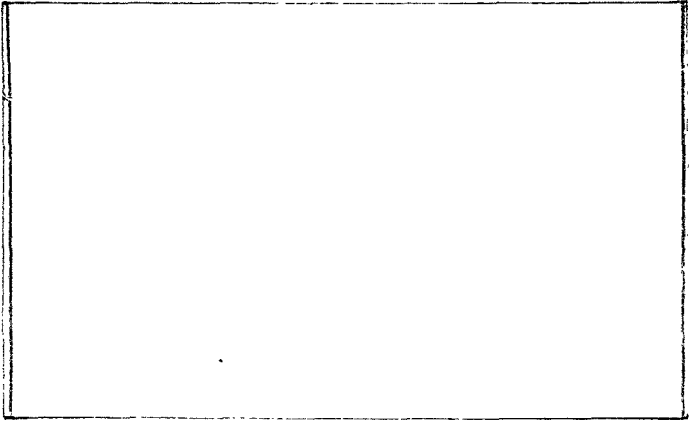


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LEAR SIEGLER, INC.

RESEARCH LABORATORIES

SANTA MONICA, CALIFORNIA

SEMICONDUCTOR THIN FILMS

CONTRACT NO. AF33(657)-7623

Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio

QUARTERLY REPORT NO. 5
November 1, 1962 to March 31, 1963

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1.0 PREVIOUS PLANS FOR FIFTH PERIOD

- 1.1 To construct a small glass system in which ultra high vacuum would be obtained by gettering or cryosorption.
- 1.2 To investigate the effect of annealing on GaAs films.
- 1.3 To make Hall mobility measurements at room temperature and to set up for low temperature measurement.
- 1.4 To deposit GaAs on BeO and to continue the search for CeO_2 and other substrates.
- 1.5 To continue improvement of electron microscopy techniques.
- 1.6 To continue improvement of the GaAs evaporation source.
- 1.7 To investigate deposition at microns per second.
- 1.8 To continue device exploration using films of available quality.



2.0 ACCOMPLISHMENTS

2.1 The tube source shown in Figure 1 of Quarterly Report No. 4 (QR4) gave thinner films for a given amount of feed than did an open boat. A new source was built (Figure 1) which gave deposits about as thick per gram evaporated as did the boat but with much less powder sticking to the substrate.

2.2 Hall measurements were made using the circuit shown on page 5 of QR4.

Sample	Type	r ohm cm	R _s cm ³ /coul.	μ cm ² /vs	T °C
J546-2P	p	4.5	21	4.6	room
J556B	p	2.5	63	25	room
J549A	p+	0.6	34	61	room
J552B	p	6.2	85	13.6	22
J547A	p	22.4	55	2.5	22
		140			- 195
J559B	p	5.7	80	12.8	22
		54	150	2.8	- 195

Better contacts are needed. The present plan is to fire silver pads (to which wires may be soldered) onto the substrate before depositing the semiconductor and then connect the pad to p or n semiconductor by vacuum depositing In or Sn respectively.

The circuit elements B₂, D, V, and R₆ have been replaced by a Kiethly Model C 660 dc differential voltmeter with null adjustment and full scale readings from 0.1 mv to 500 v; B₁ has been replaced by an Electronic



Measurement Model C 636 constant current supply, 0.0015 ma to 220 ma.

Sample geometries are shown in Figure 2.

2.3 Attempts at annealing GaAs were unsuccessful because at 700° and 600° the GaAs evaporated before any improvement of the film could be effected. Additional attempts will be made by covering the GaAs with a deposited film of CaF_2 , SiO , or SiO_2 before annealing.

2.4 A simple ultrahigh vacuum system was built and used for deposition of germanium (Figure 3). Five depositions were made.

