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STUDIES OF TANTALUM NITRIDE THIN FILM RESISTORS

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I. INTRODUCTION

Extensive interest in tantalum as a thin film material has been stimulated by its use in the microelectronics industry. Tantalum has a refractory nature which implies that any imperfection frozen in during deposition will not anneal out during the life of the thin film. In addition, tantalum belongs to a class of metals which form tough self-protective oxides either through heat treatment or through anodic oxidation.<sup>1</sup> The preferred method of deposition has been sputtering rather than evaporation because of the refractive nature of the tantalum. Sputtered tantalum films have a tendency to become contaminated since it is a reactive material although some degree of contamination is desirable in order to achieve useful properties.<sup>2</sup>

The technology of tantalum resistors is complicated by the fact that tantalum films can exist in two separate structures or a combination of both.<sup>3</sup> The conventional bcc tantalum structure corresponding to the bulk is known as the  $\alpha$  structure. The  $\beta$  structure is tetragonal in nature. The exact conditions under which  $\alpha$  or  $\beta$  tantalum will form are not fully understood; however, it has been found that the  $\beta$  form does not appear in systems in which there is an appreciable degree of gaseous contamination.<sup>4</sup> Frequently films contain a mixture of the  $\alpha$  and  $\beta$  structures. Reproducibility of the films is difficult to achieve under these conditions. Originally oxygen was used as a form of contamination which could be added in a controlled fashion. Oxygen has a profound effect on both the resistivity and the thermal coefficient of resistance (TCR) of the tantalum films. The change in resistivity with % dissolved oxygen is extremely rapid and difficult to control. A more

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attractive contaminant for deliberate introduction during the sputtering process is nitrogen. In practice film composition close to that of  $Ta_2N$  was chosen since it was thought that resistors having this composition would display the greatest stability during life tests.<sup>5</sup> Tantalum nitride films are susceptible to oxidation and will change characteristics on exposure to air. It is common to lightly anodize the film or to anneal the film in air to create a thin oxide layer on the surface which stabilizes them.

The purpose of this study was to correlate the electrical properties of tantalum nitride thin film resistors with the nitrogen concentration in the films. Previous studies have used either separately or in combination Auger sputter profiling<sup>6</sup> and chemical analysis<sup>7,8</sup> to give constituent concentrations and depth distributions in a destructive analysis. In the present experiment ion backscattering was used to provide both the constituent concentrations and depth distributions in a single nondestructive test. In addition growth of the oxide during the stabilization period was studied. The present study has accomplished this correlation by using high energy (2.0 MeV) ion backscattering to characterize the thin film tantalum nitride resistors, as to the amount of surface oxide, the nitrogen content, its depth distribution, the amount of argon sputtering gas incorporated into the film and its depth distribution.

The films for these experiments were deposited on two types of substrates: alumina and beryllium. The use of a low Z substrate in ion backscattering, namely Be, allows observation of film components of higher Z, e.g., C, N, O, and Ar. The films on the alumina were characterized both by ion backscattering and electrical measurements and the films on the beryllium were characterized only by ion backscattering. The electrical properties of the films measured were the sheet resistance, differential Seebeck potential, thermal coefficient of resistance, and stability. Preliminary results were given in a previous paper.<sup>9</sup> This

paper discusses the preparation of the substrates and resistive films, the resistor definition, the stabilization procedure, and the electrical and ion backscattering measurements. The results of the electrical and ion backscattering measurements are presented, and finally the conclusions based on the experimental results are discussed in detail.

## II. EXPERIMENTAL PROCEDURE

A. Sample preparation. Substrates of both alumina and beryllium were used in the experiment. The alumina substrates had dimensions  $2.54 \times 2.54 \times 0.068$  cm and were cleaned by ultrasonically washing successively in trichloroethylene, acetone, detergent, and deionized water followed by hot deionized water rinse and an air firing at  $900^\circ\text{C}$  for one hour. The beryllium substrates were machined from high purity bulk material which had been formed by cold isostatic pressing followed by hot isostatic pressing and whose grain sizes were of the order of  $10 \mu\text{m}$ .<sup>11</sup> The substrates were subsequently mechanically polished, electrically polished and finally rinsed in deionized water. Air exposure of these substrates at room temperature provided a stable surface oxide layer, approximately 100 Å thick, so that deposition was accomplished on similar oxide surfaces: namely alumina and beryllia. In the vacuum station the substrates were heated to  $400^\circ\text{C}$  for two hours before sputtering was accomplished. The films were prepared by dc diode reactive sputtering of tantalum in nitrogen-argon mixtures in a 15 cm diameter oil diffusion pumped system with a liquid nitrogen trap and a stainless steel bell jar. Sputtering from a  $730 \text{ cm}^2$  cathode was conducted at a potential of 5 kV, a current density of  $0.25 \text{ mA/cm}^2$ , an interelectrode spacing of 7.0 cm and a gas pressure of  $4.5 \times 10^{-2}$  Torr. The nominal deposition rate was 150 Å per minute, the argon flow rate was 20 Std  $\text{cc}^3/\text{min}$  (sccm) and the nitrogen flow rate was varied from 0.5 to 0.9 sccm. This technique resulted in film

compositions varying from 0.25 to 0.55 at.% nitrogen. A presputtering period of 20 minutes was accomplished with a shutter covering the substrates. Following this presputtering the shutter was removed and the films were deposited in approximately 180 seconds. This procedure produced tantalum nitride films of nearly the same thickness but with varying amounts of nitrogen incorporated into the film. Each deposition was made on a batch of 36 alumina substrates arranged in a square configuration. To obtain films of the same thickness only the 4 central substrates were used in this study. This was necessary because the substrates not in the center were found by ion backscattering to have minor variations in the thickness of the resistor films. This variation was attributed to perturbation of the electric field caused by fixturing in the sputtering system. Two of the four central substrates diagonal to each other had 0.95 cm holes through them to accommodate the beryllium substrates which were used in the ion backscattering study.

