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Redeposition: a factor in ion-beam etching topography

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The design of high performance SAW* resonators and reflection pulse compression filters using grooves as the reflective arrays requires that the profiles of the grooves be rectangular and reproducible. The use of neutralized ion-beam technology has largely been adopted for etching these grooves in quartz and lithium niobate because of the precision and control which can be exercised in its use. The experimental work presented here asserts the major contribution that redeposition makes in the evolution of ion-beam etched topography of amorphous, polycrystalline and crystalline materials in a way which is directly useful to the determination of groove profiles. Data are also presented on the relationship between the incidence angle of the ion beam and the etch rate of some important SAW materials indicating the connection between this function and the etching characteristics of the materials.

1. Introduction

Ion etching using argon as the etchant is the preferred method of making SAW reflective structures for resonators and pulse compression filters used in radar, and such devices are currently being investigated both in the UK and USA [1, 2]. Generally, the grooves should have a mark/space ratio of unity to preserve phase coherence, and this calls for high resolution as the width of a groove at 1.5 GHz is 0.5 μm and for a square groove profile, to give a reflectivity amplitude proportional to groove depth.

An AZ.1350J positive resist in-contact mask has generally been found to give satisfactory resolution and profiles for all practical operating frequencies of SAW filters where groove depths do not exceed 1.5 μm. High-resolution photolithographic techniques have to be used to define the patterns and to give the squareness of mask profile necessary to result in square grooves.

The experimental work reported here has been carried out using an ion-beam machine in which a fairly well collimated beam of argon ions, composed of a matrix of overlapping beamlets which are 10° divergent, is accelerated on to the target with a potential of 600 V. The relatively low energy of the ions is necessary to avoid in-depth damage to the substrate so that removal of sputtered material may be considered a surface phenomenon. The surfaces of insulating substrates are prevented from acquiring a charge by the injection of electrons into the ion beam. Insulators and metals may be ion beam etched with equal resolution.

During experiments it was observed that groove profiles made under similar conditions of resist profile and thickness were dependent on the substrate material and that whereas single crystal quartz etched with an almost ideal reflective profile, lithium niobate, the other important piezoelectric substrate material, did not, but consistently resulted in a groove sidewall with an approximately 45° slope. Many factors influence the topographical formations in ion bombarded surfaces and have interested investigators over many years [3–6], but from an engineering point of view it is apparent that sputtering from surfaces during ion bombardment must play a part in the formation of features being evolved under masked

* SAW = Surface Acoustic Wave.

conditions, and that simultaneous etching and redeposition must take place. The experiments described in this paper are designed to show the magnitude of this factor and how it influences feature shapes. It introduces the concept that all etch rates are net etch rates unless the target is infinitely large and atomically smooth and flat. The experiments also show that with a fair degree of probability ion etching takes place at angles up to 90°.

It has been shown experimentally that the ion beam etching rate of crystalline materials is orientation-dependent and evidence of this dependence has been observed in the different etching characteristics of quartz and lithium niobate.

A further major factor in etching is the ion incidence angle dependence of etch rate. This is the result of incident ion yield as a function of angle and the cos law relationship of incident ion density falling on a plane surface as the angle changes. For an amorphous material this peaks at about 55° but considerable variations in the peak rate and the shape of the curve may be expected with crystalline and polycrystalline materials. As expected, an originally square profile feature will tend to assume a facet at an angle appropriate to the angle of fastest etch rate but it is shown that the powerful influence of redeposition may result in very much modified profiles which are material-dependent.

AZ.1350J resist has many characteristics which make it the preferred masking material [7] for the low profile etched grooves required in reflective surface wave filters. It has an homogeneous polycrystalline structure of small molecular size so that its ion etching properties are non-directional and it consequently behaves like an amorphous material. The etch rate is comparable to those of both quartz and lithium niobate which limit the depth of reflective grooves to about 1.5 μm because of mechanical and optical restrictions in abnormally thick resist films. This groove depth is, however, adequate for most SAW applications down to the lowest SAW frequencies of 10 MHz. A further advantage of AZ.1350J resist is that mask-making is a one step process and does not involve any chemical etching technique which might contaminate or damage the piezoelectric substrate. By adequately cooling the substrate, the resist remains in an easily removable condition after ion etching.

