APPENDIX III

PROCESS NOTES

PROCESS NOTES

Foreword

None of the material in this appendix is to be construed as constituting a process manual. A process manual for the manufacture of quartz resonator plates must contain an abundance of detail, and any attempt to describe a general or comprehensive process for several resonator types would necessarily result in a very large and complex document.

This appendix includes three items for general guidance:-

(1) A reproduction of U. S. Army Signal Equipment Support Agency's "Guide for Preparation of Process Manual For PEM Crystal Unit Contracts." This document has been issued only on ditto sheets, and is included here because it deserves to be published in more permanent form and because it is more useful than its title suggests. It sets a minimum of written description for process sequences, which is of value whether the purpose of the description is to report technical progress or to instruct production foremen.

(2) A very brief general outline emphasizing the function or functions of each process step.

(3) A list of available PEM process manuals, arranged according to type of quartz resonator covered.

These items are followed by a few sections of factual material for use in the manufacture of crystal units.

COMPONENTS AND ELECTRON DEVICES BRANCH
FIELD ENGINEERING DIVISION
U. S. ARMY SIGNAL EQUIPMENT SUPPORT AGENCY

SIGFM/ES-PFE-4 18 September 1957

GUIDE FOR PREPARATION OF PROCESS MANUAL FOR
PEM CRYSTAL UNIT CONTRACTS

Section 2 of Final Reports for Industrial Preparedness Studies for Quartz Crystal Units, shall be prepared in accordance with the following outline:

1. Inspection and Grading
   a. Size, type and grade of quartz.
   b. Equipment, materials and procedures used.
   c. Etching materials and procedures used.

2. Orientation
   a. Equipment, materials and procedures used.

3. Cutting
   a. Description of cutting sequence.
   b. Method and materials used for mounting.
   c. Type of cut.
   d. Thickness and tolerances of wafer cut:
      (1) Diamond concentration, diameter of blade, thickness of blade, diameter of flanges, peripheral speed and rate of feed.
      (2) Life of saw blade.
   e. Type of saw and modifications.

4. Dicing
   a. Method and materials.
   b. Dimensions and thickness of diced blanks with dimensional spreads.
   c. Type of saw or other method used and coolant.
   d. Type of blade, thickness, diamond concentration, diameter, peripheral speed, rate of feed, etc. where applicable.

5. Orientation
   a. Angles: Z-Z', X-X', Y-Y':
      (1) Tolerances.
   b. Type of X-ray equipment:
      (1) Modifications.
      (2) Description of jigs and fixtures.

6. Rough Lapping
   a. Type of lap:
      (1) Speed of rotation.
      (2) Modifications.
   b. Method of conditioning plates:
      (1) Weight of upper plate.
   c. Carrier material:
      (1) Number of blanks per load.
      (2) Thickness of carrier.
      (3) Throw of carrier.
      (4) Shape and size of nest and number of nests per carrier.
   d. Type of abrasive, mixture, method and rate of feed.
   e. Pattern of transposing blanks.
   f. Speed of grinding.
   g. Thickness, or frequency, before and after lapping:
      (1) Tolerances.
      (2) Spread.
   h. Parallelism after lapping and how measured.
   i. Blank classification:
      (1) Frequency or thickness.
      (2) Equipment used.
      (3) Number of classifications.
   j. Yield.
   k. Angle change caused by lapping.

7. Intermediate Lapping
   Same as Paragraph 6.

8. Rounding

a. Type of machine:
(1) Modifications.
(2) Type of wheel, diamond concentration, thickness, and peripheral speed.
b. Feed.
c. Coolant.
d. Type of wax or cement.
e. Length of load and time per load.
f. Diameter of blank after rounding.

9. Finish Lapping
Same as Paragraph 6.

10. Semi-Polishing

Same as Paragraph 6.

11. Contouring

a. Geometry of contour.
b. Type of machine and modifications:
(1) Size of load.
(2) Speeds.
c. Methods of conditioning grinding elements and/or cups, etc.
d. Type of abrasive, mixture, method and rate of feed.
e. Frequency change.
f. Contour tolerances, and method of measurement.
g. Frequency tolerances.
h. Method of maintaining angle.

12. Cleaning prior to Etch

a. Method and materials.

13. Etching

a. Amount of change in frequency.
b. Type of etch:
(1) Grade.
(2) Concentration.
(3) Temperature.

14. Cleaning prior to Plating

a. Equipment.
b. Method and materials.

15. Base Plating

a. Method i.e., sputtered, evaporated, etc..
b. Equipment:
(1) Modifications.
(2) Type, size and placement of cathode or filament;
(a) Plating metal.
c. Description of ion-bombardment or heat cleaning method.
d. Description of masks:
(1) Number of blanks per load.
(2) Method of cleaning.
e. Amount of plate-back in kc and tolerances:
(1) Amount of metal.
(2) Time required.
(3) Frequency spread.
f. Degree of vacuum.
g. Current and voltage (sputtering).
h. Size of plated electrode area.
i. Method of storing blanks between operations.

16. Mounting

a. Method of cleaning bases and storing before use.
b. Diameter of mounting wires.
c. Method and equipment used.
d. Materials used.

17. Intermediate Plating
Same as Paragraph 14.

18. Bonding

a. Type of cement; system, kind and time of storage.
b. Method of curing:
   (1) Temperature and time.
c. Portion of mounting wire engaging blank.
d. Amount of bonding:
   (1) Bonding on one or both sides of blank.
e. Drawing of mount, with enlarged view of bonding area.
f. Handling and storage of blanks after curing of cement.

19. Frequency Plating

Same as Paragraph 14.

20. Canning

a. Method of cleaning cans, handling and storing after cleaning.
b. Method of preparing base and can:
   (1) Flux used.
   (2) Solder used.
c. Method of canning.
d. Method of evacuating and sealing.

NOTES: Where possible, drawings or photographs of special equipments or modifications should be included.

Description of any special features or techniques, such as the use of cold traps, gases,etc. should be included in the report.

This guide is intended in no way to limit the contractor in the preparation of the manual. All other pertinent information and processes not specifically referenced should be included.

PROCESS OUTLINE

PROCESS: Orientation and Sawing

MAIN PURPOSES: To obtain wafers of quartz with the desired orientation with reference to the crystallographic axes. These wafers must be thick enough to permit subsequent lapping down to the desired surface and shape without becoming too thin for the desired frequency. They can be no thinner than the saw blades will saw accurately.

OTHER FUNCTIONS: Elimination of faulty, including twinned, material.

EQUIPMENT: Conoscope, Polariscope, etc. for rough orientation; X-ray for precise orientation; diamond saws; etch tanks, ovens for melting waxes or cements.

ALTERNATIVE POSITION IN THE SEQUENCE: None

PROCESS: Dicing

MAIN PURPOSE: To obtain rectangular plates from the wafers. Usually these are square, and a little larger than the diameter intended for the finished, round plate. A few rectangular finished plates are still used. Circular plates can be cut directly from the wafers if a flattened edge or other mark is left to indicate orientation. Orientation must be indicated to enable a later X-ray test or sorting operation. It must also be retained on blanks which are to be oriented with reference to the mounting supports.

OTHER FUNCTIONS: None

EQUIPMENT: Saw, usually with multiple diamond blades; core-drill for direct cutting of round plates.

