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Abstract:

The bulk micromachining on (010), (110) and (111)A GaAs substrates in the 1 H_2SO_4:1 H_2O_2:8 H_2O system is investigated. Focus is placed on anisotropy of 3D etching shapes with a special emphasis on convex and concave undercuts which are of prime importance in the wet micromachining of mechanical structures. Etched structures exhibit curved contours and more and less rounded sidewalls showing that the anisotropy is of type 2. This anisotropy can be conveniently described by a kinematic and tensorial model. Hence, a database composed of dissolution constants is further determined from experiments. A self-elaborated simulator which works with the proposed database is used to derive theoretical 3D shapes. Simulated shapes agree well with observed shapes of microstructures. The successful simulations open up two important applications for MEMS: CAD of mask patterns and meshing of simulated shapes for FEM simulation tools.

1. Introduction

Recently new mechanical sensors based on piezoresistive and piezoelectric effects in GaAs have been investigated [1,2]. The possibility of fabricating mechanical devices in GaAs by bulk micromachining sustains the interest in these new sensors applications. Different acidic etching baths with various compositions have been studied. They include H_2SO_4:H_2O_2:H_2O [3-8], H_3PO_4:H_2O_2:H_2O [9,10] and HF:H_2O_2:H_2O [11] where H_2O_2 is the oxidant. The H_2SO_4:H_2O_2:H_2O system which at some compositions produces polished surfaces remains the most commonly used etchant. Most of these works [4,5,10] have focused on the etched (010) surface and especially on grooves aligned along <110> directions because this surface orientation offers interesting MEMS applications. Some works [3,11] have reported results on localized etchings on other (hhl) substrates covered with mask strips. These strips are aligned along two perpendicular preferential directions with one direction coinciding with a <110> direction. A previously published systematic work on GaAs etching has been concerned [7] with the 1 H_2SO_4:8 H_2O_2:1 H_2O system. To our knowledge up to now, no complete study on 3D structures etched on substrates with various orientations has been reported in literature for the etchant 1 H_2SO_4:1 H_2O_2:8 H_2O. Moreover, there is a lack on information on slow etch planes for this specific etching bath. Only the (111)A plane is found without ambiguity to be a slow etch plane for other etchants [3,7,11]. A great variety of other orientations was also cited in literature, but these orientations remain uncertain.

The etchant used in this paper is thus the 1 H_2SO_4:1 H_2O_2:8 H_2O system. The aim of this study is to give more complete information on the wet micromachining on (010), (110) and (111)A substrates. Hence, we pay attention to the micromachining of 3D structures and on convex and concave undercuts in order to characterize the anisotropy of the wet etching. Then 3D etching shapes are analysed in the framework of the kinematic and tensorial model (KT model) [12,13] which applies for a dissolution process governed by orientation. A database for the micromachining of GaAs crystal in the 1 H_2SO_4:1 H_2O_2:8 H_2O system is determined. Finally, numerical simulations of 3D etching shapes are derived using a self-elaborated simulator based on the KT model.

2. Experiments

2.1. Experimental details

Polished GaAs substrates with orientation (010), (110) and {111}A were used for etching experiments. Prior to etching these wafers were coated with a silicon dioxide film. This film was patterned with various test masks using stand photolithographic process. For each orientation the test mask is composed on the one hand, of circular patterns for the micromachining of membranes and of mesa and on the other hand, of rectangular patterns aligned along different crystallographic directions in order to evaluate concave and convex undercuts. Such test masks allow us to evaluate the degree of anisotropy. The etchant was the 1 H_2SO_4:1 H_2O_2:8 H_2O system. Etchings were performed at room temperature and under room light. After etching wafers were rinsed in desionised water. The resist was removed in acetone. The depth of etch was measured with a computer based 3D profilometer. Etched structures were observed by scanning electron microscopy (SEM).

2.2. Experimental results

At this point let us recall that depending on crystal the chemical etching produces etching shapes with anisotropy either of type 1 or of type 2. Type 1 and type 2 of anisotropy characterize the chemical etching silicon structures [14-18] and of quartz structures [19,20] respectively. In type 1, etched structures are bounded by a limited number of crystallographic planes. These planes intersect the upper surface in successive straight elements. In contrast in type 2 sidewalls of micromachined structures are bounded by curved regions. As a result the top contour is composed of rounded elements even after a prolonged etching. To determine the type of anisotropy it is convenient to fabricate membranes and mesa on differently oriented GaAs substrates patterned with circular masks. So, in the first step of this study we consider these “circular” structures. The analysis is concentrated firstly, on changes in starting circular contour of etched structures and secondly, on shapes of shoulders limiting these structures. At this point it is important to recall that the GaAs crystal belongs to the non-centro symmetric $\bar{4}3m$ class. This class is characterized by three symmetry operators:

i) <100> axes are 4-fold rotation-inversion axes.

ii) <111> axes are 3-fold rotation axes.

iii) {110} planes act as mirror planes.

