Solid-State Electronics Vol. 22, pp. 141–149

0038-1101/79/0201-0141/$02.00/0

SOLID PHASE FORMATION IN Au:Ge/Ni, Ag/In/Ge, In/Au:Ge GaAs OHMIC CONTACT SYSTEMS

A. CHRISTOU
Naval Research Laboratory, Washington, DC 20375, U.S.A.

(*Received* 10 *November* 1977; *in revised form* 21 *June* 1978)

Abstract—Solid phase epitaxy formation in Au:Ge/Ni, Ag/In/Ge, and In/Au:Ge contacts to GaAs has been identified utilizing micro-spot Auger spectroscopy and selected area electron channeling. It is shown that the lateral extent of solid phase formation directly controls the value of the specific contact resistivity. Solid phase growth occurs as a result of dissolution of GaAs by the contact constituents in the vicinity of the eutectic temperature. Solid phase growth also results in regions free of oxide layers and contaminants and hence a lower contact resistivity.

INTRODUCTION

The reliability of GaAs microwave devices is directly related to the integrity of the ohmic contacts[1, 2]. Industrial suppliers of GaAs devices such as field effect transistors have standardized on the use of Au/Ge or Ag/Ge based ohmic contacts mainly due to their initial low contact resistance. However, the reliability and long term stability of these contacts has not been clearly established. The major problems in GaAs ohmic contacts are[3, 4]: lack of uniform wetting of the metals to the GaAs, metal segregation at the metal-semiconductor interface, the extent of an epitaxial surface layer, surface roughness and the presence of a multitude of semiconductor-metal phases. It has also been shown[4, 5] that micro-pricipitates do exist in Au:Ge/Ni contacts containing an uneven distribution of Ni and Ge. In addition to the major problems in forming low resistance contacts to GaAs, work elsewhere has established that Au:Ge/Ni contact resistance increases with increasing temperature, time and bias[5, 6]. We report in the present investigation the identification of solid phase epitaxy regions in Au:Ge/Ni, Ag/In/Ge and In/Au:Ge contacts to GaAs. It will be shown that the extent of solid phase formation directly controls the value of the specific contact resistivity. The hypothesis of solid phase epitaxy in ohmic contacts has been proposed previously[7] but to date no direct experiments have been performed to verify this. We will show in the present investigation that the minimum specific contact resistivity, (r_c) occurs for optimum solid phase formation conditions. Unique to this study is the utilization of high spatial resolution Auger spectroscopy (μAES) and sputter profiling to identify micro-segregation and solid phase formation in ohmic contacts. With μAES, surface chemical changes can be analyzed with a spatial resolution of 1000 Å[8]. Microsegregation effects in these contacts has been studied as a function of contact alloying parameters. In addition, the crystallographic orientation of epitaxial regions was determined by selected area electron channeling in the scanning electron microscope.

EXPERIMENTAL CONDITIONS

Ohmic contact specimens were formed on (100) $n/n+$ GaAs wafers having a 2.0 μm thick epitaxial n layer ($n = 2\times 10^{16}\,cm^{-3}$). Square metal contact regions were defined by selective etching. Each wafer consisted of 10–15 contact squares of areas 10^{-5} to $2\times 10^{-3}\,cm^2$ for electrical evaluation. The Au:Ge/Ni contacts consisted of 1600–1700 Å of Au:Ge eutectic followed by 400 Å of Ni overlay. In/Au:Ge samples were formed by first depositing 400 Å of In followed by 1700 Å of AuGe. Thicker In/Au:Ge contacts were formed by depositing Au:Ge to a thickness of 4 μm. A layered structure of Ag/In/Ge of total thickness of 2000 Å was also deposited in order to form Ag/In/Ge contacts. The compositions of the In/Au:Ge and Ag/In/Ge contacts were 90 wt/% Au or 90 wt/% Ag, 5 wt/% Ge and 5 wt/% In, with a total thickness of 2000 Å. Sintering of each sample was accomplished either in an open tube furnace (N_2 or forming gas atmosphere) or in a vacuum tube furnace located within an ultra-high vacuum system. A typical annealing scheme for Au:Ge/Ni is shown in Fig. 1. All tem-

