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ABSTRACT

Etch rate experiments were carried out for (100) GaAs using etching solutions of $H_2SO_4:H_2O_2:H_2O$ (3:1:1, 3:1:15), $HCl:HNO_3$ (3:1), $HCl:HNO_3:H_2O$ (1:1:1) and $HCl:HNO_3$:glycerol (with various dilutions of glycerol). Several differences were seen for the (100) plane compared to previous results for other crystal orientations. The sulphuric acid solutions showed much lower activation energies for etching the (100) plane. The $HCl:HNO_3$:glycerol solutions showed considerably lower etch rates for the (100) plane, probably indicating that they etch GaAs anisotropically.

For a 1:1:2 solution of $HCl:HNO_3$:glycerol a decrease in the etch rate of (100) GaAs was observed in the presence of stirring. This is the opposite result to what is commonly assumed for this polishing etchant. It indicates that the main polishing process attributed to this etchant is not present, and in fact, the polishing quality of the etchant is probably limited by the etching process which is present.

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EDITORIAL NOTE

The Australian Nuclear Science and Technology Organisation replaced the Australian Atomic Energy Commission on 27 April 1987. Reports issued after April 1987 have the prefix ANSTO with no change of the symbol (E, M, S or C) or numbering sequence.

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1. INTRODUCTION

A great deal of effort has been expended on studies of etchants for Gallium Arsenide. Both the aqueous $H_2SO_4:H_2O_2$ etchant system and $HNO_3:HCl$ based etches have been the object of some investigation and there are a couple of reviews which give an overview of that work [1,2]. There is, however, a great amount of detail which has not been widely published for these systems. Etching rates is one area where the published information is incomplete.

Etching rates for the $H_2SO_4:H_2O_2:H_2O$ system have been reported by Iida and Ito [3] who carried out a comprehensive investigation over the temperature range from 0° to 100°C for the (001) plane. Other surfaces were studied but only with $H_2SO_4:H_2O_2:H_2O$ = 8:1:1 and 1:8:1 solutions, and then not for the (100) plane. D.W. Shaw [4] reported some etch rates for the (100) GaAs plane but using only room temperature $H_2SO_4:H_2O_2:H_2O$ etching solutions.

For $HNO_3:HCl$ based etchants the published information is very sparse, especially for etch rates. The problem with $HNO_3:HCl$ etching solutions is that, for some orientations, there is a tendency towards the formation of etch pits [5,6]. Despite this problem, however, there is still some degree of interest in this system as evidenced by such recent publications as that of Adachi and Oe [6] and Bourret et al [7].

For the experiments reported here the etch rate of (100) semi-insulating GaAs was measured at various temperatures and concentrations for the etchant systems mentioned above. These measurements were motivated by a need to control the etching rates during substrate and epitaxy preparation. For substrates, the surface damage caused by mechanical polishing requires from 50 to 80 microns of the semiconductor to be removed prior to epitaxial growth. For epitaxies, only some 10 microns is required for removal before metalisation.

2. EXPERIMENTAL PROCEDURE

For these experiments, etch rates for the aqueous $H_2SO_4:H_2O_2$ system were studied at two different dilutions, $H_2SO_4:H_2O_2:H_2O$ = 3:1:1 and $H_2SO_4:H_2O_2:H_2O$ = 3:1:15, and at a number of different temperatures. Etch rate experiments were also carried out at room temperature using aqua

regia ($HCl:HNO_3$ = 3:1), $HCl:HNO_3:H_2O$ = 1:1:1 and HNO_3:HCl:glycerol = 1:1:2.

Commercially available wafers of semi-insulating (100) GaAs were used for all of the measurements. The wafers were cut into sections; each section was degreased in xylene; given a displacement rinse in methanol, and then finally a rinse in high purity (18Mohm) H_2O. The width of each wafer section was measured with a micrometer and then weighed with a microbalance so that the initial volume and area of each wafer section could be determined. A sample density of 5.316 g/cc [8] was used for these calculations.

For each of the etching experiments a fresh solution of etchant was prepared. The chemicals used for the etchants were all reagent grade with the following compositions: HCl (37%), HNO_3 (70%), H_2O_2 (31%), H_2SO_4 (96%) and glycerol (99.5%).

When necessary the etching solutions were cooled to the appropriate temperature in an ice or water bath. Cooling usually took some time because of exothermic reactions between the etchant ingredients; this was particularly so for the $H_2SO_4:H_2O_2:H_2O$ etchants.

After the appropriate solution temperature was reached a number of the semi-insulating wafer sections were put in the etch solution. Whilst in the solution the wafer sections were hand stirred with a set of teflon tweezers (though occasionally the conditions of stirring were changed, as described below) and then each section was removed at the end of a fixed period of time. After being removed from the etch the wafer sections were immediately quenched in water. After drying, the sections were reweighed and their widths determined using the initial area as a constant. This procedure was followed for several samples using a number of time periods for each temperature and solution used.

For the 3:1:1 $H_2SO_4:H_2O_2:H_2O$ etch some further room temperature etch rate experiments were carried out, with no stirring, with continuous hand stirring (random stirring), and with the etch solution being vigorously stirred using a magnetic teflon stirring rod. These measurements were done

in order to establish what effect different types of stirring would have on the etch rates for the 3:1:1 solution.

The severity of etch pitting was observed for a number of $HCl:HNO_3$:glycerol solutions with varying glycerol dilutions. This was done in order to help choose an appropriate glycerol dilution for the etch rate experiments. The 1:1:2 $HCl:HNO_3$:glycerol solution was eventually decided upon for this purpose. Using that solution, a number of etch rate experiments were carried out with continuous hand stirring, and with no stirring at all. This was done in order to observe the effects of stirring for that very viscous solution.

3. RESULTS AND DISCUSSION

3.1 $H_2SO_4:H_2O_2:H_2O$ Etch Rates

Etching solutions generally contain an oxidant, which in this case oxidises GaAs, and a solvent which dissolves the oxidised products. For the $H_2SO_4:H_2O_2:H_2O$ system the H_2O_2 acts as the oxidant while the H_2SO_4 acts as the solvent [4]. The additional H_2O is usually thought of as a dilutant [2].

The results for the $H_2SO_4:H_2O_2:H_2O$ etches are summarised in Figures 1, 2 and 3. For these etches wafer thickness reduction was always approximately linear with time (see Figures 1 and 2). This linear relationship indicates that the hydrogen peroxide was not decomposing at higher temperatures, since if it had the etchant would have been rendered useless.