In run No. 1, the system, attached to the Veeco 4 inch vacuum system, was outgassed by placing the 8" x 1" silica tube in a tube furnace and heating to about 1000°C (temperature estimated by eye). In the tube was a fused silica supporting framework, a tungsten filament loaded with Ge, a tantalum sheet with hooks supporting a CaF_2 substrate, and barium getters designed for use in vacuum tube manufacture. One of two barium getters partly evaporated during the outgassing. When the pressure had dropped to 2×10^{-6} torr, after about 14 hours of outgassing, the tube was sealed off from the Veeco system and the pressure rose temporarily to 3×10^{-4} torr. The tube was then placed in the coil of an RF induction heater and the first getter evaporated. The pressure dropped to 1×10^{-7} . Getter No. 2 was then fired and the pressure dropped to 4×10^{-8} . The end of the tube was immersed in liquid nitrogen and



the pressure dropped to 2.4×10^{-8} . The Ge and substrate were then heated by positioning the RF coil between them, and the RF power raised gradually. The pressure rose temporarily at each increase of power until evaporation started. Temperatures were estimated by eye. The substrate heater reached 800° while the Ge was at 900° so the coil was moved and power increased until the Ge melted while the substrate heater was at about 600° . Evaporation continued for 10 minutes at a pressure of 3 to 4×10^{-6} . On cooling, the pressure was 3×10^{-8} and rose only to 4×10^{-8} on removing the liquid nitrogen. The deposit was polycrystalline.

In run No. 2, two grams of activated charcoal were inserted in place of the getters and a stainless steel optical baffle was placed between the Ge and the outlet to the Veeco system. The small system was outgassed at 650° (thermocouple outside the silica tube) for 3 days and the attached gauge at 420° for a short time until the pressure dropped to 3×10^{-4} . During seal off the pressure rose to over 10^{-3} but dropped, on cooling to room temperature, to 4×10^{-9} . The pressure read 6×10^{-6} as deposition started but rose to over 1×10^{-3} by the end of the deposition and remained above 10^{-3} for several hours after the power was turned off. The deposit was single crystal (Figure 4) with the familiar diffuse X.

In run No. 3, mica baffles were inserted between 1 gram of carbon, the



evaporation framework, and the attached vacuum gauge. The carbon was outgassed at 820° while the Ge near the end of the tube furnace remained somewhat cooler. The gauge was outgassed at 450° . The pressure before seal off was 2×10^{-4} with everything hot but, on shifting the tube in order to get at the intended seal off site, the pressure went over 10^{-3} so it was shifted back until the pressure dropped to 6×10^{-4} and sealed off with the usual pressure rise to over 10^{-3} . On cooling the pressure dropped to 4×10^{-9} . The mica had dehydrated and may have acted as a gas reservoir. The substrate heater was preheated to 900° C and deposition carried out with the substrate heater at about 750° and the pressure reading about 2×10^{-5} . The next morning the pressure was 1.6×10^{-6} and the deposit was polycrystalline.

In run No. 4, Vycor was used for baffles and Ti as the getter. The tube was outgassed at 700° - 795° for 6 hours and at 795° for 16 more hours, the pressure dropping to 4×10^{-6} . The system was then tipped off. On cooling the pressure dropped to 5×10^{-9} . During firing of the Ti getter, the pressure rose temporarily to 6×10^{-6} as the silica tube near the getter got red hot. The pressure then settled at 2×10^{-8} . During evaporation the pressure could not be read because of electrical interference but was 5×10^{-8} immediately after the RF was turned off and 6×10^{-9} when the system had cooled down. The deposit was polycrystalline.



In run No. 5, no getter was used. The system was outgassed at 750° C for about 100 hours and tipped off. The pressure was 4×10^{-9} but rose to 10^{-6} when the silica tube was flame heated to about 800° C. After cooling, the pressure settled at 2×10^{-8} . During deposition the pressure was not read but after deposition it dropped, as cooling proceeded, from 1.5×10^{-7} to 2×10^{-8} . The deposit was partly polycrystalline but there was no detectable diffuse X.

We shall repeat the depositions in a dynamic 10^{-8} vacuum system which Vactite has promised to make available.

2.5 Barrier diodes and field effect transistors were constructed by Dr. Macha and Mr. Sun using $\text{Ge}/\text{Al}_2\text{O}_3$ and Ge/CaF_2 . The work will be reported in detail in QR 3 of our contract NObsr 87634 with the Bureau of Ships and demonstrates that active devices can be made with polycrystalline films of Ge or Si.