The true etch rate of AZ.1350J resist as a function of ion incidence angle was measured experimentally and is shown in Fig. 1. A polished glass cylinder was cut in half longitudinally and coated on the outside with an evaporated layer of chromium. A 1.5 μm thick layer of AZ.1350J resist was coated onto this surface and baked for 20 min at 80° C. The half cylinder was mounted on to water-cooled copper blocks, heat transfer from the glass cylinder to the copper being effected by silicone grease. Screens of low sputter-rate material were arranged relative to the cylinder so that redeposition from other parts of the system was reduced to a very low rate. Measurements of etch rates were made by counting fringes in the resist which has a refractive index of 1.55, almost the same as that of glass, hence the use of the chrome layer to increase contrast in the fringes. Incremental measurements of 350 Å can be made by this method. SEM and TEM pictures show that the etched surface of the resist has some craters near to 0° incidence angle, reducing in density as the angle is increased, but otherwise the surface is smooth and free from induced struc-

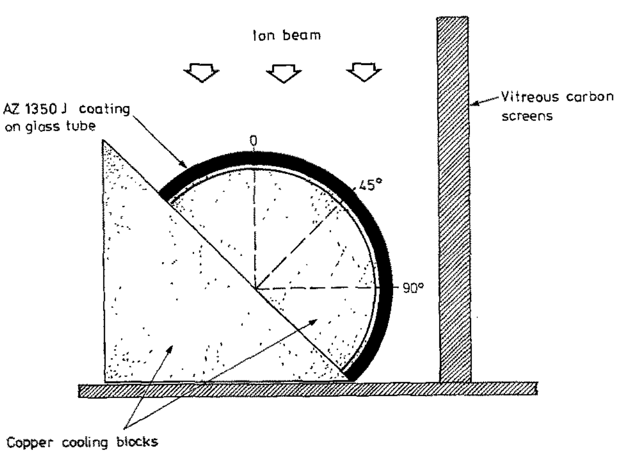


Figure 1 Experimental set up for measuring true etch rate of AZ.1350J resist. Etching conditions: Accelerating voltage 500 V, etching gas Ar^+, current density 0.70 mA cm^{-2}, chamber pressure 8×10^{-5} Torr.

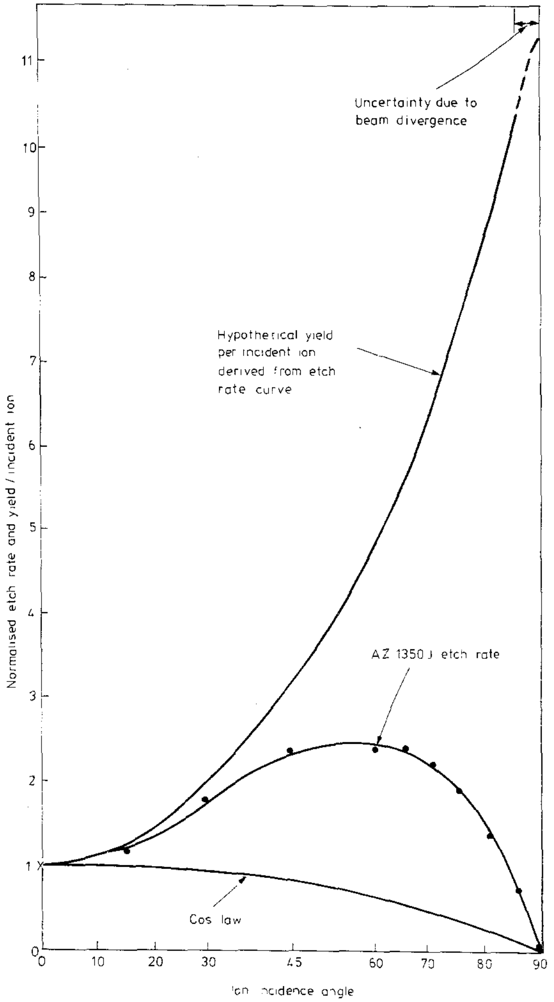


Figure 2 Experimentally measured true etch-rate versus incidence angle for AZ.1350J resist.

turing. The etch rate dependence follows the law expected from a polycrystalline material and etching rate goes to zero at 90°. (In the experiments, etching takes place beyond 90° due to the divergent gaussian distribution of the multiple beamlets of which the main beam is composed).

