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HIGH-TEMPERATURE MEASUREMENTS OF Q-FACTOR IN ROTATED X-CUT QUARTZ RESONATORS*

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The Q-factors of piezoelectric resonators fabricated from natural and synthetic quartz with a 34° rotated X-cut orientation have been measured at temperatures up to 325°C. The synthetic material, which was purified by electrolysis, retains a high enough Q to be suitable for high-temperature pressure-transducer applications, whereas the natural quartz is excessively lossy above ∿ 200°C for this application. The present results are compared to results obtained previously on AT-cut resonators.

Introduction

Quartz-resonator pressure transducers are being developed at Sandia National Laboratories for high-temperature (≈ 300°C) applications in geotechnology areas.[1] Areas of particular interest include surveying of geothermal and deep oil and gas resources. In order for a crystal resonator to be used as a pressure gauge, the effect of temperature changes on the resonator frequency must be minimized compared to the pressure-induced frequency shift. Thus it is desirable to use a resonator design that is temperature-compensated to as high a degree as possible over the temperature range of interest. Plate resonators operating in the thickness shear mode are generally utilized in temperature-compensated applications, as they have turnover points in their frequency vs temperature characteristics. This means that the derivative df/dT (f = frequency, T = temperature) is zero at some appropriate temperature. For applications around 200-300°C, a rotated X-cut orientation of the resonator plate has been shown to be more suitable than other temperature-compensated orientations because it exhibits a lower curvature of f(T) at the turnover point.[2] The geometry of the rotated X-cut plate is shown in Fig. 1. Here a rotation angle of $\theta = 34^\circ$ is shown, as this is the angle that provides compensation in the temperature range of interest.

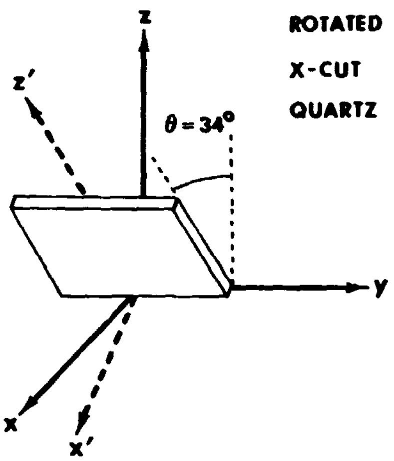

Fig. 1. Illustration of the 34° rotated X-cut orientation used for temperature-compensated pressure gauge applications.

A more detailed description of the pressure gauge being developed can be found in Ref. 1.

Although the rotated X-cut orientation has been found to be optimum from the viewpoint of its frequency vs temperature and pressure characteristics, there are no data in the literature on the Q-factor of resonators with this orientation. For stable and reliable gauge operation it is necessary to have a Q of over ∿ 10^5 for all operating temperatures.[1] It is known from previous work on quartz that the Q depends on a number of factors including crystalline orientation, growth conditions (natural or synthetic), sample preparation, and the number and type of defects present. The present work was undertaken to characterize the temperature dependent Q-factor of rotated X-cut quartz resonators fabricated both from natural and synthetic (electrolyzed) material.

Experiments

Four samples were studied in the present investigation: two natural quartz samples from Hoffman and two synthetic quartz samples from Sawyer. The Sawyer material was electrically swept (electrolyzed) at Sandia. Plano-convex resonators with deposited Au electrodes were fabricated and then mounted in hermetically sealed cans. The samples were heated in a tube furnace, and care was taken to stabilize the temperature before taking each Q measurement.

The simple apparatus used for the Q measurements is shown in Fig. 2.

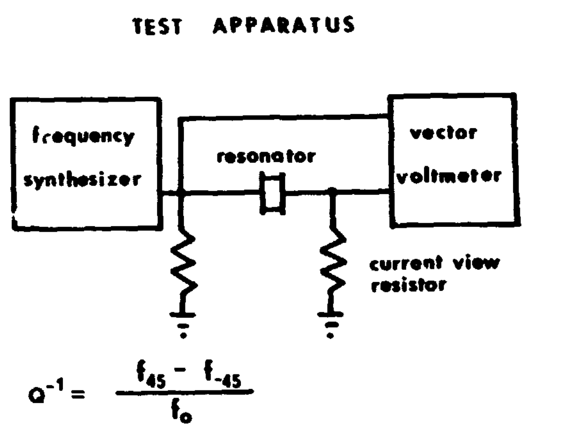

Fig. 2 Apparatus used for Q measurements

The output of a frequency sythesizer is used to excite the resonator and is simultaneously applied to the reference terminal of a vector voltmeter. Current flow through the resonator is monitored via a current viewing resistor, the voltage across which is applied to the signal input of the voltmeter. To ensure that the resonator is excited by a low impedance source, the output of the synthesizer is shunted by the resistor shown to the left of the resonator in the drawing. With this arrangement the voltmeter measures the complex admittance of the resonator. Provided that the resonance is sufficiently strong, the width (at the half power points) of the resonance is the difference of the frequencies f_{45} and f_{-45} where the current and voltage are ± 45° out of phase.[3] For the resonators used in the present work, the resonances were somewhat weaker than expected under ideal conditions. Because of this, it proved impractical to measure Q values below about 5 x 10^4 readily, as doing so would have required a point-by-point tracing out of the resonance circle in the complex admittance plane.[3]

