THE EFFECT OF RAPID THERMAL ANNEALING ON QUARTZ RESONATORS

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ABSTRACT

The temperature treatment of the quartz resonators is very important for the electrode structure and their morphology, for the acoustical losses and the aging processes.

This paper presents our results on the influence of RTA (Rapid Thermal Annealing) two side radiation heating of resonators on the equivalent dynamic parameters. The results are compared with our previous results about one-side contact heating.

The morphological changes of the electrode materials as a resulting from RTA are studied.

The effect of the RTA parameters on the equivalent dynamic parameters of the quartz resonators for the both types of heating is compared.

The RTA conditions for rapid changes of resonator amplitude-frequency characteristics and the consequent change of the basic mode are established.

On the base of the Thermal Sensitive Quartz Resonators (TSQRs) the actual temperatures of the treatment as a function of the annealing duration and the previously given heaters temperature are determined.

Conditions for a significant improvement of the acoustic parameters of the quartz resonators are established.

1.INTRODUCTION

The quartz resonator parameters can be influenced in different ways in order to improve and stabilize them..
Theoretical overview of the influence of different surface parameters on the behaviour of quartz crystal components is published. A program has been created which enables the estimation of the role of the material on electrical parameters of devices and their temperature frequency behaviour [Ref.1].
It is well known, that the basic parameter influencing resonator stability is the temperature treatment, wich is directly connected with the processes of aging.Two main parameters are involved in quartz aging behaviour: quartz cut and average operating temperature [Ref.2].
The influence of RTA on resonator behaviour has been shown in [Ref.3,4,5] previously published our articles.
The process is very complex and is connected with changes of structural, morphological and electrophysical properties of the metal electrodes and with alterations of quartz structure under the Al-SiO_2 interface. All our experiments up to now have been carried out by one side direct heating and at fixed parameters of the RTA unit heaters.
The aim of this work is to investigate the influence of RTA on the resonator structures by two-side radiation heating.

2.EXPERIMENTAL RESULTS AND DISCUSSION

All resonators were manufactured from the same quartz material (AT-cut) in the same technological process. The diameters of quartz plates and electrodes were 8 mm and 4 mm respectively. The electrodes of 120 nm thickness have been deposited on two sides of the quartz plate.Two types of electrode materials were used - Ag and Al.
The Al-electrodes have been E-beam evaporated with a rate of 8,6 Å/s.
The other type of electrodes-Ag have been obtained by vacuum resistive evaporation at an initial pressure of 10^{-3} Pa with a rate of 35 Å/s.
The resonators has been subjected to RTA in vacuum of $6{,}66.10^{-3}$ Pa at heater's temperatures 700^0C, 800^0C and 900^0C for annealing durations from 15s to 180s.
The actual temperature of the resonators during the treatment has been estimated by using TSQRs (temperature sensitive quartz resonators) as temperature sensors for calibration of the equipment. More details of the experiment is presented in [Ref.6].
The main parameters of the resonators have been measured using conventional methods by selective level Meter PSM-14 from Wandel and Golderman.
The measured parameters of all resonators were: the resonance frequency (f), the equivalent dynamic resistivity (Rs) , the static capacitivity (Co) and the relative insertion losses (Ps) .The equivalent dynamic capacitivity (Cq) , the equivalent dynamic inductivity (Lq) and the qualitative factor (Q) have been calculated.
All samples' parameters have been measured before and after RTA treatment and their equivalent dynamic parameters have been calculated respectively.
The effect of RTA treatment of resonators with Ag electrodes on the equivalent dynamic parameters has been presented in Table 1

. Table 1. Influence of RTA – process on the equivalent parameters of quartz resonators

Sample	RTA heater|′ s parameters		Tempera-ture of the resonators	Serial resonance frequency f [Hz]		Equivalent parameters of quartz resonators before and after RTA			
						Rs[Ω]		$Q.10^3$	
No	T [°C]	t [s]	T [°C]	Before RTA	After RTA	Before RTA	After RTA	Before RTA	After RTA
N – 9	800	15	175	14689284	14689534	13.60	7.80	30.52	53.33
N – 7	700	180	185	14666663	14667016	9.70	8.40	45.73	52.92
N – 21	800	30	255	14624687	14624938	7.50	5.38	55.67	77.62
N - 17	800	60	305	14623737	14625010	15.90	8.70	26.25	49.47

It is obvious that in the temperature range from 175^0C to 305^0C all equivalent parameters are influenced.The values of the serial resonance frequency of the resonators raised negligibly as result of the treatment. Probably the relaxation in the quartz crystal lattice is responsible for this behaviour. Equivalent dynamic resistivity has been affected in the whole range of heating and
become smaller in the investigated temperature interval. These alterations can be explained by changes in electrode structure and density and their adhesion to quartz wafers [Ref.5].
The values of the C_q and L_q before and after RTA have been retained in the same limits (24 to 26)fF and (4,50 to 4,80)mH respectively .In the same time the qualitative factor has been increased significantly within the limits from 16% to 88%.
The changes of the resonator parameters with Al electrodes have shown the same tendency. It can be suggested, that the improvement of resonator parameters have been connected with decreasing of residual mechanical stresses in the quartz crystal lattice as a result of the short impulse heating.
At heating temperature up to 305^0C the value of the serial resonance frequency has improved and the fundamental mode left unchanged (Table 1).
When the RTA temperature increases in the range from 305^0C to 370^0C the thickness share mode (TS) is been observed to disappear, the pure thickness mode (TE) arises as a fundamental and a third overtone is obtained. The measured values were presented in Table 2.

