FINAL FREQUENCY ADJUSTMENT OF QUARTZ OSCILLATOR-PLATES

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Contents

Abstract

The art of finishing machine lapped quartz oscillator-plates to final frequency and activity specifications is discussed. Two general methods have been practiced: the abrasive lapping technique, now abandoned because of undesirable ageing phenomena caused in the plate, and the etching-to-frequency method. The *x*-ray irradiation technique has a special field of application in making highly precise frequency adjustments and in salvaging over-frequency plates. The cause and cure of frequency and activity ageing in quartz plates is discussed, and methods of adjusting the frequency of metal plated wire-suspension type plates are described.

Introduction

The machine lapping of AT and BT quartz oscillator-plates is usually stopped somewhat over the final desired thickness. The final finishing operation as ordinarily applied involves a process of lapping or etching the individual crystals by hand, with intermittent electronic measurement of the accompanying change in frequency, until the final desired frequency is reached. Control over the thickness of the crystal cannot be had at this stage by mechanical measurement since the dimensions involved are of the order of millionths of an inch. At the end of the finishing operation the crystal is cleaned and assembled in its permanent holder. The unit is now tested over a specified range of temperature, say from −55° to +90° C., to see if the frequency and activity remain within prescribed tolerance values, and then passes through various mechanical tests and inspections before it is submitted to Government inspectors for final acceptance.

Finishing by the Abrasion Method

In this method the crystals are first lapped by machine until they are within roughly 0.04 to 0.4% of the final desired frequency. The plates are

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then lapped individually by hand with fine abrasive on a glass plate. The crystal frequency is measured from time to time during the operation, usually in a comparison oscillator against a crystal standard of the desired frequency, and the duration of the hand lapping is gauged accordingly. The crystal should be measured in the particular pair of electrodes

FIG. 1. Final abrasive finishing position, showing glass lap (held in a pie dish) with crystal and abrasive, comparison oscillator with standard crystal, polariscope (left) for determining X and Z′ directions, sink with soap solution and tooth brush for cleaning, air gun for drying, and lead dish of etching solution with plastic holding tongs for acid wash.

in which it is to be permanently mounted. It is highly desirable to use a relatively fine emery abrasive, preferably 303½, or a fine garnet finishing powder in the final lapping. The operator lightly holds the plate on two opposite corners by the finger tips. The glass lap plate is then circled with a figure-eight or epicyclic motion and the fingers are then transferred to the opposite corners. This process is repeated on both sides of the crystal. By suitably holding the crystal a skilled operator can control convexity or wedging in the plate. Generally speaking, it is desirable to finish high frequency BT plates so that they are slightly convex, the centers being higher by a few hundred thousandths of an inch, since this condition is found to promote crystal activity. Very flat plates or wedged plates usually have relatively low activity and concavity usually proves fatal. The contour can be examined by wringing down on an optical flat and observing the system of interference fringes or by sensitive surface gauges such as the Pratt and Whitney Electrolimit gauge. It is sometimes found convenient to lap the crystal by wringing down on a small optically flat glass dop which is then manipulated by hand over the lapping plate. Groups of crystals can be handled simultaneously in this way by wring-

FIG. 2. View of final finishing department in a crystal plant.

ing or cementing down on a large optical flat. Needless to say, the flatness of the glass lapping plate itself must be carefully maintained.