All the data presented in this paper were obtained at the third overtone ($f \approx 3$ MHz) of the resonators. Resonator data are typically obtained at the fifth harmonic, but for the present devices the third harmonic exhibited a higher Q and a stronger resonance than did the fifth.

Results

Typical data showing the temperature-induced shift in resonance frequency for a $\theta = 34.0^\circ$ rotated X-cut resonator at atmospheric pressure are shown in Fig. 3.

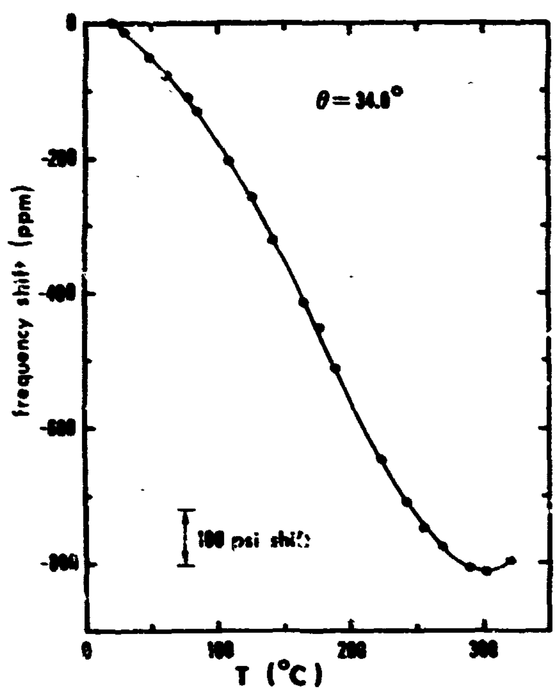

Fig. 3. Fractional change in resonant frequency vs. temperature for a (contoured) rotated X-cut resonator.

Also shown in this figure is the magnitude of the frequency shift produced by a pressure increase of 100 psi for an actual pressure gauge at 275°C.[1] For the resonator measured, the turnover point is at 300°C, compared to the value of 220°C expected from the data in Ref. 2. The shift in turnover point is believed due to the resonators in the present work being slightly contoured whereas the previous work pertains to flat plates.

Typical data for Q as a function of temperature are shown in Fig. 4. The quantity actually plotted is the loss Q^{-1} (on a logarithmic scale), as is conventional. The upper curve is for the natural (unswept) material. For this sample there is a loss peak at $\sim 70^\circ$C and a rapid increase of loss with temperature above 200°C. As mentioned above, it was not convenient with the simple apparatus utilized to measure values of Q much lower than 5×10^4. However it was observed that the loss did continue to increase with increasing temperature up to 300°C.

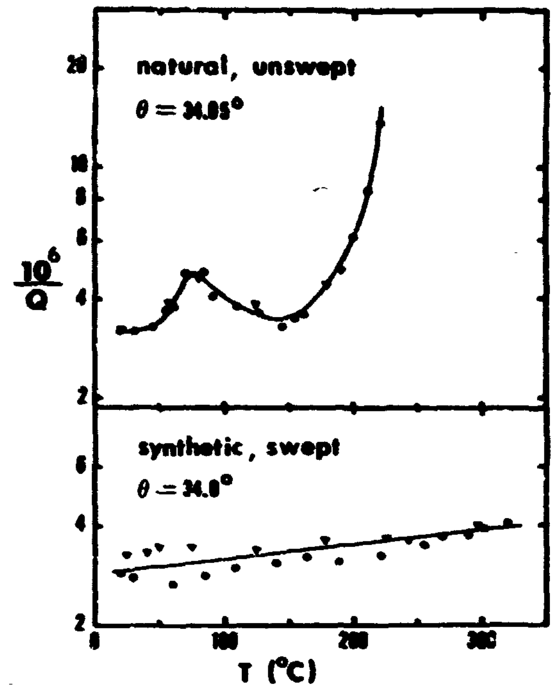

Fig. 4. Typical data for acoustic loss Q^{-1} up to 325°C. Data were taken at 3 MHz (3d harmonic).

