AD-751 748

PRECISION SSB CRYSTAL UNITS

John F. Silver, et al

CTS Knights, Incorporated

Prepared for:

Army Electronics Command

September 1972

RESEARCH AND DEVELOPMENT TECHNICAL REPORT
ECOM-0198-I

PRECISION SSB CRYSTAL UNITS

INTERIM REPORT

BY

JOHN F. SILVER
LOUIS A. DICK

SEPTEMBER 1972

CTS Knights, Inc.
Sandwich, Illinois

ECOM

UNITED STATES ARMY ELECTRONICS COMMAND · FORT MONMOUTH, N J

DISTRIBUTION STATEMENT

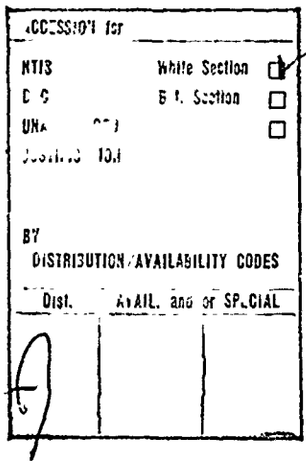
Approved for public release;
distribution unlimited.

N O T I C E S

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

The citation of trade names and names of manufacturers in this report is not to be construed as official Government indorsement or approval of commercial products or services referenced herein.



Disposition

Destroy this report when it is no longer needed. Do not return it to the originator.

TR ECOM 0198-I

REPORTS CONTROL SYMBOL
OSD-B66

PRECISION SSB CRYSTAL UNITS

Interim Technical Report

Contract No. DAAB07-71-C-0198
PPRRC/PD No. C8-1-04300-01-08-CA

Details of illustrations in this document may be better studied on microfiche.

DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited.

Prepared By

John F. Silver
Louis A. Dick

CTS KNIGHTS, INC.
SANDWICH ILLINOIS

For

U.S. Army Electronics Command
Fort Monmouth, New Jersey

ic

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1 ORIGINATING ACTIVITY (Corporate author) CTS KNIGHTS, INC. SANDWICH ILLINOIS	2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED 2b. GROUP

3 REPORT TITLE

PRECISION SSB CRYSTAL UNITS

4 DESCRIPTIVE NOTES (Type of report and inclusive dates)

INTERIM TECHNICAL REPORT, SEPTEMBER 1972

5 AUTHOR(S) (First name, middle initial, last name)

JOHN F. SILVER
LOUIS A. DICK

6 REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO OF REFS
SEPTEMBER 1972	65	-0-

8a. CONTRACT OR GRANT NO	9a. ORIGINATOR'S REPORT NUMBER(S)
DAAB07-71-C-0198 b. PROJECT NO PPRRC/PD No. C8-1-04300-01-08-CA c. d.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) REPORTS CONTROL SYMBOL OSD-1366

10 DISTRIBUTION STATEMENT

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED.

11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY
Reproduced from best available copy.	U.S. ARMY ELECTRONICS COMMAND FORT MONMOUTH, NEW JERSEY

13 ABSTRACT

THIS INTERIM REPORT DEALS WITH THE HISTORY, THEORETICAL MODEL AND DERIVED POSTULATES OF DESIGN, PROCESSING AND MEASUREMENT OF FUNDAMENTAL MODE 5 MHZ RESONATORS CAPABLE OF REACHING AN AGING RATE OF 2×10^{-10} PER WEEK WITHIN EIGHT WEEKS AND WITHSTANDING WIDE RANGE TEMPERATURE CYCLING WITH NO MORE THAN $\pm 1 \times 10^{-8}$ PERMANENT SHIFT IN FREQUENCY PER TEMPERATURE CYCLE. EXPERIMENTAL WORK HAS BEEN LIMITED TO ATTEMPTS TO REPLACE BONDING CEMENTS WITH ELECTRO-PLATED NICKEL BONDS AND THEREBY OBTAIN OPTIMAL ADVANTAGE FROM THE HIGHER TEMPERATURE PROCESSING CAPABILITIES SO AFFORDED. THE RESULTS OF THIS WORK IS GENERALLY PROMISING.

DD FORM 1 NOV 65 1473

UNCLASSIFIED

Security Classification

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
QUARTZ RESONATOR UNITS CRYSTAL RESONATOR PRECISION FUNDAMENTAL MODE						

ib

UNCLASSIFIED

Security Classification

FOREWORD

Organizational and personnel changes encountered as a result of the drastic post-defense cut-back in general quartz crystal industry activity has delayed progress on the generation of hardware required by the contract. However, it is hoped that the time spent in reviewing the state-of-the-art, formulation of a cause and effect model, and the generation of the working and testing hypotheses for hardware now in process will probe to be worth the recording; and perhaps compensate for some of the time lost by providing a more straight-forward approach for fabrication and test that is in general agreement with latest published works in this highly specialized field of endeavor.

TABLE OF CONTENTS

I

OBJECTIVE OF PROJECT

The major objective of this project is well stated in the following selected sentences from the technical guidelines:

"Precision SSB crystal units required for temperature compensated oscillator in the next generation of SSB radio equipment must not age more than 2×10^{-10}/week and when subjected to -55°C to +85°C temperature cycling must stay within 1×10^{-8} of their original frequency." Information on the design parameters necessary for obtaining the required thermal frequency repeatability is lacking and shall be provided by this program.

Detailed specifications for hardware to be submitted are as follows:

5 MHz, Fundamental mode quartz crystal resonator in HC-6/U coldweld type holder.

a) Frequency: 5 MHZ $\pm$120 Hz
b) Power Level: 100 microwatts
c) Upper Turn-over Temperature: 70°C$\pm$1°C
d) Aging: 2×10^{-10}/week, Measurements to be made at upper turn-over temperature and aging rate spec to be reached after not more than 8 weeks of operation at UTP temperature.
e) Q of resonator to be not less than 5×10^5.
f) Thermal Frequency Repeatability (TFR): 1×10^{-8} after being subjected to following temperature cycle:

    1. Start room temperature, heat to upper turning point.
    2. Measure frequency (f) to within $\pm 1 \times 10^{-9}$ upon reaching thermal equilibrium.
    3. Remove UTP temperature, plunge unit into -55°C environment and allow to remain for 30 minutes.
    4. Remove from -55°C chamber and return to UTP temperature.
    5. Re-measure (f2) to within $\pm 1 \times 10^{-9}$ upon reaching thermal equilibrium.
    6. Plunge unit into +85°C environment and allow to remain there for 30 minutes.

7. Remove from +85°C chamber and return to UTP temperature.
8. Again measure (f_3) to within $\pm 1 \times 10^{-9}$ to within $\pm 1 \times 10^{-9}$ upon reaching thermal equilibrium.

Record (f_1), (f_2), and (f_3) - variance of three readings to be not more than $\pm 1 \times 10^{-8}$ to meet letter of specification.

II

SUMMARY OF PERTINENT WORK PREVIOUSLY DONE AND LEADING TO A WORKING HYPOTHESIS.

A relatively large amount of significant work has been done and reported upon since the middle sixties, most all of which has substantially confirmed the original thesis set forth by the pioneer investigations of this period, viz:

A. Aging, the change in resonant frequency of a quartz crystal resonator versus time obtains largely if not completely as a result of either one or the other, or a vectorial summation of both, of the following:

   1. Continued change in the effective mass loading of the resonator caused by the sorption or desorption of transferrable mass.

   2. Strain relief of deformation stress introduced into the completed product during the manufacturing process.

B. Assuming freedom from discontinuities of the type generally associated with partial structure failure or discrete restructuring, both the mass transfer and the strain relief phenomena are assumed to be of the closed system type, in which the resulting action directly diminishes the force or cause of the action, and therefore, follow a logarithmic rate law of the form:

$$\Delta f/f = a + K_{r1} \ln\left(1 + \frac{t}{t_o}\right) \quad [1]$$

(where a, K_{r1} and t_o are constants independent of t); and the directly related exponential relationship:

$$f = f_\infty \left[1 - \frac{f_o}{f_\infty}\right] e - K_{r2} t \quad [2]$$

(f_∞ and f_o represent the resonant frequencies for $t = \infty$ and $t = o$ respectively).

These equations emphasize the concept that for a sealed closed system, free from the discontinuities of structural change there will be an equilibrium frequency (f_{∞}) for any fixed temperature, and that the rate of change with which the operating frequency, f, approaches f_{∞} is uniquely identified by the constant K_{r1}, in [1] or its counterpart K_{r2} in [2].

Such a working hypothesis logically generates keen interest in the pursuit of means of minimizing mass transfer phenomena and deformation stress from precision quartz crystal resonator designs. The evolution of the 5 MHz, fifth overtone design capable of withstanding high temperature (300°C approximately) processing, with near atomical cleanliness, demonstrates dramatically the apparent soundness of this basic postulation, as shown by the curves of Exhibit I. Clearly from this and other evidence, a new generation of precision resonators became producible as the deleterious effects of mass transfer and strain relief of assembly stress distortions became better understood and greatly reduced with high temperature designs and process.

However, the task demands of subject contract extend beyond these accomplishments.

In the conclusion of the report which generated the data of Exhibit I, the authors state:

> "There still exist, however, a number of undesirable effects that degrade the frequency-time characteristics of quartz resonators and warrant further study. These include the small permanent offsets of 1 to 5×10^{-10} observed after restart; short-term noise; random drift of frequency of a few parts per 10^{10}, lasting periods of several hours; and small frequency fluctuations that sometimes occur during the first month of initial stabilization."

And most significantly to the task at hand:

> "These effects probably were present heretofore but were masked by the large mass sorption effects on the frequency."

These undesirable remaining effects would seem to result from discrete structural changes and, indeed, experience suggests that there is a significant degree of correlation between the number and severity of such effects and those designs, processing disciplines and/or environmental abuse more conductive to micro-structural breakdown.

The combination of the two requirements, (1) a 5 MHz fundamental resonator of reasonably low impedance, and (2) resonant frequency retrace to within the relatively small tolerance following the specified wide range temperature excursion dictates the need for structural stability probably equal to if not beyond current state-of-the-art.

Of particular concern is the matter of mounting. The fundamental blank provides much less opportunity to trap activity in the center of the blank by contouring. The edge and particularly the surfaces near the edge of the blank are therefore much more active than the overtone design; dictating that mounting contact be minimal and confined to the outer most edge for maximum decoupling to the mounting structures; but not so minimal as to invite structural breakdown from stress generated by process handling, bake-out temperature, and environmental testing.

Moreover, as succinctly stated above, unless the effects of mass transfer and strain relief are controlled to state-of-the-art capability, the gross frequency-shift effects of these phenomena will mask the effects of the micro-structural changes under consideration and hopelessly confuse the significance of measurements made during the early life of the resonator which is the period during which the unit should exhibit most pronouncedly to what degree it is "breaking in" in logical pursiut of equilibrium or "breaking down".

It must be admitted that the exponential approach to equilibrium resulting from an inertial change or relaxation of stress through displacement of matter within the system may be considered to differ more in degree than concept from micro-structural breakdown of parts of the system. A useful analogy might be that of an R. C. relaxation oscillator consisting of an electrolytic capacitor and a metal film resistor. The circuit is such that it subjects the electrolytic capacitor to a DC potential and the metal film resistor surface is in process of continued oxidation. The DC component of current builds up the effectiveness of the dielectric film asymtotically approaching a final value of capacitance; the self-limiting outer layer oxidation of the metal film resistor likewise asymtotically decreases the change in resistance with the passage of time. If uninterrupted by physical breakdown or dielectric film minute fragmentation, peeling or loss of film, and broken strand or poorly soldered connections, the more history generated by this circuit, the more predictable future behavior becomes. Contrariwise, as physical or structural "breakdown" becomes detectable the future behavior of the circuit becomes less predictable with the passing of time, at least until the exact cause of the breakdown can be ascertained.

It is therefore, the all important difference of whether the changes taking place are such as to better or worsen the predictibility of the future behavior that in the final analysis determines the difference between the constructive equilibrium-seeking changes and destructive "structure rupturing" changes regardless of how micro-dimensioned they may be. The problem that remains is to detect the difference through practical measurement as early in the life of the resonator as possible, and it is from this conclusion that a working hypothesis is based.

III

WORKING HYPOTHESIS

The objective (I) and review of previous work (II) leads to the following working hypothesis:

The two major objectives:

Maximum possible structural stability, considered to be most significant,; and a quite short stabilization time with limited retrace following wide range temperature cycling, though dependent upon different behavior mechanisms are both believed to be most likely attainable by employing the following design and process approach:

1) Minimize to maximum degree possible incipient instabilities in the surface layers of the quartz on all surfaces and edges.

2) Deposit selected metallic films to quartz surface only after surface is cleaned to the maximum degree possible of all absorbed species.

3) Select materials, design and process steps and sequences which provide sufficiently strong but more important, most consistent and stress free interface bonds known in current state-of-the-art.

4) Exclude from the design all materials (notably organics and work hardening metals) whose elastic viability is subject to continued and/or irreversible change due to time, temperature and/or work stress.

5) Minimize reflected reaction from mounts by shaping and orienting blank, and by special design of mounting pads, located at positions of minimum activity. Provide "stress-free" attachment to a mounting structure of high structural integrity but poor acoustic coupling capability.

6) Enclose mounted resonator in hermetically sealed enclosure which together with mounted resonator has been purged of foreign species by high temperature vacuum bake-out as an integral part of sealing operation.

7) Program high temperature and other purging and strain relieving operations both during processing and stabilization phases to accelerate those changes effecting equilibrium in an ordered manner but limited to prevent gross molecular or structural breakdown.

8) Design and critically manage the use of equipment capable of making the precision measurements required to:

   a) Control the "input" of materials and process, and

   b) Measure the "output", i.e., the performance of the finished product.

Details of this general approach follow under:

IV - The Design

V - The Process

VII - Measurement

IV

THE DESIGN

1) The Blank

Cultured precision grade swept quartz.
0.530" dia., Plano convex with 2 diopter contour.
All surfaces and edges lapped, polished, and etched for improved structural stability.
Optically inspected with 20X stereo microscope and rejected if not free from detectable edge fissures, incipient cracks and raw scratches.

2) Mounting Pads

Alternate A & B: 10 kc of chromium covered with 30 kc of gold as measured at 10 MHz. Configuration as shown in Exhibit II.

3) Electrodes

Alternate A: Gold - one f^2. (to frequency).

Alternate B: Copper-gold-one f^2 - start 90% Cu, 10% Au graduating to 90% Au 10% Cu.

Both in configuration shown in Figure 1 for initial study.

4) Mounting Structures

See Exhibit II, Figures 1 and 2.

5) Enclosure:

As specified on Drawings 3 and 4.

IV (a) Design Rationale.

Item 1 of the working hypothesis focuses upon the integrity of the quartz and particularly upon the necessity of eliminating or minimizing the incipient cleavages, distorted lattice structure and stepped layer surfaces. A crude but not too distorted appreciation of the orders of magnitude involved here may be obtained from the simple dimensional relationship:

$$\Delta f/f = K\frac{\Delta t}{t}$$

i.e., the fractional frequency stability will depend directly upon the fractional dimensional deviation in the thickness dimension for thickness shear modes of vibration.

Neglecting the complication of the contoured surface the dimensional stability for a 5 MHz resonator with a stability of $1x10^{-10}$ follows:

$$\Delta f/f = 1x10^{-10} = 5x10^{-4}\text{Hz iN } 5x10^{6}\text{Hz}$$

$$= <1/2x10^{-10}\text{mm IN } 1/2 \text{ mm. (t of 5 MHz blank)}$$

$$= <1/2x10^{-7}\mu \text{ IN } 1/2 \text{ mm}$$

$$= <1/2x10^{-7} \text{ wave lengths of orange yellow light.}$$

$$= <1/2x10^{-3}\text{A IN } 1/2 \text{ mm}$$

$$= 1/6x10^{-3} \text{ atomic layers.}$$

i.e., approximately one sixth of one thousandth of an atomic layer!

Although X-rays with acceleration voltages of billions of volts and wave lengths of as short as $1x10^{-8}$A° are possible, most laboratory work using copper and silver targets emit rays having wave lengths from 1/2 to 1-1/2A°. This is some three orders of magnitude longer than the fractional dimensional deviation associated with parts $x10^{-10}$ frequency stability.

However, research in this field on silicon substrates has demonstrated that due to the strain pattern emanating from isolated cells in the crystal lattice, sufficiently large areas are affected so that X-ray scanning, particularly unique approaches such as the double crystal device developed by Bouchert and described in Exhibit III, provide means of detecting parts $x10^{-9}$ or less anomolies from topographic pictures of the surfaces.

S.I.D. (Source Image Distortion) pictures reveal comparative and semi-quantitative information as shown in Exhibits III, CK2-1,CK6-3, CK5-3, CK4-4.