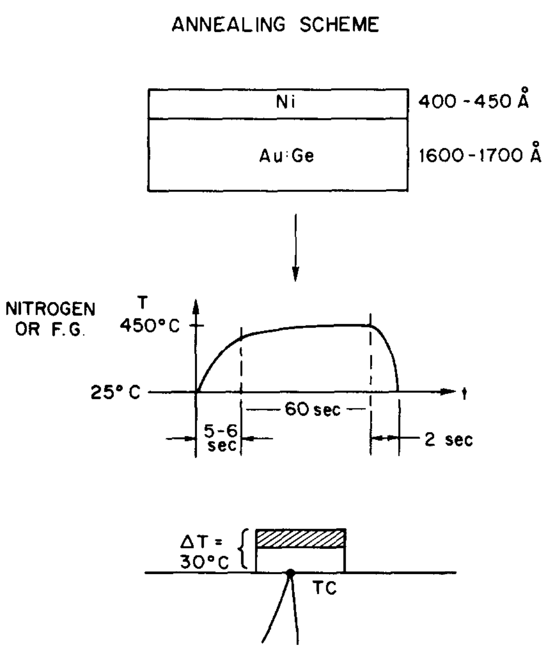

Fig. 1. Schematic showing annealing scheme for the Au:Ge/Ni–GaAs contact. All temperatures have been verified by pyrometric measurements.

peratures have been checked and compared with the temperatures recorded by an I.R. pyrometer.

The specific contact resistance, r_c, was measured using a modified form of the Cox and Stack[9] method. The total resistance of a square contact of side a in length is given by

$$R_T \simeq \frac{r_c}{a^2} + \left(\sqrt{\left(\frac{\pi}{4}\right)}\right)\frac{\rho}{\pi a} \arctan\left(4\sqrt{\left(\frac{\pi}{4}\right)}\frac{h}{a}\right) + R_0$$

where ρ is the resistivity of the epi material of thickness h and R_0 is the resistance of the back contact, n^+ substrate and probe resistance. The second term represents the spreading resistance term which is only weakly dependent on the exact shape of the contact area.

Ohmic contact surfaces were examined utilizing the μAES technique. Auger electron spectroscopy with a focused electron beam in a high resolution scanning electron microscope (SEM) has provided an extremely useful diagnostic technique for GaAs surfaces. The focused electron beam in a SEM allows one to determine surface topograph of the specimen which can be correlated with the Auger spectra of selected areas. The AES technique has been shown to have a surface resolution of 1000 Å at a beam current of 5×10^{-8} amps necessary for AES analysis[7, 8].

EXPERIMENTAL RESULTS

Microsegregation effects were observed on the three contacts investigated over the temperature region which resulted in ohmic behavior. For discussion purposes, we define solid phase epitaxy as the presence of structures which form at a sintering temperature slightly below the eutectic temperature and which have the same orientation as the substrate[10]. Precipitation structures are those which form at sintering temperatures above the eutectic temperature. Table 1 summarizes the eutectic temperatures employed in the present investigation. The ternary eutectic temperatures were determined by resistivity techniques at the Naval Research Laboratory and will be reported in a separate publication.

Table 1. Critical eutectic temperatures and compositions of GaAs contacts

	GaAs Ohmic Contacts Au:Ge/Ni	Ag/In/Ge	In/Au:Ge
Binary Eutectic	363°C	651°C	363°C
Temperature	(Au:Ge)	(Ag:Ge)	(Au:Ge)
Ternary Eutectic	425°C	651°C	520°C
Temperature	(AuGeNi)	(AgGeIn)	(AuGeIn)

