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Microsectioning: A Metallographic Technique for Semiconductor Devices

Abstract: A microsectioning technique is described that enables metallographic sectioning of fragile semiconductor devices without the difficulties and specimen damage associated with the use of conventional techniques. Major advantages are (a) maintenance of a planar surface on specimens having adjoining areas of widely varying physical characteristics, (b) preservation of boundary details between such areas by elimination of rounding-off effects, and (c) precise positioning of the sectioning plane by direct micrometer caliper measurement. Choice of sectioning plane, specimen mounting, machine lapping of mounted specimens, polishing, etching, and panoramic microphotographic techniques are described. The effectiveness with which data can be gathered from the resulting microphotographs is demonstrated by an analysis and evaluation of selected specimen photographs.

Introduction

In both development and production of semiconductor devices, metallographic sectioning of selected specimens is essential for the systematic study or routine inspection of internal structures. Observational data from these specimen sections, correlated with measurements of their electrical characteristics, are of great value in diagnosing physical or electrical defects, and in determining the changes necessary in design, fabrication, and processing to improve device performance.

Specimens selected for sectioning may fall into one or more of the following categories: (a) new devices undergoing laboratory development; (b) defunct units scheduled for post-mortems; (c) units made for the purpose of studying the surface-wetting properties of alloy-dot materials, alloy-junction formation, eutectic bond configurations, and other effects; and (d) one-of-a-kind samples having abnormal physical or electrical characteristics.

A specimen fitting category (d) is the *p-n-p* alloy-junction transistor of Fig. 1, which contains very thin sections of hard, brittle germanium adjacent to areas of soft, putty-like indium. Such specimens cannot be embedded or "potted" for sectioning by conventional pressure-molding processes because of fracturing of the germanium. They cannot be polished by resilient fabrics charged with abrasive, as in conventional metallographic techniques, because of rounding off of the edges of the germanium which obscures recrystallization effects near these edges.

This paper describes a more appropriate metallographic sectioning technique which overcomes these difficulties, and enables the preparation of specimens in a reasonable amount of time, utilizing miniature containers having a diameter more compatible with the physical size of the specimens.

Choice of the sectioning plane

The first step in the procedure is a careful inspection of the specimen under both metallurgical and stereo-microscopes for geometry, surface data, and external defects pertaining to a particular study. If the specimen is one of a kind, or one which exhibits unusual electrical characteristics, these observations are of special importance in choosing the position of the sectioning plane. This choice may not always yield the most data, but if a number of specimens are available from the same lot, various sectioning possibilities may be distributed among a sufficient number of samples for the greatest yield of information.

Selection of the sectioning plane is the next step. Taking the alloy-junction transistor of Fig. 2a as an example, let us assume that germanium crystallographic orientation error and collector eccentricities with respect to the washer hole are the major considerations, and that we are to study their effects on alloying and recrystallization behavior. Plane *A-A*, perpendicular to the page, is chosen to observe alloying behavior on the basis of maximum eccentricity between the collector and the hole in the washer, with some crystallographic orientation error present. Crystallographic orientation error in the plane of the section is the angle θ between the surface of the germanium die and the dotted lines denoting the geometri-

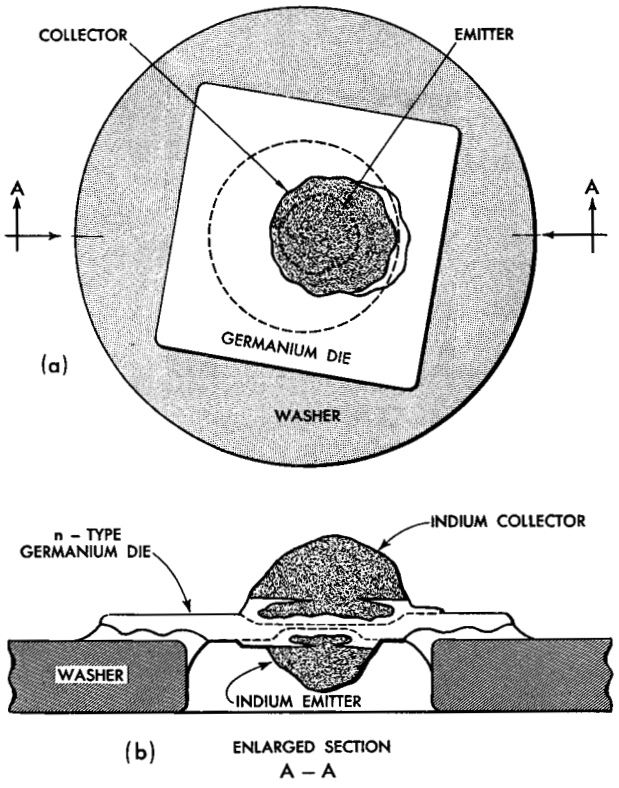


Figure 1 **Alloy-junction p-n-p transistor specimen showing abnormal recrystallization.**

cally flat and parallel alloy junctions of Fig. 2b. Plane *B-B* is an alternate sectioning plane chosen to observe maximum crystallographic orientation error of the germanium and its effect on alloying behavior, with almost maximum eccentricity between the collector and the washer hole. It is not possible to determine this particular sectioning plane on every specimen, because some transistors lack the necessary externally visible collector-emitter formations and germanium etch pit geometry. Plane *C-C* reveals alloying behavior with negligible crystallographic orientation error, since this plane is approximately 90° from section *B-B*. The resulting sections are shown respectively in Figs. 2b, c, and d.

Many specimens are sectioned only for a determination of the junction spacing or for a study of some large externally visible defect or an unusual alloying behavior. It becomes a simple matter, then, to alternately lap down and inspect until the sectioning plane appears to pass roughly through the center of the configuration being studied. If the configuration is only 0.001″ or so in size, however, a more precise location of the sectioning plane is required, and it is then necessary to note its position with respect to a reference point on the periphery of the washer. Subsequent micrometer caliper measurements from a datum plane tangent to the washer at this reference point determine when the sectioning plane is reached during lapping and polishing operations. Positional accuracy can be held consistently within ± 0.0001″.

Specimen mounting

Since the specimens are only about ⅛″ in over-all diameter, it is convenient to use small transistor encapsulation cans (0.215″ O.D., 0.3″ high) as mounting containers. This saves a great deal of the time and labor required to lap and polish samples mounted in conventional containers one inch or larger in diameter.