Although continued study and research will be conducted with the sophisticated X-ray equipment currently under construction, the following approach suggested nearly 10 years ago in the development of the 5 MHz, fifth overtone crystal has proved to be a most useful model and technique for the fabrication of high integrity blanks when conscientiously used, viz:

In spite of the most careful lapping action the uncushioned lapping plate surfaces create minute fractures in the surfaces of the quartz which can only be cleanly removed by using a soft (felt) polishing material and the finest available cerium oxide rouges operated in such a manner and at such speeds as to minimize excessive melting or "flow" of quartz projections.

Edges and corners if expected to be active must be similarly treated, applying the polishing slurry with a felt covered paddle or via alternate means.

Although such an approach is time consuming and allows greater spread of dimension and profile the surface so obtained does demonstrate its integrity since the finished blank when etched retains its highly reflective lustre until the familiar "shingles" due to anisotropic etching rates are clearly visible.

Admittedly, such treatment and inspection side-steps the fractional molecular layer dimensional evaluation suggested above and substitutes an integrated area evaluation which is detectable with microscopic examination utilizing light waves of approximately 6000 A° wave length.

Natural or swept precision grade cultured quartz is logically preferred instead of the rather poor Z growth shown in the Exhibit III.

To minimize the reaction of the mount on the resonator the design provides for mounting on the peripheal edge on the ZZ' axis. The major concern here is to obtain what might be termed macro-mechanical steadfastness over and throughout the smallest contact area practical to attain this steadfastness.

After considerable library research and critical review of in-house experience, the construction chosen is a most carefully applied chromium under gold pad deposited upon specifically cleaned, polished surfaces. The mechanism postulated takes advantage of the very high bonding strength of chromium to Si_2O resulting from the high free energy of

oxidation of chromium Cr_2O_3(-160) relative to -196 for SiO_2 plus the apparent lack of diffusion, migration or other interaction at the chrome-gold interface. The stress generated at the chromium, SiO_2 interface can be tolerated provided that an equilibrium condition is reached, which is free from hysteresis and is non-variant with time and temperature.

A desirable alternative to CR-AU pads would appear to be the tri-metal combination of Ti, palladium and gold overlay. Lack of facilities and time will exclude work on this attractive alternative unless positive evidence of failure of the CR-AU combinations evolves.

Two electrode designs are being tried. Recognizing that as the surface of the quartz approaches atomic cleanliness there is increasing evidence that only the relatively weak Van der Wallian forces hold the gold in position and that such a situation generates doubt as to the steadfastness of behavior when the designs are subjected to temperature retrace tests, an electrode material whose free energy of oxidation relative to SiO_2 provides opportunity for the stronger valence bonds is also being tried. To remain clear of the excess stress and possible continuous oxidation at the interface from oxygen extracted from the quartz, the relatively malleable copper with its relatively small bonding force potential and corresponding lower stress and faster stabilization was recommended since processing limitations prevents complete non-exposure to clean room atmosphere during processing, a gold overlay is required. To hopefully prevent deleterious migration between the copper and gold, a graduated barrier layer of CU to gold is deposited as a simultaneous part of the electrode deposition operation.

The matter of surface contamination is, of course, a processing technique consideration, but it should be mentioned that it is useless to optimize design performance through critical selection of films and expect any correlation in performance unless adsorption and particularly, adsorption of gases carrying hydrophobic materials is controlled. As previously stated, to effect the kind of control required to obtain fast stabilization and good retrace has required high temperature (300°C) processing. Accordingly, all materials and combinations thereof must withstand these temperatures without adverse effect. For designer and processer to retain proper perspective in this matter it is important to remember that at ordinary or atmospheric pressure a clean surface will adsorb a monolayer of foreign species in a few billionths of a second! And even in an ultra-high vacuum (10^{-10} torr) it takes but one hour to add an atomic monolayer of whatever gas (usually CO_2 and O_2) is present.

Exhibit II, Figures 1 and 2 are graphic representations of the designs and their mounting variations.

Common to all designs is the peripheral edge attachment and the use of sulfamate nickel electro-bonding. The sulfamate nickel solution and technique is controlled for minimum stress.

The design shown in Figure 1 is straight-forward and simple in design and process except for unequal thickness uprights.

The design shown in Figure 2 represents an attempt to make use of selected materials and arrangement of same to best decouple the resonator from the reaction effects of the mounts. The copper wire section decouplers are welded to the nickel or stainless steel uprights as .020" wire tapered to flat ribbon with a thickness of approximately .003" adjacent to the blank. As described under the section on processing, partial contact possibilities are minimized by growing a thin layer of SN nickel between the blank pad and the support member.

The choice of copper was predicated upon its quite low sound propogation velocity factor, its high acoustic losses, and its limited crystalline change due to cold working. The acoustical Q of copper is 520 VS 32,500 for nickel.

The nickel coldweld enclosure is less than ideal for the task since high temperature bakeout must be limited to approximately 285°C to prevent softening. For this reason extraordinary chemical cleaning procedures are included.

THE PROCESS

1. - Prepare Blank - (See Insert A for chemical descriptions)

1.1 Select 24°-27' to 28.5' angle

1.2 Round to 530; three steps: - Diamond wheel to 560; #500 grit to 534 and lap with 5μ to 530.

1.3 Grind 523" flats on Z axis.

1.4 Bevel edge - 2 pipe operations - 3 hours #400 grit and 3 hours # 125 grit.

1.5 Finish lap - 3M abrasive.

1.6 Partial contour - one side - 2 diopter - #125 grit.

1.7 Pipe-Bevel contour side - 400 grit 2 hours - 125 grit - 2 hours.

1.8 Finish contour - 2 diopter - 3 M abrasive.

1.9 Pipe bevel - 400 grit 2 hours - 125 grit 2 hours.

1.10 Etch - 30 sec @ 55°C.

1.11 Hand polish edge 27° off of Z axis.

1.12 Final polish both sides.

1.13 Etch - 5 sec.

1.A Visual Inspect - 20x Setero Microscope - Surface, bevel and polish

2. - Clean Blank -

2.1 Scrub - H_2O and alcohol - 4 deionized H_2O rinses @70°

2.2 Place in teflon jig and soak in trichlor 3 min @ 27°C. See Exhibit IV

2.3 Rinse in jig in running distilled H_2O - 30 sec @ 27°C.

2.4 Soak in conc. HNO_3 - 10 min @ 85°C.

2.5 Rinse as per 2.3

2.6 Soak in conc. NH_4OH - 5 min @ 85°C.

2.7 Rinse as per 2.3

2.8 Soak in dilute (25% by vol.) HNO_3 - 2 Min @ 85°C.

2.9 Rinse as per 2.3.

2.10 Soak in "quartz etch" - 5 sec @ 60°C.

2.11 Rinse as per 2.3.

2.12 Soak in conc. HNO_3 - 10 min. @ 85°C.

2.13 Rinse as per 2.3.

2.14 Soak in 30% H_2O_2 - 10 min @ 85°C.

2.15 Rinse as per 2.3.

2.16 Soak in distilled H_2O - 5 min @ 85°C.

2.17 Place in beaker of distilled H_2O and overflow.

2.A Test by subjecting to water vapor test for fractional monolayer freedom from hydrophobic contamination.

3. - Deposit Cr/Au Mounting Pads -

3.1 Place clean blanks into masks which have been hydrogen fired at 350°C. Mounting is completed and filled masks placed into plater at mask temperatures above 100°C.

3.2 Pump down to $5x10^{-6}$ or better vacuum with blanks shielded from evaporation source, vaporize surface layer of Cr from source.

3.3 Rotate rack to unmask blank and deposit Cr on peripheal edge, around corner and pad area of surface; duplicate for opposite mounting pad (27° off 2 axis.).

3.4 Repeat plating procedure to apply Au overplate. Plate back of Cr is 10 kHz at 10 MHz with Au overplate of 30 kHz at 10 MHz.

3.A - Visually inspect with 20X stereo microscope for corners and three surfaces.

4. Clean Bases and Cans. -

4.1 Place bases and cans in stainless steel basket.

4.2 Expose to chlorothene VG at 80°C for 15 min.

4.3 "Reverse current" clean in MacDermid Metex S1701 solution at 65°C using steel anode and 75 amp/ft^2 for 1 min.

4.4 Rinse in agitated distilled H_2O at 85°C for 2 min.

4.5 Place in MacDermid Metex acid salt solution M#629 for 1 min. at 150°F.

4.6 Rinse as per 4.4.

4.7 Soak in 30% H_2O_2 at 85°C for 20 min.

4.8 Rinse in running distilled H_2O.

4.A Apply water vapor test

4.9 Bake H_2 oven at 200°C for 45 min.

4.10 Store cans at 140°C positive pressure area.

5. - Mounting Blank to Base -

5.1 Orient blank in mount and using collodian cement to the B post completely covering "B" pad and contact rod. See Figures 1 and 2 of Exhibit II.

5.2 Dry for 45 min. at 50°C.

5.3 Adjust gap between "A" post coupler and "A" pad to approximately .001". Collodian in this position leaving exposed only 010 to 020" peripheal to de-coupler for electro bonding fillet.

5.4 Dry for 45 min. at 50°C.

5.5 Electro-nickel bond using Barret nickel sulfamate solution chemically adjusted for minimum mechanical stress as per manufacturer's instructions. Plate at 50°C and a current density of 15 ma/CM^2 for 1.5 hours.

5.6 Remove collodian, and clean preparatory to plating post B.

5.7 Slosh unit alternately in distilled H_2O and alcohol at 80°C.

5.8 Soak in acetone bath at 60° for 3 hours.

5.9 Boil, in acetone in closed petri dish for 30 min.

5.10 Ultrasonic clean, low power in acetone 1 minute.

5.11 Soak in alcohol bath at 60° for 3 hours.

5.12 Boil in alcohol in closed petri dish for 30 min. Air dry.

5.13 Mask entire "A" area with collodian and adjust "B" arm for .001 gap between pad and decoupler and repeat operations 5.2 through 5.12.

5.A - Visually inspect Ni bonds grain size, edges and position.

6. - Cleaning Prior to Electrode Deposition -

6.1 Fire mounted units in hydrogen oven at 400°C for 30 minutes.

6.2 Seal units in G7 bulb assemblies using "nitrogen rinse" glass sealing technique and vacuum bake at 350°C and better than 5×10^{-6} torr. vacuum. Glass parts pre-cleaned and fired in nitrogen oven at 350°C for 30 min. and checked with water vapor test.

7.1 - Filament Preparation for Alternative Electrode Designs -

Cu/Au Electrode:

7.1.1 Shape filament with two well loops.
7.1.2 Weight filaments to within .0001 grm.
7.1.3 Melt 99.999% pure Cu into wider loop and weigh.
7.1.4 Melt Au into narrow loop and weigh.
7.1.5 Measure resistance to within .0005 ohms.
7.1.6 Match in pairs according to (1) resistance, (2) weight incl. Cu only and (3) weight total Cu & Au.

Au Only Electrode:
Same as above except single well loop and Au only.

7.2 - Electrode Deposition -

7.2.1 Install previously prepared filaments in matched pairs in plater.

7.2.2 Pump vacuum to better than $1x10^{-6}$ torr.

7.2.3 Apply short pulse of full filament voltage to evaporate outer layers and observe paired response of filaments.

7.2.4 Open plater and insert crystal units in masks.

7.2.5 Pump down to $1x10^{-8}$ or better.

7.2.6 Plate both electrodes simultaneously to frequency. (Average 25 kHz at 5 MHz.)

8. - Cleaning After Electrode Deposition -

8.1 Fire units in hydrogen oven @ 300°C for 30 min.

8.2 Position units and matching cans into jig for high vacuum bakeout and coldweld seal.

8.3 Bakeout at 250°C, $2x10^{-8}$ torr. for 50 hours.

8.4 Seal units in $2x10^{-8}$ torr. vacuum.

8.A - Leak test by immersing in boiling H_2O for ten minutes.

(Identification of chemicals and commercially named materials referenced in this section is made in Insert A following Section V.)

INSERT A

Identification of chemicals and commercially named materials referenced in Section V.

Alcohol: A mixture of 95 PPM alcohol formula "3A" (a specially dentured alcohol) and 5 PPM isopropyl alcohol.

Supplier: Mallinckrodt Chemical Works.

Trichloroethylene: (Transist AR Trademark): Controlled to contain not more than 1 PPM heavy metals.

Supplier: Mallinckrodt Chemical Works.

HNO_3: Reagent grade, A.C.S. assay (HNO_3): 70.0 - 71.0%.

Supplier: Specialy Chemical Division, Allied Chemical Corporation.

NH_4OH: Reagent grade, used in concentrated solution.

Supplier: J. T. Baker Chemical Company

Quartz Etch: A commercial preparation composed in part of ammonium biflouride and sugar.

Supplier: Geo. W. Gates & Co., Inc.

H_2O_2: 30% reagent grade, A.C.S.

Supplier: J. T. Baker Chemical Company

Distilled H_2O: D.C. Resistance more than 130,000 Ohms.

Supplier: Jensen Water Company

Chlorothene VG: An inhibited 1,1,1 trichloroethane used as a vapor degreaser.

Supplier: Dow Chemical Company

Metex Acid Salt M-629: A mixture of acid salts, activators and surface active agents.

Supplier: MacDermid, Inc.

Collodian: Any commercially available non-pigmented fingernail polish.

Supplier: Dura-Gloss, T.M.

Barret Nickel Sulfamate Solution: A commercial solution, type SN, for electro deposition of nickel.

Supplier: Allied-Kelite Division of the Richardson Co.

VI

MEASUREMENTS

A - The Completed Product.

The measurement set-up shown in Exhibit V has a demonstratable precision and repeatability of $\pm 2 \times 10^{-10}$ for 5 MHz resonators with parameters such as those of subject contract.

A passive measurement system is inherently a more basic and direct method of determining the variance of resonator parameters than the alternative of recording the frequency stability of a continously running oscillator utilizing the resonator, since it provides greater opportunity to directly control and isolate the effects of resonator parameter changes from other associated circuitry changes.

Commercially available frequency synthesizers will provide the stability and resolution to manually resolve frequency changes of $\pm 2 \times 10^{-10}$.

The in-plant bridge (Figure 2) and two stage proportional control oven provides means of selectively measuring the frequency of twelve crystals on a programmed schedule.

The bridge is constructed to be inherently balanced, incorporating a double shielded transformer and vernier remote control adjustment of R such that when the motional arm of the crystal is tuned to resonance amplitude null indications of -120 db below 0 dbm are readibly discernible on the amplitude meter of the VTVM. The VTVM selectivity for this indicator is 1000 Hz.

An alternate AM null detection indicator incorporates a communications receiver operating in the selective C.W. mode with its IF signal feeding a linearly swept scope and is included as an integral part of the measurement set-up. The scope provides means for the observer to selectively null against the 5 MHz signal as selected by eye on the scope display.

The combination of the phase detector with its very narrow (1 Hz) bandwidth, its discriminator analogue output, and the complimentary analogue control of the 1×10^{-3} Hz decade of the synthesizer provides means of obtaining closed loop readings directly and conveniently.

To optimize the validity of such readings a measurement procedure is necessary to; (1) zero out the phase shift of the cables, etc., and (2) operate slightly off of absolute R balance to provide consistent sense for the feed back control. This second requirement can be met with less than 3 db of R imbalance and without significant effect on the frequency reading for reactance nulls. The built in phase shifter control of the VTVM provides direct means of accomplishing the first but to obtain maximum precision closed loop frequency readings are not to be compared to open loop readings when recording increments of change.

The computing counter printer combination will display visually, print out and plot on a continuousstrip recorder to a resolution of $\pm 2 \times 10^{-10}$ at 5 MHz. Error multiplier circuitry may be utilized to extend this resolution.

Finally, as shown the oven includes a built in precision oscillator utilizing a MOS FET with AGC capability of operating fundamental or third overtone 5 MHz crystals with high gain AGC controlled drive levels. It is not intended that these systems be used interchangeable except for analysis purposes, since minor changes in internal temperature distribution occur when switching from one to the other. However, continously running the AC cut and recording temperature may offer obvious advantage.

The set-up is expensive but universal and flexible for general purpose duty beyond subject contract. Additional oven capacity will be added.

Figure 2 is a schematic and equivalent circuit representation of the bridge against which Equation [3] can be readily examined and better understood.

VII

EXPERIMENTAL RESULTS

1. Finished Product -

To establish some reference, a 5 MHz fundamental coldweld unit manufactured under a previous ECOM R & D Contract No. DA 28-043-AMC-02183(E) which utilized careful blank preparation and limited (285°C) high temperature bakeout processing, was taken from room temperature shelf storage, operated for five (5) days at T.P. temperature to stabilize, and then the temperature cycled to determine possibility of meeting the retrace specification. To eliminate effect of removing and replacing resonator in oscillator-oven, the complete unit was cycled. As shown in Exhibit VI, the time required for the complete unit to cool off or warm up to operating temperature from the -55°C cold reference point was approximately 1.5 hours whereas the specification infers a faster thermal gradient.

