Active and Passive Elec. Comp., 1992, Vol. 14, pp. 245–249
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Printed in the United Kingdom

EVAPORATION OF ADHERENT Al-FILMS ON SINGLE CRYSTALLINE $LiNbO_3$ SUBSTRATES

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$LiNbO_3$ is used as a single crystalline substrate material for the manufacturing of surface acoustic wave devices as frequency selective components. On the polished substrate wafer, Al with a film thickness of about 400 nm is evaporated in a high vacuum.

The difficulties of the inadequate adhesion of the structured Al-film were eliminated by systematical investigations. Films that were adherent to the total surface could be prepared by evaporating an intermediate film of Cr or SiO_x or, otherwise, a mixed film of Al with Si.

An ultrasonic test for adhesion strength was developed that results in an assessment of local and also large-area film adhesion.

I. INTRODUCTION

For electronics, Lithium niobate ($LiNbO_3$) is a very interesting material. Because of its crystal class 3m, $LiNbO_3$ is trigonal and polar. It is a ferroelectric material with large pyroelectric, piezoelectric, electro-optic, and photo-elastic coefficients. Therefore, static electric charges may occur on the substrate surface because of temperature variations. A detailed description of the crystal structure and the physical characteristics of the $LiNbO_3$ are given by WEIS and GAYLORD[1].

The growth of the $LiNbO_3$ single crystals can be made by the Czochralski method. To avoid areas of different polarity in a crystal, the polarity during the growing of a crystal above the Curie-temperature of 1140°C can be set by an electric field[2,3].

A field of application of $LiNbO_3$ is the manufacturing of surface acoustic wave devices. For that purpose a metal film is evaporated onto a polished single crystalline substrate wafer in high vacuum. An interdigital structure is then generated by means of photolithography. The interdigital structure consists of two "combs" of different shape and size. The structural widths of electrodes of the interdigital structure are less than 15 μm. The frequency range of the filters depends on the structural width; frequencies of more than 300 MHz require electrode widths of about 1 μm. The electrical contact is created by bonding of Al-wires to the structured metal film.

A good adhesion strength of the evaporated metal film on the vibrating surface is absolutely necessary for the function of the filters. Concerning the yz cut there were problems of adhesion bonds with the 38°-cut.

II. EXPERIMENT

In production, the polished $LiNbO_3$ wafers are given a short treatment with various chemicals. Therefore, tests of various pretments of the $LiNbO_3$ wafers were made. After a standard cleaning with acetone in an ultrasonic bath, the wafers were treated at 20°C and 50°C for 5 min and 20 min with the following chemicals:

1. NH_3 HF, 20% solvent
2. Chromium-sulphuric acid
3. NaOH, 20% solvent
4. H_3 PO_4, concentrated solvent
5. H_2 F_2, concentrated solvent

An assessment of the efficiency of the chemicals with respect to the surface of $LiNbO_3$ wafers resulted from electron microscopic photographs.

In another test series, standardized cleaned and pre-treated glass and $LiNbO_3$ substrates were coated under the same conditions with Al by evaporization. These tests should show differences between the adhesion of Al films to amorphous glass and crystalline $LiNbO_3$.

In general, the evaporation was done from W-boats on the substrates in vacuum with and without a glow discharge. For the manufacturing of a mixed film, Al and the additives Cu, Si and Ni were evaporated from separate boats. The thickness of the mixed film was 20 nm to 50 nm. Above it, pure Al was evaporated up to a thickness of 400 nm.

For the manufacturing of intermediate films, SiO_x as an insulator or Cr as a metal were first evaporated to a maximum thickness of 30 nm, followed by pure Al evaporated up to a total thickness of 400 nm.

To investigate the adhesion of the evaporated films a test method was developed. The samples are immersed into a solvent (e.g., acetone, xylene) and ultrasonically agitated[4]. The test method is sensitive to the extent of adherence of the film to the substrate. The test method can be incorporated into a production process because it is non-distructive of fully adherent films.

By this test method, non-adherent or not sufficiently adherent films are removed from the substrate and visually obscure as spots without a film. By the proper choice of solvent for sound-transmission, ultrasonic frequency and treatment time, the test method can be adapted to the adhesion bond necessary for the product[4]. For the assessment of the investigations, a screen of 1 mm × 1 mm was used to measure the area of substrate without a film.

