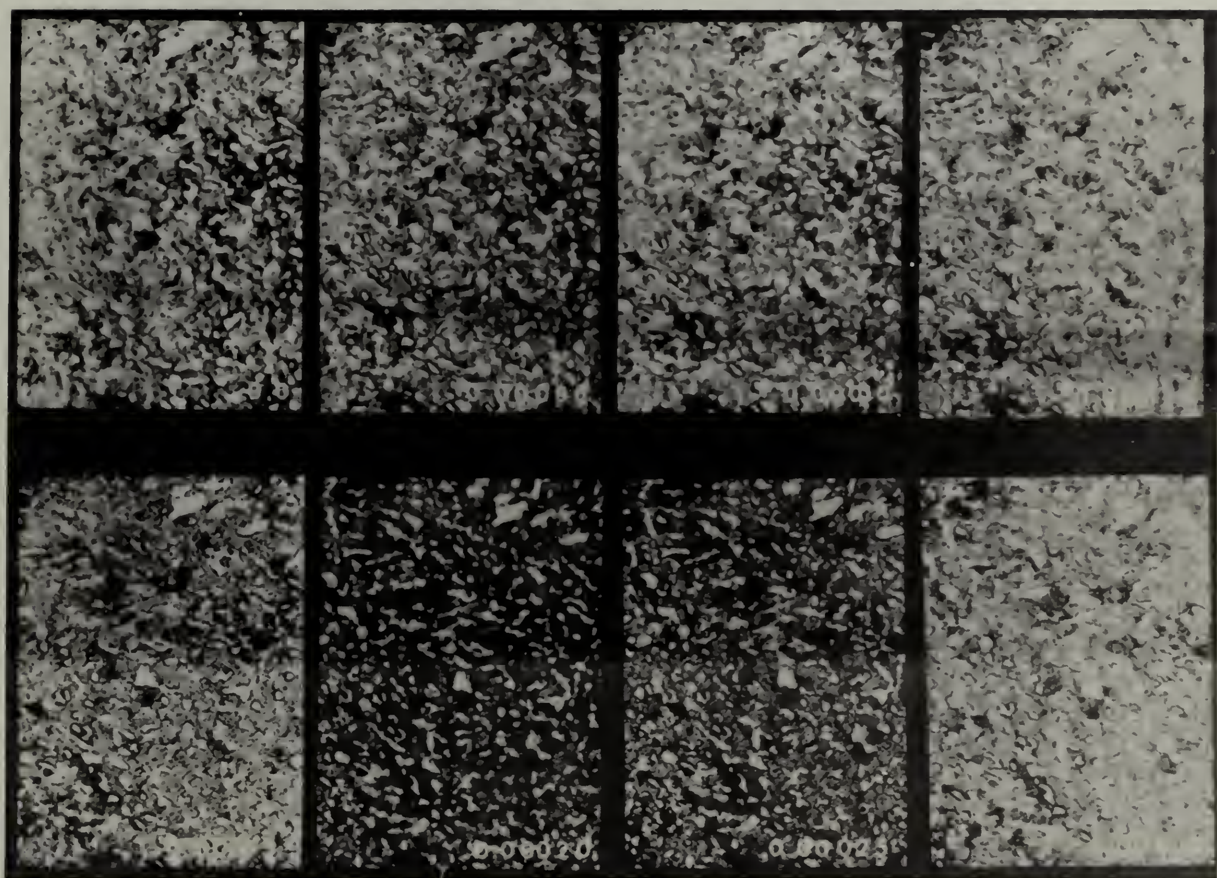


Figure 23. Depth study of alpha-beta titanium alloy which was etched with Keller's etch. Originally at X 200.

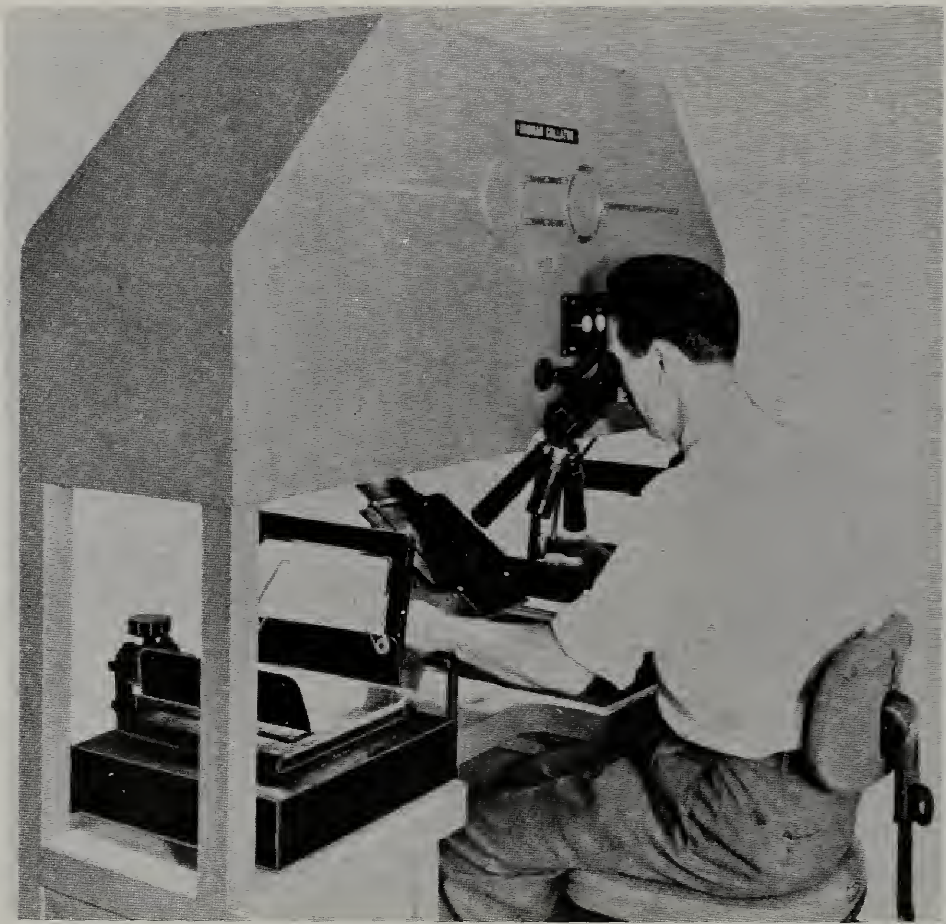


Figure 24. Hinman collator.