Recognizing this distortion in the test the initial stabilizing time and retrace as exhibited by Exhibit VI indicated that mass transfer and irreversible strain relief were sufficiently under control to probably meet the contract specifications.

The test was deemed important in demonstrating that carefully made "high temperature processed" units even containing organic cement could retain their integrity in storage for time periods of greater than one year.

2. Blank Stress Measurements -

The quartz plate geometry previously generated was free enough from coupled modes to postpone changes in design until effects of mounting configuration on temperature coefficient could be ascertained. As stated previously, minor variations in contour were expected to be traded off for the better polished surface attained by using a soft polishing matter and the minimization of an amorphous quartz coating on the surface of the blank.

From our calculations an angle spread of ± 0.6 minutes is required to obtain a ± 1°C spread in upper turnover point. However, actual variation in turning point temperature and to a lesser degree the difference between turning points is influenced by contour and especially mounting stress.

Table I of Exhibit VII presents two columns of carefully measured upper turning points. Plano-convex blanks used for both groups were screened by careful X-ray remeasurement prior to processing and targeted to yield 70°C ±1°C UTP.

The same type of mounting clips were used for both cement and electro-nickel bonding. However, the cement was critically and sparingly limited to the small areas of blank circumscribed by the holes in the mounting clips whereas the electro-bonding joined the clip to the blank at all perimeters of contact.

The greater downshifting of the electro-nickel bonded units was a first indication of greater mounting stress for this construction and the critical degree to which the electro-nickel bath need be adjusted to provide stress-free bonding.

Table 2 of this same exhibit sets forth a further attempt to quantitatively correlate temperature characteristic variation with measurable parameters of the blank and/or assembly during processing. A_1 and A_2 show two consecutive series of readings on a blank with CR/AU + Ni mounting pads. Column A_3 lists three readings taken within 1/2 hour on same blank after pads were removed with a razor blade.

Note that before pads were removed variation in readings was as much as 0.8 minutes, but only 0.1 minute after pads were removed.

Blank and coating combinations were prepared and forwarded to the engineering experiment station of Georgia Tech. for X-ray SID and topographical photos. Exhibit VIII shows selected reproductions of the group. The following conclusions were reached:-

1) The cultured quartz specimen from which the blanks were obtained contained an unusually high degree of stress due to lattice structure deformation. The topographic photo CK2-1 demonstrates this condition most explicitly.

2) 750A° of Cr followed by 2250A° of Au mounting pads with mounting clips properly aligned and affixed with DuPont 5504 cement showed the least mounting stress. See CK6-3.

3) 750A° C_r with 2250A° evaporated nickel overlay and 5504A cemented clip construction showed only slightly greater stress around the pad than the C_r/A_u pad. See CK5-3.

4) 750A° C_r with 2250A° A_u mounting pads bonded into clips with sulfamate process nickel intensified stress patterns as shown by CK4-4.

Annealing the above specimens at 300°C in the hydrogen furnace did not significantly change the patterns.

A discussion with the Barrett Company chemistry laboratory confirmed our previous understanding that the stress free characteristics of the sulfamate process result from the purity of nickel plate obtained and therefore no significant difference should be observed between evaporated pure nickel and sulfamate process deposited nickel. It is not clear and admittedly complex to understand how the stress can then be reversed under certain sulfamate conditions and this problem is under continued exploration.

3. Double Crystal X-ray Topographical Method of Photographing Lattice Strains.

The above referenced X-ray photos would indicate that more sensitive means of examining lattice strains resulting from coating and mounting stress as well as causes more intinsic to the quartz would be most desirable in the development and manufacturing control of precision crystal resonators.

Arrangements were negotiated and work is currently under way at the Turner Laboratory, Goshen College, Goshen, Indiana to complete a double crystal X-ray topographic camera. This approach as shown in Exhibit III is sufficiently sensitive to detect strain anomolies in silicon to resolutions of parts per billion.

The device was developed by Dr. Robert C. Buschert, Director of the Turner Laboratories and Professor of Physics at Goshen College. Dr. Buschert is personally designing and directing the work on the unit for quartz crystal use and it is expected that first photos can be made during the third quarter of 1972.

4 - Mounting Considerations.

As emphasized in the working hypothesis the major area of concern should be mounting the blank in such a manner as to minimize reaction and irreversible stress-strain equilibria between the quartz and the mounting structure.

Previous designs of best performance fundamental designs utilized selected conductive cements to affix the blank to the mounting clip. Repeated experience at Knights and elsewhere demonstrated that cements which were too brittle "broke down" in discrete and usually minute increments causing frequency jumps, poor retrace and "noisy" short term stability. DuPont 5504A demonstrated a capability of withstanding temperature bakeouts approaching 300°C without apparent loss of viability.

The sulfamate electro-nickel bonding process as developed by Bernstein and his co-workers at ECOM Labs. provides a most promising means of eliminating all organic materials from the design.

First designs utilized the same clip mount placement as had the cemented designs. However, no positive means of confining the electro-bond attachment was provided such as could be attained by applying cement only through the hole in the clip. The electro-nickel bond at the edges of the clip parallels a mechanical contact and with high liklihood of electrolite entrapment.

The margin of mechanical strength available with the design shown in Exhibit II, Figure 1 was determined by purposely mounting the blank .025" off center and bonding the blank on the edge with minimal bonding fillets, then subjecting the assembly to a series of impact tests in three planes. 3000G's at .35 milliseconds was required to fracture the bond and in each case the Cr/Au pads tore loose from the quartz.

The process of affixing the blank to the two points of mounting in a strain-free and homogenous manner is described in the process section of this report. The method is currently not a very straight forward technique from a production point of view, but does accomplish what is believed to be a most important aspect of obtaining an homogenous bond by completely sandwiching a layer of electro deposited sulfamate nickel between the gold overlay chromium pad and the nickel or copper decoupler which in turn is welded to the upright supports. It is hoped that noise spectrum measurements (short term noise) can be useful in evaluating the integrity of these attachments.

5 - Mounting Pads and Interfacial Bonding.

An attempted review of the state-of-the-art in the increasingly important field of interfacial relationships for thin film deposits quickly convinces the reviewer that this is a field in an unusually high state of investigation and that new techniques such as scanning X-ray and electron microscopy, low and high energy electron diffraction, plus a wide range of spectrographic analysis including, but not limited to the now popular Auger surface spectroscopy is generating data faster than it can be collated into working models that are generic enough to accept as textbook postulates.

Laboratory work on this subject was limited to the tools and facilities available. However, the quartz crystal operation has always been fortunate, or unfortunate, to the degree that the piezoelectric quartz resonator with its exceptionally high Q so dominantly and precisely determines the resonant frequency of electronic circuitry and so can be measured to degrees of precision greater than parts per 10 billion which until most recently has been better than any mechanical, optical or chemical means heretofore available.

All fundamental designs manufactured to date using electro-nickel bonded clip mounts developed what appeared to be noisy or erratic contact behavior. The electro-nickel bond was suspected and the design changed as previously discussed and shown in Exhibit II. Additional further probing disclosed that the mounting pads were not adhering well.

Poor adherence of the Cr/Au pad to the quartz was at first puzzling since design and process used were supposedly time proven for many types of precision crystals.

At first the sulfamate plating solution and the deviations in cleaning procedures adopted in the application of the electro-nickel bond was suspected but it is now reasonably certain that the problem was contamination resulting from a combination of marginal processing methods and lack of disciplined control of the special non-production lots run through established production positions.

A relatively intensive cleaning technique program was then initiated resulting in the following findings or confirmations:

1. The water break test followed by the atomized H_2O leading angle droplet test for hydrophobic contaminants is an effective and most useful test for mono-molecular and fractional layer contamination levels provided, however:

   1.1 Both use and evaluation techniques are regularly calibrated with involved personnel using freshly cleaved mica, de-ionized H_2O and disciplined means of observation.

   1.2 Misleading results can be obtained if final rinse of specimen is not de-ionized H_2O. In particular when wetting agents or reagent alcohol formulae rinses were used there was apparent greater low angle wetting on less clean surfaces.

The inclusion of a final rinse of de-ionized H_2O after so-called standard blank scrubbing operation follwed by an H_2O "boil" at controlled temperatures, and this followed by a 350°C N_2 "flow-through" bake-out, produced blanks which not only tested clean by the atomized H_2O test but provided Cr/Au mounting pad bonding strengths consistently stronger than the quartz.

An equally important operation in obtaining strong pad bonds was that of shielding the blank from the initial emission of filament so that the quartz was preserved clean for pure chromium deposit.

Exhibit IV shows the teflon cleaning jig developed for the cleaning experiments. Features of the jig are:

1. Blanks are individually positioned for easy and sure identification.

2. Nesting means designed for maximum exposure of blank (minimum masking).

3. Solvent flow-through maximized.

6. Performance Tests.

Data run on all units finished to date is thought to be misleading since the degradation logically results from the poor bonds existing between mounting pads and blanks and therefore adversely reflect upon the usefulness of electro-nickel bonding and high temperature processing.

Exhibit IX is a report and graph of one of a lot of third overtone 5 MHz units utilizing clip mount electro-nickel bonded

to Cr/Au mounting pads. These units were processed through regular production and there is no history of adherence problems of the pads when these units were built.

High temperature vacuum bakeout processing used on these units and critical customer feed-back is all positive. The units stabilize in 4 to 6 weeks to better than $1x10^{-9}$ per day and as can be seen, warmup and retrace is within less than $5x10^{-9}$.

VIII

CONCLUSIONS

The period immediately ahead should provide opportunity to evaluate the crucial question underlying the major reason for the work done under the contract, i.e., does electro-nickel bonding or some other improved bonding means provide the opportunity of utilizing high temperature processing with resulting shorter stabilizing times and improved retrace characteristics on 5 MHz fundamental crystals as is now accepted fact on high temperature processed overtone crystals?

It is the intent of this report to state that although the problem is believed to be well defined with significant and important confirmation and understanding of the basic theoretical and practical differences logically prerequisite to building a fundamental resonator approaching the precision and integrity of its overtone counterpart, no units incorporating these basic prerequisites have been completed and evaluated in time for this report.

Further it must be recognized that the success of cements in coping with the problem of affixing a non-quiessent edge of the blank to its mounting structure may lie in what this work is predetermined to eliminate, i.e., the lossy plasticity of these materials as contrasted to the more crystalline and elastic structure of metals.

Although results obtained to date offer little if any factual data in support of meeting the specifications with metallic bonding means, it is significant that considerable work has been done to remove or destroy apparent evidence that metallic bonding was the reason for performance inferior to that of selected cemented mounting bonds.

The period ahead should provide the opportunity for critical testing of resonators utlizing high temperature designs and processing techniques and manufactured with the precise control required to confirm our theoretical model.

EXHIBITS

Exhibits, Continued

EXHIBIT I

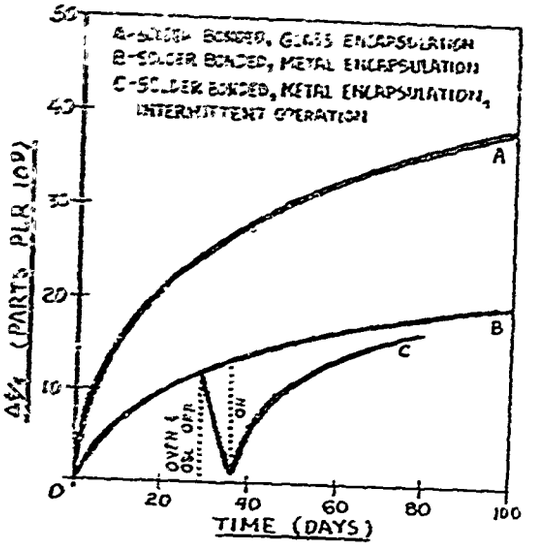


Figure 1 Frequency stabilization of low-temperature processed 5 MHz crystal units.

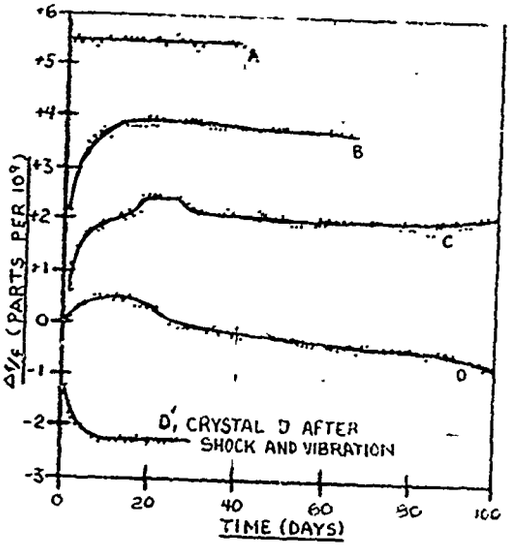


Figure 2 Frequency stability of high-temperature processed 5 MHz crystal units.

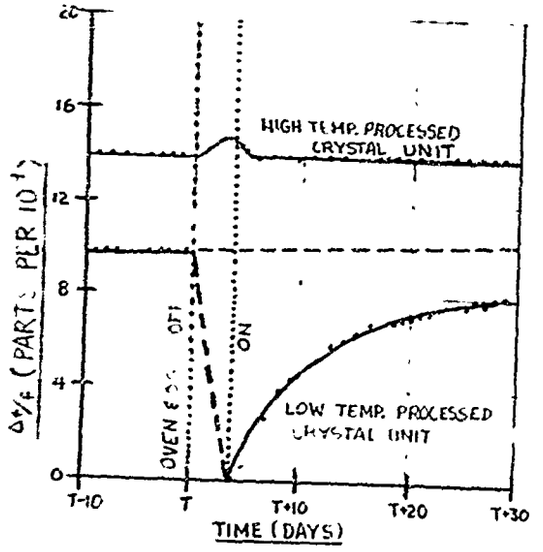


Figure 3 Frequency stability of 5 MHz crystal units aft after ove shutdown.

Data shown here is taken from article by R.J. Bryne and J.L. Hokanson "Effect of High Temperature Processing on the Aging Behavior of Precision 5 MHz Quartz Crystal Units."

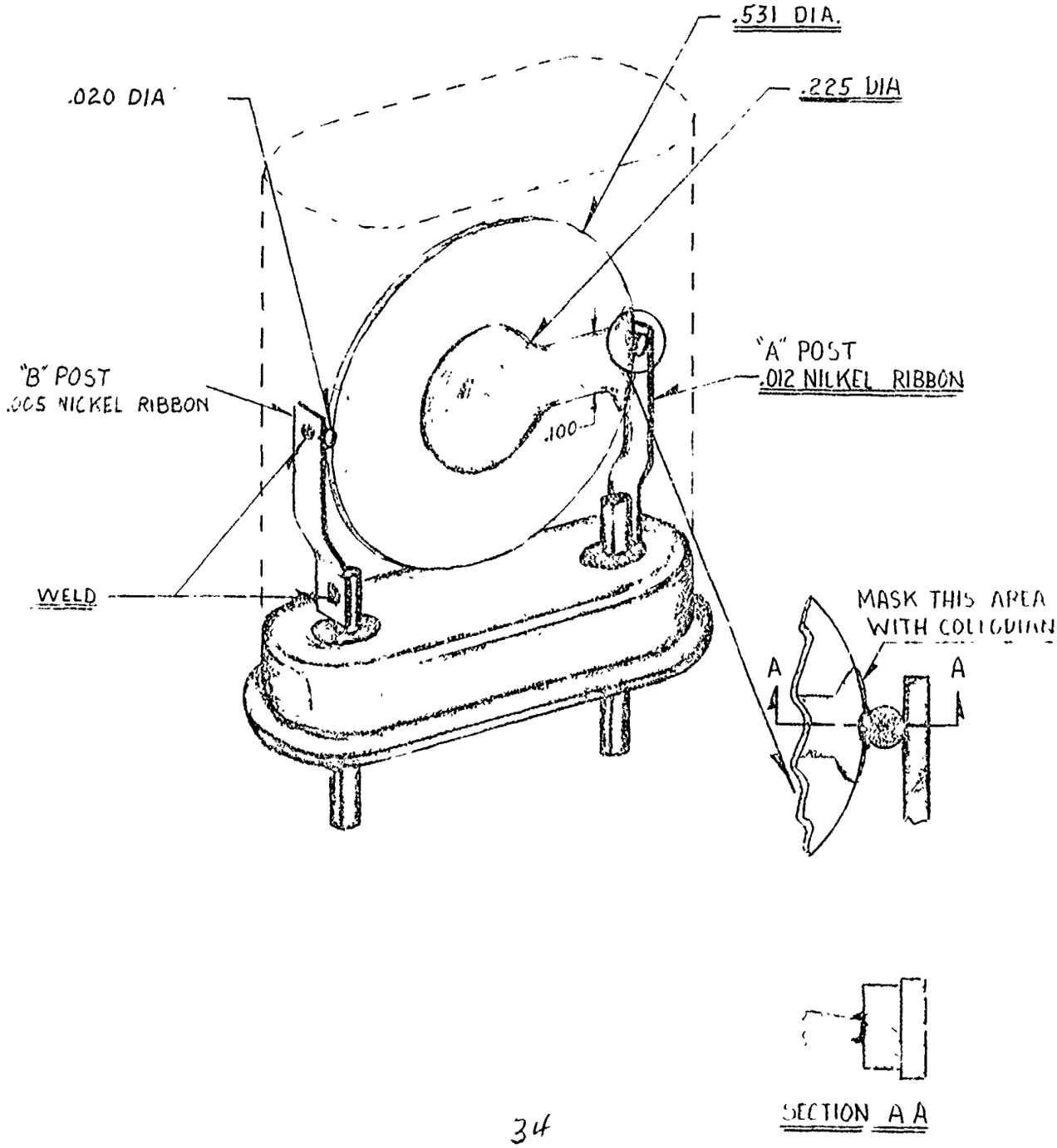
Published In
IEE TRANSACTIONS ON INSTRUMENTATION AND MEASUREMENT
Volume IM-17 Number 1
March 1968, pp 76-79'