In the finishing process attention must also be paid to the so-called activity of the crystal. The activity is primarily a function of the edge dimensions of the plate in a given electric circuit and holder, but also is influenced by the contour, the fineness of abrasive finish, cleanliness, the presence of cracks and chips on the edges, twinning, inclusions and other factors. In so-called predimensioned AT and BT plates the edge dimensions are deliberately preselected so as to give minimum coupling between the fundamental high frequency shear mode vibration and high harmonics of the Y flexural mode. Such crystals have inherent high activity and are relatively free from spurious frequencies and from activity dips over a range of temperature. Accurate control of dimensions, usually to 0.001 inch in low frequency crystals, and of cutting angle, with both the ZZ′ and XX′ angles held to at least ±15 minutes of arc, is essential. It is general practice, however, especially in the higher frequencies, to cut all crystals during manufacture to some abritrary set of dimensions. This is done because the ideal dimensions for a particular frequency usually are not known or conveniently determined, and because the dimensional and angular control involved is not easily achieved on a mass production basis. When arbitrarily dimensioned crystals are hand finished it is often necessary to bevel or grind down the edges of the plate in order to bring up activity. This changes the edge dimensions to a more favorable ratio.[1] The edging operation is variously done by lapping with abrasive on glass, or on a carborundum stone, fine emery paper, crocus cloth or a diamond wheel. Rotating hollow cylinders lined with diamond impregnated bakelite also are useful. Grinding the X edge of the plate is advantageous since this dimension more directly controls the interfering flexural mode. Unless the edge dimensioning of the plates preliminary to finishing is carefully done, a slight beveling and rounding of the sides and corners of the plate is desirable in order to remove roughness and chips. Rough edges and sharp corners are objectionable because of the danger of small particles of quartz becoming dislodged and wedging between the plate and electrodes.

After the plate has been brought to specifications it is scrubbed with a toothbrush and soap, thoroughly rinsed and dried, rechecked for frequency and activity and then mounted permanently in its holder. The holder and electrodes must also be clean. Drying should be done with a

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[1] Temperature test rejects in randomly dimensioned plates usually run between 15 and 40 per cent. The rejected crystals are disassembled and run through the finishing operation again, where the edge dimensions are changed to a more favorable ratio. Temperature test reject rates in predimensioned crystals ordinarily are only a few per cent.

jet of clean dry compressed air. Rubbing with a towel is objectionable because the surface of the plate becomes loaded with cellulose and the plate frequency is decreased thereby. Towelled plates when baked at *ca.* 500° C. gain exactly the same amount of frequency lost by the towelling operation, presumably due to burning off of the organic matter. Slight upward or downward changes in frequency can be effected by changing to electrodes with a higher or lower air gap, respectively, and a further downward change in frequency can be effected by the x-ray method described beyond. Plates can be given a crude partial temperature test during the finishing operation by heating them during measurement over an alcohol burner or electric stove or by cooling with a piece of dry ice. Activity dips recognized in this way can be removed by edge grinding, the changes in the plate dimension moving the dip to higher temperatures until it goes beyond the upper temperature limit.

Various mechanical lapping contrivances have been devised to speed up the initial stages of the finishing operation. The Sipp-Eastwood or Seco lap comprises two rotating lapping discs with a thin perforated workholder containing a single quartz plate. The workholder, ordinarily made of vinylite plastic or zinc, is held in position by a metal post and has a rotatory motion imparted by the lapping discs. The Empire Electronics Co. lap is a small scale version of the drill press type of mechanical lap. These laps are described in an accompanying paper.[2]

Electrodes and Springs. Most of the high frequency crystals currently in mass production, notably the CR-1 and FT-243, are clamped between stainless steel electrodes about 0.050 to 0.060 inches thick. The corners of the electrodes are raised somewhat, giving an air gap which usually ranges between 0.0004 and 0.0008 inches. Maximum activity is obtained in the lower gaps and the plate will ultimately refuse to oscillate as the air gap is increased. Both the lands and the air gap areas must be flat and parallel to at least within 0.0001 inches. This is necessary in order that the crystal withstand drop and vibration tests and to avoid certain undesirable modes of vibration which result in activity and frequency failures in the temperature testing. The electrodes are held in place by a spring exerting a force of about 5 to 8 pounds. Excessive spring pressure may damp out undesirable subsidiary modes of vibration but tends to reduce activity, while low spring pressure may result in failure in the drop and vibration tests.