The experimentally measured etch rate as a function of incidence angle is shown in Fig. 2 together with the derived yield of sputtered atoms per incident argon ion. This latter curve shows that the yield of sputtered atoms removed from the target by a single incident ion continues to increase until it reaches a value of about 10 times the normal yield at an angle of 85°. The yield per incident ion then enters a grey area where it is reasonable to expect some reflection of the impinging ions due to inter-atomic collisions, and some deflection of the ions due to imperfect neutralization of the charge on the resist target surface, but from a practical point of view the ion etching continues to an incidence angle of 90°.

2. Net etch rate as a function of ion incidence angle for different SAW materials

The true etch rate of AZ.1350J resist as a function of incidence angle, together with its net etch rate curve relative to copper, is shown in Fig. 3. The four remaining curves for AZ.1350J resist, an evaporated crystalline film of aluminium and bulk crystals of quartz and $LiNbO_3$, are relevant SAW materials and their properties are of interest in the prediction of groove profiles. The experimental

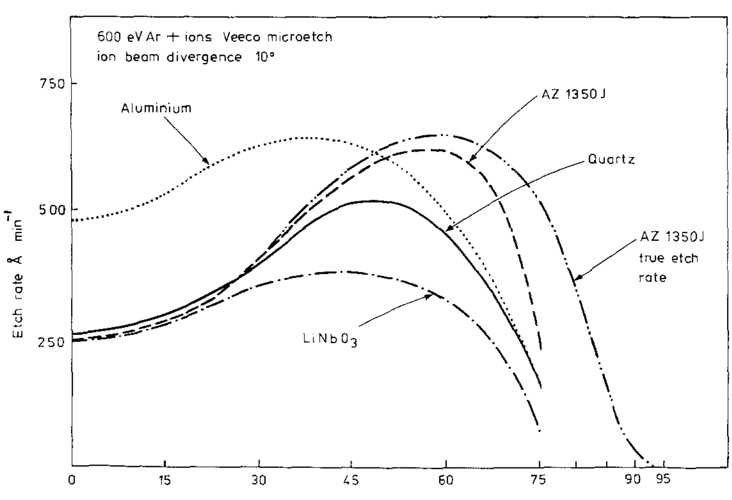


Figure 3 Experimentally measured true etch rate versus incidence angle for AZ.1350J resist and the net etch rate versus incidence angle of several other materials.

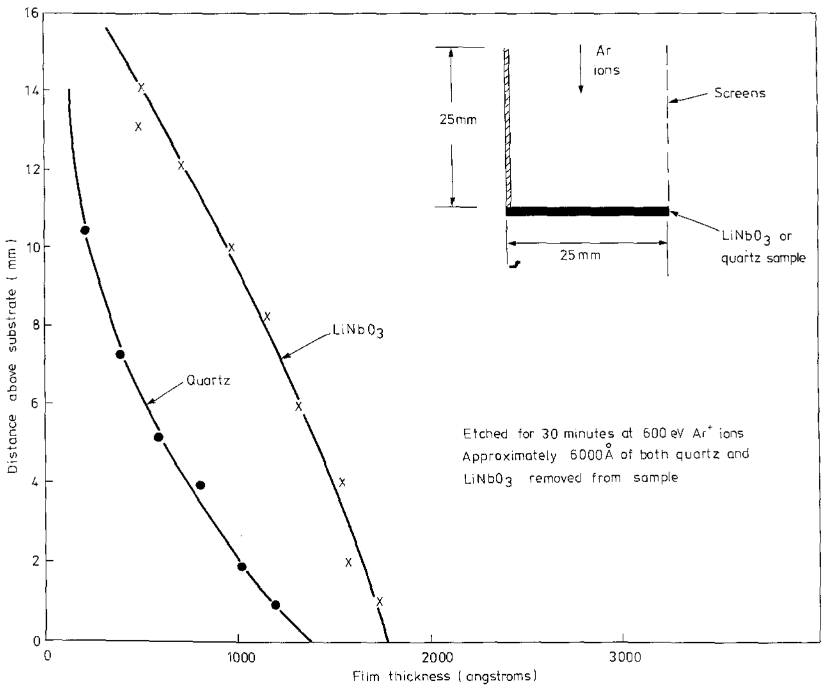


Figure 4 Redeposition from a groove base onto a vertical side wall.

measuring technique was to etch steps into plates (25 mm x 25 mm) and to measure step heights on a Talysurf. Screens were not used for these tests and therefore they are net etch rate curves relative to the redeposition of copper from the surrounding water-cooled target holder. It is possible to obtain true etch rates for these materials but they are included in this form to demonstrate the importance of net etch rates and because the relationships approximate more nearly to those found in practice. The shape of the curves indicates the etching characteristics of the material. $LiNbO_3$ for example has a poorly defined etch rate enhancement by a factor of 30% at about 45°, relative to that as 0°. This relates to the development of surface irregularities such as terraces and trenches. Its deviation from the amorphous case of AZ.1350J resist is probably due to the crystalline structure of the $LiNbO_3$. Quartz on the other hand has a well-defined etch rate enhancement at 45° relating to a smoother surface finish and steeper sidewalls in groove structures.

