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Improved Process for Making Thin-Film Sodium Niobate Capacitors

Sodium niobate (NaNbO_3), a material of nominally high dielectric constant, has been used as a thin film dielectric in microelectronic capacitors. The films formed by conventional vapor deposition processes are generally amorphous, lack chemical and structural homogeneity, and are often contaminated with foreign metals from the vapor deposition apparatus. Such sodium niobate films have a lower dielectric constant and a higher dissipation factor than the sodium niobate films formed by a new process involving three conventional evaporation methods. These entail high vacuum, flash, and reactive evaporations.

In the new process, the sodium niobate film is formed from high purity sodium carbonate and metallic niobium. The heat source is a commercially available electron beam apparatus, incorporating a tungsten filament which is shielded to prevent the deposition of tungsten and its oxides. The copper hearth is lined with a sheet of niobium. The niobium evaporant together with sodium carbonate is placed on this hearth liner, which prevents alloying of the molten evaporant with copper or evaporation of the copper by the electron beam. The reaction occurring in the flash evaporation can be represented as follows:

- (1) $\text{Na}_2\text{CO}_3 \rightarrow \text{Na}_2\text{O} + \text{CO}_2$
- (2) $2\text{Na}_2\text{O} + \text{O}_2 \rightarrow 2\text{Na}_2\text{O}_2$
- (3) $\text{Nb} + \text{O}_2 \rightarrow \text{NbO}_2$
- (4) $\text{Na}_2\text{O}_2 + 2\text{NbO}_2 \rightarrow 2\text{NaNbO}_3$

The Na_2O formed (equation (1)) is deposited on the capacitor base electrode, and the CO_2 gas is pumped from the vacuum system. Oxygen present in the system combines with the Na_2O to form sodium peroxide (Na_2O_2) on the base electrode. The molten niobium is simultaneously evaporated from the hearth and is deposited on the base electrode where it is oxidized (by the residual oxygen in the system) to niobium oxide

(NbO_2). The sodium peroxide then combines with the niobium oxide to form the sodium niobate film on the base electrode.

The reaction will more readily correspond to equation (4) if the evaporation is conducted by alternate depositions of sodium oxide and niobium. The process can be summarized in accordance with the following steps:

1. Vapor deposit base electrode metal using standard high vacuum techniques.
2. Increase residual pressure by introducing oxygen to the vacuum chamber, and maintain at 5×10^{-4} torr.
3. Deposit sodium oxide by flash evaporation of sodium carbonate from hot niobium slug. The temperature of the niobium is kept below the decomposition temperature of Na_2CO_3 and below the melting point of the metal. A temperature range of $900^\circ\text{--}2000^\circ\text{C}$ has been found usable.
4. Deposit niobium.
5. Alternate Steps 3 and 4 to obtain desired thickness.
6. Raise substrate temperature from approximately 350°C to 460°C and maintain oxygen pressure. One hour has been found adequate for stress relief, should the recrystallization be completed in air subsequent to this step. Four to five hours have been found satisfactory for a deposit thickness of approximately 10,000 angstroms. Oxygen pressure can also be increased, should this be desired.
7. Repeat Step 1 to form the counter electrode. A complete capacitor will have been formed if masks were used to define the geometry during deposition. Photolithographic techniques can be employed to form desired patterns by standard masking and chemical etching.

(continued overleaf)

Notes:

1. Crystalline sodium niobate films can be formed on amorphous or crystalline materials.
2. High purity films are formed from relatively inexpensive, pure starting materials.
3. Stoichiometric chemical composition of the film is assured by control of substrate temperature and deposition time; the composition is virtually independent of deposition rates.
4. Inquiries concerning this process may be directed to:

Technology Utilization Officer
Manned Spacecraft Center
Houston, Texas 77058
Reference: B68-10163

Patent status:

This invention is owned by NASA and a patent application has been filed. Royalty-free, nonexclusive licenses for its commercial use will be granted by NASA. Inquiries concerning license rights should be made to NASA, Code GP, Washington, D.C. 20546.

Source: E. Z. Micka
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