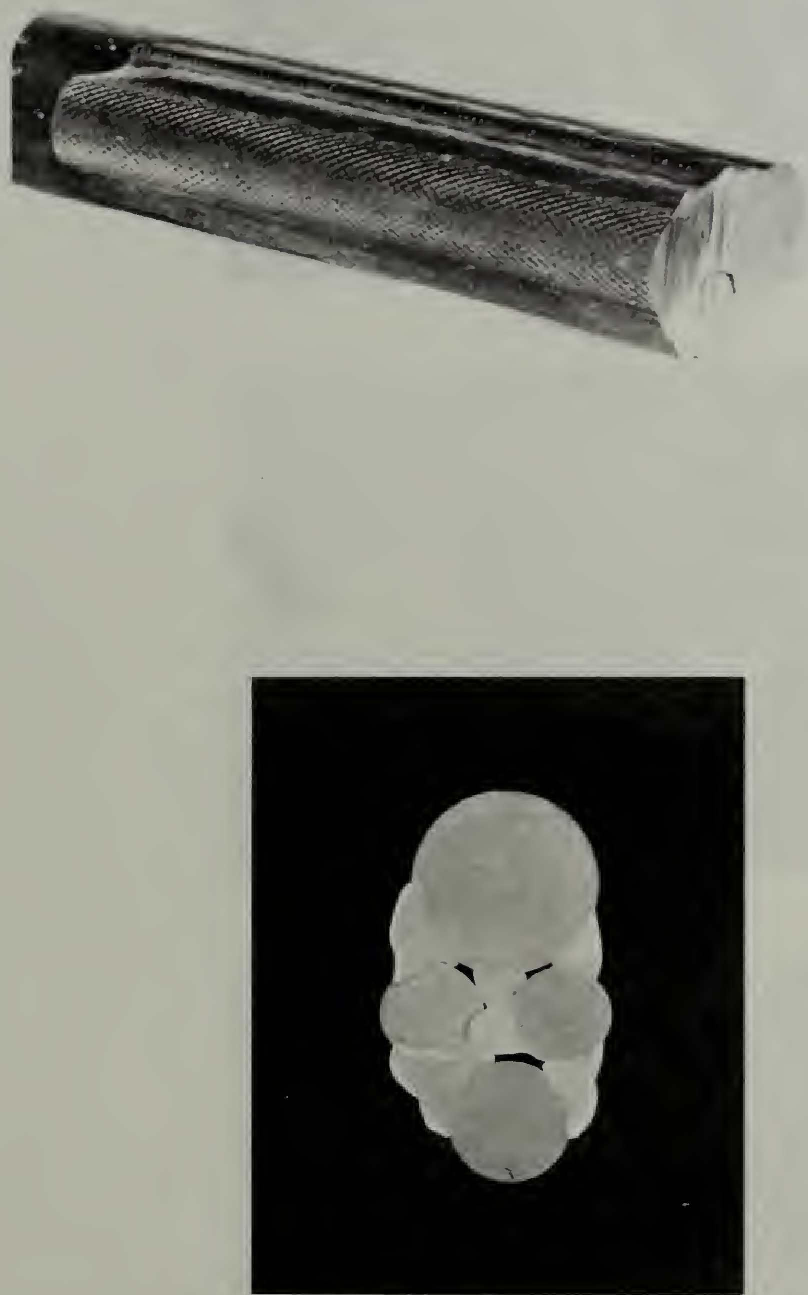
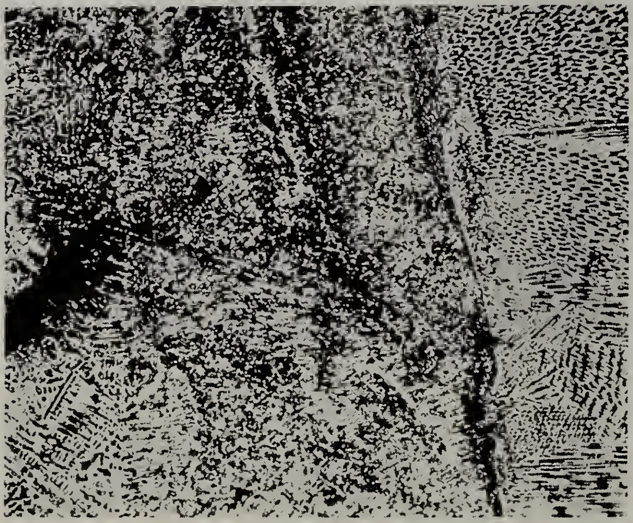


Figure 25. Weldments such as these can be used as transmittable tags. The patterns formed by the interaction between the welded rods and the weld metal are "fingerprint" characteristics, as are the microstructure of the welds, the configuration of the assembly, the composition of the bars and weld metal and the surface characteristic of each bar, when considered as a unit. Approx. X 1.

a

b



c

Figure 26. Effect of higher magnification on identification of weldments.

a. X 1.

b. X 10.

c. X 100.

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