Table 2. Influence of RTA on the mode transformation

Sample	RTA heater′s parameters		Resonators′ temperature	Resonator′s frequency, f [Hz]		
				Before RTA (Ts mode) fundamental	After RTA (TE mode)	
No	T [°C]	t [s]	T [°C]		Fundamental	3 - th overtone
A – 13	800	60	305	14667821	21603796	64913956
A – 19	800	180	345	14539541	21430659	64385210
A - 14	900	15	255	14666539	21529438	64568110
N – 12	800	60	305	14679070	21626512	64973802
N – 13	800	180	345	14669649	21638649	64964800
N – 14	900	30	300	14631027	21556305	64773650
N - 24	900	60	370	14637848	21571084	64805847

This phenomenon has been observed for both types of resonators with Al and Ag electrodes.
It's interesting to note, that at the temperature of 305^0C some resonators have kept the frequency fundamental mode after RTA (N17) ,while the others (A-13) have changed it.
This behaviour of resonance frequency as a result of RTA treatment is in agreement with assumption, that partial amorphization at the surface takes place in this process.
It has been established that 305^0C is a critical temperature for this process. The observed difference in f-measurements have been explained by a different degree of amorphization of the samples.

The morphological studies of the resonators electrodes were carried out by Scanning Electron Microscopy (SEM) on JSM 5300.
The surfaces of all samples proved to be rough (fig.1a,fig.1b). At low magnification of 2000, no difference has been observed between Al and Ag surfaces, but the difference is clearly seen at the magnification in the order of 20000 (fig.2c, fig.2d). The structure of Al electrodes is small grained , while Ag electrodes have smooth surfaces. No significant changes of the surfaces as a function of RTA parameters of two-side radiation heating have been observed.

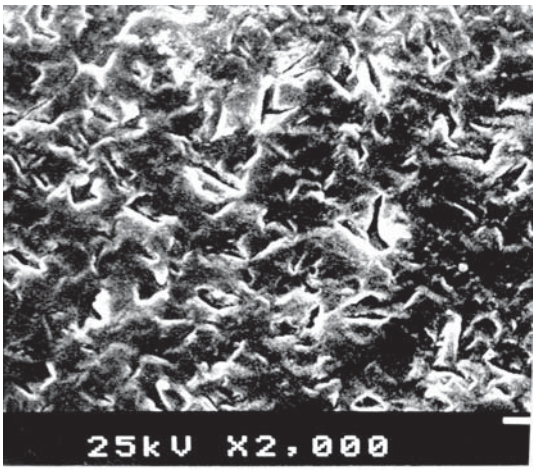

Fig. 1a Morphology of thin films after RTA at 305^0C for 60s. Magnification x 2000, Al – film

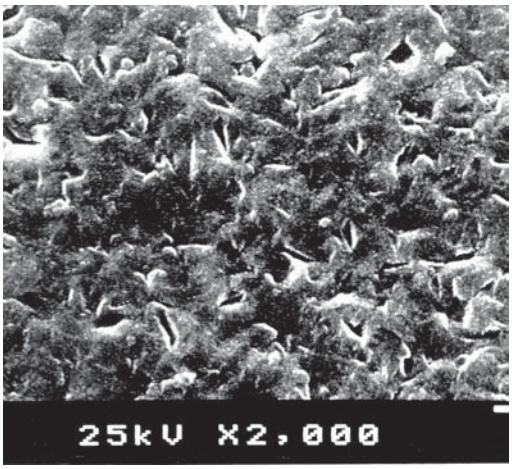

Fig. 1b Morphology of thin films after RTA at 305^0C for 60s. Magnification x 2000, Ag – film

These results are in contrast to our previous studies [Ref.4] on electrode morphology after contact heating.

3.CONCLUSION

.In has been shown, that the rapid thermal annealing of resonator structures by two side radiation heating influence the equivalent dynamic parameters and the resonance frequency .

Two temperatures ranges of change have been determined: the first up to 305^0C in which Rs improves and the second between 305^0C and 370^0C in which the fundamental frequency mode changes.
These results could be useful in improving resonator parameters for very short temperature treatments

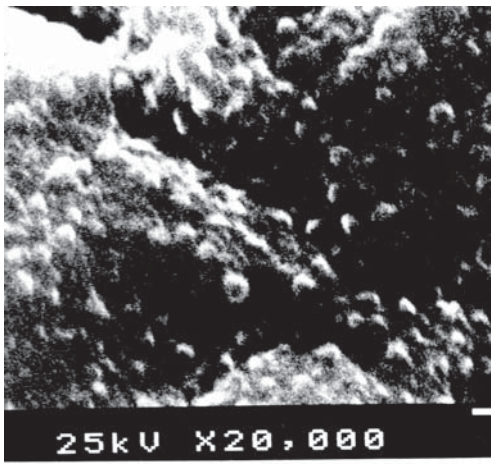

Fig. 2 c Morphology of thin films after RTA at 305^0C for 60s. Magnification x 20000, Al - film

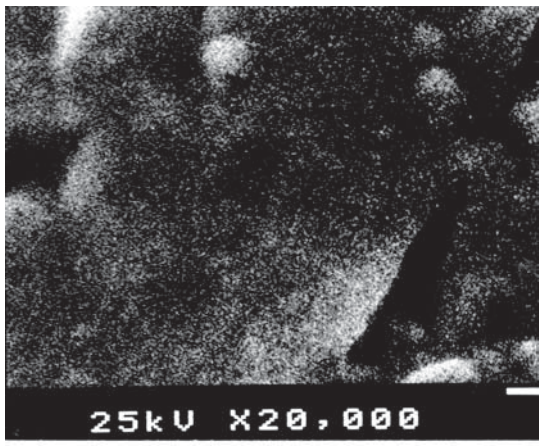

Fig. 2 d Morphology of thin films after RTA at 305^0C for 60s. Magnification x 20000, Ag – film

4.ACKNOWLEDGEMENTS

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