The mounting material in which the specimen is potted should have the following properties:

a) Low viscosity to permit flow into microscopic crevices in the specimen without trapping air bubbles.
b) Negligible shrinkage during hardening.
c) Sufficient hardness and toughness for approximately the same rate of wear as the specimen during lapping and polishing.
d) Good adherence to surfaces to preserve specimen edge details.
e) Negligible tendency for the surface to load up with grit particles during lapping and polishing.
f) Negligible attack by strong acid mixtures.

The first specimens prepared for microsectioning were potted in a rather makeshift manner in quartz cement, a material of low melting point used in the piezoelectric industry to secure quartz crystals for wafering and lapping. This cement was the only material then available that melted into a liquid sufficiently thin to flow into at least some of the larger crevices in the specimens. It was very unsatisfactory, however, because of its softness and excessive shrinkage and cracking upon cooling. Its tendency to form small bubbles while liquid was a serious problem, because grit particles, lodged in the resultant cavities during lapping, worked out again and caused severe scratch damage to the specimen surface during final polishing. Acid absorbed into the shrinkage cracks during etching later crept out onto the surface of the specimen, rendering it unfit for microscopy within a few days after preparation. In addition, the cement itself was not acid resistant, was prone to load up with grit particles during lapping and polishing, and was too soft to support and preserve germanium edge details.

During a search for better potting materials, it was noted that stop-off lacquer,* commonly used in the electroplating industry for selective masking, had desirable qualities of hardness, toughness and tenacity when used as a coating material for specimens. When diluted half-and-half with lacquer solvent, it effectively wets metal surfaces and flows into narrow crevices without trapping air bubbles. The coating thickness can be built up by successive dippings until corner radii are large enough to permit the use of an epoxy resin compound†

*"Miccrostop" stop-off lacquer, reducer, Michigan Chrome and Chemical Co., Detroit, Michigan.
†"Araldite" CN-502 resin, with HN-951 hardener; Ciba Co., Inc., Kimberton, Pennsylvania.

which is otherwise too viscous to flow into these crevices. Except for its viscosity, epoxy resin is an ideal potting material for microsectioning.

If stop-off lacquer dipping is impracticable for any reason, perhaps prefilling small recesses in the specimen with a globule of epoxy resin on the point of a sharp needle, or drawing a momentary vacuum on specimens precoated with resin, just before potting, may be more convenient. Potted specimens are allowed to harden overnight at room temperature in order to avoid thermal distortion and pulling away of the potting material from the specimen. Greater care is used in potting specimens that require lapping and polishing to within ± 0.0001″ of the desired sectioning plane, since the reference plane mentioned previously must be normal to the axis of the mounting container.

Rough lapping on the "microsectioner"

If there are many specimens to be microsectioned, it is more practicable to resort to a mechanized rough lapping operation to secure uniformly good quality than to attempt to do it by hand labor. A machine called the *microsectioner,* developed for this purpose, is shown in Fig. 3. It consists essentially of a horizontal motor-driven turntable surfaced with abrasive paper, on which rides a rapidly oscillating arm carrying the specimen being lapped. A small worm-gear speed reducer drives the turntable at approximately 50 rpm through a V-belt and also actuates the specimen-carrying arm by means of a crank linkage and vertical shaft. The arm oscillates approximately 3.1 cycles for each turntable revolution. The speed reducer is driven in turn by a d-c shunt motor with variable armature voltage for control of lapping speed. A simple spring-loaded latch allows quick and easy removal of the arm for insertion and removal of specimens.

Since there are slight variations in the O.D., roundness, and taper of the specimen mounting cans, a reference surface must be provided for precise control of lapping and polishing operations. This surface is provided by an accurately machined brass sleeve into which the mounted specimen is snugly fitted. The brass sleeve closely fits both the hole in the oscillating arm of the microsectioner and the bore of a cylindrical holder used in subsequent polishing operations. Since only about 0.0002″ is removed in a polishing operation, the rough-lapped surface of the specimen must be square with the axis of the brass sleeve. This squareness is effected by initially adjusting two tapered driving screws on the microsectioner until a test rod held in the oscillating arm is square with the turntable surface. Both the test rod and the *final* lapped surface of the specimen must project from the underside of the arm the same distance; otherwise, squareness of the specimen surface with the sleeve axis cannot be maintained.

Specimens requiring lapping and polishing to within ± 0.0001″ of the desired sectioning plane are first lapped on the microsectioner until a previously designated reference point lies in the lapping plane. A micrometer caliper measurement of the initial over-all length of the specimen and sleeve is then noted, and lapping is continued until the final measurement is reached, the difference between these measurements being the distance from the reference point to the desired sectioning plane with an allowance for polishing.