ALTERNATIVE POSITION IN THE SEQUENCE: None

PROCESS: X-Ray Sort

MAIN PURPOSE: To determine the angle of the plate and to reject all blanks outside one of the useable tolerances before cost of further processing has been incurred.

OTHER FUNCTIONS: None

EQUIPMENT: Precision X-ray, equipped with holder for quartz plates (preferably a double crystal X-ray).

ALTERNATIVE POSITION IN THE SEQUENCE: The earlier in the sequence the X-ray sort is placed, the less the expenditure wasted on off-angle blanks, and the less the difficulty in preserving the orientation. The later in the sequence the X-ray sort is placed, that is, the nearer the plate has its final shape and the better its surface is, the more accurate the X-ray sort. Current specifications are usually such as to make X-ray sorting of the rough blanks (surfaces as left by the saw) inadvisable. The earliest practical position for the X-ray sort is after the first lapping stage. When precision resonators are being manufactured there are often two X-ray sorts, one after the first lapping stage, a second after one of the last lapping or polishing stages.

PROCESS: Angle Correction

MAIN PURPOSE: To correct the orientation of off-angle blanks.

OTHER FUNCTIONS: None

EQUIPMENT: Fixtured grinder and planetary lap, precision X-ray.

ALTERNATIVE POSITION IN SEQUENCE: Must follow X-ray sort. Probably most useful for large corrections.

PROCESS: Rounding or Edge Grinding

MAIN PURPOSE: To produce a round or rectangular plate of the desired size.

OTHER FUNCTIONS: None

EQUIPMENT: Grinder

ALTERNATIVE POSITION IN THE SEQUENCE: Circular plates may be cut directly from the wafers. Position of rounding operation also depends upon method used to preserve indication of orientation for the X-ray sort process and upon the position in the sequence of the X-ray sort. A few manufacturers who believe that the flatness of very thin, high frequency blanks, near their edges, effects their performance, introduce a second rounding process, very late in the lapping and polishing sequence, in order to remove the outer portions of the plate which normally show the most curvature and most lack of parallelism.

PROCESS OUTLINE (cont'd)

PROCESS: Lapping

MAIN PURPOSE: To produce blanks of the desired thickness, degree of parallelism, and surface finish.

OTHER FUNCTIONS: To prepare blanks for more accurate X-ray sorting.

EQUIPMENT: Lapping machines, equipment for conditioning the plates of lapping machines, abrasive stirrers and devices for feeding abrasive, comparators or other equipment for determining thickness, radio receivers for monitoring change of frequency (thickness), automatic radio shut-off, tumbling equipment.

ALTERNATIVE POSITION IN SEQUENCE: None.
At least two lapping stages are commonly used. Three to five stages are typical. Each successive stage employs a finer abrasive. X-ray sort and rounding may occur between lapping stages.

PROCESS: Contouring and Bevelling

MAIN PURPOSE: To produce a curvature or bevel on plates which are thick in proportion to their diameter, and thus to minimize mounting losses, control coupled modes (activity dips), and control inharmonics.

OTHER FUNCTIONS: To adjust frequency (thickness) and to improve surface finish.

EQUIPMENT: Lens grinders, edge grinders, drum contouring equipment, spherical lap plates, lathe for conditioning spherical laps, abrasive stirrers (radio receivers and automatic shut-off as under Lapping).

ALTERNATIVE POSITION IN THE SEQUENCE: None

PROCESS: Polishing

MAIN PURPOSE: To produce a fine surface finish on high frequency plates.

OTHER PURPOSES: To adjust to frequency; to preserve shape.

EQUIPMENT: Lapping machines (usually of the pin type), specially prepared lap plates (formerly pitch, now usually plates faced with plastic), equipment for preparing and reconditioning plates, abrasive stirrers, etching equipment for measuring surface finish.

ALTERNATIVE POSITION IN SEQUENCE: None

PROCESS: Etching

MAIN PURPOSE: To improve the surface of plates, unless they have a very high polish, and thus to prevent severe upward shift in frequency as the plate loses small particles of quartz with age.

OTHER PURPOSES: To adjust to frequency.

EQUIPMENT: Etch tank, work carrier.

ALTERNATIVE POSITION IN SEQUENCE: None

PROCESS: Cleaning

MAIN PURPOSE: To remove from the blank all foreign material, especially viscous material, which may load the resonator, reducing its activity, and causing both its frequency and activity to be unstable.

OTHER FUNCTIONS: None

EQUIPMENT: Chemicals, detergents, supersonic cleaners, spin dryers, vacuum dryers, ion bombardment equipment in plating machines, vacuum ovens.

ALTERNATIVE POSITION IN SEQUENCE: Plates are repeatedly cleaned throughout the sequence, to enable frequency measurement and inspection, and to improve process control. The most critical cleaning processes are those between final etch or polish and final sealing.

NOTE: Base and mount, and cover must also be cleaned.

PROCESS OUTLINE (cont'd)

PROCESS: Frequency Measurement

MAIN PURPOSE: To determine that plates are of correct thickness at any process point and to enable sorts to reduce thickness spreads of in-going loads, thus improving process control.

OTHER FUNCTION: None

EQUIPMENT: Temporary electrode devices, oscillators, radio receivers, frequency counters, frequency standards.

ALTERNATIVE POSITION IN THE SEQUENCE: Mechanical measurements are sometimes made of thickness in connection with very early lapping stages, but thickness is measured throughout all later stages in terms of frequency.

PROCESS: Plating

MAIN PURPOSES: To provide the resonator with electrodes and to adjust it to frequency.

OTHER FUNCTIONS: Cleaning by means of ion bombardment in evaporation platers.

EQUIPMENT: Evaporation platers, sputtering machines, electroplating equipment, oscillators and frequency standards, frequency counters.

ALTERNATIVE POSITION IN THE SEQUENCE: Usually there are two plating processes, sometimes three, or even four. The first process puts electrodes on both sides of the unmounted plates. The second process comes after the plates are mounted, and adjusts each plate individually to the desired frequency, by adding metal to each side in the case of difficult high frequency resonators. Reverse plating, by chemical or electronic removal of plating (such as reverse sputtering) is also used for final adjustment to frequency.

PROCESS: Mounting and Bonding

MAIN PURPOSE: To place the plate in its permanent supports, which, except in the case of some designs for very low frequency plates, are also the electrical leads to the electrodes.

OTHER FUNCTIONS: None

EQUIPMENT: Usually manual tools and jigs.

ALTERNATIVE POSITION IN THE SEQUENCE: Unless there is a single plating stage, mounting and bonding must come between the two plating stages.

PROCESS: Sealing

MAIN PURPOSE: To seal the resonator hermetically in an inert atmosphere, thus preventing contamination, or corrosion, and stabilizing the unit.

OTHER FUNCTIONS: The atmosphere may also be selected to insure maximum activity (vacuum) or good heat dissipation (helium).

ALTERNATIVE POSITION IN SEQUENCE: None

PROCESS: Final Testing

MAIN PURPOSE: To determine if units meet specifications.

OTHER FUNCTIONS: To provide information for better process control.

EQUIPMENT: According to specifications: leak testing equipment, vibration testers, shock testers, salt spray baths, temperature control chambers, means of continuous measurement of resistance and frequency over temperature ranges.