3. Redeposition as a mechanism of groove profile determination

Two experiments were carried out to determine redeposition rates in a typical groove configuration. The first of these was to determine the redeposition rate of quartz and $LiNbO_3$ on the vertical sidewall of a groove from the groove base (Fig. 4). A large scale model was made using 25 mm^2 plates of the piezoelectric materials. The redeposition rates were measured on a Talysurf after an in-contact resist mask had been removed from the measuring plate. The total material removed from the piezoelectric base plates was in both cases 6000 Å. The thicknesses of material deposited on the walls of the model from quartz and $LiNbO_3$ were significantly different and would result, from this factor alone, in the development of a groove wall angle of about 20° in quartz and 30° in $LiNbO_3$. This implies that the spatial distribution of sputtered quartz and lithium niobate are generally different, due to crystallographic and topographic effects. Some experiments were carried out to determine this point by measuring the thickness of a film of $LiNbO_3$ deposited on the inside surface of a hollow hemisphere with a circular hole at the top, through which an argon ion beam was projected onto a flat plate of lithium niobate. Difficulties were encountered in measuring the resultant deposition thickness on the inside surface of the hemisphere and the results were inconclusive. It was noted that in the case of Y cut lithium niobate the 2 μm deep depression in the surface due to the ion-beam etching was terraced into a regular,

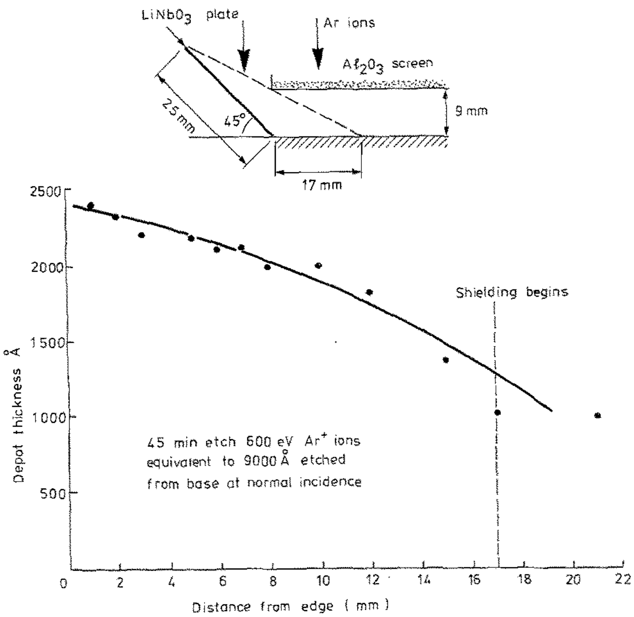


Figure 5 Redeposition from a 45° sloping side wall onto the base of a groove.

six-sided figure which would probably correspond to the structure of the crystal planes and directions, thus giving rise to different, directionally dependent, sputter rates. However, this is not the entire picture and the second experiment (Fig. 5) shows the redeposition of $LiNbO_3$ from a 45° slope side wall onto the base of a groove, etching of the base being prevented by using a suitable screen of alumina to intercept the incident ions. At the bottom of the slope, the net etch rate of the base is some 27% slower than the true etch rate for the same material ($LiNbO_3$). It is interesting to note that a trench may appear at the base of the wall of a groove or step due to shadowing in that zone during the evolution of the feature, because this zone has a faster net etch rate than the unshadowed areas. This may be seen in Fig. 6 which is an SEM picture of a typical edge of a $LiNbO_3$ groove, etched to a depth of 8000 Å at normal incidence. About 2000 Å of resist remains on top of the $LiNbO_3$ and this has a steep slope. This sloping edge is also subject to direct etching, but, due to the rather flat etch rate as a function of ion incidence angle, it is able to reach an equilibrium shape