(a) *The* Ag/In/Ge *system*

The Ag/In/Ge contacts exhibited solid phase facet growth up to sintering temperature of 650°C with a typical (100) orientation. Figure 2 shows facet growth and μAES analysis of Ag/In/Ge, indicating the presence of epitaxial (100) oriented Ge/In regions ("square particles"). The (100) particles are Ga–In–Ge–As regions with surface composition as indicated by the "square particle" analysis. After sputtering for approximately 20 minutes, the particles become Ge rich. The "agglomerated regions" shown by the arrow on the right, are Ag particles with adsorbed sulfur. In both the matrix (region surrounding the "square particles") and the epitaxial square particles, Ge is shown to have migrated into the GaAs substrate. By scanning the electron beam over all areas of interest a complete map of elements as a function of depth and lateral extent has been obtained and is shown in Fig. 3. Nonuniform sputtering effects were not observed. Also shown is the selected area electron channeling insert indicating that the "square regions" have a (100) orientation as does the GaAs substrate and are aligned in the [110] direction. The sinter temperature range for which solid phase epitaxy was observed was determined and correlated with variation in specific contact resistivity, r_c. Figure 4 shows that the minimum r_c of $10^{-5}\Omega\text{-cm}^2$ was attained within the temperature region (620°C, 1 min) for solid phase formation which is below the eutectic temperature of 651°C for Ag–Ge. It is suggested in Fig. 4 that the extent of solid phase formation in Ag/In/Ge contacts also controls the contact resistance. Increases in r_c were only observed when the microstructure morphology changed from oriented to random precipitation above 640°C.

The extent of solid phase epitaxy (as determined by dimensions of the "square regions" was also determined as a function of sinter temperature and specific contact resistivity. For purposes of uniformity, the square particles will be referred to as solid phase particles. A variation in average solid phase particle size from less than 0.5 to 2 μm was attained by varying sintering temperature while maintaining the sinter duration at 60 seconds. The results summarized in Fig. 5 indicate an excellent correlation between the 2.0 μm particle size and the low contact resistance samples, with the average number of particles per mm^2 being constant. A degraded

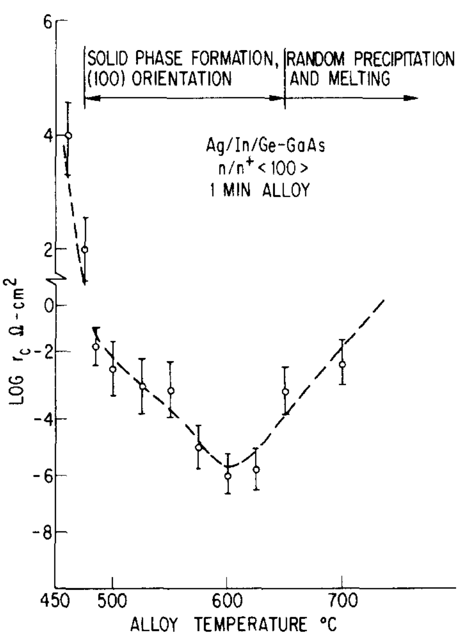

Fig. 4. Variation of log r_c with alloy temperature for constant one minute sintering times. Random melting occurred above the ternary eutectic temperature.