Figure 1 shows SEM images of membranes and mesa micromachined on (010), (110) and {111}A surfaces. Etching shapes reflect the symmetry of the GaAs crystal. Obviously, the {111}A membrane and mesa satisfy the 3-fold symmetry attached to the <111>A direction. The mirror symmetry connected with {110} planes can be easily detected on the various SEM images etched on (010), (110) and {111}A surfaces. Moreover it appears that the out-of-roundness of lower and of upper contours of structures produced by the anisotropic dissolution is not marked. Effectively, contour shapes do not deviate very much from the starting circular contour of masks. Never the etching of “circular” mesas in the 1 H_2SO_4:1 H_2O_2:8 H_2O system produces significant convex undercutting as observed for silicon crystal [14,17,18]. This behaviour which is typical of anisotropy of type 2 has been also observed [8] in the case of GaAs etching with the 1 H_2SO_4:8 H_2O_2:1 H_2O. We have now to determine if some crystallographic planes limit some etched structures. Examination of undercutting at corners of rectangular membranes (Fig. 2) and mesa (Fig. 3) provides interesting information. Let us recall that the formation of facets at a concave corner is due to the divergence of trajectories for moving surface elements in the vicinity of a protuberance of the dissolution slowness surface. Only the presence of a peaky protuberance within the angular sector of the concave corner leads to the development of a perfectly planar facet. A smooth maximum gives rise to a curved facet. Turning attention to Fig. 2, it appears that square corners of rectangular membranes have curved concave undercuts. Only few corners corresponding to specific directions of alignment are free of undercutting (Fig. 2d). Moreover the formation of curved facets at corners is restricted to few orientations only. Figure 2a shows a rather curved facet (f_A) with an outward normal whose mean orientation corresponds to an {111}A plane. This plane cuts the (010) substrate along a round intersect. This observation agrees with previous works [3-5] that concluded that {111}A surfaces are principal slow etching surfaces. The inspection of convex “square” corners (Fig. 3) leads to quite similar observations. When present, the convex undercutting affecting mesa is always moderate. Magnified SEM images (3b, 3c and 3d) indicate that at corners the successive intersects of sidewalls with the upper surface do not make sharp angles. In fact, at convex corners upper contours are also partly curved (see Figs. 3b and 3c for example). Since the importance of convex undercutting is associated with features of valleys in the dissolution slowness surface, we can conclude that deep valleys are absent. Finally, the fact that localized etching at “square” corners give rise to rather identical behaviour (curved upper contours) for concave and convex undercuts calls also for an anisotropy of type 2.

3. Database and simulations

3.1. Database

As outlined in previous section, the anisotropy of type 2 produces micromachined structures bounded by rounded sidewalls that intersect upper and lower surfaces along curved contour elements. So it is obvious that numerous surface elements participate to final 3D etching shapes. Consequently simulations must be able to carry out calculations for successive surface elements whose orientations vary by one degree or less. This condition implies a precise knowledge of variations in etch rate $R_{(hkl)}$ with orientation (hkl). This requirement may be easily fulfilled provided we use a simulator that is based on an analytical model.