LIST OF PROCESS MANUALS

Nearly all detailed process manuals are limited in scope to a few, usually one, crystal type. There is one excellent general manual, Handbook -- Fabrication Quartz Crystal Oscillator Units, T.O. 12-1-45, prepared by the Crystal Engineering Branch, Dayton Air Force Depot (15 March, 1957). This volume deals with fabrication machinery and procedures in general, and is supplemented by Planning and Processing Data for Quartz Oscillator Units, T.O. 12-1-56 (27 April, 1959), covering military thickness shear types; and Planning and Fabrication Data on Face Shear Crystal Units (2 May, 1958). These volumes are substantially the work of Mr. Leland T. Sogn.

Union Thermolectric's Cumulative Manufacturing Manual for VHF Crystal Units is a model for minute detail, such as would be used by a production line foreman, but it is limited to one type of crystal unit, except that some material has been added later on the CR-18/U. Union has also published a small, illustrated pamphlet, properly called Introductory Process Manual for Military Quartz Crystal Units, Contract DA36-039-sc-71061 (3 October,1960).

In the following list of some of the more important process manuals prepared under government contracts the manuals have been grouped in a minimum number of categories. They are first divided according to harmonic order, and then according to whether the units are designed for temperature control or for a wide temperature range such as -55° to +90° or to +105°C. The manuals concerned with units not designed for temperature control are subdivided according to the allowed frequency deviation, less than ±0.005% being noted as "tight" tolerance. Manuals concerned with units to operate on the fundamental and to be enclosed in the very small HC-18/U holder are listed separately. This distinction is not made in connection with manuals for units intended for harmonic operation. Many other distinctions are not made in this short list.

INC means that the editor's files do not include a final report or process manual or "Completion Report, Step I."

1. Units Operating on Fundamental - No Temperature Control

Midland, DA36-039-sc-11994, Order 25643-Ph-51-ISD

Midland, DA36-039-sc-81273, Order 7620-PP-59-81-81
[CR-(XM-26)/U, high shock and vibration] INC

Radio Corporation of America, W36-039-sc-38244
[1949]

Radio Corporation of America, DA36-039-sc-5460
[1952]

2. Units Operating on Fundamental - No Temperature Control - TIGHT TOLERANCE

Keystone, DA36-039-sc-72691, Order 50799-PP-56-81-81
[CR-(XM-3)/U] INC

Pan Electronics, DA36-039-sc-72689, Order 50797-Phila-56-81
[CR-18(XM-3)/U]

Scientific Radio Products, DA36-039-sc-70287,
Order 26674-Phila-56-81(s)
[CR-(XM-3)/U]

3. Units Operating on Fundamental with Temperature Control

Piezo Crystal Co., DA36-039-sc-75970, Order 43785-PP-58-81-81
[CR-(XM-14)/U]

Reeves-Hoffman, DA36-039-sc-72405

Scientific Radio Products, DA36-039-sc-73007

4. Units Operating on Fundamental - MINIATURE (HC-18/U) Holder

James Knights, DA36-039-sc-75974, Order 43789-PP-58-81-81, INC

5. <u>Units Operating on Third Harmonic - No Temperature Control</u>

Bulova Watch Co., DA36-039-sc-81271, Order 7618-PP-59-81-81
[CR-(XM-27)/U, high shock and vibration]

Keystone, DA36-039-sc-54696, Order 10687 Ph-55-81

McCoy, DA36-039-sc-66073, Order 16204-Phil-55-81

Midland [see 11994 in Section 1 above]

Pan Electronics, DA36-039-sc-54695, Order 10688-Phil-55-81

6. <u>Units Operating on Third Harmonic - No Temperature Control-TIGHT TOLERANCE</u>

Keystone, DA36-039-sc-75982, Order 43797-PP-58-81-81
[CR-(XM-22)/U] INC

Piezo Crystal Co., DA36-039-sc-72771, Order 53254-Phila-57-81
[CR-52(XM 8)]

7. <u>Units Operating on Third Harmonic with Temperature Control</u>

Bliley Electric, DA36-039-sc-75972, Order 43787-PP-58-81-81
[CR-(XM-15)/U]

8. <u>Units Operating on Fifth Harmonic - No Temperature Control</u>

Bliley Electric, DA36-039-sc-70283, Order 26670-Phil-56-81
[CR-56(XM-4)]

Bliley Electric, DA36-039-sc-81270, Order 7617-PP-59-81-81
[CR-(XM-25)/U, low resistance] INC

P.R. Hoffman, DA36-039-sc-72678, Order 50786-PP-56-81-81

McCoy, DA36-039-sc-70284, Order 26671-P-56-81-81
[CR-56/U]

McCoy, DA36-039-sc-70290, Order 26677-PP-56

Midland, DA36-039-sc-70276, Order 26663-Phil-56-81
[CR-56(XM-1, XM-4)]

Midland, DA36-039-sc-46601, Order 12667-Phila-54-81

Pan Electronics, DA36-039-sc-72680, Order 50788-Phil-56-81
[CR-56]

Piezo Crystal Co. [Hupp], DA36-039-sc-70275,
Order 26662-Phila-56-81
[CR(XM-4)/U] INC

Scientific Radio Products, DA36-039-sc-70280,
Order 26667-Phila-56-81
[CR-(XM-4)]

Scientific Radio Products, DA36-039-sc-46602,
Order 12668-P-54-81
[CR-54]

Union Thermoelectric, DA49-025-sc-118, File 550-OSigO-52
[CR-54]

9. Units Operating on Fifth Harmonic - No Temperature Control - TIGHT TOLERANCE

Bulova Watch Co., DA36-039-sc-75940, Order 56377-PP-57-81-81
[CR-(XM-9)]

McCoy, DA36-039-sc-75942, Order 54276-PP-57-81
[CR-54(XM-9)]

Midland, DA36-039-sc-46601, Order 12667-Phil-54-81
[CR-54]

Midland, DA36-039-sc-75936, Order 54272-Phil-57-81
[CR-54(XM-9)]

10. Units Operating on Fifth Harmonic with Temperature Control

Bliley Electric, DA36-039-sc-75972, Order 43787-PP-58-81-81
[CR-(XM-15)/U]

Wright Electronics, DA36-039-sc-70277, Order 26664-P-56-81
[CR-56/U(XM-6)]

11. Units Operating on Seventh Harmonic

Piezo Crystal Co., DA36-039-sc-81272, Order 7619-PP-59-81-81
[XM-18 also high shock and vibration] INC

12. Glass Enclosed Units

Keystone, DA36-039-sc-81275, Order 7622-PP-59-81-81
[CR-(XM-28)/U] INC

James Knights, DA36-039-sc-81274

McCoy, DA36-039-sc-81269, Order 7616-PP-59-81-81
[CR-(XM-17)/U] INC

McCoy, DA36-039-sc-74942

Scientific Radio Products, DA36-039-sc-74999

Scientific Radio Products, DA36-039-sc-46602,
Order 12668-P-54-81

13. Special Units

Bliley, DA36-039-sc-54697, Order 10688-Phila-55(IPS)-81
[high precision, 5 mc, 5th overtone] INC

Hycon Eastern [Hermes], DA36-039-sc-73234
[filter crystals]

Hycon Eastern [Hermes], DA36-039-sc-81249,
Order 43804-PP-58-81-81
[filters]

James Knights, DA36-039-sc-74884
[phase-stable units] INC

James Knights, DA36-039-sc-70176,
[control at +200°C]

Midland, DA36-039-sc-78273,
[phase-stable units] INC

Union Thermoelectric, DA36-039-sc-71061, Order 27473-Phila-56-51
[VHF Crystal Units, 108 mc, low resistance]

14. Special Process Items

Bulova R & D, DA36-039-sc-54667,
[mechanization]

Bulova R & D, DA36-039-sc-72775, Order 54258-PP-57-81-81
[projection welding]

P.R. Hoffman, DA36-039-sc-75957, Order 56393-Phila-57-81
[saw blades]

Raw Quartz

Natural Raw Quartz

The following paragraphs are excerpts from MIL-C-15729A (18 October, 1951). They constitute a convenient classification of natural quartz stones.