Care should be taken in low frequency AT clamped plates to avoid particular air gap dimensions. Acoustic waves generated by the oscillator-plate are reflected back from the electrode and at certain gaps resonance

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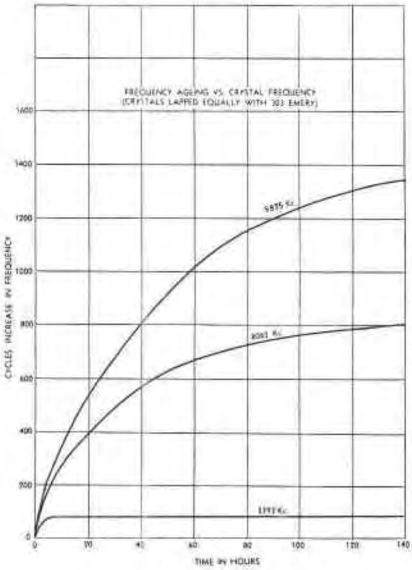
[2] Parrish, W.: Machine Lapping of Quartz Oscillator-Plates, *Am. Mineral.* this issue.

will occur resulting in damping and diminished activity of the crystal. The air gap dimensions to be avoided are given by

$$l = \frac{pv}{2f}$$

where l is the air gap in inches, v the velocity of acoustic waves in air at room temperature and pressure (=1083 ft/sec.), f the plate frequency in cycles per second, and $p = 1, 2, 3. \ldots$ The effect is of little or no consequence in high frequency BT plates.



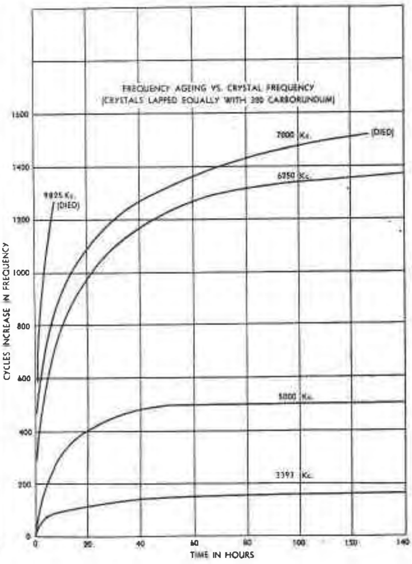


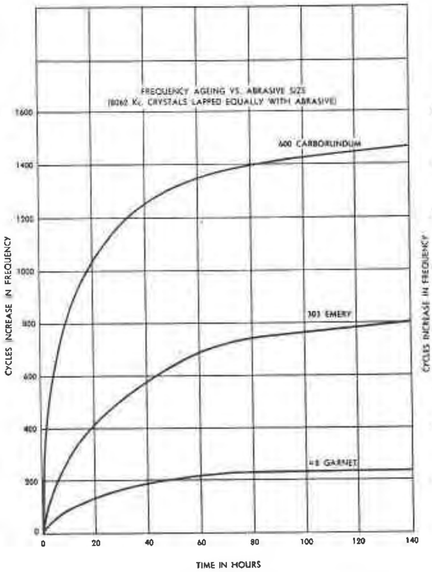
FIG. 3. Relation of frequency ageing to crystal frequency. All are BT crystals lapped identically with 303 emery.

FIG. 4. Relation of frequency ageing to crystal frequency. All are BT crystals lapped identically with 320 carborundum.

AGEING PHENOMENA IN ABRASIVE-LAPPED PLATES

The Armed Services have had serious difficulties with abrasive-finished high frequency crystals due to deterioration in storage after acceptance from the manufacturer. The ageing consists of a spontaneous increase in frequency, often to such a degree that the upper frequency tolerance is exceeded. There is an accompanying decrease in activity, usually at a slower rate, and this may continue until the plate will no longer oscillate in the test circuit. Probably several million high frequency crystals which initially passed all manufacturers and Signal Corps tests were lost by ageing before the cause and cure of the effect were found. Most of these crystals have since been salvaged. The principal features of quartz ageing are as follows:

1. In a series of plates of different frequencies that have been lapped identically with abrasive the amount of frequency ageing increases with increasing plate frequency. (Figs. 3 and 4.)
2. In a series of plates of the same frequency the frequency effect is more marked the coarser the abrasive used (Fig. 5).