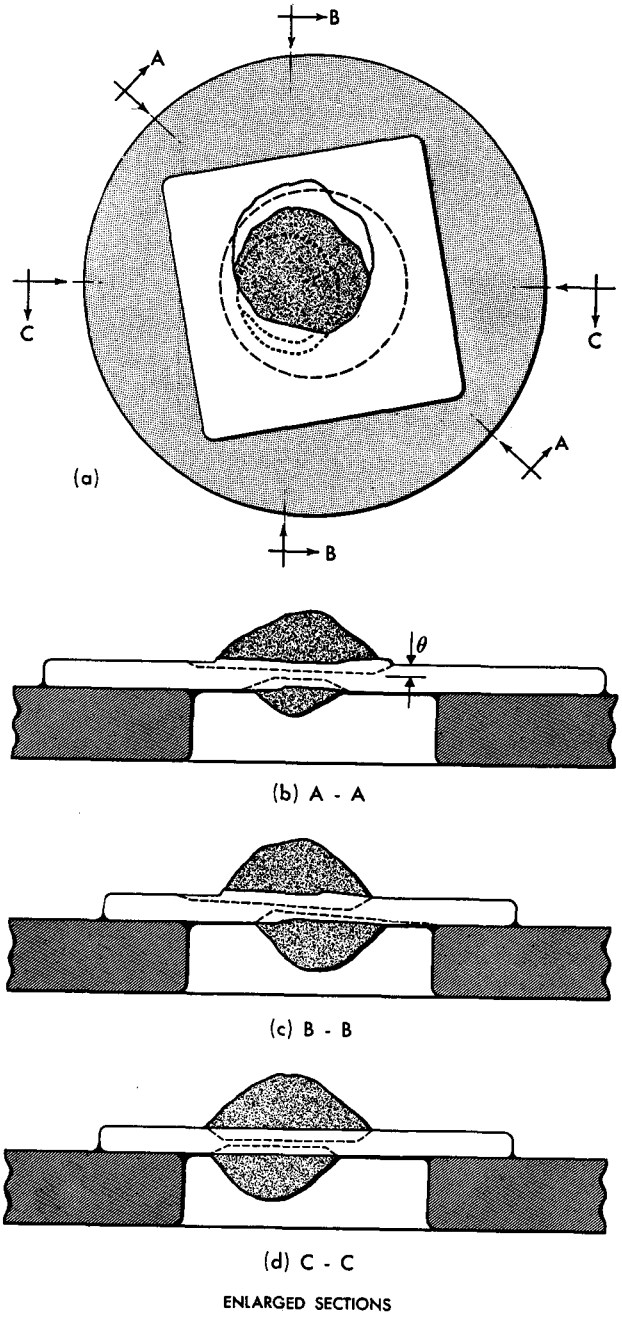


Figure 2 **Alloy-junction transistor specimen illustrating various sectioning plane possibilities.**

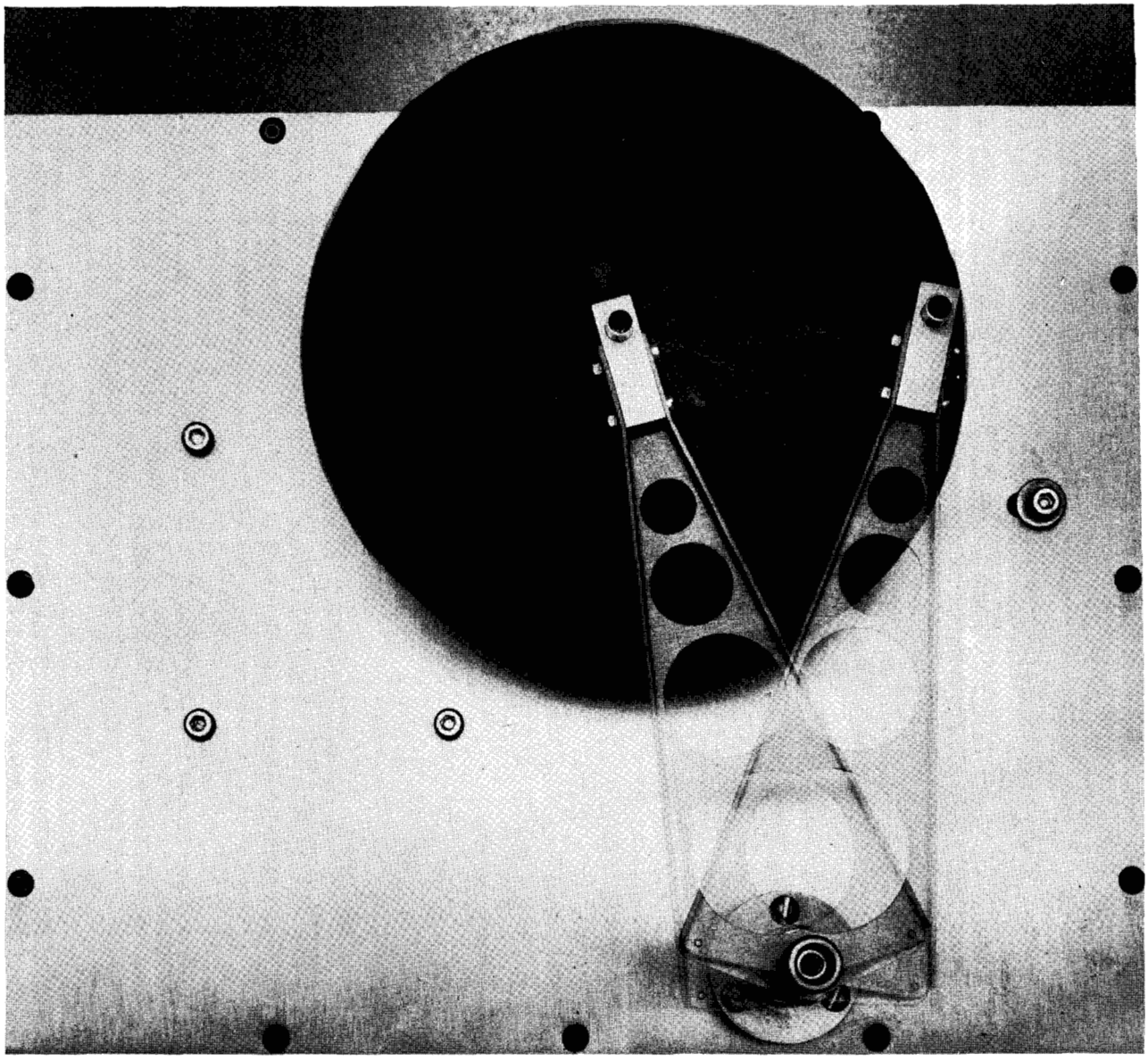


Figure 3 **Machine for rough lapping specimens to desired metallographic sectioning plane.** *A Lissajous figure was purposely traced on a dry abrasive disc surface by running the machine several revolutions without the lapping vehicle to show that the specimen never retraces the same path. Ordinarily, the machine is never run without the lapping vehicle.*

Polishing techniques

In most semiconductor specimens, the rough-lapped surface is characterized by hard, brittle areas adjoining very soft areas, putty-like in comparison. The surface must be polished to a high degree of perfection in both flatness and optical reflectivity before it can be observed under a metallurgical microscope for the study of cross-sectional configurations.

Alloy junctions and other recrystallization effects with which we are most frequently concerned are usually situated very close to the edges of the hard, brittle areas. If resilient polishing fabrics or papers charged with abrasive are used, as in conventional metallographic techniques, there is a tendency for softer areas to wear down faster and the edges of the hard material to round off, thereby obscuring these effects in the microscope field of view.