2.6 No deposits of GaAs on BeO were made but will be when an adequate supply of BeO crystals is obtained. CeO_2 crystal growth will be attempted by the Société Générale Métallurgique de Hoboken, Belgium.

We are able to deposit single crystal GaAs on mica, though with some twinning and polycrystalline material, (Figure 5), but mica seems an unsatisfactory substrate because it is mechanically liable to delamination



(Figure 6) so the approach has not been pursued.

2.7 Electron microscope stereophotographs made of replicas of GaAs film surfaces demonstrated that replication with polystyrene did not show enough detail and may well have introduced artifacts due to local stretching of the polystyrene. Replications were made with evaporated carbon films on pre-shadowed surfaces. The replicas were removed by floating on $\text{HF-HNO}_3\text{-H}_2\text{O}$ which dissolves the GaAs. Shadowing has been done with Pt and with tungsten. Of four methods of evaporating platinum, soaking graphite rod in chloroplatinic acid, wrapping a graphite rod with Pt wire, evaporating Pt from between parallel, touching W wires, and evaporating Pt-C pellets between C electrodes, the last has given the thickest films and therefore best results so far, but efforts to improve the wrapped wire method will be made. Tungsten as shadowing material may be sublimed just below the melting point. A wire with a short thinned segment burned out before much W evaporated but a method using massive heat sinks consisting of pairs of half inch square copper bars two centimeters apart to clamp a 20 mil wire (see R. G. Hart, Journal of Applied Physics, Vol. 34, p. 434, 1963) worked well if the temperature of the wire was kept just below the melting point. This was done by continually adjusting the voltage to keep the space current from the hot W to the ground of the vacuum system at a few microamperes.



3.0 PLANS FOR THE SIXTH PERIOD

- 3.1 To deposit films in a dynamic 10^{-8} vacuum system.
- 3.2 To investigate annealing of GaAs films.
- 3.3 To make Hall measurements at a series of temperatures.
- 3.4 To investigate deposition at microns per second.
- 3.5 To improve the resolution in electron microscope surface studies and to investigate a setup for etching thicker films down to thicknesses suitable for electron microscopy.
- 3.6 To continue device studies with films of available quality.