At this point let us recall that the wet anisotropic etching is frequently described by kinematic models [12,13,21,22]. With these models geometrical constructions and numerical simulations of etching shape require to establish an accurate database on etch rate $R_{(hkl)}$ [21,22] or on dissolution slowness $L_{(hkl)}=1/R_{(hkl)}$ [12,13]. Two different methods can be adopted to determine the database. The first one consists to have coherent and experimental data on etch rates. In particular an elegant way [23,24] for determining a rather complete orientation dependence of the etch rate is to etch a hemispherical GaAs crystal. On this hemisphere all possible surface orientations are exposed to etchant. But care must be taken to kept the maximum etch depth small with respect to hemisphere radius in order to avoid errors in etch rate measurements. The second method that is preferred here because it needs less data is based on a kinematic and tensorial model (KT model [12,13]). In this model a dissolution slowness vector $\vec{L}_{(hkl)}$ (magnitude $L_{(hkl)}=1/R_{(hkl)}$) is associated to a surface element of orientation (hkl). When the orientation varies the vector $\vec{L}_{(hkl)}$ describes in space a representative surface called the dissolution slowness surface that characterizes the anisotropy of the chemical etching. The analytical equation for the dissolution slowness surface of the GaAs crystal is expressed on the one hand, in terms of dissolution constants that are components of tensors of rank N_R and on the other hand, in terms of Cartesian components (n_1,n_2, n_3) of the unit inward normal to the surface element. Consequently, with the KT model we can follow continuously changes in the dissolution slowness with orientation. The number of independent dissolution constants is reduced by the crystal symmetry. Applied to the GaAs crystal [25] the reduction gives for tensors of ranks 9 to 12 the dissolution constants listed in table 1. Note that in this table we adopt the notation $i(N_i)j(N_j)k(N_k)$ where N_i, Nj and N_k are the numbers of subscripts i, j and k respectively. Previous works [14,16] showed that tensors of relatively high ranks (N_R>8) are needed to reproduce the anisotropy observed for quartz and silicon crystals. Owing to this remark the database for the GaAs crystal contains dissolution constants that belong to very tensors of relatively high rank. At this time we retain for the 1 H_2SO_4: 1 H_2O_2: 8 H_2O system a set of dissolution constants (15 constants with non-zero value the constant of rank zero D_o included) for which the anisotropy ratio

L{111}A/L{111}B is found to be close to 2.4 for the two types A and B of {111} planes. Table 2 gives for each tensor rank the number N_o of constants with non-zero value and the values of constants with smallest and largest absolute values. Moreover Fig. 4 shows polar plots of the dissolution slowness corresponding to (100), (110) and (111) cross sections. We observe that:

i) The more pronounced protuberances are for the {111}A surfaces (Fig. 4B). Moreover these protuberances are not very peaky and consequently, divergence of trajectories affects a relatively wide angular sector (more than 30°).

ii) Minima in the dissolution slowness are relatively flat. This explains the rounded convex undercutting observed on SEM images of mesas.

iii) The two faces A and B of {111} and {112} plates (Figs. 4B and 4C) dissolve with different dissolution slowness. This remark applies for all {hhℓ} plates until the Miller index h or ℓ takes a non-zero value.

3.2. Simulations

The major interest of the KT model is that the Cartesian components of the displacement $\vec{P}$ of a moving surface element can be calculated from the analytical equation of the dissolution slowness surface. Consequently, this model allows us to construct numerically final shapes of structures micromachined on substrates with any orientation through masks with any shape. A simulator TENSOSIM [14,26,27] whose flow chart is given in Fig. 5 was developed. The database of the simulator is composed of the selected dissolution constants. Of course, a good adjustment of dissolution constants must be met to obtain satisfactory agreement between theoretical and experimental etching shapes. In order to undertake a comprehensive discussion simulations of small holes, of “circular” membranes and mesa and of concave and convex undercuts are performed. Figures 6 to 11 show an almost complete overall picture of these simulations. Let us first put attention on experimental and theoretical etching shapes (Fig. 6) for membranes and mesas micromachined with circular masks of finite radius (250 µm for (110) and {111}A structures). Theoretical shapes for (010) and (110) membranes give evidence for the action of {111}A protuberances which are at the origin:

i) Of two curved facets f_A with outward normal that can be easily depicted on the theoretical (010) membrane. Note that {111} protuberances produce also the two inward edges (IE) of experimental and theoretical (010) membranes.

ii) A {111}A protuberance is also responsible of the generation of the curved outward region (OR) which bounds (110) membranes (Figs. 1b and 6b).

Clearly theoretical etching shapes for membranes agree well with experimental shapes. If we compare SEM images of mesas (Fig. 1) with corresponding simulations (Fig. 6) we can adopt a similar conclusion: theoretical shapes look as experimental shapes. In particular for mesas the agreement concerns:

i) The formation two inward regions (IR) and two outward regions (OR) for the (010) mesa (Fig. 6A). The development of acute (AE) and obtuse (OE) edges at 90° of each other may be seen as the resulting effect of the four {111}A protuberance because firstly, beneath the mask we are concerned with a potentially concave profile and secondly, the anisotropy is not so great.

ii) The obtuse angled shoulder (OS) which limits the (110) mesa (Fig. 6B) on an angular sector of about 180°.

iii) The presence of three equivalent and slightly inclined shoulders (S) which joint the reference {111}A surface (Fig. 6C).

iv) General shapes of mesas that are essentially composed of rounded shoulders that cut curved contours on the top surface.