"Faced crystals are crystals each of which must have at least one identifiable natural face; this may be a side (prism) face or a cap (pyramid) face. Faced material 100 to 200 grams in weight shall have at least one identifiable natural face with an area not less than $\frac{1}{4}$ square inch. Faced material 200 grams and over in weight shall have at least one identifiable natural face with an area not less than 3/4 square inch.

"The term "usable portion," as applied to crystal weighing less than 5,000 grams, shall be defined as the largest single continuous volume conforming to the requirements of a given grade. Those portions terminating in a thin section from which a quartz plate of $\frac{1}{2}$ inch cross section cannot be cut, are considered defective in calculating the usable portion of crystals.

"Optical twinning is a detrimental defect readily discernible when the crystal is submerged in an oil bath and examined with polarized light. All strained and twinned volumes shall be considered defective, and such volumes shall be subtracted from the total volume of the crystal when estimating the usable portion.

"Other defects are all those that can ordinarily be detected when a crystal is submerged in an oil bath and examined by moderate arc-lamp illumination. Under these conditions, the defects will deflect some of the light and thereby become luminous and detectable.

"Moderate arc-lamp illumination shall be defined as the light furnished by a $4\frac{1}{2}$ ampere d.c. arc-lamp such as that made by Bausch and Lomb and listed as catalog No. 31-33-81-02 and 31-33-81-03, or equal.

"Polarized light shall be defined as light derived from a type EH-4, 100-watt reflector flood, mercury vapor lamp, transmitted through the polarizing device on the inspection oil bath.

"The oil in the bath shall be free from color (a yellow color absorbs the blue of the arc-light, making it more difficult to pick up defects which scatter blue light) and shall have a refractive index as near as practicable to the refractive index of quartz.

"Quartz crystals weighing 200 grams or more shall be graded as follows:

GRADE I -- Grade 1 quartz is quartz which, in the usable portion shall be free from all defects that can ordinarily be detected in an oil bath, when examined with polarized light and with moderate arc-lamp illumination.

"Grade 1 quartz crystals shall be classified as faced (F) and unfaced (U), divided into percentage limits of usability, and designated as follows:

Designation	Limits of Usability Percent
13	30 to 45
14	45 to 60
16	60 to 100

"The first figure in the designation indicates the grade; the second, the lower percentage limit of usability, except that the figure "4" is used to indicate 45 to 60 percent usability.

GRADE II -- Grade 2 quartz is quartz which does not meet the requirements of grade 1, but which in the usable portion shall be free from all defects that can ordinarily be detected in an oil bath when examined with polarized light and with moderate arc-lamp illumination, except that when examined with moderate arc-lamp illumination the following inclusions are permitted in the usable portion:

(a) Hard blue needles (all types, except when large or present in great numbers).

(b) Soft blue needles (all types, except when present in great numbers).

(c) Color (including color phantoms), provided the light absorption is not so great as to materially interfere with the examination.

(d) Tyndall effect.

(e) Scattered fine or small inclusions (often referred to as "specks" or "points").

"Grade 2 quartz crystals shall be classified as faced (F) and unfaced (U), divided into percentage limits of usability, and designated as follows:

Designation	Limits of Usability Percent
23	30 to 45
24	45 to 60
26	60 to 100

"Each crystal weighing 100 to 200 grams shall have a median length of not less than 2 inches in a direction parallel to the optic axis and an average diameter of not less than 1 inch in a direction perpendicular to the optic axis. It shall be faced and shall have a continuous useable volume, comprising not less than 60 percent of its total volume, equal to or better than grade 2 material.

"Crystals weighing 100 to 200 grams shall be designated as follows:

Designation	Limits of Usability Percent
L6	60 to 100

"The letter "L" indicates the grade of the crystals and the number "6" indicates the lower percentage limit of usability."

<u>NOTE:</u> The most economical size to use to produce plates of a specified size must be determined in each case by a consideration of at least two factors:

1. Yield of inspected blanks per dollar value of quartz.

2. Labor cost per blank.

Cultured ("synthetic") Quartz

Figure 87 shows dimensions of rough cut, AT, plates relative to the economical dimensions of cultured Y-bars.

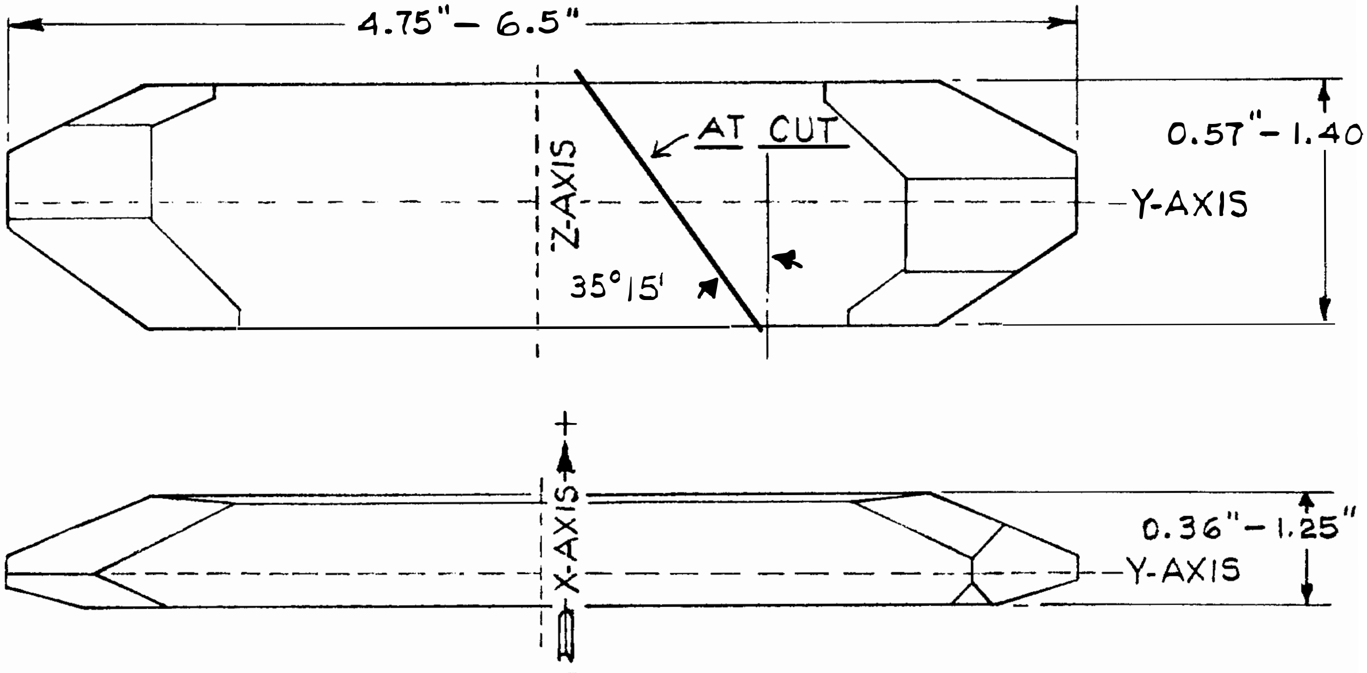
Because the AT cut wafers are usually at an angle of over 35° to the width of the bar, they have one dimension which is approximately 22% greater than the width of the bar, so that the sum of the width of two plates can be greater than the width of the bar even after allowing for a saw cut through the seed area. This is shown by the projection and table at the bottom of Figure 87.