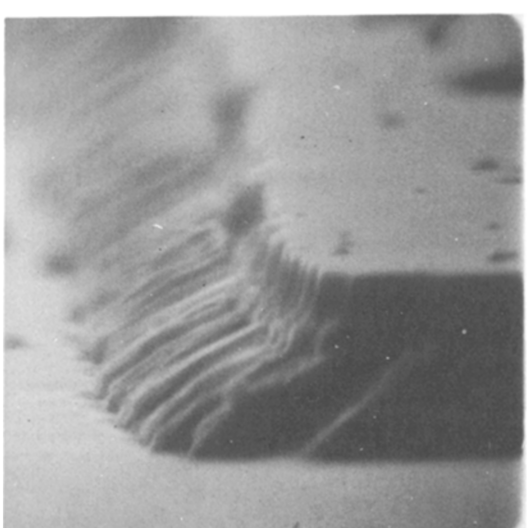


Figure 6 Typical edge formation in $LiNbO_3$ using AZ.1350J resist mask, SEM × 19.5 K.

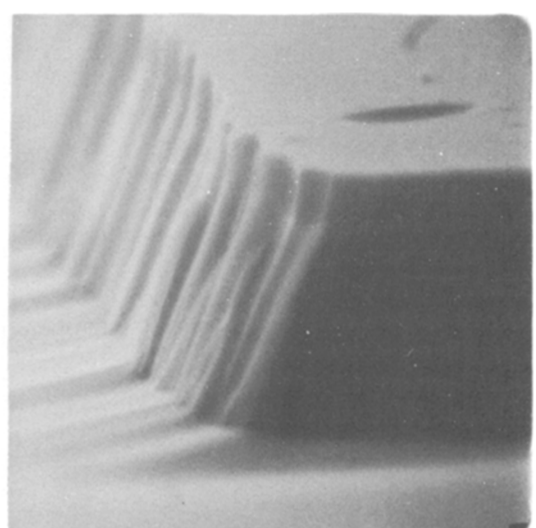


Figure 7 Typical edge formation in single crystal quartz using AZ.1350J resist mask SEM × 18 K.

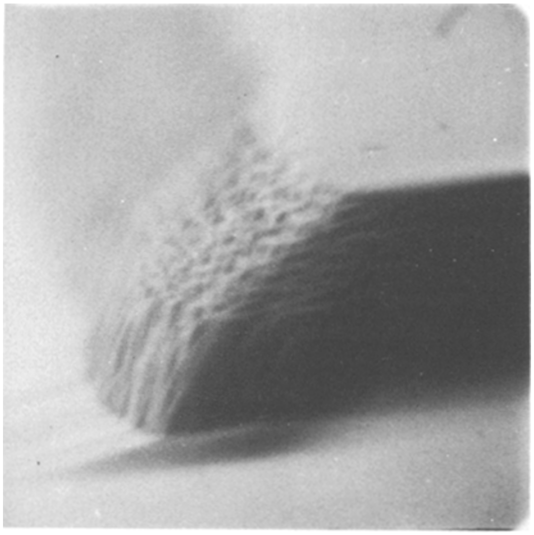


Figure 8 Terrace formation in AZ.1350J resist resulting from a poorly defined photolithographic mask after etching.

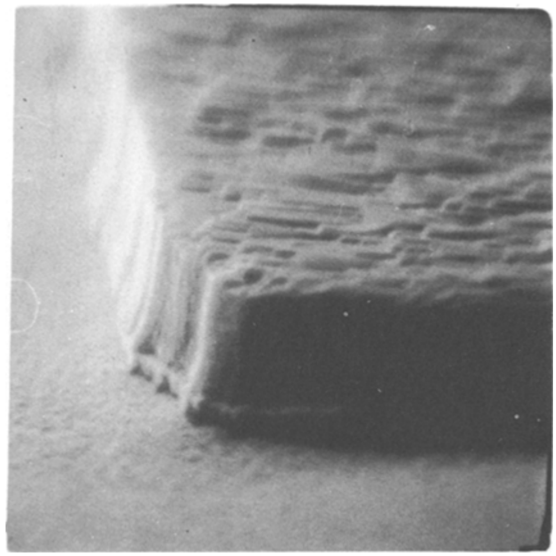


Figure 9 Terrace formation in AZ.1350J resist resulting from 60° incidence angle etching from the left. The irregularities of the terraces is caused by the irregularity of the photolithographically defined edge from which they form.

over a large range of angles. The straight slope is, of course, a stable condition under true etch rate conditions.