The linear relationship between wafer thickness reduction and time may also indicate that the etching reactions were all reaction limited rather than diffusion limited [9,10]. The distinctions between these two types of reaction are explained further on in the text. For a diffusion limited reaction, however, the thickness reduction is related to the square root of time [9].

The rate removal for the 3:1:1 etchant was greater than that of the more dilute 3:1:15 etchant by a factor of 2. From the data plotted in Figure 3 (and using a linear approximation, as found by a least squares analysis) the rate of thickness removal per degree celsius was calculated

for both of the etchants. A value of 0.217 $\mu m/min/^\circ C$ was obtained for the 3:1:1 etch and a value of 0.128 $\mu m/min/^\circ C$ for the more dilute 3:1:15 etchant. (Notably, the room temperature etch rate found by Shaw [4] for a 3:1:1 solution agrees well with a room temperature etch rate found from the rate thickness removal value shown above).

Considering the amount of dilution between the two solutions a greater difference in etch rates might have been expected. There are, however, two competing factors which determine the etch rates observed. First of all for the $H_2SO_4:H_2O_2:H_2O$ 3:1:1 solution the H_2SO_4 would be incompletely ionised due to the lack of H_2O. On the other hand, the H_2SO_4 in the more dilute 3:1:15 solution must be completely ionised so that it would be more effective in that solution. The second factor, the factor which acts to lessen the effect of the greater degree of H_2SO_4 ionisation in the 3:1:15 solution, is that the H_2O_2 in that solution is more dilute, and therefore, less effective.

3.2 Stirring Effects for $H_2SO_4:H_2O_2:H_2O = 3:1:1$

Stirring has been noted to affect the etching rate for a mixture of 8:1:1 $H_2SO_4:H_2O_2:H_2O$ [3]. The increase in etch rate with stirring is thought to occur because the high concentration of sulphuric acid gives the solution a high viscosity. The high viscosity ensures that the diffusion of reactants becomes an important step in limiting the etch rate of the solution [11]. Therefore, without stirring, a layer depleted of reactants eventually builds up around the GaAs sample. Stirring the solution ensures that this layer is reduced and a fresh supply of reactants comes in contact with the GaAs so that the etch rate increases [2].

The 3:1:1 solution, used for these experiments, and commonly used by other investigators [see, for example, 6 and 7], although not as viscous as the 8:1:1 mixture, still has a high concentration of sulphuric acid. It might therefore be expected to show a similar dependence of etch rate on stirring. This was indeed found to be the case. At room temperature stirring the 3:1:1 $H_2SO_4:H_2O_2:H_2O$ solution was found to increase the measured etch rates by a considerable amount, as shown in Figure 4. The actual method of stirring used did not make that much difference - though hand stirring gave slightly higher etch rates. Notably high viscosity

solutions, such as those with a high H_2SO_4 content, are often used to improve the polishing quality of an etch [1,4,10]. These solutions are thought to preferentially etch the high points of the semiconductor compared with the low points (diffusion of reactants to the low points takes longer because a viscous solution tends to be more stagnant at the low points). This can result in a very smooth surface which is desirable for the etching of epitaxies and substrates.

For the 3:1:15 $H_2SO_4:H_2O_2:H_2O$ solution it was not known whether stirring would have affected the etch rates. Based on the past experience of Iida and Ito [3], it might have been expected that in this lower H_2SO_4 (lower viscosity) solution, the etching rate of the GaAs would have been reaction limited and therefore, unaffected by stirring [3]. However, Iida and Ito also noted a significant difference in the activation energy for high and low H_2SO_4 solutions. This difference has since been interpreted as being due to the two major processes controlling etching reactions. A high activation energy, is therefore, usually attributed to a reaction limited etch rate and a low activation energy is attributed to a diffusion limited etch rate [2]. As shown in Figure 5, this thinking is not reflected in the data presented here, since a lower activation energy was found for the 3:1:15 solution - the solution more likely to be reaction limited when etching GaAs.

Another point regarding activation energies is that the activation energies measured here for the 3:1:1 and 3:1:15 etchants (with stirring) are much lower than what might have been expected based on the findings of Iido and Ito. For the 3:1:1 solution, the activation energy was found to be 5.0 kcal/mol and for the 3:1:15 solution it was 3.5 kcal/mol. These lower activation energies are attributed to the experiments being performed on a (100) plane, a plane not examined by Iida and Ito. If this interpretation is correct then despite the low activation energies, which would normally indicate otherwise, it is possible that in the presence of stirring both the 3:1:1 etchant and the 3:1:15 etchant could have been reaction limited. This idea is confirmed by the linearity of the semiconductor thickness removal with time for both of the etchants since such linearity is indicative of a reaction limited etch rate [9,10]. It would also help explain why the 3:1:15 etchant did not have a higher etching activation energy than the 3:1:1 etchant. Of course, in the

absence of stirring the etching action of the 3:1:1 etchant would still have to be diffusion limited. This would explain the change in etch rate when stirring took place. Further measurements would, of course, have to be made to confirm the above ideas.

3.3 $HCl:HNO_3$ Etches

$HCl:HNO_3$ based etchants were examined here as an alternative to the $H_2SO_4:H_2O_2:H_2O$ system. The results of etching experiments for $HCl:HNO_3$ 3:1 and $HCl:HNO_3:H_2O$ 1:1:1 are shown in Figures 6 and 7. The etch rate for the $HCl:HNO_3$ 3:1 etchant at 26°C was found to be 6.2 microns/min. The etch rate for the $HCl:HNO_3H_2O$ 1:1:1 etchant was found to be 1.8 microns/min.

HNO_3 can act as an oxidant on its own, however, for a solution of $HCl:HNO_3$ there is believed to be a reaction between the two acids which results in the formation of chlorine. It is the chlorine which is apparently responsible for the main etching action of $HCl:HNO_3$ based solutions [2]. The reduction in etch rate between the two etches may therefore be explained by the reduction in the concentration of HCl (and hence chlorine).

As an alternative to the $H_2SO_4:H_2O_2:H_2O$ etchant system the main drawback of aqua regia based etches is that they tend to cause etch pitting on certain orientations of GaAs [5,6]. Severe etch pitting was also observed here for these experiments with the (100) plane. This etch pitting would seem to be related to the vigorous formation of gas bubbles around the GaAs during etching [2]. The more effective etchant, the $HCl:HNO_3$ 3:1 solution, of course, produced more bubbling and a greater degree of etch pitting than the $HCl:HNO_3:H_2O$ 1:1:1 etchant.