The military specification for Synthetic Quartz of the Y-bar type, No. SQ-3A, specifies for 125 gram bars (0.6" minimum in X direction, 1.00" minimum in the Z direction) a seed not less than 5" long with a cross section not more than 0.04 square inches. In current practice the seed is much smaller than this: 0.090" to 0.100" in the Z direction by less than 0.2" in the X direction, less than 0.02 square inches.

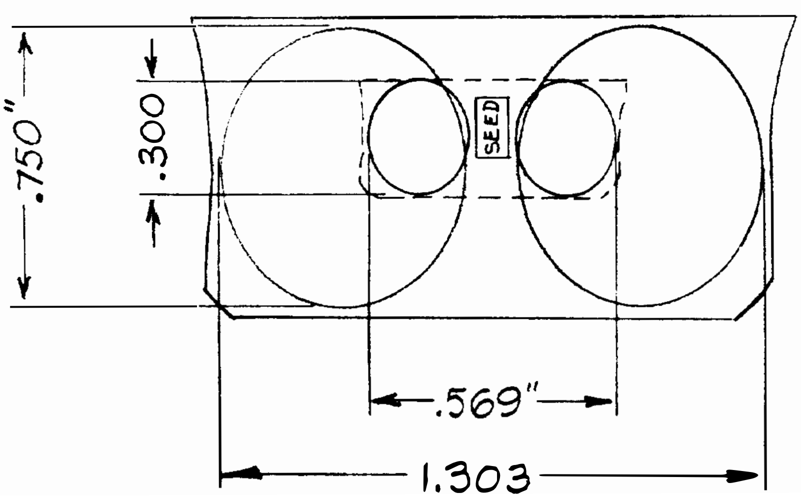
Figure 88 shows a special effort in the direction of using cultured quartz to obtain a high yield. Although avoidance of breakage may cause a production process to aim a little short of this yield, a yield of seven to thirteen times as many good rough blanks per pound from cultured quartz as from natural quartz can be achieved. The ratio is dependent upon the quality, shape, and size of the natural quartz stones. In addition to the greater yield from cultured quartz, the great saving in labor and tooling resulting from using uniform shapes and sizes, grown to economical sizes, and free from twinning and other imperfections is very significant.

FIGURE 87

CULTURED QUARTZ CRYSTAL BARS FOR AT, BT, CT CUTS, ETC.



PROJECTION OF AT ROUGH CUT BLANKS OF 0.750" DIAMETER AND 0.300" DIAMETER ON PLANE SECTION OF CORRESPONDING CRYSTAL BAR LYING PERPENDICULAR TO Y-AXIS.



ROUGH CUT AT BLANK DIAMETER	MIN. WIDTH OF CRYSTAL BAR IN THE DIRECTION OF THE Z-AXIS
.300"	.569"
.320"	.602"
.430"	.775"
.500"	.896"
.600"	1.058"
.650"	1.140"
.700"	1.222"
.750"	1.303"

Sawyer Research Products
Eastlake, Ohio

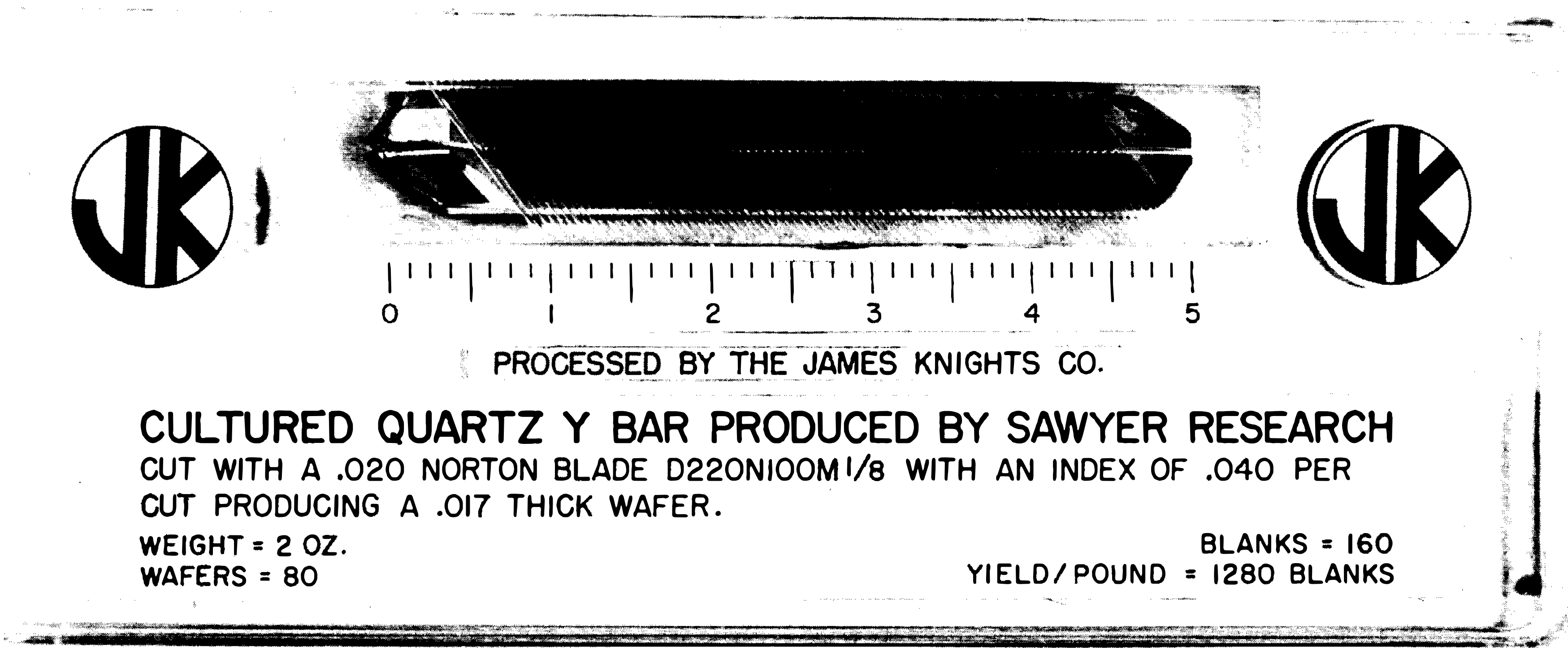

0 1 2 3 4 5
PROCESSED BY THE JAMES KNIGHTS CO.
CULTURED QUARTZ Y BAR PRODUCED BY SAWYER RESEARCH
CUT WITH A .020 NORTON BLADE D220N100M1/8 WITH AN INDEX OF .040 PER CUT PRODUCING A .017 THICK WAFER.
WEIGHT = 2 OZ.
WAFERS = 80
BLANKS = 160
YIELD/POUND = 1280 BLANKS

FIGURE 88

Etch Solutions

Etching is used for the following purposes:

1. X-bars are etched to reveal twinning and determine handedness.
2. Wafers are etched to reveal twinning.
3. Resonator plates are etched to produce final surface, and to adjust to frequency.
4. Samples of polished resonator plates are etched to evaluate surface finish.