#### B. Resistor Definition and Stabilization.

In order to make certain of the electrical tests, it was necessary to deposit conductor films on the resistor material so that leads could be attached. This was carried out by vacuum evaporation of approximately 300 Å of chromium followed by a deposition of 30,000 Å of gold. During these depositions the substrate temperature remained at 375°C. Following these depositions resistor patterns were photolithographically defined on the alumina substrate for subsequent determination of the thermal coefficient of resistance and the long-term stability. Photolithography is discussed extensively in both references 2 and 5 and will not be discussed further here. Following resistor definition, stabilization of the resistors was accomplished by annealing in air for 2 hours

at 300°C. This formed a passivating oxide layer on the films. Both electrical and backscattering measurements were made before and after this anneal. Fifteen parallel resistors were defined on each substrate. They were 1.27 cm long and had a width of 0.13 cm with a nominal resistance of 10 kOhms each. Following the photolithography process and the resistor stabilization, gold-plated copper leads were attached by thermal compression bonding.<sup>11</sup>

C. Electrical Measurements. Sheet resistance and the differential Seebeck potential measurements were performed on the thin film resistors before metallization. Measurements of the sheet resistance,  $R_s$ , were performed using an A & M Fell Ltd. four-point probe system. The resistivity,  $\rho$ , of the thin film resistors was determined from the sheet resistance and the film thickness,  $T$ , as determined by ion backscattering, by the equation

$$\rho = R_s \cdot T$$

Four resistance measurements were made on each substrate and the results from the four substrates of each deposition run were averaged to give a sheet resistance for the run. Seebeck thermoelectric measurements were made using two gold-plated copper contact blocks, 13 x 13 mm, separated by 22 mm. One contact was thermostated at 10°C using a Peltier cooler and the other contact was thermostated at 60°C. This configuration allowed measurements of the differential Seebeck potential. Two measurements were made on each substrate along the diagonal axes of the resistor. Again the results from the four substrates of each deposition run were averaged.

The thermal coefficient of resistance (TCR) measurements were performed after resistor definition and stabilization by measuring the resistance of each resistor at three temperatures:  $-195^{\circ}\text{C}$ ,  $23^{\circ}\text{C}$  and  $+150^{\circ}\text{C}$ . From these measurements, TCR can easily be calculated.<sup>12</sup> Stability tests were run at  $150^{\circ}\text{C}$  for 1000 hours. The resistance was sampled at 30, 100, 300 and 1000 hours. In each case the 15 resistors on one substrate were averaged to give an average value of TCR and stability. On those substrates which had the hole in them to accommodate the beryllium substrates, only 8 resistors could be defined. For each deposition there were values obtained for 46 resistors. These were averaged to give a value for TCR and stability for that particular deposition run.

D. Backscattering Measurements. Ion backscattering has been used extensively to determine composition vs. depth in solids.<sup>13</sup> A well-collimated monoenergetic beam of  $\text{He}^+$  ions was directed onto the sample. The incident ions lose energy in the solid by two processes: (i) numerous collisions with electrons of the sample material, and (ii) individual elastic scattering events due to the repulsive Coulomb force between the nuclei of the incident ion and the target atom. The stopping power of the electrons of the sample material provide information about the depth distribution of the elements in the sample since ions scattered from atoms deep in the sample will have lost more energy than those nuclei scattered from atoms of the same element nearer the surface. The nuclear scattering allows identification of the constituent elements of a thin film by the amount of energy the incident ion loses to recoil of the target atom. Light ions such as nitrogen and oxygen can take up a large portion of the energy of the ion, while heavy atoms like tantalum absorb relatively little energy in recoiling. Beryllium is an ideal substrate to use because the low backscattering yield from this low-Z element is relatively small and occurs at low energy.

The higher energy backscattering yield from a higher-Z element is therefore easily resolved. This backscattering technique has the important feature of being nondestructive.<sup>14</sup>

The ion backscattering measurements were made with 2 MeV Helium ions obtained from a Van de Graaff accelerator. The experimental apparatus and the basic equations pertinent to ion backscattering have been described elsewhere.<sup>15</sup> The beam had dimensions of 1 x 1 mm and a current in the range of 10-50 nA. Typically, the charge deposited per spectrum was 10  $\mu\text{C}$  for the alumina substrates and 40  $\mu\text{C}$  for the beryllium substrates. Particles backscattered at an angle of  $170^\circ$  were energy-analyzed with a surface-barrier Si detector whose energy resolution corresponded to  $\sim 120 \text{ \AA}$  depth resolution. For each deposition, eight backscattered energy spectra were taken: one on each of the alumina substrates, and one on each of the two beryllium substrates before stabilization and one on each after stabilization.

### III. RESULTS

A typical backscattered energy spectrum for an unannealed sputtered tantalum nitride film on a beryllium substrate is shown in Fig. 1. For energies less than 1.7 MeV the vertical scale is expanded by a factor of 50. The solid vertical lines indicate the energies with which 2.0 MeV He ions are backscattered from Ta, Ar, O, N, C and Be atoms when these elements are on the surface. These energies correspond to the half-heights of the leading edges of the respective scattering peaks.