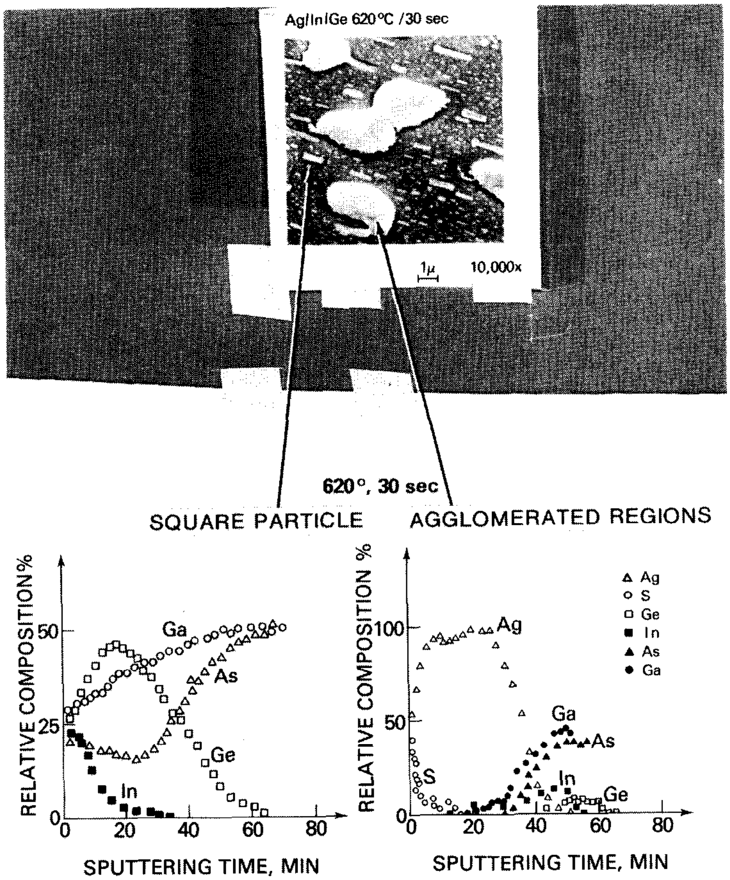

Fig. 2. Micro-spot SES profiles of Ag/In/Ge–GaAs contact system, showing "square" epitaxial particles and agglomerated regions.

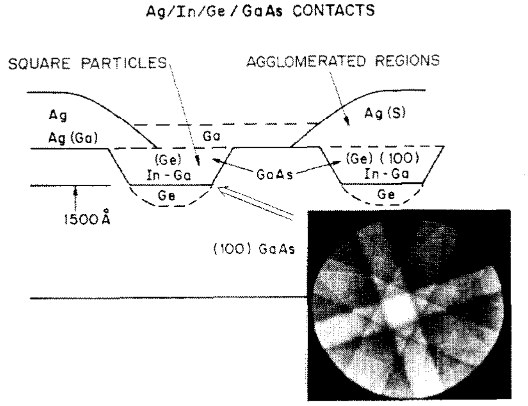

Fig. 3. Cross section of Ag/In/Ge contact with selected area channeling pattern insert of solid phase epitaxial region. The channeling pattern is typical of a square particle showing a (100) orientation.

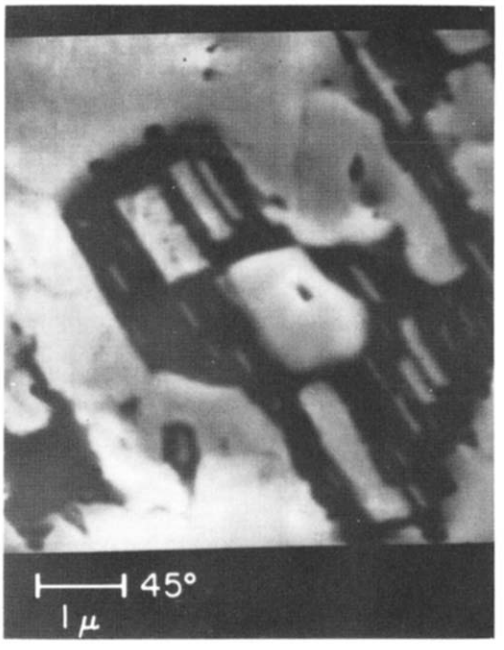

Fig. 6. Solid phase epitaxy of Au:Ge/Ni contacts is indicated by the rectangular regions. The contact was formed by sintering at 450°C for one minute.

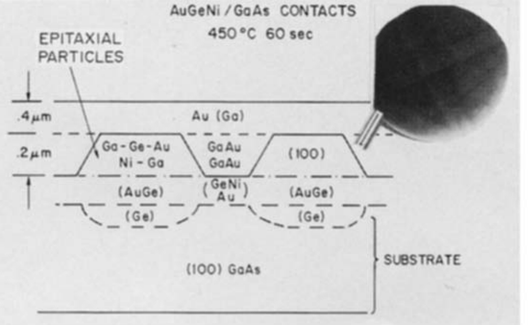

Fig. 10. Cross section of Au:Ge/Ni contact with selected area channeling pattern showing a (100) orientation.