3.4 $HCl:HNO_3$:Glycerol Etches

In order to improve the quality of $HCl:HNO_3$ based etchants, glycerol has been suggested as a useful dilutant by Packard [11]. A more dilute $HCl:HNO_3$ solution has the advantage of reduced etch pitting, and the very high viscosity of the glycerol would seem to be an advantage for polishing (the above discussion for the H_2SO_4 etchant system explains the advantages of viscous etchants). Great care must, of course, be used when experimenting with nitric acid and glycerol since these ingredients can, under certain circumstances, react to form nitroglycerin - a very unstable

explosive. The use of sulphuric acid with nitric acid and glycerol is especially dangerous [12].

In his work Packard used a 1:1:8 HNO_3:HCl:glycerol solution for which he obtained an etching rate of 0.37 mg/cm^2 min, or 0.70 microns/min. This etchant was re-examined for this investigation but it was found that the etch rate with stirring for the (100) plane was far lower than that found by Packard for a (111) crystal orientation. Less than 0.2 microns of material was removed from (100) samples spending from 3 to 4 minutes in this etching solution.

Obviously a lesser amount of glycerol was called for in order to raise the etch rates for the (100) GaAs plane. However, a compromise had to be reached whereby the severity of etch pitting was at an acceptable level for GaAs etching. To achieve this compromise, glycerol was added to 1:1 HCl:HNO_3 to make mixtures of greater dilution. For each of the mixtures the bubbling on the surface of GaAs samples was observed during etching (without stirring) and the severity of etch pitting was observed after the etching. Room temperature solutions of 2:2:1, 1:1:1, 1:1:2 and 1:1:3 HCl:HNO_3:glycerol were all examined in this manner. For the 1:1:2 solution, some bubbling was still observed on the (100) GaAs in the absence of stirring, however, the bubbles formed were small in size and took some time to appear. Certainly the bubbling and consequent etch pitting observed on the GaAs appeared to be no worse than that observed for the standard 3:1:1 H_2SO_4:H_2O_2:H_2O solution. The 1:1:2 HCl:HNO_3:glycerol solution was therefore selected for further GaAs etch rate experiments.

The 1:1:2 HCl:HNO_3:glycerol etch rate experiments were carried out at room temperature with vigorous hand stirring and with no stirring. The results of these experiments are shown in Figure 8.

A fresh solution of the 1:1:2 etching solution had to be used for all of the above measurements because when left for long enough the solution would spontaneously boil, probably due to an exothermic reaction with a very long time constant. First all of the HCl, and then all of the HNO_3 would boil off leaving a water and glycerol solution. The source of this exothermic reaction was not clear. A rise in temperature to about

40°C was noted during the initial mixing of the 1:1:2 solution. However, unless a large batch of the solution was made, the temperature would subsequently drop so that the solution remained stable for a period of some hours.

Although stirring this etchant was very effective in stopping bubbles from forming on the surface of the GaAs, stirring was also shown to reduce the etch rate for the GaAs (see Figure 8). This indicates that some form of autocatalytic reaction [2] was probably occurring: an autocatalytic reaction being one for which some of the reacting species form a catalyst during the initial etching process. It may also indicate that, at least for this particular plane, the HNO_3 plays a more significant role in the etching of GaAs since it is known to take part in autocatalytic reactions [2]. Regardless, however, of the source of the reaction, the effect of removing the catalytic species by stirring is to reduce the etching rate.

Of course, one observation that tends to confirm the importance of the HNO_3 during etching with this solution, is that the differences in the etch rate depend on how the solution is mixed. Mixing HNO_3 and glycerol first, and then adding HCl, produces a clear solution apparently free of NO_2 (a product of the decomposition of HNO_3). This solution was shown to be affected more by stirring than a solution for which the HCl and HNO_3 were first added together with the glycerol being mixed in later (see Figure 8). This second solution showed a characteristic yellow colour indicating the presence of NO_2 [13]. NO_2 is a strong oxidising agent [14] and its presence may well facilitate an important step during the etching of the (100) GaAs. In a solution which is NO_2 rich it might well be expected that stirring has a lesser affect than in a solution where NO_2 is only present at the surface of the GaAs.

One of the consequences of the effect of stirring on the HCl:HNO_3:glycerol solutions is that they are probably a poor choice for polishing (100) GaAs since the etching would occur in an opposite sense to that supposed by Packard [11]: i.e. the low points of the GaAs surface would actually be etched in preference to the high points because greater concentrations of the catalytic species would build up in the stagnant layer of liquid near the lower areas of the semiconductor.

On the positive side, there appears to be significant anisotropy for this etchant system. This is evidenced by a comparison of the etch rate results presented here using 1:1:8 $HCl:HNO_3$:glycerol and the results of Packard [11]. An etching rate of ~ 0.06 microns/min for (100) GaAs was found for these experimeents compared to 0.7 microns/min for {111} GaAs as found by Packard. Both sets of experiments were carried out at room temperature with stirring present. The slow etch rates of these glycerol diluted solutions would be an advantage in obtaining good reproducability during preferential etching. Slow preferential etches are usually very hard to attain with wet chemicals since most preferential etches are usually reaction limited rather than diffusion limited [4].

4. CONCLUSIONS

Etch rate experiments were carried out using etching solutions of 3:1:1 and 3:1:15 $H_2SO_4:H_2O_2:H_2O$. For these solutions the activation energies were well below other values found for the etching of GaAs. Such low activation energies are usually attributed to a diffusion limited etching process. An observed increase in the etch rate with stirring would tend to confirm that the 3:1:1 solution was diffusion limited. However, with stirring both the 3:1:1 solution and the 3:1:15 solution had similar etching activation energies. Given the lower viscosity of the 3:1:15 solution, it should have had a greater activation energy than the 3:1:1 solution if their etching rates were both diffusion limited. What's more the depth of semiconductor removal was linear with the time of removal, indicating that the etchants were reaction limited. It was therefore thought that the 3:1:1 and 3:1:15 solutions were both reaction limited in the presence of stirring, while without stirring the 3:1:1 etchant was diffusion limited. The low activation energies for these etchants were therefore attributed to the use of the (100) plane for these experiments. This hypothesis, of course, needs further measurements in order to be confirmed.

A 1:1:8 $HCl:HNO_3$:glycerol solution was found to etch the (100) plane of GaAs at a much lower rate than other crystal orientations. This indicates that the highly viscous $HCl:HNO_3$:glycerol system etches GaAs anistropically. This is a surprising result since high viscosity solutions tend to be isotropic.