III. RESULTS

From electron microscopic investigations, the surface of the $LiNbO_3$ wafer appears to be affected in different ways by different chemicals. There is no surface effect due to NH_3 HF. The largest etching pits, which are arranged according to the crystal symmetry, are produced by H_2 F_2. Fig. 1 shows the classification of the chemicals

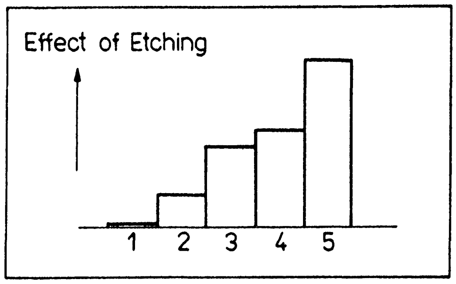

FIGURE 1 Classification of chemicals according to the etching effect on the $LiNbO_3$ wafer.
1—NH_3 HF (20% solvent),
2—Chromium-sulphuric acid
3—NaOH (concentrated solvent)
4—H_3 PO_4 (concentrated solvent)
5—H_2 F_2 (concentrated solvent).

according to the etching effect on the $LiNbO_3$ wafer. It was found that the treatments of the $LiNbO_3$ wafer with chromium-sulphuric acid and H_3 PO_4 will improve the adhesion of the Al film.

Fig. 2 shows clearly that the adhesion of the Al film is essentially worse on $LiNbO_3$ than on glass. After a test time of 10 min the Al film has been nearly 60% dissolved from the $LiNbO_3$, while it is still on the glass surface up to more than 90%.

For the film systems with an underlying film on the substrate, it was found that the adhesion of the Al film along with an AlSi mixed film is excellent as Fig. 3 shows. Al films with an underlying mixed film of AlCu have better adhesion than with AlNi mixed films or films of pure Al. To retain the filter function of the component, the thickness of the mixed film must not succeed 30 to 40 nm[5].

An excellent adhesion is also to be found with the Al films with an intermediate

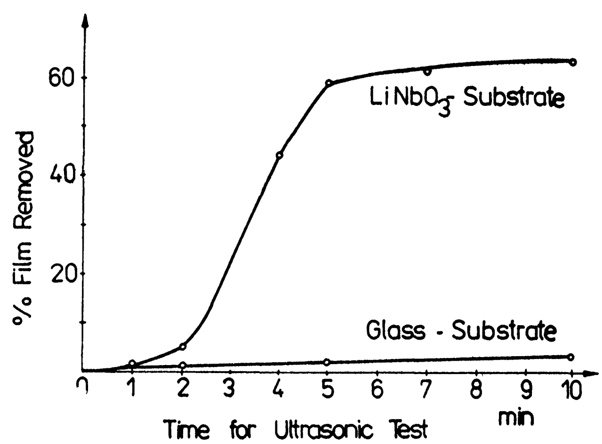

FIGURE 2 Area without layer after ultrasonic test of pure Al film on glass- and $LiNbO_3$-substrate in percent.

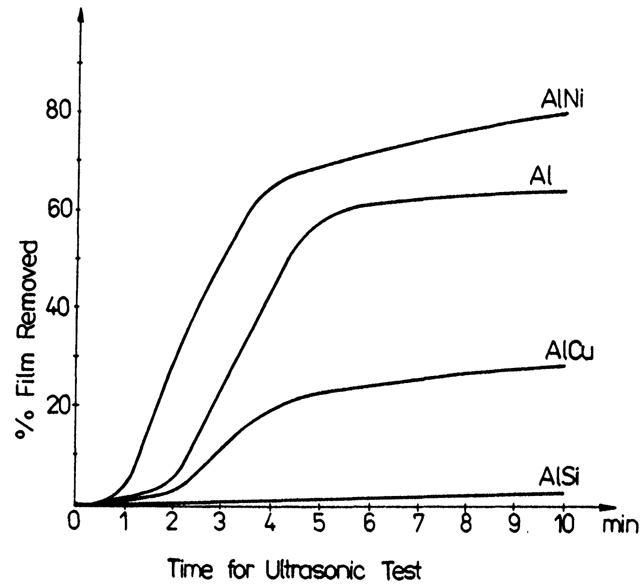

FIGURE 3 Area without layer after ultrasonic test of Al film and Al films with below mixed films of AlSi, AlCu and AlNi on $LiNbO_3$ in percent.

layer of the materials SiO_x or Cr. After a test time of 10 min there are no dissolutions of the Al film to be seen. A glow discharge is necessary before the evaporization of Al on the intermediate layer SiO_x[6]. The glow discharge is not necessary at the evaporization of Al on the intermediate Cr layer. The excellent adhesion of the Al film can be reached with very thin intermediate layers of less than 20 nm. With thicker intermediate layers, determined changes of the filter component can be reached according to the patents[7,8].

IV. CONCLUSION

The investigation of the adherent metallization of $LiNbO_3$ substrates with Al by evaporization show that by a suitable combination of substrate pretreatment, film system, and technological methods, total-area well-adhered films can be evaporated in a reproductible way. For composite film systems, Al films with underlying films of AlSi, SiO_x, or Cr are suitable. To get results of the adhesive bond of films, a test method—employing ultrasonic agitation, was developed. The test method is applicable in the manufacturing process, as well as for testing the adhesion of films that have been structured by a photolithographic method.

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