CTS Knights, Inc.
Sandwich, Illinois

SIZE	CODE IDENT NO.	DWG NO.
A	75378	
REV.	AUTH.	SHEET

TSX COF-4207-0-3/68

5 MHz Fundamental 2 Point
Edge Mount, Nickel Decouplers

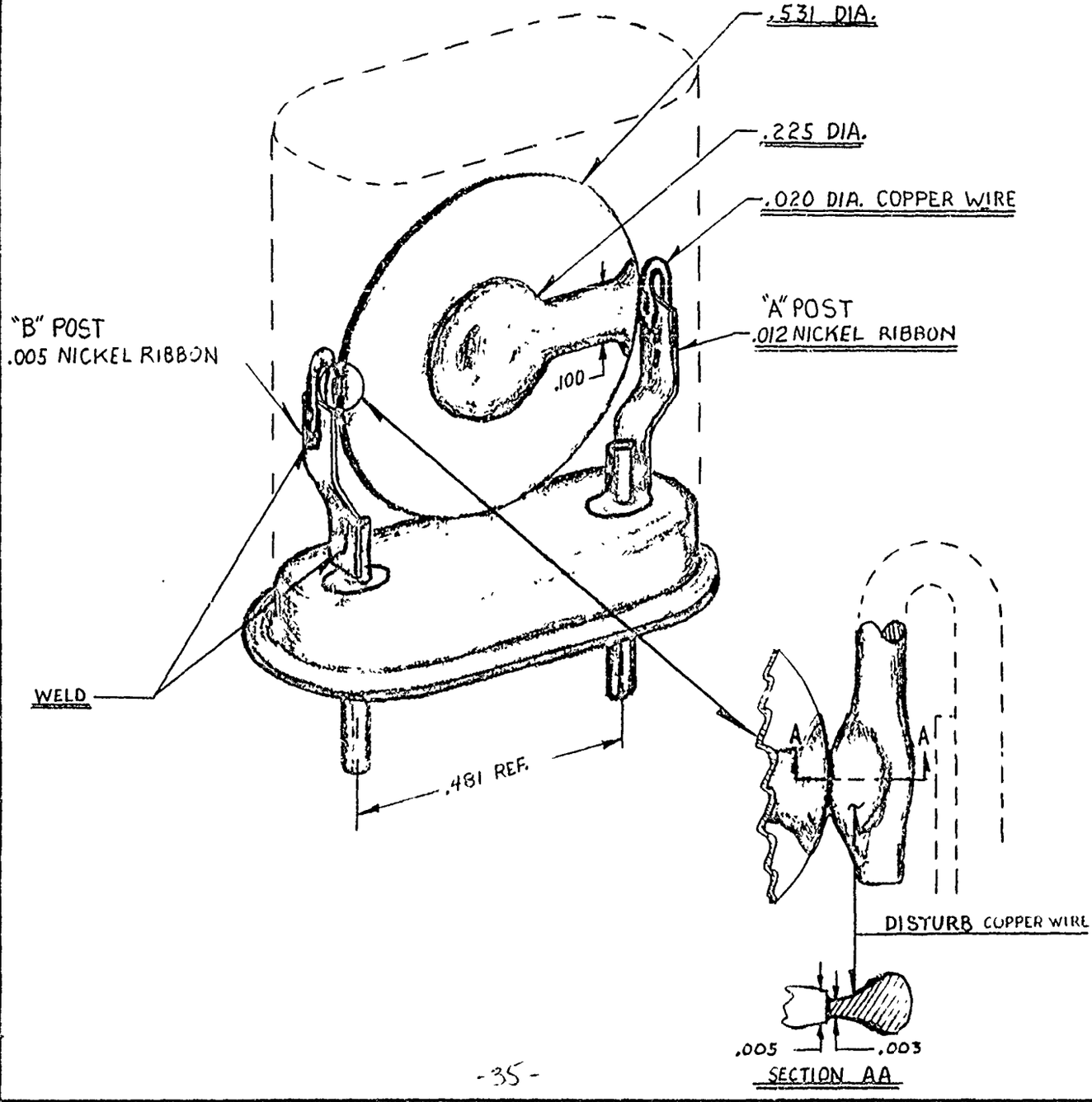


SECTION A A

CTS Knights, Inc. Sandwich, Illinois	SIZE A	CODE IDENT NO. 75378	DWG NO. EXHIBIT II Fig. 1
	REV	AUTH.	SHEET

CTSK 005-4207-O-3/68

5 MHz Fundamental, 2 Point
Edge Mount, Copper Decouplers

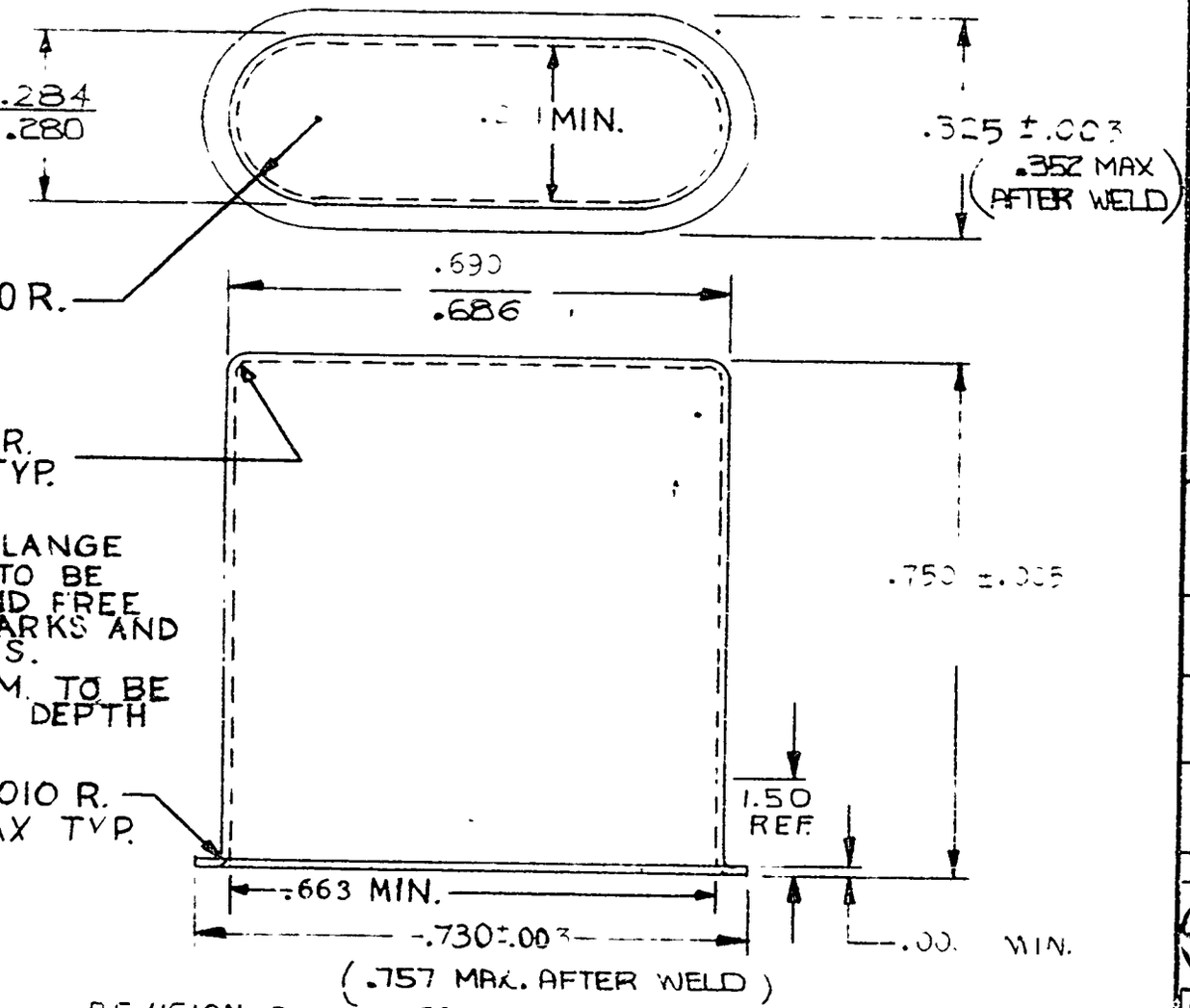


CTS Knights, Inc. Sandwich, Illinois	SIZE A	CODE IDENT NO. 75378	DWG NO. EXHIBIT II FIG. 2
	REV.	AUTH.	SHEET

005-4207-0-3/68

EXHIBIT II
CRYSTAL COVER
FIG 3
PAGE 36

TOLERANCE UNLESS OTHERWISE SPEC	PART NAME	PART NUMBER
ALLOWABLE VARIATION ON DIMENSION	COVER, CRYSTAL	341E-701-3
HOLE DIAMETER .003	MATERIAL OR SOURCE: GRADE A NICKEL #270 .010 THICK — GTI CORP. — TEMPER OR HARDNESS	USED ON: COLDWELL
WHEN ADDITIVE FINISH IS SPECIFIED TOLERANCE & LIMITS APPLY OVER FINISH	ADDITIVE FINISH SPEC OR COLOR	PROCESS IDENTIFYING PART NO'S
ANGULAR TOLERANCES SPEC AT DIMENSION	DRAWN/DATE: 10MAY67	CHECKED DATE: 2-22-68



NOTES:
1. BOTTOM FLANGE SURFACE TO BE SMOOTH AND FREE OF TOOL MARKS AND SCRATCHES.
2. INSIDE DIM. TO BE HELD FOR DEPTH OF .150".

ISSUE	RELEASE AND REVISIONS	AUTH
X1	WAS SK56705 .29 ±.002 WAS .285 .310 ±.002 WAS .310 .755 ±.008 WAS .748 .732 ADDED SOURCE E FINISH. 2-27-68 RELEASE FOR PRODUCTION	2820
A	.284 .280 WAS .282 .280 .325 ±.003 WAS .324 ±.002 .730 ±.00- WAS .729 ±.002 ADDED .261 MIN., .140 R .015 R MAX TYP. .664 MIN. .010 R. MAX. TYP NOTES 1 & 2.	3641
B	Change .261 to .260 also .664 to .663 July 18, 1969	3694
C	CHGD. FINISH TO MIL SPEC. 11/18/7[illegible]	4343
D	CHANGED HEIGHT DIM. & [illegible]	[illegible]
E	ELIMINATED PLATING ADDED SOURCE. CHGD. DIMENSIONS. 10/31/72	6008

SCALE

REVISION D SEE OBSOLETE FILE

DO NOT SCALE DRAWING / REVISIONS INDICATED BY CIRCLED LETTERS / ALL DIMENSIONS ARE [illegible]

THE JAMES KNIGHTS COMPANY
SANDWICH ILLINOIS
PRODUCTS

KBE 1851

MAKE SURE THIS IS THE LATEST ISSUE

TOLERANCE UNLESS OTHERWISE SPEC		
ALLOWABLE VARIATION ON DIMENSION	± .005	
HOLE DIAMETER	.003	

WHEN ADDITIVE FINISH IS SPECIFIED TOLERANCE & LIMITS APPLY OVER FINISH

ANGULAR TOLERANCES SPEC AT DIMENSION

PART NAME: CRYSTAL MOUNTING HEADER

MATERIAL OR SOURCE: ASTA. COMPONENT. INC. G.T.I. CORP. — TEMPER OR HARDNESS:

ADDITIVE FINISH SPEC OR COLOR: ELECTROLESS NICKEL PER MIL-C-25074 CLASS 1 .0001 TO .0002 INCHES THICK

PROCESS IDENTIFYING PART NO'S:

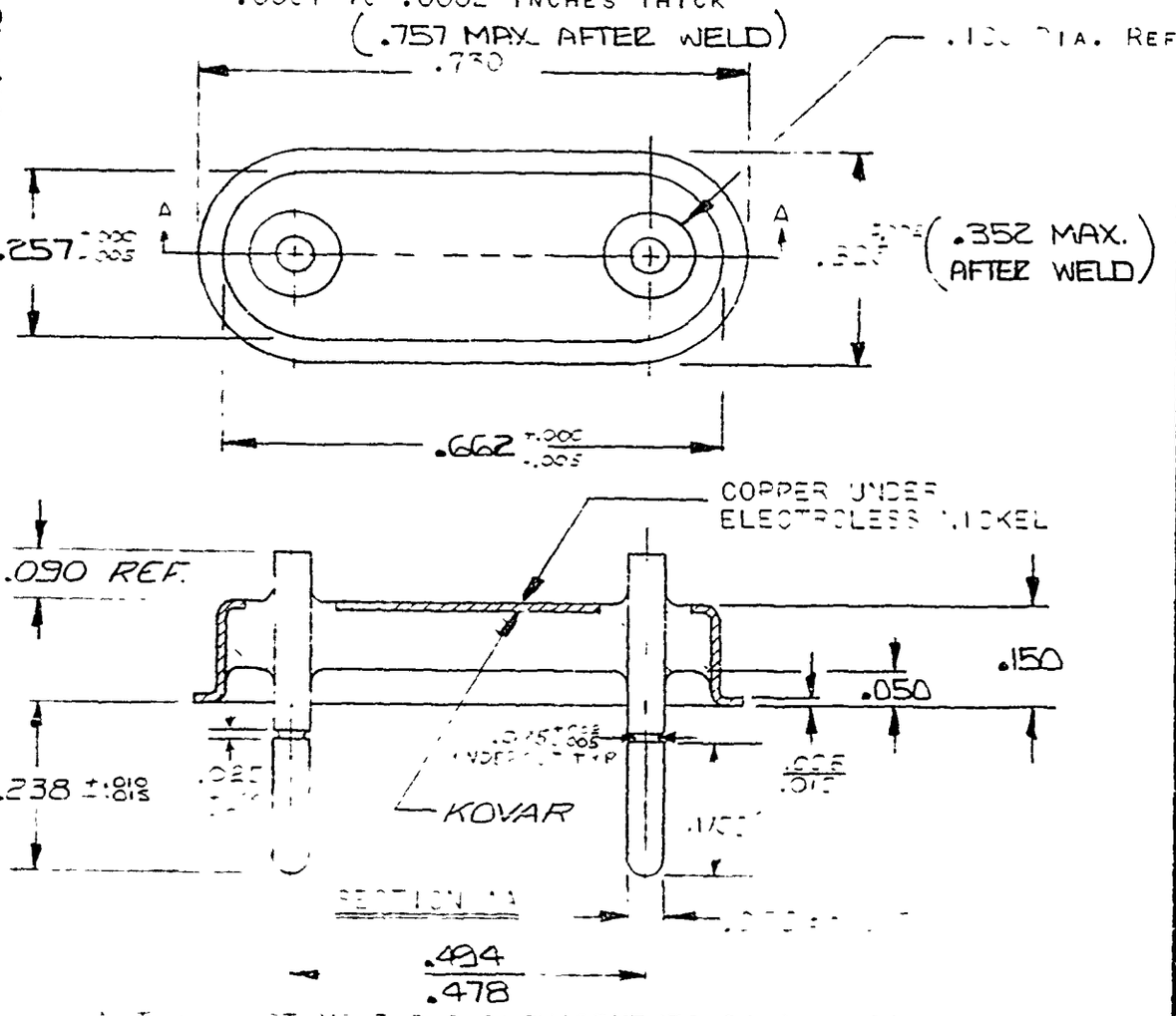
PART NUMBER: 23C-1702-0

USED ON: COLDWELD HC-36[illegible]