An even more surprising result for the HCl:HNO_3:glycerol system was that the etch rate of a 1:1:2 solution was shown to decrease with stirring. This is the opposite result to what is commonly assumed for a high viscosity, diffusion limited etchant. It indicates that the system undergoes an autocatalytic reaction while etching the (100) plane of GaAs. This may explain why the system etches GaAs anisotropically, since the etching action may in fact be reaction limited by the effect of the catalytic species formed at the surface of the GaAs. Diffusion may, of course, still play an important role in limiting the effect of other etching reactions competing with the autocatalytic reaction.

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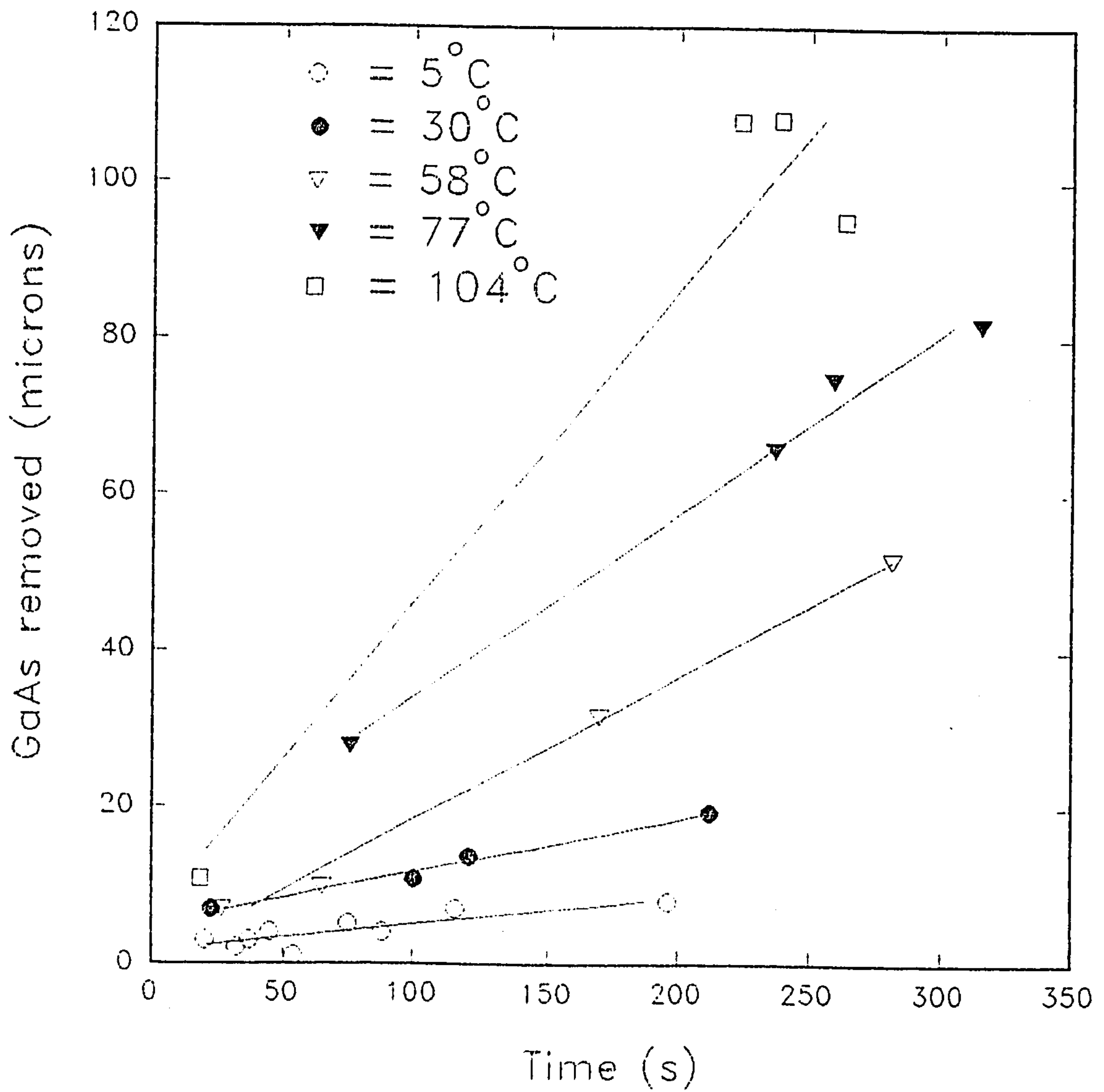

Figure 1: Thickness of (100) GaAs removed, versus time, using 3:1:1 $H_2SO_4:H_2O_2:H_2O$ at various temperatures.