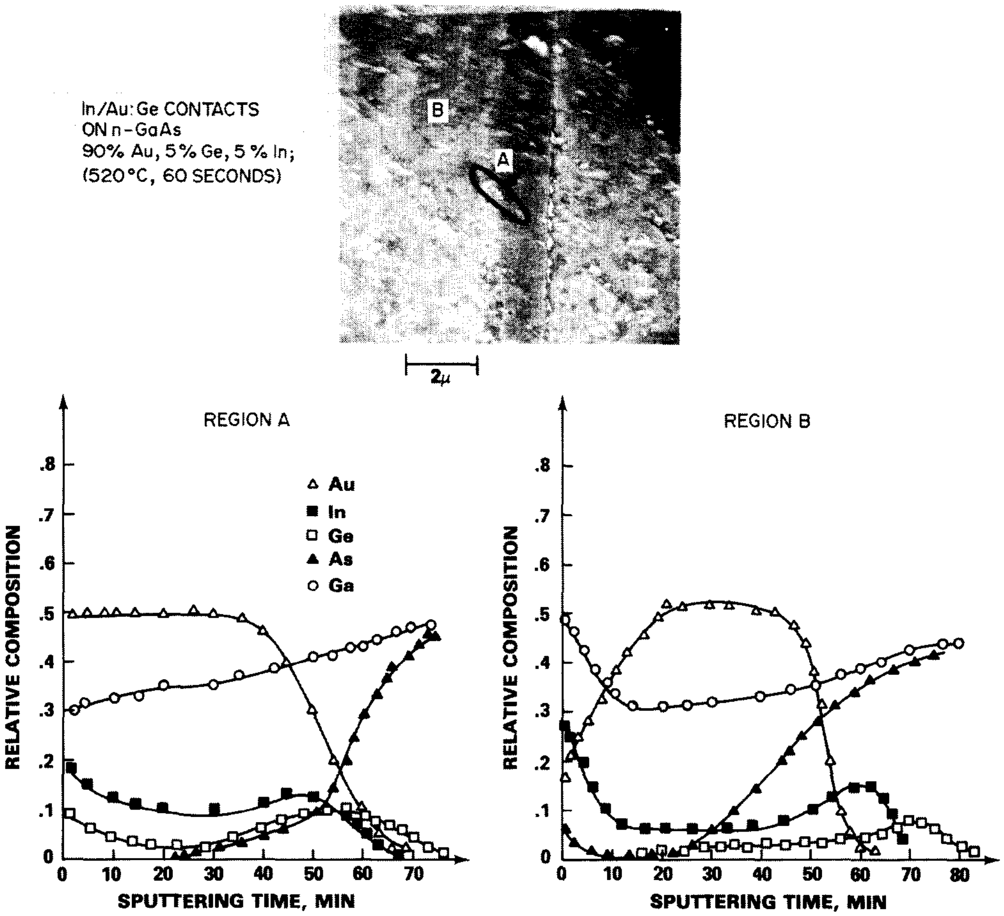

Fig. 11. Micro-spot AES sputter profiles of In/Au:Ge–GaAs contacts.

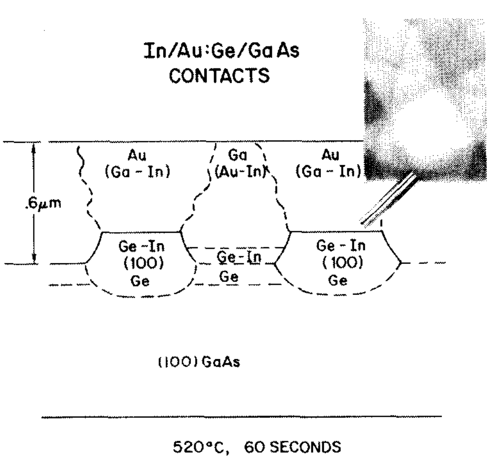

Fig. 12. Cross section of In/Au:Ge–GaAs contact with channeling pattern insert showing (100) orientation.