The ion backscattering yield from Ta is shown at high energy,  $\sim 1.8$  MeV. The width of the peak is the measure of the film thickness. The yield from the argon incorporated into the film in the deposition process is easily distinguished and is evenly distributed throughout the depth of the film at a concentration of 2 a/o. This is in agreement with the results of Morabito.<sup>6</sup> The oxygen peak located at 0.7 MeV results from a tantalum oxide surface layer, presumably  $Ta_2O_5$ , which formed on room temperature air exposure of the film after deposition. The high energy edge of this peak is energy-resolution limited while the low energy edge is not, indicating that the average spatial oxygen concentration is decreasing with greater depths in the film. The low-energy oxygen peak is associated with the substrate as BeO. The shape of the high-energy edge of the nitrogen peak indicates that nitrogen is depleted from the surface region but is probably distributed uniformly throughout the remainder of the film. This uniformity is difficult to ascertain since the resistive film thickness is only three times the depth resolution. A small surface carbon peak is shown, its presence was probably due to the breakup of residual hydrocarbons in the vacuum system caused by the ion beam bombardment. The integrated composition of the film is obtained from the elemental scattering

cross section.<sup>13</sup> The atomic percentage nitrogen, oxygen and argon in the film with respect to the total amount of tantalum in the film shown in Fig. 1 is 38%, 11%, and 2% respectively. For very low Z targets, e.g., beryllium, the elastic cross section is enhanced over the Rutherford cross section, but this is not the case for nitrogen.<sup>16</sup>

Because of possible differences in films deposited on different types of substrates it was necessary to show that as near as possible the films deposited on the beryllium substrates and those deposited on the alumina substrates were the same. The backscattering spectra taken on the alumina substrates appeared similar to that in Fig. 1 except the aluminum yield from the alumina substrate occurred at 1.08 MeV and together with the oxygen in the substrate obscured all information about the surface oxygen layer and the nitrogen incorporated into the film. However, the tantalum and the argon in the spectrum are well resolved. The measured film thicknesses as determined by ion backscattering for all substrates, alumina and beryllium, were the same for each deposition and the distribution of argon and the amount of argon incorporated into the films were the same within an experimental error of 2%. It was therefore assumed that the films deposited on the beryllium substrates were the same as those deposited on the alumina substrate and had essentially the same compositional content.

Ion backscattering spectra were taken on each of the beryllium substrates before and after stabilization to measure the growth of the surface oxide layer. It was found that the amount of oxygen in the surface layer increased by approximately a factor of two for all deposition runs. The oxide layer appeared to become more dense with oxygen and only increased slightly in depth. One cannot determine the thickness of the surface oxide layer from the width of the oxygen peak since it is energy-resolution limited, but one can deter-

mine an average thickness from the area under the oxygen peak. In order to obtain a thickness in Å it is necessary to make two assumptions: (i) the stoichiometry for this oxide layer, namely  $Ta_2O_5$ , and (ii) its density,  $8.73 \text{ gm/cm}^3$ . Using these assumptions the oxide thickness layers on the films before stabilization were determined to have an average thickness of  $40 \text{ Å}$  and varied from  $35 \text{ Å}$  to  $45 \text{ Å}$ . After stabilization this oxide layer had grown to a thickness of about  $80 \text{ Å}$  and varied between  $75\text{-}85 \text{ Å}$ . For all of the films investigated, it appeared as though the nitrogen was rather uniform with depth, but depleted somewhat from the surface region, i.e.,  $\sim 50 \text{ Å}$ . Morabito has found that nitrogen is homogeneous beneath the surface oxide layer.<sup>6</sup>

It is important to point out the distinction between the film thickness and the resistor thickness. The film thickness being the resistor thickness plus the surface oxide layer thickness. The nitrogen content of the resistor film is determined not by the total amount of tantalum present in the film, but by the amount of tantalum available for nitriding, i.e., the total amount of tantalum minus that associated with the surface oxide (assumed to be  $Ta_2O_5$ ). All backscattering data were reduced in this manner. The uncertainty associated with the nitrogen content varies with the absolute nitrogen content and is mainly due to counting statistics:  $\pm 5\%$  for the highest nitrogen content of  $Ta_2N_{1.07}$  and  $\pm 16\%$  for the lowest nitrogen content of  $Ta_2N_{0.5}$ . Fig. 2 shows the relationship between the nitrogen flow rate in the deposition system in the nitrogen content in the resistor film. Within experimental error the dependence is linear and a best-fit straight line has been drawn to guide the eye. Fig. 3 shows the sheet resistance vs nitrogen content and also the resistance values of the defined resistors vs nitrogen content. The correlation between the two is quite good.

In Fig. 4 is shown the dependence of the differential Seebeck potential on the nitrogen content in the resistor film. Within experimental error the dependence is linear and a best-fit straight line has been drawn to guide the eye. The probable error for the differential Seebeck potential was set equal to the rms deviation and was  $< \pm 1\%$  for all deposition runs. A recent work by Trudel correlated nitrogen partial pressure in the deposition chamber with Seebeck ratios.<sup>17</sup> Qualitative agreement with the present results and those of Trudel was found.

The resistivity vs. nitrogen content is shown in Fig. 4. Resistivity is a bulk property and therefore to determine it from the sheet resistance, one must account for both the resistive film thickness and its roughness caused by the substrate roughness. The thickness was determined by measuring the energy width of the tantalum peak and applying Bragg's Rule<sup>18</sup> to the stopping cross sections<sup>19,20</sup> and accounting for the various constituents of the film. This yielded an areal density of tantalum in the film. Taking only that portion of the tantalum associated with the nitrogen and assuming a density for tantalum nitride of  $15.8 \text{ grams/cm}^2$  a thickness in Å was obtained. The effect of surface roughness was taken into account by noting the increase in sheet resistance of films deposited on alumina substrates over that for single crystal  $\text{Al}_2\text{O}_3$  (sapphire) in the same deposition. This effect was measured in a separate experiment. This correction was determined by averaging the difference obtained from 12 deposition runs and was  $25 \pm 1\%$ . The resistance increased with increasing surface roughness. A measure of the surface roughness could be made by observing the slope of the trailing edge of the tantalum peak in the ion backscattering spectra. The slope was very high for both the polished beryllium and single-crystal  $\text{Al}_2\text{O}_3$  but somewhat less for the alumina substrate. The resulting

dependence of resistivity on nitrogen content is linear within the experimental uncertainty which is shown for all data points. The rms deviation for the sheet resistance was less than  $\pm 1\%$ , but due to the assumption of a constant density, the  $\pm 5\%$  error for the stopping cross sections and the correction for film roughness, the experimental uncertainty for the resistivity is estimated to be  $\pm 5\%$ . Previous experiments by Gulner<sup>7</sup> and Waterhouse<sup>8</sup> have correlated film resistivity and nitrogen content. Both experiments used some form of chemical analysis to determine nitrogen content. The results of Gulner are shown in Fig. 4 and agree quite well with the present results while those of Waterhouse are about 60% higher and are not shown.