Quartz, as seen by its etch rate versus incidence angle relationship, will have a more precisely defined edge, and this can be seen in the edge of a quartz groove etched 9000 Å deep and with an overlying residual resist film of 4000 Å (Fig. 7). In this particular configuration the redeposition of material from the groove base has not only strongly influenced the slope and curvature of the wall, but has totally inhibited the formation of facets in that edge which would be attributed to incidence-angle/etch-rate considerations. Redeposition onto the base from the sidewalls may be seen as a low ramp at the intersection of the base and the wall. These fortuitous characteristics of AZ.1350J resist and single crystal quartz combine to make high-resolution etched grooves in quartz easy to achieve.

Other single materials and combinations of materials will have their own peculiar etch properties and it should be possible, in theory at least, to explain the etching properties of single materials and in some cases to optimise the characteristics of multi-material systems by (a) measuring the etch rate as a function of incidence angle, (b) the sputter rate and distribution of the redepositioning material as dependent on incidence angle and (c) the orientation dependence of these

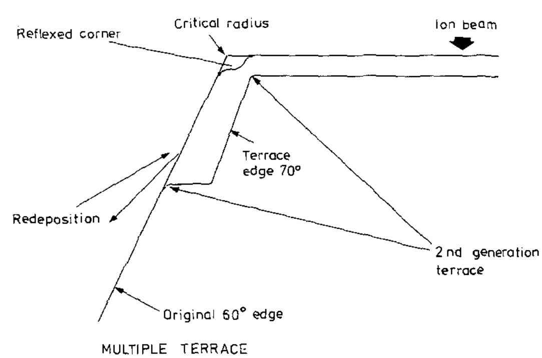
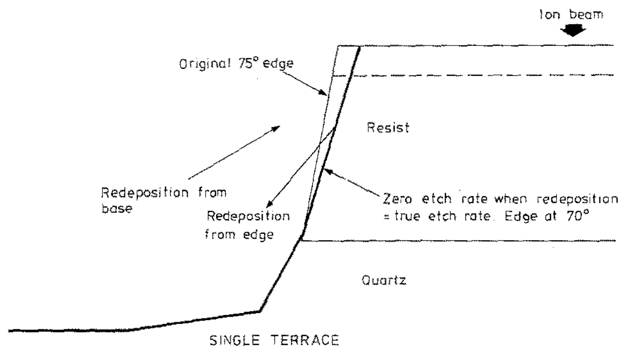


Figure 10 The equilibrium condition is shown diagramatically for multiple terracing of AZ.1350J resist overlaying single crystal quartz.

parameters on the crystalline or polycrystalline structure of the component parts. There may well be other effects such as reflected ions at angles close to 90°, flux enhancement, and reduction in the mean free path because of sputtered substrate atoms, but from an engineering topographical point of view the factors discussed are of major importance.

Terraced formations in resist occur under certain conditions. Because resist is polycrystalline of carefully controlled chain size of about 5 molecules length, the terraces cannot be due to crystallographic effects for dimensional reasons. Neither

Figure 11 Evolution of a single terrace in AZ.1350J resist overlaying quartz.



are they due to standing wave patterns set up during photolithography of about 1290 Å periodicity above the reflecting interface between resist and substrate. The terraces (Fig. 8) are perhaps not important in themselves but they help in the understanding of equilibrium conditions set up in the evolution of ion beam etched topographies generally.

It is likely that in a polycrystalline material terraces are initiated by a small radius corner being sputtered off to form a reflexed surface, which is an unstable condition until a right-angled reflex corner is formed with the correct angles, this being a stable condition. There is only one other stable condition and that is a perfectly flat plane at normal incidence to the ion beam. Fig. 9 shows

