JOURNAL OF MATERIALS SCIENCE 15 (1980) 3066–3070

Strained surface layers of quartz plates produced by lapping and polishing and their influence on quartz resonator performance

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The strained surface layers of AT-cut quartz plates produced by lapping and polishing have been studied using the X-ray double crystal method. In addition, the influence of surface strain on the characteristics of quartz resonators has been investigated. Experimental results from X-ray double crystal measurements indicate that a residual stress layer in the lapped and polished surface is created and that the quartz resonator performance is also affected.

1. Introduction
Quartz resonator plates have been processed by lapping and polishing and it has been suggested that a strained surface layer is created by these processes [1–3]. It is likely that the influence of this layer on the electrical properties of the quartz resonator is greater with thin quartz plates and that this layer affects the frequency stability, equivalent resistance and ageing [4]. In order to make a high frequency AT-cut quartz resonator, the quartz plates need to be thin. Consequently, the effect of the strained layer on quartz resonator performance is important.

In the present paper, the properties of the strained layer have been investigated and the correlations between this layer and the resonator performance were studied.

2. Experimental procedures
2.1. Specimen
The specimen used was a synthetic Y-bar of quartz and the growth conditions were: temperature 350° C, autoclave pressure 1.2 kbar, growth rate 0.7 mm day^{-1} in the 1N NaOH solution. The AT-cut resonator plates from a Z-growth sector of Y-bar block shown in Fig. 1 were studied. The AT-cut plates lay within 35°21′ ± 30″ of the Z-axis, the flatness is $\lambda/4$, the parallelism is ± 15′ and the fundamental frequency mode for thickness shear vibration is 10 MHz. The process of lapping and polishing is shown in Table I.

2.2. X-ray measurements
The double crystal method is very sensitive to minute lattice strains and the assymmetric parallel arrangement [5, 6] was used in the present work. The $(10\bar{1}1)$ plane cut from a synthetic quartz was used for the first crystal with the assymmetric factor [7] of $b = 1/27$ for MoKα_1 X-rays.

TABLE I Lapping and polishing process of AT-cut quartz plates by various abrasive powders. Initial cut thickness from as-grown Y–bar synthetic quartz is 1 mm and the specimens are lapped and polished by various particle sizes of abrasive powder (no. 800, no. 1500, no. 3000 and CeO_2). The final thickness with each abrasive powder is 0.167 mm which corresponds to 10 MHz at AT-resonators at the fundamental mode

Particle size No.	no. 800 (16 μm)	no. 1500 (8 μm)	no. 3000 (5 μm)	CeO_2 ($\lesssim 1\,\mu$m)
no. 800	0.167	0.317	0.357	0.361
no. 1500		0.167	0.207	0.211
no. 3000			0.167	0.171
CeO_2				0.167

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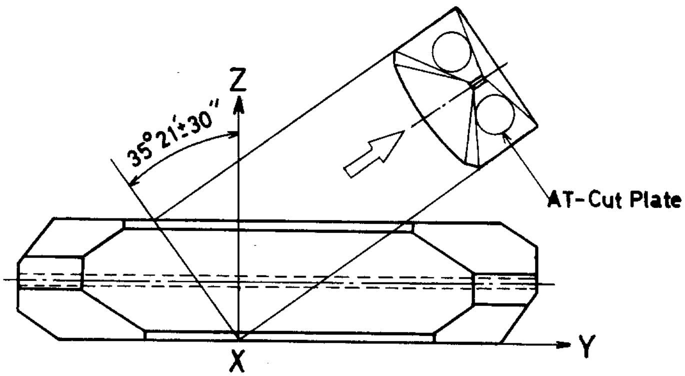

Figure 1 AT-cut quartz resonator plates cut from synthetic Y-bar quartz.

The specimen for lattice strain measurement is prepared by CeO_2 powder polishing to a final thickness of 1 mm.

2.3. Measurements of the quartz resonator performance

The oscillators were prepared the same size as the commercial oscillators shown in Fig. 2. The thickness of the Ag evaporated film electrodes was about 700 Å and the connection between the quartz plate and the wire was made by conductive Ag adhesive. The quartz oscillators were finally packaged in vacuum ($\simeq 10^{-4}$ torr) with a cold weld technique.