DRAWN/DATE	CHECKED/DATE	APPROVED/DATE
C. Gontzos 10MAY67	5-22-68	5-22-68

ISSUE	RELEASE AND REVISIONS	AUTH.
X1	WAS SKETCH [illegible]	
	.729 WAS .725 .324 WAS .320 ADDED .090 REF. [illegible] FINISH - SEE NOTE - .005 TO .002 WAS .005 RELEASED FOR PRODUCTION 5-27-68	2520
A	ADDED PLATING SPEC & 016-1045. 11/4/70	4880
B	CORRECTED DIMENSIONS PER MIL-[illegible] 10056/23 7/19/72	5433

EXHIBIT II FIG 4
CRYSTAL BASE
PAGE 37



NOTE: [illegible] REQUIREMENTS OF [illegible]

SCALE

DO NOT SCALE DRAWING. REVISIONS INDICATED BY CIRCLED LETTERS/ALL DIMENSIONS ARE IN INCHES

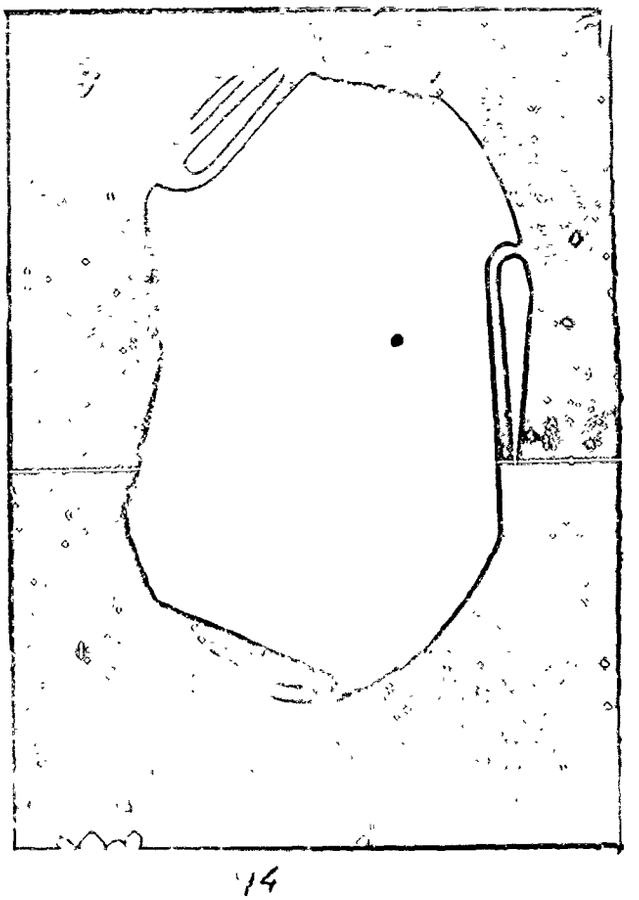
CTS KNIGHTS, INC.
FORMERLY

THE JAMES KNIGHTS COMPANY
SANDWICH ILLINOIS

PRODUCTS

E 1951

Exhibit III
Buschert Double Crystal
X-Ray Topographs

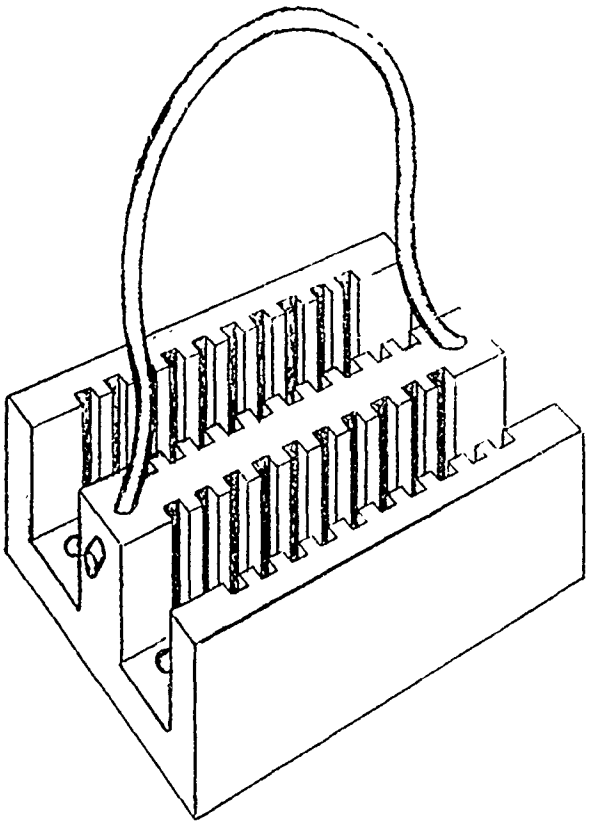


These are X-ray double crystal topographs made at Bell Laboratories (Allentown) in the summer of 1969. The crystals are (111) slices of silicon 1" in diameter and about .1" thick. The pronounced growth rings of the one picture (#77) shows the non-uniform doping in the bulk of this crystal. It was intentionally doped with phosphorous to about one part per million. The other picture (#94) shows very faint growth rings of a similar crystal doped to 10 parts per billion. This shows the extreme sensitivity to bulk strains (lattice parameter variations) of the method since these were highly polished syton finish which showed to be strain free since subsequent itching produced no changes. Surface scratches, mounting strains and surface diffusions were also highly visible in similar topographs if great care was not taken to avoid them.

The extreme sensitivity of the double crystal method to surface strains and the unimportance of sample thickness should make this double crystal method better than the Lang method for quartz.

Reference: Robert C. Buschert
Chairman, Physics Department
Goshen College
Goshen, Indiana

CLEANING JIG

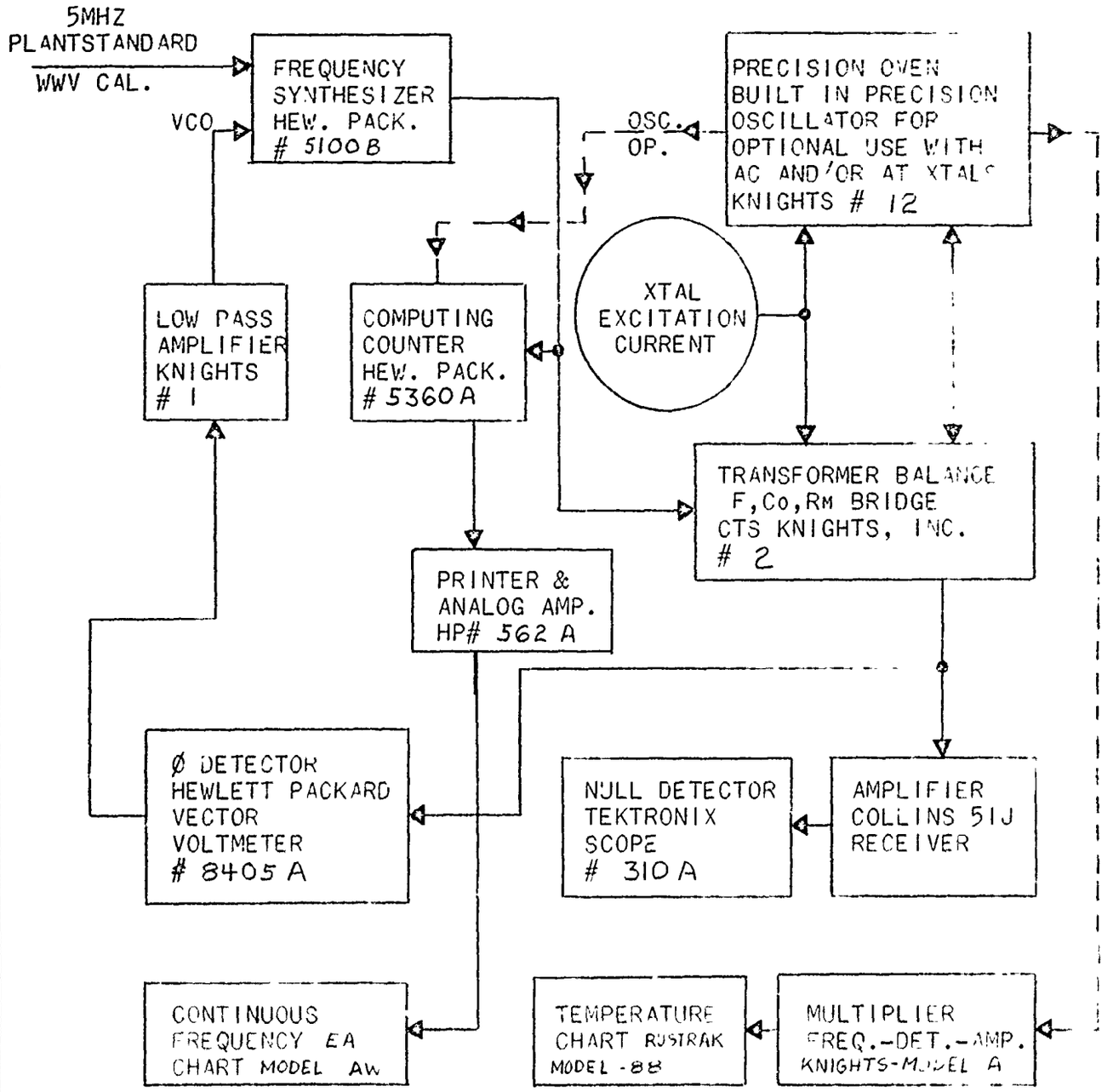


CTS Knights, Inc. Sandwich, Illinois	SIZE A	CODE IDENT NO. 75378	DWG NO EXHIBIT IV
	REV.	AUTH.	SHEET

005-4207-0-3/68

Exhibit V, Figure 1

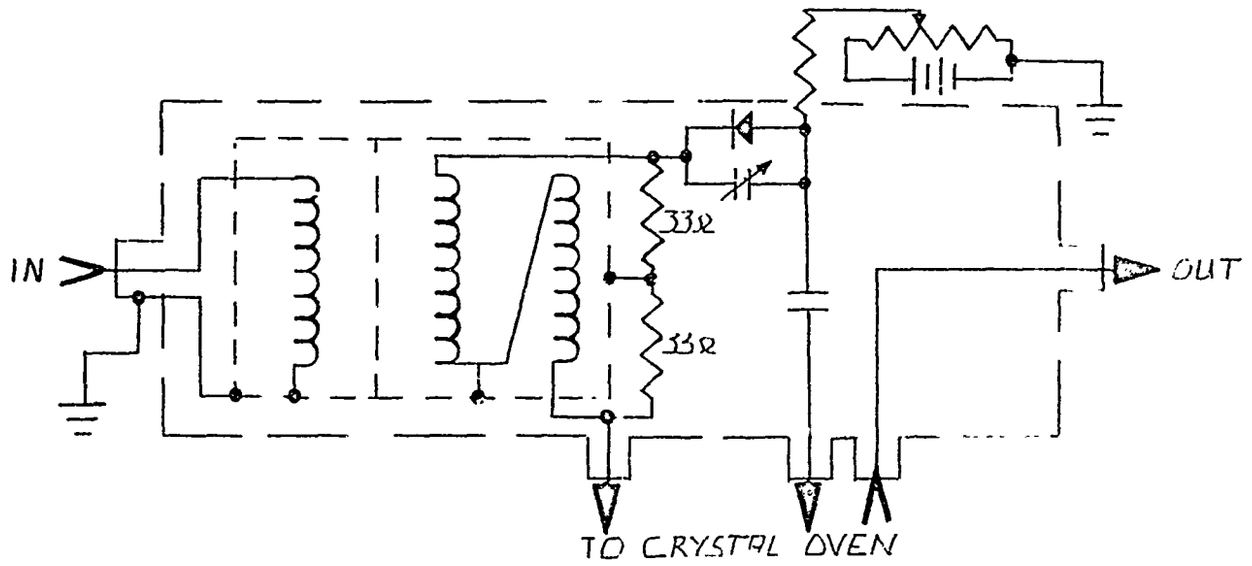
COMPLETED PRODUCT TEST STATION

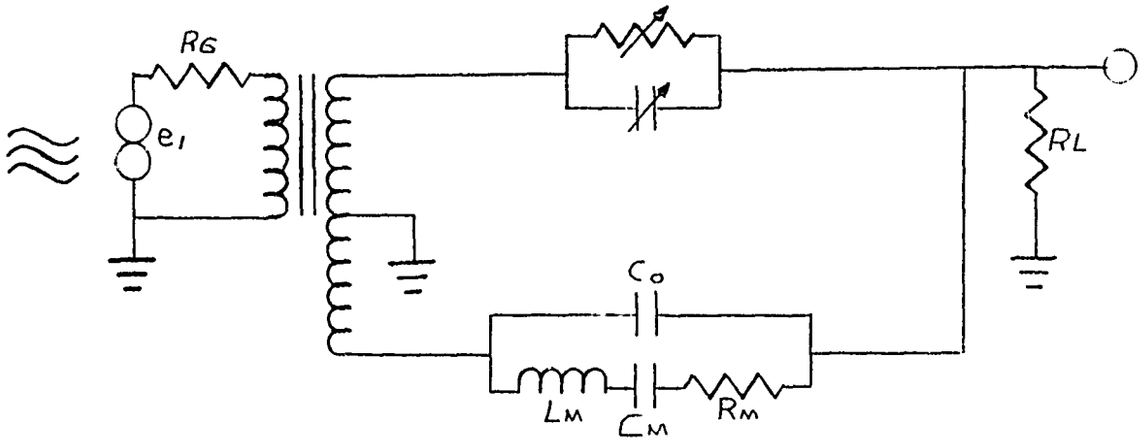


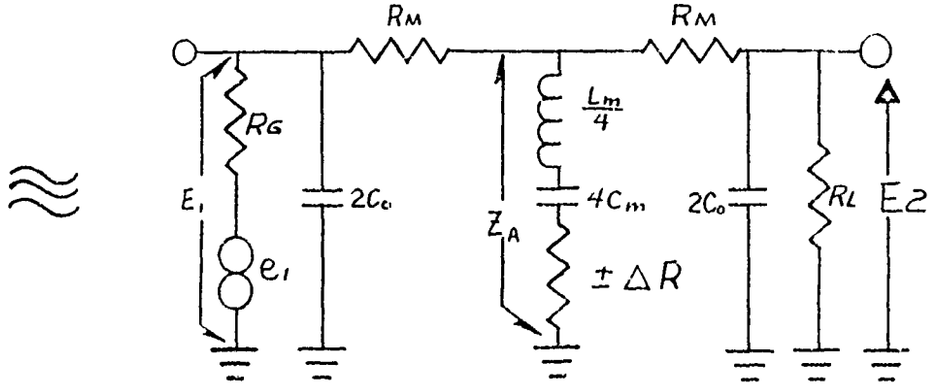
CTS Knights, Inc. Sandwich, Illinois	SIZE A	CODE IDENT NO. 75378	DWG NO. EXHIBIT V Fig. 1
	REV.	AUTH.	SHEET

Exhibit V, Figure 2

BALANCE TRANSFORMER TYPE BRIDGE







CTS Knights, Inc. Sandwich, Illinois	SIZE A	CODE IDENT NO. 75378	DWG NO. EXHIBIT V FIG. 2
	REV.	AUTH.	SHEET

The probes of the Vector Voltmeter sense at points A & B and indicate amplitude of voltage at Point B on the -70 db range 1000 Hz BW amplitude meter and degrees of phase difference between voltage sensed at points A and B. This information is filtered through a 1 Hz BW filter and indicated on the phase reading meter with a $\pm 6^{\circ}$ full scale maximum sensitivity.

The equivalent T circuit is that of a notch filter with the output E_2 attenuated in accordance with the following expression:-

$$\frac{E_2}{E_{2\ max}} = \frac{\Delta R + j\Delta X_a}{R_p + \Delta R + j\Delta X_a}$$

where $\Delta X_a = \pi f L_m \frac{\Delta f}{f_o} = \frac{X_{cm}}{2} \Delta f/f_b$ and $R_p = (P_G + P_{m/2})(R_L + R_{m/2})$

For crystals under consideration $R_p \cong 15\Omega$ & $X_{cm} \cong 2.x10^6$ Therefore with ΔX_a tuned out ($\Delta f = o$) and an amplitude null of 100 db (obtained by use of auxilliary attenuator in series with synthesizer ΔR is $R_p \times 10^{-5} \cong 15x10^{-5}\Omega$

To reduce the null by 3 db would increase R_p to $\cong 20x10^{-5}\Omega$

To generate $20x10^{-5}$ of reactance (ΔX_a) we have

$$\frac{X_{cm}}{2} \Delta f/f = 20x10^{-5}$$

$$\Delta f/f = (20x10^{-5})(1x10^{-6})$$

$$= 2x10^{-10}$$

At this frequency the phase difference between the current through the arm A and the voltage across it should be 45°, and accordingly a $\Delta f/f$ of $2x10^{-11}$ could be expected to produce a phase shift of 4.5°

Since Z_a is so small as compared to R_G and R_L which are 50Ω it can be seen that E will be in phase with I_a and E_2 in phase with E_a. The Vector Voltmeter therefore should exhibit a phase sensitivity of better than $1x10^{-11}$ per degree, except that random noise may reduce this as much as 10/1.