An alternate polishing technique with negligible rounding-off action utilizes a sheet of smoothly stoned, hard-surfaced vellum or tracing paper dry-charged with 0.3μ aluminum oxide polishing powder, and a specimen holder one inch in diameter to keep the specimen surface

parallel to the polishing plane. The brass sleeve in which the specimen was positioned for lapping closely fits the bore of the polishing holder, and is held in place by means of a small thumbscrew. The charged vellum is held in slight tension on a metal backing plate ground to 16-microinch smoothness. The specimen projects from the underside of the holder approximately 0.0002″ and is moved about on the charged surface with a circular motion until this amount is polished away and the holder begins to bear on the vellum.

Alternate polishing and inspection is continued until the desired sectioning plane is reached or until the surface is of the desired quality. Specimens requiring extreme accuracy in sectioning plane position are also checked with micrometer calipers during polishing to determine when the sectioning plane is within 0.0001″ of the desired position. Specimens containing fragile recrystallization formations are polished very carefully with a straight-line motion in a direction parallel to these configurations to avoid damage.

Whenever the vellum becomes darkened it is cleaned with trichlorethylene or other suitably volatile solvent and is recharged with polishing abrasive.

After specimen polishing is completed, significant surface configurations are noted and photographed, since they will be partially obliterated by a subsequent junction-etch treatment. The specimens are left undisturbed in the sleeves until all lapping, polishing, and preliminary microscopy are completed, after which the specimens are removed for etching, to avoid damaging the sleeves.

Junction etching

The purpose of etching is to create line patterns which indicate such things as alloy- or diffused-junction positions within the semiconductor material, alloying behavior during processing, crystallographic misorientation, processing temperatures, upper limit of high-frequency response, and interelectrode capacitance.

The lapped and polished semiconductor surface is attacked by the etching solution at a rate depending on items such as impurity types and concentrations, crystallographic orientation, residual stresses, and the severity of lapping and polishing operations. If a number of sample specimens made from material of the same origin are sectioned with reasonable care, impurity content then becomes the predominant factor governing the etch rate, and the optimum etching time for best etch line definition is easily determined. Accordingly, one-of-a-kind specimens are given several very short etchings and inspections to avoid overetching and loss of etchline definitions.

The etching solution most commonly used consists of 4 parts HNO_3 (70%), 4 parts HF (48%), and 1 part distilled H_2O, by volume. Another solution sometimes used consists of 2 parts HNO_3, 1 part HF, 1 part concentrated H_2SO_4, and 1 or 2 parts distilled H_2O, depending on the desired etching time. The specimen, minus the brass sleeve, is held under a fine stream of the etching solution for the appropriate etching time (usually 1½ or 2 seconds), rinsed and dried, and inspected for etch line definition. Care is taken to avoid overetching, otherwise the specimen must be repolished and the etching repeated.

Microscopy and photography

A Bausch & Lomb Model CMET metallurgical microscope with vertical beam illuminator, 8× objective, and binocular eyepieces was fitted with a special micrometer stage of 0.1″ × 0.1″ movement reading to 0.00001″ on the verniers. One 20× widefield eyepiece contains a 5 × 5 square net reticle through which the specimen image is photographed by means of a 35-mm eyepiece camera, and the other eyepiece contains a graduated cross-scale reticle by which specimen image position and focus are maintained.

Measurement of such specimen configurations as junction separation, and base and recrystallization region thicknesses are customarily made to the nearest 0.00001″ on the micrometer stage. Other specimen dimensions of minor importance may be noted in terms of reticle cross-scale readings.

Occasionally, large panoramic "billboard" photographs of specimen cross sections are needed for demonstrational or educational purposes. Since the length of the semiconductor portion of the specimen is considerably greater than the width of the field of view, the image must be moved in equal increments along the reticle scale while a sequential series of frames is exposed on the 35-mm film, starting at the left end of the image. The result is a series of overlapping pictures in proper order on the negative by which the specimen cross section is viewed in its entirety as in Fig. 4, and from which 4″ × 6″ positive print enlargements can be made for "billboard" mounting. In this figure and in all the following figures, except Fig. 5, the frames have been cut apart and joined together to form continuous panaromic photographs.

Interpretation of data

The great value of microsectioning lies in the skill with which microphotographs are analyzed and evaluated, and in the effectiveness of recommended changes made to correct abnormalities observed in the sectioned specimens. Within limits, the degree of skill increases rapidly as the total number of processed specimens increases.

A number of microphotographs of microsectioned specimens have been selected to illustrate the manner in which an analysis is made. In each figure, two views of the specimen are shown, the "A" view being the as-polished section, and the "B" view the junction-etched section. The superimposed net reticle squares are approximately 0.0047″ in size.

Fig. 5 shows an early version of an *n-p-n* transistor consisting of lead-antimony dots (a) and (b) alloyed on the (100) faces of a thin germanium die (c). This crystallographic orientation leads to an unstable alloying

and recrystallization behavior that is difficult to control, the result being nonplanar *n-p* boundaries (d) and complex (111)-plane configurations (e) between the recrystallized germanium and the dot material. Recrystallization failure and possibly poor voltage breakdown characteristics are indicated by the dark areas (f) under the emitter and collector dots.

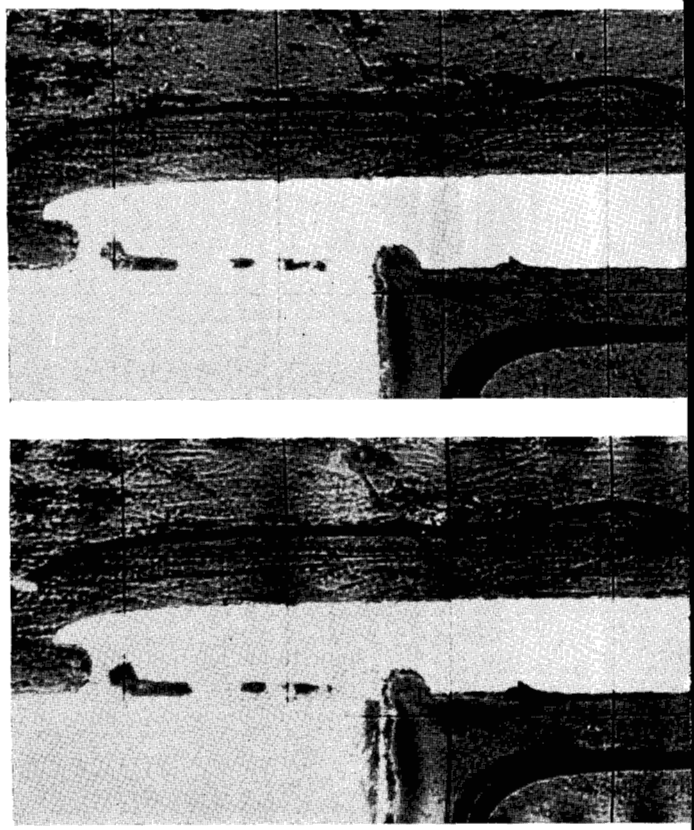
The geometry of another *n-p-n* transistor was greatly improved by use of (111) oriented germanium, as shown in Fig. 6. In view A, the germanium underneath the dots had recrystallized to practically its original thickness at (a). This transistor had been cleanup etched to reduce back-current leakage, so that the germanium outside the dots at (b) is only 60% of its original thickness, and considerable undercutting around the dot peripheries has reduced the germanium thickness to a fragile 20 or 30% of the original at (c) and (d). The bond between the germanium and the washer at (e) and (f) had numerous pits and blowholes. These are objectionable because contaminants lodged therein during cleanup etching are not only difficult to remove, but migrate out of these crevices at some later period of time after the transistor has been encapsulated, destroying its electrical characteristics.