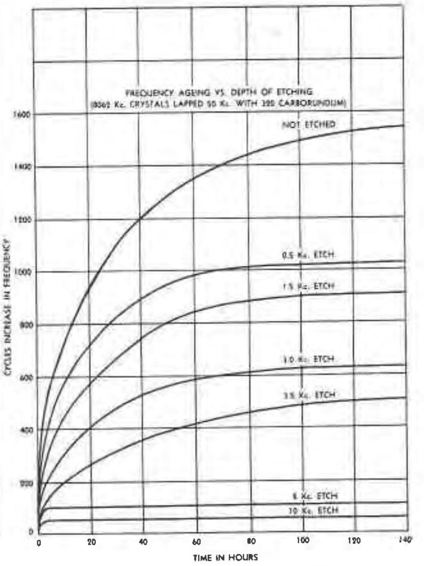


FIG. 5. Relation of frequency ageing to abrasive size. 8062 Kc BT crystals lapped 50 Kc by hand with abrasive of the type indicated.

FIG. 6. 8062 Kc crystals lapped 50 Kc with 320 carborundum and then etched varying amounts. The frequency ageing decreases with increased depth of etching up to a limit of roughly 0.5 micron of quartz.

3. In a group of plates lapped identically with abrasive the frequency ageing is both accelerated and accentuated if the plates are kept wet. The reverse is true if the plates are kept dry over a powerful desiccant. Immersion in various organic liquids has little effect on the ageing and no correlation is found with the dielectric constants or other properties of the liquids.
4. Abrasive lapped plates allowed to age over a lengthy period are found on examination to have a loose dust-like deposit of quartz over their surface. Further, the plate may be covered by a thin adherent film of silica that can be scraped up by a pointed medium. This film may be thick enough to visibly reduce the air gap between clamped electrodes; when the plate is rubbed with a towel the air gap increases, and the frequency may increase as much as several kilocycles over the original value. The film consists of hydrated (?) silica together with remnants of finger grease, abrasive, and soap or other agents used in cleaning.

5. Activity ageing is more marked and more rapid the coarser the abrasive finish and the higher the plate frequency. Generally speaking, the decrease in activity with time in crystals lapped with 303 emery or finer abrasive is measurable but does not commonly exceed tolerances in frequencies below about 6500 Kc. The deterioration becomes more serious with increasing frequency and the great majority of abrasive finished, unetched, crystals over about 7500 to 8000 Kc either drop below activity tolerance or go dead entirely with time. In a series of BT crystals finished with 320 carborundum it was found that in frequencies from 3400 to 5000 Kc many of the crystals still oscillated, with diminished activity, after standing five months, but that over about 6000 Kc all crystals soon went dead. In a series of 9825 Kc crystals, those finished 50 Kc with #8 garnet were still weakly active after five months while those finished with 600 or 320 carborundum went dead after periods of a few hours up to a few weeks.

The principal cause of ageing is believed to be a deterioration of the surface of the quartz oscillator-plate itself. Due to the abrasive action in lapping the surface of the plate becomes strained and cracked on a submicroscopic scale. This thin surface skin of misaligned quartz tends to spall off or recrystallize with time, aided apparently by adsorbed and capillary water, and the increase in frequency reflects the decrease in effective thickness of the plate. Direct evidence of the damaged surface layer has been obtained by *x*-ray, electron diffraction, and electron microscope studies; this work, done by others, will be published and need not be described here.[3] Other effects which may also contribute to undesirable frequency and activity changes with time are fatigue and corrosion of the springs and other metal parts of the holder, absorption of moisture, and deterioration of the phenolic material composing the body of the holder itself. These result in loading of the crystal by water or organic material and in decreased spring pressure.