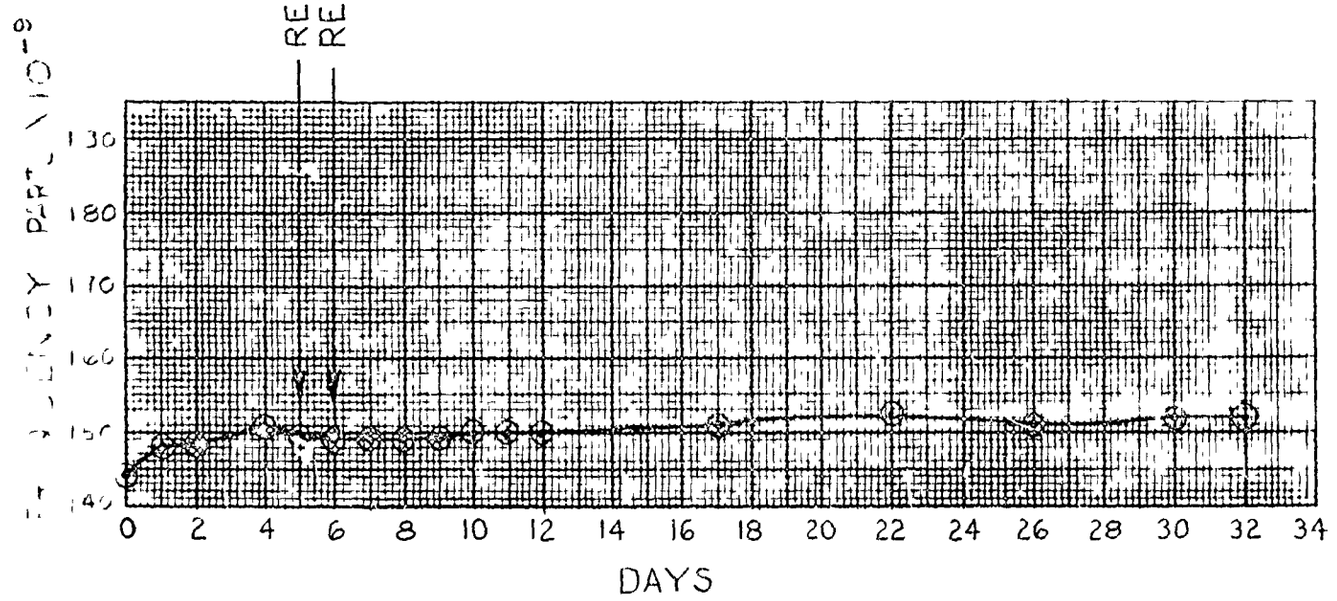
APPLICATION		REVISIONS			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED

Exhibit VI, Figure 1

AGING DATA
POSITION
SERIAL NO.
FREQUENCY
FIRST READING
LAST READING

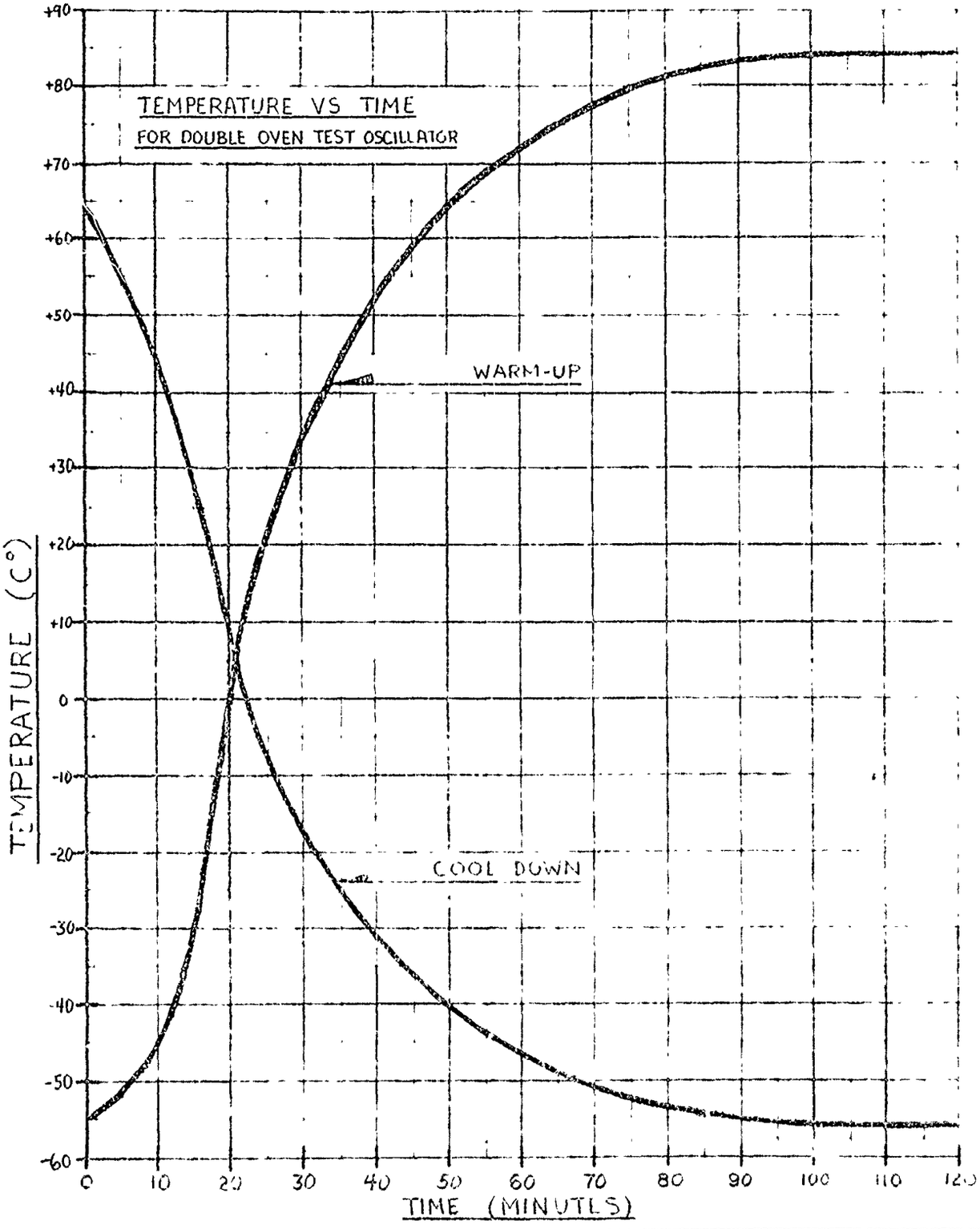
5MHz CRYSTAL, FUNDAMENTAL MODE,
5504A CEMENT BONDING

DESIGNED AND FABRICATED
IN ACCORDANCE WITH PROCESS
DEVELOPED ON CONTRACT DA-28-043-AMC-02183E



REV. STATUS OF SHEETS	REV. / SHEET		
ELEC. ENG. DATE	CONTRACT NO.	CTS Knights, Inc. Sandwich, Illinois	
MECH. ENG. DATE	DRAWN BY DATE		
Q.C. DATE	CHECKED BY DATE	DESCRIPTION: EXHIBIT VI FIG 1	
SALES DATE	DSGN. ACTIVITY APPD. DATE	SIZE: A	CODE IDENT NO: 75378 / DWG. NO.
	AUTH. DATE	SCALE	SHEET

EXHIBIT VI FIG. 2



CTS Knights, Inc.
Sandwich, Illinois

SIZE	CODE IDENT NO.	DWG NO
A	75378	
REV.	AUTH	SHEET

CTSK 005-4207-0-3/68

EXHIBIT VII

Table 1

Upper Turning Point Temperature	
Nickel Electrobond	5504A Cement
70.2 C	71.5 C
69.6	74.3
66.7	69.0
68.5	69.7
68.4	67.5
	68.1
	67.2

The quantity is too few for rigorous statistical analysis but the following observations are believed to be significant.

The average of the nickel group is 68.4

The average of the cement group is 70.2 - very close to the 70 our experience curve (gained with use of cements of one kind or another) predicts.

The dispersion of the nickel bonded group is ± 1.7 C A 0.6' spread in angle from our calculations should have resulted in a ± 1 C spread. Two and maybe 3 out of the 5 would meet the ± 1 ¢C variation in T.P. required. To adjust the nominal from 68.4 C to 70 C would require approximately a 0.6' increase in angle.

Only two out of the seven blanks of the cemented group would fall within the ± 1 C T.P. requirement even though the average is close to nominal.

Table 2

X-Ray Readings vs. Mounting Stress

A-1		A-2		A-3	
30	25.25'	30	25.0'	30	24.6'
30	24,70'	30	24.4'	30	24.7'
30	24.30'			30	24.7'

Exhibit VIII

Topographical and Source
Image Distortion X-Ray Photos

The 21.0 reflection was used in every case. The 21.0 direction is along the X-axis (piezoelectric technology convention) so that the diffracting planes are both perpendicular to the X-axis and perpendicular to the crystal surface (for the AT cut). This 21.0 direction is the direction of motion for pure thickness-shear modes in AT cut quartz crystals. This reflection is most sensitive to lattice defects with burgers vector (or fault vectors) in the 21.0 direction (i.e., normal to the diffracting planes). Defects with fault vectors in the Z-Z' or Y-Y' directions (i.e., parallel to the diffracting planes) would not be imaged. Dauphine twins or twin lamellae would be observed. Localized lattice defects are shown by variations (usually increases) of diffracted intensity.

Variations in the "strightness" of the SID lines provides information about the larger scale lattice deformation. For the conditions under which the present patterns were made, all deviations of the SID lines can be attributed to tilts, i.e, bending of the crystal. The patterns were sensitive to the component of tilt (rotation) about an axis parallel to the undistorted SID lines (in our case about the Z-Z axis). On the prints supplied, a deviation of the SID lines of 2.4 mm (i.e., a displacement of the line in a direction perpendicular to itself) corresponds to a relative tilt of 2×10^{-4} radians.

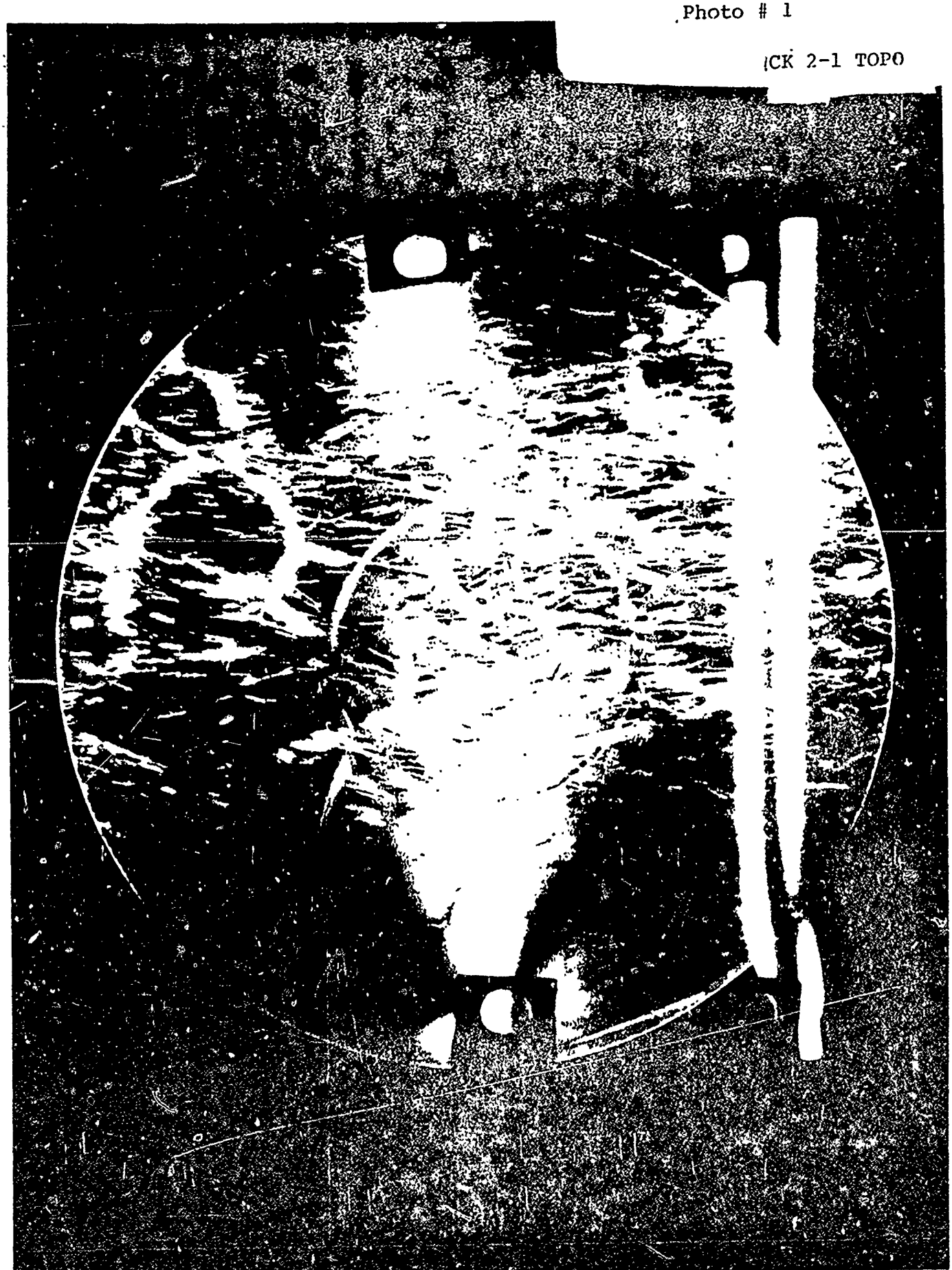
A preliminary topograph of Unit 2-1 is included to give some idea of how much better the defects are shown by the topographic technique. The 21.0 reflection was used. (Figure CK 2-6)

Reference: Photos were taken at Georgia Tech of blanks submitted by CTS Knights, Inc.