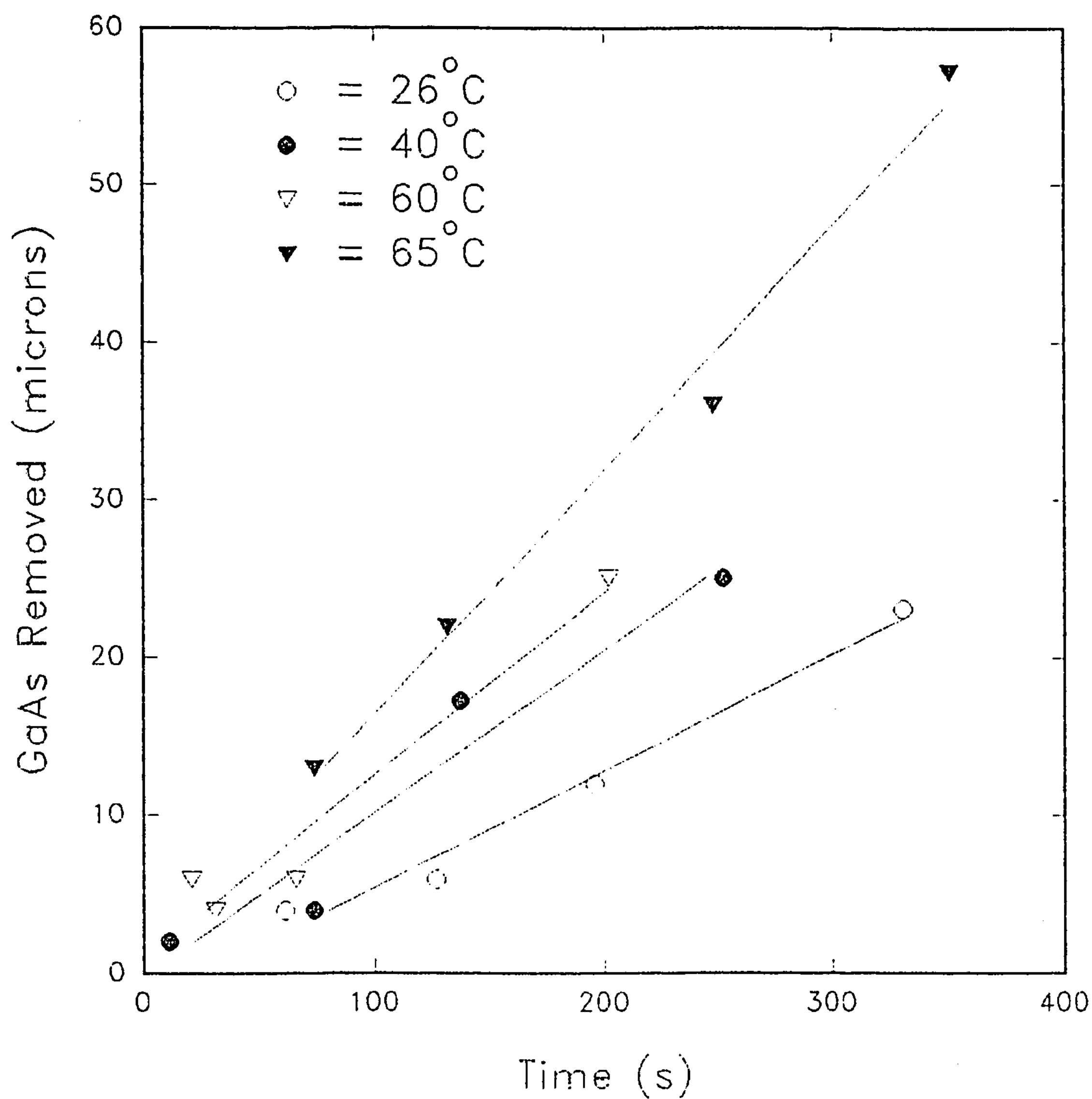

Figure 2: Thickness of (100) GaAs removed, using 3:1:15 H_2SO_4:H_2O_2:H_2O at various temperatures.

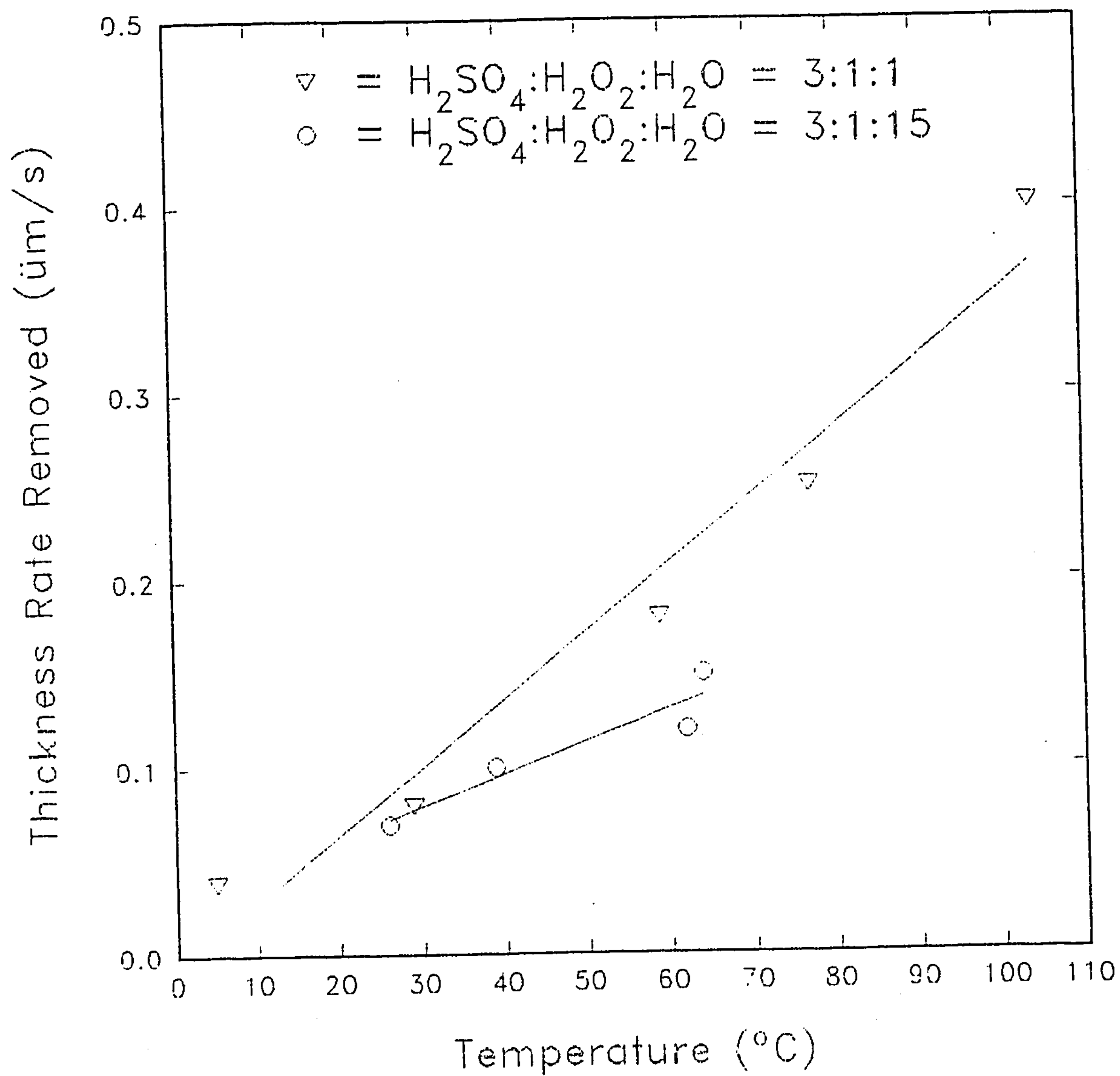

Figure 3: The rate removal of (100) GaAs for 3:1:1 and 3:1:15 $H_2SO_4:H_2O_2:H_2O$.