It is found that ageing effects can be eliminated if the oscillator-plate is etched in a solvent as the last step in manufacture, thus removing the strained and misaligned material and exposing solid bedrock. Data illustrating the decrease in frequency ageing accompanying various depth of etching are shown in Fig. 6. This discovery led the Signal Corps to specify that all high frequency crystals must be etched instead of abrasive lapped to frequency. The minimum etch specifies one micron of quartz and contains a safety factor of roughly 2 to 3.

FINISHING BY THE ETCH METHOD

In the etching-to-frequency technique, the machine lapping is stopped when the crystals are a little more than one micron above final thickness. The crystals are now etched in bulk, in a single continuous operation, to remove one micron of quartz—the minimum Signal Corps requirement.

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[3] See also Bottom, V., Ageing of Quartz Crystal Units, Camp Coles Signal Labs., Crystal Branch, Eng. Memo, No. 4, 13 pp., June 29, 1944.

In this operation, the crystals may be racked together in notched copper, plastic or Pyrex trays (Fig. 7), or positioned on endless chains or suspended baskets, and then immersed for a predetermined time in a tank or shallow dish of etching solution. The solution used in mass etching usually is saturated with NH_4HF_2 and is kept at a temperature of 40 to 50° C. The amount of etching is ordinarily so controlled as to bring the crystals to about 0.02 to 0.08 per cent less than final frequency. The crystals are now issued to the finishing operators who etch them individually in a small bowl of solvent, with accompanying measurement of frequency, until the desired frequency is reached. The solution used in final

FIG. 7. Crystals racked in notched Pyrex trays, preliminary to being transferred successively to shallow tanks containing sulfuric-chromic acid cleaning solution, running rinse water, etching solution, and running water for final rinsing.

finishing usually contains about 15 to 40 per cent by weight of NH_4HF_2 and is kept at room temperature. Hot concentrated solutions are sometimes used but small frequency changes are not then easily controlled. A typical finishing position is shown in Fig. 8. After etching, the crystals are washed, dried, and mounted in holders. If the activity drops to a low value during final etching the crystal may be edge lapped on a carborundum stone or rotating abrasive cylinder (Fig. 8). Cleanliness is essential to ensure uniformity of etching. Machine lapped crystals should be cleaned before mass etching first in organic solvents or soap and water to remove most of the lapping vehicle and carborundum and then immersed in hot solutions of trisodium phosphate plus "soapless soap." Washing before the final finishing etch also is advisable, especially if the crystals have been handled; and a final scrubbing with toothbrush and soap followed by thorough rinsing in warm running water is requisite.

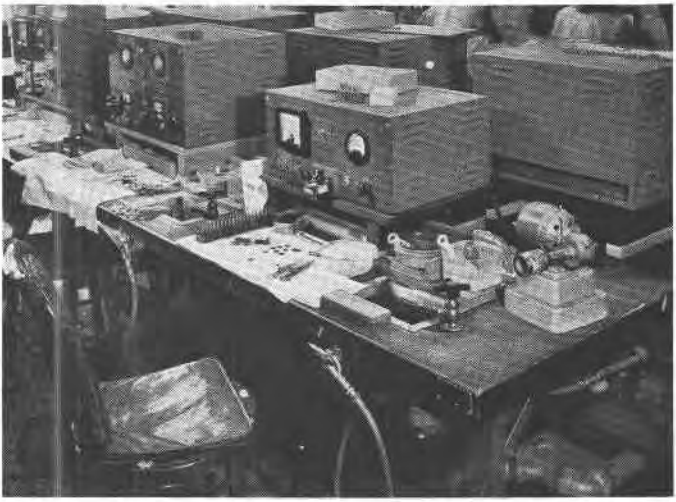

FIG. 8. Final etch finishing position. Note plastic tongs for immersing crystal in Pyrex bowl of etch solution, toothbrush and bowl of soap solution for cleaning, air gun, bowl of dilute ammonia water, comparison oscillator, and abrasive lined cylinder for beveling crystal edges.

