Mini-device for controlled quartz etching

G. Pentovolis, P. Collet

► To cite this version:

G. Pentovolis, P. Collet. Mini-device for controlled quartz etching. Journal de Physique IV Proceedings, 1994, 04 (C2), pp.C2-73-C2-77. 10.1051/jp4:1994209 . jpa-00252477

HAL Id: jpa-00252477

<https://hal.science/jpa-00252477>

Submitted on 1 Jan 1994

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Mini-device for controlled quartz etching

G. PENTOVELIS and P. COLLET

Rue Keller, Z.I., BP. 16, 10151 Pont-Sainte-Marie, France

Abstract. We propose a mini-device for an individual quartz chemical etching. It is based on an electrolyte(1)/ solide/ electrolyte(2) double sided contact. When the electrolyte is an etching solution on either one or both sides of quartz (solide), frequency variation can be observed. The implementation of the mini-device is simple and makes automatisisation of the etching procedure possible. We expose the mini-device operating and the span of its application.

1. Introduction.

In the recent past many laboratories have studied the effects of Quartz/liquid Junctions⁽¹⁻³⁾. When a quartz is in contact with a liquid, the quartz frequency is modified. This effect is caused by the electrochemical equilibrium on the interface. These previous works were initiated to the analytical chemistry applications.

In electrochemistry, semiconductors/electrolyte junctions are used to the study of the semiconductors corrosion by impedance measurements.

In the chemical etching for quartz manufacturing application, the control of the etched surface evolution is difficult. Usually temperature and concentration control of the liquid are used. Then, two or more steps are needed for a good final frequency precision.

2. Results.

We have studied an electrolyte(1)/quartz/electrolyte(2) double sided contact to solve this problem. The device of figure 1 has been worked out. Its dimensions are smaller than 50x50x50 mm. We called it mini-device.

In the either one or both compartments, the electrolyte is an etching solution, like ammonium bifluorure or sodium hydroxide aqueous solutions.

FIGURE 1. Mini-device.

Two screws are used for the network analyzer connection and for the calorific energy transmission from a hot source to the solution. We can maintain the solution's temperature via the metal screws.

In the figure 2 the impedance-frequency graph of a quartz is drawn (air gap system measurements at room temperature). The resonance frequency is 19707 KHz.

When this quartz is introduced in the mini-device the impedance graph changes (see figure 3). We can see that span of the graph changes from 20 KHz to 100 KHz per division and from 1 KOhms to 20 Ohms per division. Quality factor Q ⁽⁴⁾ and resonance frequency decrease. The impedance modification is more important when a more concentrated solution is used (see figure 4). It is very difficult to attribute these modifications to one or other motionel parameters. But the resonance frequency measurement is always possible.

In the case of a quartz only, the equivalent electrical circuit can be a RLC classical circuit. When the quartz is introduced into the mini-device, the electrical circuit of the metal/electrolyte and the quartz/electrolyte contacts are added in serie. The parameters that change the quartz mode vibration are liquid concentration, liquid viscosity, charge transfer in the quartz/electrolyte and metal/electrolyte interfaces, and stress of the device on the liquid/quartz contact boundary.

When a physico-chemical reaction exists between quartz and liquid, the quartz vibrations persist (figure 4). When the etching rate is important the quartz thickness is reduced and the quartz frequency is increased. On the network analyzer we can see the quartz/solution interface modification, because this is the only real modification into mini-device system. The graph of the figure 5 is obtained.

It's obvious that mini-device resonance frequency variation can be observed in real time.

Also we see the quality factor modification during the etching. During the first 6 microns of the etching (so 3 microns per side) Q factor is increasing. After, Q factor is decreasing continuously. This is attributed: firstly to the modification of quartz/etching solution interface improvement because the polycrystalline superficial layer is removed, and secondly to the modification of the increasing quartz sensibility at stress, concentration, and viscosity of the liquid.

Above 40 MHz the Q factor becomes very low. The resonance frequency measurement is difficult. We can see the spectrum of the mini-device on the network analyzer, but resonance frequency measurements are not precise.

3. Conclusion.

Quartz frequency variation measurements are possible in real time during the etching process, when the mini-device is used. Thus, we neither need to know the etchant concentration nor the temperature to know the quartz frequency or thickness variation.

We have seen that liquid contact modifies the quartz frequency, but for a given temperature and liquid concentration we can determine the frequency variation caused by the liquid contact thereby enabling close control over the final frequency.

In the mini device only a central area of the quartz is etched. The other surface area is masked by the O-ring. So the crystal produced by the mini-device has a reduced thickness in the central area surrounded by a thicker outer ring.

The implementation of the mini-device is simple and makes automatisisation of the etching procedure possible.

4. References.

- [1] K.Kanazawa, J.G.Gordon, Anal. Chem. **57**(1985)1771-1772
- [2] R.A.Cranet, G.Fischer, J.Phys.D.; Appl.Phys. **12**(1979)2019-2026
- [3] J.Hlavay, G.G.Gulbault, Anal.Chem. **49**(1977)1890-1898
- [4] G.Bardeche, C.Filiatre, J.-P.Valentin, Proceeding of the 6th European Frequency and Time Forum, Noordwijk, NL, 17-19 March 1992, pp 265-268

FIGURE 2. Impedance-frequency graph of quartz. Air gap system measurement at room temperature.

FIGURE 3. Impedance-frequency graph of the above quartz in the mini-device. Low concentrated solution at room temperature.

FIGURE 4. Impedance-frequency graph of the above quartz in the mini-device. High concentrated solution at temperature higher than 100°C .

FIGURE 5. Impedance-frequency graphs of the above quartz in the mini-device. High concentrated solution at temperature higher than 100°C (same than figure 4)