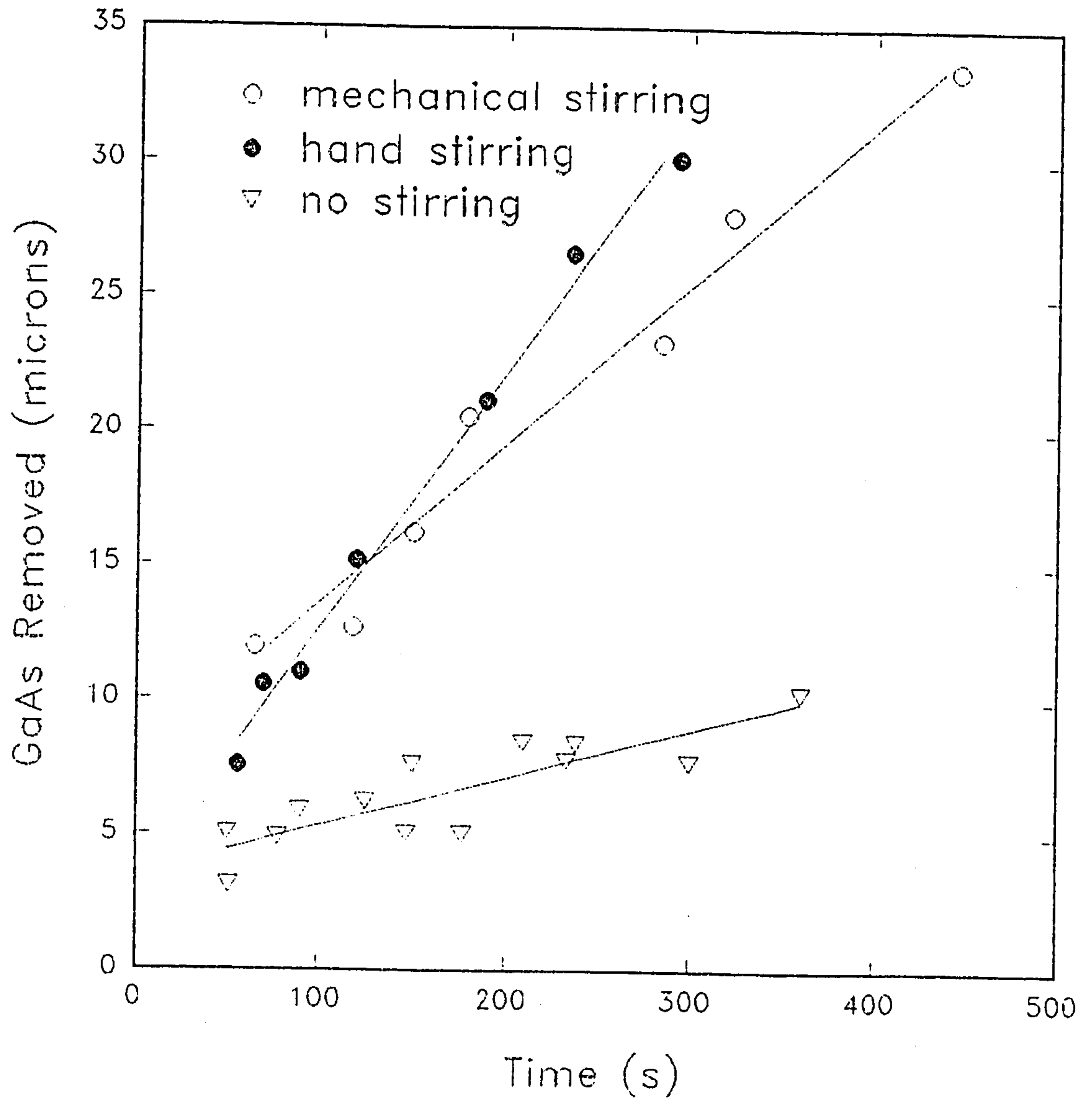

Figure 4: Effect of stirring on the etch rate of (100) GaAs using 3:1:1 H_2SO_4:H_2O_2:H_2O.