Exhibit VIII

Photo # 1

CK 2-1 TOPO



CK2-1 TOPO

Exhibit VIII

Photo # 2

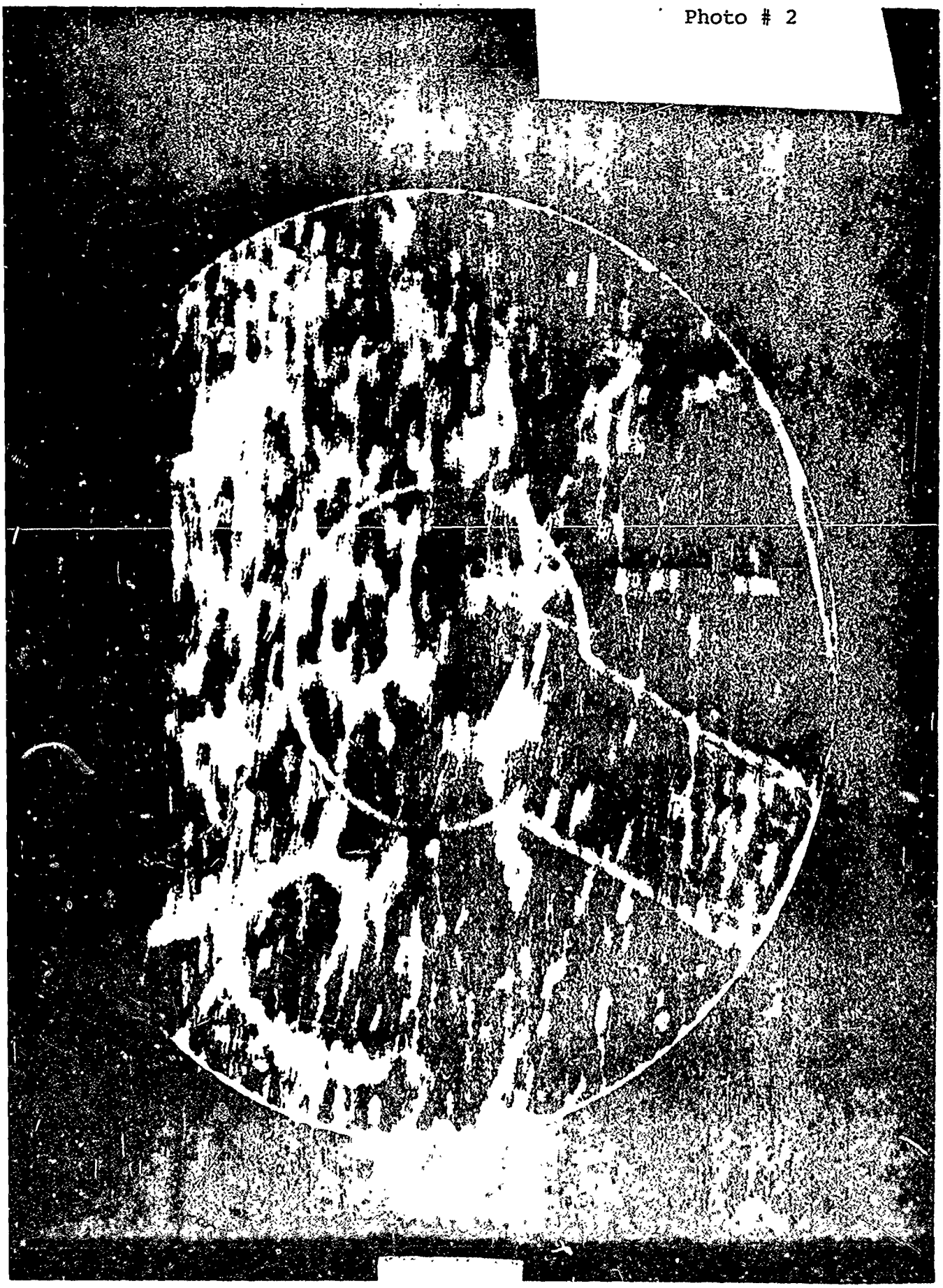


Exhibit VIII

Photo # 3

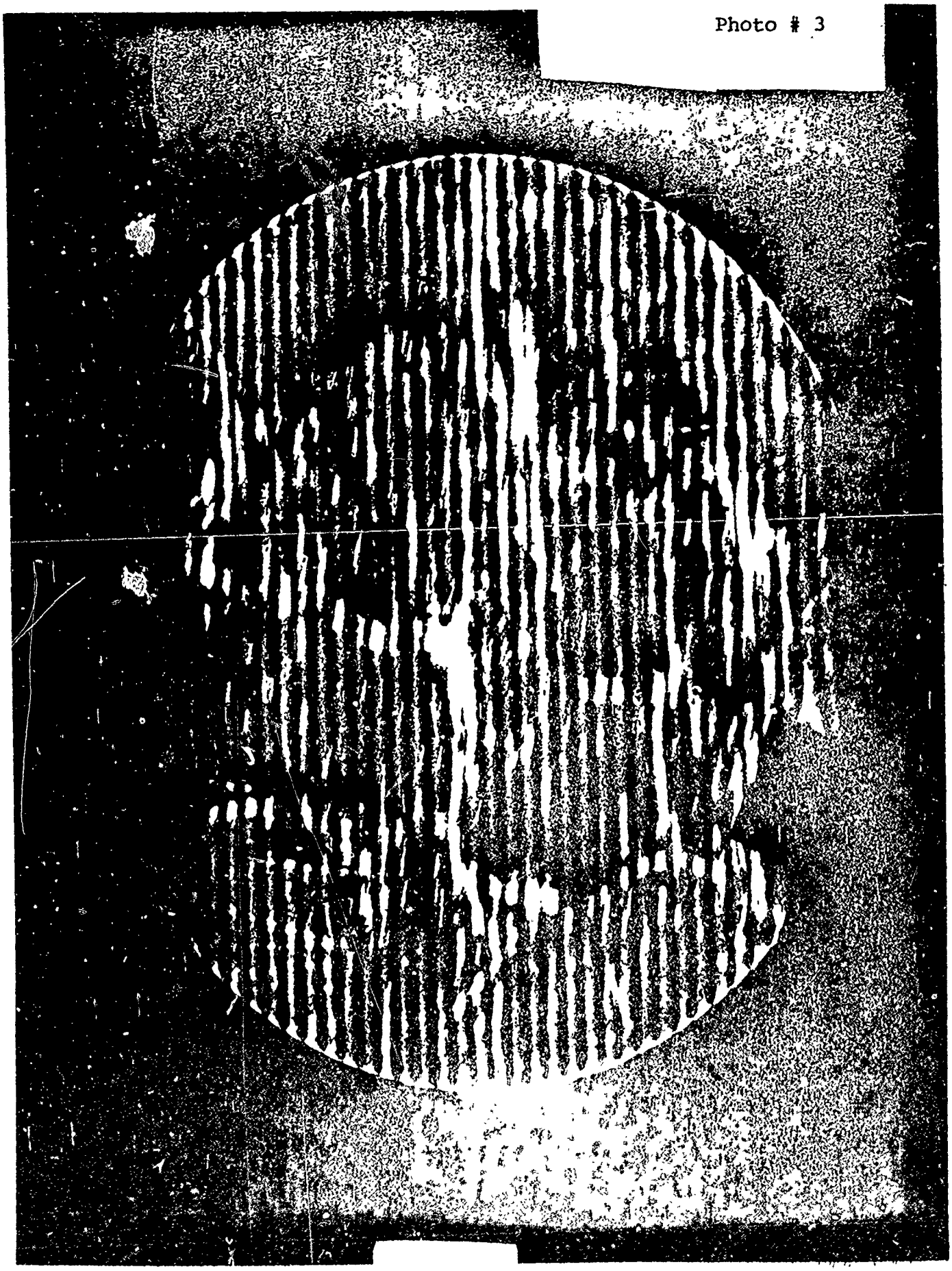


Exhibit VIII

Photo # 4

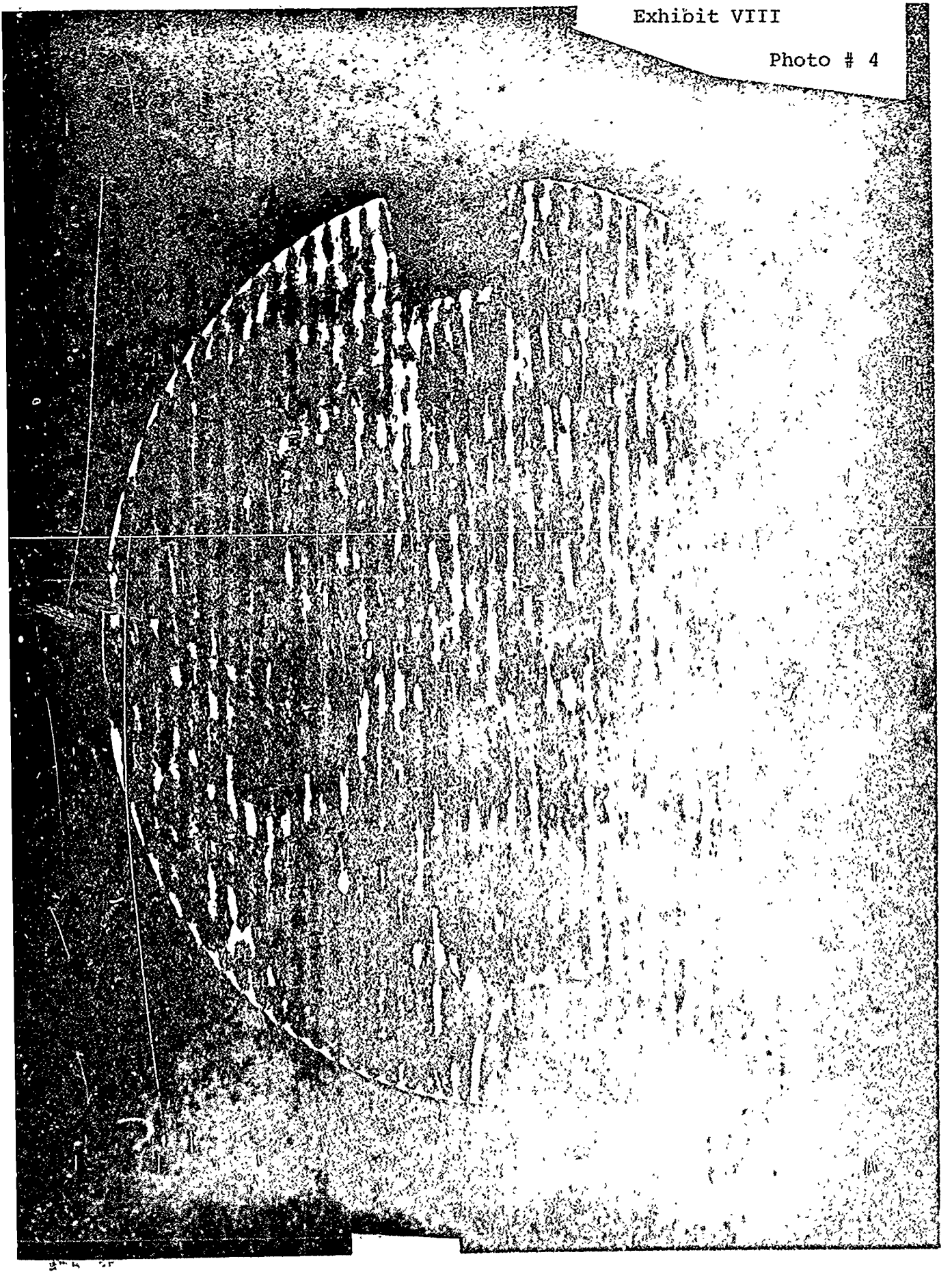


Exhibit VIII

Photo # 5

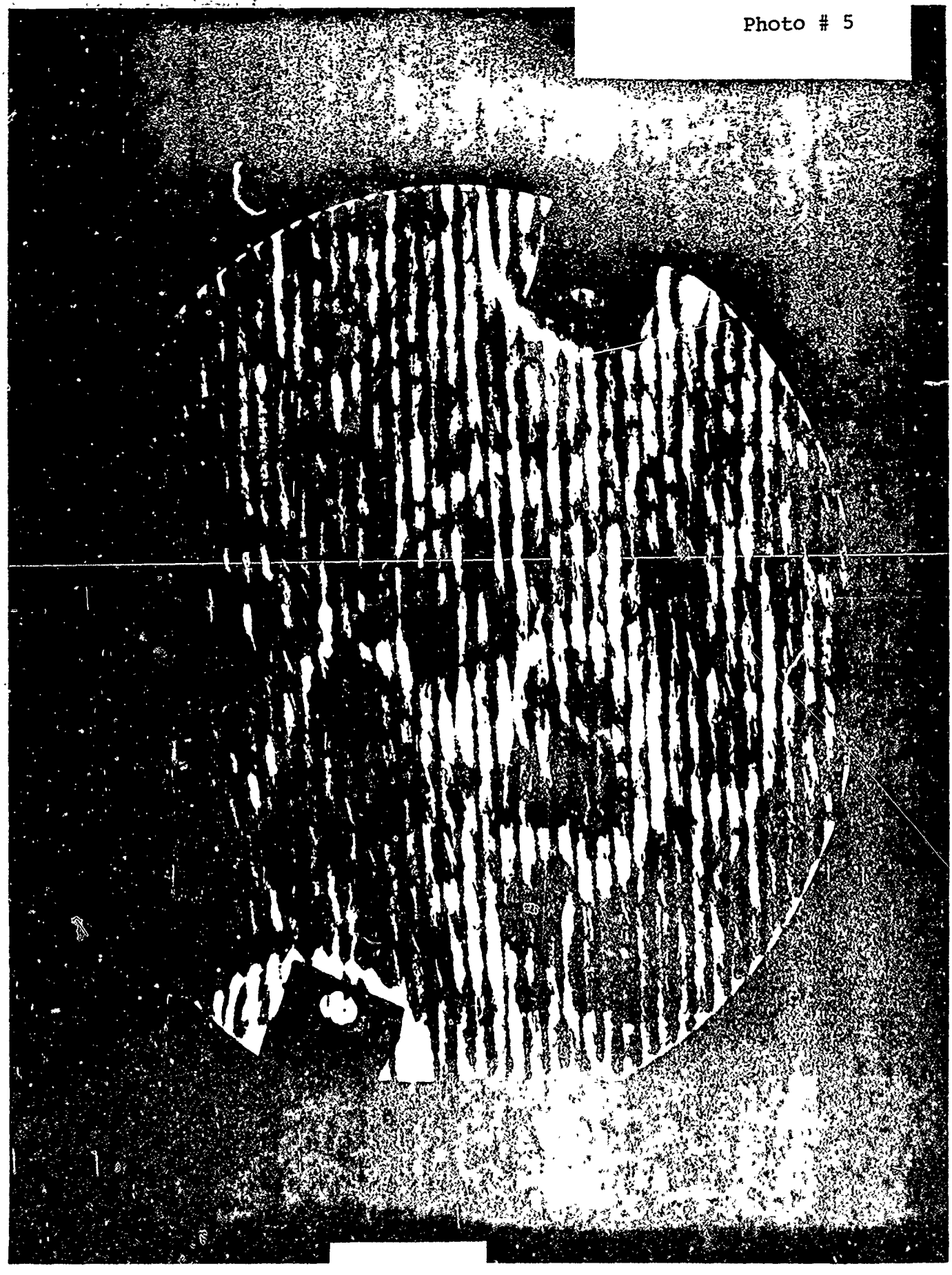
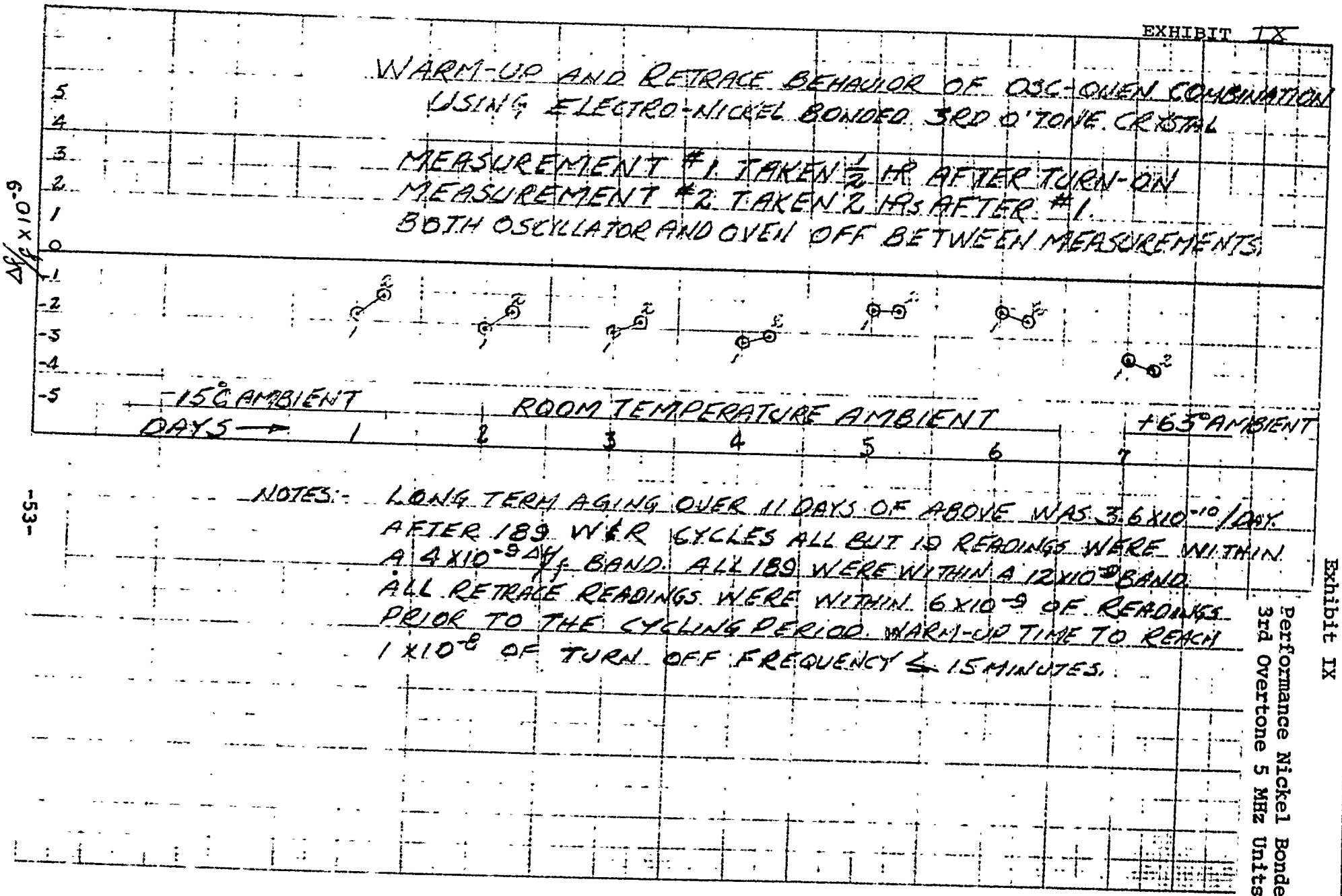


EXHIBIT IX

WARM-UP AND RETRACE BEHAVIOR OF OSC-OVEN COMBINATION
USING ELECTRO-NICKEL BONDED 3RD O'TONE CRYSTAL

MEASUREMENT #1 TAKEN ½ HR AFTER TURN-ON
MEASUREMENT #2 TAKEN 2 HRS AFTER #1.
BOTH OSCILLATOR AND OVEN OFF BETWEEN MEASUREMENTS.