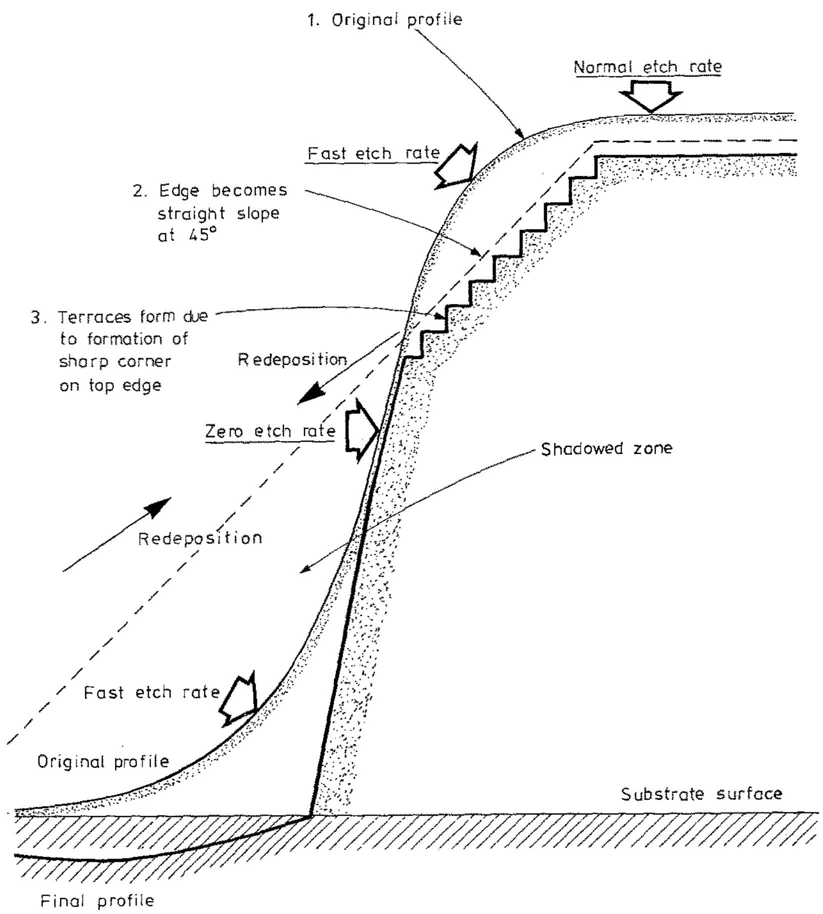


Figure 12 The evolution of multiple terraces on a sloping resist edge overlaying single crystal quartz.

multiple terraces forming from the leading edge, which has an angle of 60° to the ion beam and thus is not in equilibrium with the redeposition rate. The faces of the new terraces form at the angle of equilibrium and are thus geometrically stable. The single terrace seen in Fig. 7 is also stable and depends on the correct redeposition and true etch-rate ratio to determine the exact angle that the edge makes.

The evolution of multiple and single terraces are shown diagramatically in Figs. 10 to 12. The material depicted is AZ.1350J resist on top of quartz and it should be borne in mind that other combinations of materials will have different profiles.

4. Surface topography after ion beam etching at 45°

Whereas normal-angle ion-beam etching always has a tendency to level the surface due to the enhanced etch rate on sloping surfaces between 10° and 70° this is not necessarily the case when the incidence angle of the ion beam is not normal to the surface. The crystalline structure will initiate changes in etching rate due to its orientation dependence. The crystallites will then be brought into low relief simultaneously with a rounding off of the structure due to modified etching angles and re-deposition. Fig. 13 shows this diagramatically. Transmission electron micrographs show that a polished quartz surface remains smooth and featureless after angled etching, but lithium niobate (Fig. 14) and evaporation deposited aluminium (Fig. 15) show their characteristic crystalline structures. These surface topographies are related to the specific etch-rate versus incidence angle plots in Fig. 4 and together give the etching properties of a particular material. For example, the 1 μm grain size of the fine-grain aluminium film could be expected to produce ion beam feature edges with a sub-μm RMS roughness distributed with a random mean periodicity of 1 μm. It is fundamental character-