FORMULAE FOR AMMONIUM BIFLUORIDE ETCH

Nr.1
4 lbs. ammonium bifluoride
2 quarts denatured alcohol
4 lbs. of fluoboric acid (42%-45%)
3 quarts water

Nr.2
3.48 liter/gal. fluoboric acid, 47%
6.95 lb./gal. ammonium bifluoride

Nr. 3
1 part ammonium bifluoride, by volume
5 parts water
At 60°C ±2°C.

FORMULA FOR HYDROFLUORIC ACID ETCH

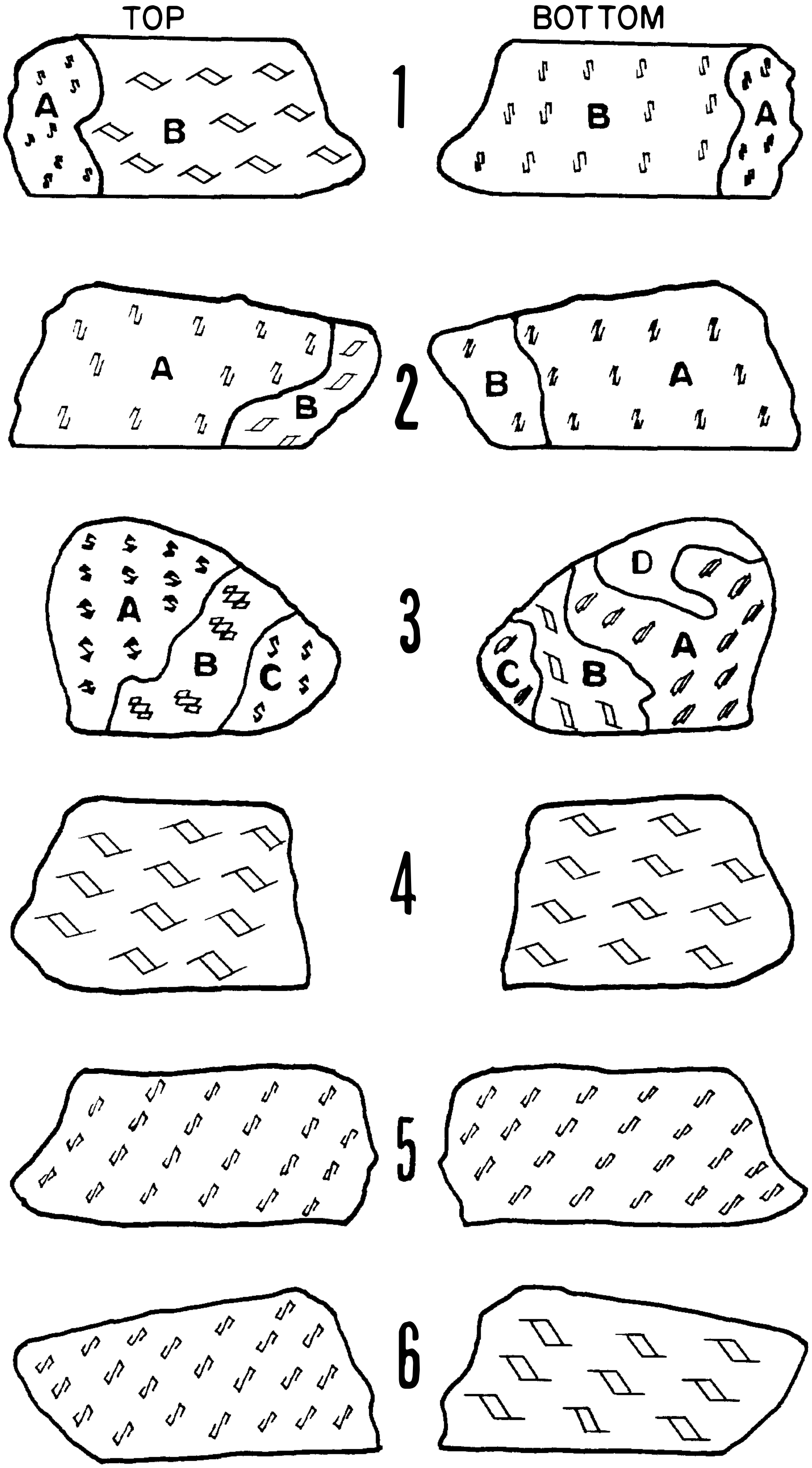
48% Hydrofluoric acid by weight
52% water

The hydrofluoric acid is much faster. Safety regulations prevent its use in some areas.

NOTE: Gates "Quartz Etch" is an effective commercial preparation of proprietary formulation.

TYPICAL X-BAR ETCH PATTERNS

FIGURE 89



1. A BAD: ON TOP ON BOTTOM
   B GOOD: ON TOP ON BOTTOM

2. A BAD: ON TOP ON BOTTOM
   B GOOD: ON TOP ON BOTTOM

3. AREAS A, B, C, AND D ARE ALL BAD, WITH DISTORTED FIGURES THROUGHOUT.

4. SAME PATTERN ( ) ON BOTH TOP AND BOTTOM. BAD.

5. SAME PATTERN ( ) ON BOTH TOP AND BOTTOM. BAD.

6. GOOD BAR: ON TOP FACE, ON BOTTOM FACE; SHARP, UNDISTORTED FIGURES; NO VISIBLE IMPERFECTIONS.

Abrasives

The size of abrasive grains or grit is expressed in the mesh size by some manufacturers, and in the average particle size in microns by others. Mesh size is determined by the number of holes per square inch in the sieve used to screen the grains. The micron size is a linear measurement. One micron is equal to one millionth of a meter(39.37 inches) or approximately forty-millionths of an inch.

The relation between the mesh and grit sizes is given by the following formulas from Heising, Quartz Crystals for Electrical Circuits (1946):

Let "N" equal mesh and "S" equal grit or grain size in microns.

Then:

$$S_{maximum} = 19000/N,$$
$$S_{average} = 15000/N,$$
$$S_{minimum} = 11000/N.$$

Thus: For N = 3000, we have

$$S_{maximum} = 19000/3000 \approx 6 \text{ micron},$$
$$S_{average} = 15000/3000 = 5 \text{ micron},$$
$$S_{minimum} = 11000/3000 \approx 4 \text{ micron}.$$

By transposing the formulas we can determine the mesh for average particle size. For example:

$$5 \text{ microns} = 15000/5 = 3000 \text{ mesh}.$$

A graph for converting between micron size and mesh size follows. Manufacturers and suppliers of abrasives follow different practices with respect to the grading of abrasives, the tendency being to separate out the finer grains more carefully if they are marketable as a finer grade. Moreover, abrasives in bulk, subjected to the movement of the container, or to vibration, tend to regrade themselves, so that there may be a significant difference between abrasives at the top and at the bottom of a container. Generally, lapping on a time basis is unsatisfactory control-wise unless the abrasive has been very carefully graded. Abrasive suppliers attach their own nomenclature to abrasive grades; the nomenclature thus may relate to micron size, to mesh size, or to neither.