The junction-etched specimen of Fig. 6B shows the more desirable geometrically flat and parallel alloy junctions produced by the stable (111) recrystallization plane. Alloy penetration depth (and hence junction spacing) is more easily and precisely controlled by adjustment of the firing temperature, and such junction configurations provide better high-frequency response without sacrificing voltage breakdown limits between emitter and collector. The 1.5° crystallographic misorientation of this specimen contributed to the "troughing" effect seen under the periphery of the collector dot at (g). This configuration produces deep, narrow crevices during cleanup etching that are difficult to clean out. Poor dot concentricities also contributed to unsatisfactory alloying and electrical characteristics.

The specimen of Fig. 7A had been selected for sectioning without cleanup etching so as not to obliterate recrystallization behavior in the vicinity of the dot peripheries. Bond contours are excellent but several cavities are noticeable at (a) and (b). In view B, recrystallization failure and peripheral troughing are evident at (c) and (d) respectively. Poor dot concentricity and a crystallographic misorientation of 0.80° in the plane of sectioning are also noted.

The *n-p-n* unit of Fig. 8 is another poor specimen, with dot material inclusions (a), recrystallization failure (b), and the peripheral trough (c) being greatly accentuated by excessive dot eccentricity and crystallographic misorientation (0.95°). The bond at (d) is excellent, but that at (e) is unusually bad.

The unetched specimen of Fig. 9A was sectioned through a bond blowhole to determine the extent of this type of cavity. Visual examination of the surface of the specimen before potting showed fine globules of bond material splattered over both the emitter dot and the surface of the germanium within the washer hole. Investigation revealed that the washers had been improperly cleaned before plating, and as the plating alloyed with the germanium to form the bond, vaporization pressure of the contaminants under the plating at (a) actually blew the bond material out of the joint. In view B, dot material inclusions (b) and the peripheral trough effect (c) were minimized because of good dot concentricity and fairly good crystallographic orientation (0.6° error). Two gas bubbles and an emitter dot recrystallization failure are noted at (d) and (e) respectively.



The *p-n-p* transistor of Fig. 10A was selected to show an undesirable laminar recrystallization behavior within the indium collector dot at (a) that had plagued developmental efforts for a time. Since this configuration was extremely fragile, straight-line lapping and polishing only were employed. Polishing was held to a minimum and over-all specimen section quality was partially sacrificed to avoid damaging the 0.0002″ layer of recrystallized germanium within the indium collector dot. Measurements made on the junction-etched specimen in view B showed the alloying or wetting diameter of the collector to be 0.0230″, and the recrystallization diameter to be 0.0256″. Since no cleanup etching had been done, it is apparent that an initial recrystallization in a direction