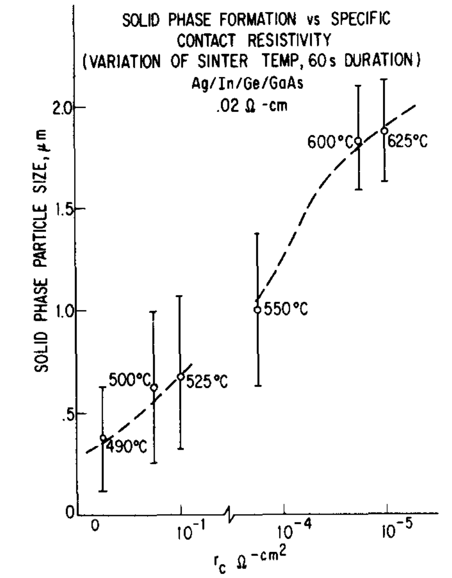

Fig. 5. Variation of solid phase particle size with specific contact resistivity, r_c.

contact resistance of $10^{-1}\Omega\text{-cm}^2$ was observed for specimens with solid phase particle size of $\simeq 0.5\,\mu\text{m}$. The results for Ag/In/Ge contacts summarized in Figs. 2–5 show that the extent of solid phase formation directly controls the value of the specific contact resistivity. The minimum r_c occurs for optimum solid phase formation conditions.

(b) *The* Au:Ge/Ni *contact system*

The Au:Ge/Ni contacts displayed typical indications of solid phase formation with facet growth of Ni–Ga and Ni–Ge in Au films having the same orientation as the (100) substrate and aligned in the [110] direction. A typical solid phase epitaxy growth structure at the Au:Ge/Ni–GaAs interface is shown in Fig. 6. The rectangular aligned regions typical of solid phase formation were observed at temperatures well below the eutectic temperature. These rectangular regions will be referred to as solid phase epitaxy regions. Auger electron spectroscopy analysis of the (100) oriented rectangular regions (7a) and the surrounding surface area (7b) is shown in Fig. 7. The sputter profiles of the solid phase epitaxy regions indicate a significant increase in nickel concentration at the contact-substrate interface (50–70 min of sputtering) probably in the form of Ni–Ga and Ni–Ge compounds. These compositions are evidence that the metal Ni is transported through the Au:Ge to the GaAs surface where (100) oriented nickel rich regions form in a fashion similar to a previously described solid phase epitaxy process[10]. The surface composition of regions surrounding the solid phase epitaxy consists of Au–Ga solid solutions as shown in Fig. 7.

The variation of r_c with alloy temperature for Au:Ge/Ni was found to depend directly on the presence of solid phase epitaxy regions. Figure 8 indicates the variation of log r_c with alloy temperature showing three different microstructure morphologies with a degradation in r_c observed only in the regions of random facets or random precipitation (above the eutectic temperature). The size of the solid phase epitaxial regions was found to vary from less than 1 μm (350°C) for $r_c = 10^{-1}\Omega\text{-cm}^2$ to 3.0 μm for $r_c = 10^{-6}\Omega\text{-cm}^2$ at 425°–450°C. Figure 9 which shows the variation of particle size with r_c indicates the presence of a particle size plateau at 3.0 μm and 420°–450°C which corresponds with the temperatures for attaining the lowest contact resistivity. This experiment provides further empirical evidence that the greater the extent of solid phase formation is, the lower is the resultant contact resistance.

The Au:Ge/Ni contact formed by annealing at 450°C for 60 sec. was further analyzed by μAES sputter profiling in order to determine the lateral and depth distribution of Au, Ge, Ni, Ga and As. A schematic of the results is summarized in Fig. 10, and indicates the presence of Ge in the GaAs substrate. Germanium migration into GaAs has been hypothesized[7] as the