In most microelectronic circuit applications it is desirable to have resistors which have a small temperature coefficient of resistance, TCR, defined as  $1/R (dR/dT)$ . The TCR can be useful for film characterization and it can be important in circuit functions where ambient temperature changes would cause unwanted changes in circuit performance. The studies relating TCR to nitrogen content reveal that it is a strong function of nitrogen content below  $[N]/[Ta] = 0.35$ , but remains almost constant throughout the range 0.35 - 0.55 as shown in Fig. 5. The units of TCR are parts per million (ppm) per degree centigrade, and values less than 100 ppm/ $^{\circ}C$  are normally considered acceptable. Thin film resistors may drift from their initial resistance values especially if they are operated at higher temperatures. This effect is expressed as percent resistance change per unit time. In reality the percent drift is not linear with time but logarithmic. Thin film resistors usually become more stable as they age partly due to self-limiting processes such as surface oxidation which forms a passivating layer. The results of the stability tests also shown in Fig. 5 indicate that the stability of the thin film resistors is independent

of the nitrogen content to within the experimental error. The stability is given as percentage change and resistance for 1000 hours at 150°C. Values for the stability less than 0.5%/1000 hr-°C are acceptable.

#### IV. DISCUSSION AND CONCLUSIONS

Brauer and Zapp<sup>21</sup> studied the tantalum nitride system using both x ray and chemical analytical methods and found only two stable stoichiometric tantalum nitrides:  $Ta_2N$  and  $TaN$ . The solubility of nitrogen in metallic tantalum was observed not to exceed 4 at.% and from this concentration through  $TaN_{0.35}$  mixed phases of  $Ta_2N$  and metallic tantalum were observed. They found that  $Ta_2N$  is homogeneous from  $TaN_{0.41}$  to  $TaN_{0.50}$ , while at greater nitrogen concentrations mixed phases of  $Ta_2N$  and  $TaN$  occur until  $TaN$  is reached. Based on the linear dependence of both the differential Seebeck potential and the resistivity on nitrogen content no abrupt change from a single phase to another single phase can be detected in the present study. However, the present results are not necessarily in conflict with the findings of Bower and Zapp since sputtered thin films are notorious for not being in thermodynamic equilibrium, i.e., they are multiphase and have numerous defects. Presumably a combination of at least two phases exist. The differential Seebeck voltage is an indication of the disparities of carrier concentration and their mobilities at the two temperature junctions. Thus the linear decrease of the differential Seebeck potential with increasing nitrogen content is indicative that barrier height is increasing with nitrogen content, i.e., the metallic character of the film is changing to a semiconductor character.

Our results lead to the following important conclusions: 1. A surface oxide layer grows on air exposure of the tantalum film at room temperature. Assuming the oxide is composed entirely of  $Ta_2O_5$ , then the equivalent oxide thickness is about 40 Å. After stabilization of the thin film resistors the surface oxide layer thickness increases to approximately 80 Å. Nitrogen was found to be somewhat excluded from this surface layer region. 2. The sputtering gas argon appears to be included uniformly throughout the film at a concentration of 2 at.%. 3. For sputtered thin tantalum nitride films both the resistivity and the differential Seebeck voltage ratio appear to exhibit a linear dependence on nitrogen content in the range  $0.25 < [N]/[Ta] < 0.55$ . Since the differential Seebeck potential is a strong function of nitrogen content, it is quite possible to use this parameter as a measure of nitrogen content when the remaining parameters in the deposition system remain constant. 4. The TCR of tantalum nitride film was found to decrease sharply below  $[N]/[Ta] = 0.35$  and to be relatively constant from 0.35 to 0.55. 5. The stability of the resistors was independent of nitrogen content in the range covered by this experiment. 6. Use of ion backscattering using low Z substrates can give absolute depth concentrations of light elements in thin films nondestructively.

#### ACKNOWLEDGMENT

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## FIGURE CAPTIONS

- Fig. 1 Energy spectrum of 2.0 MeV  $^4\text{He}$  ions backscattered from a reactively sputtered Ta-N film about 500-Å thick on a beryllium substrate. The low-energy side of the spectrum is magnified by a factor of 50. The shaded areas are the scattering yields of the elements in the film. The energies with which the incident ions are backscattered from the film elements Be and O. The edge labeled O(Ta) is associated with the Ta-N film as a surface oxide layer and the edge labeled O(Be) is associated with the Be substrate as BeO.
- Fig. 2 Nitrogen flow rate vs. nitrogen content in the resistive film. The tantalum content is that available for nitriding (i.e., excluding Ta surface oxide). (See text for further explanation.)
- Fig. 3 Sheet resistance and strip resistance vs. nitrogen content in the resistive film.
- Fig. 4 Differential Seebeck potential vs. nitrogen content.
- Fig. 5 Film resistivity vs. nitrogen content.
- Fig. 6 Thermal coefficient of resistance and stability vs. nitrogen content.