NOTES:- LONG TERM AGING OVER 11 DAYS OF ABOVE WAS 3.6×10^{-10}/DAY.
AFTER 189 WARM CYCLES ALL BUT 10 READINGS WERE WITHIN
A 4×10^{-9} Δf/f BAND. ALL 189 WERE WITHIN A 12×10^{-9} BAND.
ALL RETRACE READINGS WERE WITHIN 6×10^{-9} OF READINGS
PRIOR TO THE CYCLING PERIOD. WARM-UP TIME TO REACH
1×10^{-8} OF TURN OFF FREQUENCY ≤ 15 MINUTES.

Performance Nickel Bonded
3rd Overtone 5 MHz Units

Exhibit IX

Exhibit X

Crystals in the Interim Stage

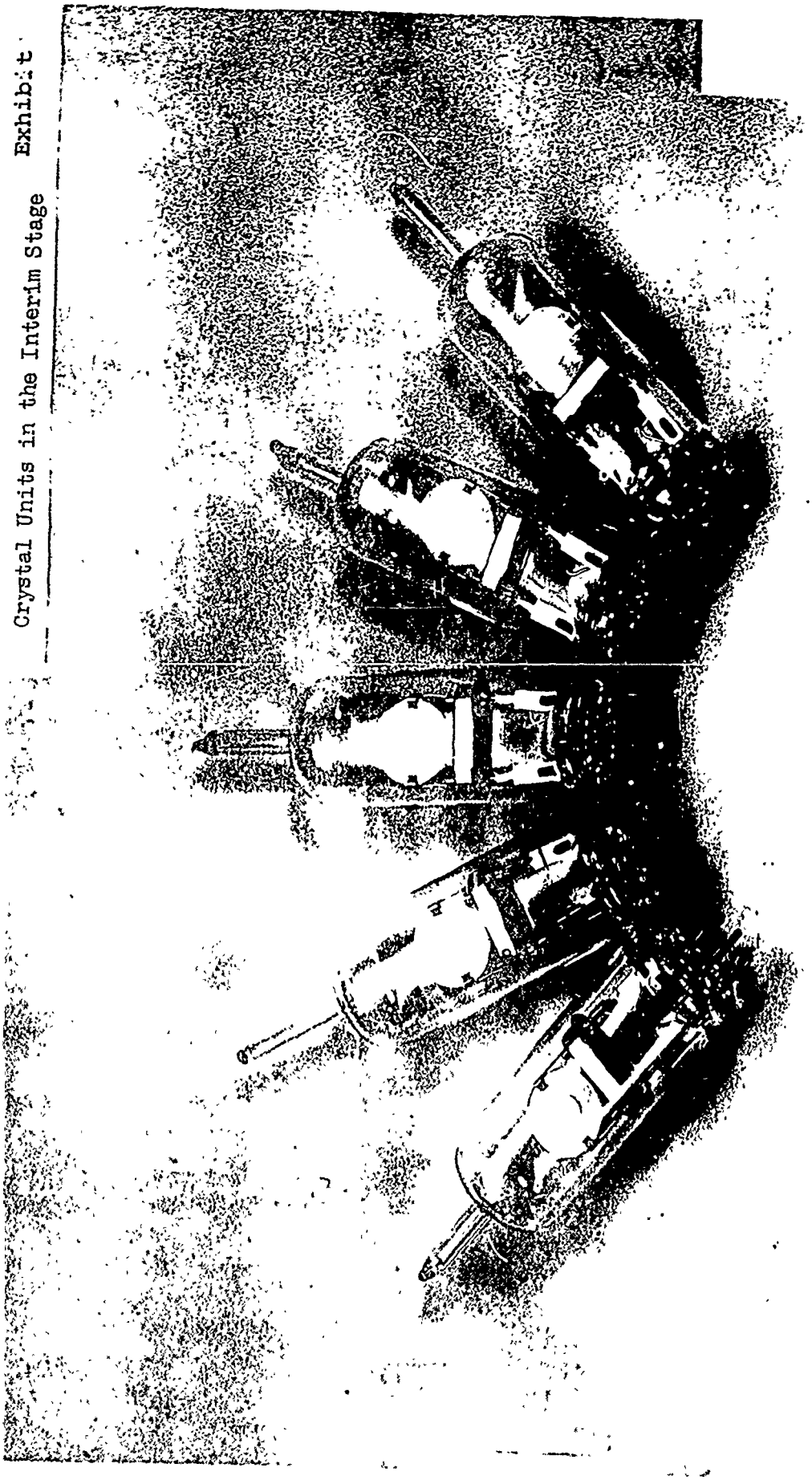


Exhibit t

Crystal Units in the Interim Stage

Exhibit XI

High Temperature High Vacuum Glass Enclosure Processing Machine

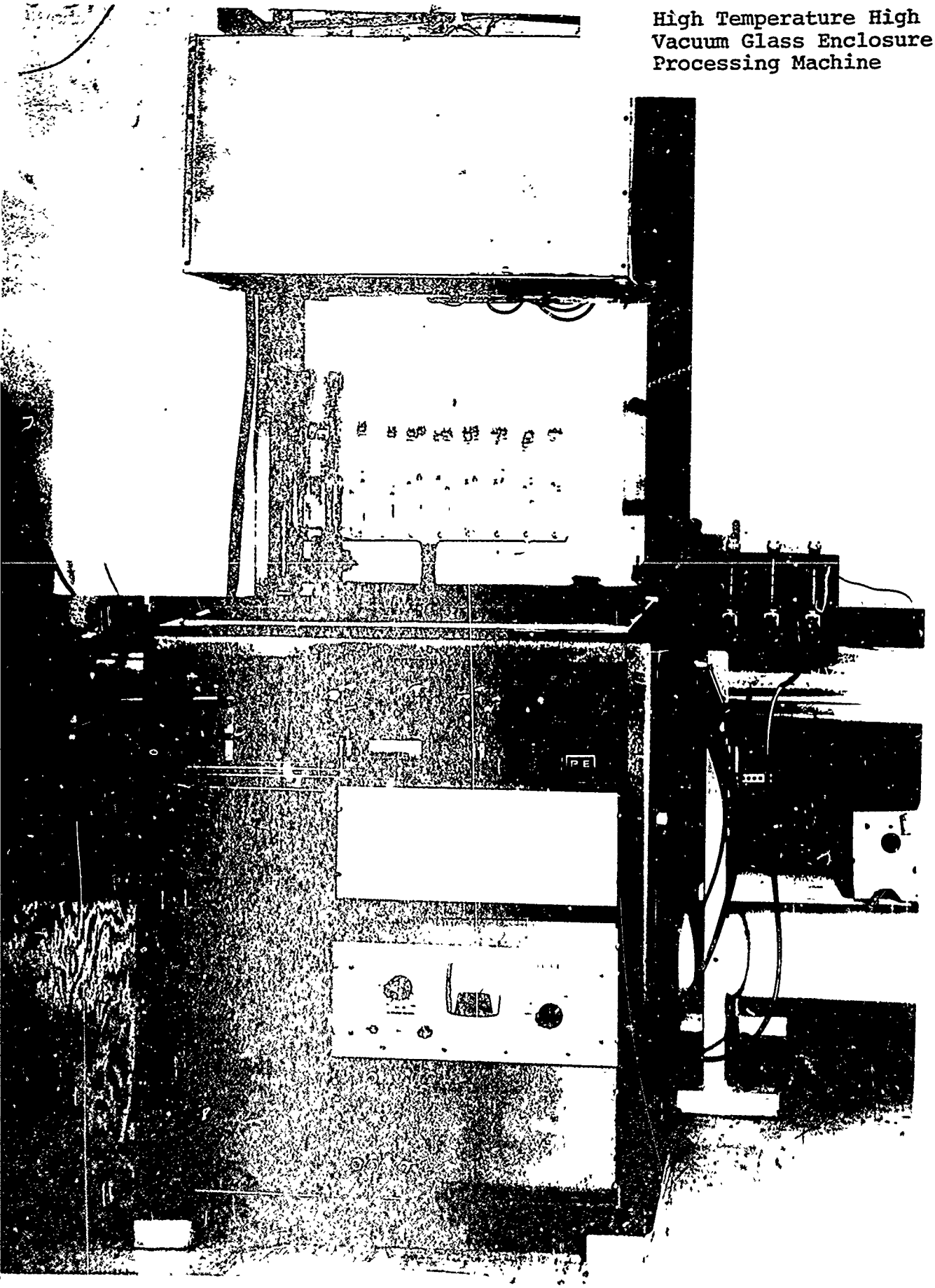
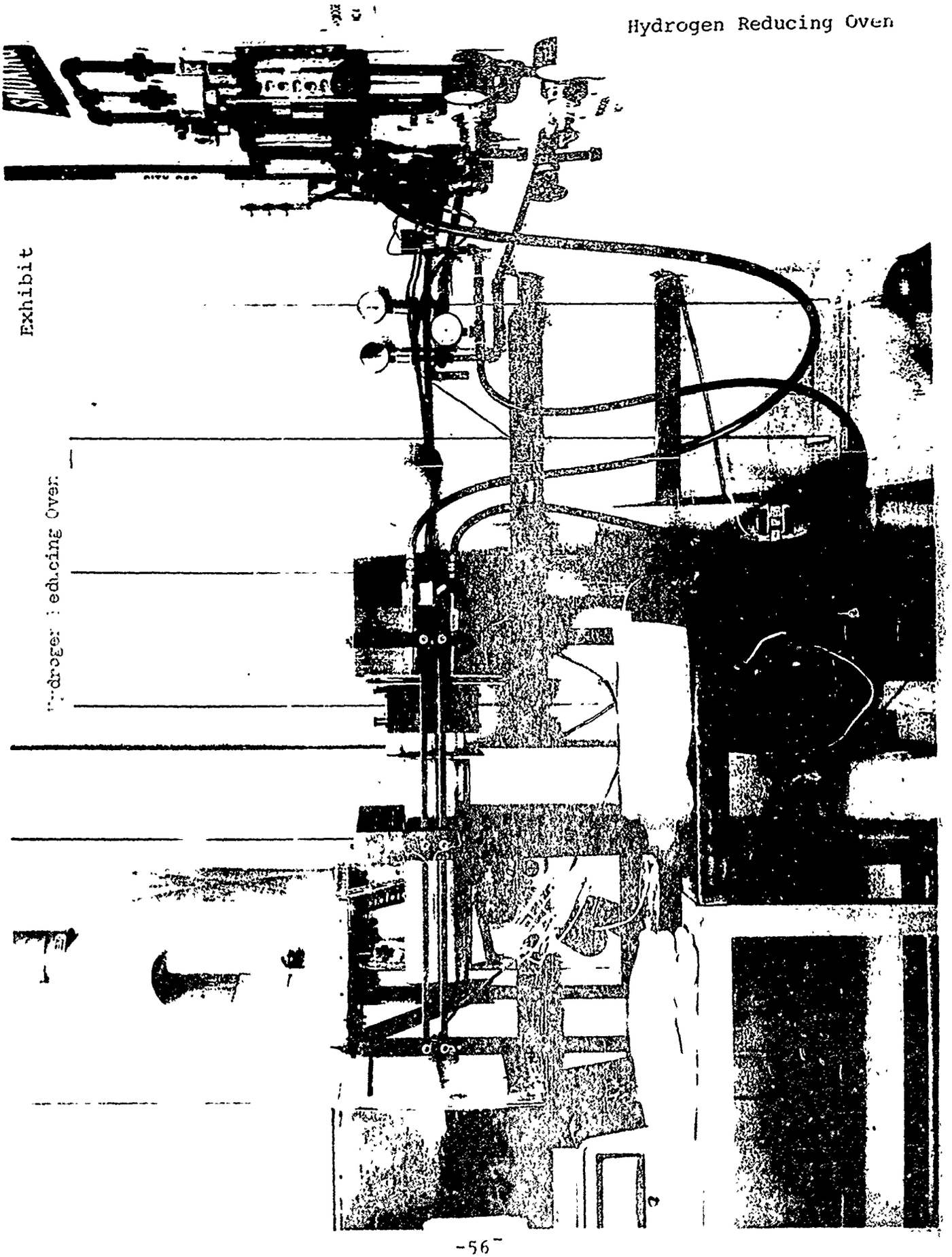


Exhibit XII

Hydrogen Reducing Oven



Exhibit

Hydrogen Reducing Oven

Exhibit XIII.

Single Shot High Temperature
Vacuum Plater

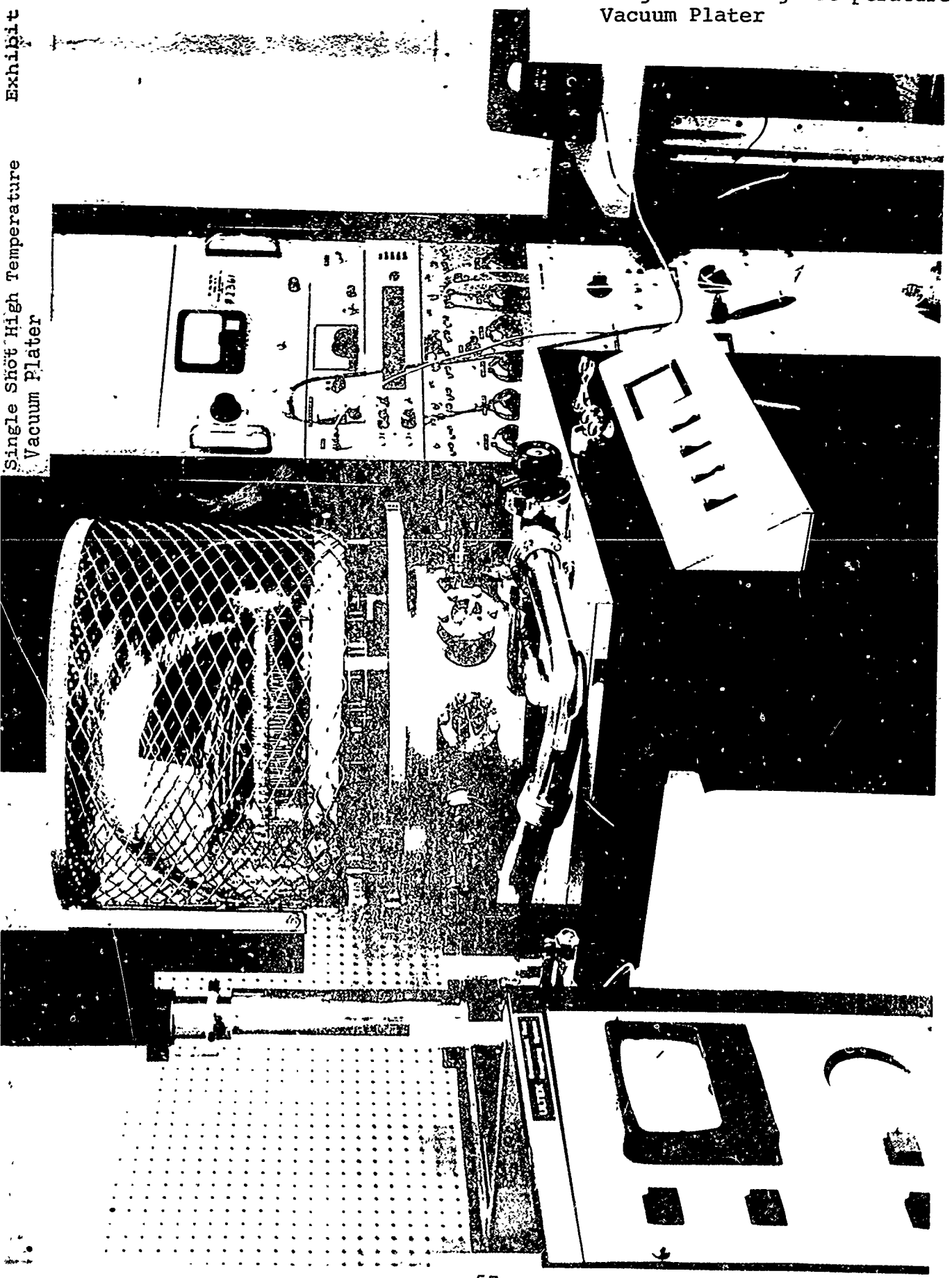


Exhibit XIII.

Single Shot High Temperature
Vacuum Plater