Data obtained for a sample of synthetic swept quartz are shown in the bottom part of Fig. 4. The dramatic improvement due to the electrolytic purification is immediately apparent. Since the Q is more than 2.5×10^5 over the entire range of temperature, it appears that synthetic swept quartz is suitable for pressure gauge applications. Data obtained on the other sample of swept synthetic material are similar to those shown in Fig. 4. An important point to mention with regard to the present data is that the actual intrinsic Q of the swept synthetic material may be higher than the data indicate. This is because the resonators were plated and the resulting stresses may dominate the loss for high quality quartz. Previous workers have noticed this effect,[4] and for reliable measurements of $Q \geq 10^6$ it is advisable to drive the resonator by capacitive coupling across a gap.

Discussion

It is of interest to compare the present results with those obtained in previous studies of resonator loss as a function of temperature. Most of the previous work in this area has been done on AT-cut thickness shear resonators. The extensive early work that was done has been reviewed by Fraser,[4] who discusses in detail the effects of impurities, radiation, and electrical sweeping on the temperature dependent acoustic loss. Nowick and Stanley[5] have given a group-theoretical analysis of dielectric and acoustic relaxation in quartz and have used the results to interpret data in the literature.

From symmetry considerations, Nowick and Stanley have argued that all pure shear mode deformations will couple to relaxational normal modes transforming according to the doubly degenerate E representation of the crystalline point group (D_3). Since the AT-cut thickness shear mode involves a pure shear deformation, and since the rotated X-cut thickness shear mode is very nearly a pure shear mode,[2] one would expect similar anelastic behavior for the two different orientations. Of course, the magnitudes of the anelastic relaxations cannot be deduced from symmetry arguments. Nonetheless, it is not particularly surprizing that the data of Fig. 4 are somewhat similar to previously published data on AT-cut resonators fabricated from natural and swept-synthetic quartz.

The previous work[4,5] on anelastic loss in quartz resonators has led to a partial understanding of the relation between various impurities in the samples and the various loss peaks observed. Unfortunately, the behavior at low temperature is better understood than at high temperature. All quartz, natural or synthetic, contains a significant number ($\gtrsim$ 5 ppm) of aluminum (Al^{3+}) impurities which substitute for silicon in the lattice. These defects are charge-compensated by alkali ions (Na^+, Li^+ or K^+) at interstitial positions adjacent to the Al. Compensation by protons is also possible. The motion of the interstitial ion among equivalent positions in response to the acoustic stress is an important mechanism for producing acoustic loss. Careful electrolytic sweeping can remove the alkali impurities, and it is believed that protons or, in certain cases, holes provide charge compensation of the Al^{3+}. Water may also be incorporated into the quartz lattice (eg during growth) by replacing a Si-O-Si bridge with two Si-O-H structures.

For natural quartz the rapid rise in loss above 200°C as seen in Fig. 4 is believed due to alkali diffusion in response to the applied stress. Removal of alkalis by electrolysis is necessary to reduce this source of loss. The loss peak shown at $\sim$ 70°C in Fig. 4 may be of the same origin as a similar peak observed in natural Brazilian opaline quartz and in fast Z-growth synthetic quartz.[4] It appears to be associated with OH bonds.

The data for synthetic quartz in Fig. 4 do not show any evidence of acoustic loss peaks. The previous work on AT-cut resonators has shown that loss peaks usually are observed, but that they are quite weak. It appears that the background loss due to electroding and mounting may have obscured any small loss peaks in the present measurements.

Conclusions

Two main conclusions may be drawn from the present work. The first is that the acoustical loss properties of rotated X-cut resonators appear similar to those of the widely studied AT-cut. Thus most past experience on AT-cut resonators may provide a valuable guide in designing devices using the rotated X-cut. The second conclusion is that electrolytically swept synthetic quartz appears to have sufficiently high Q for pressure gauge applications, whereas natural quartz is unsuitable for temperatures above $\sim$ 200°C.

References

1. E. P. EerNisse, Sandia Laboratories Report No. SAND78-2264, (1979).

2. E. P. EerNisse, Proc. 32nd Annual Frequency Control Symposium, U.S. Army Electronics Command (Elect. Ind. Assoc.), p. 255 (1978).

3. R. Holland and E. P. EerNisse, IEEE Transactions on Sonics and Ultrasonics SU-16, 173 (1969).

4. D. B. Fraser, in Physical Acoustics, ed. by W. P. Mason, (Academic, New York), vol V, p. 59 (1968).

5. A. S. Nowick and M. W. Stanley, in Physics of the Solid State, ed. by S. Balakrishna et al., (Academic, New York and London), p. 183 (1969).

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