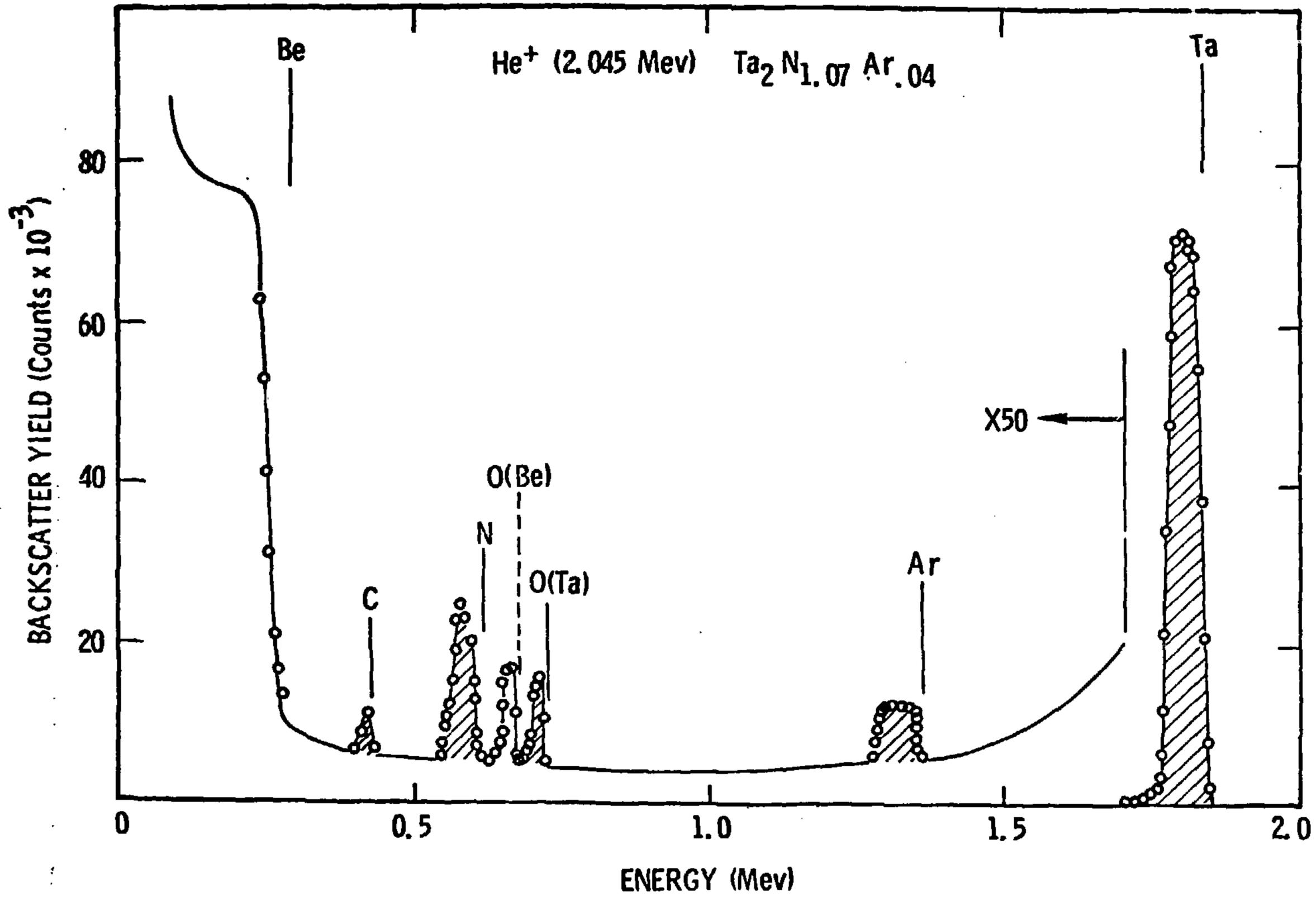


Fig. 1

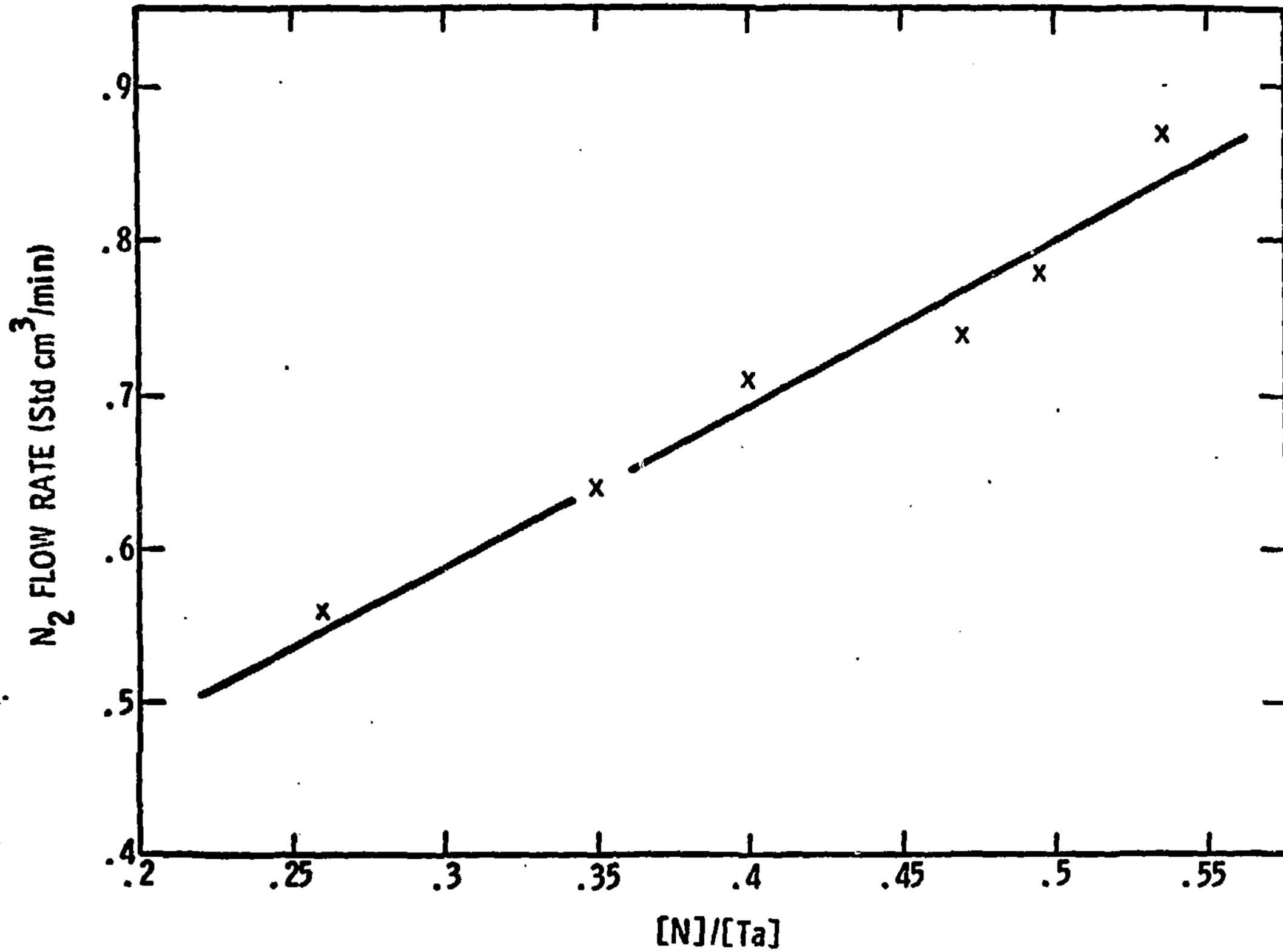