Table 10 includes the most commonly used abrasives, and some others. Silicon carbide and aluminum oxide are commonly used for lapping. Many different materials, including very fine aluminum oxide, and cerium oxide, are used for polishing.

The usual practice is to use an abrasive between 50 and 25 microns (300 to 600 mesh) for the first or rough lapping process. One to three intermediate lapping stages employ abrasives from between 25 and 5 microns (600 to 3000 mesh). The final lapping stage (not polishing) employs abrasives from about $12\frac{1}{2}$ to 4 microns (1400 to 3700 mesh). For polishing, abrasives are usually selected by commercial brand. The nominal micron sizes range between 2 and 0.1. For contouring, relatively coarse abrasives are used, 25 to 9.5 microns (600 to 1600 mesh). The contouring process proper may be followed by a surface finishing process employing a finer abrasive, and certain high precision, contoured units are given a full polish.

FIGURE 41

MICRONS VS. MESH SIZE

S = MESH SIZE
N = MICRON SIZE

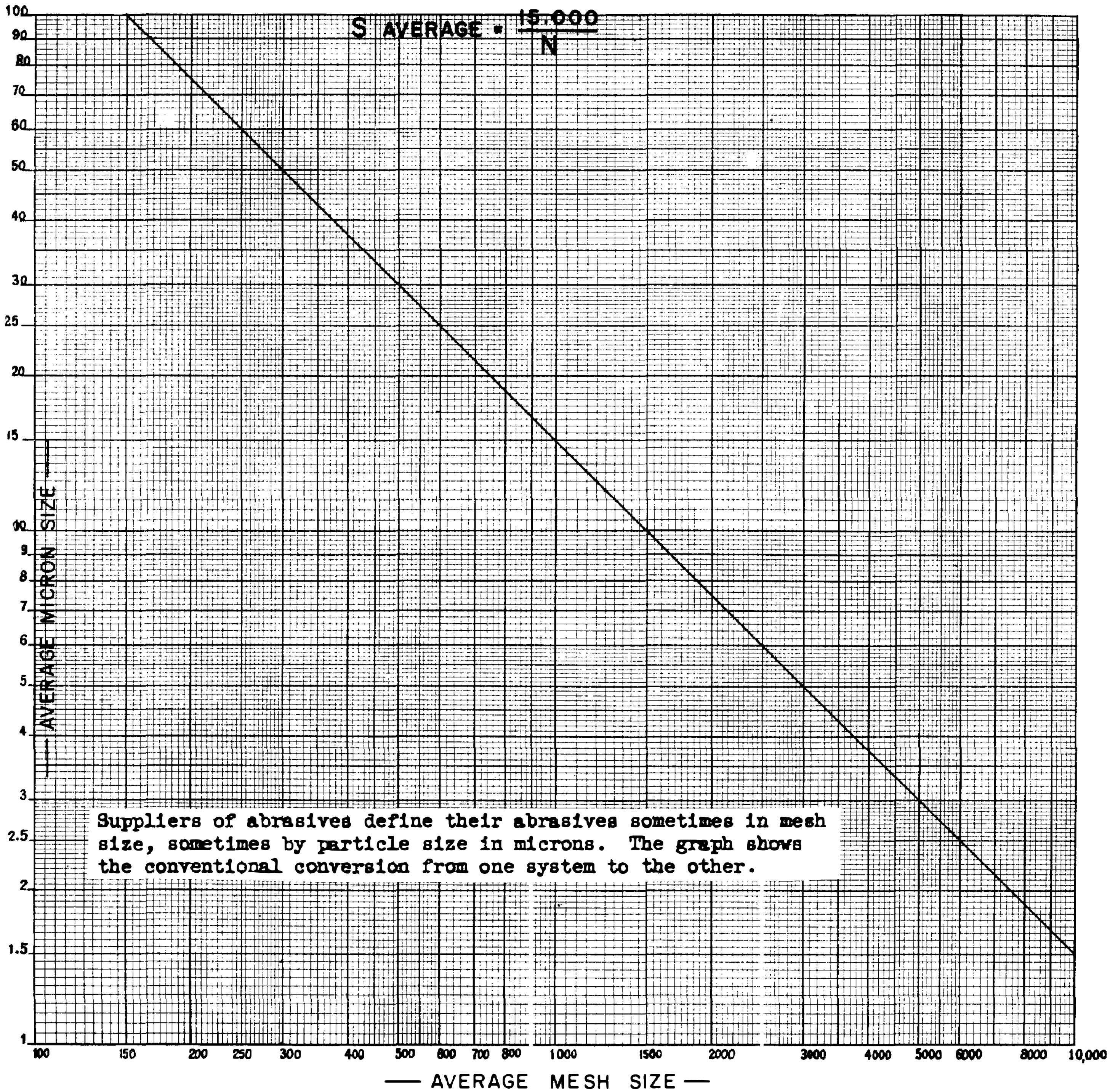


TABLE 10

ABRASIVES

MATERIAL	HARDNESS-MOHS' SCALE	HARDNESS-KNOOP SCALE	TRADE NAMES
Aluminum oxide, natural, or corundum	9	2000	Alumina, Ruby Powder, Carborundum
Aluminum oxide, synthetic	9 to 9½		Aloxite, Alumina, Aluminide, Alundum, Bionite, Boralon, Bolox, Sira
Boron carbide	9½ to 9 3/4	2800	Norbide
Cerium oxide	5½		
Diamond	10	over 7000	
Emery (impure, natural aluminum oxide)	7 to 9		
Ferrous sulphate or anhydrous ferric oxide	4 to 7		Rouge (red from top of roasting crucible, purple or crocus from bottom)
Quartz	7	800	Silex
Quartz, Crypto-Crystalline	7		Jasper Powder
Silicon carbide	9½	2500	Carbolon, Carborundum Crystolon, Carbonite Dicarbo, Electrolon, Silcaride
Zirconium oxide	5.5 to 6.5		Zirox

Cleaning and Prevention of Aging

Changes Within a Resonator Unit

The interior parts of a resonator unit need to be as clean as possible in order that there will be no significant transfer of material onto or away from the vibrating surface of the piezoid, resulting in changes of the electrical parameters. By "clean" we mean, therefore, free from any unnecessary material which is capable of chemical change, of creating chemical change, or of mechanical movement.

Since extremely minute amounts of foreign material are capable of creating significant changes in the electrical parameters of the quartz crystal,* it is usually impossible to deal with contaminants in analytical and quantitative terms. Some of the most troublesome types of contamination are:

> Contaminants resulting from flux used in soldering.
>
> Oil on the surface of or in the metal of the enclosure or mounting support, resulting from the processing of these parts, or from handling.
>
> Foreign material given off at the time of sealing, or later, from the bonding cement.
>
> Various residues from processing steps, including residues from the cleaning agents, themselves.