Notes on the Etching Process. The rate of change of frequency during etching is a function of the initial frequency of the plate and increases with increasing frequency according to the frequency-thickness relation. The rate of etching is very much faster in the first minute or so than thereafter (cf. Fig. 12). This is due to the relatively rapid solution of projecting edges and corners on the lapped surfaces and is primarily an unloading effect. The effect is especially marked in dilute solutions. In crystals of the same frequency the rate of etching is faster the coarser the abrasive finish on the crystal (Fig. 9). The rate of etching also increases with increasing temperature of the etching solution (Fig. 10), and with increasing concentration (Figs. 11 and 12). Agitating the solution speeds the action. Crystals from which 5 to 10 microns or so of quartz have been etched, depending on the kind of abrasive finish, become transparent with a glazed appearance. Prolonged etching commonly results in decrease of activity due partly to relatively deep etch pits, enlargement of surface flaws, and change of contour. Different oscillator-cuts change frequency at different rates during etching because of the vectoriality of solution rates in quartz and in part, because of the differences in modes of vibration.

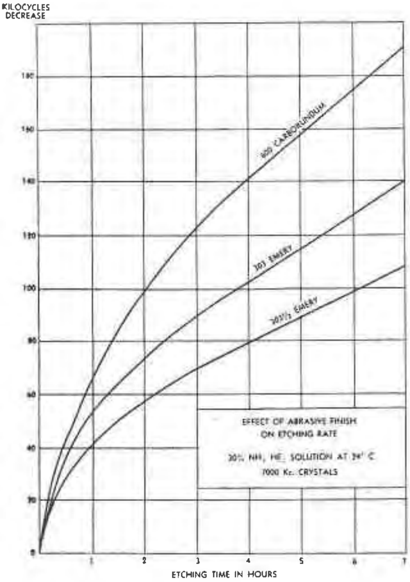

FIG. 9

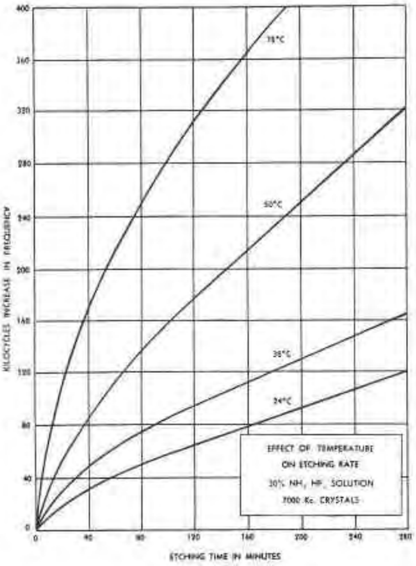

FIG. 10

Hydrofluoric acid formerly was used as the etching medium but this dangerous compound was later replaced by ammonium bifluoride and by proprietary compounds[4] based on ammonium bifluoride but containing additional ingredients to improve the quality of the work. General speaking, hydrofluoric acid gives a coarse deeply pitted effect while the ammonium and alkali bifluorides, especially when containing fluoborates,

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[4] "Safe-T-Etch" and "Frequency-Etch," sold by the Hudson American Corp., 25 West 43rd Street, New York, N. Y.

give a fine-grained even effect. Care should be taken to maintain constancy of concentration and temperature of etching solutions in order not to lose the calibration of etching rate. The solutions should be neutralized with soda ash before they are discarded to prevent damage to the plumbing.