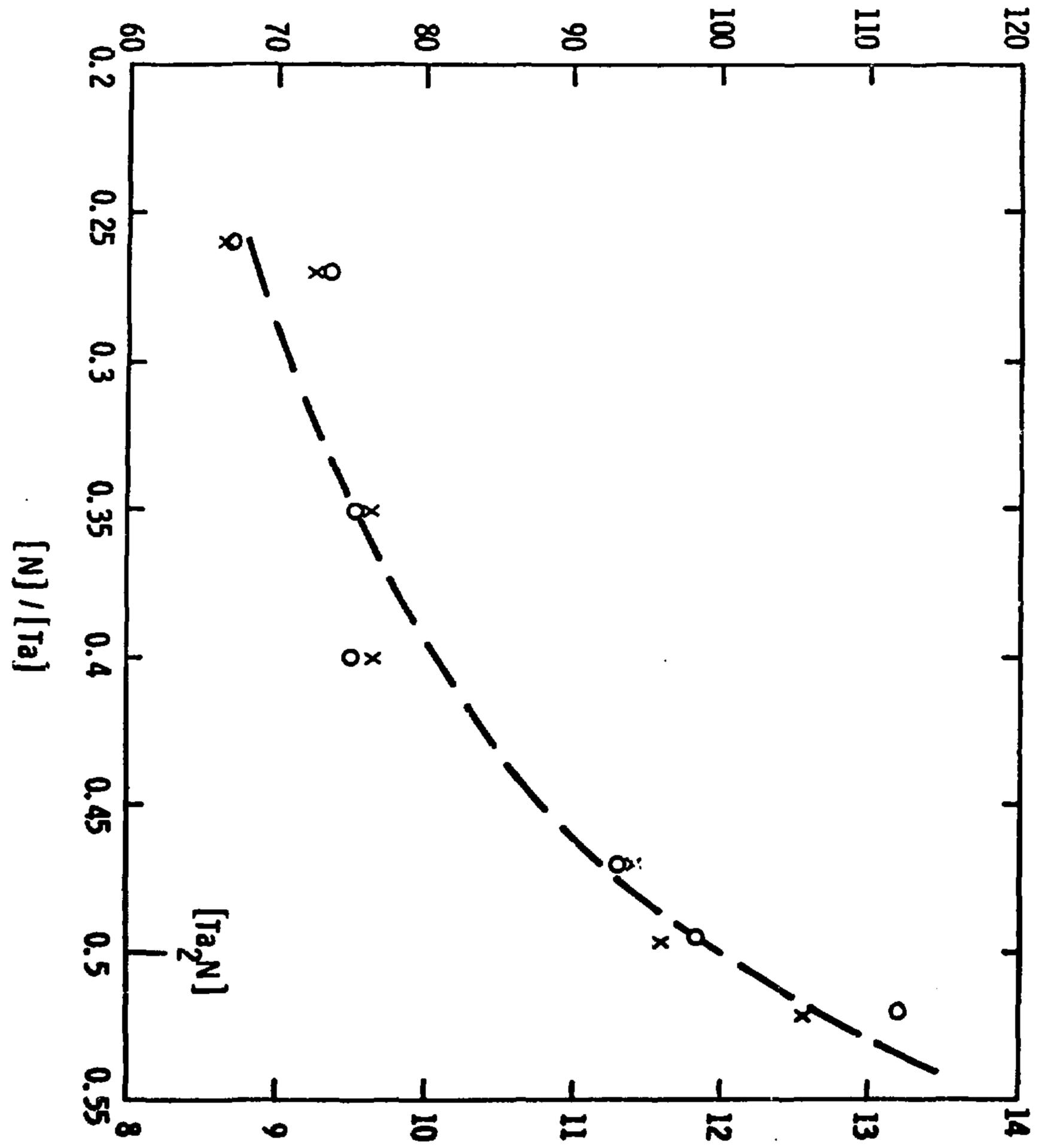


Fig. 2

SHEET RESISTANCE ( $\Omega/\text{sq}$ )



STRIP RESISTANCE ( $\text{K}\Omega$ )