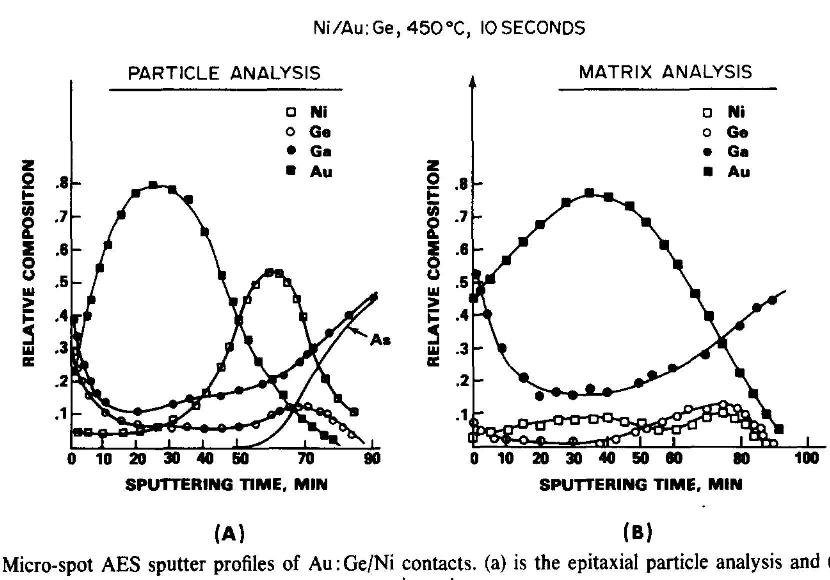

Fig. 7. Micro-spot AES sputter profiles of Au:Ge/Ni contacts. (a) is the epitaxial particle analysis and (b) is the matrix region.

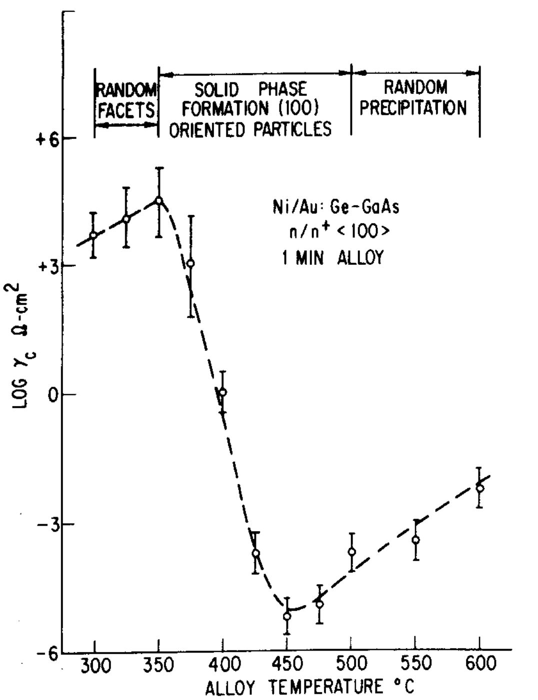

Fig. 8. Variation of log r_c with alloy temperature for constant one minute sintering times.

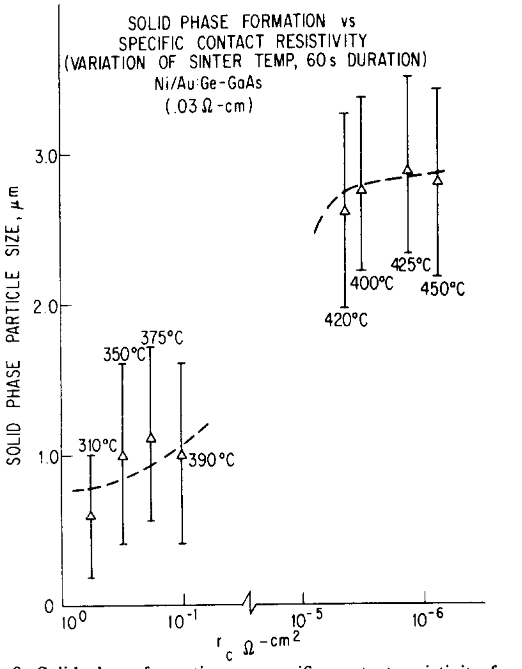

Fig. 9. Solid phase formation vs specific contact resistivity for Au:Ge/Ni contacts. The low contact resistivity was obtained for samples with the largest solid phase epitaxy area.

process for increasing impurity concentration necessary for attaining a low contact resistivity. In addition, Ga has outdiffused to the Au overlay region and is shown to be present at the same concentration level as Au in the regions between the epitaxial particles. The AuGe, GeNi, Au and Ge in parentheses denotes that these were minor constituents only with GaAs being the major component. The selected area channeling pattern insert of the epitaxial regions is also shown in the figure. The fourfold symmetry of the channeling pattern is indicative of a (100) orientation.