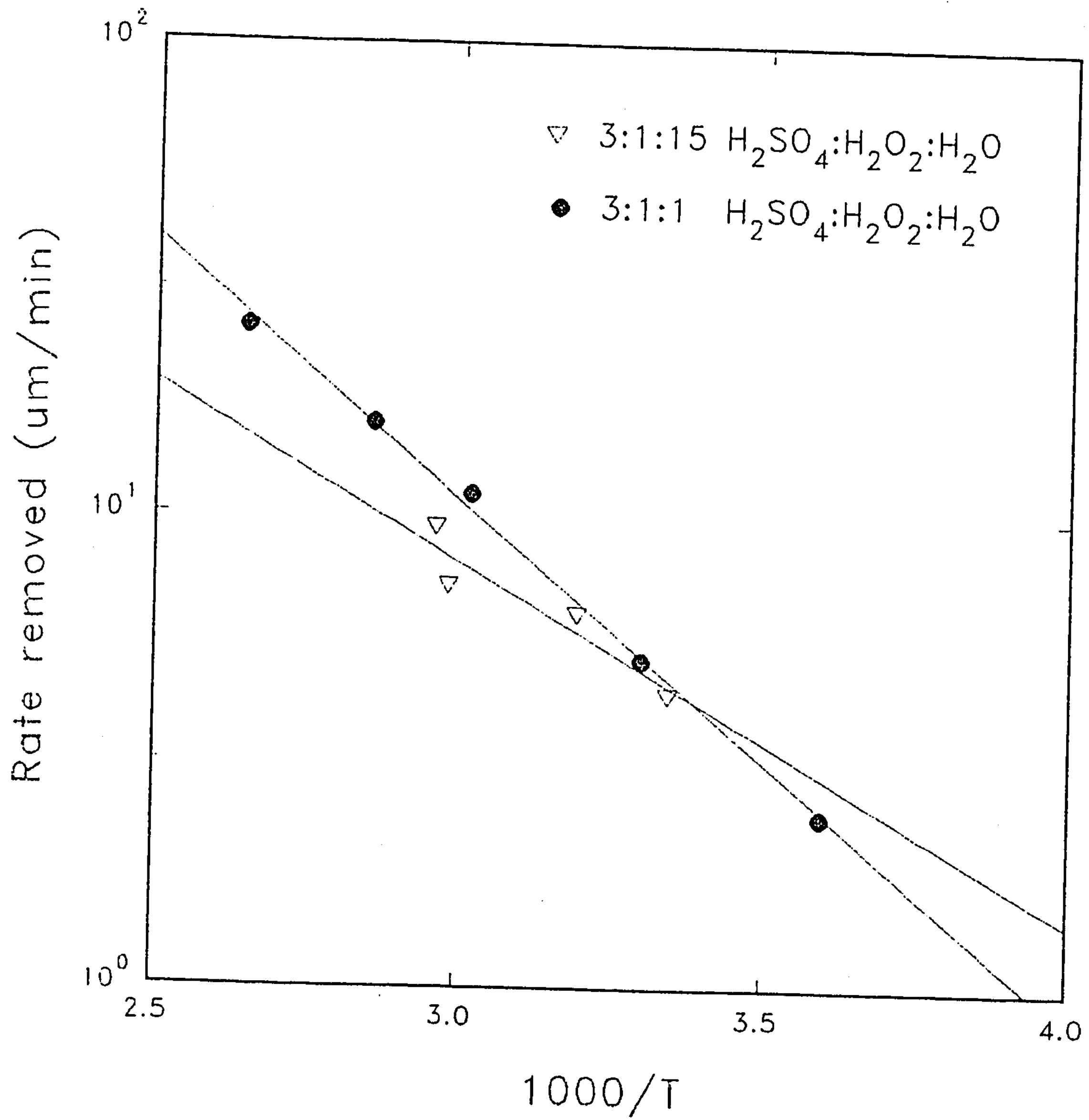

Figure 5: Arrhenius plots for the etching of (100) GaAs using 3:1:1 and 3:1:15 $H_2SO_4:H_2O_2:H_2O$ solutions.

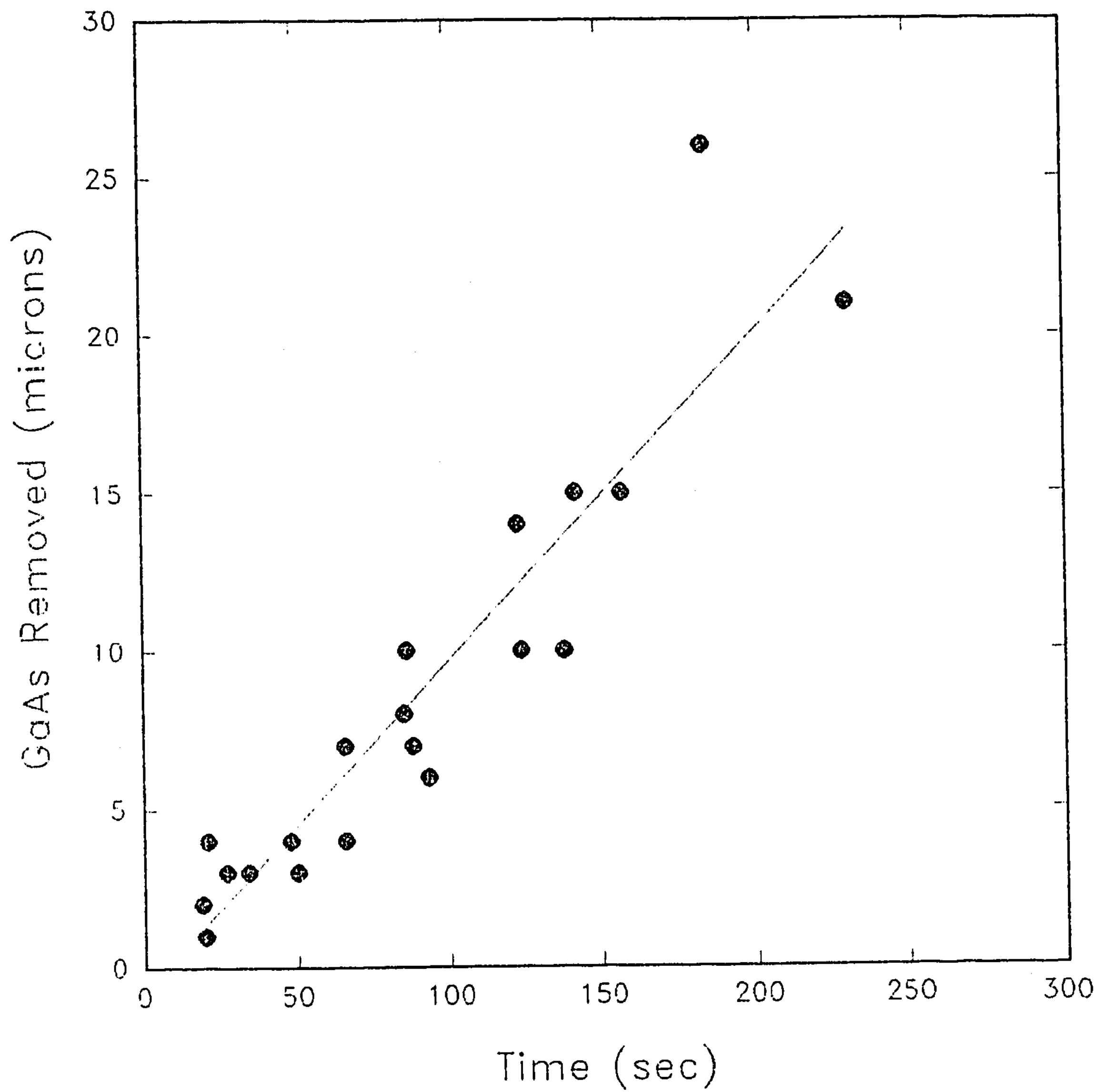

Figure 6: Thickness of (100) GaAs removed, at room temperature, using 3:1 $HCl:HNO_3$.