Some of the more important changes which can occur inside a resonator cannot be prevented by cleaning:

> Non-inert materials which are designed into the unit (such as the metal or other active material used for the holder or the mounting supports, the bonding cement or solder, and the metal electrodes) cannot be rendered inert by cleaning. This should be understood to include the possible migration of metals, such as solders.

---

* See A. W. Warner in Proceedings of the I.R.E., vol. 43 (July, 1955), p. 792.

Failure of the hermetic seal can result in various kinds of change. Any change in the viscosity or density of the atmosphere within a holder will result in a change in the electrical parameters of the unit (see Atmosphere). Moreover, the new atmosphere contains non-inert gasses (oxygen may be assumed as always entering through a leak) which will create chemical changes in certain electrode materials and in bonding cements. The presence or entrance of moisture has particularly large effects upon a unit, especially as the ambient temperature passes through the dew point.

There are two important types of mechanical change which cannot be prevented by cleaning as such. The quartz plate, particularly in the case of a pressure mounted plate, can lose particles of quartz from a disturbed layer left by the lapping processes. This is largely prevented by etching.

The metal electrodes of plated units may change. Such changes, including changes in the crystalline structure of evaporated, sputtered, or electro-plated metal electrodes, are not perfectly understood. In the case of certain processes for the electro-plating of nickel, the relaxation of strains with time has been advanced as a cause of aging.

Temperature changes can result in mechanical changes within the unit. For example, the mounting supports and bonding material may be altered more or less permanently by a change in temperature, resulting in a difference in the stress applied to the quartz plate. Some of the apparent hysteresis phenomenon have this origin.

All of these many and varied changes within a resonator unit which effect its electrical characteristics, whether resulting from contamination, or from the use of materials which are not chemically inert, or of materials which are capable of being changed by temperatures within specified use or storage ranges, are, when their effects occur after the completed unit receives its first test, lumped together under the term "aging."

It is generally assumed that it is easier to prevent aging if the resonator unit is composed of a minimum number of different materials, which materials are as inert as possible, such as gold rather than aluminum or silver whenever possible, or properly cured and oxidized aluminum, glass instead of a metal alloy, etc. On the other hand, an inert material such as a gold electrode or a glass enclosure, may, if inadequately cleaned, be in effect less inert than inferior materials such as nickel-silver alloy and silver which are very clean and enclosed in an inert, dry, atmosphere, in a good hermetic seal.

Since a complete analysis of kinds of contamination and their effects is not possible, the subject can only be covered by listing some of the more common methods and materials used in cleaning. Manufacturers select among these according to economy, the types of resonators which they are producing, the kinds of contamination most troublesome in their plants, etc.

In general, the quartz plates are repeatedly cleaned throughout the process sequence, not only with a view to achieving cleanliness at the end of the sequence, but also in order to make electrical measurements possible at various process stages, and in order to keep the processes under control. For example, if quartz plates are allowed to enter an intermediate lapping stage contaminated by abrasive particles and the abrasive vehicle from a previous stage, electrical measurements will not only be difficult and inaccurate, but the intermediate lapping stage will be largely wasted since the particles of coarser abrasive from the preceding stage will determine the surface finish.

Many solvents are dangerous if improperly handled. In setting up safety procedures some handbook such as N. Irving Sax, _Handbook of Dangerous Materials_ (N.Y., Reinhold, 1951) should be consulted.

Cleaning Processes

Certain crucial cleaning operations are enumerated below.

1. Cleaning Quartz Blanks After Lapping

Early stages of lapping leave a film of oil and abrasive adhering to a blank. The removal of this film is usually done with a simple degreasing operation in which a number of blanks to be cleaned are placed in a wire basket and submerged and agitated in a tank of solvent such as trichlorethylene. Other solvents which may be used are carbon tetrachloride or a ketone such as acetone.

2. Cleaning Before Etching

Since the main reason for etching a blank is to remove a uniform layer of quartz of prescribed thickness from each side of a blank in a pre-determined length of time, it is necessary that the surfaces of the blank be free from any material, such as oil, which is insoluble in the etchant. Oils may be removed by submerging the blank to be cleaned in ethanol, methanol, trichlorotrifluoroethane, trichloroethylene, tetrachloroethylene, or chromic acid. For the best results, some agitation of the quartz surfaces should be employed, as with an ultrasonic generator. Such agitation is imperative if a soap or detergent is used instead of a solvent or acid cleansing agent. After washing, the blanks must be thoroughly rinsed in pure water (distilled or filtered and de-mineralized). The blanks may be dried by spinning in a simple centrifuge, or by drawing a vacuum on their container so as to quickly evaporate the water. Evaporation of the water by heating requires little in the way of equipment, but may result in some breakage.

3. Cleaning Before Plating

The same method may be used to clean blanks before plating that was used before etching, with either one or two additional steps. The first involves heating the blanks to drive off volatile materials. This can be done just before the plating masks containing the blanks are inserted into the vacuum chamber, or the chamber can be equipped with an internal heater. The blanks should be plated at a temperature of about 200°C to insure best adhesion of the electrode metal. An additional refinement of the final cleaning method is the well-known ion bombardment of all interior parts of the vacuum chamber during the first stages of evacuation. This is known to be beneficial not only for cleaning the exposed surfaces of the quartz blanks, but also for cleaning the surfaces of the filament wires, crucibles, electrode raw material, and all other potential sources of contamination.

4. Curing of Bonding Cement

The conductive cements used for rigid attachment of the quartz plate to the support wires contain volatile organic materials which are released from the cement at the elevated curing temperatures. All such material must be removed from the bonds before the resonator is sealed into its cover, in order to prevent its condensation upon the resonator surfaces after exposure of the completed unit to high temperatures. Cement-curing operations, which harden the cement material, remove volatile materials, and increase the electrical conductivity of the cement, are usually simple baking cycles, in which the baking time and maximum temperature to which the cement is heated depend upon the type of cement which is used. The baking should take place in open air or in an evacuated chamber, so that the material evaporated from the cement is carried away from the quartz plates, rather than condensing upon them. Bakelite cement is normally completely cured by baking for one hour at a temperature of 150°C, while Bondmaster M-640 is satisfactory for most purposes after being heated to 175°C for 90 minutes. If Bondmaster cemented units are to be heated to temperatures higher than about 150°C, the cement should be vacuum-baked beforehand.

When electroplating is used for final frequency adjustment, the resonator plate may need to be cleaned after the base-plating and cement-bonding operations have been completed. In this event, it is usually sufficient to brush the plate's surfaces lightly with a camels hair brush while the plate is wetted with ethyl alcohol. Ketone solvents should be avoided here because of their tendency to dissolve the bonding cement.

5. Cleaning Holders (Cans and Bases)

The principal contaminants most likely to be found on the base and cover of a crystal holder are petroleum oils and solder flux. Oils may be removed by washing the parts in a degreasing solvent such as trichlorethylene or alcohol. If an alcohol is used, it will also remove any type of water soluble or rosin flux which may be present. Nickel silver cans may be brightened in appearance by rinsing in solutions of either sodium phosphate tribasic (10% water solution) or ammonium hydorxide (6% water solution). Parts should be thoroughly rinsed in pure water after cleaning or brightening.