Fig. 3

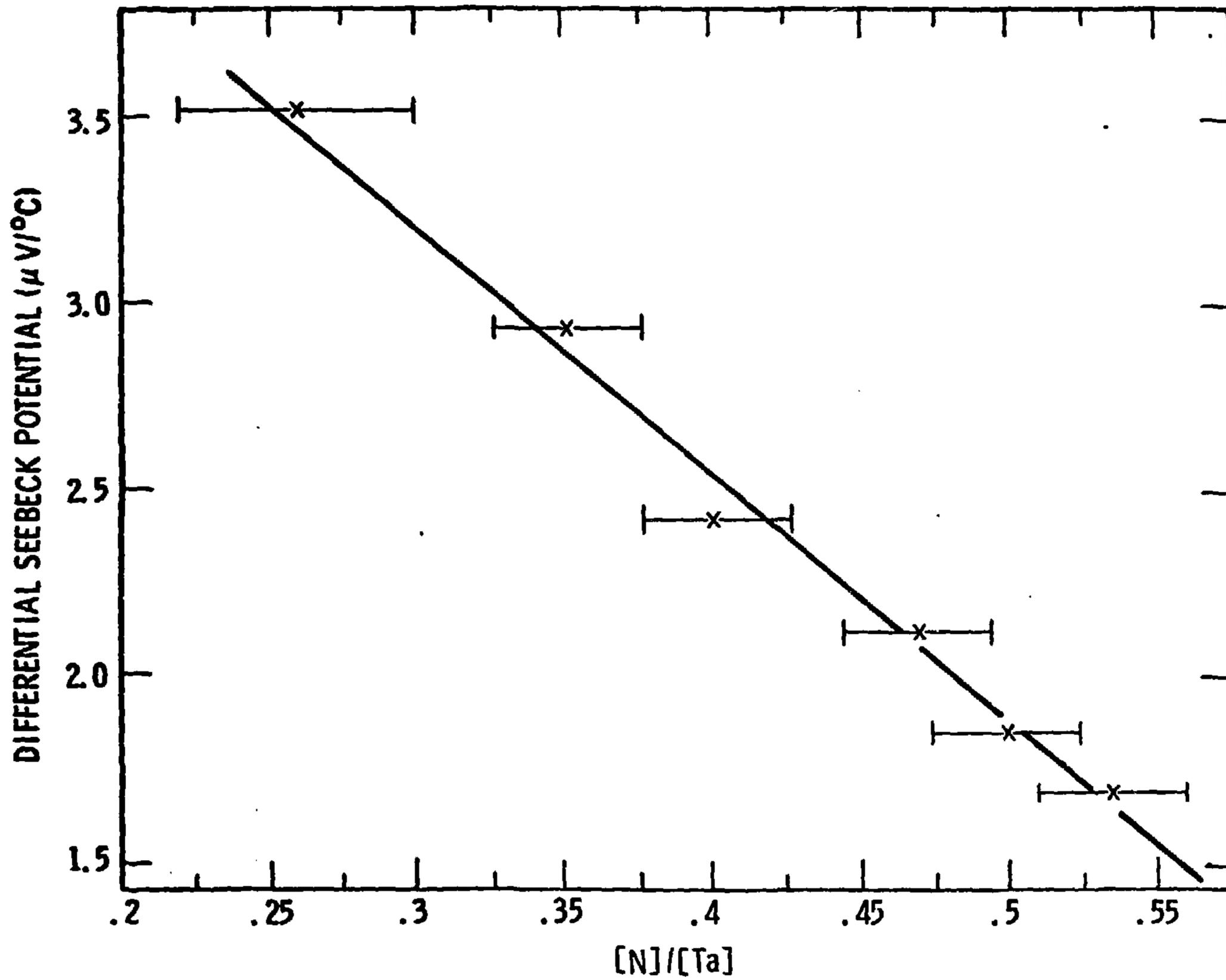


Fig. 4

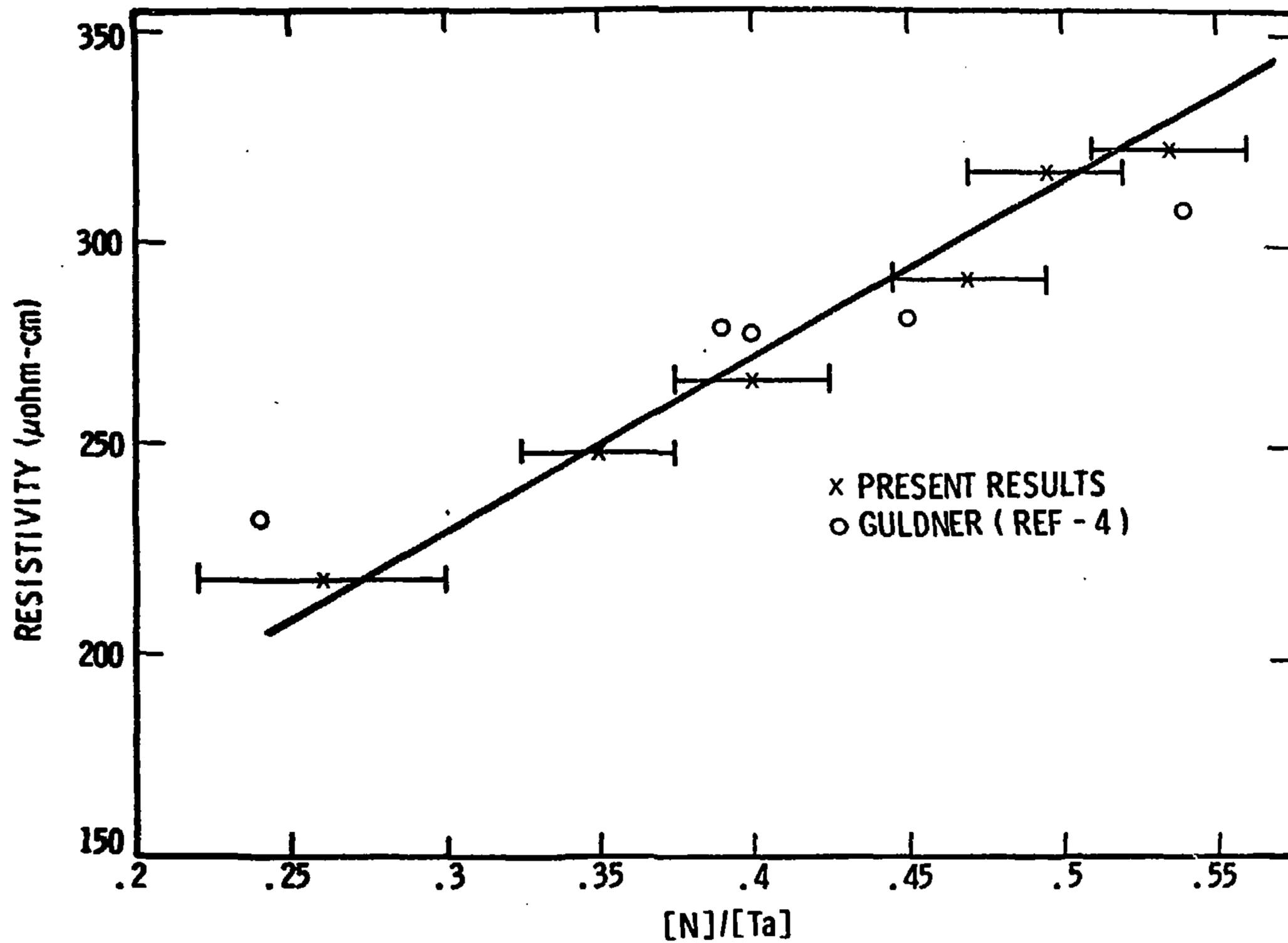