The electrical properties of the quartz resonators were measured by means of a π-circuit method [8]. According to this method, the equivalent electric circuit constants of resistance, capacitance and inductance, R_1, C_1, L_1, respectively, and Q-value are calculated by the following equations

$$C_1 = 2(C_0 + C_L) \cdot [(f_{rL} - f_r)/f_r]$$
$$L_1 \doteq (4\pi^2 f_r C_1)^{-1}$$
$$R_1 = (V_a - V_b) \cdot (R/V_b)$$
$$Q = (2\pi \cdot f_r \cdot C_1 \cdot R_1)^{-1},$$

where f_{rL} and f_r indicate the resonance frequency whether the additional capacitance (C_L) is inserted or not in the π-circuit, R is the fixed resistance of the π-circuit, and V_a and V_b are the voltages of the quartz resonator before and after insertion of the π-circuit.

3. Results and discussion

Fig. 3 shows the rocking curves of diffracting X-rays from the $(10\bar{1}1)$ plane for AT-cut polished and etched quartz surfaces, and Fig. 4 shows the half-width of the rocking curves for lapped and polished quartz surfaces. Lapped and polished surfaces indicate a broad rocking curve caused by the strained layer. After 1 min etching by HF, the rocking curves become nearly equal to that of perfect crystal surfaces and the surface state shows a single crystal pattern (Fig. 3c). For the surface polished with CeO_2, 1 min etching by HF corresponds to the thickness of about 800 Å.

Fig. 5 shows the Bragg angle displacement between as-polished and etched quartz surfaces, which is caused by the lattice strain with a residual stress from polishing. These peaks corresponding to the Bragg angle are determined by measuring the X-ray intensity with a dark X-ray film and a

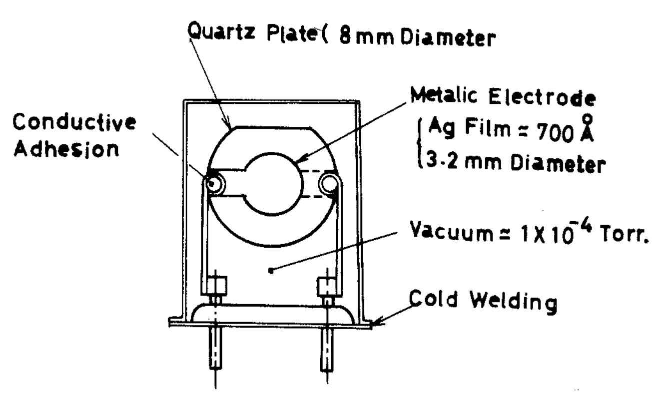

Figure 2 AT-quartz resonator.