Finishing by the *X*-Ray Irradiation Technique

A new method for making a precise final adjustment of frequency, still under development and not yet widely used in the crystal industry, also may be mentioned. When quartz oscillator-plates are irradiated with a broad beam of *x*-rays the quartz gradually becomes smoky in color and the elastic constants are concomitantly altered in such way as to reduce the oscillation frequency. The general nature of the effect is described in an accompanying paper.[5] Briefly stated: (1) the total change of frequency that can be effected in BT plates is limited to roughly 0.006 to 0.12 per cent of the nominal frequency with an average downward change of approximately 0.02 per cent; (2) the total response varies in different specimens of quartz, for reasons not known, between the limits stated; (3) the color of irradiated plates can be discharged and the frequency restored to the original value by baking at temperatures over about 180° C.; (4) the rate of change of frequency during irradiation depends primarily on the wave-length and intensity of the *x*-ray beam

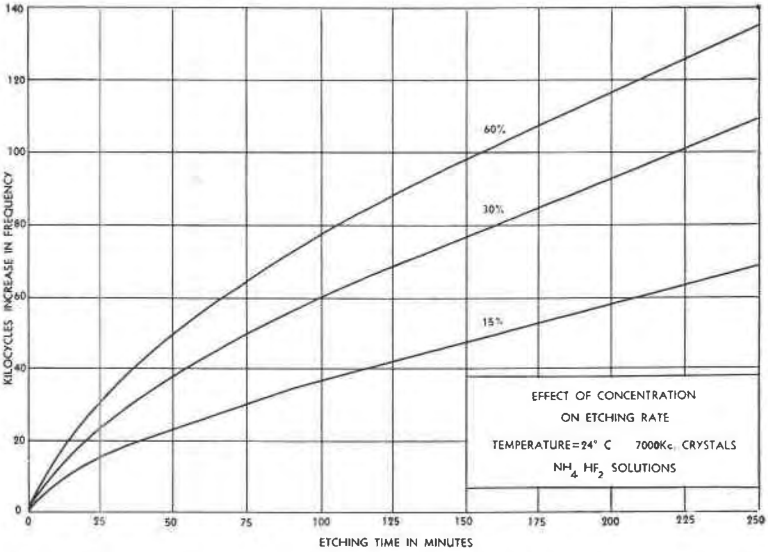

Fig. 11

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[5] Frondel, C., Effect of Radiation on the Elasticity of Quartz, *Amer. Min.*, this issue.

employed, the anode to plate distance, the thickness of the plate being irradiated, and on the total response inherent in the particular plate being treated.

The irradiation technique offers a number of important practical advantages. The frequency of the plate can be exactly adjusted under continuous visual control by oscillating it in the x-ray beam until it reaches the desired frequency. The downward change in frequency permits the salvage of plates that have gone over the upper frequency tolerance due to ageing or to over-finishing during manufacture. Further, surface stabilized or metal-plated crystals can be adjusted in frequency without disturbing the surface condition of the quartz; and plates can be finished to have a desired frequency at a specific temperature by irradiation at that temperature. The technique is of special advantage in the manufacture of ultra high frequency plates with tight frequency tolerances. The principal factors which limit the application of the method are the relatively long irradiation time needed with present equipment when