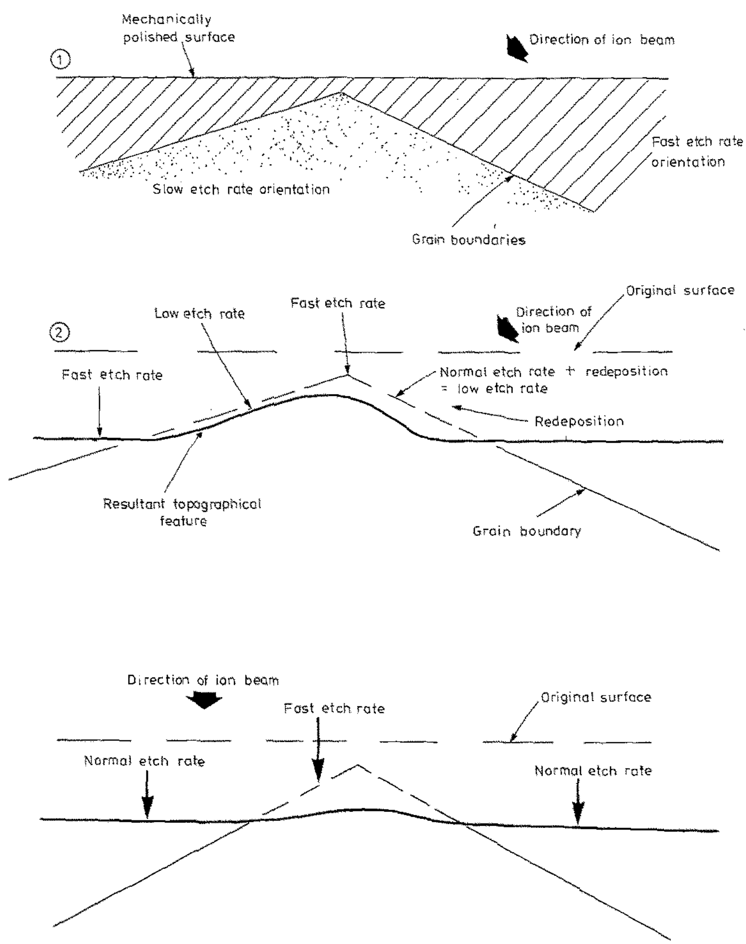


Figure 13 Evolution of topography of a polished polycrystaline orientation dependent etch-rate material at ion incidence angles of 45°, (a) and (b), and 0°, (c).

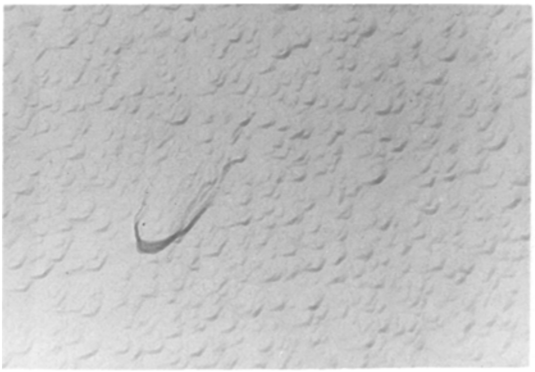


Figure 14 Topography of mechanically polished $LiNbO_3$ after ion beam etching at an incidence angle of 45°. TEM Pt/C. X 50 000.

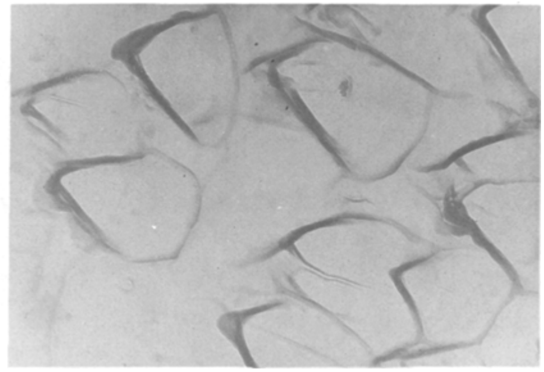


Figure 15 Topography of a fine-grain evaporated aluminium film on polished quartz after ion beam etching at an incidence angle of 45°. TEM Pt/C. X 50 000.

istics of this nature which help to determine the choice and etching properties of materials being considered for a particular application.

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References

1. R. C. M. LI, R. C. WILLIAMSON, D. C. FLANDERS and J. A. ALUSOW, "On the Performance and Limitations of Surface-Wave Resonators Using Grooved Reflectors", Proceedings of IEEE Ultrasonics Symposium (1974) 245.
2. R. C. WILLIAMSON and H. I. SMITH, *IEEE Trans. Microwave Theory and Techniques*, **MTT21** (1973).
3. A. R. BAYLEY, *J. Mater. Sci.* 7 (1972) 404.
4. D. J. BARBER, F. C. FRANKS, M. MOSS, J. W. STEEDS and I. S. T. TSONG, *J. Mater. Sci.* 8 (1973) 1030.
5. PER GLOERSON, *J. Vac. Sci. Tech.* **12** (1975).
6. H. DIRMINGER and H. LUTHJE, *Philips Tech. Review* **35** (1975) 199.

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