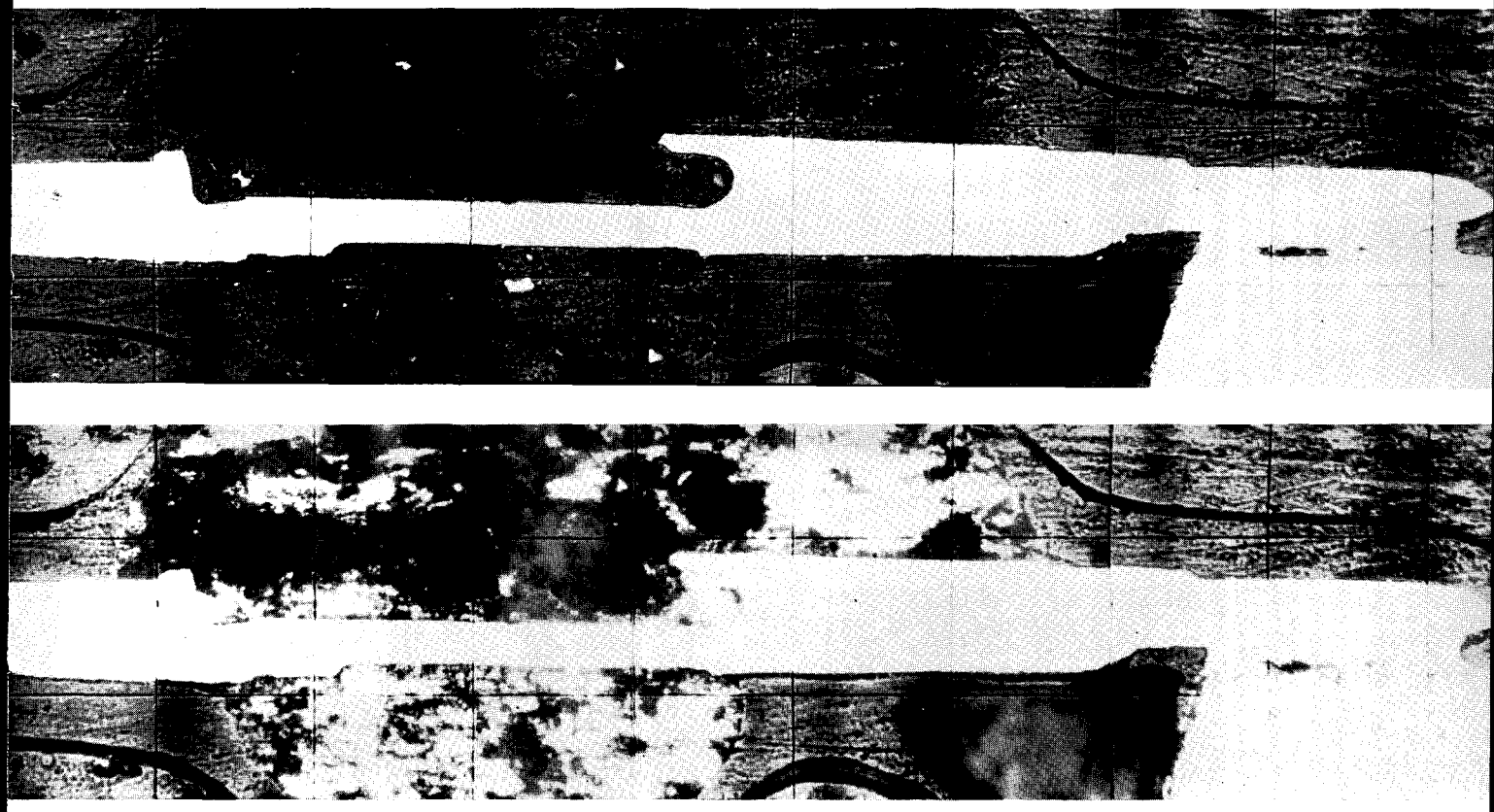


Figure 4 **Sample 35-mm microphotographs showing panoramic view of a specimen.**

normal to the (111) face of the germanium die had taken place around the collector periphery at (b) and (c), before laminar recrystallization in easier directions parallel to the (111) face had begun. The upper surface of this lamination is actually 0.0008″ above the original surface of the germanium die.

Measurements made on the emitter dot of Fig. 10B showed that dot retraction had occurred during recrystallization. The alloying diameter was 0.0120″, the recrystallization diameter was 0.0190″, and the final diameter was 0.0160″, indicating that the dot had retracted 0.003″ at (d). Evidence of laminar recrystallization within the emitter dot 0.0003″ above the die surface is visible, although efforts to preserve the formation were unsuccessful. The 0.00011″ junction spacing was one of the closest ever observed in sectioned specimens. Bond quality was satisfactory in the sectioning plane, although three blowholes were observed in the bond fillet before the specimen was potted. Crystallographic misorientation was 0.35°, and some collector eccentricity was noted.

Figure 11A shows another example of a more common incomplete laminar recrystallization found in some *p-n-p* specimens. Alloying and recrystallization behavior was almost identical to that of the specimen of Fig. 10, except that optimum junction spacing was attained and the dots were nicely centered. However, life expectancy for this unit would have been nil because of numerous crevices within the bonding region.

In general, since *p-n-p* transistors that had laminar recrystallization exhibited increasing back-current leakage as cleanup etching progressed, the units eventually had to be discarded. Analysis of a typical laminar recrystallization serves to explain this behavior and shows why such recrystallization is objectionable. First of all, any extraneous recrystallization of germanium tends to reduce the total amount that would have otherwise recrystallized upon the parent material of the die, so that an undesirable reduction in the thickness of the recrystallized *p*-region occurs. Second, a greater reduction occurs in more critical areas immediately under the periphery of the dot, by reason of the shielding effect of the laminar formation just above. The longer the cleanup etch is allowed to attack this periphery, the deeper and narrower the crevice becomes, and the greater the number of very thin recrystallized regions reached by the etchant under the dot periphery; hence an increase in back-current leakage due to cleaning is inevitable.

In *n-p-n* transistors having dot material inclusions and peripheral trough effects (both contributing seriously to reduction in thickness of the recrystallized *n*-type region), deep, narrow cleanup etch crevices were almost always formed.

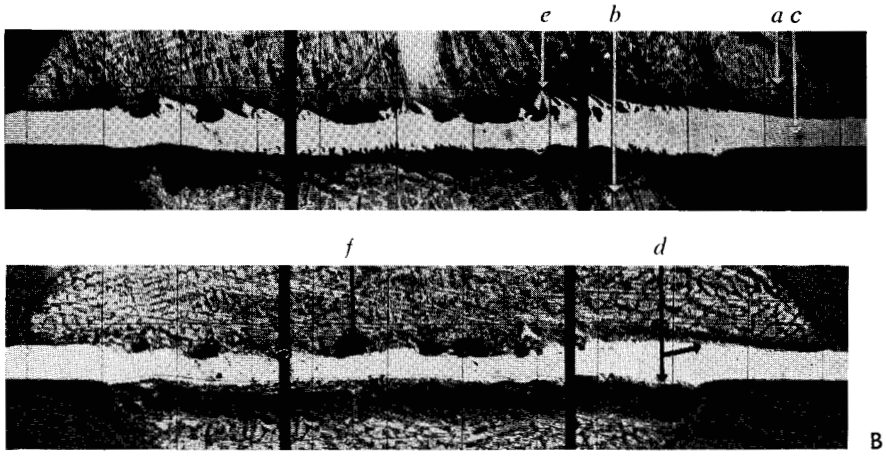


Figure 5 **N-p-n transistor cross-section with (100) oriented germanium showing abnormal recrystallization and non-planar n-p boundaries.**

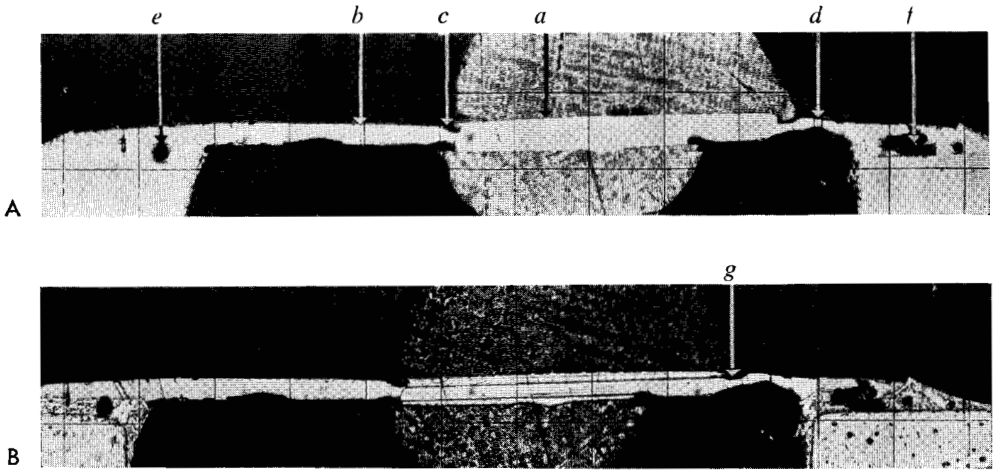


Figure 6 **N-p-n transistor cross-section showing parallel-plane n-p boundaries produced by more stable recrystallization of (111) oriented germanium.**