(c) *The In/Au:Ge contact system*

The In/Au:Ge system also exhibited solid phase epitaxy formation at sintering temperatures of 400°–530°C (1 min sinter duration). Elongated faceted regions were observed on the top surface of the 0.6 μm thick contact and epitaxial regions were observed at the contact—GaAs interface. The epitaxial (100) regions consisted of Ge–In in addition to Ga at approximately 40% concentration. The decrease in As concentration at the interface has been accompanied by an increase in Ge, in concentration as shown in Fig. 11. In both regions *A* and *B*, Ga outdiffusion to the contact surface has been observed. Region *B* also shows 7% As on the contact surface. The migration of Ge into the GaAs substrate was observed in both the *A* and *B* regions and seems to be a general phenomenon with all sintered GaAs contacts.

Lateral and depth profile mapping results of the In/Au:Ge contact are shown in Fig. 12. The epitaxial (100) regions consist of equal concentrations of Ge and In. A thinner layer of Ge–In was also found in the region surrounding the epitaxial particles. The inserted selected area electron channeling pattern taken after sputter etching to the contact–GaAs interface indicates that the solid phase regions are single crystal with a (100) orientation. Channeling patterns of the matrix region indicated the presence of polycrystalline material. Outdiffusion of Ga and In is also shown to be present throughout the Au region of the contact.

DISCUSSION AND CONCLUSIONS

The experimental results show that Au:Ge/Ni, Ag/In/Ge and In/Au:Ge contacts to GaAs undergo precipitation and solid phase formation over a temperature region which corresponds to ohmic behavior. The extent of solid phase formation is directly correlated with the value of the specific contact resistance. "Solid" phase growth occurs as a result of the dissolution of GaAs in the vicinity of the eutectic temperature. As the ohmic contact composite structure is brought up to a temperature below the eutectic melting temperature, dissolution of the GaAs will take place until the solubility limit is reached. The composite structure, when cooled undergoes precipitation and growth of the dissolved atoms onto the substrate. The epitaxial layers thus formed always incorporate either Ni or In and Ge. The cleanliness of the substrate/metal interface will determine the ultimate extent of the epitaxial layer. If the interface is not free of any contaminants, such as surface oxides, the epitaxial layer will not be laterally uniform.

The driving force for solid phase growth in GaAs ohmic contacts is the reduction in chemical free energy[10] between the amorphous or near amorphous state of the Au:Ge and the crystalline epitaxial state. Once Au:Ge and Ni have undergone interdiffusion and crystallization on the substrate, further growth occurs because of the difference in chemical potential between the regrown crystallities and the substrate. In general, the transport mechanism for epitaxial growth is hypothesized to be precipitation of crystals out of a supersaturated solid solution. Experiments are underway to determine the transport mechanism for the Au:Ge/Ni system.

In summary, the present investigation has clearly established a correlation between the extent of "solid" phase epitaxial growth and specific contact resistivity.

Acknowledgements—The investigation was supported by the Office of Naval Research and Naval Electronic Systems Command.

REFERENCES

1. A. M. Andrews and N. Holonyak, *Solid-St. Electron.* **15**, 601 (1972).
2. G. Y. Robinson, *Solid-St. Electron.* **18**, 331 (1975).
3. C. R. Paola, *Solid-St. Electron.* **18**, 1189 (1970).
4. P. C. Moutou, J. J. Godard, J. M. Montel and B. Dixneuf, 1975 *Cornell Conf. Proc.*, p. 225 (1975).
5. H. M. Macksey, Gallium arsenide and related compounds 33b. 254 (1976) *Inst. of Physics Conf. Series.*
6. A. Christou and K. Sleger, 1977 *Cornell Conf. on Active Microwave Devices*, Ithaca, N.Y. (August 17–19, 1977).
7. T. Sebestyen, H. L. Hartnagel and L. H. Herron, *IEEE Trans on E.D.* **ED-22**(12), 1073 (1975).
8. A. Christou, *J. Appl. Phys.* **47**(12), 5464 (1976).
9. R. H. Cox and H. Stack, *Solid-St. Electron.* **10**, 1213 (1967).
10. D. Sigurd, G. Ottaviani, V. Marrello, J. W. Mayer and J. O. McCaldin, *J. Non-Cryst. Solids* **12**, 135 (1972).