Fig. 5

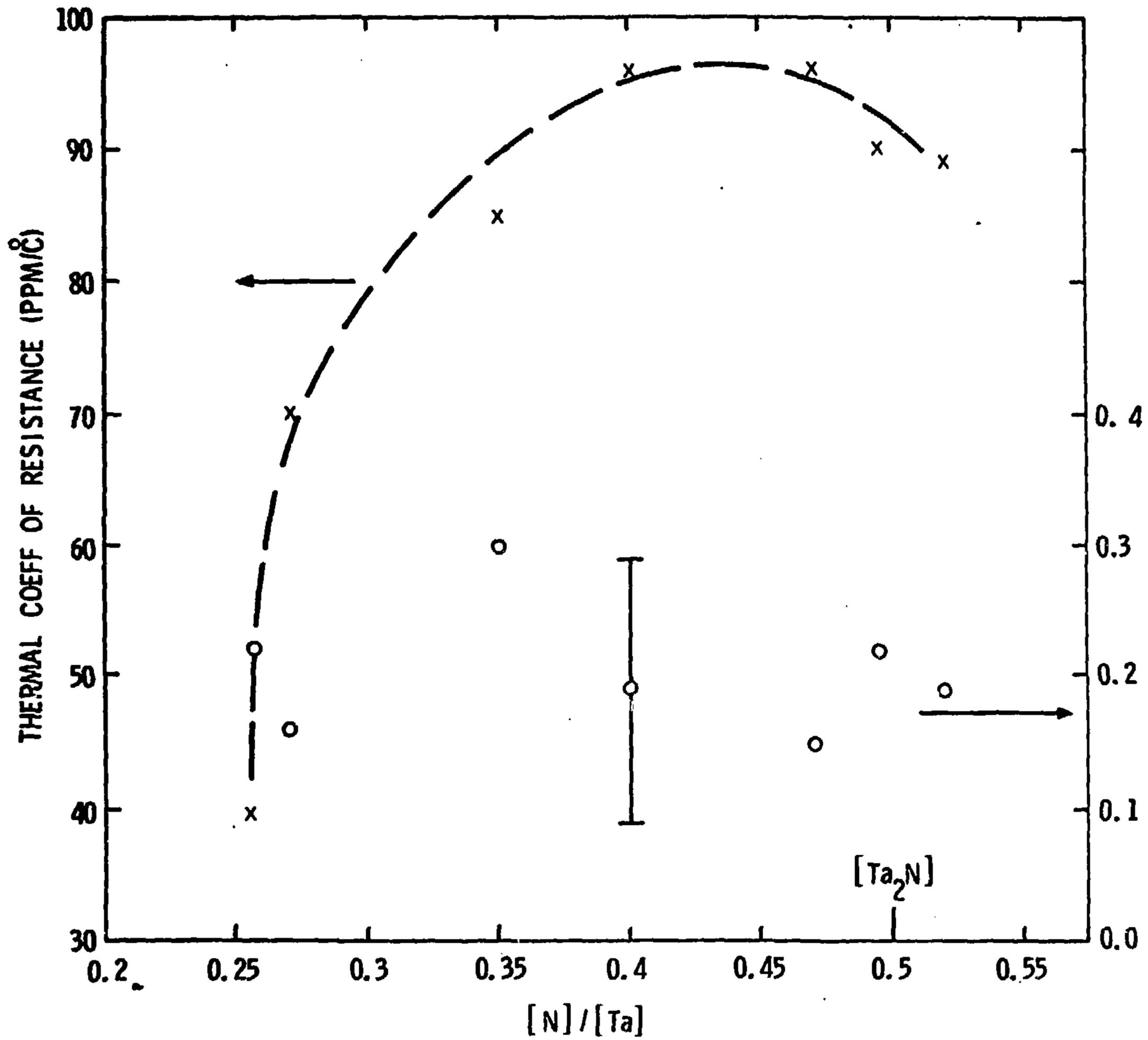


FIG. 6

STABILITY,  $\Delta R/R$ , 150°C for 1000 hrs. (%)