At this point it should be remarked that for an anisotropy of type 2 it may be also of interest to investigate theoretical changes in shape of the starting circular top contour induced by prolonged etching (Fig. 7). We observe that theoretical top contours shown in Fig. 7A resemble to experimental contours. Moreover contours derived for small holes are also composed of slightly curved elements aligned along <110> directions as expected for a chemical etching governed by an anisotropy of type 2 where the slow etching planes {111}A play a role.

It is clear that with the proposed database the simulator is capable to generate realistic 3D etching shapes for “circular” membranes and mesas. So let us outline that it is of prime importance to study theoretical shapes of concave and convex undercuts. For some orientations micromachining produces mechanical structures non non-symmetric shape. In the case of a resonant structure the mechanical performances can be significantly degraded and the asymmetry can generate spurious resonant frequencies. Hence in the following we report some results on the undercutting that can affect square corners of membranes and mesas. Figure 8 shows experimental and theoretical concave undercuts formed at various corners on a (110) substrate. Simulations yield theoretical undercuts at corners and lateral shoulders (S1, S3, S*1, S*2) that accurately represent observed concave corners. A similar conclusion can be drawn for concave undercuts produced on (010) and (111)A substrates (Fig. 9). In Fig. 9A the inward shoulder (IS) can be observed on SEM image and on corresponding simulation. In Fig. 9B we depict the presence on the SEM image of a shoulder slightly inclined (S2) that is effectively generated by the simulator. SEM images and on corresponding simulations of convex “square” corners produced on (110) substrate are shown in Fig. 10. As above mentioned some corners are free of undercutting. It is the case for corners a1 and a2 and for the related theoretical shapes (Fig. 10A). However, we can distinguish on magnified representations (Figs. 10B and 10b) a very moderate bunching which affects the corner a2. For active undercuts the simulator provides satisfactory representations (Figs. 10C and 10c for example) on the one hand of the convex undercutting and on the other hand of the accompanying bunching. Comparison of experimental and theoretical convex undercuts produced on other substrates (Fig. 11) speaks also for fitly simulations with an agreement which covers:

i) The shape of the convex undercut for the {111}A corner (Fig. 11B).

ii) The development of inward (IS) and outward (OS) shoulders (Fig. 11A).

4. Discussion and conclusion

Clearly the micromachining of GaAs in the 1 H_2SO_4:1 H_2O_2:8 H_2O system produces structures whose shape depends on orientation. But the dependence differs markedly to that observed for silicon crystal. GaAs structures exhibit curved limiting shoulders and more and less rounded concave undercuts. Moreover a very moderate undercutting, where frequently a curved top contour can be detected, affects convex regions of structures. Hence for this etchant the anisotropy can be classified as anisotropy of type 2. As a consequence an analytical model such as the KT model can account for the observed anisotropy. Dissolution constants that constitute the database of a self-elaborated simulator are thus determined. With this database the dissolution slowness exhibits the more pronounced protuberances for {111}A surfaces. But we are concerned with a weak anisotropy and with relatively wide protuberances. It is the reason why for totally concave structures the simulator generates limiting rounded facets with mean {111}A orientations. These predictions agree well with experimental observations made in particular for structures micromachined on (010) and (110) substrates and with previous studies [3-5] on localized etching at mask edges where in {110} sections revealed planes were identified as {111}A planes. For the different substrates investigated here numerical simulations yield theoretical etching shapes that are quite consistent with observed 3D shapes of etched structures, mixed concave/convex shapes included. Because in the case of mixed concave/convex shape maxima and minima in the dissolution slowness play simultaneously a role in the formation of the final etching shape it is more difficult to simulate mixed concave/convex shapes than completely concave or completely convex shapes. Such predictions require a precise adjustment of amplitude of successive minima and maxima. So, a cause of deviation is frequently the use of a not entirely accurate database. Convex corners constitute mixed concave/convex shapes because the top contour is convex and cross-sectional profiles are concave. In the present study the simulator predicts the convex undercutting with a sufficient accuracy. The database can be once more adjusted but clearly here we demonstrate on the one hand, the satisfying validity of the proposed database and on the other hand, the quite successful applicability of the simulation tool to GaAs micromachining. Two different possible applications can now be contemplated. The CAD of mask patterns is obviously straightforward. Because the anisotropy is weak, the possible adjustment of the mask dimensions with etching depth and with lateral under-etches beneath the mask constitutes a very interesting way to control the final dimensions of mechanical structures. A more sophisticated application is the design of mask compensation patterns even if for GaAs the micromachining produces a convex undercutting only for some specific orientations. Moreover, it may be interesting for anisotropy of type 2 to extend the design of compensation patterns to concave corners for which significant rounded undercuts are observed. The second important application consists to use predicted 3D etching shapes of mechanical structures in FEM simulation tools. Actually the carrying out of realistic meshings for micromachined structures remains a challenge. At this point, let us note that it is effectively essential to work with theoretical etching shapes. Consider a (114) wafer and the micromachining of "rectangular" cantilevers located at the four corner of a square. The starting mask pattern is shown in Fig. 12. Possibility of piezoelectric activation of new resonant sensors [28] motivates the choice of a (114) crystallographic orientation. Figure 13 shows just at the opening of the structure final shapes of the four cantilevers as viewed on the two faces. The asymmetry of shapes can be easily depicted on this figure with in particular different sidewalls formed on the two faces of the (114) wafer. In this condition the simulator offers a suitable opportunity for introducing this asymmetry in the meshing of GaAs mechanical structures.