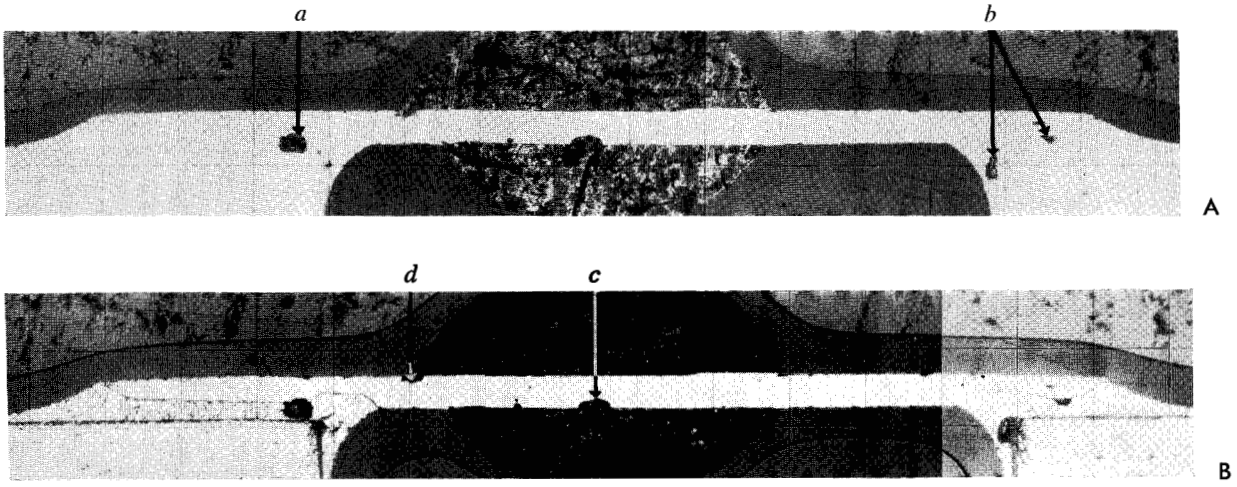


Figure 7 **N-p-n transistor cross-section showing recrystallization defects and bond cavities.**

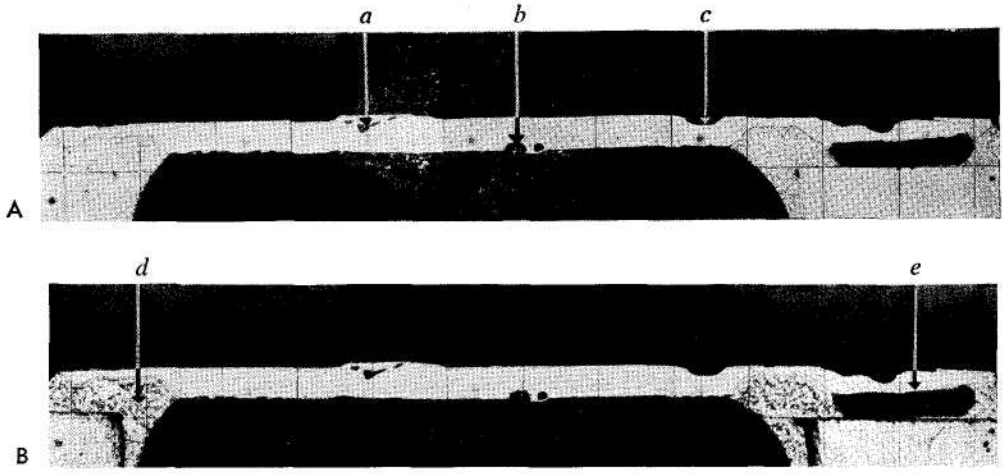


Figure 8 **N-p-n transistor cross-section showing effect of collector eccentricity and germanium orientation error on recrystallization behavior.**

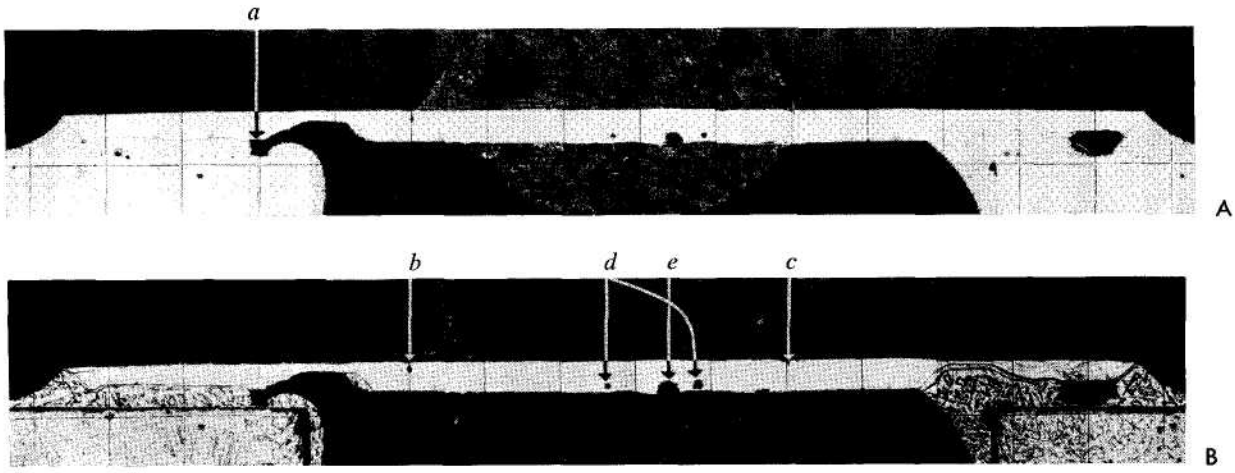


Figure 9 **N-p-n transistor cross-section showing extent of bond blowhole formation and improved recrystallization behavior.**