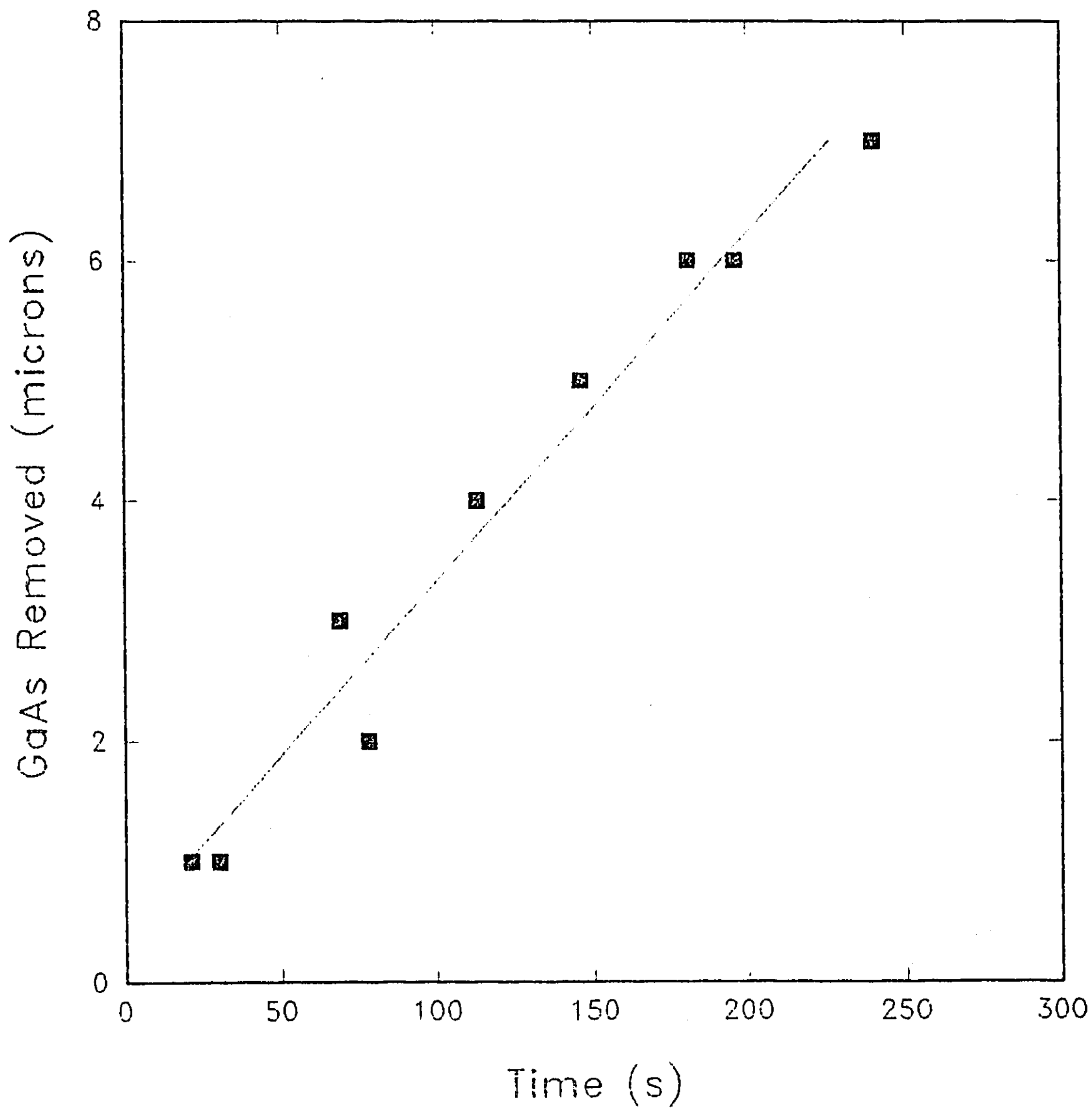

Figure 7: Thickness of (100) GaAs removed, at room temperature, using 1:1:1 $HCl:HNO_3:H_2O$

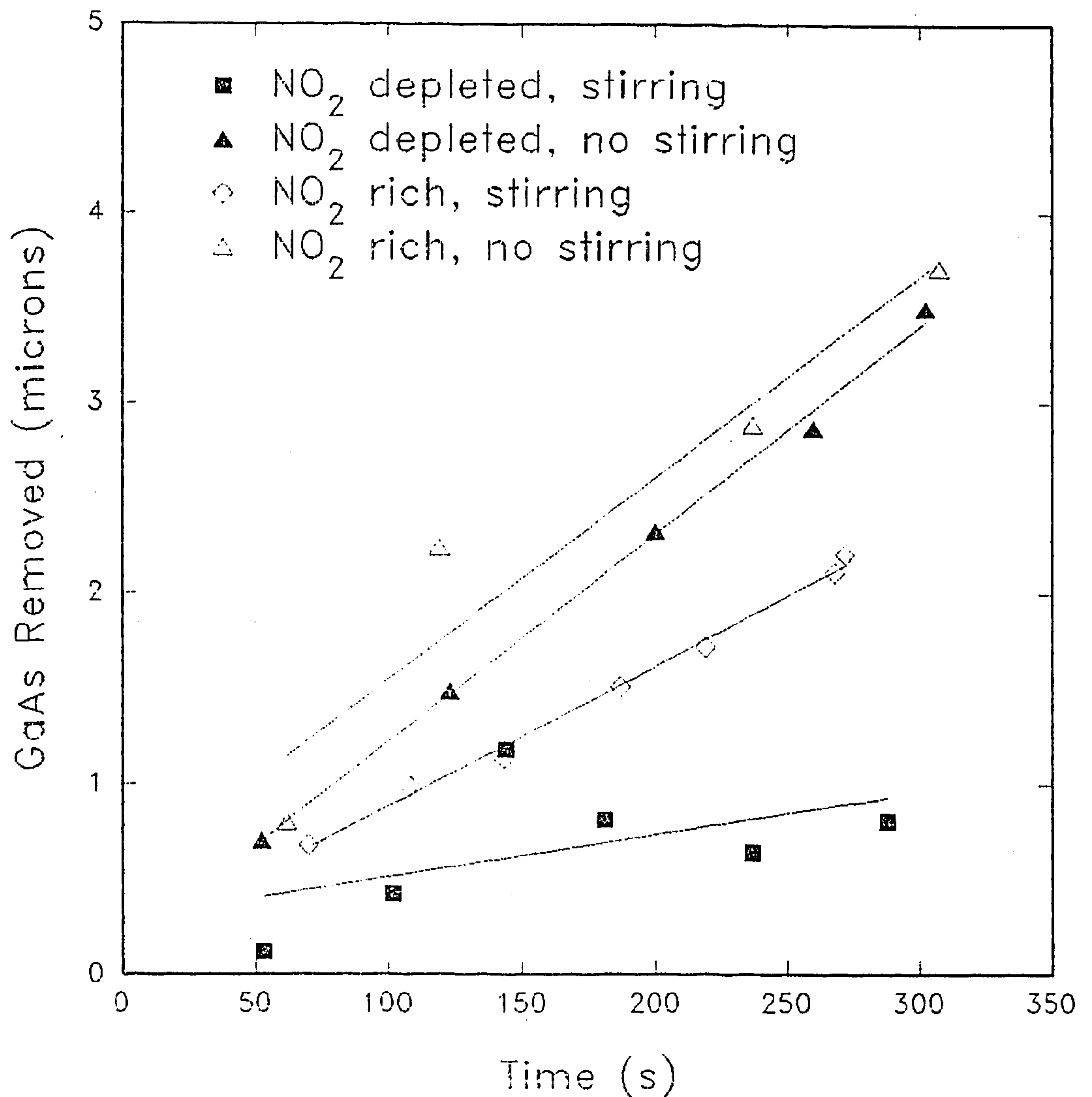

Figure 8: Effect of stirring on the etch rate of (100) GaAs using 1:1:2 $HCl:HNO_3$:Glycerol, at room temperature.