In conclusion CAD of mask patterns including compensation masks and realistic meshings of mechanical structures are extensions of simulations that open up new interesting possibilities for mechanical GaAs sensors. These possibilities will be explored in future works.

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CAPTIONS TO FIGURES

Figure 1: SEM images of “circular” membranes (a,b,c) and mesas (A,B,C).(a,A), (b,B) and (c,C) are for (010), (110) and {111}A substrates respectively. OR and IR denote regions (R) with outward (O) and inward (I) normal respectively.

Figure 2: SEM images of concave undercuts at square corners. (a), (b) and (c,d) are for (010), (110) and {111}A substrates respectively.

Figure 3: SEM images of convex undercuts at square corners. (a,b) and (c,d) are for (110), and {111}A substrates respectively.

Figure 4: Cross sections of the dissolution slowness surface. (A), (B) and (C) represent polar plots for (010), (110) and (111) cross sections respectively.

Figure 5: The flow chart of the simulator TENSOSIM.

Figure 6: Simulations of “circular” membranes (a,b,c) and of “circular” mesas (A,B,C). (a,A), (b,B) and (d, D) are for (010), (110) and {111}A substrates respectively.

Figure 7: In (A) theoretical shapes for upper contours of membranes, small holes and mesas etched on various substrates. In (B) SEM images of small holes etched on (010), (110) and {111}A surfaces.

Figure 8: Simulations (A,B) of concave undercuts (a,b) formed at square corners of membranes etched on a (110) substrate.

Figure 9: Simulations (A,B) of concave undercuts (a,b) formed at square corners of membranes etched on various substrates. (a,A) and (b,B) are for (010) and {111}A substrates respectively.

Figure 10: Simulations (A,B,C) of convex undercuts (a,b,c) formed at square corners of mesas etched on a (110) substrate.

Figure 11: Simulations (A,B) of convex undercuts (a,b) formed at square corners of mesas etched on various substrates. (a,A) and (b,B) are for (010) and {111}A substrates respectively.

Figure 12: Shape of starting mask.

Figure 13: Final theoretical shapes (just at the opening of the structure) of cantilevers micromachined on two faces of a (114) wafer. To make the comparison more easy shapes are derived with similar durations of etching for the two faces. This signifies that starting etching times for the two faces differ.

N_R	INDEPENDENT CONSTANTS $D_{i(ni)j(nj)k(nk)}$
9	$D_{1(7)2(1)3(1)}$, $D_{1(5)2(3)3(1)}$, $D_{1(3)2(3)3(3)}$
10	$D_{1(10)}$, $D_{1(8)2(2)}$, $D_{1(6)2(4)}$, $D_{1(6)2(2)3(2)}$, $D_{1(4)2(4)3(2)}$
11	$D_{1(9)2(1)3(1)}$, $D_{1(7)2(3)3(1)}$, $D_{1(5)2(5)3(1)}$, $D_{1(5)2(3)3(3)}$
12	$D_{1(12)}$, $D_{1(10)2(2)}$, $D_{1(8)2(4)}$, $D_{1(6)1(6)}$, $D_{1(8)2(2)3(2)}$, $D_{1(6)2(4)3(2)}$, $D_{1(4)2(4)3(4)}$

Table 1. The independent dissolution constants for tensors of rank N_R. Subscripts ni, nj and nk in brackets denote the number of subscripts i, j and k. For example, read D_{113333} for $D_{1(2)3(4)}$.

N_R	N_o	Smallest D*	Largest D*
9	3	− 0.65	1.34
10	4	0.31	1.06
11	3	− 0.14	1.09
12	4	0.08	1.96

Table 2. Number N_o of constants for each tensor rank. Values (at room temperature) of dissolution constants with smallest and largest absolute values D* (in min/µm). The dissolution slowness of the {100} plane is about 0.77 min/µm.