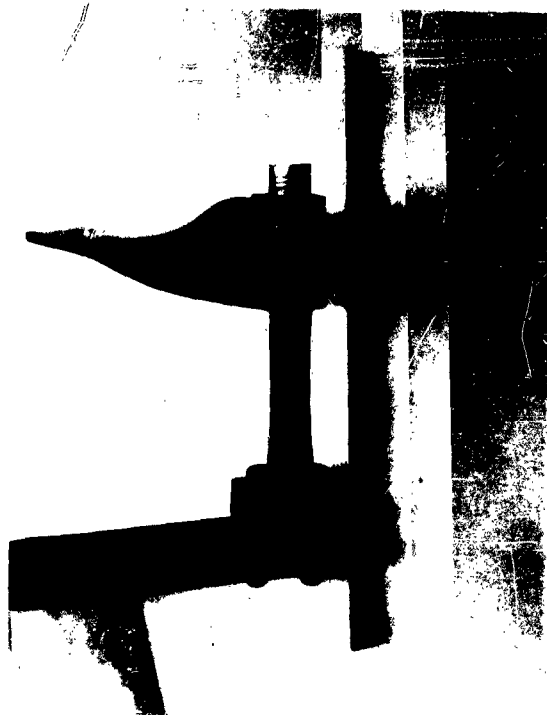


FIGURE 1

Vertical flash evaporation source

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FIGURE 2

Sample structures for Hall and resistivity measurements

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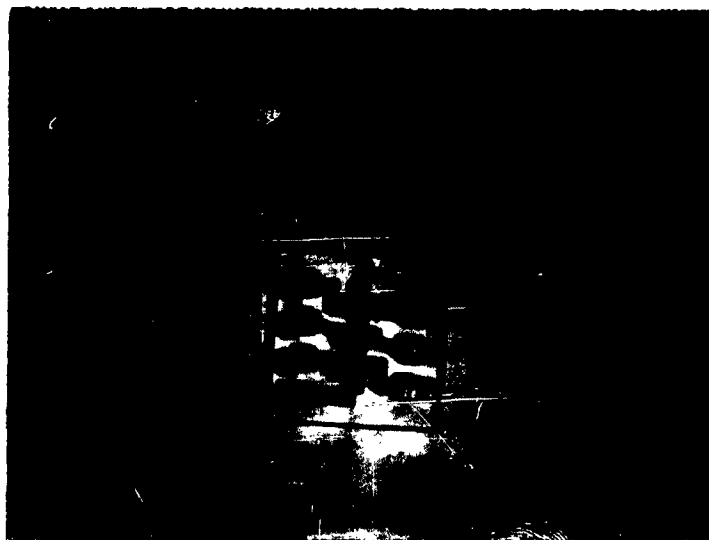


FIGURE 2

Sample structures for Hall and resistivity measurements

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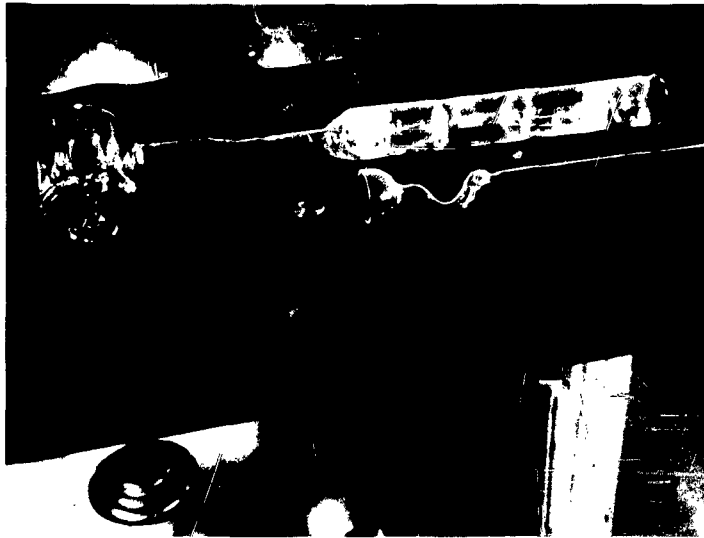


FIGURE 3

Glass system for deposition in higher vacuum

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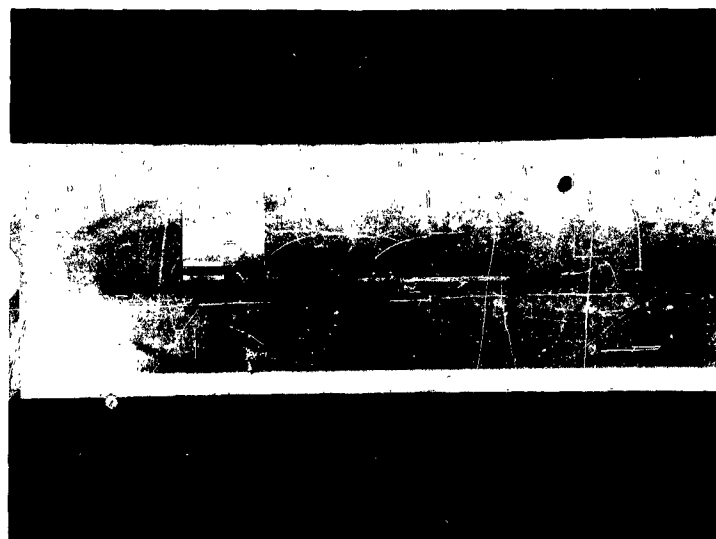


FIGURE 4

Glancing angle oscillation x-ray diffraction pattern of Ge/CaF_2 deposited in a system like that in Figure 3 (UHV-2, film 1710)



FIGURE 5

Glancing angle oscillation x-ray diffraction pattern of GaAs/mica (J557 A, film 1681)



FIGURE 6

Ga As/mica, J557A, 12 X

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