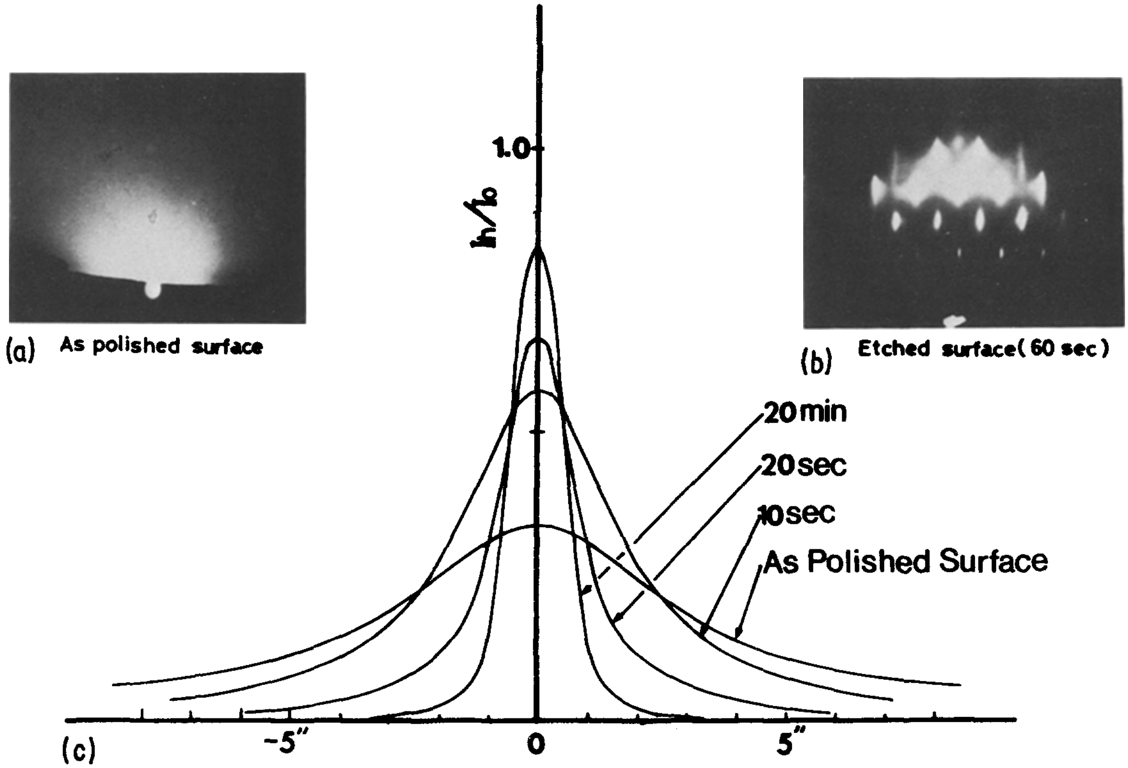

Figure 3 (a) As-polished surface state shows a harrow pattern (an amorphous state) by electron microscopic observation (100 kV). (b) After 1 min etching, the etched surface state shows a perfect single-crystal pattern. (c) An example of observed rocking curves from (1 0 $\bar{1}$ 1) diffracting plane for polished and etched quartz surface by the double crystal method.

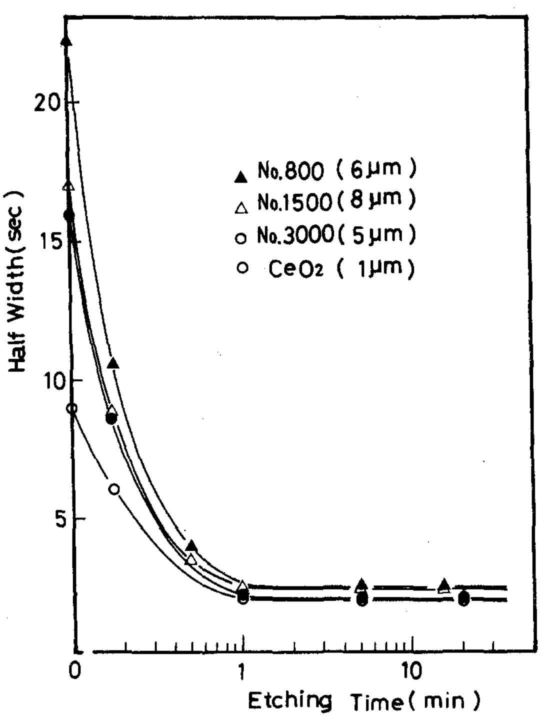

Figure 4 Variations of half-width of rocking curves with etching time by HF from (1 0 $\bar{1}$ 1) diffracting plane.

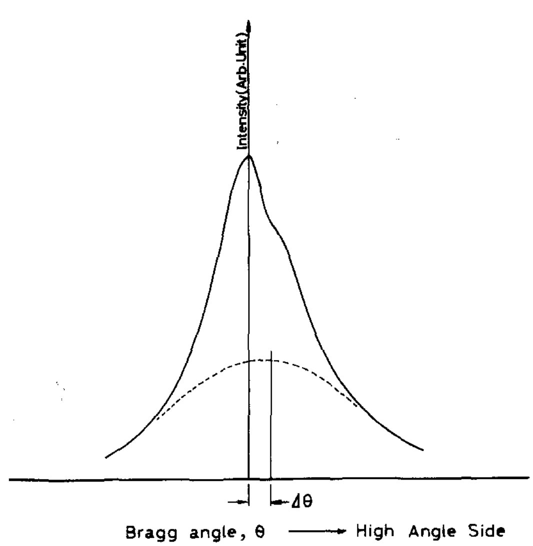

Figure 5 Displacement of Bragg angle from (1 0 $\bar{1}$ 1) diffracting plane by double crystal method with assymmetric parallel setting between as-polished and etched quartz surface.