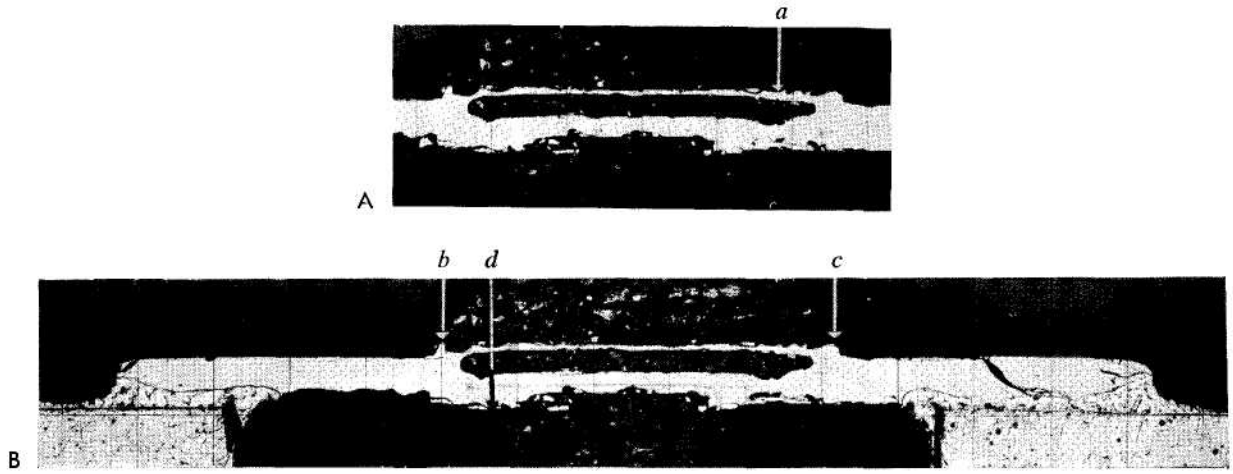


Figure 10 **P-n-p transistor cross-section showing severe laminar recrystallization and emitter retraction.**

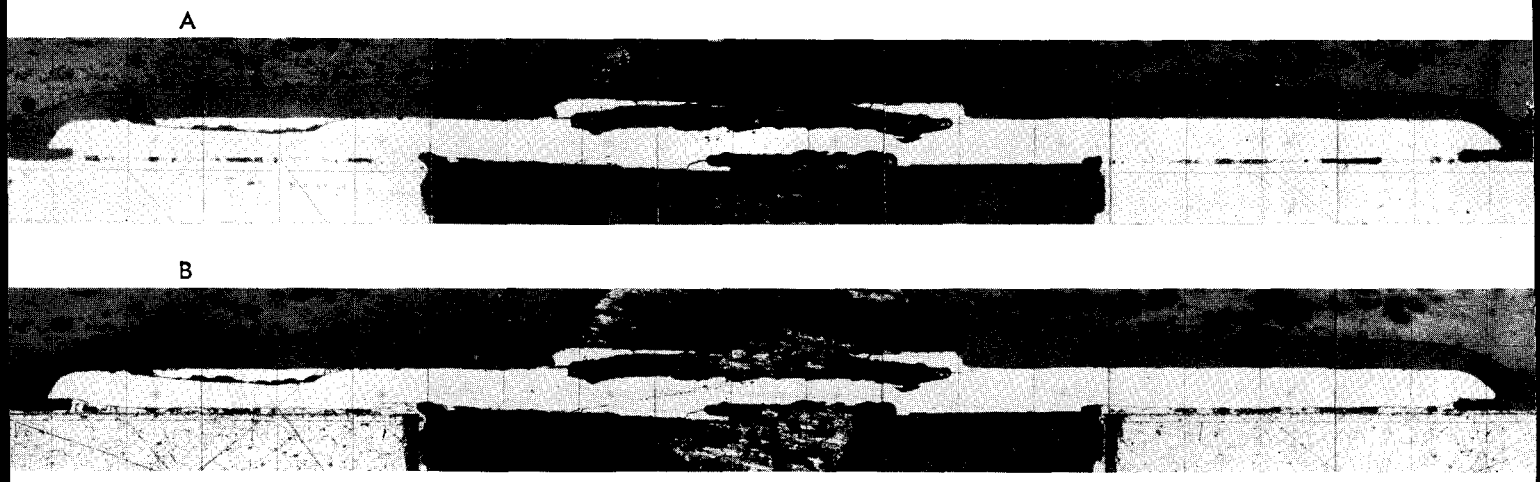


Figure 11 **P-n-p transistor cross-section showing laminar recrystallization and bond cavitation.**

On the basis of the foregoing observations and microphotograph analyses, the following recommendations were made:

1. Adhere to $\pm 0.1°$ tolerance on crystallographic orientation of the germanium dies.
2. Rework firing jigs to secure better accuracy in centering collector and emitter dots with respect to each other and to the hole in the washer.
3. Revise firing temperature cycle, both in heating and in cooling to obtain better alloying and recrystallization behavior.
4. Make necessary changes in initial surface contact conditions between the germanium die and the washer to secure satisfactory bond fillet radii and freedom from blowholes and crevices.

Conclusions

Microsectioning, with appropriate minor variations, can be applied to almost any subminiature item. Major advantages of this technique are (a) precise positioning of the sectioning plane by direct micrometer caliper measurement, (b) maintenance of a planar surface on specimens having adjoining areas of widely varying physical characteristics, (c) preservation of boundary details between such areas, and (d) multiple sectioning in any number of parallel planes to secure a three-dimensional picture of internal contours, *n-p* boundaries, recrystallization regions, et cetera.

The microsectioning technique enables preparation of anywhere from several to a dozen specimens per machine per day, depending on the skill of the technicians, and is considerably more rapid than conventional metallographic techniques.

Use of the microsectioner need not be limited to one specimen at a time, nor only to rough lapping, for that matter. Depending on the extent of the activity, the microsectioner can be easily modified for a multiplicity of synchronized lapping arms, each with its own individual on-off control, or for mechanized polishing operations, either wet or dry. In addition, the convenient small-scale nature of microsectioning permits each laboratory or plant activity to have its own individual equipment setup as the need arises.

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