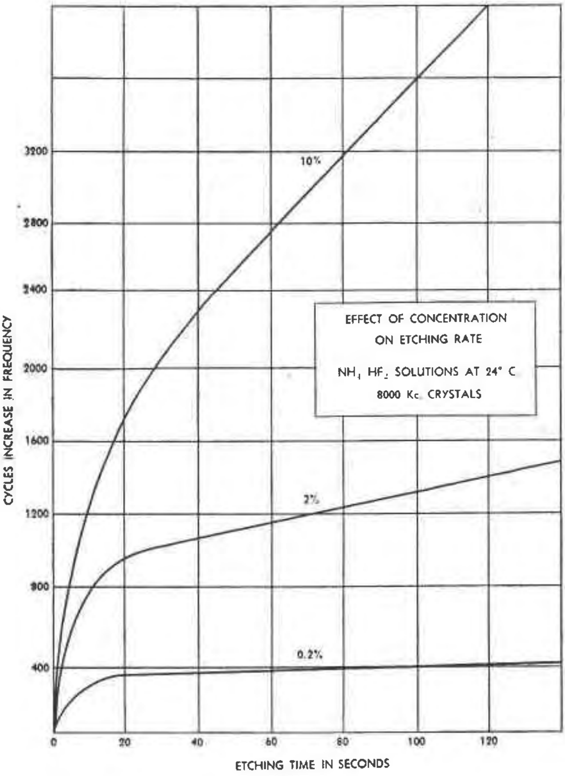

FIG. 12

large frequency changes are desired, and the high initial cost of installing sufficient equipment to handle a large volume of work. Generally speaking, the crystals have to be finished by conventional methods to within roughly 0.02 per cent of nominal frequency before irradiation since the total frequency change that can be effected is of this order.

Commercial x-ray equipment for irradiating oscillator-plates has been marketed by the North American Philips X-Ray Corporation. A photograph of three Philips irradiation units used on the production line at one of the quartz plants of Reeves Sound Laboratories in New York City is shown in Fig. 13. The equipment comprises a broad focus copper anode tube designed to operate continuously at 25 ma and 60 KV. The water-cooled tube is housed in a casting to which are attached two insulated jigs opposite the windows of the tube. The jigs consist of a rotatable shielded disc containing six variously sized electrode positions into which oscillator-plates may be inserted and in turn rotated into the x-ray beam (Fig. 14). The outer electrode facing the window is pierced with a large hole to permit the beam to strike the central area of the crystal. The jig is so constructed that the plate can be oscillated during irradiation and

FIG. 13. Philips x-ray irradiation units used to adjust the frequency of finished quartz oscillator-plates

the frequency change followed visually on a meter. The average rate of downward change of frequency in this equipment in high frequency BT plates averages between 20 and 40 cycles a minute. Crystals mounted by wire suspension in bakelite or other holders relatively transparent to x-rays can be oscillated and irradiated to frequency directly through the holder.

FIG. 14. Close-up view of jig used to hold and oscillate crystals during irradiation.

FREQUENCY ADJUSTMENT OF METAL PLATED CRYSTALS

A number of types of crystals are plated on the surface with a thin film of metal, usually gold, silver or aluminum. In crystals whose frequency is determined by edge dimensions, as in the GT, CT, DT cuts and X-cut bars, the frequency is relatively insensitive to the amount of metal deposited and to the thickness dimension of the quartz plate itself. Minor frequency adjustments of such crystals are effected by edge grinding, leaving the metallized surface untouched. These crystals ordinarily are mounted by clamping electrodes to or soldering wires to the nodal points. In plated AT and BT shear mode crystals, however, the frequency decreases with the amount of metal deposited and unless the plating operation itself is very carefully calibrated minor frequency adjustments are necessary. These are effected by operating on the metal plated surface itself. Shear mode plated crystals are preferably mounted by wire suspension, the wires being soldered to opposite and opposing edges or corners of the plate.

In the manufacture of plated AT and BT crystals, the plate is lapped and etched a number of kilocycles over the final desired frequency and then metal is superdeposited by cathode sputtering, evaporation, chemical deposition or ceramic processes until the frequency is brought back close to the desired value. If a further downward change is then required, a metal such as nickel can be electroplated upon the base coat or minor adjustments can be effected by *x*-ray irradiation. If an upward adjustment is desired the metal film can be carefully abraded with a rubber ink eraser or the film can be partly etched off with a solvent. Metal also can be deplated electrochemically. These adjustments can be made after the crystal has been soldered to its supporting wires. Efforts to remove large amounts of metal often result in a loosening of the film and if a large upward frequency is desired it is best to dissolve off all the metal and start over. Methods recently have been devised in which the plate can be oscillated and measured during the metal plating operation and brought directly to frequency. It is desirable to bake plated crystals at *ca.* 550° C. to bring about an increased particle size in the film and enhance its strength and adherence.