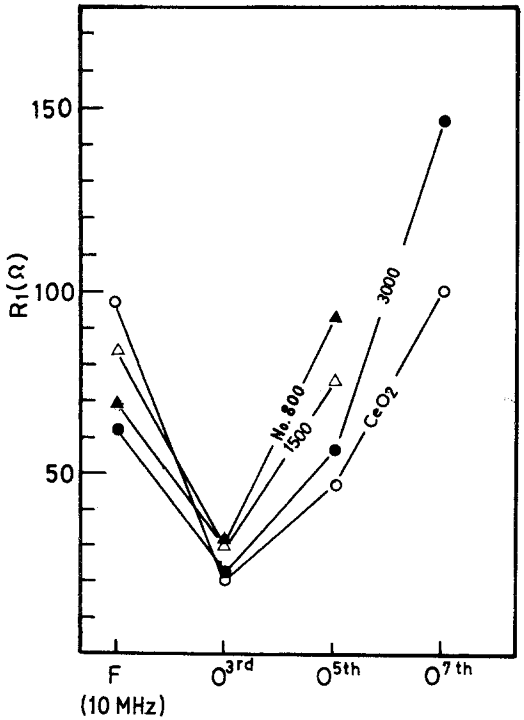

Figure 6 Correlation between oscillating modes and equivalent electric constant (R_1) of as-lapped and polished AT-quartz resonators.

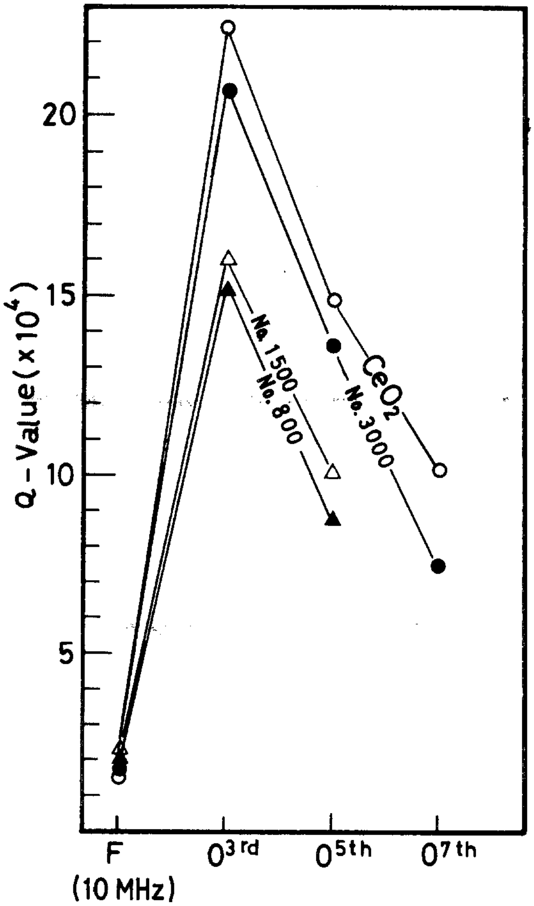

Figure 7 Correlation between oscillating modes and *Q*-values of as-lapped and polished AT-quartz resonators.

scintilation counter for a fixed time. Therefore, this difference in Bragg angle ($\Delta\theta_B \simeq 0.7''$) corresponds to $\Delta d/d \simeq 3.3 \times 10^{-5}$ lattice strain for a (1 0 1 1) plane. Using the quartz elastic constants given by Mason [9], the residual plane stress caused by this lattice strain in the polished surface has been shown to be about 34 kg cm^{-2}.

Figs 6 and 7 show the correlation between oscillating modes and equivalent electric resistance, *Q*-value of the quartz oscillators. The equivalent electric resistance (R_1) and the *Q*-value are evidently affected by the strained layer. In the case of an oscillator, the minimum equivalent resistance (the maximum *Q*-value) exists at a third overtone mode. Although the effect of the strained layer at the fundamental mode is not much compared with the overtone mode, the maximal performance of a quartz oscillator is obtained at the third overtone ($\simeq$ 30 MHz) mode. This indicates that these effects, in addition to the strained layer at the fundamental mode, predominantly control the characteristics of the quartz oscillator. X-ray topographic observations on the oscillating quartz plate, show some inharmonic vibrations (flexure, thickness twist etc.) in the fundamental mode rather than harmonic vibrations (thickness shear mode). These inharmonic modes disappear more quickly than the third overtone mode. The results indicate that the quartz oscillator performance is affected by the strained layer and this behaviour appeared as the overtone mode increases because the effective thickness of the quartz oscillator decreases with increasing overtone mode. The ratio of the strained layer thickness to the effective thickness of quartz oscillator increases. Consequently, as the thickness of the quartz plate becomes less, the influences of the strained layer with respect to the performance of the quartz oscillator become important.

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Received 28 February and accepted 8 May 1980.